August 28, 2020

Skunk Camp
Water Quality Monitoring Program

PINAL AND GILA COUNTIES, ARIZONA

Prepared for:

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Contents

Tables

Figures

Appendices

1 INTRODUCTION

Montgomery & Associates (M&A) has prepared this Water Quality Monitoring Program on behalf of Resolution Copper (RC) for groundwater and surface water monitoring of the Skunk Camp site investigation area. The program (Program) has been developed to characterize and monitor groundwater and surface water chemistry upgradient, downgradient, and in the vicinity of the proposed Skunk Camp Tailings Storage Facility (TSF).

This document details the Skunk Camp monitoring network and the procedures for sample collection and analysis. The monitoring network includes wells, springs, and surface water locations situated within the Upper Mineral Creek and Dripping Spring Wash watersheds, and along the Gila River.

Selection of monitoring locations was determined based on development of a conceptual hydrogeologic model for the Skunk Camp site, and fate and transport modeling of seepage from the proposed TSF (M&A 2020). Initial water quality sampling has been conducted at most locations included in this program, as outlined in the Skunk Camp Sampling and Analysis Plan (M&A, 2019); however, six additional wells and three additional springs are incorporated into the Program beginning in 2020.

The sampling and monitoring plan provides for ongoing construction and operational monitoring of groundwater and surface water quality upgradient and downgradient from the proposed TSF. A final sampling and monitoring plan will be completed by the Arizona Department of Environmental Quality (ADEQ) after the completion and issuance of a final Aquifer Protection Permit (APP) for the Skunk Camp TSF. Per the APP, compliance with water quality standards will be a condition of continued construction and operation of the TSF. As such, the final locations and composition of the monitoring network discussed herein may be adjusted.

This document establishes the schedule and list of chemical analytes to be collected during three phases of mine activities:

1. Pre-construction—the primary objective during this phase is to continue baseline water quality sampling and characterize water chemistry at the site. A minimum of four (4) quarterly samples will be collected from upgradient spring locations for site characterization, and a minimum of

ten (10) quarterly samples will be collected from wells and downgradient springs in support of the APP.

2. Construction and Operation—water quality monitoring at groundwater and surface water locations will continue on a quarterly basis, with removal of sites within the proposed TSF footprint from the Program.

3. Post-Closure—water quality monitoring at groundwater and surface water locations will continue on a semi-annual basis.

Quarterly monitor reports will be submitted by RC to the Arizona Department of Environmental Quality (ADEQ) as required to support the APP application, and will continue through construction and operations to document compliance with water quality standards.

ADEQ will have regulatory oversight of the APP program; consequently, monitoring locations and sampling schedules will be subject to review, modification, and/or addition by ADEQ in support of APP issuance for the facility. In some instances, final well monitoring locations may depend on authorizations from landowners.

2 WATER QUALITY MONITORING PROGRAM

The following sections describe the Skunk Camp monitoring network and proposed Program schedule.

2.1 Monitoring Network

The monitoring network includes wells, springs, and surface water locations that are intended to document baseline water chemistry conditions prior to the development of a TSF, and be used for ongoing water quality monitoring through post-closure. In total, the monitoring network comprises 44 monitoring locations consisting of 17 springs, and surface water sites and 27 wells. Proposed sampling locations are listed in **Table 1** and shown on **Figure 1**.

Between November 2018 and July 2019, Resolution Copper installed 13 pumping and monitoring wells at Skunk Camp. The wells, designated as "RC wells", are constructed of steel and PVC blank and screened casing that vary from four to six inches in diameter. A detailed description of lithologic data, construction, and testing results for the wells are included in the Skunk Camp Site Investigation report (KCB, 2019). Based on well location and construction characteristics, ten of the wells were selected for inclusion in the sampling Program.

In early 2020, RC installed an additional seven monitoring wells, including two dual-completion wells. The dual-completion wells consist of two 2-inch PVC wells installed in the same drilling bore, with a shallow well completed in Quaternary alluvium (Qal) and a deeper well completed in Tertiary Gila Conglomerate (Tcg). The remaining five wells consist of 4-inch diameter blank and screened PVC casing. Drilling, construction, and testing results for the wells are described in detail in the Summary of Results for 2020 Site Investigations report (M&A, 2020a). Of the seven wells completed in 2020, three wells—including the shallow and deep completions of both dual completion wells—have been added to the Program. RC wells that are included in the Program are listed in **Table 1** and shown on **Figure 1**.

Nine private regional wells have been selected for inclusion in the Program (**Table 1**). The regional wells are primarily used for domestic and livestock water supply. Prior to the start of the Program, the wells were inspected for access, operation, and suitability. Seven of the wells were sampled for preliminary water chemistry screening prior to the start of the Program (**Table 1**).

Springs and surface water sampling locations along the Gila River and Mineral Creek will be used to assess surface water quality upgradient and downgradient from the proposed tailings area. With the addition of Big Spring to the Program, a total of 16 springs and surface water sites have been identified for water quality monitoring. The 16 sampling locations are listed in **Table 1** and shown on **Figure 1**.

Additional surface locations where surface water runs onto the area of the proposed TSF footprint following precipitation may be sampled opportunistically during the Program. Water chemistry data from run-off samples in the vicinity of the TSF may be useful for geochemical simulations of initial chemistry conditions.

2.2 Groundwater and Surface Water Monitoring Program

The groundwater and surface water monitoring Program consists of quarterly sample collection for a proposed duration of ten consecutive quarters (2.5 years). The proposed Program schedule is based on the recommendation of implementing a quarterly sampling frequency for APP data collection objectives. Ten sampling rounds are recommended for monitoring locations that are considered relevant to APP (**Table 1**). For locations that are not considered relevant to the APP, four quarterly samples are recommended to characterize seasonal variability.

Groundwater sampling of wells will be conducted with the use of submersible pumps and HydraSleeve samplers. Sampling methods selected for each well are presented in **Table 2** and are described in more detail in Section 3 of this document. The surface water monitoring program includes visits to springs and selected surface water locations. Samples will be collected at springs and surface water sites when water is present.

2.2.1 Monitoring Constituents

Analytical constituents for groundwater and surface water samples are detailed in **Table 3**. Analytes include common, trace metal, radiological constituents, stable isotopes, and radiogenic isotopes. Each water quality sample will be analyzed at a minimum for common, trace, and radiological constituents. At selected wells and surface water locations, samples will be analyzed for stable isotope analyses and/or radiogenic isotopes. The analyte suites and sampling schedule for each monitoring location is given in **Table 1**.

Routine field water quality parameters including temperature, pH, electrical conductivity (EC), and turbidity will be measured at each location prior to sampling. At well locations, groundwater levels will be measured prior to purging or sampling activities, and at the time of sample collection.

2.3 Monitoring Program Schedule

2.3.1 Pre-construction Phase

Prior to construction of the TSF, samples will be collected at wells, springs, and surface water locations in order to continue gathering baseline water quality data throughout the Skunk Camp study area. Baseline monitoring activities began in 2018 and are scheduled to continue through 2022. Monitoring during pre-construction includes a minimum of ten quarterly samples at wells, and a minimum of four quarterly samples at springs and surface water locations. Water quality at the Gila River will include 16 samples through the end of 2022. The proposed monitoring schedule is shown in **Table 1.**

2.3.2 Construction and Operation Phases

Water quality monitoring is scheduled to continue on a quarterly basis during the construction and operation of the mine, as indicated in **Table 1**. RC and regional wells that are located within the proposed TSF footprint are assumed to be covered by the facility during this phase, and have been removed from the monitoring Program. In addition, monitoring of springs and surface water locations upgradient of the facility that will not be impacted by mining activities are not included in the monitoring schedule during this phase, however additional samples may be collected if required.

2.3.3 Post-Closure Phase

Ongoing water quality monitoring during post-closure of the mine is scheduled on a semiannual basis at well, spring, and surface water locations that will be monitored during the construction and operation phases (**Table 1**).

3 WATER QUALITY MONITORING PROCEDURES

Water quality monitoring will be performed by qualified and trained personnel. Procedures for data acquisition, quality assurance/quality control (QA/QC), groundwater level measurement, groundwater sampling, spring and surface water sampling, quality assurance sampling, and sample control are described in the following sections.

3.1 Quality Assurance

Groundwater sampling and analysis will be conducted in a manner that is consistent with general field operating procedures and standard operating procedures (SOP) for groundwater sampling (**Appendix A**) and RC quality assurance / quality control (QA/QC) procedures (**Appendix B**) (M&A, 2017). These documents describe the duties of personnel involved with data collection and establish sampling and analytical protocols and documentation requirements to ensure the groundwater and surface water monitoring data are collected, reviewed, and analyzed in a consistent manner. These appendices also include data quality objectives for sampling procedures, sample and document custody procedures, laboratory analytical methods, internal quality control checks, data validation and reporting procedures, and corrective action procedures.

QA/QC procedures will be conducted in the field and laboratory. Field procedures will comprise field documentation, blind code labeling, and collection of quality control samples, including sample duplicates, field sampling blanks, and sampling equipment rinsate blanks. Laboratory QA/QC procedures will comprise achievement of laboratory performance criteria, including sample holding times, matrix spike/matrix spike duplicate recoveries, and laboratory method blank results.

3.2 Groundwater Level Measurement

Water levels will be measured in wells prior to purging or sampling, and at the time of sample collection. Construction details and any previous measurements for each well will be reviewed by the field staff before obtaining measurements. The field data sheet for recording water level measurements is included in **Appendix C**.

3.2.1 Materials and Equipment

The following materials and equipment are needed to measure water levels and well depth. All equipment that comes in contact with the well should be decontaminated prior to commencing field activities.

- Records of well construction details and previous measurements
- Electronic water level indicator with accuracy of 0.01 feet
- Water sampling record form (**Appendix C**)
- Weighted tape graduated to the nearest 0.01 feet

3.2.2 Measuring Point

Well depth and water level measurements will be referenced from the top of well vault centerline, determined by measuring from the vault lid cross bar or other flat object laid flat across the top of the well vault casing. RC has provided surveyed elevations of well vault tops for all RC wells.

3.2.3 Well Depth Measurements

The total depth of each new well will be measured with a weighted measuring tape immediately after construction and will be verified periodically thereafter. The weighted tape will be lowered into the well until the tape becomes slack indicating that it has encountered the bottom of the well. Care will be taken to lower the tape slowly to avoid damaging the tape or well bottom. The tape will be raised until it becomes taut, and with the tape in this position, the total depth of the well will be measured to the nearest 0.01 feet below the measuring point.

3.2.4 Water Level Measurements

Manual water level measurements will be obtained from wells with an electronic water level indicator prior to purging or sampling. The SOP for measuring water levels with an electronic water level indicator is summarized follows:

- Open the protective outer cover of the monitoring well and remove any debris that has accumulated around the riser near the well plug. If water is present above the top of the riser and well plug, remove the water prior to opening the well plug. Do not open the well until the water above the well head has been removed.
- Allow the well to equilibrate for at least one minute before measuring the water level.

- Using an electronic water level indicator accurate to 0.01 feet, determine the distance between the established measuring point and the surface of the standing water present in the well. Repeat as necessary until two successive readings agree to within 0.01 feet. Record date and time of each water level measurement and the serial number of the water level indicator used.

Additionally, if feasible a water level measurement will be collected at the time of sample collection. This measurement provides additional information regarding the conditions at the time of the sample, which may influence interpretations of water quality data.

The accuracy of electronic water level indicators will be verified at least annually as part of routine maintenance. The entire length of the graduated tape/cable will be compared to a steel surveyor's tape of the same or greater length to determine accuracy at 100-foot increments. Water level indicators will be checked more frequently if there is reason to suspect the tape/cable was damaged or altered during field operations.

3.3 Groundwater Sample Collection

The following sections provide the procedures to be utilized during the collection of groundwater samples from monitoring wells.

3.3.1 Field Instrument Calibration

At the beginning of each day of sampling, field instruments will be calibrated following manufacturer's recommended procedures using known, standard solutions. **Appendix E** includes the manuals and procedures for field instruments. Calibration procedures, date, and time will be recorded on equipment calibration data sheets (**Appendix C**). Back-up instruments will be available in case of malfunction. Instrument maintenance will be performed as deemed appropriate by the manufacturer (**Appendix E**).

3.3.2 Groundwater Sampling Methods

General methods and procedures to be used during groundwater sample collection are presented in the groundwater sampling SOP (**Appendix A**). The groundwater samples will be collected using dedicated or portable sampling pumps (standard three bore volume purge) or HydraSleeve™ samplers (no purge).

For samples collected following purging with pumps, the wells should be purged a minimum of three times the bore volume of standing water, or until EC, temperature, and pH field parameters stabilize. The bore volume of water present in each well shall be computed based on the length of water column in the well casing and include the volume of water in the filter pack. The casing water volume can be calculated using the following formula:

$$\mathbf{V_c = (0.041)d_c^{\wedge 2} \times h}$$

Where: V_c = casing water volume in gallons, d_c = casing diameter in inches, h = height of the water column from the well bottom in feet.

The filter pack water volume shall be computed using the following formula:

$$\mathbf{V_f = [((0.041)d_b^{\wedge 2} \times l) - ((0.041)d_c^{\wedge 2} \times l)] \times 0.28}$$

Where: V_f = volume in gallons, d_b = bore diameter in inches, d_c = casing diameter in inches, l = length of saturated filter pack in feet, and 0.28 = approximate filter pack porosity.

The total bore volume of water is then computed as $\mathbf{V_c + V_f}$.

Wells will be purged through dedicated discharge tubing at wells where dedicated equipment is installed. New syringes and 45-micron filters will be used for collection of samples that will be analyzed for dissolved metals and stable isotopes. At wells where portable sampling equipment is used, the pumps and discharge tubing will be decontaminated prior to each sampling event. Decontamination procedures are described in **Section 3.5.2** below.

During purging, field parameters will be monitored and recorded on water sampling record forms (**Appendix C**). Temperature, pH, and EC will be measured using a Myron L Ultrameter II instrument or equivalent. Turbidity will be measured using a Hach 2100Q turbidimeter or equivalent. Dissolved oxygen (DO) and oxidation-reduction potential (ORP) may be measured as requested by RC, using a YSI Professional Plus instrument with an attached flow-through cell. Standard operating procedures for field instruments are included in **Appendix E**.

Most groundwater samples will be collected using dedicated and portable pumps after three borehole volumes are purged and field parameters have stabilized to within ±0.1 standard units for pH, ±3 percent for EC and temperature,

±10 millivolts (mv) for ORP, and ±10 percent for turbidity and DO. Stabilization of field parameters will be recognized when a minimum of three consecutive measurements collected with at least three minutes between measurements meet the stabilization criteria. Field sampling data will be recorded on water sampling record form (**Appendix C**).

For wells that do not have adequate water level recovery to sample within 24 hours of purging, HydraSleeve samplers may be used. The HydraSleeve sampling method includes the use of disposable HydraSleeve samplers that are deployed at the screened interval midpoint of the well. Sampling personnel will record the date and time of HydraSleeve deployment and retrieval on the water sampling record form, in addition to the water level prior to deployment and prior to retrieval of the HydraSleeve. Typically, the HydraSleeve will remain in the well for approximately three months until the next sampling round before being retrieved. After retrieval of the HydraSleeve, a new sampler will be deployed for the following sampling round. Advantages and limitations of HydraSleeves are described by Interstate Technology & Regulatory Council (ITRC, 2007). Detailed standard operating procedures for HydraSleeve sample collection methods are provided in **Appendix E**.

The only well currently planned for HydraSleeve sampling is RC19-10 (**Figure 1, Table 2**). Based on preliminary temporal dependence analysis conducted for well RC19-10, sampling events at RC19-10 should be spaced at approximately 2.5 years. **Table 1** shows samples planned for RC19-10 during the first three years of the Program in Q4 of 2019 and Q1 of 2022.

3.4 Spring and Surface Water Sample Collection

Samples collected from springs and surface water locations will require the same field instrument calibration procedures previously outlined in **Section 3.3.1**.

Springs will be documented with photographs or videos during sampling visits, whether or not water is present. For springs with identifiable flow during visits, field parameters will be recorded on water sampling field forms (**Appendix C**) and a sample will be collected. Field parameters will be measured using flowing water near the spring source, and will include temperature, pH, EC, and turbidity. The field parameters will be collected with a Myron L Ultrameter II and Hach 2100Q, or equivalent instrumentation. Field parameters will be measured and recorded a minimum of three times over no less than five minutes to verify

consistency between readings. Once three sets of field parameters have been recorded and determined to be consistent, a sample will be collected. For springs with standing water but no identifiable flow during visits, field parameters will be recorded but no sample will be collected.

Spring samples will be collected using flowing water near the spring source (first emergence of groundwater). The sampling locations at each site will be noted on field data forms and remain consistent between sampling events to the extent that is feasible given the variability of climatic conditions. Care will be given to minimize the amount of turbidity and organic matter collected in samples.

Surface water sites will be documented with photographs and videos during scheduled sample visits. As with spring samples, field parameters will be measured and recorded a minimum of three times over five minutes to verify consistency before a sample is collected. Field parameters will include temperature, pH, EC, and turbidity collected with a Myron L Ultrameter II and Hach 2100Q, or equivalent instrumentation.

Surface water sampling will be conducted from dry ground using a long handle pole and dipper, with samples collected just below the water surface. Sampling personnel are instructed to wear rubber boots and life vests to approach muddy areas surrounding surface water sampling locations. This sampling procedure reduces the safety risk of flowing water to sampling personnel.

Surface water sampling methods and locations will remain as consistent as possible between sampling events. Sampling locations will not be chosen in or around upstream eddies, where water can become stagnant. In addition, care will be given to minimize the amount of turbidity and organic matter collected in surface water samples.

3.5 Quality Assurance Sample Collection

QA/QC samples will be collected in the field to evaluate laboratory and field precision and the effectiveness of sampling equipment decontamination. QA/QC sampling will require the same field instrument calibration procedures previously outlined (**Section 3.3.1**), and will be conducted in accordance with the general RC QA/QC procedures (**Appendix B**). QA/QC samples will consist of duplicate samples, field blanks, and equipment rinsate blanks, and will be clearly identified on field sampling forms.

Duplicate Samples

Duplicate water quality samples will be collected at a frequency of 10 percent of the total number of samples collected during monitoring events. Specific locations will be strategically selected for duplicate samples during planning and coordination of the monitoring Program. The duplicate samples will be collected at the same locations as the corresponding primary samples, and immediately following collection of primary samples using identical sampling techniques. Duplicate samples will be treated in an identical manner as the primary samples during storage, transportation, and analysis. The duplicate sample containers will be assigned an identification number in the field so that they cannot be identified (blind duplicate) as duplicate samples by laboratory personnel performing the analysis.

Field Blanks

Field blank samples will be collected to assess the effectiveness of sampling practices or impacts by ambient conditions at a frequency of 10 percent of the total number of samples collected during monitoring events. Field blank samples will be prepared by filling sample containers with deionized water in the field using the same methods used for normal sampling operations. Field blanks will include a dissolved metals analysis of deionized water that has passed through a new syringe. The syringe will be discarded after use and will not reused for any environmental samples. The water will be collected and transported to the laboratory for analysis for common, trace, and radiological constituents.

Equipment Rinsate Blanks

In order to assess the effectiveness of equipment decontamination procedures, a minimum of one equipment blank will be collected during each monitoring event where decontamination of sampling equipment is required (once per quarter). Equipment blanks will be prepared by pouring reagent-grade de-ionized water over or through decontaminated field sampling equipment prior to the collection of environmental samples. The water will be collected and transported to the laboratory for the equivalent analysis as the primary samples. Use of portable sampling equipment is only anticipated at well RC19-8C.

3.5.1 Sample Designation and Labeling

All samples submitted for analysis, including primary samples, duplicate samples, and equipment rinsate blanks, will be given a unique, blind, RESH sample identifier. Sample identifiers will be recorded on field sampling data sheets.

Sample containers will be labeled with the sample identifier, date and time of sampling, and sampler's initials. Blind sample identifiers will be paired to sample identifiers on field sampling data. A similar naming protocol will be used for all quality control sampling.

3.5.2 Equipment Decontamination Procedures

Before use at each location, the submersible pumps and depth to water sensors will be washed using a solution of water and Liquinox (or other laboratory-grade cleaner) and water, then rinsed with potable water, and finally rinsed a second time with distilled/deionized water. Polyethylene tubing will be decontaminated after each well is sampled by pumping a solution of water and Liquinox through the pump and tubing, followed by potable water, and finally distilled/deionized water. All decontamination will be performed on new polyethylene sheets to avoid contact with the ground. Sampling personnel will use new, disposable gloves at each well location.

3.6 Sample Control

3.6.1 Sample Containers/Sample Handling

The sample containers will be prepared and provided by the analytical laboratory. Samples will be preserved consistent with requirements of the analytical methods presented in **Table 3**. Preservatives will be added to the sampling containers in the field at the time of sampling. Expiration dates of the preservatives will be inspected prior to use and no expired preservatives shall be used. The type and size of container used for each parameter and the type of preservative added by the sampler, if any, will be recorded on the field sampling data form (**Appendix C**). Sample containers will be placed in an iced cooler immediately after sample collection. The sample containers will be kept tightly closed, maintained under custody, and continuously chilled until analysis. Maximum holding times from the time of sample collection until sample analysis are provided in **Table 3**.

3.6.2 Sample Custody

At the end of each sampling day and before samples are transferred off site, sample information will be documented on Chain of Custody (COC) forms. The forms used for each laboratory involved in the Program are provided in

Appendix D. Once samples are collected, they will remain in the custody of the sampler or other authorized personnel until they are shipped to the laboratory. Upon transfer of sample possession to subsequent custodians, the persons transferring custody will sign the COC form.

During transport, the COC form will be placed in a resealable plastic bag and accompany each sample cooler to the laboratory. Signed and dated COC seals will be placed on coolers prior to shipping. When the samples are received at the laboratory, the custody seal on the cooler will be broken and the condition of the samples recorded by the laboratory custodian. COC records will be included in the analytical report prepared by each laboratory.

Upon receipt of the samples, the laboratory will complete the COC record. The condition of each sample container will be noted. The laboratory will also maintain a sample-tracking record that will follow each sample through the laboratory process. The sample-tracking record must show the dates of sample collection and sample analysis for each sample.

3.6.3 Packaging and Shipping

Samples will be shipped to the analytical laboratory by overnight delivery. Samples will be packaged and shipped using the following procedures:

- Sample containers will be placed in resealable plastic bags in a sealed, insulated cooler. A sufficient amount of ice will be placed around the samples to ensure that samples will remain chilled throughout shipment.
- If used, glass bottles will be separated in the cooler by shock-absorbent packaging material to prevent breakage.
- Sample shipments will be accompanied by a COC form, which will be sealed in a plastic bag and placed inside each cooler.

3.7 Laboratory Analysis

Water quality samples will be submitted for hydrochemical analysis to analytical laboratories selected by RC. Laboratory analyses will be performed using EPA approved methods. Analytical methods for required analyses are summarized in **Table 3**.

At a minimum, samples will be analyzed for common, trace metal, and radiological constituents using the methodologies specified in **Table 3**.

When laboratory reports are received, data will be verified in accordance with the RC QA/QC procedures (**Appendix B**) by verifying that all internal performance standards and measures established by the laboratory have been achieved. Blank and duplicate samples will be checked. For each well, cations and anions will be evaluated to ensure that the balance is within 20 percent. Following the first sampling round, results for specific wells will be compared with previous monitoring data for consistency with historic values or observed trends. Questions that arise during the validation process will be addressed to the laboratory and may require reanalysis or resampling. Once verified, sample results will be incorporated into the site water quality database.

4 REFERENCES CITED

KCB Consultants Ltd., 2019, Skunk Camp Site Investigation, report prepared for Resolution Copper Mining LLC, November 1, 2019.

Montgomery & Associates, 2017, Summary of Quality Assurance/Quality Control Procedures for Water Chemistry Data, technical memorandum prepared for Resolution Copper Mining LLC, July 5, 2017.

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Montgomery & Associates, 2020, Conceptual Hydrogeologic Model: Skunk Camp Tailings Storage Facility Alternative, report prepared for Resolution Copper Mining LLC, June 29, 2020.

Montgomery & Associates, 2020a, Summary of Results for 2020 Site Investigations at the Skunk Camp Tailings Storage Facility Site, report prepared for Resolution Copper Mining LLC, July 3, 2020.

ITRC, 2007, Protocol for Use of Five Passive Samplers to Sample for a Variety of Contaminants in Groundwater, February 2007.

TABLE 1. MONITORING LOCATIONS AND SCHEDULE
SKUNK CAMP WATER QUALITY MONITORING PROGRAM

MONITORING TYPE & LOCATION		BASELINE & CHARACTERIZATION DATA																												MONITORING		LAND OWNERSHIP	OBJECTIVES
		2018								2019									2020				2021				2022				Sample Count end of 2022	Construction & Operations	Post-Closure		
		M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4					
RC WELLS	RC-POC (proposed)																													-	quarterly	quarterly	Resolution Copper	Completed to monitor uppermost aquifer
	RC19-3												D				T		X	X	X	X	X	X		X	X				10			Resolution Copper	APP background of western TSF
	RC18-4								D										X	X	X	X	X	X	X	X	X				10	quarterly	semiannually	Resolution Copper	Background Mineral Creek watershed, monitoring
	RC19-7														D	2T			X	X	X	X	X	X		X	X				10			Resolution Copper	APP background, understand fault chemistry
	RC19-8									D	T	T							X	X	X	X		X	X	X	X				11			Resolution Copper	Upper 250 feet of Tg (just below RC19-8B)
	RC19-8B													D			T		X	X	X	X	X	X		X	X				10			Resolution Copper	Upper 50 ft of Tg
	RC19-8C																		dry	X	X	X	X	X	X	X	X	X				10			Resolution Copper	Qal location
	RC18-9								D	2T									X	X	X	X	X		X	X	X				10	quarterly	semiannually	Resolution Copper	APP background upgradient well, monitoring
	RC19-10													D					X								X				2	every 2.5 yrs	every 2.5 yrs	Resolution Copper	APP background upgradient well (Hydrasleeve), monitoring
	RC19-13										D				T				X	X	X	X	X		X	X	X				10			Resolution Copper	APP background northern TSF
	RC19-15															D	T		X	X	X	X	X	X		X	X				10	quarterly	semiannually	Resolution Copper	APP background upgradient well, monitoring
	RC20-2B(S)[1]																					X	X	X	X	X	X	X	X			10	quarterly	semiannually	Resolution Copper	Downgradient Qal monitoring
	RC20-2B(D)[2]																					X	X	X	X	X	X	X	X			10	quarterly	semiannually	Resolution Copper	Downgradient Tcg monitoring
	RC20-2D																					X	X	X	X	X	X	X	X			10	quarterly	semiannually	Resolution Copper	APP background, understand fault chemistry, monitoring
	RC20-18(S)[1]																					X	X	X	X	X	X	X	X			10	quarterly	semiannually	Resolution Copper	Downgradient Qal monitoring
	RC20-18(D)[2]																					X	X	X	X	X	X	X	X			10	quarterly	semiannually	Resolution Copper	Downgradient Tcg monitoring
REGIONAL WELLS	55-808287																X		X	X	X	X	X	X		X	X				10	quarterly	semiannually	Resolution Copper	Background downgradient, monitoring
	35-17570																X		X	X	X	X	X	X		X	X				10	quarterly	semiannually	Harris	Background downgradient, monitoring
	55-632794									X									X	X	X	X		X	X	X	X				10			Resolution Copper	Background northern TSF, possible fault connectivity
	55-807702																X		X	X	X	X	X	X		X	X				10	quarterly	semiannually	Resolution Copper	Background downgradient, monitoring
	55-632793									X									X	X	X	X		X	X	X	X				10			Resolution Copper	Background Mineral Creek watershed
	55-632800																		X	X	X	X	X	X	X	X	X				10	quarterly	semiannually	Resolution Copper	Possible contamination from livestock, monitoring
	55-622471																				D,T	X	X	X	X	X	X	X	X			10	quarterly	semiannually	Resolution Copper	Downgradient monitoring
	55-205266							X											X	X	X	X	X	X		X	X				10			Resolution Copper	Background northern TSF, possible fault connectivity
	55-632797												X						X	X	X	X	X		X	X	X				10			Resolution Copper	Background northern TSF
	55-615260																X		X	X	X	X	X	X		X	X				10	quarterly	semiannually	AZ State Land Dept.	Upgradient Mineral Creek, monitoring
	55-502917															X			X	X	X	X	X	X		X	X				10	quarterly	semiannually	Wind Spirit Comm.	Background downgradient, monitoring
SPRINGS AND SURFACE WATER	Big Springs																			X		X	X	X	X	X	X	X	X	X	X	11	quarterly	semiannually	Resolution Copper	Downgradient of TSF, fault chemistry, monitoring
	Chimney Spring		X												X				X					X								5			Resolution Copper	Upgradient western TSF
	Elkins Spring																			X			X	X	X	X	X	X	X	X	X	10	quarterly	semiannually	Resolution Copper	Downgradient of TSF, near community, monitoring
	Government Springs																X		X		X			X								4			Gvt. Spr. Ranch	APP downgradient stream
	Haley Spring																		X		X			X		X						4			Resolution Copper	Upgradient western TSF
	Indian Spring														X				X					X		X						4			Resolution Copper	Upgradient western TSF
	Laguna Spring																		X		dry			X	X	X						4			AZ State Land Dept.	Downgradient Mineral Ck
	Looney Spring	X																	X					X		X						4			Resolution Copper	Upgradient western TSF
	Skunk Spring																		dry		dry			X	X	X	X					4			AZ State Land Dept.	Upgradient western TSF
	Stone Cabin Box Spring		X																X					X		X						4			Resolution Copper	Upgradient eastern TSF
	Stone Cabin Spring										X								X		X					X						4			Resolution Copper	Upgradient eastern TSF
	Sump Spring																X		X		X			X								4			AZ State Land Dept.	Downgradient Mineral Ck
	Upper Mineral Creek - MC 8.4C																		dry		X		X	X		X						4			Gvt. Spr. Ranch/AZ State	APP downgradient stream
	Walnut Spring									X									X		X					X						4			Resolution Copper	Upgradient Mineral Ck
	Well Spring																				X		X	X	X	X	X	X	X	X	X	10	quarterly	semiannually	Resolution Copper	Downgradient of TSF, monitoring
	Woodchopper Spring									X									X		X					X						4			Resolution Copper	Upgradient western TSF
	Gila River downstream of DSW							X					X					X	X	X	X	X	X	X	X	X	X	X	X	X	16	quarterly	semiannually	BLM or private	APP downgradient stream, monitoring

NOTES:

(S)[1] shallow completion of dual-completion well

(D)[2] deep completion of dual-completion well

LEGEND:

X COM/TR; RL (common, trace, and radiologicals)

X COM/TR; RL; ST; RG (all of the above + radiogenic isotopes)

D Development. COM/TR; RL (no low-level Hg; total U; total isotopic U)

T/T Test sample (analyte suite indicated by letter color)

scheduled sample not collected

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TABLE 2. SAMPLING METHODS FOR WELLS, SPRINGS, AND SURFACE WATER LOCATIONS
SKUNK CAMP WATER QUALITY MONITORING PROGRAM

TYPE	SAMPLING LOCATION	WELL TOTAL DEPTH (feet)	CASING NOMINAL DIAMETER (inches)	SCREENED INTERVAL (feet bls)	APPROX. WATER LEVEL (feet bls)	RECOMMENDED SAMPLING EQUIPMENT	SAMPLING METHOD	PUMP SAMPLING DEPTH (feet bls)	THREE BORE VOLUMES (gallons)	TARGET FLOW RATE (gpm)	ESTIMATED PURGE TIME (mins)	HYDROGEOLOGIC UNIT(S)	ARC. MONITOR REQUIRED?	COMMENTS
RC Wells	RC-POC (proposed)	TBD	TBD	TBD	TBD	TBD	TBD	TBD	TBD	TBD	TBD	TBD	-	Completed to monitor uppermost aquifer
	RC19-3	750	6	610 - 750	261	Dedicated pump: Grundfos 25S100-52D	Purge 3 volumes & stable parameters	370	2650	35	76	Gila Conglomerate (Tcg)	-	Requires towed generator (three phase)
	RC18-4	269	4	227-267	215	Dedicated pump: Grundfos 5SQ07-230	Purge 3 volumes & stable parameters	264	250	2.5	100	Gila Conglomerate (Tcg)	-	Requires towed generator (single phase)
	RC19-7	755	6	670 - 750	298	Dedicated pump: Grundfos 62S75-17	Purge 3 volumes & stable parameters	350	2290	50	46	Gila Conglomerate (Tcg)	-	Requires towed generator (three phase)
	RC19-8	250	6	148 - 168 208 - 248	67	Dedicated pump: Grundfos 25S20-11	Purge 3 volumes & stable parameters	146	1100	30	37	Gila Conglomerate (Tcg)	-	Requires towed generator (three phase)
	RC19-8B	130	6	85 - 125	70	Dedicated pump: Grundfos 10SQ05-160	Purge 3 volumes & stable parameters	116	390	5	78	Gila Conglomerate (Tcg)	-	Requires towed generator (single phase)
	RC19-8C	71	6	31 - 71	70	Portable pump: Mega Monsoon	Purge 3 volumes & stable parameters	70	variable	1	variable	Quaternary Alluvium (Qal)	-	To be monitored with pressure transducer and only sampled when enough water present in the alluvium (WL above ~ 67 ft bls)
	RC18-9	402	5	320 - 400	238	Dedicated pump: Grundfos 77S100-20	Purge 3 volumes & stable parameters	307	780	50	16	Gila Conglomerate (Tcg)	-	Requires towed generator (three phase)
	RC19-10	647	4	465 - 647	563	HydraSleeve	HydraSleeve	600 - 620	840	NA	NA	Gila Conglomerate (Tcg)	Yes	Stack of three hydrasleeves (4"- 2L) spread out over 20 feet; 2.5 years between sampling events based on temporal dependence analysis
	RC19-13	350	6	268 - 348	215	Dedicated pump: Grundfos 6SQF-3 (solar)	Purge 3 volumes & stable parameters	267	890	8	111	Gila Conglomerate (Tcg)	-	Solar power supply; may be pre-purged if used recently; sampler should try to maintain WL above screen
	RC19-15	985	6	595 - 975	410	Dedicated pump: Grundfos 62S100-22	Purge 3 volumes & stable parameters	450	0	50	0	Gila Conglomerate (Tcg)	-	Requires towed generator (three phase)
	RC20-2B(S)[1]	90	2	40 - 90	70	Dedicated pump: Grundfos Redi-Flo2	Purge 3 volumes & stable parameters	85	60	2	40	Quaternary Alluvium (Qal)	-	Requires 120V generator
	RC20-2B(D)[2]	155	2	115 - 155	115	Dedicated pump: Grundfos Redi-Flo2	Pump to dryness & sample recovered	150	100	2	50	Gila Conglomerate (Tcg)	-	Requires 120V generator
	RC20-2D	139	4	99 - 139	80	Dedicated pump: Grundfos Redi-Flo2	Purge 3 volumes & stable parameters	115	110	4	28	Fault (Dripping Spring)	-	Requires 120V generator
	RC20-18(S)[1]	91	2	41 - 91	90	Dedicated pump: Grundfos Redi-Flo2	Purge 3 volumes & stable parameters	91	10	0.5	20	Quaternary Alluvium (Qal)	-	Requires 120V generator
	RC20-18(D)[2]	130	2	110 - 130	89	Dedicated pump: Grundfos Redi-Flo2	Purge 3 volumes & stable parameters	110	60	2	30	Gila Conglomerate (Tcg)	-	Requires 120V generator
Regional Wells	55-808287	254	---	---	81	Permanent pump	Stable parameters	confirm pump depth	pre-purged (used daily)	---	NA	Gila Conglomerate (Tcg)	-	Well is pumped daily to fill domestic water supply tanks
	35-17570	125	TBD	TBD	77	Permanent pump	Stable parameters	confirm pump depth	pre-purged (used daily)	---	NA	Gila Conglomerate (Tcg)	-	Well owner has requested results for each sample
	55-632794	392	8	---	175	Permanent pump	Stable parameters	confirm pump depth	pre-purged (used daily)	---	NA	Gila Conglomerate (Tcg)	-	Solar-powered pump installed
	55-807702	310	6	285 - 310	65	Permanent pump	Stable Parameters	confirm pump depth	pre-purged (used daily)	---	NA	Gila Conglomerate (Tcg)	-	Recorded pump capacity is 20 gpm
	55-632793	200	8	---	51	Permanent pump	Purge 3 volumes & stable parameters	confirm pump depth	---	8	---	Gila Conglomerate (Tcg)	-	Coordinate generator with Morty; pumps at ~8 gpm
	55-632800	250	8	---	73	Permanent pump	Purge 3 volumes & stable parameters	confirm pump depth	---	4.5	---	Gila Conglomerate (Tcg)	-	Yanmar motor with crank that requires diesel; pumps at ~4.5 gpm
	55-622471	1475	14 - 16	260 - 600 615 - 1418	145	Permanent pump	Purge 3 volumes & stable parameters	589	35000	300	117	Gila Conglomerate (Tcg)	-	Notify and coordinate well use with RC and rancher
	55-205266	230	6	205 - 225	180	Permanent pump	Purge 3 volumes & stable parameters	confirm pump depth	---	5	---	Gila Conglomerate (Tcg)	Yes	Arc. Monitor required; solar-powered pump installed; pumps at ~5 gpm; WL draws down to pump after 1.5 - 2 hrs
	55-632797	300	6	---	164	Permanent pump	Purge 3 volumes & stable parameters	confirm pump depth	---	---	---	Gila Conglomerate (Tcg)	-	Well has pump installed that requires generator
	55-615260	150	---	---	46	Permanent pump	Purge 3 volumes & stable parameters	120	---	3.6	---	Qal (above Ya)	-	Solar-powered pump installed; pumps at ~3.6 gpm; discharge water into tanks during purge (per Mickey's request)
	55-502917	365	8	180 - 200 260 - 300 320 - 360	160	Permanent pump	Stable parameters	confirm pump depth	pre-purged (used daily)	---	NA	Gila Conglomerate (Tcg)	-	Used daily for domestic supply and irrigation. Coordinate to have pump run for a couple of hours prior to arrival
Springs and Surface Water	Big Springs	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Fault	-	Dripping Spring fault within Paleozoics
	Chimney Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Yds, Yd	-	rheocrene spring; probably persistant
	Elkins Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Tcg	-	hillslope spring; typically reliable flow
	Government Springs	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	---	-	requires coordination with Mickey, and ideally same sampler
	Haley Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Yds, Yd	-	evidence of seasonal flow
	Indian Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Yds, Yd	-	rheocrene spring; probably persistant
	Laguna Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	---	-	near Sump Spring
	Looney Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Yds, Ym (fault zone)	-	rheocrene spring; probably persistant
	Skunk Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Yds, Yd, Qal	-	may have seasonal water; has not been sampled
	Stone Cabin Box Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Qal (above Cb)	Yes	rheocrene spring; may have seasonal flow
	Stone Cabin Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Ym, Yd	Yes	rheocrene spring; probably persistant
	Sump Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	---	-	excavated sump near Laguna Spring
	Upper Mineral Creek - MC 8.4C	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	NA	-	upper Mineral Creek station MC 8.4C; often dry
	Walnut Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Yds, Yd	-	hillslope spring; reliable flow
	Well Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Qal, Cb	-	excavated sump below cement mixer container; probably seasonal flow
	Woodchopper Spring	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	Yds	-	rheocrene spring
	Gila River downstream of DSW	NA	NA	NA	NA	NA	Stable parameters - grab	NA	NA	NA	NA	NA	-	use sampling pole and cup; wear boots and do not enter water

NOTES:

(S)[1] shallow completion of dual-completion well
(D)[2] deep completion of dual-completion well
bls = below land surface
gpm = gallons per minute

TBD = to be determined
NA = not applicable
--- = information not available

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TABLE 3. ANALYTE SUITES, CONTRACT LABORATORIES, SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
SKUNK CAMP WATER QUALITY MONITORING PROGRAM

ANALYTE SUITES							
CATEGORY	**ANALYTICAL METHOD**	**PARAMETER**	**LABS**	**SAMPLE CONTAINERS**	**PRESERVATIVES**	**HOLDING TIMES**	**NOTES**
FIELD PARAMETERS	*Observation*	Electrical Conductivity (Field)	NA	NA	NA	NA	
	Observation	pH (Field)	NA	NA	NA	NA	
	Observation	Temperature (Field)	NA	NA	NA	NA	
	Observation	Turbidity (Field)	NA	NA	NA	NA	
	Observation	Dissolved Oxygen (Field)	NA	NA	NA	NA	For selected samples; groundwater samples will use flow-through cell
	Observation	ORP (Field)	NA	NA	NA	NA	For selected samples; groundwater samples will use flow-through cell
COMMON CONSTITUENTS (COM)	*SM 2320B*	Total Alkalinity (as CaCO3)	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	14 days	
	SM 2320B	Bicarbonate (Calculated by M&A)	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	14 days	
	SM 2320B	Bicarbonate Alkalinity (as CaCO3)	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	14 days	
	EPA 300.0	Bromide	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	28 days	
	EPA 200.7	Calcium	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	6 months	
	SM 2320B	Carbonate (Calculated by M&A)	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	14 days	
	SM 2320B	Carbonate Alkalinity (as CaCO3)	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	14 days	
	EPA 300.0	Chloride	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	28 days	
	EPA 300.0	Fluoride	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	28 days	
	SM 2340B	Hardness (as CaCO3)	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	6 months	
	EPA 200.7	Magnesium	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	6 months	
	EPA 353.2	Nitrate+Nitrite as N	SVL	250 mL - Nutrients	H2SO4 - 1.25 mL; **Keep On Ice**	28 days	
	SM 4500-H-B	pH (Laboratory)	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	15 minutes	
	EPA 200.7	Potassium	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	6 months	
	EPA 200.7	Silica	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	6 months	
	EPA 200.7	Sodium	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	6 months	
	EPA 120.1	Specific Conductance (Laboratory)	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	28 days	
	EPA 300.0	Sulfate	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	28 days	
	SM 4500-S-F	Sulfide	SVL	500 mL	NaOH+Zn Ac - 2 mL; **Keep On Ice**	7 days	
	SM 2540C	Total Dissolved Solids (Laboratory)	SVL	500 mL - Inorganic (General Chemistry)	No chemical preservative; **Keep On Ice**	7 days	

TABLE 3. ANALYTE SUITES, CONTRACT LABORATORIES, SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
SKUNK CAMP WATER QUALITY MONITORING PROGRAM

ANALYTE SUITES							
CATEGORY	**ANALYTICAL METHOD**	**PARAMETER**	**LABS**	**SAMPLE CONTAINERS**	**PRESERVATIVES**	**HOLDING TIMES**	**NOTES**
TRACE METALS (TR)	*EPA 200.7*	Aluminum	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.8	Antimony	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.8	Arsenic	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.7	Barium	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.7	Beryllium	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.7	Boron	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.8	Cadmium	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.7	Chromium	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.7	Cobalt	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.8	Copper	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 335.4	Cyanide, Total	SVL	250 mL	NaOH - 1.25 mL; **Keep On Ice**	14 days	
	EPA 200.7	Iron	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.8	Lead	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.7	Manganese	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	CVAA 245.7	Mercury	SVL (subcontracted to Anatek Labs)	Two 50 mL glass vials - Total Hg and Dissolved Hg	Both vials pre-preserved with HCl; **Keep On Ice; Filter in Field**	6 months	
	EPA 200.7	Molybdenum	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.7	Nickel	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.8	Selenium	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.8	Silver	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.8	Thallium	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.8	Vanadium	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	
	EPA 200.7	Zinc	SVL	500 mL - Total Metals and/or Dissolved Metals	HNO3 - 2.5 mL; **Keep On Ice; Filter in Field for Dissolved Metals**	6 months	

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TABLE 3. ANALYTE SUITES, CONTRACT LABORATORIES, SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
SKUNK CAMP WATER QUALITY MONITORING PROGRAM

ANALYTE SUITES							
CATEGORY	**ANALYTICAL METHOD**	**PARAMETER**	**LABS**	**SAMPLE CONTAINERS**	**PRESERVATIVES**	**HOLDING TIMES**	**NOTES**
RADIOLOGICALS (RL)	*EPA 900*	Gross Alpha	ACZ	4 L cube, 250 mL	HNO3; pre-preserved	6 months	Fill cube to ≥ 2/3 full and 250 mL to ≥ 50 mL
	Calculated	Gross Alpha, Adjusted	ACZ	4 L cube, 250 mL	HNO3; pre-preserved	6 months	Fill cube to ≥ 2/3 full and 250 mL to ≥ 50 mL
	EPA 900	Gross Beta	ACZ	4 L cube, 250 mL	HNO3; pre-preserved	6 months	Fill cube to ≥ 2/3 full and 250 mL to ≥ 50 mL
	EPA 903.1	Radium 226	ACZ	4 L cube, 250 mL	HNO3; pre-preserved	6 months	Fill cube to ≥ 2/3 full and 250 mL to ≥ 50 mL
	EPA 903.1	Radium 226 + Radium 228	ACZ	4 L cube, 250 mL	HNO3; pre-preserved	6 months	Fill cube to ≥ 2/3 full and 250 mL to ≥ 50 mL
	EPA 904	Radium 228	ACZ	4 L cube, 250 mL	HNO3; pre-preserved	6 months	Fill cube to ≥ 2/3 full and 250 mL to ≥ 50 mL
	EPA 200.8	Uranium	ACZ	4 L cube, 250 mL	HNO3; pre-preserved	6 months	Fill cube to ≥ 2/3 full and 250 mL to ≥ 50 mL
	EPA 200.8	Thorium, total	ACZ	4 L cube, 250 mL	HNO3; pre-preserved	6 months	Fill cube to ≥ 2/3 full and 250 mL to ≥ 50 mL
STABLE ISOTOPES (ST)	*Unknown*	Delta Carbon-13 of DIC	Beta Analytic	Only a few mls required when only sampling for Carbon-13. Suggest filling at least 50 ml in 250 ml bottle	No chemical preservative and no refrigeration needed		
	Unknown	Delta Deuterium	Isotech	1 L - same bottle as other (ST) analyses for Isotech	No chemical preservative; **Keep On Ice; Filter in Field**		
	Unknown	Delta Oxygen-18 of H2O	Isotech	1 L - same bottle as other (ST) analyses for Isotech	No chemical preservative; **Keep On Ice; Filter in Field**		
	Unknown	Delta Oxygen-18 in dissolved sulfate	Isotech	1 L - same bottle as other (ST) analyses for Isotech	No chemical preservative; **Keep On Ice; Filter in Field**		May be collected periodically based on input from management
	Unknown	Delta Sulfur-34	Isotech	2 L - same bottle as other (ST) analyses for Isotech	No chemical preservative; **Keep On Ice; Filter in Field**		
RADIOGENIC ISOTOPES (RG)	*Unknown*	Carbon-14	Beta Analytic	1 L	No chemical preservative and no refrigeration needed		
	Unknown	Tritium	Isotech	500 mL - if sending for just Tritium. Can be in same 1 L bottle if sending for all Isotech analyses (ST) and (RG)	No chemical preservative. If sending for just tritium, no refrigeration needed and no field filtering required.		

R. 13 E. | R. 14 E. | R. 15 E. | R. 16 E. | R. 17 E.

T. 02 S. | T. 03 S. | T. 04 S.

Upper Mineral Creek - MC 8.4C
Government Spring
Mineral Creek
55-615260
Laguna Spring
55-632793
Sump Spring
Walnut Spring
RC18-4
RC19-15
RC19-10
Stone Cabin Spring
Stone Cabin Box Spring
Indian Spring
Chimney Spring
RC18-9
Woodchopper Spring
55-632794
RC19-13
55-632797
Skunk Spring
Looney Spring
55-205266
Mineral Creek Rd.
Haley Spring
RC19-7
55-632800
RC19-8B
RC19-8
RC19-8C
RC19-3
RC20-2B(S)
RC20-2B(D)
Dripping Springs Rd.
Well Spring
55-622471
Big Spring
RC-POC (preliminary)
RC20-2D
55-807702
55-502917
35-17570
55-808287
RC20-2B(S)
RC20-2B(D)
Elkins Spring
Dripping Spring Wash
77
177
Gila River
KEARNY
GILA COUNTY
PINAL COUNTY
Gila River below Dripping Springs Wash

EXPLANATION

- RC Well
- Regional Well
- Spring
- Surface Water Location
- RC-POC Well (preliminary)
- Seepage Collection Pond
- Streams and Drainages
- Road
- Fault
- Proposed Skunk Camp TSF Footprint
- City
- Counties

0 2,500 5,000
Meters

0 2 4
Miles

RESOLUTION COPPER

SKUNK CAMP MONITORING PROGRAM LOCATIONS

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Water Resource Consultants

2020

FIGURE 1

G:\GIS-Tuc\Projects\605\605.85\AnalysisMaps\SAP\Figure 1_Locations Map_26Aug20_v2.mxd

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Appendix A

Standard Operating Procedures for Field Work and Water Quality Sampling

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HEALTH AND SAFETY STANDARD OPERATING PROCEDURES

TABLE OF CONTENTS

Page

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GENERAL FIELD STANDARD OPERATING PROCEDURES

1.0 SOP - GENERAL FIELD SAFETY

- Always be alert to possible risks or anything that just “does not seem right”.
- Ask questions about anything you do not understand.
- Follow all safety rules.
- Refrain from “horseplay”.
- Inspect all field equipment and tools, personal protective clothing (PPC), and personal protective equipment (PPE) before mobilizing to work site and before use each work day, and complete applicable field equipment checklist(s).
- If a hazard is identified during a pre-use inspection, mitigate or eliminate the hazard before using the equipment
- Immediately report to the project manager or health and safety manager any defective, malfunctioning, or “just-not-right” tool, machine, or equipment.

- Follow instructions for safe use of tools and machines.
- Keep the work area neat and the aisles clear.
- Walk; do not run.
- Wear assigned PPC/PPE.
- Immediately report any field accident, injury, or illness to the project manager or health and safety manager.

2.0 SOP - GOOD HOUSEKEEPING FOR THE FIELD

- Keep alert so that tripping, falling, impact, puncture, and splinter hazards can be eliminated.
- Do not overload electrical circuits.
- Do not leave extension cords near heat or water.
- Do not let grease or dirt build up; they are fire hazards, and are detrimental to equipment.
- Keep food, drinks, and cigarettes out of the work area. They can become contaminated by chemicals, attract bugs, and add to the general clutter.
- Keep cords, wires, and ropes untangled. Getting knotted is damaging to cords, wires, and ropes, as well as a nuisance to undo.
- Make sure all containers and materials are labeled. If you do not know what something is, find out.
- Keep portable lights and vehicle lights clean. Dirty lights do not give off sufficient light and can be a fire hazard.
- Report holes, loose boards, and other floor or terrain problems so they can be fixed before someone trips and gets hurt.
- Throw away trash promptly and properly. Be sure that hazardous trash is placed in proper containers and that incompatible trash is placed in correct containers. Trash should also be emptied frequently. If that is not happening at the site, contact the project manager or health and safety manager.
- Keep all tools and materials in their proper places when not in use.
- Keep sharp edges covered.
- Keep walkways and floors clear at all times.
- Keep aisles, passageways, and sprinklers clear.
- Keep all field vehicle and tool drawers closed when not in use.
- Use permanent wiring, not extension cords, whenever possible.
- Keep flammable liquids in approved airtight metal containers equipped with a flame arrestor.
- Keep flammable liquids away from ignition sources.
- Clean up spills immediately.
- Do not let dust or lint build up on machinery or work surfaces.
- Dispose of flammable scrap in tight, closed metal containers, and empty the containers daily.
- Remove only necessary quantities of chemicals from containers.
- Make sure all chemical containers are labeled.
- Keep chemical containers closed when not in use.
- Check chemical containers regularly for leaks.
- Environmental samples shall not be stored in food/drink coolers or refrigerators.

3.0 SOP - MUSCULOSKELETAL DISORDER (MSD) PREVENTION

- Ensure prevention through stretching exercises.
- Try to avoid or reduce repetitive movements.
- Switch tasks periodically to use different movements and muscles.
- Change positions regularly.
- Keep wrists straight rather than bent or flexed.
- Reduce the number of repetitive movements in a task.
- Change awkward positions to reduce strain on your body.
- Break in slowly to a new repetitive task.
- Use tilting or adjustable-height work surfaces.
- Choose and use only the right tool for the task.
- Use a power tool instead of a manual tool when possible.
- Maintain tools properly to reduce vibration.
- Use tools that are lightweight or have rounded or cushioned handles or textured grips.
- Use two hands for a task rather than one.
- Wear gloves with a smooth, comfortable fit.
- Grasp objects with your full hand and all fingers.
- Carry materials with a palm-down grip (especially if they’re an awkward size).
- Take early action when symptoms appear.

4.0 SOP - BACK AND LIFTING SAFETY

- Use the right lifting position and technique. Keep the object you are lifting as close to your body as possible.
- Estimate the weight of the object you want to lift. If it is difficult to move, it is too heavy to lift. Do not lift it alone. Get someone to help you or use a lifting aid such as a cart, hand truck, wheelbarrow, dolly, hoist, or a lift truck. Heavy, bulky, and oversized loads include portable generators.
- Determine the frequency of lifting. Get a lifting aid for tasks that must be repeated often.
- Avoid bending, reaching, and twisting as you lift. Turn your whole body in the direction you want to move, again, avoid twisting.
- Use a stepstool or platform to reach loads above your head.
- Grasp the object correctly and firmly. If available, use lifting handles.
- Lift with your legs. Bend your knees. Keep your back straight.
- Stay on secure footing. Be extra cautious on surfaces that are slippery, unstable, or uneven.
- Squat to set loads down.
- Keep fit. Exercise regularly and keep your weight under control. Your body needs strength and flexibility to lift.
- Take plenty of time to lift objects. Do not hurry.

5.0 SOP - SLIP, TRIP, AND FALL SAFETY

- Keep everything in its proper place and put things away after use.
- Repair or report any terrain and floor problems: uneven terrain, slippery mud, loose or missing tiles, warped wood planks, buckled carpet, and turned-up edges.
- Keep walkways and aisles clear of obstacles.
- Keep equipment drawers closed.
- Dispose of trash promptly and properly.
- Do not leave machines, tools, or other materials on the ground or floor. Block off and mark areas that are being cleaned or repaired.
- Keep cords, power cables, and air hoses out of walkways.
- Clean up spills and leaks right away.
- Be sure there is enough lighting before you move ahead. If need be, use a flashlight.
- Walk; do not run. Walk slowly, with a sliding motion, on slippery or uneven terrain or surface. Wear safety boots with nonskid soles.
- Beware of loose pant cuffs; you could trip over them.
- Do not carry a load you cannot see over, especially on stairs.
- Keep your hands at your sides, not in your pockets, for balance. Use railings when climbing up or down stairs.
- Keep all four chair legs on the ground or floor when you sit in a chair.
- Do not jump off platforms or loading docks.
- Step around obstructions.
- Do not engage in horseplay or practical jokes.
- Pay attention to where you are going and what might be in your way.

6.0 SOP - MOTOR VEHICLE AND DRIVING SAFETY

- Abide by all traffic laws and signs when operating any vehicle. Other safe driving rules adopted by clients, prescribed by state or local laws, or applicable Department of Transportation (DOT) safety regulations shall be adhered to.
- Be mentally and physically rested and alert prior to each trip.
- Be sure to allow adequate driving time for the road conditions.
- Check to see if appropriate insurance cards are in the M&A-owned field vehicles.
- Check your vehicle daily before each trip, and check the vehicle visually each time before driving. Complete a daily vehicle checklist for all company and rental vehicles.
- Do not drive any vehicle on M&A business that you know or suspect to be unsafe.
- Do not drive faster than a rate consistent with existing speed laws, site regulations, road traffic, and weather conditions. Obey posted speed limits.
- Do not give rides to strangers or hitchhikers.
- Do not operate an unsafe vehicle until repairs are made.
- Do not ride in the bed of a pickup truck.
- Do not take chances while operating motor vehicles.
- Do not use a cellular phone while driving. This includes texting and use of hands-free devices.
- Ensure that the fire extinguisher is securely fastened in the company vehicle.
- Immediately report any motor vehicle accident to your supervisor or health and safety manager.
- Keep to the right except when overtaking slow-moving vehicles, or when getting into a position to make a left turn.
- Never attempt to exercise the right-of-way; let the other driver go first.
- Never follow another vehicle so closely that you shall not be able to make a safe stop under any conditions. Observe timed interval and following distance guidelines.
- Report all accidents immediately, as required by law, site regulations, and M&A policy.
- Report all arrests and traffic convictions to HR.
- Report any required vehicle maintenance or repair to your supervisor (e.g., oil change, tire replacement, and window or light repair/replacement).
- Report any unusual and/or unsafe condition and/or operation of any vehicle used for M&A business to your supervisor or health and safety manager.
- Restrict use of M&A vehicles to M&A business.
- Slow down and watch for children in school zones.
- Use turn signals to show where you are heading while going into traffic and before every turn or lane change.
- Vehicles are to be driven by authorized drivers only.
- **Wear a seat belt when operating or occupying any vehicle.**

7.0 SOP - TOWING SAFETY

- Chock wheels of the trailer, when not in tow. Wheel wedges shall be used whenever a rolling hazard exists.
- Ensure you are trained prior to towing a trailer. Document this training on the daily safety briefing attendance record or other appropriate M&A training log.
- If possible, have the rental company deliver and pick up the trailer from the field site.
- Ensure the correct size tow ball is used for the trailer being towed.
- Never tighten the winder or coupler with a wrench or hammer. Only hand-tighten the coupler.
- Hook safety chains to the vehicle. Crisscross the safety chains, under the trailer tongue. This prevents the trailer from hitting the ground in the event of a trailer separation. Leave enough slack to permit tight turns.
- Check the safety chains and ensure they are secure and adjusted properly. This prevents the trailer from separating from the towing vehicle in the event of an accident.
- The tongue of the trailer must be level. Before going on the road, check that the tow ball and coupler are securely fastened and the latching mechanism is locked. Also, check that bolts with washers are tightly secured as the vibration of road travel can loosen them.
- Check to see if lights are working properly. The law requires brake lights, taillights, and turn signals.
- Check electrical wiring for broken wires or terminals, and proper connections. Have worn or broken wiring repaired or replaced, if necessary.
- Adjust mirrors. Extended mirrors are required if vision is blocked by the trailer.
- Do not leave anything on the trailer that could blow off during transit.
- Riding on a towed trailer is strictly prohibited.
- To become familiar with the changing driving characteristics in acceleration, braking, and handling, test-drive the vehicle with the trailer prior to starting the fieldwork.
- If it is necessary to back up the field vehicle with a trailer in tow, seek assistance. Have another person, who is located out of the field vehicle, provide hand signals to guide you while backing up.
- Check the hitch installation before, during, and after use.
- Swing wider. Wider swings at curves and corners are needed because the trailer's wheels are closer to the inside of a turn than the wheels of the field truck.
- Avoid sudden starts and stops. Start slowly. Drive slowly. Check spacing. Stop slowly. Never forget the extra weight that needs to be managed.
- Drive safely. Allow more time when stopping, passing, and changing lanes.
- Signal your intentions.
- Always be courteous. Stay in the right lane except when passing.
- Pass with extra care. It takes more time to pass with a trailer in tow.
- Shift to a lower gear to help ease the load on the transmission and engine when going over steep hills, sand, gravel, or dirt roads.

- Watch the wind. Be aware of sudden changes in air pressure caused by winds, or passing vehicles.
- If there is any doubt as to the operation or safety of the trailer, do not use it. Call the rental company immediately.

8.0 SOP - GENERATOR SAFETY

- Do not lift a generator by yourself. Get help or use a lifting aid/dolly.
- Make sure the unit provides adequate power for your needs.
- Operate generators only in well-ventilated areas.
- Do not touch the generator with sweaty or wet hands.
- Always use a transfer switch when a generator is connected to a circuit breaker box.
- Make sure you have good fuel (an oil change is sometimes recommended).
- Refuel the generator when it is off and is cool.
- Make sure the generator is operating properly and is placed on level ground.
- Use grounded extension cords, when applicable, and use cords with sufficient wire gauge for the application.
- Do not store the generator near a gas water heater or furnace.
- Do not operate a generator at maximum power output for more than 30 minutes.
- Follow manufacturer's use instructions and grounding recommendations.
- Locate the generator as close as possible to a wellhead to minimize the distance between the generator, the starter box, and the pump cable.
- Ensure all safety precautions are addressed with the generator (grounding, wire configurations, cable inspections, knockouts, lock-out/tag-out). Follow the established safety procedures for your project, which may include having an electrician conduct an inspection before using the equipment.
- Start up generator and allow to warm up for several minutes before operating pump.
- Make sure generator, electric cords, and control panel are placed in a location that will not be wetted by pumped water or precipitation run-off.
- Do **not** rest the control panel on the generator; vibration from the generator may cause the panel to fall, possibly severing a cable and creating an electrical fire hazard.

9.0 SOP - FIRE PREVENTION FOR THE FIELD

- Keep machinery lubricated so the equipment does not run too hot.
- Keep motors and machine tools clean and free of dust and grease that could burn.
- Use a nonflammable material instead of a flammable one, whenever possible.
- Clean up flammable and combustible liquid leaks and spills immediately.
- Repair any flammable and combustible liquid leaks immediately.
- Remove any clothing that has absorbed a flammable liquid immediately.
- Use proper, approved containers for flammable liquids.
- Store flammable materials only in designated locations.
- Check container labels and MSDSs to make sure incompatible substances are not stored close together.
- Assume an empty container that held a flammable liquid still has flammable residue.
- Ground and bond containers when transferring flammable liquids to prevent static electricity from igniting materials.
- Dispose of flammable waste in covered, airtight metal containers, whenever possible.
- Dispose of all waste promptly and properly.
- Keep work areas clean and free of dust and lint.
- Keep walkways and passageways clear.
- Provide adequate ventilation when flammable liquids or gases are being used.
- Properly store and dispense flammable liquids and gases.
- Remove combustible waste from the workplace daily.
- Transport flammable liquids and gases in safety cans or compressed gas cylinders that have been properly labeled.
- Clean up flammable liquid spills immediately.
- Use only properly grounded electrical equipment.
- Use a container that is intended and labeled for that flammable liquid.
- Use only labeled containers in good condition to store flammable materials; keep the containers closed when not in use.
- Do not:
    - Accumulate combustible waste.
    - Leave heating equipment or machinery running unattended or overnight.
    - Leave a circuit breaker blocked in a closed position.
    - Store oxygen cylinders near combustible materials.
    - Handle oxygen cylinders with oily hands or gloves.
    - Refill a generator or gasoline motor unless it is turned off and cool.
    - Store materials so high that they block fire sprinklers.
    - Let waste materials accumulate in the work area.
    - Smoke outside designated areas.
    - Heat a flammable or combustible liquid.
    - Store flammable liquids in coolers or domestic-type refrigerators.
    - Pour flammable liquids down ditches or sink drains.

- Smoke, use open flames, or create sparks where there is a possibility of igniting flammable or combustible materials.

10.0 SOP - ELECTRICAL SAFETY FOR THE FIELD

- Inspect electrical equipment and wires before use to make sure the equipment is properly insulated and grounded and that electrical connections are tight.
- Report all electrical hazards:
  - Loose electrical connections.
  - Cords with no insulation or frayed insulation.
  - Plugs that do not match your outlet (*e.g.*, a three-pronged plug in a two-pronged outlet).
  - Non-waterproof cords used outdoors.
  - Equipment running over capacity.
  - Tools that smoke, smell, spark, or shock.
  - Extension cords instead of permanent wiring.
  - Wires running across the ground or floor.
  - Electrical cords left near heat or water.
  - Electrical cords used around hazardous, flammable, or explosive materials and not designed for that use.
- Never use a metal ladder around live electricity.
- Always obey barriers, signs, and other warnings to stay away from electrical equipment.
- Use PPC/PPE such as special rubber gloves or boots when you work with electrical equipment.
- Avoid using extension cords whenever possible.
- Check with the site safety officer or project manager before you handle anything electric.
- Inspect portable equipment, including extension cords, before each use and turn in anything that is defective or damaged.
- Be sure electric plugs match your receptacles; never alter a plug.
- Do not fasten cords with staples.
- Make sure your hands are dry before you handle anything electric.
- Do not use any piece of electrical equipment that sparks, smokes, smells, or shocks. Lock out, tag out, label “out of order”, and immediately report the hazard.
- Be especially cautious around flammable liquids, vapors, or dusts, or any area that might have contained them. Ventilate the air before starting work and use only electrical equipment identified as safe for that use. Keep an eye on the equipment to avoid sparks or high heat that could start a fire.
- When using long conductive objects like pipes around exposed live parts, be sure to use insulation, guarding, and other precautions.
- Keep machines and tools properly lubricated.
- Do not let grease, dust, or dirt build up on machinery.
- Do not leave machinery or heating equipment operating unattended after working hours.

- Leave at least three feet of workspace around electrical equipment so it can be reached for repair or maintenance.
- Keep the work area clean. It is especially important to properly dispose of paper, sawdust, oily rags, or anything that could burn.
- Do not leave cords tangled or lying across an area where people walk.
- Check wiring to make sure it's properly insulated and the right choice for the job (*e.g.*, labeled for use outdoors or in work areas with hazardous substances).
- Check that electrical connections are tight.
- Match plugs to outlets (*i.e.*, three-pronged plugs in three-pronged outlets only).
- Read and follow manufacturer's instructions for electrical equipment.
- Leave work on energized equipment to **qualified electricians**.
- Obey warnings to stay away from electrical circuits and equipment that is locked out and tagged out (LOTO).
- Inspect electrical tools before each use.
- Report any electrical tool, equipment, or wire problems immediately.
- Do not overload motors, circuits, or outlets.
- Do not use temporary wiring except in emergencies.
- Do not put anything but a plug into an electrical outlet.
- Do not place cords near heat or water.
- Do not let cords get twisted or tangled.
- Do not get closer than 30 feet to a power line; a greater distance may be required depending on the voltage.
- Do not reach blindly into a space that may contain energized equipment.
- Do not wear metal jewelry when working with electrical equipment.
- Provide first-aid for electrical accidents:

    - Shock - Do not touch a shock victim. Call for professional medical help and, if you can do it safely, turn off the power that is giving the shock. Use a stick or something that will not conduct electricity to push the person away from the wire or equipment that caused the shock. Have the victim lie down, and lightly cover the victim, until help comes. If breathing has stopped, give artificial respiration to the victim. If the heart has stopped, give CPR to the victim.

    - Electrical Burn - Rinse with cold water and cover with a clean dry cloth and get medical attention. If it is a major burn, cover it with a clean, dry cloth. Get immediate medical attention.

    - Electrical Fire - Notify firefighters immediately. Do not touch the burning object or use water on it. If you can do it safely, unplug or turn off the power. You can put out a very small fire with a multipurpose ABC extinguisher. Unless the fire is very small, field employees seek the assistance of trained firefighters.

11.0 SOP - PERSONAL PROTECTION

- Identify hazards that require PPC/PPE before starting a job.
- Select and use PPC/PPE that protects against the identified hazards. Double check that PPC/PPE protects against identified hazards.
- Maintain PPC/PPE in good working condition.
- Prepare and inspect PPC/PPE before donning.
- Inspect for rips, tears, dents, disintegration, or other damage. Turn in and replace any damaged PPC/PPE.
- Notify the HSM or HSC if PPC/PPE is lost or damaged so new and replacement PPC/PPE can be ordered.
- Know the correct way to don and doff PPC/PPE; if you do not know, ask.
- Practice performing field tasks while wearing PPC/PPE in a non-hazardous environment and make any needed adjustment or replacements.
- Be especially careful when removing PPC/PPE that has been contaminated by hazardous chemicals. Remove contaminated PPC/PPE one piece at a time, from the top down.
- Keep your gloves on while removing the other clothing so you do not get anything on your hands.
- Properly clean and store PPC/PPE.
- Dispose of contaminated clothing in assigned containers.
- Clean off non-contaminated PPC/PPE and return to assigned storage.
- Store and maintain PPC/PPE according to manufacturers' instructions.

11.1 HEAD PROTECTION

- Wear a hard hat in any situation where there might be falling or flying objects, potential for bumping into objects, or electricity hazards.
- Adjust the headband so there's space between hat and head to absorb shock.
- Check the label inside the hat to determine protection capability.
    - Wear a Class A rated hard hat in most field situations.
    - Wear a Class B rated hardhat in field situations with electrical hazards.
- Do not wear a hard hat over a cap or other hat.
- Do not abuse the hat.
- Replace a hat that has cracks or holes.
- Clean the hat occasionally in soap and warm water.
- Store the hat away from sun or extreme heat or cold.
- Avoid placing stickers on the hard hats as some adhesives weaken the material.

11.2 EYE AND FACE PROTECTION

- Identify potential hazards and select proper eyewear and face protection before starting a field task.
- Select eye and face protectors that:
  - Provide the best protection against identified hazards.
  - Are reasonably comfortable.
  - Fit snugly enough to keep out hazards.
- Before using eye and face protectors inspect for:
  - Damage such as knotted, twisted, worn, or stretched goggle straps.
  - Pitted or scratched lenses/face shields.
- Turn in and replace damaged eyewear.
- Wear safety eyeglasses equipped with side shields to keep out flying objects.
- Use a face shield plus goggles when handling corrosive materials (*i.e.*, acids or caustics).
- Use tinted protective eyewear that protects against harmful ultraviolet (uV) light.
- Never use metal-framed eyewear around live electricity.
- Use prescription protective eyewear or safety goggles designed to fit over prescription glasses.
- Avoid wearing contact lenses in areas with hazardous dusts or chemicals.
- After removing eye and face protectors:
  - Inspect for damage.
  - Clean lenses with water plus soap or mild detergent.
  - Decontaminate if contaminated by chemicals or disinfect if other people may use it.
  - Store in proper place in closed containers.
- Follow safe work procedures that prevent eye and face injuries such as:
  - Adhering to MSDS precautions when working with chemicals.
  - Not opening a container just to see what is inside.
  - Wearing eye and face shields when around all potential flying objects and splashing hazards.
  - Containing as many flying or splashing hazards as possible.
  - Ensuring workers in the area are protected **before starting tasks** that create eye or face hazards.
- Get medical attention for eye and face injuries and be familiar with first-aid treatment:
  - Chemical splash in eye: go immediately to emergency eyewash and flush eye for at least 15 minutes; get water under eyelids; get immediate medical attention.
  - Something in the eye: if it doesn't blink out, do not rub it; keep eye closed and covered; get immediate medical attention.
  - Receive a blow to the eye: get immediate medical attention.

11.3 HEARING PROTECTION

- Be aware of noise levels requiring hearing protection.
- Avoid high noise levels whenever possible.
- Wear assigned hearing protectors (i.e., earplugs or earmuffs).
- Report hearing protectors that do not fit well or work well.
- Make sure hands are clean before inserting or putting on hearing protectors.
- Keep hearing protectors clean.
- Take special care when wearing hearing protection around drill rigs, other motorized equipment, or when working in high traffic areas as the ability to verbally communicate a warning is minimized.
- Maintain visual contact with co-workers when wearing hearing protection.
- Maintain machinery and equipment to reduce noise levels.
- Attend annual hearing tests as part of the Hearing Conservation Program (HCP).
- Have your hearing tested if you have:
    - Noise or ringing in your ears.
    - Trouble hearing people speaking.
    - Trouble hearing certain high-pitched or soft sounds.
    - TV or radio volume turned so high others complain.
- Do not:
    - Tamper with hearing-testing or noise-reduction equipment.
    - Ignore or forget to use hearing protection.
    - Use hearing protectors that are loose or cracked.
    - Use hearing protectors that do not fit properly.

11.4 HAND PROTECTION

- Wear the glove material(s) specified in the site HASP.
- Work gloves must be worn at all times while working with equipment. The only time gloves are not required is when the sole task is documenting data.
- Make sure gloves fit comfortably.
- Wear insulated or leather gloves for hot and cold. Glove fabric should be fire-retardant for open flame work and reflective for radiant heat tasks.
- Wear special insulated rubber gloves for electricity hazards.
- Wear metal mesh or other cut-resistant gloves to handle sharp objects.
- Wear leather gloves for rough surfaces.
- Wear fabric gloves for handling slippery objects.
- Wear nitrile, neoprene, rubber or other suitable chemical-resistant gloves to handle toxic and/or corrosive materials.
- Check the MSDS before selecting gloves when handling chemicals.
- Use long cuffs or duct tape to keep chemicals or heat from getting into gloves.

11.4.1 Chemical–Protective Gloves

- Inspect before wearing to make sure gloves are clean, with no rips or holes.
- Bandage any small cuts or scrapes on your hands before putting on gloves.
- Clean gloves thoroughly before removal, disposal, or storage.
- Store gloves in a cool, dark, dry place, right side out, with cuffs unfolded.
- Wash hands frequently and thoroughly with soap and water or skin cleanser. Do not wash hands with solvents or industrial detergents after working with chemicals.

11.4.2 Working with Machinery and Tools

- Pay full attention to the job and the equipment.
- Follow manufacturer and M&A instructions for using tools and equipment.
- Always cut away from your body when using a knife.
- Use the right tool for the job and use it correctly.
- Store tools so no sharp edges are exposed.
- Pass tools handle first. Do not throw tools to other workers.
- Wear gloves at all times for protection against cuts and abrasions while handling and operating equipment. Gloves must be close fitting and not have large cuffs or loose ties that can catch on moving parts.
- Keep machine and tool guards in place.
- Use lock out/tagout (LOTO) procedures when machines have to be repaired or maintained.
- Keep your hands away from moving machine parts.
- Feed materials into moving machinery with a push stick, not your hands.

11.4.3 Handling Materials

- Check materials for sharp edges, burrs, and splinters before handling them.
- Use brushes, not hands, to sweep up metal or wood chips.
- Make sure you know how hot or cold an object is before handling it.
- Wipe off greasy or slippery objects before handling them.
- Lift an object so your hands are not near the pinch points.
- Keep fingers on the sides, not the top or bottom, of spacers when you are stacking materials.
- Put materials down carefully so you do not smash your fingers.

11.4.4 Prevention of Carpal Tunnel

- Use power tools whenever possible on repetitive jobs.
- Try to vary your movements.
- Keep wrists straight rather than bent or flexed as much as possible.
- Use the full hand and all fingers to hold objects.
- Shake out your hands occasionally.

11.5 SKIN PROTECTION

- Cover as much skin as possible when on the job.
- Wear sunscreen with an SPF of 15 or higher. Reapply sunscreen as necessary throughout the day.
- Wear chemical-resistant PPC/PPE as assigned in the site HASP. Wear the right kind of PPC/PPE for the job, and make sure it is in good condition (*e.g.*, Tyvek® coveralls, face shield, and splash apron).
- Follow instructions on labels and MSDSs.
- Bandage any small scrapes or cuts before putting on gloves or protective clothing.
- Wash promptly and thoroughly after working with hazardous substances, even if there was no direct contact.
- Do not use solvents or industrial detergents to clean your hands; solvents and detergents can create skin problems of your own.
- Change your work clothes every day, and wash the clothes separately from street clothes.
- Protect pre-existing skin conditions from further aggravation.
- Report any skin reactions or problems that develop on the job.
- Understand and follow proper first-aid procedures for cuts, burns, and exposure to hazardous substances.

11.6 FALL PROTECTION

- Use ladders, railings/guardrails, and fall arrest equipment as designed and intended.
- Do not use metal ladders near electrical circuits. Ensure portable metal ladders are legibly marked “Caution – Do Not Use Around Electrical Equipment”.
- Do not store wood ladders near radiators, stoves, steam pipes, or other places subject to excessive heat or dampness.
- Ensure ladder rungs are kept free of grease and oil.
- Do not place ladders on boxes, barrels, or other unstable bases to obtain additional height.

- Face the ladder when ascending, working upon, or descending.
- Do not stand on the top rung of a stepladder.
- Complete training in fall arrest equipment prior to use.
- Use personal fall arrest equipment when there is a free fall hazard of six feet or greater and where its use does not create a greater hazard.
- Do not use a fall arrest system unless you have been trained and authorized to do so.
- Inspect the fall arrest equipment for signs of tearing, cracking, or deformation before each use.
- Ensure fall arrest equipment fits you properly and securely.
- Do not use fall arrest equipment if you are uncertain about the safety or strength of the equipment; consult a qualified person, such as the manufacturer or HSM.

11.7 RESPIRATORY PROTECTION

- Use only National Institute for Occupational Safety and Health (NIOSH)–approved respirators.
- Check the following:
    - Cartridge/canister color-coding and label to be sure it protects against the specific contaminant.
    - End of service life indicator or follow the change schedule for cartridges/canisters described by the manufacturer.
    - Fit and seal of the respirator before each wearing.
    - Condition of the respirator before each wearing.
- Understand the limitations of a respirator.
- Know how to properly put on your respirator.
- Recognize that a respirator may slow the work pace or make it more difficult to do certain tasks.
- Pay attention to the quality of the air supply and get to fresh air when an air contaminant is tasted or smelled.
- Report and do not wear a respirator that shows damage.
- Remove a respirator in such a manner that you do not contaminate yourself.
- Clean and disinfect respirators after each use.
- Store respirators carefully in its assigned location.
- Use an atmosphere-supplying respirator whenever there is not enough oxygen.
- Get to fresh air when atmosphere-supplying respirator signals that the air supply is low.

12.0 SOP - CHEMICAL SAFETY FOR THE FIELD

- Follow handling, storage, transportation, and disposal instructions provided on the MSDS or label.
- Ensure an MSDS is on file and is readily available for hazardous substances used at, brought onto, or stored at field sites. This applies to those hazardous substances used by contractors/subcontractors.
- Append applicable MSDSs to the site HASP or provide MSDSs in a readily available MSDS field binder.
- Know where the MSDS collection is located at the site (*e.g.*, appended to the site HASP or compiled in an on-site MSDS binder).
- Label field storage rooms, work areas, and containers including gasoline fuel cans. Indicate chemical contents of containers including any hazards such as, but not limited to, corrosive, toxic, flammable, or non-potable water.

13.0 SOP - PERSONAL HYGIENE AND SANITATION

- Eating, drinking, chewing gum or tobacco, and smoking only in designated areas.
- Drinking only from potable water dispensers or systems that have been clearly labeled as potable water.
- Drinking from disposable cups or closed-lid bottles.
- Ensuring that potable water dispensers are tightly closed after each use.
- Ensuring that single-service cups are properly disposed of in the designated waste receptacle.
- Refraining from dipping containers into potable water supplies and using nonpotable water for drinking or washing.
- Disposing of waste only in designated containers.
- Ensuring that a portable toilet is provided when required and is maintained in a sanitary condition.
- Knowing the location of the nearest toilet and washing facility.

14.0 SOP - EMERGENCY PREPAREDNESS AND ACTION FOR FIELD OPERATIONS

- Be familiar with the following field hazards:
  - Flammable liquids and the circumstances in which they could catch fire.
  - Reactive chemicals and the types of reactions they could cause.
  - Explosive agents and what could cause them to explode.
  - Electrical hazards that could cause fires.
  - Chemical gases, vapors, and dusts that could be toxic, burn, or explode.
- To be prepared for emergencies, be aware of the following:
  - Emergency escape procedures and routes.
  - First-aid kit location.
  - Rescue and medical duties for first aid and CPR.
  - Fire extinguisher location and use.
  - Procedures to account for employees following emergency evacuation.
  - People to contact for further information. That is, names and phone numbers of people inside and outside M&A to contact in an emergency.
  - Procedures for reporting emergencies.

14.1 ESCAPE ROUTES AND EVACUATION PROCEDURES

- Know how to report a fire, spill, or other accident.
- Recognize the sound of the emergency alarm.
- Immediately stop work upon notification that an evacuation is required. Notification may be by an alarm system, verbal instruction, or other means of notification.
- Use the nearest and safest exit from the workplace and evacuate in a safe and orderly manner.
- Do not endanger or threaten your health or the health of others by remaining in the workplace during an evacuation.
- Alert other workers around you to evacuate.
- Know the procedures for shutting down operations or systems.
- Follow the assigned evacuation route and meet at the designated assembly point.
- If emergency response (ER) duties are assigned, follow instructions on where to go and what to do.
- Know where to find first-aid supplies and fire extinguishers.
- Follow directions and instructions given by the site safety officer.
- Stay in the designated meeting area until the site safety officer or other authorized supervisor has accounted for all field employees.
- Do not re-enter the workplace for any reason until authorized to do so by the site safety officer or authorized supervisor.

14.2 RESCUE AND MEDICAL TREATMENT

- Assess and prioritize the medical treatment to be administered to injured or ill employees.
- Administer or direct the administration of first aid and/or CPR.
- Designate auxiliary staff members who are trained in first aid and CPR to assist with medical treatment, if necessary.
- Ensure that first aid and/or CPR are administered until ER professionals have arrived and are able to take over these duties.

14.3 FIRST AID FOR INJURIES

- Call immediately for emergency medical help.
- Bring help to the victim; do not bring the victim to help.
- Do not move an injured person unless it is necessary to save his or her life.
- Know where the first-aid kits are kept.
- Check to see if the victim is breathing.
- Do not use medication without a doctor's supervision.
- If you are not sure what to do, wait for medical assistance.

- Bleeding
    - Apply constant pressure to the wound with a cloth or your hand to stop the flow.
    - If that is not enough, apply pressure to, and elevate the wound.
    - If that is still not enough, apply pressure to, elevate the wound, and press firmly on the appropriate pressure points (*e.g.*, inside of the upper arm or the crease of the groin).

- Amputated Limb
    - Place limb in plastic bag with ice and rush to hospital with victim.

- Broken Bones
    - Wait for medical help.
    - Do not move the person unless absolutely necessary.

- Shock
    - Elevate the victim's feet, unless victim has suffered a head injury or is having trouble breathing.
    - Cover victim to maintain normal body temperature.
    - Maintain an open airway.
    - Loosen tight or restrictive clothing and do not provide fluids.

- Eye Injuries
  - Treat immediately.
  - Flush chemical splashes for at least 15 minutes with eyewash or water.
  - Cover closed eyes with clean cloth and take victim to the doctor.
  - Wait for medical help to remove object stuck in eye.

- Electrical Shock
  - Do not touch a victim who is in contact with a live electric current.
  - Turn off or have an electrician turn off the main electric switch or fuse.
  - Remove victim from the source of electricity.
  - Check for heartbeat and breathing.
  - Administer CPR, if necessary.
  - Cover and keep warm.

- Burns
  - For chemical burns, flush with water for 15 minutes and carefully remove contaminated clothing.
  - For other burns:
    - Wrap a victim who is on fire in a blanket or coat or make the victim drop and roll.
    - Cut away loose clothing, but do not touch clothing that is stuck to a burn.
    - Do not rub the body.
    - Immerse first- and second-degree burns in cold water to relieve pain; then cover the skin with a moist sterile dressing.
    - Elevate burned limbs
    - Treat the victim for shock and check for breathing problems.
    - Do not use lotion or ointment on a burn.

- Chemical Exposure
  - Refer to the chemical's label and MSDS for proper treatment.
  - Flush eyes and skin with water for 15 minutes.
  - Move inhalation victim to fresh air and administer artificial respiration or CPR, if necessary.
  - Get medical assistance in cases of ingestion and check MSDS or call the poison control center listed in the site HASP.

14.4 FIRST AID FOR ILLNESSES

- Call for medical help immediately. Explain the kind of illness or symptoms and where the victim is. Stay calm and act fast. Use emergency numbers for the medical facility, hospital, and paramedics.
- Use employees with first-aid training.
- Bring help to the victim; do not bring the victim to the help.
- Do not move an injured person unless it is necessary to save the victim's life.
- Know where the first-aid kits are kept.
- Check to see if the victim is breathing.
- Do not use medication without a doctor's supervision.
- If you are not sure what to do, call for professional help and wait.

- Not Breathing
  - Shout at and gently shake the victim (unless there is a possible back or neck injury) to determine if he or she is conscious.
  - If no response, call for medical assistance.
  - Look, listen, and feel for signs of breathing.
  - Place victim on your back, loosen clothes around the neck, and make sure nothing is blocking the mouth or throat.
  - Tilt the victim's head slightly, holding the mouth open with your thumb.
  - Pinch the nose and cover the mouth with yours.
  - Give the victim a full breath once every five seconds until you see the chest rise and fall, and see and feel breathing from mouth.

- Shock
  - Call for medical assistance.
  - Lie victim down.
  - Elevate the feet, unless victim has suffered a head injury or is having trouble breathing.
  - Cover victim to maintain normal body temperature.
  - Maintain an open airway.
  - Loosen tight or restrictive clothing and do not provide fluids.

- Heart Attack
  - Call for medical assistance.
  - Place victim in comfortable reclining or sitting position.
  - Loosen tight clothing at waist and neck.
  - Ask if the person has medication (*e.g.*, nitroglycerin tablets) that you can give them.
  - Keep the victim, still until help arrives.
  - If the heart stops, start CPR.

- Heatstroke
  - Call for medical assistance if victim is unresponsive or condition appears serious.
  - Remove the victim from the hot environment.
  - Cool the person down immediately with water from a hose or other source. You can also apply ice to victim's neck, armpits, and groin.

- Stroke
  - Call for medical assistance.
  - Cover the victim with light blanket.
  - Turn the victim's head to side if vomiting.
  - Do not give stimulant or anything to eat or drink.
  - Let professionals handle treatment.

- Epileptic Seizure
  - Call for medical assistance.
  - Move harmful objects out of victim's way.
  - Roll victim onto your left side. Maintain airway if vomiting.
  - Do not put anything in the victim's mouth.
  - Keep victim comfortable and keep other people away while waiting for medical assistance.

- Diabetes/Insulin Reaction
  - Call for medical attention if victim is unresponsive or condition appears serious.
  - If conscious, give victim candy, sugar, or honey.
  - Give nothing by mouth if victim is unconscious.
  - Keep victim warm until medical assistance arrives.

15.0 SOP - HEAT STRESS PREVENTION AND MONITORING

15.1 HEAT STRESS PREVENTION

Heat stress is a concern when wearing PPC/PPE and when extreme temperature and humidity exist. All field personnel must be aware of the symptoms of heat stress so that treatment can be immediately provided. The use of impermeable protective clothing may cause excessive heat stress, resulting in profuse sweating. To prevent dehydration, water lost during the work period will be replaced. Plain water is an excellent rehydrator and ample supply of cool water will be available. Gatorade® or other electrolyte drinks will also be available. The following intervals are recommended:

- Before shift and at each meal, drink a minimum of 16 ounces of water
- During break periods, drink a cup or two of water every 15 to 20 minutes or at each monitoring break. Larger workers may need larger quantities of fluids.

When warranted and feasible, the following controls will be implemented to prevent heat-related illnesses:

- Field personnel will have access to shaded rest areas.
- Field vehicles will be equipped with air conditioning.
- Power equipment will be used to reduce strenuous physical activities, when applicable.
- Field personnel will be rotated during work and rest cycles established by the site safety officer.
- Attendance at daily field safety briefings will be required.
- Intermittent rest periods will be taken with frequent water breaks.
- Frequent rest breaks will be taken in a cooler environment.
- First-aid provisions will be readily available.
- A rapid cool-down system, such as a water hose or water container, will be available.
- Emergency medical information will be readily available.
- Transportation to a medical facility will be readily available.

15.2 HEAT STRESS MONITORING

The primary mechanisms for monitoring heat stress during routine field activities include visual observation and heart rate. The use of the Wet Bulb Globe Temperature Index established by the American Conference of Governmental Industrial Hygienists is not anticipated and is not a valid indicator when PPC/PPE is worn. Heat stress will be monitored

by observing field personnel and documenting heart rate when either of the following weather conditions exist:

- Ambient temperature exceeds 70°F and impermeable clothing is being worn.
- Ambient temperature exceeds 85°F.

Each field person is responsible for maintaining contact with his or her buddy and being aware of signs of heat distress. M&A field personnel are trained to recognize heat-related problems and to provide first-aid treatment. The buddy system will be used to observe field person's eyes and facial expression, gait, and demeanor. Unusual changes in appearance, actions, or gait, as well as changes in demeanor or irrational behavior will be noted and appropriate heat stress treatment will be rendered.

Any field personnel experiencing or displaying heat stress symptoms will immediately be removed from the work activity and his/her heart rate will be determined. The heart rate can be measured by counting the radial pulse during a 30-second period as early as possible in the rest period. Any field employee whose heart rate exceeds 110 beats per minute at the beginning of the rest period will shorten the next work cycle by one-third and keep the rest period the same. If the field employee's heart rate still exceeds the 110 beats per minute at the beginning of the next rest period, the following work cycle will be shortened by another one-third. Use the Heat Stress Monitoring Log to record data.

Work/rest periods will be determined by the site safety officer. The work/rest period will be specific to the particular field activity and atmospheric conditions. In the event that impermeable PPC/PPE is used during field activities, pulse-rate monitoring will be used to determine heat-stress potential when the ambient temperature exceeds 70°F. Monitoring of pulse rate is also required when ambient temperature exceeds 85°F regardless of the PPC/PPE being worn.

TASK	GROUNDWATER SAMPLING		
PRECEDING OR CONCURRENT TASK	Preparation of Field Notes (**SOP 2.01**) Production Well Installation (**SOP 2.02**) Monitor Well / Exploration Well Installation (**SOP 2.03**) Zonal Sampling **(SOP 2.04)** Air-Lift Tests **(SOP 2.05)** Well Development **(SOP 2.06)** Pumping Tests **(SOP 2.07)**		
REVISION (DATE)	Rev 0 (September 21, 2012)	SOP Reference #	SOP 2.08
PREVIOUS (DATE)	Rev A (October 15, 2011)		
DESCRIPTION	Groundwater samples are collected as representative samples of water in the aquifer or target parts of the aquifer. It is imperative that the samples have not been altered or contaminated by the sampling and handling procedures.		
PERSONNEL	Field Managers and Field Staff		
FREQUENCY	As needed	Timing:	As needed

PURPOSE AND APPLICABILITY

This Groundwater Sampling SOP establishes uniform procedures for collection, documentation, and handling of groundwater samples. The procedures described herein ensure that groundwater samples are obtained in a credible, uniform, properly documented, and legally defensible manner when using the following devices: (1) dedicated pumps; (2) temporarily installed pumps; (3) air-lift pumping equipment; and (4) bailers. In cases where the sample will be collected from a discharge port as part of a discharge assembly, consideration must be paid to access to the sample port.

EQUIPMENT AND SUPPLIES

Before going to the sampling site(s), it is important to know what constituents you will be testing for and what kind of pump and discharge set up you will be using to collect your samples. It is also important to talk to your project manager, and familiarize yourself with any special sampling procedures. This will affect the type and amount of equipment you bring with you. Sampling equipment will generally consist of the following:

Project Information and Documents

- Technical well specifications, sampling plan, schematic well diagram, SOPs, and related documents
- Sampling paperwork:
  - List of laboratory analyses (with hold times and lab receiving hours)
  - M&A Traffic Reports
  - COC forms and custody seals
- Forms for recording water level readings, flow rate observations, and discharge observations (field parameters, color, clarity, sediment content, comments) printed on waterproof paper
- Clipboards and banker clips
- Field notebook and note pads
- If applicable, equipment details:
  - Pump and motor specifications
  - Pump installation notes and diagrams
- Applicable permits:
  - AZPDES De Minimis Discharge Permit
  - Copies of staff MSHA, OSHA, or other required certifications
  - Project-specific permits, such as Extra Permit to Work and Hot Work Permit

EQUIPMENT AND SUPPLIES (continued)

- Applicable safety documents, if applicable:
  - Emergency Response Plan with contacts and hospital location
  - Adequate road maps of region
  - MSDSs for all chemicals on site (e.g. calibration solutions, sample preservative)
  - Vehicle Inspection forms
  - Site Induction, Safety Tailgate, and Risk Assessment forms

Safety and Personal Items

- Appropriate clothing and PPE
- Rain gear, sunscreen, extra water, and extra food
- Digital wristwatch / stopwatch
- Table, chair, sunshade (canopy, umbrella, wide-brimmed hat)
- Nitrile or latex gloves
- Heavy-duty kitchen gloves to keep discharge fluids off of the hands and forearms and provide some insulation if production water is very hot
- Fleece liner gloves that can slip under sampling or kitchen gloves
- Work towels and rags
- Garbage bags
- Hand measuring tape (with engineering scale: 0.01' divisions)
- Headlamp / flashlights
- Cell phone / sat phone / radio
- GPS unit
- Camera
- Calculator
- Electrical chargers, DC/AC converter, Batteries (9V, AA, D-cell, etc.)
- Tool box (basic hand tools)
- Vehicle Safety Kit (first aid kit, fire extinguisher, chocks, tie-downs, tire gauge, air compressor, fix-a-flat, jumper cables, flares, 1-quart motor oil)

Sampling Equipment and Supplies

- Sanitized 5-gallon and 15-gallon buckets, if necessary
- Sample bottles and containers (See Section 3.1)
  - Include correct pre-preserved bottles for suite of analyses
  - Include extra sample containers for unplanned sample collection and/or mistakes during sampling
  - For VOC samples, one field blank and one duplicate sample should be obtained for every 10 samples
  - For VOC samples, one trip blank per shipped ice chest
  - Equipment blanks, if necessary
- Sample numbers and bottle labels (extra labels)
- Coolers / Ice Chests
  - Include a sufficient number of coolers to hold the anticipated number and volume of samples, plus any blanks, duplicate samples, and ice.
  - Know in advance which containers must be kept on ice following collection. In general, most environmental and drinking water samples should be stored

EQUIPMENT AND SUPPLIES (continued)	on ice.
	• Sample Filter Pump (Peristaltic Pump) and Pump Tool Kit
	• Sample Filters (0.45 micron)
	  o Include at least one sample filter per sample source, although more may be needed if the water to be sampled contains substantial sediment. Never reuse filters.
	• Sample preservative, if preservative not previously added to sample bottles
	• Pens, pencils, permanent markers, and a Rite-In-The-Rain® pen
	• Collapsible water cubes (cubitainers) for obtaining additional sample volume or transferring water to sample containers
	• Clean towels and/or paper towels
	• Clear packing tape and electrical tape
	• Bubble wrap, if sample kit includes glass containers
	• Water level sounder and backup
	• Field parameter meter with buffer and standards
	• Imhoff cone
	• Depending on sampling technique:
	  o Complete pump installation / testing equipment.
	  o Sample pump, connector cords, electrical panels, generator, and fuel
	  o Discharge assembly with valves, hoses, sampling port, flowmeter, pipe wrenches
	  o Clean bailers (one per sample source plus extras) with sufficient length of clean lanyard cord and reel to lower bailer
	  o Sample filter pump (peristaltic pump) and pump tool box
	  o Adaptor for attaching filters directly to hose bib / sampling port
	• Well locks and project keys
	• Project specific equipment (see project manager)

PREPARATION	Prior to field sampling activities, review pertinent sampling information including project-specific sampling plan, site safety plan, and all applicable SOPs. In addition, conduct the following tasks: • Obtain table of well and pump specifications and details for each well to be sampled, including estimated amount of water to be purged from each well • Review maps and UTM coordinates for monitoring wells; organize sampling route • Identify specific wells to be sampled • Identify specific analytical methods to be used • Review sample container requirements (See Section 3.1) • Contact laboratory to coordinate sampling event, review sample methods, and order appropriate type and number of sample containers, including duplicate and blank samples (See Section 3.1); notify lab of anticipated shipment dates for samples with short holding times • Prepare and print sampling data sheets, chain of custody documents, traffic report forms, analytical request schedules, shipping documents, and water level record sheets; place forms in field notebook • If shipping samples from outside the office, compile supplies for sample shipment including shipping containers, packing material, ice or ice packs, tape, pens, plastic bags, etc.

PREPARATION (continued)

- Compile all applicable field equipment and supplies (See Section 5.0)
- Determine where and how purged water will be discharged or stored at each well; ensure that appropriate discharge permit(s) (e.g. AZPDES deminimus discharge permit) have been issued or are in effect

Sample Containers

All sample containers should be ordered from the appropriate laboratories several days to 1 week before the scheduled sampling event. The containers should be placed in coolers or boxes for transport to the field site. Laboratories should provide documentation indicating which samples should be filtered, which containers contain preservative, maximum holding times, volume of each sample container, and any other pertinent details. The sampler(s) should review all documentation prior to sampling. Be sure to include enough sample kits to allow one field duplicate and one trip blank for every ten samples, as well as equipment blanks, if appropriate.

It is critical to be aware of all holding times prior to sampling, and to make sure the samples will be delivered to the laboratories with enough time for analyses to avoid conflicts with holding times. If uncertain of holding times, contact project manager or laboratory for information. In some cases, (for example, collection of isotope samples) sample containers and chemical preservatives are available in-house from the Montgomery & Associates Tech Room. All such supplies are purchased from established laboratory and environmental supply vendors (e.g., Fisher Scientific, Cole-Parmer, etc.).

Considerations

Before sampling, two important things to consider: 1) the maximum holding time for a sample, and, 2) the hours of operation at the receiving laboratory. If you are taking samples that have a maximum holding time of 3 days or less, you should not collect the samples immediately before the weekend or a major holiday, unless you make special arrangements with the laboratory for receiving the samples and analyzing those constituents with short holding times.

If possible, try to allow 1 hour before collecting samples to label containers and to fill out traffic reports and Chain of Custody documents. If you are collecting an extensive suite of samples for environmental or drinking water analyses, you may need to allow more than 1 hour of preparation time.

GROUNDWATER SAMPLING PROCEDURES

Procedures for groundwater sampling comprise purging and sampling. Purging removes potentially stagnant water in the well or borehole prior to sampling, which may represent water quality parameters unrepresentative of groundwater in the aquifer or target zone of the aquifer. After purging, the well or borehole is recharged with groundwater from the aquifer or target zone of the aquifer; this groundwater is more representative sample of actual water quality conditions.

Generally, purging is conducted using dedicated or temporary pumps, however use of bailers or air-lift procedures are often part of sampling plans. In all cases pH, temperature, and specific conductance will be monitored during purging. The data values shall be recorded into the field log book.

Purging:

1. If installing pumping equipment, review scope of work and project directives. After pump installation, set up discharge assembly/hoses, connect generator cables and control panel, and measure and record water level on appropriate field form.

GROUNDWATER SAMPLING PROCEDURES (continued)

2. If generator is used for powering, ensure all safety precautions are addressed with the generator (grounding, wire configurations, cable inspections, knockouts, lock-out/tag-out). Follow the established safety procedures for your project, which may include having an electrician conduct an inspection before using the equipment.
    a. Start up generator and allow to warm up for several minutes before turning pump control panel switch or lever to "on" position. Make sure generator, electric cords, and control panel are placed in a location that will not be wetted by purged water, precipitation, or runoff.
    b. Do **not** rest the control panel on the generator; vibration from the generator may cause the panel to fall, possibly severing a cable and creating an electrical fire hazard.

3. During pumping, periodically (every 5 to 10 minutes) measure and record the following on appropriate field forms:
    a. Depth to water in well
    b. Field parameters of purged water, including temperature, pH, and electrical conductance; depending on the project, it may be necessary to measure additional parameters such as ORP, dissolved oxygen, and/or turbidity
    c. Pumping rate (via flowmeter or manual measurements)
    d. For each set of measurements, note sample color and clarity/sediment content

Monitor wells should be purged, at a minimum, the equivalent of three times the bore volume of standing water or continue evacuating water until specific conductance, temperature, and pH stabilize. The bore volume of water present in each well shall be computed based on the length of water column in the well casing and include the volume of water in the filter pack. The casing water volume shall be computed using the following formula given below:

$$\mathbf{V_c = (0.041)d_c \times d_c \times h}$$

Where: V_c = casing water volume in gallons, d_c = casing diameter in inches, h = height of the water column from the well bottom in feet.

The filter pack water volume shall be computed using the following formula:

$$\mathbf{V_f = [((0.041)d_b \times d_b \times l) - ((0.041)d_c \times d_c \times l)] \times 0.28}$$

Where: V_f = volume in gallons, d_b = bore diameter in inches, d_c = casing diameter in inches, l = length of saturated filter pack in feet, and 0.28 = approximate porosity of coarse sand.

The total bore volume of water is then computed as $V_c + V_f$.

4. Field notes should reflect the single well volume calculations or determinations that clearly identify the purge volume goal. Dividing 3 wellbore volumes (in gallons) by the average pumping rate (in gpm) will yield the minimum number of minutes of pumping.
5. Stabilization should be achieved within 3 well volumes, if not continue until it does or to a maximum of 5 well volumes. The field project manager should determine when the most representative sample can be obtained based upon available site information.
6. With respect to ground water chemistry the following conditions can generally be used to determine purge stabilization: Stabilization is defined as 3 consecutive readings where

GROUNDWATER SAMPLING PROCEDURES (continued)

all of the following apply:

- pH varies ≤ 0.1 unit
- Electrical conductance varies ≤ 3%
- Temperature varies ≤ 0.2ºC

7. In some instances a well may be pumped or bailed dry (evacuated). When this occurs, the well can be assumed to be adequately purged and the well can be sampled following sufficient recovery (enough volume to allow filling all sample containers). Sampling must commence as soon as possible after purging and within 24 hours of pumping or bailing the well dry. If there is not an adequate volume available, sampling should occur as soon as sufficient volume is achieved. For special occurrences in low permeability settings, low-flow sampling equipment may be used as addressed by project manager.
8. Low-flow sampling may be conducted in wells where the water level in the well during pumping can be maintained within 0.3 feet of the static level. Prior to the start of pumping, the static water level is recorded. Pumping should begin at a relatively slow rate, and drawdown within the well should be minimized. If drawdown in the well exceeds 0.3 ft, the sampling event may continue if the drawdown can be stabilized at less than 0.3 ft of drawdown during pumping. The minimum purge volume should be greater than the drawdown volume of water in the borehole plus the water volume in the discharge line. As with the purge method, pumping should continue until parameters have stabilized prior to collecting a sample.

Sampling:

1. Before initiating sampling, measure and record water quality field parameters a final time. Record these readings on the Traffic Report and Analytical Request Schedule forms.

2. Prepare and label all sample containers prior to filling with water. Be aware that for large sample suites (e.g., sampling for analysis of drinking water standards), you may need up to an hour for labeling and preparation. **As part of QA/QC standards, do not label or identify sample containers with the site or well identifier**. Use the M&A sample ID stickers or write a unique four-digit code on the label provided by the lab. If M&A sample ID stickers are not available, create your own code such as: 060711A (the date "6July2011" and "A" for first sample). Cross reference this information to the sampling site in your field notes.

3. Make sure each sample label contains, at a minimum:

    a. Sample ID
    b. Date and Time - use the same time on all sample containers for each sample even if sampling occurs over a length of time; use the expected midpoint as the time for all labels.
    c. Sampler's Initials
    d. Montgomery & Associates
    e. Type of Sample
    f. Type of Preservative, if applicable

    Label samples using indelible ink such a ball-point pen. Permanent markers can be used in some situations, but should be avoided if sampling for VOCs. Never use pencils or ink that will dissolve, bleed, or smudge when wet.

An example sample label follows:

Trace Constituents/Metals/Cations Example Laboratories 5555 Denargo Drive (800) 555-5555 Denver, CO 80808 Fax (555) 555-5555
Company: **Montgomery & Associates** Sample ID: **4136** Sample Date: **06 July 2011** Time: **15:45** Sampler: **DRG**
Inorganic Preservative: HNO3 ☒ HCL ☐ NaOH ☐ H2SO4☐ Zn Acetate ☐
Organic Analysis: VOA ☐ BNA ☐ BTX ☐ TPH ☐ Pests ☐ PCB ☐ Herbs ☐
Sample Type: Raw ☐ Filtered ☒ Solid ☐

4. Cover the sample labels with clear packing tape to prevent them from getting wet.

5. Check each sample container and determine the specifics for each type. Review lab instructions for each sample container before filling. Know before sampling which containers have preservative, which containers/vials have to be filled with no air space (e.g., VOA vials), and which containers need filtering. Review lab instructions for each sample. Check sample documentation provided by the lab for any questions.

    g. For inorganic and radiochemical samples, do not overfill containers. This will avoid the spilling and potential contact with the harsh chemicals that are typically used as preservatives. Samples are also less likely to leak during shipment if they have some headspace.

    h. For VOCs, SVOCs, and other organic samples, use a very low flow to fill these containers so that entrapment of bubbles is avoided or minimized. Completely fill the containers so that a convex meniscus bulges from the neck of the container. Place the cap on the container so that the container has zero headspace or entrapped air. After capping, tip each sample container upside down and shake it to ensure no headspace (bubbles) exist. An air bubble less than 2 mm in diameter is acceptable. If an air bubble is larger than 2 mm in diameter, empty and refill the container or if the bottle contained preservative, replace it with a pre-preserved container.

    i. Filtered Samples: To filter samples, use one of the following methods:

        - If sampling from a hose bib (spigot), attach a hose bib adaptor into which you can thread the filter. Water can then flow directly from the filter into the sample container.

        - If a hose bib (spigot) or adaptor is not available, place water into a

clean holding container, insert clean flexible tubing from the peristaltic pump into the holding container, attach a filter to the opposite end of the flexible tubing, and use a peristaltic pump to push the sample water through the tubing/filter and into the sample container. Note the flow direction indicated on the peristaltic pump, and if necessary, switch the direction as necessary to ensure the water flows toward the filter and sample container. Adjust the flow rate of the peristaltic pump so that the filter pressure does not exceed the filter specifications; disk filters have a maximum allowable pressure of 14 psi and capsule filters have a maximum allowable pressure of 40-60 psi.

To avoid contamination, do not touch the ends of the filter housing. Always allow water to flow through the filter for approximately 30 seconds before filling containers. Dispose of filters after they have been used once; never reuse them at a different sample site. If the water being sampled contains excessive sediment, it may be necessary to use more than one filter per sample source. Upon completion of filtering, decontaminate and rinse all adaptor and/or pump components that came into contact with sample water.

6. On the field sample forms, record all pertinent detailed field notes, including but not limited to: date and time; site and/or well name; sample identifier; pumping rate; type of pump; depth to pump intake; water disposal method; site conditions; weather; sample collection method; analytical suite; lab name; and water level.

7. If using a sample pump (or any other sampling equipment) it is generally necessary to obtain an equipment blank. Use distilled or deionized water to fill all containers of the sample suite. The distilled / deionized water should be run through the sampling equipment (i.e. the sample pump) in the same exact manner as all other sample water. Distilled or deionized water can be obtained from the laboratory or at most grocery stores.

8. Trip blanks, field blanks, field replicates (duplicates), shipping container temperature blanks may also be part of sampling plans. Consult scope of work and project QA/QC protocols for specific directives.

9. Avoid contamination of samples. Wear clean latex or Nitrile gloves when handling and filling the sample containers. Do not touch the lip of sample containers or allow the lip of the sample container to touch the spigot or sample port. Do not touch the inside of the container caps or allow dirt, dust, rain, or other potential contaminants to come in contact with them; when filling bottles, set the caps aside, face down, on a clean secure surface. All sample containers provided by laboratories are sanitized and ready to be filled without rinsing. Sample containers which are not provided by the laboratory, if necessary, should be rinsed three (3) times with the water to be sampled, before being filled (caps should be rinsed also).

10. Make sure all sample container caps are securely tightened and all sample labels are correct and complete. Never use electrical tape to seal VOC or SVOC samples. Date, sign, and place custody seals on the containers and over the caps to prevent (or provide evidence of) tampering with samples after collection.

11. Store sample containers in coolers with ice or ice packs (at $\leq 4^{o}C$), until they can be

unloaded into a refrigerator. Make sure a temperature blank is also stored in cooler. In general, all samples should be stored on ice or refrigerated until received by the laboratory, unless laboratory instructions indicate otherwise.

12. If sampling at a new site or well, take photographs to document site conditions and features, including sampling port, discharge assembly, water disposal, weather, and other pertinent features. Also, record well location using hand-held GPS unit.

Decontamination: When sampling for organic compounds or other constituents in trace concentrations, decontamination protocols should be part of QA/QC protocols. Consider that for non-dedicated purging equipment, decontamination procedures should be implemented after use to ensure against cross-contamination from one well to the next well. If a bailer is used to purge the well, use dedicated one time use bailers. If not, develop protocols for cleaning, such as using detergent solution and washing of all interior surfaces. Detergent solution should be disposed of properly. After using detergent, completely rinse exterior and interior surfaces three times with distilled water. Sometimes a specific solvent (typically isopropyl alcohol or methanol), can be used in the cleaning process after rinsing with distilled water. The wetted or contaminated portion of braided nylon or cotton cord on the bailer can be cut and disposed of.

If a portable submersible pump is used, pump distilled water through immediately after use. Wash or wipe the exterior surface of the pump hose with a detergent solution and rinse or wipe three times with distilled water. Air dry before reuse.

Shipping and Delivery:

1. Fill out all necessary Chain of Custody forms, including Traffic Reports and Analysis Request Schedules. Make sure all sample documentation matches sample IDs, dates, and times recorded on container labels and in field notes. Each lab will require its own Chain of Custody detailing required analysis. Draw a diagonal line through all unused or inapplicable portions of the Chain of Custody forms.

2. Deliver or ship all sample containers according to their laboratory instructions by placing them in insulated shipping containers (coolers) to maintain sample temperatures of ≤4ºC. In general, shipping ice should be prepared in advance by filling the ice containers furnished by the laboratory and placing them in a freezer. If ice cubes or nuggets are used to maintain temperatures during shipment, place it in resealable bags to prevent it from spilling in the event the shipping container is breached during shipment. Do not place loose ice in the shipping containers. Place at least 2 custody seals on each shipping container to prevent (or provide evidence of) sample tampering. Place clear shipping tape over the custody seals to ensure they do not come off during shipment. Finally, tape each shipping container shut by looping the packing tape around completely around the lid and body of the container and back on itself. Make sure that all samples are delivered or shipped to laboratories before holding times are exceeded.

3. The laboratory documents (chain of custody/analytical request schedules) should be signed by the sampler (and/or sample custodian) along with the date and time the samples were relinquished (delivered or shipped). If substantial space remains in any of the shipping containers after the ice and samples are packed, fill the space with clean packing material such as bubble wrap or packing "peanuts". Place the laboratory documents into a watertight, resealable bag in the uppermost part of the shipping container(s), on top of the ice and packing material. Photocopy and scan the signed

	laboratory documents and retain for sample records. 4. If samples are being shipped from the office by a service such as Federal Express, coordinate shipping arrangements with M&A clerical staff: a. Check with M&A clerical staff to confirm there is ample time to prepare and ship samples that day. If not, store samples in an appropriate refrigerator, and then prepare and ship sample containers the following work day. b. Weigh each shipping container using scales in the front office. Notify clerical staff of the weight(s) of each container c. Notify clerical staff of the project number, the lab(s) the samples are being shipped to, the lab address(es), the shipping option desired (e.g. overnight or 2-day), and the weight of each shipping container. 5. Copies of sampling documentation should be distributed as follows: a. QA/QC Administrator – Original traffic reports, carbon copy of COCs b. EnviroData Manager – Photocopies of traffic reports, station data with UTM coordinates if new sampling location c. Project Sampling Manager – Photocopies of traffic reports, COCs, other sample tracking documents (e.g. sample number cards) d. Well/Site activity notebook or folder – Photocopies of sampling notes, traffic reports, COCs, and other sample tracking documents (e.g. sample number cards) 6. If you are shipping samples from the field to the M&A office for processing and distribution (e.g. isotope sample sets): a. Copy or scan all documents and retain a copy prior to shipping (in case paperwork is lost or damaged in transport). Provide labeled sets of copies as above in shipment. b. Follow normal shipping guidelines for icing, signing off on COCs, and attaching custody seals. c. **Email project manager, QA/QC administrator, EnviroData manager, and technical staff with sample details, estimated time of arrival, and shipping schedule. To the extent possible, utilize and direct hydrologic technicians to conduct the necessary processing and preparation of samples, shipment, and distribution of sample documents.**
FORM(S) REQ'D	See text
REPORTING PROCEDURE	See text
TOOLS REQ'D	See text
REVISION FREQUENCY	Annual
WRITTEN BY	Field Managers
REVIEWED BY	D. Weber
SOP FILENAME	SOP_2.05_GroundwaterSampling_Rev0
FILE LOCATION	s:\Admin\SOPs_Standard_Operating_Procedures
REFERENCES	American Society for Testing and Materials, 1999, *ASTM Standards on Design, Planning, and Reporting of Ground Water and Vadose Zone Investigations:* Sponsored by ASTM Committee D-18 on Soil and Rock, Second Edition, July 1999. American Society for Testing and Materials, 1996, *ASTM Standards on Ground Water and Vadose Zone Investigations: Drilling, Sampling, Well Installation and Abandonment*

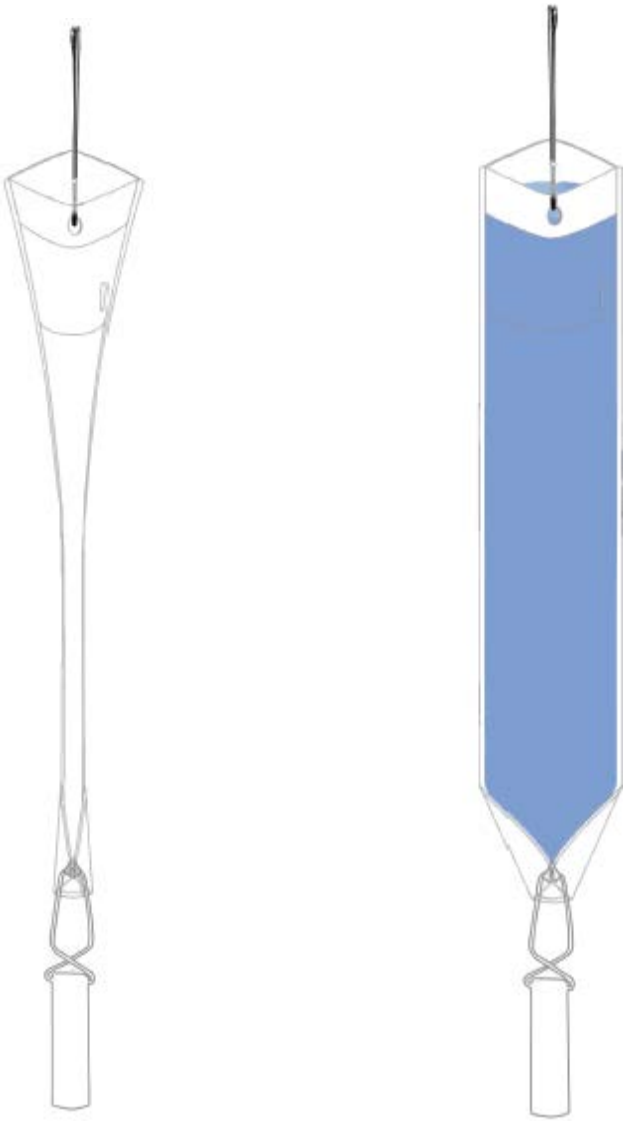
	Procedures: Sponsored by ASTM Committee D-18 on Soil and Rock, April 1996. Lurry, D.L. and C.M. Kolbe, 2000, *Interagency Field Manual for the Collection of Water-Quality Data:* U.S. Department of the Interior and the U.S. Geological Survey, Open-File Report 00-213. Wilde, F.D., D.B. Radtke, J. Gibs, and R.T. Iwatsubo, 1998, *National Field Manual for the Collection of Water-Quality Data, Book 9, Handbooks for the Water-Resources Investigations*: U.S. Department of the Interior and the U.S. Geological Survey, Chapter A1: Preparations for Water Sampling.

HYDRASleeve™

Simple by Design

US Patent No. 6,481,300; No. 6,837,120 others pending

Standard Operating Procedure: Sampling Groundwater with a HydraSleeve



This guide should be used in addition to field manuals and instructions appropriate to the chosen sampling device (i.e., HydraSleeve, SpeedBag or Super/Skinny Sleeve and W3 HybridSleeve).

Find the appropriate field manual and instructions on the HydraSleeve website at http://www.hydrasleeve.com.

For more information about the HydraSleeve, or if you have questions, contact:
GeoInsight, P.O. Box 1266, Mesilla Park, NM 88047
800-996-2225, info@hydrasleeve.com.

Table of Contents

Introduction

The HydraSleeve is classified as a no-purge (passive) grab sampling device, meaning that it is used to collect groundwater samples directly from the screened interval of a well without having to purge the well prior to sample collection. When it is used as described in this Standard Operating Procedure (SOP), the HydraSleeve causes no drawdown in the well (until the sample is withdrawn from the water column) and only minimal disturbance of the water column, because it has a very thin cross section and it displaces very little water (<100 ml) during deployment in the well. The HydraSleeve collects a sample from within the screen only. It excludes water from any other part of the water column in the well through the use of a self-sealing check valve at the top of the sampler. It is a single-use (disposable) sampler that is not intended for reuse, so there are no decontamination requirements for the sampler itself.

The use of no-purge sampling as a means of collecting representative groundwater samples depends on the natural movement of groundwater (under ambient hydraulic head) from the formation adjacent to the well screen through the screen. Robin and Gillham (1987) demonstrated the existence of a dynamic equilibrium between the water in a formation and the water in a well screen installed in that formation, which results in formation-quality water being available in the well screen for sampling at all times. No-purge sampling devices like the HydraSleeve collect this formation-quality water as the sample, under undisturbed (non-pumping) natural flow conditions. Samples collected in this manner generally provide more conservative (i.e., higher concentration) values than samples collected using well-volume purging, and values equivalent to samples collected using low-flow purging and sampling (Parsons, 2005).

Applications of the HydraSleeve

The HydraSleeve can be used to collect representative samples of groundwater for all analytes (volatile organic compounds [VOCs], semi-volatile organic compounds [SVOCs], common metals, trace metals, major cations and anions, dissolved gases, total dissolved solids, radionuclides, pesticides, PCBs, explosive compounds, and all other analytical parameters). Designs are available to collect samples from wells from 1” inside diameter and larger. The HydraSleeve can collect samples from wells of any yield, but it is especially well-suited to collecting samples from low-yield wells, where other sampling methods can’t be used reliably because their use results in dewatering of the well screen and alteration of sample chemistry (McAlary and Barker, 1987).

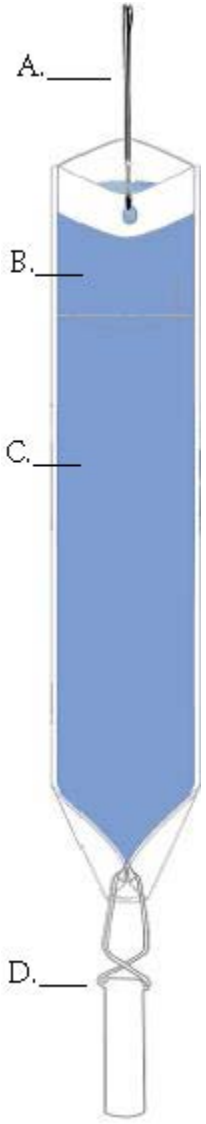
The HydraSleeve can collect samples from wells of any depth, and it can be used for single-event sampling or long-term groundwater monitoring programs. Because of its thin cross section and flexible construction, it can be used in narrow, constricted or damaged wells where rigid sampling devices may not fit. Using multiple HydraSleeves deployed in series along a single suspension line or tether, it is also possible to conduct in-well vertical profiling in wells in which contaminant concentrations are thought to be stratified.

As with all groundwater sampling devices, HydraSleeves should not be used to collect groundwater samples from wells in which separate (non-aqueous) phase hydrocarbons (i.e., gasoline, diesel fuel or jet fuel) are present because of the possibility of incorporating some of the separate-phase hydrocarbon into the sample.

Description of the HydraSleeve

The basic HydraSleeve (Figure 1) consists of the following components*:

- A suspension line or tether (A.), attached to the spring clip or directly to the top of the sleeve to deploy the device into and recover the device from the well. Tethers with depth indicators marked in 1-foot intervals are available from the manufacturer.
- A long, flexible, 4-mil thick lay-flat polyethylene sample sleeve (C.) sealed at the bottom (this is the sample chamber), which comes in different sizes, as discussed below with a self-sealing reed-type flexible polyethylene check valve built into the top of the sleeve (B.) to prevent water from entering or exiting the sampler except during sample acquisition.
- A reusable stainless-steel weight with clip (D.), which is attached to the bottom of the sleeve to carry it down the well to its intended depth in the water column. Bottom weights available from the manufacturer are 0.75" OD and are available in a variety of sizes. An optional top weight may be attached to the top of the HydraSleeve to carry it to depth and to compress it at the bottom of the well (not shown in Figure 1);
- A discharge tube that is used to puncture the HydraSleeve after it is recovered from the well so the sample can be decanted into sample bottles (not shown).
- Just above the self-sealing check valve at the top of the sleeve are two holes which provide attachment points for the spring clip and/or suspension line or tether. At the bottom of the sample sleeve are two holes which provide attachment points for the weight clip and weight.



*Other configurations such as top weighted assemblies, Super/SkinnySleeves, Speedbags, and W3 Hybrids are available.

Note: The sample sleeve and the discharge tube are designed for one-time use and are disposable. The spring clip, weight and weight clip may be reused after thorough cleaning. Suspension cord is generally disposed after one use although, if it is dedicated to the well, it may be reused at the discretion of the sampling personnel.

Selecting the HydraSleeve Size to Meet Site-Specific Sampling Objectives

It is important to understand that each HydraSleeve is able to collect a finite volume of sample because, after the HydraSleeve is deployed, you only get one chance to collect an undisturbed sample. Thus, the volume of sample required to meet your site-specific sampling and analytical requirements will dictate the size of HydraSleeve you need to meet these requirements.

Table 1. Dimensions and Volumes of HydraSleeve Models.

Diameter	Volume	Length	Lay-Flat Width	Filled Dia.
2-Inch HydraSleeves				
Standard 600 mls HydraSleeve	~600mls	30”	2.5”	1.4”
Standard 1-liter HydraSleeve	~1 Liter	38”	3”	1.9”
Super/SkinnySleeve 1-liter	~1 Liter	38”	2.5"	1.5”*
Super/SkinnySleeve 1.5-liter	~1.5 Liters	52”	2.5”	1.5”*
Super/SkinnySleeve 2-liter	*~2 Liters*	*66"*	*2.5"*	*1.5"**
4-Inch HydraSleeves				
Standard 2.5 liter	~2 Liters	38”	4”	2.7”

* outside diameter on the Heavy Duty Universal Super/SkinnySleeves is 1.5" however when using with schedule 40 hardware the O.D. of the assembly will be 1.9"

It's also recommended that you size the diameter of the HydraSleeve according to the diameter of the well (i.e. use 2-inch HydraSleeves in 2-inch wells). Using smaller sleeves in larger diameter wells (i.e. 2-inch HydraSleeves in 4-inch wells) will result in a longer fill rate and will require special retrieval instructions (explained later).

The volume of sample collected by the HydraSleeve varies with the diameter and length of the HydraSleeve. Dimensions and volumes of available HydraSleeve models are detailed in Table 1.

HydraSleeves can be custom-fabricated by GeoInsight in varying diameters and lengths to meet specific volume requirements. HydraSleeves can also be deployed in series (i.e., multiple HydraSleeves attached to one tether) to collect additional sample to meet specific volume requirements, as described below.

If you have questions regarding the availability of sufficient volume of sample to satisfy laboratory requirements for analysis, it is recommended that you contact the laboratory to discuss the minimum volumes needed for each suite of analytes. Laboratories often require only 10% to 25% of the volume they specify to complete analysis for specific suites of analytes, so they can often work with much smaller sample volumes that can easily be supplied using a HydraSleeve.

HydraSleeve Deployment

Information Required Before Deploying a HydraSleeve

Before installing a HydraSleeve in any well, you will need to know the following:

- The inside diameter of the well
- The length of the well screen
- The water level in the well
- The position of the well screen in the well
- The total depth of the well

The inside diameter of the well is used to determine the appropriate HydraSleeve diameter for use in the well. The other information is used to determine the proper placement of the HydraSleeve in the well to collect a representative sample from the screen (see HydraSleeve Placement, below), and to determine the appropriate length of tether to attach to the HydraSleeve to deploy it at the appropriate position in the well.

Most of this information (with the exception of the water level) should be available from the well log; if not, it will have to be collected by some other means. The inside diameter of the well can be measured at the top of the well casing, and the total depth of the well can be measured by sounding the bottom of the well with a weighted tape. The position and length of the well screen may have to be determined using a down-hole camera if a well log is not available. The water level in the well can be measured using any commonly available water-level gauge.

HydraSleeve Placement

The HydraSleeve is designed to collect a sample directly from the well screen. It fills by pulling it up through the screen a distance equivalent to the length of the sampler when correctly sized to the well diameter. This upward motion causes the top check valve to open, which allows the device to fill. To optimize sample recovery, it is recommended that the HydraSleeve be placed in the well so that the bottom weight rests on the bottom of the well and the top of the HydraSleeve is as close to the bottom of the well screen as possible. This should allow the sampler to fill before the top of the device reaches the top of the screen as it is pulled up through the water column, and ensure that only water from the screen is collected as the sample. In short-screen wells, or wells with a short water column, it may be necessary to use a top-weight on the HydraSleeve to compress it in the bottom of the well so that, when it is recovered, it has room to fill before it reaches the top of the screen.

Example

2" ID PVC well, 50' total depth, 10' screen at the bottom of the well, with water level above the screen (the entire screen contains water).

Correct Placement (figure 2): Using a standard HydraSleeve for a 2" well (2.5" flat width/1.5" filled OD x 30" long, 600 ml volume), deploy the sampler so the weight (a 5 oz., 2.5" long weight with a 2" long clip) rests at the bottom of the well. The top of the sleeve is thus set at ~34" above the bottom of the well. When the sampler is recovered, it will be pulled upward approximately 30" before it is filled; therefore, it is full (and the top check valve closes) at approximately 64" (5.3 feet) above the bottom of the well, which is well before the sampler reaches the top of the screen. In this example, only water from the screen is collected as a sample.

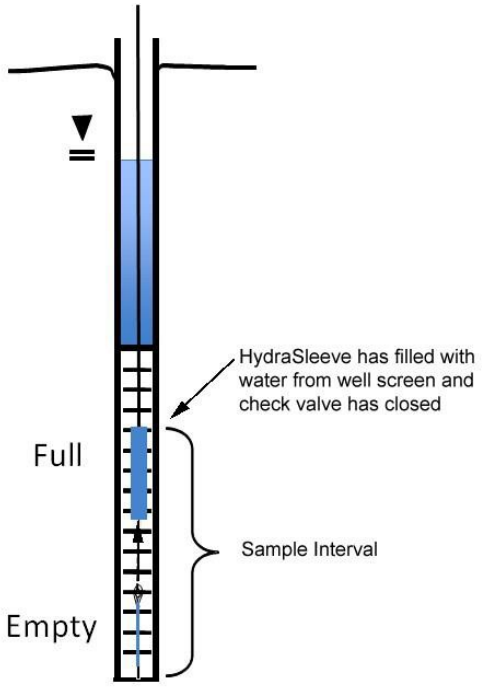


Figure 2. Correct Placement of HydraSleeve.

Incorrect Placement (figure 3): If the well screen in this example was only 5' long, and the HydraSleeve was placed as above, it would not fill before the top of the device reached the top of the well screen, so the sample would include water from above the screen, which may not have the same chemistry.

The solution? Deploy the HydraSleeve with a top weight, so that it is collapsed to within 6" of the bottom of the well. When the HydraSleeve is recovered, it will fill within 36" (3 feet) from the bottom of the well, or 2-feet before the sampler reaches the top of the screen, so it collects only water from the screen as the sample.

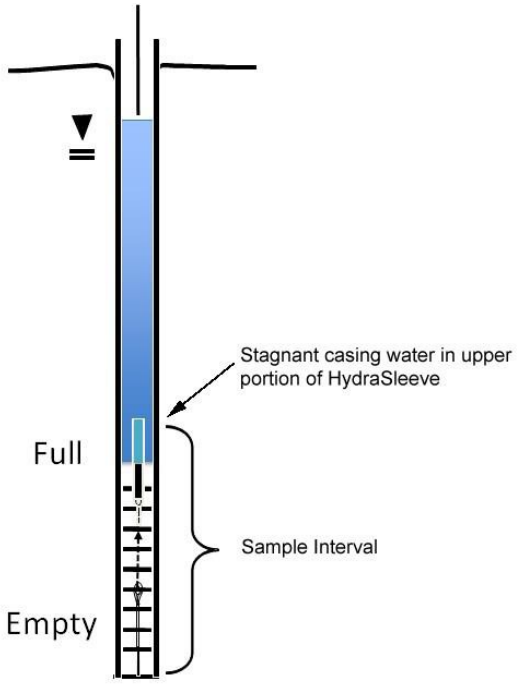


Figure 3. Incorrect placement of HydraSleeve.

This example illustrates one of many types of HydraSleeve placements. More complex placements are discussed in a later section.

NOTE: Using smaller diameter HydraSleeves (2-inch) in larger diameter wells (4-inch) causes a slower fill rate. Special retrieval methods are necessary if this is your set up (shown later in this document).

Procedures for Sampling with the HydraSleeve

Collecting a groundwater sample with a HydraSleeve is usually a simple one-person operation.

Note: Before deploying the HydraSleeve in the well, collect the depth-to-water measurement that you will use to determine the preferred position of the HydraSleeve in the well. This measurement may also be used with measurements from other wells to create a groundwater contour map. If necessary, also measure the depth to the bottom of the well to verify actual well depth to confirm your decision on placement of the HydraSleeve in the water column.

Measure the correct amount of tether needed to suspend the HydraSleeve in the well so that the weight will rest on the bottom of the well (or at your preferred position in the well). Make sure to account for the need to leave a few feet of tether at the top of the well to allow recovery of the sleeve.

Note: Always wear sterile gloves when handling and discharging the HydraSleeve.

I. Assembling the Basic HydraSleeve*

1. Remove the HydraSleeve from its packaging, unfold it, and hold it by its top.
2. Crimp the top of the HydraSleeve by folding the hard polyethylene reinforcing strips at the holes.
3. Attach the spring clip to the holes to ensure that the top will remain open until the sampler is retrieved.
4. Attach the tether to the spring clip by tying a knot in the tether.

Note: Alternatively, if spring clips are not being utilized, attach the tether to one (NOT both) of the holes at the top of the Hydrasleeve by tying a knot in the tether.

5. Fold the flaps with the two holes at the bottom of the HydraSleeve together to align the holes and slide the weight clip through the holes.
6. Attach a weight to the bottom of the weight clip to ensure that the HydraSleeve will descend to the bottom of the well.

*See Super/SkinnySleeve assembly manual and HydraSleeve Field Manual for other assembly instructions.

II. Deploying the HydraSleeve

1. Using the tether, carefully lower the HydraSleeve to the bottom of the well, or to your preferred depth in the water column

   During installation, hydrostatic pressure in the water column will keep the self-sealing check valve at the top of the HydraSleeve closed, and ensure that it retains its flat, empty profile for an indefinite period prior to recovery.

Note: Make sure that it is not pulled upward at any time during its descent. If the HydraSleeve is pulled upward at a rate greater than 0.5'/second at any time prior to recovery, the top check valve will open and water will enter the HydraSleeve prematurely.

2. Secure the tether at the top of the well by placing the well cap on the top of the well casing and over the tether.

Note: Alternatively, you can tie the tether to a hook on the bottom of the well cap (you will need to leave a few inches of slack in the line to avoid pulling the sampler up as the cap is removed at the next sampling event).

III. Equilibrating the Well

The equilibration time is the time it takes for conditions in the water column (primarily flow dynamics and contaminant distribution) to restabilize after vertical mixing occurs (caused by installation of a sampling device in the well).

- Situation: The HydraSleeve is deployed for the first time or for only one time in a well

  The basic HydraSleeve is very thin in cross section and displaces very little water (<100 ml) during deployment so, unlike most other sampling devices, it does not disturb the water column to the point at which long equilibration times are necessary to ensure recovery of a representative sample.

  In some cases, like when useing the SpeedBags, the HydraSleeve can be recovered immediately (with no equilibration time) or within a few hours. In regulatory jurisdictions that impose specific requirements for equilibration times prior to recovery of no-purge sampling devices, these requirements should be followed.

  NOTE: If using top weights additional equilibration time is needed to allow the top weight time to compress the HydraSleeve into the bottom of the well.

- Situation: The HydraSleeve is being deployed for recovery during a future sampling event.

  In periodic (i.e., quarterly, semi-annual, or annual) sampling programs, the sampler for the current sampling event can be recovered and a new sampler (for the next sampling event) deployed immediately thereafter, so the new sampler remains in the well until the next sampling event.

Thus, a long equilibration time is ensured and, at the next sampling event, the sampler can be recovered immediately. This means that separate mobilizations, to deploy and then to recover the sampler, are not required. HydraSleeves can be left in a well for an indefinite period of time without concern.

IV. HydraSleeve Recovery and Sample Collection

1. Hold on to the tether while removing the well cap.
2. Secure the tether at the top of the well while maintaining tension on the tether (but without pulling the tether upwards)
3. Measure the water level in the well.
4. Use one of the following 3 retrieval methods. In all 3 scenarios, when the HydraSleeve is full, the top check valve will close. You should begin to feel the weight of the HydraSleeve on the tether and it will begin to displace water. The closed check valve prevents loss of sample and entry of water from zones above the well screen as the HydraSleeve is recovered.

   a. In one smooth motion, pull the tether up 30"-60" (the length of the sampler) at a rate of about 1 foot per second (or faster). The motion will open the top check valve and allow the HydraSleeve to fill (it should fill in about 1:1 ratio or the length of the HydraSleeve if the sleeve is sized to fit the well). This is analogous to coring the water column in the well from the bottom up.

   b. There are times it is recommended that the HydraSleeve be oscillated in the screen zone to ensure it is full before leaving the screen area. Pull up 1-3 feet, let the sleeve assembly drop back down and repeat 3-5 times before pulling the sleeve to the surface. The collection zone will be the oscillation zone. ***When in doubt use this retrieval method.***

   c. SpeedBags require check valve activation and oscillation during recovery: When retrieving the SpeedBag, pull up hard 1-2 feet to open the check valve; let the assembly drop back down to the starting point; REPEAT THIS PROCESS 4 TIMES; and then quickly recover the SpeedBag through the well sceen to the surface.

5. Continue pulling the tether upward until the HydraSleeve is at the top of the well.
6. Discard the small volume of water trapped in the Hydrasleeve above the check valve by pinching it off at the top under the stiffeners (above the check valve).

v. Sample Discharge

NOTE: Sample collection should be done immediately after the HydraSleeve has been brought to the surface to preserve sample integrity.

Be sure you have discarded the water sitting above the check valve – see step #6 above.

1. Remove the discharge tube from its sleeve.
2. Hold the HydraSleeve at the check valve
3. Puncture the HydraSleeve at least 3-4 inches below the reinforcement strips with the pointed end of the discharge tube. NOTE: For some contaminants (VOC's/sinkers) the best location for discharge is the middle to bottom of the sampler. This would be representative of the deeper portion of the well screen.
4. Discharge water from the HydraSleeve into your sample containers. Control the discharge from the HydraSleeve by either raising the bottom of the sleeve, by squeezing it like a tube of toothpaste, or both.
5. Continue filling sample containers until all are full.

Measurement of Field Indicator Parameters

Field indicator parameter measurement is generally done during well purging and sampling to confirm when parameters are stable and sampling can begin. Because no-purge sampling does not require purging, field indicator parameter measurement is not necessary for the purpose of confirming when purging is complete.

If field indicator parameter measurement is required to meet a specific non-purging regulatory requirement, it can be done by taking measurements from water within a HydraSleeve that is not used for collecting a sample to submit for laboratory analysis (i.e., a second HydraSleeve installed in conjunction with the primary sample collection HydraSleeve [see Multiple Sampler Deployment below]).

Alternate Deployment Strategies

Deployment in Wells with Limited Water Columns

For wells in which only a limited water column needs to be sampled, the HydraSleeve can be deployed with an optional top weight in addition to a bottom weight. The top weight will collapse the HydraSleeve to a very short (approximately 6" to 24") length, depending on the length and volume of the sampler. This allows the HydraSleeve to fill in a water column only 3' to 10' in height (again) depending on the sampler size. Note the SuperSleeves accomplish the same thing but provide greater sample volume at a lower per sample cost.

Multiple Sampler Deployment

Multiple sampler deployment in a single well screen can accomplish two purposes:

1. It can collect additional sample volume to satisfy site or laboratory-specific sample volume requirements.
2. It can be used to collect samples from multiple intervals in the screen to allow identification of possible contaminant stratification.

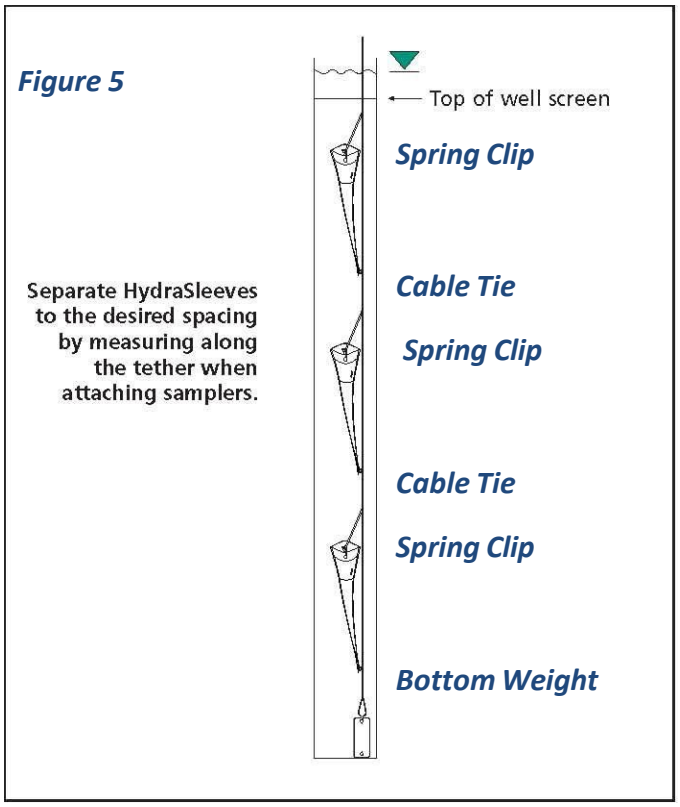


Figure 5. Multiple HydraSleeve deployment

If there is a need for only 2 samplers, they can be installed as follows. The first sampler can be attached to the tether as described above, a second attached to the bottom of the first using your desired length of tether between the two and the weight attached to the bottom of the second sampler (figure 6). This method can only be used with 2 samplers; 3 or more HydraSleeves in tandem need to be attached as described above.

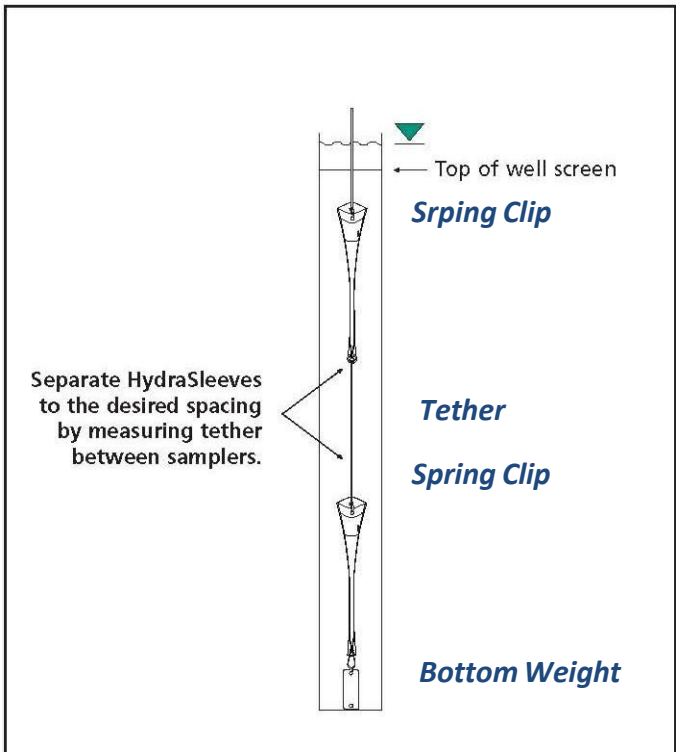


Figure 6. Alternative method for deploying multiple HydraSleeves.

In either case, when attaching multiple HydraSleeves in series, more weight will be required to hold the samplers in place in the well than would be required with a single sampler. Recovery of multiple samplers and collection of samples is done in the same manner as for single sampler deployments.

Post-Sampling Activities

The recovered HydraSleeve and the sample discharge tubing should be disposed as per the solid waste management plan for the site. To prepare for the next sampling event, a new HydraSleeve can be deployed in the well (as described previously) and left in the well until the next sampling event, at which time it can be recovered.

The weight and weight clip can be reused on this sampler after they have been thoroughly cleaned as per the site equipment decontamination plan. The tether may be dedicated to the well and reused or discarded at the discretion of sampling personnel.

References

McAlary, T. A. and J. F. Barker, 1987, Volatilization Losses of Organics During groundwater Sampling From Low-Permeability Materials, groundwater Monitoring Review, Vol. 7, No. 4, pp. 63-68

Parsons, 2005, Results Report for the Demonstration of No-Purge groundwater Sampling Devices at Former McClellan Air Force Base, California; Contract F44650-99-D-0005, Delivery Order DKO1, U.S. Army Corps of Engineers (Omaha District), U.S. Air Force Center for Environmental Excellence, and U.S. Air Force Real Property Agency

Robin, M. J. L. and R. W. Gillham, 1987, Field Evaluation of Well Purging Procedures, groundwater Monitoring Review, Vol. 7, No. 4, pp. 85-93

MONTGOMERY & ASSOCIATES

Appendix B

Quality Assurance/Quality Control Procedures for Water Chemistry Data

MONTGOMERY & ASSOCIATES
Water Resource Consultants

www.elmontgomery.com
1550 East Prince Road
Tucson, AZ 85719
520.881.4912

TECHNICAL MEMORANDUM

DATE: July 5, 2017 **PROJECT #**: 605.1513

TO: Heather Gluski, RESOLUTION COPPER MINING LLC

FROM: Marla Odom, Leilani Bew, and Tim Bayley

PROJECT: RESOLUTION COPPER MINING LLC, PINAL COUNTY, ARIZONA

SUBJECT: SUMMARY OF QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES FOR WATER CHEMISTRY DATA

Introduction

This technical memorandum has been prepared to summarize on-going data review and management procedures for water quality data collected in support of the planned Resolution copper (RC) mine. Montgomery & Associates (M&A) implements procedures to ensure that high quality data is safely stored and readily available for users. M&A carefully tracks and documents sample data through an extensive quality assurance/quality control (QA/QC) review. As data have been collected and analyzed and the scope of the project defined more clearly, the program and associated procedures have been refined accordingly. Once review is complete, verified chemical data are stored in a relational database designed to facilitate accurate and understandable data export and reporting. These procedures provide a clear, auditable trail linking data from original field and laboratory sources to import into the Resolution database.

The purpose of the QA/QC process is to establish a reliable, high quality body of chemical data. Verification of sample documentation ensures that data are associated with the correct sample. Verification of laboratory chemical results provides confidence that the quality, integrity, and usability of the data are maintained.

Database management procedures provide users of the database with a systematic way to store, modify, extract, and manage RC data.

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VERIFICATION OF SAMPLE DOCUMENTATION

Traffic Reports

For each water quality sample, field personnel generate an internal document called a traffic report (TR) that is given to QA/QC and data management staff. RC uses blind field IDs; one very important function of a TR is to correctly record and match the sample location with the blind field ID. Other information on the TR includes specifics of sampling, intended analyses, field parameters, and notable field conditions.

TRs are used in the QA/QC process in a number of ways. They alert both QA/QC and data management staff to the existence of a water quality sample, are used to crosscheck information on the Chain-of-custody (COC), and may be used during the QA/QC process to interpret data.

Chain of Custody Forms

The COC is a legal document that records who had possession of a sample, when they had it, and for how long. This information is checked against traffic reports, and any discrepancies are resolved with field staff. Any changes to the COC are documented in the QA/QC notes and relayed to data management staff and the lab as appropriate. Any special instructions are also noted on the COC.

Sample and Data Tracking

M&A maintains an internal tracking table to follow a sample throughout the QA/QC process from collection to final data storage. When a TR is received from the field, the blind field ID, station name, and sample date/time is used to establish the sample in the table. Following a specific tracking protocol ensures that QA/QC staff is aware of a given sample and what data are expected. Throughout the QA/QC process, it is easy to check the status of a sample with respect to how many labs the sample has been submitted to, when to expect data, if the data have been received, and follow-up with the lab for any specific questions or issues. Built in notifications assure that all aspects of the QA/QC process are completed in an organized and timely manner.

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VERIFICATION OF LABORATORY CHEMICAL RESULTS

Administrative Details

For each lab report, M&A compares the COC with the lab report to ensure consistency between the two. In addition to the sample date/time, M&A checks that the proper analyses were conducted, requested methods were used, project specific reporting limits (RL) or method detection limits (MDL) were met, and that special instructions provided to the laboratory were adhered to.

Historical Data

M&A's evaluation includes comparison of new data to available historical results. If results seem aberrant in any way, the concern is resolved through communication with the lab and/or field personnel until an explanation is found. Because RC has implemented a robust sampling program, there is a substantial body of data in the database for reference and comparison.

If irregularities are found, there are a number of things that are evaluated. Reporting mistakes can account for things like dilution errors or typographical errors. Communication with the lab may identify potential analytic interferences or other lab issues, and all such communication is documented.

Seasonal variations, field conditions, and site activities that could impact the results are all considered. M&A documents all communication with lab and field personnel, and tracks any corrections or re-issues of lab reports as a result of the QA/QC process.

When there is not a plausible explanation for aberrant data, it may be necessary to reanalyze the sample or obtain another field sample for analysis.

Laboratory QC Review

M&A evaluates laboratory chemical results for groundwater and surface water samples in accordance with a compilation of guidelines described in U.S. Environmental Protection Agency (EPA) documents including National Functional Guidelines for Inorganic Superfund Data Review (2010), and Guidance on Environmental Data Verification and Data Validation, (2002). Guidelines have also been adapted from the standard operating procedures (SOPs) of the accredited laboratories that analyze the samples.

Typical laboratory reports include QA/QC analyses conducted by the lab. M&A staff reviews the laboratory QC data provided with each report following an in-house checklist and determines how they reflect upon sample results. M&A's evaluation includes:

- Review the method blank (MB) to rule out possible lab contamination.
- Review the laboratory control standard (LCS) to confirm that percent recoveries meet lab-specific criteria. Analysis of the LCS indicates the accuracy of results.
- Review analysis of matrix spike/matrix spike duplicates (MS/MSD) to confirm that percent recoveries meet lab-specific criteria. Analysis of the MS/MSD addresses precision between the duplicates by calculating the relative percent differences (RPD) between the two. Possible matrix effects are also identified.
- Review laboratory duplicate data. The lab conducts duplicate analyses of a sample, calculates the RPD, and compares them to lab-specific criteria.
- Confirm that necessary laboratory data qualifiers are present and correctly assigned.
- Review the case narrative for accuracy and completeness. For example, the case narrative should address any corrective actions taken by the lab, results of reanalysis, correction of typos, or assignment of updated qualifiers.

Data Relationships

There are certain data relationships that indicate the overall integrity of laboratory data. If any of these relationships appear irregular, M&A communicates with the lab until a resolution is reached. This may include reanalysis of the sample or obtaining another field sample for analysis.

M&A's evaluation includes both qualitative and quantitative comparisons. Relationships such as percent error in the ion balance, the ratio of total dissolved solids (TDS) to specific conductance, comparison of pH to alkalinity, and comparison of total or total recoverable (TRC) results to dissolved results can contribute to overall confidence in the analyses.

Field Duplicates

Part of the QA/QC process in a sampling program is collection, analysis, and interpretation of field duplicate samples. M&A reviews duplicate sample data to assess reproducibility of field and sampling procedures, reproducibility of analytic processes, and to identify systemic issues. Reproducibility between an original sample and a field duplicate is evaluated based on calculation of the RPD for the duplicate pairs. For RC samples, RPD for a field duplicate should be no greater than 20%. If it is, the sample may be reanalyzed

or additional field samples obtained. In the case where matrix interference is suspected, another analytic method may be chosen. This review may result in recommendations for corrective strategies going forth.

Field, Equipment, and Trip Blanks

In addition to field duplicates, other kinds of blanks help address the integrity of the body of data. Any blank can identify possible lab contamination. As the names indicate, these blanks are expected to have non-detects of associated constituents. M&A reviews field blanks to address if ambient conditions or sample practices at a sampling site impact the data. Equipment blanks can identify if sampling equipment is responsible for cross contamination between samples, as well as if sample practices impact the data. Trip blanks identify impacts from shipping and transport, as well as lab contamination.

Laboratory Follow-Up

The purpose of the QA/QC process is to determine the integrity and usefulness of laboratory data. In each phase of M&A's QA/QC review process, the need for follow-up with the lab may arise.

When an aberrant result is reported, good communication between the QA/QC staff and the lab helps resolve the issue, and is documented through QA/QC notes and emails. Actions may include:

- Request additional lab review to determine if there is a reporting error.
- Determine if corroborating data might be available from other analytical methods.
- Assess if the sample condition upon receipt at the lab indicates anything about the results.
- Request re-analysis when no plausible explanation for a questionable result exists.

Data are accepted, qualified, superseded, or rejected based upon the findings of the review.

Documentation of Review Process

The entire QA/QC process and final verification of the results is documented in a number of ways. M&A's documentation procedures include storing original electronic and/or paper copies of all PDFs and electronic data deliverables (EDDs), including those from revised reports. M&A maintains physical notebooks with QA/QC packages for each lab

report. These packages may include TRs and COCs, QA/QC checklists, communication between lab and field staff, and all pertinent QA/QC notes.

Final PDFs are modified to include labels linking field station names to blind IDs and notes describing any rejections and/or supersedes. Qualifiers are assigned as a result of the QA/QC evaluation, and the additional notes explain how these decisions were made, and how any items of concern were resolved. Standardized unambiguous naming conventions ensure version control.

DATA MANAGEMENT

Electronic Data Deliverable File Preparation

The Resolution database is organized into four relational tables that fully describe data within the Resolution project (Sites, Stations, Samples, and Analyses). Data are prepared for import in files that enforce the data integrity of those four relational tables within the database. The file structure used for import is called an EDD, and is usually provided by the laboratory. For RC, M&A requests EDDs in Data Transfer Standard version 2008 (DTS 2008) or greater (Geotech, 2008).

Establishing Samples

Sampling personnel provide sample parameters such as pH, temperature, and electrical conductivity on the TR for each sample. M&A manually prepares an EDD in DTS 2008 format for import into the database for these data. This information is used to correctly establish the sample within the database, allowing other data to be attached to it when imported. Data management staff confirms that the station and site corresponding to the sample exist within the database prior to file import.

EDDs Provided Directly By Laboratory

Several of the labs routinely used on the RC project provide data in both PDF and EDD formats. The EDDs may be provided in the DTS 2008 format or in a format specific to the laboratory. Using a detailed checklist prepared by M&A, EDDS are carefully reviewed for compatibility with database structure, completeness, and reporting preferences. All original EDDs are preserved, but some modification is usually needed before import in order to make the import file consistent with the structure needed for the database and to reflect the results of the data verification process. For example, field sample identifiers are blind to the lab, and therefore sample station associations must always be prepared within the import files after receipt from the lab. EDDS are also modified to identify rejected,

superseded, or qualified data. All final import files are compared to the PDF report to ensure consistency.

Even when EDDS are received in DTS 2008 format, M&A may need to make some modifications. EDDs are checked for missing information, correct assignment of QC codes and data qualifiers, and specifications of appropriate MDLs and reporting RLs.

In addition to the steps described above, EDDs not received in DTS 2008 format require more formatting. Field headings are copied from an import file compatible with the database structure, and data are assigned to correspond with the appropriate field headings.

EDDs Manually Prepared From Laboratory Data

Data is regularly received from laboratories which do not have EDD generation capability, and who provide report results in PDF format only. M&A manually prepares EDDs in DTS 2008 format for these data.

In addition to the steps described above, field headings are copied from an import file compatible with the database structure, and data are hand-entered from field and lab sources to correspond with the appropriate field headings.

Enviro Data Database

M&A has established, implemented, and is maintaining a system and procedure for the systematic control, access, and use of information using Enviro Data, a leading software program for water quality data management (Geotech Computer Systems, Inc). Data records are maintained in secure registries that are easily available for inspection on the M&A server. Data from the RC project has been housed by M&A in this database since 2010. Data collected prior to 2010 was provided to M&A by RC and by Golder Associates (a consultant to RC) in the form of laboratory EDD files and electronic field data files, which were imported into EnviroData in 2010. M&A staff hand-prepared import files for pre-2010 subcontracted lab results that did not have corresponding EDD files from the lab.

Import Process

Once the QA/QC process is complete and EDDs are properly formatted and checked, data are imported into the database. Using Enviro Data import settings, data are imported according to data type, data source, and the level of QA/QC conducted. Enviro Data employs a data cross-checking tool upon import to force consistency between samples already present in the database, and incoming data for the same samples. This cross-check tool eliminates sample duplication within the database, and often identifies inconsistent

sample data, such as erroneous sample date and time assigned by the lab in EDD files. If differences in field headings, constituent names, or method numbers exist, these are identified and standardized using dropdown menus. This forced data consistency keeps the database structurally clean and organized, ensuring database integrity and unambiguous data output.

For each import, Enviro Data generates reports and dialogue boxes. M&A reviews these to confirm that duplicate or superseded results were correctly recognized, and that the expected number of records was imported. After an EDD has been imported, Enviro Data-generated database statistics are compared with an internal tracking table designed by M&A. If disparities are found, problems are identified and corrected.

Import Recordkeeping

In order to maintain the integrity of the database and easily track the progress of data through the QA/QC and import process, M&A maintains careful records. By updating both paper and digital lab-specific notebooks, details of the import are documented.

M&A designed an import-tracking table that keeps a running total of samples and records imported into the database. It also documents the presence of duplicates and supersedes, and the reason why they exist. A hyperlink is included within the tracking table to allow easy access to the final import file.

Routine Database Checks

Comprehensive database checks are conducted routinely to ensure database upload consistency and reporting accuracy. Database checks include, but are not limited to:

- A reported value is present in the database for every detected result (no blank values listed).
- Blank values in the database are checked for validity and that an appropriate corresponding flag is present (such as absent/present, insufficient sample, etc.).
- Non-detect values are checked to ensure that listed values are equal to the RL or MDL, as applicable.
- Any values reporting “<0” are investigated and fixed.
- Lab assigned “j” flags are checked to ensure that reported value is between the RL and MDL.
- Negative values in the database are checked to ensure that values are valid and match laboratory reports.

- Roughly 20% of the total database is checked against laboratory reports at any given time, as a secondary check of database accuracy.

Storage and Security

M&A protects customer data and databases with various technical and procedural measures to maintain information storage and security.

Original Source Data Storage

Data received directly from laboratories and historic data received from the client are stored within a designated file structure on the main M&A company server.

Server Database Storage

M&A databases are stored on a secure Structured Query Language (SQL) server, which has the following measures in place:

- Protection from hazards such as fire, smoke, dust, vibration, chemicals, electrical interference, food, drink, and nearby industrial processes
- Control of temperature and humidity
- Protection of power supply to critical computer equipment
- Restriction of physical access to critical information processing facilities to only authorized individuals
- Storage on a RAID 5 (Redundant Array of Independent Disks) Array that is backed up daily. RAID divides and replicates data among multiple hard disks. Drive failure requires replacement, and the array is not destroyed by a single drive failure.
- Backup tapes are rotated weekly to a climate-controlled, secure facility with an attendant. The storage facility has ground-level windows encased with security bars and access to the building requires check in with the facility attendant. The storage unit's only point of access is a secured door. Backup tapes are stored in anti-static bags and placed in a protective toolbox. The facility is air conditioned and equipped with fire-protection sprinklers. Humidity is regulated with the air conditioning system, anti-static bags, and enclosure in a secured toolbox.

Database Security

M&A database security is maintained using the following measures:

- Relevant security patches, updates, service packs, and configuration changes are kept up to date. As updates become available, they are tested and deployed on production servers.
- A managed firewall is in place to protect the system from security threats.
- Wireless local-area network transmissions are encrypted.
- Logging is implemented on all Critical Network Systems. Incident management procedures are in place at M&A so that unexpected or suspicious activity is investigated and managed in a timely manner by qualified personnel.
- Security incidents are reported immediately to IT support contractor (Connectivity Systems) personnel via requests through M&A's help desk system. Requests are immediately opened, tracked, and completed, with security incidents assigned highest importance.
- Anti-virus and client security applications are deployed to all M&A computers that access client databases. The configuration of the Anti-Virus and client security software maintains the integrity of the approved settings and cannot be changed by unauthorized personnel.
- M&A utilizes Microsoft Active Directory on their own domain to create and manage user accounts and their rights. M&A uses group policies to control who, how, and when the users can network resources. In addition, M&A uses SQL Server security in mixed mode so active directory users or individual accounts created in each database have only the rights needed to connect, manage, or view the data that the user has been authorized to access.
- All M&A staff and IT support contractor (Connectivity Systems) personnel are under Non-Disclosure Agreements (NDA) with M&A.

References Cited

Geotech Computer Systems, Inc., 2008, Data Transfer Standard EnviroData Version 2008.

U.S. Environmental Protection Agency, 2002, *Guidance on Environmental Data Verification and Data Validation (EPA QA/G-8)*, EPA/240/R-02/004, Office of Environmental Information, November 2002.

U.S. Environmental Protection Agency, 2010, *National Functional Guidelines for Inorganic Superfund Data Review*, OSWER 9240 1-51, USEPA-540R-10-011, Office of Superfund Remediation and Technology Innovation, January 2010.

Acronyms & Abbreviations

COC ..... chain-of-custody
DTS 2008 ..... Data Transfer Standard version 2008
EDD ..... electronic data deliverables
EPA ..... U.S. Environmental Protection Agency
LCS ..... laboratory control standard
M&A ..... Montgomery & Associates
MS/MSD ..... matrix spike / matrix spike duplicate
MB ..... method blank
MDL ..... minimum detection limit
NDA ..... non-disclosure agreement
QA/QC ..... quality assurance / quality control
RAID ..... redundant array of independent disks
RC ..... Resolution Copper
RPD ..... relative percent difference
RL ..... reporting limit
SQL ..... structured query language
SOP ..... standard operating procedures
TDS ..... total dissolved solids
TR ..... traffic report
TRC ..... total recoverable

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Appendix C

Field Forms: Water Sampling Record and Equipment Calibration Record

WATER SAMPLING RECORD

PROJECT: _____ DATE: _____

SITE ID: _____ Well / Spring / SW (circle one) SAMPLED BY: _____

WELL/PUMP INFORMATION

Total depth:	_____	feet BGS
Approximate DTW:	_____	feet ALS
Screened interval:	_____	feet BGS
Pump / Intake depth:	_____	feet BTOC
Three bore volumes:		gallons

SAMPLING INFORMATION

Pre-pumping DTW:	_____	feet BTOC
DTW at sampling:	_____	feet BTOC
Purge start time:	_____	hours
Sample collection time:	_____	hours
Approx. vol. purged before sampling:		gallons

Visual inspection of well cap and casing: _____ Good _____ Needs repair (Describe) _____

Time	Depth to Water (ft, bmp)	Temp. (^{o}F)	pH (s.u.)	EC (µS/cm)	ORP (mV)	DO (mg/L)	Turbidity (NTU)	Pump Control #	Pump Rate (gal/min)	Purge Volume (gal)	Comments

Sample ID: RESH_____ Date: _____ Time: _____ Analyses Requested: _____

Duplicate/Blank/Rinsate ID: RESH_____ Date: _____ Time: _____

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Water Resource Consultants

WATER QUALITY EQUIPMENT – CALIBRATION RECORD

Date	Time	MYRON L ULTRAMETER II								HACH 2100Q				Personnel / Signature
		pH Calibration				Specific E.C. Calibration		T Calibration		Turbidity Calibration				
		4.00	7.00	10.00	pH 7 check	1417 (µS/cm)	EC 1417 check (µS/cm)	Check	Probe	20 NTU	100 NTU	800 NTU	100 NTU check	

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Appendix D

Laboratory Chain of Custody Forms

CHAIN OF CUSTODY RECORD

Page ____ of ____

SVL Analytical, Inc. • One Government Gulch • Kellogg, ID 83837 • (208) 784-1258 • FAX: (208) 783-0891

FOR SVL USE ONLY
SVL JOB #

TEMP on Receipt:

Report to Company: _____
Contact: _____
Address: _____
_____
Phone Number: _____
FAX Number: _____
E-mail: _____

Invoice Sent To: _____
Contact: _____
Address: _____
_____
Phone Number: _____
FAX Number: _____
PO#: _____

Table 1. -- Matrix Type
1 = Surface Water, **2** = Ground Water
3 = Soil/Sediment, **4** = Rinsate, **5** = Oil
6 = Waste, **7** = Other_____

Project Name: _____

Sampler's Signature: _____

Indicate State of sample origination: _____ **USACE?** ☐ Yes ☐ No

	Sample ID	Collection			Misc.		Preservative(s)							Analyses Required										Rush Instructions (Days)	Comments
	Please take care to distinguish between: 1 and I 2 and Z 5 and S Ø and O Thanks!	Date	Time	Collected by: (Init.)	Matrix Type (From Table 1)	No. of Containers	Unpreserved	HNO_3 Filtered	HNO_3 Unfiltered	HCl	H_2SO_4	NaOH	Other (Specify)												
1																								
2																								
3																								
4																								
5																								
6																								
7																								
8																								
9																								
10																								

Relinquished by:	Date:	Time:	Received by:	Date:	Time:
Relinquished by:	Date:	Time:	Received by:	Date:	Time:

* Sample Reject: ☐ Return ☐ Dispose ☐ Store (30 Days)

White: LAB COPY Yellow: CUSTOMER COPY

ACZ Laboratories, Inc.

2773 Downhill Drive Steamboat Springs, CO 80487 (800) 334-5493

CHAIN of CUSTODY

Report to:

Name:	Address:
Company:	
E-mail:	Telephone:

Copy of Report to:

Name:	E-mail:
Company:	Telephone:

Invoice to:

Name:	Address:
Company:	
E-mail:	Telephone:

If sample(s) received past holding time (HT), or if insufficient HT remains to complete analysis before expiration, shall ACZ proceed with requested short HT analyses? YES ☐ NO ☐

If "NO" then ACZ will contact client for further instruction. If neither "YES" nor "NO" is indicated, ACZ will proceed with the requested analyses, even if HT is expired, and data will be qualified

Are samples for SDWA Compliance Monitoring? **Yes** ☐ **No** ☐

If yes, please include state forms. Results will be reported to PQL for Colorado.

Sampler's Name: _____ **Sampler's Site Information** **State**_____ **Zip code**_____ **Time Zone**_____

***Sampler's Signature:** _____

***I attest to the authenticity and validity of this sample. I understand that intentionally mislabeling the time/date/location or tampering with the sample in anyway, is considered fraud and punishable by State Law.**

PROJECT INFORMATION

Quote #:

PO#:

Reporting state for compliance testing:

Check box if samples include NRC licensed material? ☐

ANALYSES REQUESTED *(attach list or use quote number)*

SAMPLE IDENTIFICATION	DATE:TIME	Matrix	# of Containers								

Matrix: SW (Surface Water) · GW (Ground Water) · WW (Waste Water) · DW (Drinking Water) · SL (Sludge) · SO (Soil) · OL (Oil) · Other (Specify)

REMARKS

Please refer to ACZ's terms & conditions located on the reverse side of this COC.

RELINQUISHED BY:	DATE:TIME	RECEIVED BY:	DATE:TIME

Chain of Custody Form

Address: Resolution Copper
102 Magma Heights Rd.
Superior, AZ 85173

Phone: 520.689.3411
Email: Tom.white@riotinto.com

PO Number: 3102656919

Laboratory: Beta Analytic

Sample ID	Sample Matrix	Sampling Date	Sampling Time	Analysis Required

Signature	Company	Date	Time
Relinquished by			
Received by			
Relinquished by			
Received by			
Relinquished by			
Received by			

BETA ANALYTIC INC.
DR. M.A. TAMERS AND MR. D.G. HOOD
4985 S.W. 74th COURT
MIAMI, FLORIDA USA 33155
TELE: (01) 305-667-5167 FAX: (01) 305-663-0964
E-MAIL: beta@radiocarbon.com
WEB SITE: http://www.radiocarbon.com

RADIOCARBON SAMPLE DATA SHEET

Please contact us at any time for advice, assistance or discussion of results.

SUBMITTER NAME: _____ **DATE:** _____

ADDRESS: _____

_____

TELEPHONE: _____ **FAX:** _____ **E-MAIL:** _____

METHOD OF PAYMENT: PURCHASE ORDER ___ / CREDIT CARD ___ / CHECK ___ / BANK-WIRE TRANSFER ___

OTHER (SPECIFY) _____ **PURCHASE ORDER #** _____

CREDIT CARD #: _____ **EXP. DATE** _____ **AUTH. CODE** _____

ZIP CODE TO WHERE YOUR CREDIT CARD COMPANY SENDS YOUR BILL: _____

YOUR SAMPLE CODE NUMBER:	| | | | | | | | | | | | _____
	PLEASE CHOOSE ≤ 12 INITIAL CHARACTERS TO APPEAR ON THE DATA REPORT SHEET — ADDITIONAL LABELING IF NEEDED

INSTRUCTIONS TO LABORATORY

TECHNIQUE:	❑ **AMS**	❑ **RADIOMETRIC**
DELIVERY SERVICE:	❑ **STANDARD within 14 BUSINESS DAYS** ❑ **PRIORITY within 6 BUSINESS DAYS** ❑ **TIME-GUIDE 2-3 BUSINESS DAYS**	❑ **STANDARD within 25 BUSINESS DAYS**
ISOTOPE RATIOS	**13C/12C is included in the analysis price** ❑ **15N/14N – bones only** ratio analysis (diet indicator for bones)	

COMPLEX / NON-STANDARD **SERVICES ADDITIONAL FEES APPLY**

- ❑ **EXTENDED COUNTING** – RADIOMETRIC ANALYSIS ONLY – enhanced / optimal precision
- ❑ **MICRO-SAMPLE AMS COUNTING** – AMS ONLY – 100-300 micrograms final carbon
- ❑ **NON-BURNED BONE DATING – COLLAGEN EXTRACTION REQUIRED** – AMS ONLY
- ❑ **CREMATED / BURNED BONE DATING – CARBONATE EXTRACTION REQUIRED** – AMS ONLY
- ❑ **CELLULOSE EXTRACTION** – Non-burned wood and textiles with extreme contamination
- ❑ **SOLVENT EXTRACTIONS** – AMS ANALYSIS ONLY – contamination by varnishes, preservatives, oils, tar, etc.

SAMPLE MATERIAL TYPE: _____ **SAMPLE WEIGHT:** _____

FOR CARBONATE SAMPLES: MARINE _____ **(Delta-R =** _____**+/-**_____**) or FRESH WATER** _____

For Marine carbonates, visit http://calib.qub.ac.uk/marine to get the Delta-R value for the general geographical region of your site.

GENERAL GEOGRAPHIC LOCATION: _____

(REQUIRED FOR CALIBRATION OF CARBONATE SAMPLES – NOT REQUIRED FOR CALIBRATION OF ORGANIC SAMPLES)

(OVER)

FOR ADDITIONAL INFORMATION FROM FRONT PAGE

EVIDENCE OF CONTAMINATION: _____
(ROOT PENETRATION, LEACHING, HUMIC ACIDS, ETC.)

COLLECTION, TREATMENT AND STORAGE PROCEDURES: _____

_____

STRATIGRAPHIC AND ENVIRONMENTAL DETAILS: _____
(PLEASE PUT DRAWINGS AND ADDITIONAL TEXT HERE)

_____

ADDITIONAL INFORMATION

GENERAL SAMPLE SIZE REQUIREMENTS

Smaller quantities than those listed can be analyzed. Size generally does not affect precision for AMS samples but does affect precision for radiometric samples. You are welcome to contact us before sending samples or to send the optimal sample size for your research and ask to be contacted with the best method of analysis.

AMS samples providing less than 300 micrograms final carbon require the Micro-Sample AMS service. Radiometric samples providing less than the optimal 3 grams final carbon are recommended for Extended Counting for enhanced precision and AMS for best precision.

_____

QUANTITIES LISTED PRESUME MATERIALS ARE DRY AND FREE OF ADHERING / ASSOCIATED MATRIX.

Material	AMS	Radiometric
Charcoal	50 milligrams	20 grams
Wood	50 milligrams	50 grams
Dung	50 milligrams	20 grams
Plant, Seeds	20 milligrams	20 grams
Peat / Gyttja	1-2 grams	100 grams
Shell / coral / CaCO3	50 milligrams	50 grams
Organic sediment	2-5 grams	< 1000 grams
Inset (chitin)	50 milligrams	not available
Hair	20-50 milligrams	not available
Fish Otolith	5-10 mg	not available
Bone / Antler	2-10 grams	not available
Teeth	single tooth	not available
Burned / cremated bone	4-40 grams	not available
Phytoliths (extracted)	300 milligrams	not available
Pollen (extracted)	50 milligrams	not available
Forams	20 milligrams	not available
Water DIC as SrC03	50 milligrams	30 grams
Water for DIC extraction	1 liter	not available

QUOTED DELIVERY DATES

You can depend on our commitment to prompt delivery of results. Only in rare instances due to acts of nature, interruption in essential services or other unforeseen circumstances would we anticipate any delay in the meeting of our delivery commitments. Please allow for this in your expectations and contract obligations.

LIMITATION OF DAMAGES – REPAYMENT SERVICE PRICE

It is agreed that in the event of any breach of any warranty or breach of contract, or negligence of Beta Analytic Inc., as well as its agents or representatives, the liability of Beta Analytic Inc., shall be limited to the repayment, to the purchaser (submitter), of the individual analysis price paid by him/her to Beta Analytic Inc. Beta Analytic Inc., shall not be liable for any damages, either direct or consequential.

Stable Isotopes Submission Form

CLIENT DETAILS		METHOD OF PAYMENT	SHIPPING ADDRESSES
Submitter's Name		__ Purchase Order / __ Credit Card	
Affiliation		__ Check / __ Bank Wire Transfer	Beta Analytic Inc. 4985 SW 74th Court Miami, FL 33155 USA Tel: (1) 305-662-7760 Fax: (1) 305-663-0964 Email: lab@radiocarbon.com
Address		Other (Specify):	
Zip Code / Post Code		Purchase Order #	
Country		Credit Card #	International forwarding addresses: Australia - Brazil - China - India Japan - Korea - Europe
Phone number		Expiration Date:	
Fax number		Authorization Code:	For recommended sample sizes, turnaround times and submittal information go to www.radiocarbon.com
Email address		Billing Zip Code:	

General Purpose of Testing

Sample Code (name to be listed on the report)	Sample Material Type	Sample Weight	Analysis Requested				Ready for Measurement? Yes/No	Require Lab Pretreatment? Yes/No	Pretreatment Instructions / Comments
			δ13C	δ15N	δ18O	δD			

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Phone: _____

Email: _____

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Name: _____

Company: _____

Address: _____

Phone: _____

Email: _____

Project: _____

Purchase Order #: _____

Location: _____

Sampled By: _____

Circle one: Standard Priority Rush

Sample Description

Container Number	Sample Identification	Date Sampled	Time	Analysis Requested			Comments

Chain-of-Custody Record

Signature	Company	Date	Time
Relinquished by			
Received by			
Relinquished by			
Received by			
Relinquished by			
Received by			

Weatherford® LABORATORIES

MONTGOMERY & ASSOCIATES

Appendix E

Equipment Manuals and Procedures

ULTRAMETER II™

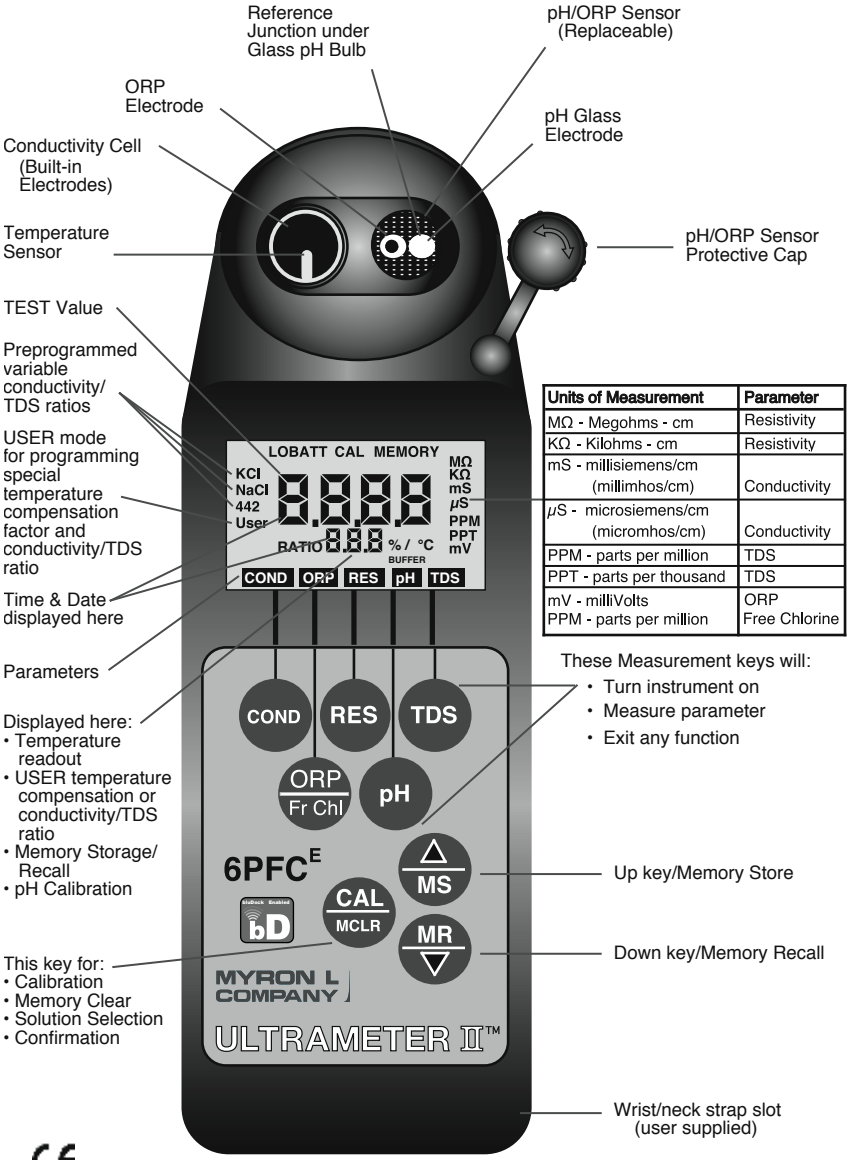
Operation Manual

MODELS 6PFC^{E} & 4P

MYRON L COMPANY
Water Quality Instrumentation
Accuracy • Reliability • Simplicity

20 Sep. 2013

Instrument Illustration



Units of Measurement	Parameter
MΩ - Megohms - cm	Resistivity
KΩ - Kilohms - cm	Resistivity
mS - millisiemens/cm (millimhos/cm)	Conductivity
µS - microsiemens/cm (micromhos/cm)	Conductivity
PPM - parts per million	TDS
PPT - parts per thousand	TDS
mV - milliVolts PPM - parts per million	ORP Free Chlorine

CE

MODEL 6PFC^{E}

Shown with bluDock™ option installed

For detailed explanations see Table of Contents

22nov11

I. INTRODUCTION

Thank you for selecting the feature-packed Ultrameter II™, one of the Myron L Company's latest in an increasing line of instruments utilizing advanced microprocessor-based circuitry and SMT manufacturing processes. This circuitry makes the instrument extremely accurate, reliable and very easy to use.

Model 6PIIFC^{E} includes Myron L Company's exclusive Free Chlorine Equivalent (FC^{E}) feature for making ORP-based free chlorine measurements. Both Ultrameter IIs now also feature optional *Bluetooth®* wireless data transfer. Other features include a clock with time and date, memory of up to 100 locations with time and date stamp, the ability of the user to adjust the timeout "Auto oFF", and enhanced performance. See Features and Specifications on pages 2 & 3.

The most exciting new feature is data logging with the ability to download the memory or stored test data wirelessly with its corresponding time, date and unit name. This feature allows the user to create spreadsheets and graphs with ease and quickly and accurately manipulate data more effectively. The optional bluDock™ and accompanying U2CI software is compatible with most computers using either Microsoft Windows XP, Vista, or 7™ or Macintosh OSX™. The data may be imported into a variety of spreadsheet formats like Microsoft Excel CSV™.
Please Note: Although the Myron L Company has performed extensive testing, we cannot guarantee compatibility of all applications and formats. We suggest testing your application and format for compatibility before relying on it.

For your convenience, a brief set of instructions is provided on the bottom side of your Ultrameter II. A waterproof pocket-sized card with abbreviated instructions is also included with the instrument.

Special note ... Conductivity, resistivity, and TDS require mathematical correction to 25°C values (ref. Temperature Compensation, pg. 39). On the left of the Ultrameter II's liquid crystal display is shown an indicator of the salt solution characteristic used to model temperature compensation of conductivity and its TDS conversion. The indicator may be KCl, NaCl, 442™ or User. Selection affects the temperature correction of conductivity, and the calculation of TDS from compensated conductivity (ref. Conductivity Conversion to Total Dissolved Solids (TDS), pg. 41). The selection can affect the reported conductivity of hot or cold solutions, and will change the reported TDS of a solution. Generally, using KCl for conductivity, NaCl for resistivity, and 442 (Natural Water characteristic) for TDS will reflect present industry practice for standardization. This is how your instrument, as shipped from the factory, is set to operate. For use in sea water desalination for example, both the conductivity and TDS may easily be changed to NaCl.

II. FEATURES and SPECIFICATIONS

A. Features

- ORP-based FC^{E} free chlorine measurement, displayed as ppm concentration (6PFC^{E})
- Superior resolution 4 digit LCD displays full 9999 μS/ppm
- Cond/TDS accuracy of ±1% of READING in a handheld instrument ±0.1% at calibration point
- All electrodes are internal for maximum protection
- Improved 4 electrode sensor technology
- Waterproof to 1 meter/3 feet
- Autoranging conductivity/TDS/resistivity
- Prompts for easy pH calibration (6PFC^{E})
- Factory calibrations stored in microprocessor
- 3 conductivity/TDS solution conversions preprogrammed into microprocessor
- User mode feature allows:
  Programming your own cond/TDS conversion factor
  Programming your own temperature compensation factor
  Disabling temperature compensation
- Real Time Clock with Time and Date
- Data Logging with TIME and DATE in memory
- Memory stores 100 readings
- User adjustable timeout “Auto oFF”
- *Bluetooth®* wireless download capability with optional bluDock™

B. General Specifications

Display	4 Digit LCD
Dimensions (LxWxH)	196 x 68 x 64 mm/ 7.7 x 2.7 x 2.5 in.
Weight	352 g/12.4 oz.
Case Material	VALOX*
Cond/Res/TDS Cell Material	VALOX*
Cond/TDS Electrodes (4)	316 Stainless Steel
Cond/Res/TDS Cell Capacity	5 ml/0.2 oz.
pH/ORP Sensor Well Capacity	1,2 ml/0.04 oz. (6PFC^{E})
Power	9V Alkaline Battery
Battery Life	>100 Hours/5000 Readings
Operating/Storage Temperature	0-55°C/32-132°F
Protection Ratings	IP67/NEMA 6 (waterproof to 1 meter/3 feet)
CE EMI/EMC Ratings (Conformité Européenne)	EN61326-1: 2006 + Annex A: 2008 (hand-held devices) CISPR 11: 2003 IEC 61000-4-2: 2001 and, IEC 61000-4-3: 2002

* ™ SABIC Innovative Plastics IP BV

Additional information is available on our website:
www.myronl.com

MADE IN USA

C. Specification Chart

	pH(6PFC^E)	ORP(6PFC^E)	Free Chlorine (6PFC^E)	Conductivity	TDS	Resistivity	Temperature
Ranges	0-14 pH	±999 mV	0.00-9.99 ppm** 350 mV ≤ ORP < 725 mV and 0.0 ≤ pH < 9.9 725 mV ≤ ORP < 825 mV and 0.0 ≤ pH <8.9	0-9999 µS/cm 10-200 mS/cm in 5 autoranges	0-9999 ppm 10-200 ppt in 5 autoranges	10KΩ - 30MΩ	0-71 °C 32 - 160 °F
Resolution	±.01 pH	±1 mV	0.01 ppm	0.01 (<100 µS) 0.1 (<1000 µS) 1.0 (<10 mS) 0.01 (<100 mS) 0.1 (<200 mS)	0.01 (<100 ppm) 0.1 (<1000 ppm) 1.0 (<10 ppt) 0.01 (<100 ppt) 0.1 (<200 ppt)	0.01 (<100 KΩ) 0.1 (<1000 KΩ) 0.1 (>1 MΩ)	0.1 °C/F
Accuracy	±.01 pH*	±1 mV*	±0.3 ppm <1.00ppm ±0.2 ppm ≥1.00ppm *	±1% of reading	±1% of reading	±1% of reading	±0.1 °C
Auto Temperature Compensation	0-71 °C 32-160 °F		0-71 °C 32-160 °F	0-71 °C 32 - 160 °F	0-71 °C 32 - 160 °F	0-71 °C 32 - 160 °F	
Adjustable Temperature Compensation				0 - 9.99%/ °C	0 - 9.99%/ °C	0 - 9.99%/ °C	
Cond/TDS Ratios Preprogrammed				KCl, NaCl, 442™			
Adjustable Cond/TDS Ratio Factor				0.20 - 7.99			

*± .2 pH in presence of RF fields ≥ 3 V/m and > 300 MHz ** If either ORP or pH is outside the specified limits, the instrument will display "-**Or**-".

D. Warranty/Service

The Myron L Ultrameter II, excluding the pH/ORP sensor (6PFC^E), has a Two (2) Year Limited Warranty. The pH/ORP sensor (6PFC^E) has a Six (6) Month Limited Warranty for materials and workmanship. If an instrument fails to operate properly, see Troubleshooting Chart, pg. 36. The battery and pH/ORP sensor are user-replaceable. For other service, return the instrument prepaid to the Myron L Company.

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Carlsbad, CA 92010-7226 USA
+1-760-438-2021
E-Mail: info@myronl.com
techquestions@myronl.com
www.myronl.com

If, in the opinion of the factory, failure was due to materials or workmanship, repair or replacement will be made without charge. A reasonable service charge will be made for diagnosis or repairs due to normal wear, abuse or tampering. This warranty is limited to the repair or replacement of the Ultrameter II only. The Myron L Company assumes no other responsibility or liability.

E. Ultrameter II Models

MODEL	4P	6PFC^E
PARAMETERS	Conductivity/TDS Resistivity/Temp.	Conductivity/TDS/pH/Resistivity/Temp. ORP mV/Free Chlorine Equivalent ppm (FC^E)

TABLE OF CONTENTS

TABLE OF CONTENTS

TABLE OF CONTENTS

III. RULES of OPERATION

A. Operation

Using the instrument is simple:

- Individual or multiple parameter readings may be obtained by filling individual sensors or entire cell cup area.
- Rinse the conductivity cell or pH/ORP sensor (6PFC^{E}) well with test solution 3 times and refill. Temperature and/or measurement extremes will require additional rinses for maximum accuracy.
- Press the desired measurement key to start measurement. Pressing the key again restarts the 15 second “Auto oFF” timer.
- Note the value displayed or press the MS key to store the reading (ref. Memory Storage, pg. 23). It’s that simple!

B. Characteristics of the Keys

- Though your Ultrameter II has a variety of sophisticated options, it is designed to provide quick, easy, accurate measurements by simply pressing one key.
- All functions are performed one key at a time.
- There is no “off” key. After 15 seconds of inactivity the instrument turns itself off (60 seconds in CAL mode). User adjustable up to 75 seconds (ref. Auto oFF, pg. 28).
- Rarely is it necessary to press and *hold* a key (as in Procedure to Select a Solution, pg. 14; or Conductivity or TDS Calibration, pg. 18).

C. Operation of the Keys (See Instrument Illustration, pg. i)

1. Measurement Keys in General

Any of the 5 measurement keys in the upper part of the keypad turns on the instrument in the mode selected. The mode is shown at the bottom of the display, and the measurement units appear at the right. Pressing a measurement key does this even if you are in a calibration sequence and also serves to cancel a change (ref. Leaving Calibration, pg. 17).

2. COND, RES and TDS Keys

These 3 keys are used with solution in the Conductivity Cell.
Precautions:

- While filling cell cup ensure no air bubbles cling on the cell wall.
- If the proper solution is not selected (KCl, NaCl, 442 or User), refer to Why Solution Selection is Available, pg. 14 and Procedure to Select a Solution, pg. 14.

a. COND Key

Solution to be tested is introduced into the conductivity cell and a press of (COND) displays conductivity with units on the right. On the left is shown the solution type selected for conductivity.

b. RES Key

A press of (RES) displays resistivity with units on the right. On the left is shown solution type selected for resistivity (ref. Solution Selection, pg. 14). The range of display of resistivity is limited to between 10 kilohms (KΩ) and 30 megohms (MΩ). A solution outside that range will only show [- - - -] in the display.

c. TDS Key

A press of (TDS) displays Total Dissolved Solids with units on the right. This is a display of the concentration of material calculated from compensated conductivity using the characteristics of a known material. On the left is shown solution type selected for TDS (ref. Solution Selection, pg. 14).

3. pH and ORP/Fr Chl Keys (6PFC^{E})

Measurements are made on solution held in the pH/ORP sensor well (ref. pH and ORP, pg. 44). The protective cap is removed and the sensor well is filled and rinsed with the sample enough times to completely replace the storage solution.

After use, the pH/ORP sensor well must be refilled with Myron L Storage Solution, and the protective cap reinstalled securely (ref. Maintenance of the pH/ORP Sensor, pg. 9 and Cleaning Sensors, 2. pH/ORP, pg. 34).

a. pH Key (6PFC^{E})

A press of (pH) displays pH readings. No units are displayed on the right.

b. ORP/Fr Chl Key (6PFC^{E})

In ORP mode, a press of (ORP / Fr Chl) displays Oxidation-Reduction Potential/REDOX reading in millivolts; “mV” is displayed.

When the FC^{E} mode is activated, a press of (ORP / Fr Chl) displays the Free Chlorine Equivalent reading in “ppm” alternating with the FC^{E} predictive ORP reading in “mV”.

4. CAL/MCLR Key

A press of (CAL / MCLR) allows you to enter the calibration mode while measuring conductivity, TDS or pH. Once in CAL mode, a press of this key accepts the new value. If no more calibration options follow, the instrument returns to measuring (ref. Leaving Calibration, pg. 17).

If (CAL / MCLR) is held down for about 3 seconds when the ORP or FC^{E} functions are active, CAL mode is not entered. Instead either “**OrP**” or “**Chl**” will appear depending on which mode is active. Change modes by pressing the Up or Down buttons. Press any parameter key to exit ORP unit preference selection or let the unit time out. ORP unit preference will be saved.

If 

is held down for about 3 seconds at any other time, CAL mode but "**SEL**" appears to allow Solution Selection (ref. pg. 14) with the Up or is not entered, Down keys. As in calibration, the CAL key is now an "accept" key.
While reviewing stored records, the MCLR side of the key is active to allow clearing records (ref. Clearing a Record/Memory Clear, pg. 24).

5. UP or DOWN Keys

While measuring in any parameter, the 

or 

keys activate the Memory Store and Memory Recall functions.
While in CAL mode, the keys step or scroll the displayed value up or down. A single press steps the display and holding either key scrolls the value rapidly.

While in Memory Recall, the keys scroll the display up and down through the stack of records (ref. Memory Recall, pg. 23).

IV. AFTER USING THE ULTRAMETER II

A. Maintenance of the Conductivity Cell

Rinse out the cell cup with clean water. Do not scrub the cell. For oily films, squirt in a foaming non-abrasive cleaner and rinse (ref. Cleaning Sensors, pg. 34). Even if a very active chemical discolors the electrodes, this does not affect the accuracy; leave it alone.

B. Maintenance of the pH/ORP Sensor (6PFC^{E})

The sensor well must be kept wet with a saline solution. Before replacing the rubber cap, rinse and fill the sensor well with Myron L pH Sensor Storage Solution. If unavailable, use an almost saturated KCl solution, pH 4 buffer or a saturated solution of table salt and tap water (ref. pH and ORP Practices to Maintain Calibration, pg. 23). NEVER USE DISTILLED WATER.

V. SPECIFIC RECOMMENDED MEASURING PROCEDURES

If the proper solution is not selected (KCl, NaCl, 442 or User), see Solution Selection, pg. 14.

NOTE: *After sampling high concentration solutions or temperature extremes, more rinsing may be required. When sampling low conductivity solutions, be sure the pH cap is well seated so that no solution washes into the conductivity cell from around the pH cap.*

A. Measuring Conductivity & Total Dissolved Solids (TDS)

1. Rinse cell cup 3 times with sample to be measured. (This conditions the temperature compensation network and prepares the cell.)
2. Refill cell cup with sample.

3. Press (COND) or (TDS).

4. Take reading. A display of [- - - -] indicates an overrange condition.

B. Measuring Resistivity

Resistivity is for low conductivity solutions. In a cell cup the value may drift from trace contaminants or absorption from atmospheric gasses, so measuring a flowing sample is recommended.

1. Ensure pH protective cap is secure to avoid contamination.

2. Hold instrument at 30° angle (cup sloping downward).

3. Let sample flow continuously into conductivity cell with no aeration.

4. Press (RES) key; use best reading.

NOTE: *If reading is lower than 10 kilohms display will be dashes: [- - - -]. Use Conductivity.*

C. Measuring pH (6PFC^{E})

1. Remove protective cap by rotating while grasping and pulling up.

2. Rinse pH/ORP sensor well and conductivity cell cup 3 times with sample to be measured. Shake out each sample to remove any residual liquid.

3. Refill both sensor well and cell cup with sample.

4. Press (pH).

5. Note value displayed.

6. **IMPORTANT:** After use, fill pH/ORP sensor well with Myron L pH Sensor Storage Solution and replace protective cap. If Myron L pH Sensor Storage Solution is unavailable, use a strong KCl solution, a pH 4 buffer, or a saturated solution of table salt and tap water (ref. Cleaning Sensors, 2. pH/ORP, pg. 34). *Do not allow pH/ORP sensor to dry out.*

D. Measuring ORP

The Ultrameter II features the ability to measure the activity of oxidizing or reducing chemicals in solution as ORP mV. The instrument also includes an innovative Free Chlorine Equivalent (FC^{E}) feature (Measuring Free Chlorine Using FC^{E}, pg. 12) that uses ORP and pH to measure free available chlorine (FAC) concentration in ppm. ORP mV and ppm of free available chlorine (FAC) are the two most commonly used sanitizer units of measure in water quality management.

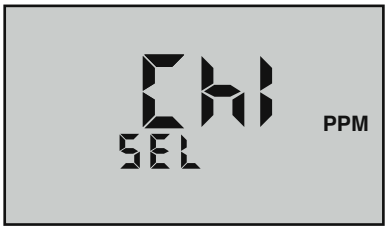
1. ORP / FC^{E} Mode Selection

The Ultrameter II allows the user to choose between measuring oxidizing sanitizers using either ORP mV or as parts per million (ppm) of equivalent free chlorine. Use ORP to directly measure the oxidizing power of all sanitizers like ozone, bromine, peracetic acid or chlorine. Use FC^{E} to measure the strength of oxidizing sanitizers as ppm of equivalent free chlorine. To select between ORP and Free Chlorine modes:

1. Press ORP / Fr Chl.
2. Press and hold CAL / MCLR for approximately 3 seconds.

The current preference for ORP units of measure is displayed. Factory setting for this preference is ORP mV.

3. Press the ▲ MS or MR ▼ keys to toggle between mV (standard ORP mode) and FC^{E} ppm. The setting chosen is displayed.



4. Press any parameter key to exit ORP unit preference selection or let the unit time out. ORP unit preference will be saved.

2. Measuring ORP

1. Ensure the 6PFC^{E} is in ORP mode (ref. ORP Mode Selection, pg. 10).
2. Remove protective cap by rotating while grasping and pulling up.
3. Rinse sensor well and cell cup 3 times with sample to be measured. Shake out each sample to remove any residual liquid.

4. Refill both sensor well and cell cup with sample.

5. Press 

.

6. Take reading.

7. Press **MS** to store reading in memory, if desired.

IMPORTANT: After use, fill pH/ORP sensor well with Myron L pH Sensor Storage Solution and replace protective cap. If Myron L pH Sensor Storage Solution is unavailable, you can use a strong KCl solution, a pH 4 buffer, or a saturated solution of table salt and tap water (ref. Cleaning Sensors, 2. pH/ORP, pg. 34). Do not allow pH/ORP sensor to dry out.

E. Measuring Free Chlorine Using FC^E

The FC^E function can be used to measure discrete samples, flowing solution and bodies of water. Measurement technique is particular to the type of sample. For accurate results, use the FC^E Flow Method described in section 2 below to measure discrete or flowing samples. Use the FC^E Immersion Method described in section 3 below in situations where the $6PFC^E$ can be dipped to obtain a sample. Read through section 4. FC^E Best Practices before you begin.

1. Prepare for FC^E Measurement

1. For ease of measurement, set the instrument's Auto oFF feature to 75 sec (ref. Auto oFF, pg. 28).

2. Ensure the FC^E mode has been activated (ref. ORP/FC^E Mode Selection, pg. 10).

3. Remove protective cap from the pH/ORP sensor by rotating while grasping and pulling up.

2. FC^E Flow Method

1. Empty the pH/ORP sensor well of all storage solution.

2. Hold the $6PFC^E$ at a 30^o angle (cup sloping downward).

3. Thoroughly flush the sensor well and cell cup with a steady stream of the solution you intend to measure by allowing the solution to flow into and out of the sensor well and cell cup for at least 10 seconds.

4. Let sample flow continuously into conductivity cell with no aeration.

5. Allow both the sensor well and cell cup to remain filled with sample.

6. Press ORP / Fr Chl. The instrument will begin alternating between a predicted final ORP value and a free chlorine equivalent concentration in ppm. Both readings will change rapidly at first.

7. Wait for the readings to stabilize. When the mV and ppm values are unchanging for 5 consecutive readings, the FC^E reading has reached a stable level. This may take 1 to 2 minutes. **NOTE:** If the reading takes more than 1 minute to stabilize, press the ORP / Fr Chl after 1 minute to prevent Auto oFF feature from disturbing the measurement process. Annunciators will alert you when either the pH or ORP of the final FC^E ppm value are Out of Range ("**-Or-**").

8. Press **MS** to store reading in memory if desired.

3. FC^E Immersion Method

NOTE: Use this method for pools, spas and other large standing bodies of water.

1. Hold instrument beneath the surface of the water to avoid surface effects on the water's chemistry.

2. Swirl the instrument around for at least 10 seconds to thoroughly rinse the cell cup and sensor well.

3. Continue holding the instrument under the surface while taking the reading.

4. Press ORP / Fr Chl.

5. The instrument will begin alternating between a predicted final ORP value and a free chlorine equivalent concentration in ppm. Both readings will change rapidly at first.

6. Wait for the readings to stabilize. When the mV and ppm values are unchanging for 5 consecutive readings, the FC^E reading has reached a stable level. This may take 1 to 2 minutes. **NOTE:** If the reading takes longer than 1 minute to stabilize, press ORP / Fr Chl after 1 minute to prevent Auto oFF feature from disturbing the measurement process. Annunciators will alert you when either the pH or ORP of the final FC^E ppm value are Out of Range ("**-Or-**").

7. Press **MS** to store reading in memory if desired.

4. FC^{E} Best Practices

For best results it is recommended that you:

1. Take 3 consecutive FC^{E} measurements and record the readings.

2. Calculate the average of the 3 measurements. Use this value.

3. Ignore measurements that are significantly different from the others. Ex: 3.20 ppm, ~~1.15 ppm~~, 3.10 ppm

IMPORTANT: After use, fill pH/ORP sensor well with Myron L pH Sensor Storage Solution and replace protective cap. If Myron L pH Sensor Storage Solution is unavailable, you can use a strong KCl solution, a pH 4 buffer, or a saturated solution of table salt and tap water (ref. Cleaning Sensors, 2. pH/ORP, pg. 34). Do not allow pH/ORP sensor to dry out.

VI. SOLUTION SELECTION

A. Why Solution Selection is Available

Conductivity, resistivity, and TDS require temperature correction to 25°C values (ref. Standardized to 25°C, pg. 39). Selection determines the temperature correction of conductivity and calculation of TDS from compensated conductivity (ref. Cond. Conversion to TDS, pg. 41).

B. The 4 Solution Types

On the left side of the display is the salt solution characteristic used to model temperature compensation of conductivity and its TDS conversion. Generally, using KCl for conductivity, NaCl for resistivity, and 442 (Natural Water characteristic) for TDS will reflect present industry practice for standardization. This is how your instrument is shipped from the factory (ref. Solution Characteristics, pg. 42).

The User selection allows a custom value to be entered for the temperature compensation of conductivity and also the conversion ratio if measuring TDS.

C. Calibration of Each Solution Type

There is a separate calibration for each of the 4 solution types. Note that calibration of a 442 solution does not affect the calibration of a NaCl solution. For example: Calibration (ref. Conductivity or TDS Calibration, pg. 18) is performed separately for each type of solution one wishes to measure (ref. Conductivity/TDS Standard Solutions, pg. 38).

D. Procedure to Select a Solution

NOTE: Check display to see if solution displayed (KCl, NaCl, 442 or

User) is already the type desired. If not:

1. Press COND, RES or TDS to select the parameter on which you wish to change the solution type.

2. Press and hold CAL/MCLR key for 3 seconds to make "**SEL**" appear (see Figure 1). For demonstration purposes, all 4 solution types are shown simultaneously.

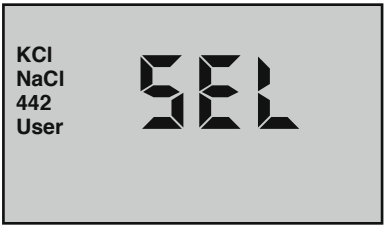


Figure 1

3. Use the ▲ MS or MR ▼ key to select type of solution desired (ref. Solution Characteristics, pg. 42). The selected solution type will be displayed: KCl, NaCl, 442 or User.

4. Press CAL/MCLR to accept new solution type.

E. Application of User Solution Type

1. User Programmable Temperature Compensation (Tempco)

This feature allows you to change your Ultrameter II's temperature compensating factor to another factor between 0-9.99%/°C (ref. Temperature Compensation, pg. 39). This feature does not apply to pH or ORP.

a. As in D. Procedure to Select a Solution above, select "User" mode.

b. With User mode now selected, press CAL/MCLR. You may now adjust a temperature compensation from .00%/°C to 9.99%/°C, by pressing ▲ MS or MR ▼. See example in Figure 2.

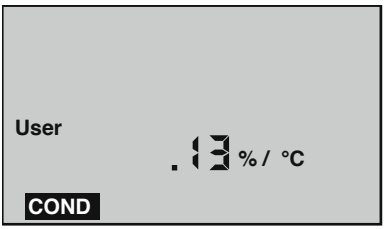


Figure 2

c. Press CAL/MCLR twice to skip calibration adjustment and accept the new tempco (3 times if in TDS mode). You are now ready to measure samples with your new temperature compensation factor.

2. Disabling Temperature Compensation

a. Select User mode (ref. Procedure to Select a Solution, pg. 14).

b. With "**User**" selected, press CAL/MCLR. If the display does not show .00%/°C, hold long enough to bring the tempco to .00%/°C (see Figure 3).

c. Press CAL/MCLR twice (3 times if in TDS mode). Temperature compensation is now disabled (=0) for measurements in User mode.

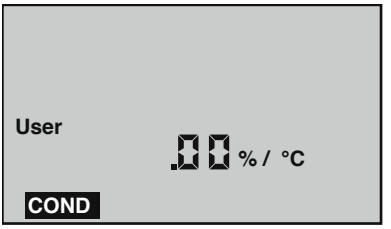


Figure 3

3. User Programmable Conductivity to TDS Ratio

This feature allows you to select a custom conductivity to TDS conversion ratio within the range of 0.20-7.99 for User mode measurements.

To determine the conversion ratio for a custom solution of **known** TDS ppm value, measure the solution conductivity **at 25ºC** with the Ultrameter II and divide the ppm value by the μS value. For example, a solution of known 75 ppm TDS and measured 100 μS conductivity at 25ºC would have a conversion ratio of 75/100 or 0.75. Enter the new conversion ratio as follows:

a. While in User mode, press TDS.

b. Press CAL/MCLR twice (to skip over tempco adjustment) and "**RATIO**" will appear (see Figure 4).

c. Adjust with ▲/MS or MR/▼ key until new conversion ratio is displayed.

Figure 4

d. Press CAL/MCLR twice (to skip over calibration adjustment) to accept new conversion ratio. You are now ready to measure samples with the new conductivity/TDS ratio.

In these first six sections, you have learned all you need to make accurate measurements. The following sections contain calibration, advanced operations and technical information.

VII. CALIBRATION

A. Calibration Intervals

Generally, calibration is recommended about once per month with Conductivity or TDS solutions. Calibration with pH solutions should be checked twice a month. Calibration of ORP is not necessary (ref. CALIBRATION INTERVALS, pg. 22).

B. Rules for Calibration of the Ultrameter II

1. Calibration Steps

a. Starting Calibration

Calibration is begun by pressing while measuring Conductivity, TDS or pH. Measuring continues, but the "**CAL**" icon is on, indicating calibration is now changeable.

The reading is changed with the and keys to match the known value. The calibration for each of the 4 solution types may be performed in either conductivity or TDS mode.

Depending on what is being calibrated, there may be 1, 2 or 3 steps to the calibration procedures.

	KCl, NaCl or 442	User
Cond	Gain only	Tempco, then Gain
Res	Done in conductivity	Done in conductivity or TDS
TDS	Gain only	Tempco, Ratio, then Gain
pH	7, acid and/or base (6PFC^{E})	
ORP	Zero set with pH 7 automatically (6PFC^{E})	

Once in CAL mode, the CAL/MCLR key becomes an "ACCEPT" key. At each point, pressing CAL/MCLR accepts the new calibration value and steps you to the next adjustment (or out of CAL mode if there are no more steps).

To bypass a calibration step, simply press CAL/MCLR to accept the present value as is.

b. Leaving Calibration

Calibration is complete when the "**CAL**" icon goes out. Pressing any measurement key cancels changes not yet accepted and exits calibration mode.

Leaving pH after the 2nd buffer results in the same gain being entered in place of the 3rd buffer.

2. Calibration Limits

There are calibration limits. A nominal "FAC" value is an ideal value stored by the factory. Attempts to calibrate too far, up or down, from there will cause the displayed value to be replaced with "**FAC**". If you accept it (press the "Cal" key), you will have the original default factory calibration for this measurement. The need to calibrate so far out that "**FAC**" appears indicates a procedural problem, incorrect standard solution, a very dirty cell cup or an aging pH/ORP sensor (ref. Troubleshooting Chart, pg. 36).

C. Calibration Procedures

1. Conductivity or TDS Calibration

a. Rinse conductivity cell three times with proper standard (KCl, NaCl, or 442) (ref. Cond/TDS Standard Solutions, pg. 38). For user calibration see User Calibration Conductivity/TDS below.

b. Refill conductivity cell with same standard. KCl-7000 shown.

c. Press (COND) or (TDS), then press (CAL/MCLR); "**CAL**" icon will appear on the display (see Figure 5).

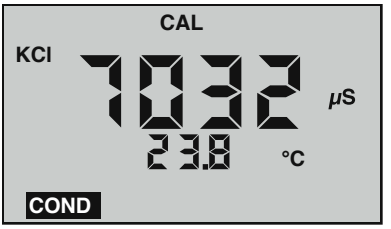


Figure 5

d. Press (▲ MS) or (MR ▼) to step the displayed value toward the standard's value (7032 > 7000) or hold a key down to scroll rapidly through the reading.

e. Press (CAL/MCLR) once to confirm new value and end the calibration sequence for this particular solution type. If another solution type is also to be measured, change solution type now and repeat this procedure.

2. User Calibration Conductivity/TDS

Instrument must be in User mode, see Solution Selection, pg. 14.

a. Rinse conductivity cell three times with your standard.

b. Refill conductivity cell with same standard.

c. Press (COND) or (TDS), then press (CAL/MCLR) twice in COND/ three times in TDS. The "**CAL**" icon will appear on the display.

d. Press (▲ MS) or (MR ▼) to step the displayed value toward the standard's value or hold a key down to scroll rapidly through the reading.

e. Press CAL/MCLR once to confirm new value and end the calibration sequence for this particular solution type.

3. Resistivity Calibration

Resistivity is the reciprocal of conductivity. To calibrate resistivity, calibrate conductivity for the solution type you wish to measure (ref. Conductivity or TDS Calibration, pg. 18).

4. Reloading Factory Calibration (Cond or TDS)

If calibration is suspect or known to be incorrect, and no standard solution is available, the calibration value can be replaced with the original factory value for that solution. This "FAC" value is the same for all Ultrameter IIs, and returns you to a known state without solution in the cell. The "FAC" internal electronics calibration (which bypasses the electrodes and cell) is not intended to replace calibration with conductivity/TDS standard solutions. If another solution type requires resetting, change solution type and repeat this procedure.

a. Press COND or TDS.

b. Press CAL/MCLR. (If in User solution mode. Press **CAL** key twice if in Conductivity, and three times if in TDS to skip over tempco and ratio adjustments.)

c. Press ▲/MS key until "**FAC**" appears and release.

d. Press CAL/MCLR to accept the factory calibration setting.

5. pH Calibration (6PFC^{E})

IMPORTANT: Always "zero" your Ultrameter II with a pH 7 buffer solution before adjusting the gain with acid or base buffers, i.e., 4 and/or 10, etc.

a. pH Zero Calibration (6PFC^{E})

1. Rinse sensor well and cell cup 3 times with 7 buffer solution.

2. Refill both sensor well and cell cup with 7 buffer solution.

3. Press pH to verify the pH calibration. If the display shows 7.00, skip the pH Zero Calibration and proceed to section b. pH Gain Calibration.

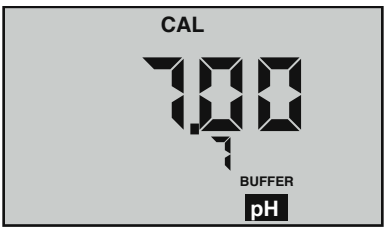


Figure 6

4. Press CAL/MCLR to enter calibration mode. The "**CAL**", "**BUFFER**" and "**7**" annunciators will appear (see Figure 6, page 19). Displayed value will be the uncalibrated sensor.

NOTES: *If a wrong buffer is added (outside of 6-8 pH), "**7**" and "**BUFFER**" will flash, and the Ultrameter II will not adjust.*

The uncalibrated pH value displayed in step 4 will assist in determining the accuracy of the pH sensor. If the pH reading is above 8 with pH 7 buffer solution, the sensor well needs additional rinsing or the pH sensor is defective and needs to be replaced.

5. Press ▲/MS or MR/▼ until the display reads 7.00.

NOTE: *Attempted calibration of >1 pH point from factory calibration will cause "**FAC**" to appear. This indicates the need for sensor replacement (ref. Troubleshooting Chart pg. 36) or fresh buffer solution. The "FAC" internal electronic calibration is not intended to replace calibration with pH buffers. It assumes an ideal pH sensor. Each "FAC" indicates a factory setting for that calibration step (i.e., 7, acid, base).*

You may press CAL/MCLR to accept the preset factory value, or you may reduce your variation from factory setting by pressing ▲/MS or MR/▼.

6. Press CAL/MCLR to accept the new value. The pH Zero Calibration is now complete. You may continue with pH Gain Calibration or exit by pressing any measurement key.

b. pH Gain Calibration (6PFC[E])

IMPORTANT: Always calibrate or verify your Ultrameter II with a pH 7 buffer solution before adjusting the gain with acid or base buffers, i.e., 4 and/or 10, etc. Either acid or base solution can be used for the 2nd point "Gain" calibration and then the opposite for the 3rd point. The display will verify that a buffer is in the sensor well by displaying either "**Acd**" or "**bAS**".

1. The pH calibration mode is initiated by either completion of the pH Zero Calibration, or verifying 7 buffer and pressing the CAL/MCLR key twice while in pH measurement mode.
2. At this point the "**CAL**", "**BUFFER**" and "**Acd**" or "**bAS**" annunciators will be displayed (see Figures 7 and 8).

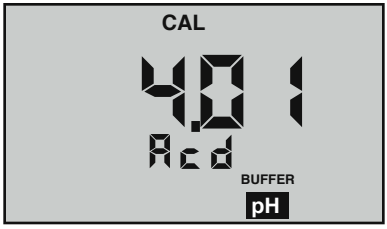


Figure 7

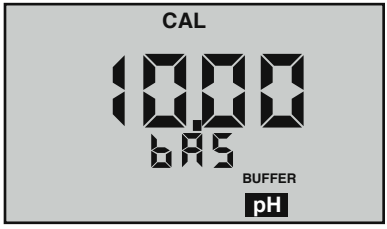


Figure 8

NOTE: *If the "**Acd**" and "**bAS**" indicators are blinking, the unit is indicating an error and needs either an acid or base solution present in the sensor well.*

3. Rinse sensor well 3 times with acid or base buffer solution.

4. Refill sensor well again with same buffer solution.

5. Press ▲/MS or MR/▼ until display agrees with buffer value.

6. Press CAL/MCLR to accept 2nd point of calibration. Now the display indicates the next type of buffer to be used.

Single point Gain Calibration is complete. You may continue for the 3rd point of Calibration (2nd Gain) or exit by pressing any measurement key. Exiting causes the value accepted for the buffer to be used for both acid and base measurements.

To continue with 3rd point calibration, use basic buffer if acidic buffer was used in the 2nd point, or vice-versa. Again, match the display to the known buffer value as in step 2 and continue with the following steps:

7. Repeat steps 3 through 6 using opposite buffer solution.

8. Press CAL/MCLR to accept 3rd point of calibration, which completes the Calibration procedure. Fill sensor well with Myron L Storage Solution and replace protective cap.

6. ORP/FC^{E} Calibration (6PFC^{E})

ORP electrodes rarely give false readings without problems in the reference electrode. For this reason, and because calibration solutions for ORP are highly reactive and potentially hazardous, your Ultrameter II has an electronic ORP calibration. This causes the zero point on the reference electrode to be set whenever pH 7 calibration is done.

7. Temperature Calibration

Temperature calibration is not necessary in the Ultrameter II.

VIII. CALIBRATION INTERVALS

There is no simple answer as to how often one should calibrate an instrument. The Ultrameter II is designed to not require frequent recalibration. The most common sources of error were eliminated in the design, and there are no mechanical adjustments. Still, to ensure specified accuracy, any instrument must be checked against chemical standards occasionally.

A. Suggested Intervals

On the average, we expect calibration need only be checked monthly for the Conductivity, RES or TDS functions. The pH (6PFC^E) function should be checked every 2 weeks to ensure accuracy. Measuring some solutions will require more frequent intervals.

B. Calibration Tracking Records

To minimize your calibration effort, keep records. If adjustments you are making are minimal for your application, you can check less often. Changes in conductivity calibration should be recorded in percent. Changes in pH calibration (6PFC^E) are best recorded in pH units.

Calibration is purposely limited in the Ultrameter II to ±10% for the conductivity cell, as any change beyond that indicates damage, not drift. Likewise, calibration changes are limited to ±1 pH unit (6PFC^E), as any change beyond that indicates the end of the sensor's lifetime and replacement is recommended.

C. Conductivity, RES, TDS Practices to Maintain Calibration

1. Clean oily films or organic material from the cell electrodes with foaming cleaner or mild acid. Do not scrub inside the cell.

2. Calibrate with solutions close to the measurements you make. Readings are compensated for temperature based on the type of solution. If you choose to measure tap water with a KCl compensation, which is often done (ref. An Example of 2 different solution selections and the resulting compensation, pg. 40), and you calibrate with 442 solution because it is handy, the further away from 25°C you are, the more error you have. Your records of calibration changes will reflect temperature changes more than the instrument's accuracy.

3. Rinse out the cell with pure water after taking measurements. Allowing slow dissolving crystals to form in the cell contaminates future samples.

4. For maximum accuracy, keep the pH sensor cap on tight so that no fluid washes into the conductivity cell.

D. pH and ORP Practices to Maintain Calibration (6PFC[E])

1. Keep the sensor wet with Myron L Storage Solution.

2. Rinse away caustic solutions immediately after use.

ORP calibration solutions are caustic, and ±5% is considered very accurate. By using the pH zero setting (0 mV = 7 pH) for ORP and precision electronics for detection, the Ultrameter II delivers better accuracy without calibration than a simpler instrument could using calibration solutions.

IX. MEMORY

This feature allows up to 100 readings with their temperatures to be stored simultaneously for later recall. At the same time, the TIME and DATE are also recorded. To download the memory to a computer, (ref. bluDock™ WIRELESS DATA TRANSFER INSTRUCTIONS, pg. 32).

A. Memory Storage

1. While displaying a measurement, press ▲ MS to record the displayed value.

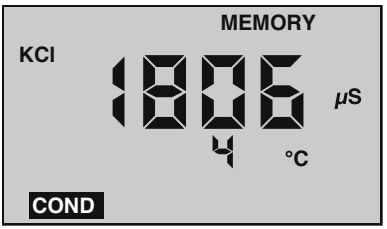


Figure 9

2. "**MEMORY**" will appear and the temperature display will be momentarily replaced by a number (1-100) showing the position of the record. Figure 9 shows a reading of 1806 µS stored in memory record #4.

B. Memory Recall

1. Press any measurement key.

2. Press MR ▼, "**MEMORY**" will appear, and the display will show the last record stored.

3. Press ▲ MS or MR ▼ to scroll to the record location desired (the temperature display alternates between temperature recorded and location number).

4. Press CAL MCLR to display time and date stamp.

5. Press any measurement key to leave memory recall or allow to automatically turn off.

C. Clearing a Record/Memory Clear

After recalling a certain record location, press and HOLD CAL/MCLR to clear that memory. This space will be the place for the next memory record, unless you scroll to another empty position before ending the recall sequence. The next memory stored will go into the next highest available memory location.

Example: You have locations 1-7 filled and wish to clear the conductivity reading stored in record location #**3** and replace it with a pH reading.

1. Press MR/▼ and scroll to location #**3**.
2. Press and HOLD CAL/MCLR to clear old record #**3**.
3. Fill pH/ORP sensor well with sample.
4. Press pH to measure sample and press ▲/MS to store reading in location #**3**.
5. The next memory stored will go into location #**8**.
6. To clear **all** records: After pressing MR/▼, scroll down. "**CLr ALL**" will be displayed (see Figure 10).
7. Press CAL/MCLR. All records will be cleared.

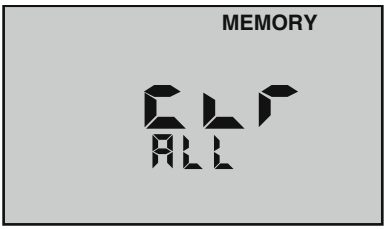


Figure 10

X. TIME and DATE

The Time and Date may easily be changed as you travel.

A. Setting TIME

Time is always displayed in 24 hour time.

Example shown in Figure 11, 16:05 equals 4:05 PM.

Figure 11

1. Press COND.
2. Press MR/▼ until the time is displayed (scrolling through

stored readings, PC OFF, and CLr ALL to time, e.g., "**16:05**").

3. Press CAL/MCLR to initiate.

"**CAL**" will be displayed along with the time (see Figure 11).

4. Press ▲/MS or MR/▼ to change the time.

5. Press CAL/MCLR to accept the change (new time).

B. Setting DATE

Example shown in Figure 12 is in US format, i.e., mo/dy/yr. **NOTE:** The default format is US. Date format may be changed (ref. Date Format "US and International (Int)", pg. 26).

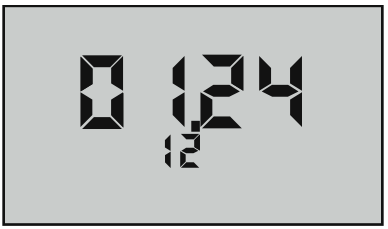


Figure 12

1. Press COND.

2. Press MR/▼ repeatedly until the date is displayed (scrolling through stored readings, PC OFF, CLr ALL and time to the date, e.g., "**11.18 11**" (Figure 12), November 18, 2011).

3. Press CAL/MCLR to initiate. "**CAL**" will be displayed along with the YEAR (see Figure 13).

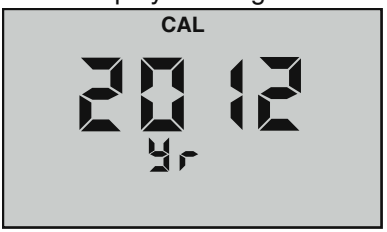


Figure 13

4. Press ▲/MS or MR/▼ to change the YEAR.

5. Press CAL/MCLR to accept the change (new year).

6. Press ▲/MS or MR/▼ to change the month.

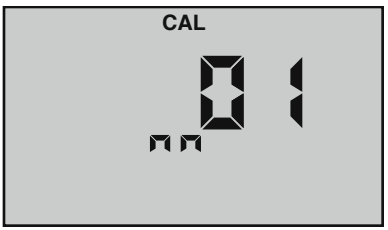


Figure 14

7. Press CAL/MCLR to accept the

change (new month),
(see Figure 14).

8. Press ▲ MS or MR ▼ to change the day.

9. Press CAL MCLR to accept the change (new day) (see Figure 15).

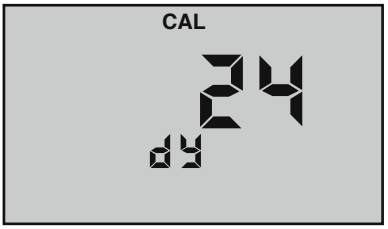


Figure 15

C. DATE FORMAT "US & International (Int)"

1. Press COND.

2. Press MR ▼ repeatedly until the format is displayed (scrolling through stored readings, PC OFF, CLr ALL, time and date to date **format**).

3. Press CAL MCLR to change. Display will now indicate other format (see Figures 16 & 17).

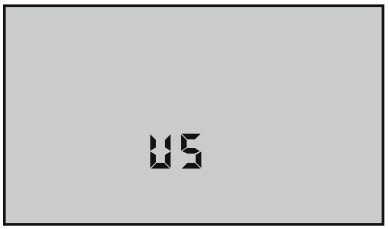


Figure 16

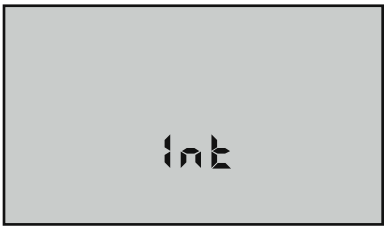


Figure 17

4. Press any measurement key or allow to automatically turn off.

XI. TEMPERATURE FORMAT "Centigrade & Fahrenheit"

1. Press COND.

2. Press MR ▼ to display the stored memory records.

3. Press MR ▼ repeatedly until you pass the "**US**" or "**Int**" date

format location. The display will show a "**C**" or "**F**" (see Figures 18 and 19).

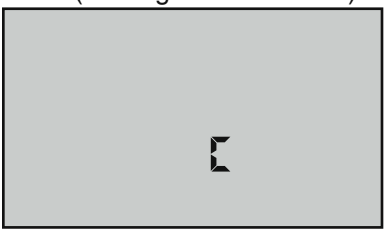


Figure 18

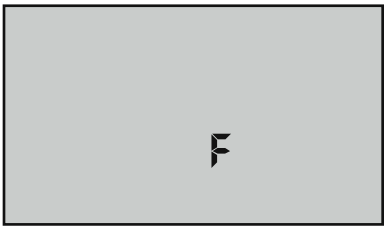


Figure 19

4. Press CAL/MCLR to switch units.

5. Press COND to accept unit preference for all temperature readings.

NOTE: *Tempco will still be shown in %/°C.*

XII. TOTAL RETURN to FACTORY SETTINGS "FAC SEL"

There may come a time when it would be desirable to quickly reset all the recorded calibration values in the instrument back to the factory settings. This might be to ensure all calibrations are set to a known value, or to give the instrument to someone else free of adjustments or recorded data for a particular application.
NOTE: All stored data will be lost.

1. Press COND.

2. Press MR ▼ to display the stored memory records.

3. Press MR ▼ repeatedly until you pass the CLr ALL and the C-F locations. The display will show a "**FAC SEL**" (see Figure 20).

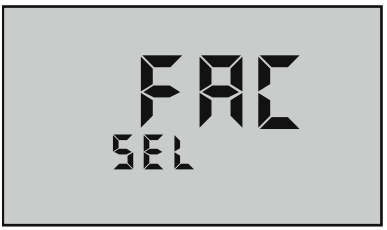


Figure 20

4. Press CAL/MCLR to accept the resetting. Display will return to Conductivity.

XIII. CELL CHECK

The cell check verifies the cleanliness of the conductivity/TDS/resistivity sensor. In normal use the cell may become dirty or coated and require cleaning. If the display is showing "**.00**" when the cell cup is dry, the sensor is probably clean. However, when testing high purity water in resistivity ("RES") mode improved accuracy may be desired. No matter what a manufacturer claims, a sensor can and will become contaminated or coated and, therefore, require cleaning. A true 4-wire sensor, as in the Ultrameter II, helps to mitigate contamination, but NO SENSOR IS 100% IMMUNE.

1. Press (COND).

2. Press (MR ▼) to display the stored memory records.

3. Press (MR ▼) repeatedly until you pass the FAC SEL location. The display will show a "**CELL ch**" (see Figure 21).

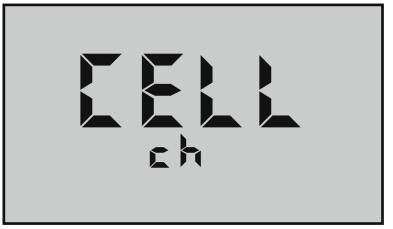


Figure 21

4. Press (CAL/MCLR) to test.

   If cell is clean, "**Good**" will momentarily be displayed (see Figure 22). If cell is dirty, "**CELL cLn**" will be displayed (see Figure 23) (ref. Cleaning Sensors, pg. 34).

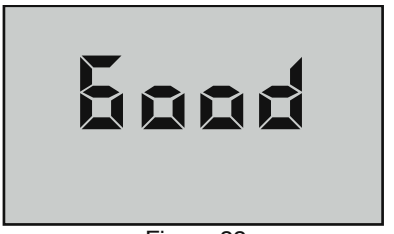


Figure 22

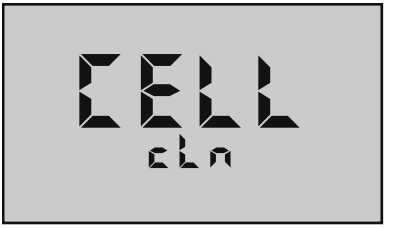


Figure 23

XIV. AUTO oFF

Auto oFF allows the user to adjust the time the instrument is ON (up to 75 seconds) after each press of a key. Default time is 15 seconds with 60 seconds in CAL (calibration) mode.

1. Press (COND).

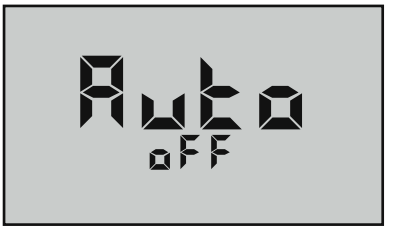


Figure 24

2. Press MR ▼ to display the stored memory records.

3. Press MR ▼ repeatedly until you pass the CELL ch location. The display will show "**Auto oFF**" (Figure 24).

4. Press CAL MCLR to initiate. "**CAL**" will be displayed along with "**15 SEC**" or current Auto oFF value (see Figure 25).

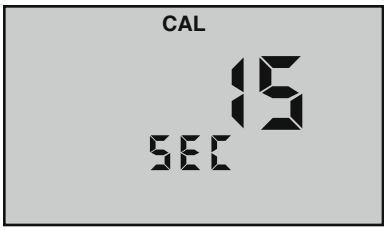


Figure 25

5. Press ▲ MS or MR ▼ to change the amount of time (see Figure 26). Maximum time of 75 seconds is shown.

6. Press CAL MCLR to accept the change (new time).

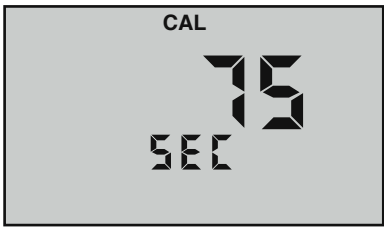


Figure 26

XV. USER MODE CALIBRATION LINC™ FUNCTION

The Linc™ function allows easy calibration when in User mode and the user does not have a user standard solution to calibrate the instrument. This function will ensure more repeatable and accurate measurements than many other calibration methods. It is recommended that this function be used to provide the highest degree of confidence when the Ultrameter II is used in User mode. When Linc is used, the User mode is linked to another standard, i.e., if User and KCl are linked, a KCl standard solution is used to calibrate the instrument. It is that simple.

A. Calibration of Ultrameter II for use in User mode

1. Press COND or TDS key.
2. Calibrate the unit using a Standard Solution (ref. CALIBRATION, pg. 17).
3. Place the Ultrameter II in User mode (ref. SOLUTION SELECTION, pg. 14).
4. Verify/Set the calibration linc. (See B. Setting User Mode Calibration "Linc", pg. 30.)

B. Setting User mode Calibration "Linc"

The Linc function sets or "links" the calibration gain factor of a Standard Solution to the User solution mode. Once set, the "Linc" will stay intact with future calibrations unless the Linc has been canceled. For more information on canceling the User mode Calibration Linc refer to C. "Canceling User mode Calibration "Linc".

Follow the steps below to set either the KCl, NaCl or 442 calibration factor to the User solution mode.

1. Press measurement key desired to be "Linked", i.e., (COND), (RES) or (TDS).

2. Place the Ultrameter II in User mode (ref. SOLUTION SELECTION, pg. 14, for selecting the User mode).

3. Press (MR ▼) arrow key until the menu "**Linc**" appears (see Figure 27).

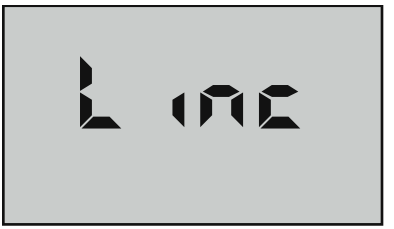


Figure 27

4. Press (CAL/MCLR) key. The instrument will display "**SEL**" and the "**User**" Icon (see Figure 28).

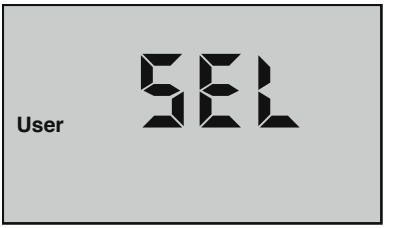


Figure 28

Any additional display of KCl, NaCl or 442 icons indicates a "Linc" between the User solution and the other solution displayed.

5. Press (▲ MS) or (MR ▼) keys to select a Standard Solution to be linked to the User mode calibration constant. In Figure 29 the display indicates that "**User**" is linked to "**KCl**".

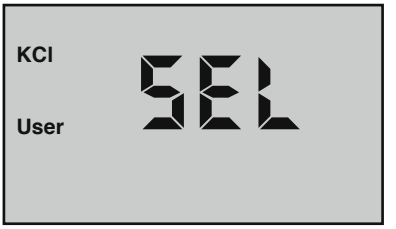


Figure 29

If none of the Solution Selection icons are displayed (i.e., KCl, NaCl or 442), nothing has been linked to User mode.

6. Press (CAL/MCLR) key to accept the setting. Pressing any of the

measurement keys will exit without changing the setting. User mode "Linc" is now complete. The User mode will now use the calibration gain constant used for the calibration of the Standard Solution as outlined above.

C. Canceling User mode Calibration "Linc"

The Ultrameter II must be in "User" linked mode in order to cancel the "Linc" (ref. SOLUTION SELECTION, pg. 14).

1. Press "Linked" measurement key (COND), (RES) or (TDS).

   Two solution icons will be shown in the left side of display - "**User**" and another, e.g., "**KCl**".

2. Press (MR ▼) key until the menu "**Linc**" appears (see Figure 27).

3. Press (CAL/MCLR) key; the instrument will display both "**SEL**" and the "**User**" Icon.

4. Press (MR ▼) key until "**User**" is the only solution icon being displayed.

5. Press (CAL/MCLR) key.

6. The User mode calibration "Linc" has now been canceled.

NOTES:

1. To maintain repeatability, use the same standard solutions for future calibrations.

2. Calibration of the Ultrameter II Gain Factor for User mode is not available when the calibration linc has been established. The other calibration functions (i.e., Temperature Compensation %/C settings and TDS Ratio settings) are still intact. To perform a calibration of the User mode as described in User Calibration Conductivity/TDS, pg. 18, the User mode Linc should be canceled. See above, "Canceling User mode calibration "Linc"".

3. Once a "Linc" has been established for User mode , the "Linc" will apply to all measurement modes using User solution selection (i.e., TDS/User, Cond/User or Res/User).

XVI. bluDock™ WIRELESS DATA TRANSFER INSTRUCTIONS

NOTE: *Bluetooth®* is a registered trademark of Bluetooth SIG. The bluDock Bluetooth module is a registered Bluetooth device.

Requires Myron L bluDock™ accessory package, Model # BLUDOCK. Package includes Ultrameter II hardware modification that allows the unit to communicate wirelessly with a personal computer configured for wireless device communication. Package also includes U2CI software application that will operate on Windows XP, Vista and 7*, and Macintosh OSX** based computer systems and Bluetooth USB adapter (dongle) for computers that do not have Bluetooth capability.

A. Software Installation

Follow the instructions in the "U2CI Software Installation Guide" that was shipped with your blueDock equipped instrument or download it from the Myron L Company website.
http://www.myronl.com/main/U2CI_Application_DL.htm

B. Hardware Setup

For a computer without *Bluetooth®* capability:
If you don't have the dongle that came with the BLUDOCK, one can be ordered separately from the Myron L Company. Order Model # BDDO. Plug in your dongle and install per manufacturer's instructions.

For computers with Bluetooth capability/Bluetooth dongle installed:

First time use of the bluDock:

1. Press any parameter button to turn the Ultrameter II on.
2. Put the Ultrameter II in "**PC On**" mode by pressing the (MR ▼) key until "**PC OFF**" appears (see Figure 30).
3. Then press the (CAL / MCLR) key. "**PC On**" will be displayed (see Figure 31).

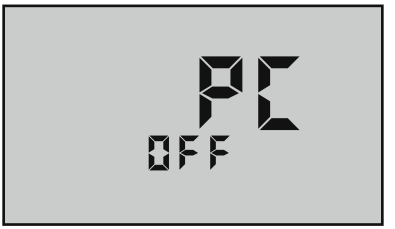


Figure 30

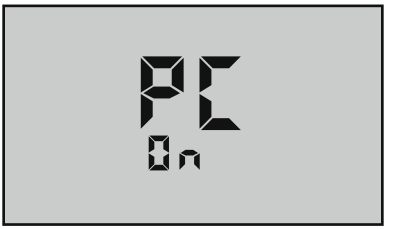


Figure 31

NOTE: "**PC Ini**" may momentarily be displayed while initializing (see Figure 32).

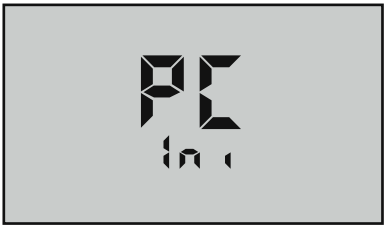


Figure 32

4. Add bluDock to your Bluetooth devices per your operating system procedure. **THE BLUDOCK DEVICE PASSKEY IS 1234.**
5. After pairing, note the number of the COM port assigned by the computer. In Windows XP, note the number of the outgoing COM port assigned by the computer.

NOTE: The unit will automatically power down after 60 sec. If the unit powers down during pairing, repeat steps 1-3 above and continue.

C. Memory Stack Download

1. With the Ultrameter II in "**PC On**" mode, open the U2CI software application.
2. Verify that the port selected matches the COM port number noted (first time only). This is the outgoing COM port on Windows XP.
3. In the U2CI application, click on the data download button. A data transfer bar will appear while the data is being downloaded.

   Once downloaded, the data may be manipulated, printed or stored within the Myron L U2CI application, or the data may be exported to another more powerful spreadsheet***, such as Excel*.

   Additional features such as assigning a name to the unit, setting time and date and erasing data are available. See U2CI Operation Manual or visit our website for the latest instructions: http://myronl.com/main/U2CI_Application_DL.htm

4. Upon completion, click on the "disconnect" icon.
5. Turn off Ultrameter II PC download mode by selecting any measurement function. Failure to do so will reduce battery life.

* Windows 2000, 2007, XP & Vista and Excel are registered trademarks of Microsoft Corporation.

** Macintosh OSX is a registered trademark of Apple Computer Inc.

*** **Please Note:** Although the Myron L Company has performed extensive testing, we cannot guarantee compatibility of all applications and formats. We suggest testing your application and format for compatibility before relying on it.

XVII. CARE and MAINTENANCE

Ultrameter IIs should be rinsed with clean water after use. Solvents should be avoided. Shock damage from a fall may cause instrument failure.

A. Temperature Extremes

Solutions in excess of 71°C/160°F should not be placed in the cell cup area; this may cause damage. The pH sensor may fracture if the Ultrameter II temperature is allowed to go below 0°C/32°F. Care should be exercised not to exceed rated operating temperature.

Leaving the Ultrameter II in a vehicle or storage shed on a hot day can easily subject the instrument to over 66°C/150°F. This will void the warranty.

B. Battery Replacement

Dry Instrument THOROUGHLY. Remove the four (4) bottom screws. Open instrument carefully. Carefully detach battery from circuit board. Replace with 9 volt alkaline battery. Replace bottom, ensuring the sealing gasket is installed in the groove of the top half of case. Re-install screws, tighten evenly and securely.

NOTE: *Because of nonvolatile EEPROM circuitry, all data stored in memory and all calibration settings are protected even during power loss or battery replacement. However, loss of time and date may occur if battery is removed for longer than 3 minutes (180 seconds).*

C. pH/ORP Sensor Replacement (6PFC^{E})

Order model RPR. When ordering, be sure to include the model and serial number of your instrument to ensure receipt of the proper type. Complete installation instructions are provided with each replacement sensor.

D. Cleaning Sensors

1. Conductivity/TDS/Resistivity

The conductivity cell cup should be kept as clean as possible. Flushing with clean water following use will prevent buildup on electrodes. However, if very dirty samples — particularly scaling types — are allowed to dry in the cell cup, a film will form. This film reduces accuracy. When there are visible films of oil, dirt, or scale in the cell cup or on the electrodes, use isopropyl alcohol or a foaming non-abrasive household cleaner. Rinse out the cleaner and your Ultrameter II is again ready for accurate measurements.

2. pH/ORP (6PFC^{E})

The unique pH/ORP sensor in your Ultrameter II is a nonrefillable combination type that features a porous liquid junction. *It should not be*

allowed to dry out. However, if this occurs, the sensor may sometimes be rejuvenated by first cleaning the sensor well with Isopropyl alcohol or a liquid spray cleaner such as Windex™ or Fantastic™ and rinsing well. Do not scrub or wipe the pH/ORP sensor.

Then use one of the following methods:

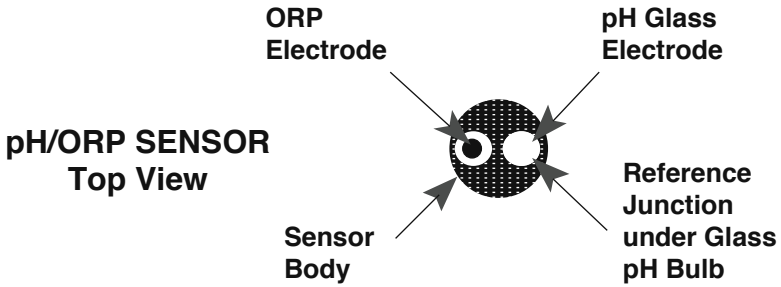
1. Pour a HOT salt solution ~60°C/140°F — a potassium chloride (KCl) solution such as Myron L pH/ORP Sensor Storage Solution is preferable, but HOT tap water with table salt (NaCl) will work fine — in the sensor well and allow to cool. Retest.

or

2. Pour DI water in the sensor well and allow to stand for no more than 4 hours (longer can deplete the reference solution and damage the glass bulb). Retest.

If neither method is successful, the sensor must be replaced.

"Drifting" can be caused by a film on the pH sensor bulb and/or reference. Use isopropyl alcohol (IPA) or spray a liquid cleaner such as Windex™ or Fantastic™ into the sensor well to clean it. The sensor bulb is very thin and delicate. Do not scrub or wipe the pH/ORP sensor.



Leaving high pH (alkaline) solutions in contact with the pH sensor for long periods of time is harmful and will cause damage. Rinsing such liquids from the pH/ORP sensor well and refilling it with Myron L Storage Solution, a saturated KCl solution, pH 4 buffer, or a saturated solution of table salt and tap water, will extend the useful life.

Samples containing chlorine, sulfur, or ammonia can "poison" any pH electrode. If it is necessary to measure the pH of any such sample, thoroughly rinse the sensor well with clean water immediately after taking the measurement. Any sample element that reduces (adds an electron to) silver, such as cyanide, will attack the reference electrode.

Replacement sensors are available only from the Myron L Company or its authorized distributors (see Replacement pH/ORP Sensor (6PFC^{E}), pg. 39).

XVIII. TROUBLESHOOTING CHART

Symptom	Possible Cause
No **display**, even though measurement key pressed	Battery weak or not connected.
Inaccurate **pH** readings (6PFC^{E})	1. pH calibration needed. Ref. pH Cal., pg. 19. 2. Cross-contamination from residual pH buffers or samples in sensor well. 3. Calibration with expired pH buffers.
No response to **pH** changes (6PFC^{E})	Sensor bulb is cracked or an electromechanical short caused by an internal crack.
Will not adjust down to **pH** 7 (6PFC^{E})	pH/ORP sensor has lost KCl.
pH readings drift or respond slowly to changes in buffers/samples or "**FAC**" is displayed repeatedly (6PFC^{E})	1. Temporary condition due to memory" of solution in pH sensor well for long periods. 2. Bulb dirty or dried out. 3. Reference junction clogged or coated.
Unstable **Conductivity/TDS/ Resistivity** readings	1. Dirty electrodes. 2. Actual resistance is changing due to atmospheric contamination.
Unable to calibrate **Conductivity/TDS**	Film or deposits on electrodes.
Resistivity readings much lower than expected	1. Contamination from previous sample or from pH sensor well. 2. Carbon dioxide in test sample.
Low **ORP** Reading Slow or no response to **ORP** changes (6PFC^{E})	ORP platinum electrode is dirty.
FC^{E} responds very slowly or returns an atypically high Predictive **ORP** value (6PFC^{E}).	1. Dirty platinum electrode (see above). 2. ORP sensor memory/battery effect. Some ORP sensors exhibit a residual charge when measuring LOW Free Chlorine concentrations soon after measuring a HIGH Free Chlorine concentration.

	Corrective Action
	Check connections or replace battery. Ref. Battery Replacement, pg. 34.
	1. Recalibrate instrument. 2. Thoroughly rinse sensor well. 3. Recalibrate using fresh buffers. Ref. pH Buffer Solutions, pg. 38.
	Replace pH/ORP sensor. Ref. Replacement pH/ORP Sensor, pg. 39.
	Clean and rejuvenate sensor (ref. Cleaning Sensors, pg. 34) and recalibrate. If no improvement, replace pH/ORP sensor (ref. Replacement pH/ORP Sensor, pg. 39).
	Clean and rejuvenate sensor (ref. Cleaning Sensors, pg. 34) and recalibrate. If no improvement, replace pH/ORP sensor (ref. Replacement pH/ORP Sensor, pg. 39).
	1. Clean cell cup and electrodes. Ref. Cleaning Sensors, pg. 34. 2. Minimize test sample exposure to air by taking a flowing sample. Ref. Measuring Resistivity, pg. 10.
	Clean cell cup and electrodes. Ref. Cleaning Sensors, pg. 34.
	1. Rinse cell cup more thoroughly before measurement. Ensure pH cap is snugly in place. 2. See Measuring Resistivity, pg. 10.
	Check the ORP sensor functioning. Take an ORP reading of Myron L pH/ORP Sensor Storage Solution (ref. pH Sensor Storage Solution (6PFC^{E}), pg. 38). If the reading is outside the range of 350-400 mV, clean ONLY the platinum ORP electrode with Myron L ORP Conditioner solution-soaked cotton swab (ref. ORP Sensor Conditioner Solution (6PFC^{E}), pg. 38), being careful not to touch the swab to the glass bulb of the pH sensor.
	1. Rinse the pH/ORP sensor well briefly with a small amount of ORP Sensor Conditioner Solution. **DO NOT** leave the conditioning solution in the sensor well for more than 10 seconds. 2. Rinse the pH/ORP sensor 3 times with Sensor Storage Solution. 3. Fill the sensor well with Sensor Storage Solution and let rest for 5 minutes.

XIX. ACCESSORIES

NOTE: MSDSs are available on the Myron L website for all solutions: **http://www.myronl.com/main/Material_Safety_DS_DL.htm**

A. Conductivity/TDS Standard Solutions

Your Ultrameter II has been factory calibrated with the appropriate Myron L Company NIST traceable KCl, NaCl, and our own 442™ standard solutions. Most Myron L conductivity standard solution bottles show three values referenced at 25°C: Conductivity in microsiemens/micromhos, the ppm/TDS equivalents (based on our 442 Natural Water™) and NaCl standards. All standards are within ±1.0% of reference solutions. *Available in 2 oz., quarts/liters, and gallon/~3.8 liter bottles.*

1. Potassium Chloride (KCl)

The concentrations of these reference solutions are calculated from data in the International Critical Tables, Vol. 6. The 7000 µS is the recommended standard. *Order KCL-7000*

2. 442 Natural Water™

442 Natural Water Standard Solutions are based on the following salt proportions: 40% sodium sulfate, 40% sodium bicarbonate, and 20% sodium chloride, which represent the three predominant components (anions) in freshwater. This salt ratio has conductivity characteristics approximating fresh natural waters and was developed by the Myron L Company over four decades ago. It is used around the world for measuring both conductivity and TDS in drinking water, ground water, lakes, streams, etc. 3000 ppm is the recommended standard.
Order 442-3000

3. Sodium Chloride (NaCl)

This is especially useful in sea water mix applications, as sodium chloride is the major salt component. Most Myron L standard solution labels show the ppm NaCl equivalent to the conductivity and to ppm 442 values. The 14.0 mS is the recommended standard. *Order NACL-14.0*

B. pH Buffer Solutions (6PFC^{E})

pH buffers are available in pH values of 4, 7 and 10. Myron L Company buffer solutions are traceable to NIST certified pH references and are color-coded for instant identification. They are also mold inhibited and accurate to within ±0.01 pH units @ 25°C. Order 4, 7 or 10 Buffer.
Available in 2 oz., quarts/liters, and gallon/~3.8 liter bottles. Order SS.

C. pH Sensor Storage Solution (6PFC^{E})

Myron L pH Sensor Storage Solution prolongs the life of the pH sensor. *Available in 2 oz., quarts/liters, and gallon/~3.8 liter bottles.*

D. ORP Sensor Conditioner Solution (6PFC^{E})

Myron L ORP Conditioner Solution removes contaminants and conditions the ORP electrode. *Available in 1 oz. Order ORPCOND1OZ.*

E. Soft Protective Carry Cases

Padded Nylon carrying case features a belt clip for hands-free mobility. Two colors to choose from:
Blue - Model #: UCC
Desert Tan - Model #: UCCDT

F. Hard Protective Carry Cases

Large case with 2 oz. bottles of calibration standard solutions (KCl-7000, 442-3000, 4, 7, & 10 pH buffers and pH storage solution). *Model #: PKUU*
Small case (no calibration standard solutions) - *Model #: UPP*

G. Replacement pH/ORP Sensor (6PFC[E])

pH/ORP sensor is gel filled and features a unique porous liquid junction. It is user-replaceable and comes with easy to follow instructions.
Model #: RPR

H. bluDock™ Wireless Data Transfer Accessory Package

This accessory allows the operator to download the Ultrameter II memory stack to a spreadsheet on a computer. The package includes bluDock modified circuit board in the unit, software CD, installation and operating instructions, and dongle. *Model #: BLUDOCK*

XX. TEMPERATURE COMPENSATION (Tempco) of Aqueous Solutions

Electrical conductivity indicates solution concentration and ionization of the dissolved material. Since temperature greatly affects ionization, conductivity measurements are temperature dependent and are normally corrected to read what they would be at 25°C.

A. Standardized to 25°C

Conductivity is measured with great accuracy in the Ultrameter II using a method that ignores fill level, electrolysis, electrode characteristics, etc., and features a microprocessor to perform temperature compensation. In simpler instruments, conductivity values are usually assigned an average correction similar to that of KCl solutions for correction to 25°C. The correction to an equivalent KCl solution is a standard set by chemists that standardizes the measurements and allows calibration with precise KCl solutions. In the Ultrameter II, this correction can be set to other solutions or tailored for special measurements or applications.

B. Tempco Variation

Most conductivity instruments use an approximation of the temperature characteristics of solutions, perhaps even assuming a constant value. The value for KCl is often quoted simply as 2%/°C. In fact, KCl tempco varies with concentration and temperature in a non-linear fashion. Other solutions have more variation still. The Ultrameter II uses corrections that change with concentration and temperature instead of single average values. See Chart 1, pg. 40.

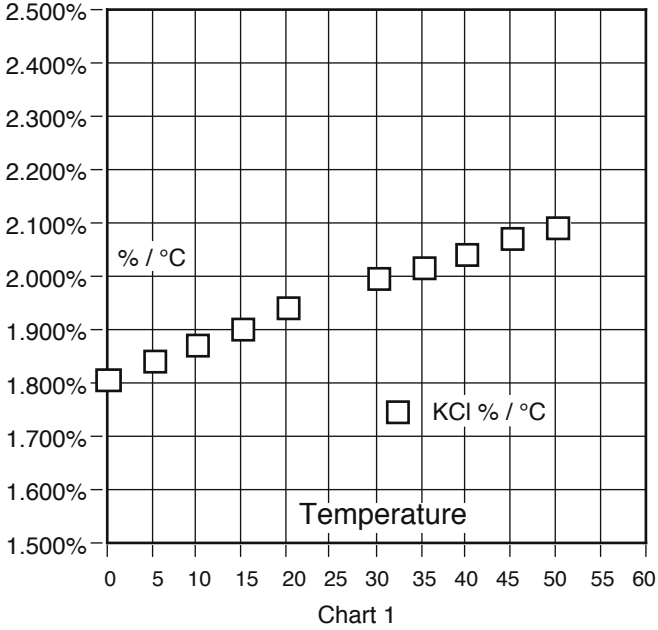


Chart 1

C. An Example of 2 different solution selections and the resulting compensation

How much error results from treating natural water as if it were KCl at 15°C?

A tap water solution should be compensated as 442 with a tempco of 1.68 %/°C, where the KCl value used would be 1.90 %/°C.

Suppose a measurement at 15°C/59°F is 900 microsiemens of true uncompensated conductivity.

Using a 442 correction of 10 (degrees below 25) x 1.68% indicates the solution is reading 16.8% low. For correction, dividing by (.832) yields 1082 microsiemens as a compensated reading.

A KCl correction of 10 (degrees below 25) x 1.9% indicates the solution is reading 19% low. Dividing by (.81) yields 1111 microsiemens for a compensated reading. The difference is 29 out of 1082 = 2.7%.

D. A Chart of Comparative Error

In the range of 1000 μS, the error using KCl on a solution that should be compensated as NaCl or as 442, is illustrated in Chart 2 on pg. 41. Users wanting to measure natural water based solutions to 1% would have to alter the internal compensation to the more suitable preloaded “442” values or stay close to 25°C. Users who have standardized to KCl- based compensation may want to stick with it, regardless of increasing error as you get further from 25°C. The Ultrameter II will provide the repeatability and convertibility of data necessary for relative values for process control.

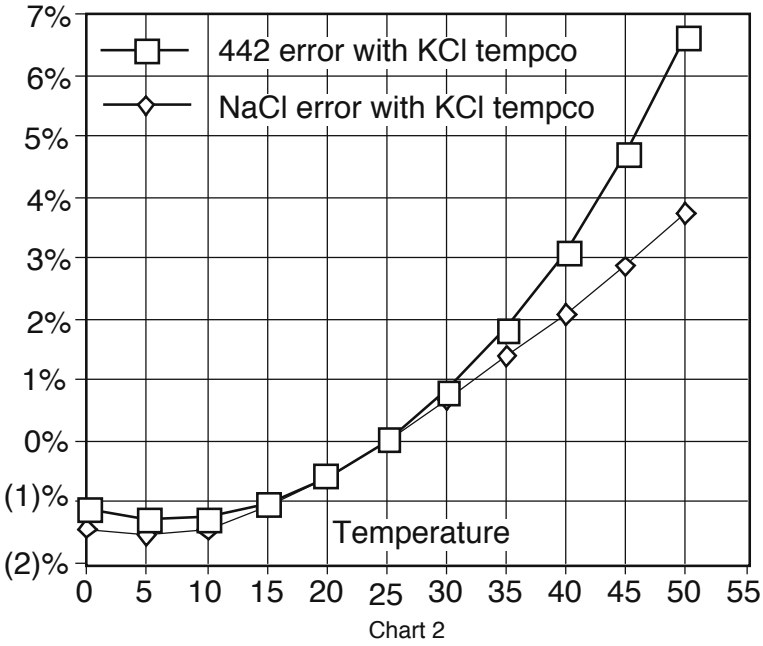


Chart 2

E. Other Solutions

A salt solution like sea water or liquid fertilizer acts like NaCl. An internal correction for NaCl can be selected for greatest accuracy with such solutions. Many solutions are not at all similar to KCl, NaCl or 442. A sugar solution, or a silicate, or a calcium salt at a high or low temperature may require a User value peculiar to the application to provide readings close to the true compensated conductivity.

Clearly, the solution characteristics should be chosen to truly represent the actual water under test for rated accuracy of ±1%. Many industrial applications have historically used relative measurements seeking a number to indicate a certain setpoint or minimum concentration or trend. The Ultrameter II gives the user the capability to collect data in "KCl conductivity units" to compare to older published data, in terms of NaCl or 442, or as appropriate. The Ultrameter II can be used to reconcile data taken with other compensation assumptions, especially with its ability to allow custom characteristics through the User mode.

XXI. CONDUCTIVITY CONVERSION to TOTAL DISSOLVED SOLIDS (TDS)

Electrical conductivity indicates solution concentration and ionization of the dissolved material. Since temperature greatly affects ionization, conductivity measurements are temperature dependent and are normally corrected to read what they would be at 25°C (ref. Temperature Compensation, pg. 39).

A. How it's Done

Once the effect of temperature is removed, the compensated conductivity is a function of the concentration (TDS). Temperature compensation of the conductivity of a solution is performed automatically by the internal processor with data derived from chemical tables. Any dissolved salt at a known temperature has a known ratio of conductivity to concentration. Tables of conversion ratios referenced to 25°C have been published by chemists for decades.

B. Solution Characteristics

Real world applications have to measure a wide range of materials and mixtures of electrolyte solutions. To address this problem, industrial users commonly use the characteristics of a standard material as a model for their solution, such as KCl, which is favored by chemists for its stability.

Users dealing with sea water, etc., use NaCl as the model for their concentration calculations. Users dealing with freshwater work with mixtures including sulfates, carbonates and chlorides, the three predominant components (anions) in freshwater that the Myron L Company calls "Natural Water". These are modeled in a mixture called "442™" which the Myron L Company markets for use as a calibration standard, as it does standard KCl and NaCl solutions.

The Ultrameter II contains algorithms for these 3 most commonly referenced compounds. The solution type in use is displayed on the left. Besides KCl, NaCl, and 442, there is the User choice. The benefit of the User solution type is that one may enter the temperature compensation and TDS ratio by hand, greatly increasing accuracy of readings for a specific solution. That value remains a constant for all measurements and should be reset for different dilutions or temperatures.

C. When does it make a lot of difference?

First, the accuracy of temperature compensation to 25°C determines the accuracy of any TDS conversion. Assume we have industrial process water to be pretreated by RO. Assume it is 45°C and reads 1500 μS uncompensated.

1. If NaCl compensation is used, an instrument would report 1035 μS compensated, which corresponds to 510 ppm NaCl.
2. If 442 compensation is used, an instrument would report 1024 μS compensated, which corresponds to 713 ppm 442. The difference in values is 40%.

In spite of such large error, some users will continue to take data in the NaCl mode because their previous data gathering and process monitoring was done with an older NaCl referenced device.

Selecting the correct Solution Type on the Ultrameter II will allow the user to attain true TDS readings that correspond to evaporated weight.

If none of the 3 standard solutions apply, the User mode must be used. Temperature Compensation (Tempco) and TDS Derivation below, details the User mode.

XXII. TEMPERATURE COMPENSATION (Tempco) and TDS DERIVATION

The Ultrameter II contains internal algorithms for characteristics of the 3 most commonly referenced compounds. The solution type in use is displayed on the left. Besides KCl, NaCl, and 442, there is the User choice. The benefit of User mode is that one may enter the tempco and TDS conversion values of a unique solution via the keypad.

A. Conductivity Characteristics

When taking conductivity measurements, the Solution Selection determines the characteristic assumed as the instrument reports what a measured conductivity would be if it were at 25°C. The characteristic is represented by the tempco, expressed in %/°C. If a solution of 100 μS at 25°C increases to 122 μS at 35°C, then a 22% increase has occurred over this change of 10°C. The solution is then said to have a tempco of 2.2 %/°C.

Tempco always varies among solutions because it is dependent on their individual ionization activity, temperature and concentration. This is why the Ultrameter II features mathematically generated models for known salt characteristics that also vary with concentration and temperature.

B. Finding the Tempco of an Unknown Solution

One may need to measure compensated conductivity of some solution unlike any of the 3 standard salts. In order to enter a custom fixed tempco for a limited measurement range, enter a specific value through the User function. The tempco can be determined by 2 different methods:

1. Heat or cool a sample of the solution to 25°C, and measure its conductivity. Heat or cool the solution to a typical temperature where it is normally measured. After selecting User function, set the tempco to 0 %/°C as in Disabling Temperature Compensation, pg. 15 (No compensation). Measure the new conductivity and the new temperature. Divide the % decrease or increase by the 25°C value. Divide that difference by the temperature difference.

2. Heat or cool a sample of the solution to 25°C, and measure its conductivity. Change the temperature to a typical measuring temperature. Set the tempco to an expected value as in User Programmable Temperature Compensation, pg. 15. See if the compensated value is the same as the 25°C value. If not, raise or lower the tempco and measure again until the 25°C value is read.

C. Finding the TDS Ratio of an Unknown Solution

Once the effect of temperature is removed, the compensated conductivity is a function of the concentration (TDS).

There is a ratio of TDS to compensated conductivity for any solution, which varies with concentration. The ratio is set during calibration in User mode as in User Programmable Conductivity to TDS Ratio, pg. 16.

A truly unknown solution has to have its TDS determined by evaporation and weighing. Then the solution whose TDS is now known can be measured for conductivity and the ratio calculated. Next time the same solution is to be measured, the ratio is known.

XXIII. pH and ORP (6PFC^{E})

A. pH (6PFC^{E})

1. pH as an Indicator (6PFC^{E})

pH is the measurement of Acidity or Alkalinity of an aqueous solution. It is also stated as the Hydrogen Ion activity of a solution. pH measures the effective, not the total, acidity of a solution.

A 4% solution of acetic acid (pH 4, vinegar) can be quite palatable, but a 4% solution of sulfuric acid (pH 0) is a violent poison. pH provides the needed quantitative information by expressing the degree of activity of an acid or base.

In a solution of one known component, pH will indicate concentration indirectly. However, very dilute solutions may be very slow reading, just because the very few ions take time to accumulate.

2. pH Units (6PFC^{E})

The acidity or alkalinity of a solution is a measurement of the relative availabilities of hydrogen (H^{+}) and hydroxide (OH^{-}) ions. An increase in (H^{+}) ions increases acidity, while an increase in (OH^{-}) ions increases alkalinity. The total concentration of ions is fixed as a characteristic of water, and balance would be 10^{-7} mol/liter (H^{+}) and (OH^{-}) ions in a neutral solution (where pH sensors give 0 voltage).

pH is defined as the negative logarithm of hydrogen ion concentration. Where (H^{+}) concentration falls below 10^{-7}, solutions are less acidic than neutral, and therefore are alkaline. A concentration of 10^{-9} mol/liter of (H^{+}) would have 100 times less (H^{+}) ions than (OH^{-}) ions and be called an alkaline solution of pH 9.

3. The pH Sensor (6PFC^{E})

The active part of the pH sensor is a thin glass surface that is selectively receptive to hydrogen ions. Available hydrogen ions in a solution will accumulate on this surface and a charge will build up across the glass interface. The voltage can be measured with a very high impedance voltmeter circuit; the dilemma is how to connect the voltmeter to solution on each side.

The glass surface encloses a captured solution of potassium chloride holding an electrode of silver wire coated with silver chloride. This is the most inert connection possible from a metal to an electrolyte. It can

still produce an offset voltage, but using the same materials to connect to the solution on the other side of the membrane causes the 2 equal offsets to cancel.

The problem is, on the other side of the membrane is an unknown test solution, not potassium chloride. The outside electrode, also called the Reference Junction, is of the same construction with a porous plug in place of a glass barrier to allow the junction fluid to contact the test solution without significant migration of liquids through the plug material. Figure 33 shows a typical 2 component pair. Migration does occur, and this limits the lifetime of a pH junction from depletion of solution inside the reference junction or from contamination. The junction may be damaged if dried out because insoluble crystals may form in a layer, obstructing contact with test solutions. See pH and ORP, pg. 44.

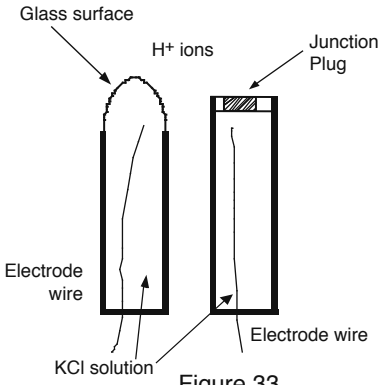


Figure 33

4. The Myron L Integral pH Sensor (6PFC^{E})

The sensor in the Ultrameter II (see Figure 34) is a single construction in an easily replaceable package. The sensor body holds an oversize solution supply for long life. The reference junction "wick" is porous to provide a very stable, low permeable interface, and is located under the glass pH sensing electrode. This construction combines all the best features of any pH sensor known.

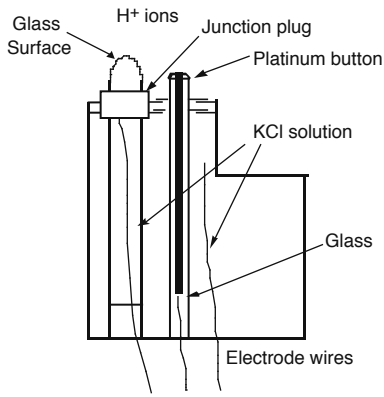


Figure 34

5. Sources of Error (6PFC^{E})

The basics are presented in pH and ORP, pg. 44.

a. Reference Junction

The most common sensor problem will be a clogged junction because a sensor was allowed to dry out. The symptom is a drift in the "zero" setting at 7 pH. This is why the Ultrameter II 6PFC^{E} does not allow more than 1 pH unit of offset during calibration. At that point the junction is unreliable.

b. Sensitivity Problems

Sensitivity is the receptiveness of the glass surface. A film on the surface can diminish sensitivity and cause a long response time.

c. Temperature Compensation

pH sensor glass changes its sensitivity slightly with temperature, so the further from pH 7 one is, the more effect will be seen. A pH of 11 at 40°C would be off by 0.2 units. The Ultrameter II 6PFC^{E} senses the sensor well temperature and compensates the reading.

B. ORP/Oxidation-Reduction Potential/REDOX (6PFC^{E})

1. ORP as an Indicator (6PFC^{E})

ORP is the measurement of the ratio of oxidizing activity to reducing activity in a solution. It is the potential of a solution to give up electrons (oxidize other things) or gain electrons (reduce).

Like acidity and alkalinity, the increase of one is at the expense of the other, so a single voltage is called the Oxidation-Reduction Potential, with a positive voltage showing, a solution wants to steal electrons (oxidizing agent). For instance, chlorinated water will show a positive ORP value.

2. ORP Units (6PFC^{E})

ORP is measured in millivolts, with no correction for solution temperature. Like pH, it is not a measurement of concentration directly, but of activity level. In a solution of only one active component, ORP indicates concentration. Also, as with pH, a very dilute solution will take time to accumulate a readable charge.

3. The ORP Sensor (6PFC^{E})

An ORP sensor uses a small platinum surface to accumulate charge without reacting chemically. That charge is measured relative to the solution, so the solution “ground” voltage comes from a reference junction - same as the pH sensor uses.

4. The Myron L ORP Sensor (6PFC^{E})

Figure 34, pg. 45, shows the platinum button in a glass sleeve. The same reference is used for both the pH and the ORP sensors. Both pH and ORP will indicate 0 for a neutral solution. Calibration at zero compensates for error in the reference junction.

A zero calibration solution for ORP is not practical, so the Ultrameter II 6PFC^{E} uses the offset value determined during calibration to 7 in pH calibration (pH 7 = 0 mV). Sensitivity of the ORP surface is fixed, so there is no gain adjustment either.

5. Sources of Error (6PFC^{E})

The basics are presented in pH and ORP, pg. 44, because sources of error are much the same as for pH. The junction side is the same, and though the platinum surface will not break like the glass pH surface, its protective glass sleeve can be broken. A surface film will slow the response time and diminish sensitivity. It can be cleaned off with detergent or acid, as with the pH glass.

C. Free Chlorine

1. Free Chlorine as an Indicator of Sanitizing Strength

Chlorine, which kills bacteria by way of its power as an oxidizing agent, is the most popular germicide used in water treatment. Chlorine is not only used as a primary disinfectant, but also to establish a sufficient residual level of Free Available Chlorine (FAC) for ongoing disinfection.

FAC is the chlorine that remains after a certain amount is consumed by killing bacteria or reacting with other organic (ammonia, fecal matter) or inorganic (metals, dissolved CO_2, Carbonates, etc) chemicals in solution. Measuring the amount of residual free chlorine in treated water is a well accepted method for determining its effectiveness in microbial control.

The Myron L Company FC^E method for measuring residual disinfecting power is based on ORP, the specific chemical attribute of chlorine (and other oxidizing germicides) that kills bacteria and microbes.

2. FC^E Free Chlorine Units

The 6PIIFC^E is the first handheld device to detect free chlorine directly, by measuring ORP. The ORP value is converted to a concentration reading (ppm) using a conversion table developed by Myron L Company through a series of experiments that precisely controlled chlorine levels and excluded interferants.

Other test methods typically rely on the user visually or digitally interpreting a color change resulting from an added reagent-dye. The reagent used radically alters the sample's pH and converts the various chlorine species present into a single, easily measured species. This ignores the effect of changing pH on free chlorine effectiveness and disregards the fact that some chlorine species are better or worse sanitizers than others.

The Myron L Company 6PIIFC^E avoids these pitfalls. The chemistry of the test sample is left unchanged from the source water. It accounts for the effect of pH on chlorine effectiveness by including pH in its calculation. For these reasons, the Ultrameter II's FC^E feature provides the best reading-to-reading picture of the rise and fall in sanitizing effectivity of free available chlorine.

The 6PIIFC^E also avoids a common undesirable characteristic of other ORP-based methods by including a unique Predictive ORP value in its FC^E calculation. This feature, based on a proprietary model for ORP sensor behavior, calculates a final stabilized ORP value in 1 to 2 minutes rather than the 10 to 15 minutes or more that is typically required for an ORP measurement.

XXIV. SOFTWARE VERSION

Contact the Myron L Company to see if a software upgrade is available.

1. Press COND key.
2. Press MR ▼ key until three numbers are displayed as shown in Figure 35.
3. Press COND key, instrument will time out in ~15 seconds.

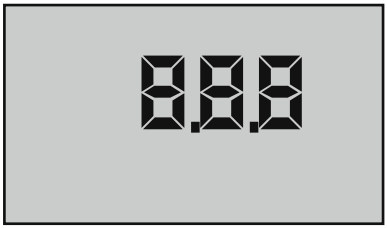


Figure 35

XXV. GLOSSARY

Anions Negatively charged ions.
See Solution Characteristics, pg. 42.

Algorithm A procedure for solving a mathematical problem.
See Temperature Compensation (Tempco) and TDS Derivation, pg. 43.

FAC Free Available Chlorine. The amount of chlorine that remains active in solution and is available for ongoing disinfection. See Free Chlorine as an Indicator, pg. 47.

FC^{E} FC^{E}™ directly measures ORP, the germ killing property of chlorine and other oxidizing germicides. It displays both the ORP reading (in mVDC) as well as an equivalent free chlorine concentration (in familiar ppm). For more information see ***FCE™: Groundbreaking Measurement of Free Chlorine Disinfecting Power in a Hand-Held Instrument*** on the Myron L Company website.

Logarithm An arithmetic function. The inverse of an exponential function. See pH Units, pg. 44.

ORP Oxidation-Reduction Potential or REDOX, See ORP/ Oxidation-Reduction Potential/REDOX, pg. 46.

REDOX Reaction An abbreviation for Reduction-Oxidation reactions. This is the basic electrochemical process by which chlorine destroys microbes by grabbing electrons from the microbe's proteins, denaturing the protein and killing the organism. ORP directly measures the strength of a solutions' REDOX potential and, therefore, sanitizing strength.

TDS Total Dissolved Solids or the Total Conductive Ions in a solution. See Conductivity Conversion to Total Dissolved Solids (TDS), pg. 41.

Tempco Temperature Compensation
See Temperature Compensation of Aqueous Solutions, pg. 39.

User A mode of operation that allows the instrument user (operator) to set a tempco and/or a TDS factor for their specific solution type. See Temperature Compensation of Aqueous Solutions, pg. 39 and Temperature Compensation (Tempco) and TDS Derivation, pg. 43.

For details on specific areas of interest refer to the Table of Contents.

PT1 POCKET TESTER

Conductivity/TDS/Salinity Pen

TEMPERATURE REPORTED WITH EVERY READING

MYRONL.COM

NEW!

EVERYBODY NEEDS ONE

HIGH PERFORMANCE FEATURES:

- Accuracy of ±1% of READING
  ±.2% at Calibration Point
- Reliable Repeatable Results
- KCl, NaCl and 442™ Natural Water Modes
- Automatic Temperature Compensation
- Autoranging
- Durable, Fully Encapsulated Electronics
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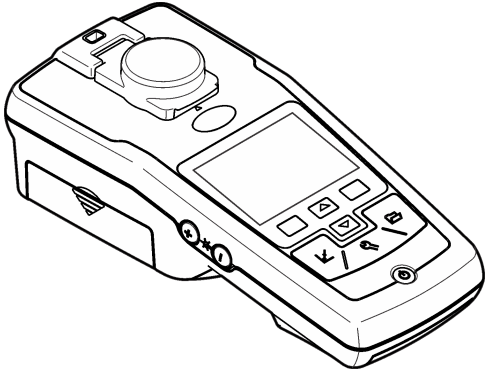
2100Q and 2100Q*is*

04/2013

User Manual



Specifications

Specifications are subject to change without notice.

Specification	Details
Measurement method	Ratio turbidimetric determination using a primary nephelometric light scatter signal (90°) to the transmitted light scatter signal.
Regulatory	**2100Q:** Meets EPA Method 180.1 **2100Q*is*:** Meets ISO 7027
Lamp source	**2100Q:** Tungsten filament lamp **2100Q*is*:** Light-emitting diode (LED) at 860 nm
Range	0–1000 NTU (FNU)
Accuracy	±2% of reading plus stray light from 0–1000 NTU (FNU)
Repeatability	±1% of reading or 0.01 NTU (FNU), whichever is greater
Resolution	0.01 NTU on lowest range
Stray light	≤ 0.02 NTU (FNU)
Signal averaging	Selectable on or off
Detector	Silicon Photodiode
Reading modes	Normal (Push to Read), Signal Averaging or Rapidly Settling Turbidity™
Calibration options	Single step RapidCal™ for Low-Level Regulatory Reporting from 0–40 NTU (FNU) Full range calibration from 0–1000 NTU (FNU) Calibration to degrees of turbidity
Calibration logger	Records the last 25 successful calibrations
Verification logger	Logs the last 250 successful verifications
Data logger	500 records
Power requirement	AC 100–240 V , 50/60 Hz (with power or USB/power module) 4 AA alkaline batteries Rechargeable NiMH (for use with USB/power module)
Operating conditions	Temperature: 0 to 50 °C (32 to 122 °F) Relative Humidity: 0–90% at 30 °C, 0–80% at 40 °C, 0–70% at 50 °C, noncondensing
Storage conditions	–40 to 60 °C (–40 to 140 °F), instrument only
Interface	Optional USB
Sample required	15 mL (0.5 oz.)
Sample cells	Round cells 60 x 25 mm (2.36 x 1 in.) borosilicate glass with screw caps
Dimensions	22.9 x 10.7 x 7.7 cm (9.0 x 4.2 x 3.0 in.)
Weight	530 g (1.17 lb) without batteries 620 g (1.37 lb) with four AA alkaline batteries
Meter enclosure rating	IP67 (closed lid, battery and module compartment excluded)
Protection class	Power supply: Class II
Certification	CE certified
Warranty	1 year (EU: 2 years)

General information

In no event will the manufacturer be liable for direct, indirect, special, incidental or consequential damages resulting from any defect or omission in this manual. The manufacturer reserves the right to make changes in this manual and the products it describes at any time, without notice or obligation. Revised editions are found on the manufacturer's website.

Safety information

NOTICE

The manufacturer is not responsible for any damages due to misapplication or misuse of this product including, without limitation, direct, incidental and consequential damages, and disclaims such damages to the full extent permitted under applicable law. The user is solely responsible to identify critical application risks and install appropriate mechanisms to protect processes during a possible equipment malfunction.

Please read this entire manual before unpacking, setting up or operating this equipment. Pay attention to all danger and caution statements. Failure to do so could result in serious injury to the operator or damage to the equipment.

Make sure that the protection provided by this equipment is not impaired. Do not use or install this equipment in any manner other than that specified in this manual.

Use of hazard information

⚠ DANGER

Indicates a potentially or imminently hazardous situation which, if not avoided, will result in death or serious injury.

⚠ WARNING

Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

⚠ CAUTION

Indicates a potentially hazardous situation that may result in minor or moderate injury.

NOTICE

Indicates a situation which, if not avoided, may cause damage to the instrument. Information that requires special emphasis.

Precautionary labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed. A symbol on the instrument is referenced in the manual with a precautionary statement.

	This is the safety alert symbol. Obey all safety messages that follow this symbol to avoid potential injury. If on the instrument, refer to the instruction manual for operation or safety information.
	This symbol indicates that a risk of electrical shock and/or electrocution exists.
	Electrical equipment marked with this symbol may not be disposed of in European public disposal systems after 12 August of 2005. In conformity with European local and national regulations (EU Directive 2002/96/EC), European electrical equipment users must now return old or end-of-life equipment to the Producer for disposal at no charge to the user. ***Note:*** *For return for recycling, please contact the equipment producer or supplier for instructions on how to return end-of-life equipment, producer-supplied electrical accessories, and all auxiliary items for proper disposal.*

Certification

Canadian Radio Interference-Causing Equipment Regulation, IECS-003, Class A:

Supporting test records reside with the manufacturer.

This Class A digital apparatus meets all requirements of the Canadian Interference-Causing Equipment Regulations.

Cet appareil numérique de classe A répond à toutes les exigences de la réglementation canadienne sur les équipements provoquant des interférences.

FCC Part 15, Class "A" Limits

Supporting test records reside with the manufacturer. The device complies with Part 15 of the FCC Rules. Operation is subject to the following conditions:

1. The equipment may not cause harmful interference.

2. The equipment must accept any interference received, including interference that may cause undesired operation.

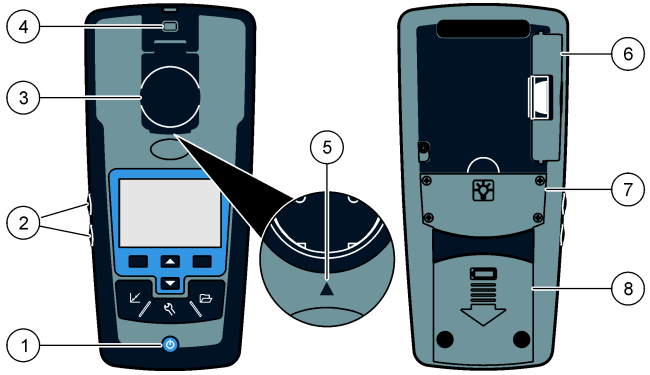
Changes or modifications to this equipment not expressly approved by the party responsible for compliance could void the user's authority to operate the equipment. This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference, in which case the user will be required to correct the interference at their expense. The following techniques can be used to reduce interference problems:

1. Disconnect the equipment from its power source to verify that it is or is not the source of the interference.
2. If the equipment is connected to the same outlet as the device experiencing interference, connect the equipment to a different outlet.
3. Move the equipment away from the device receiving the interference.
4. Reposition the receiving antenna for the device receiving the interference.
5. Try combinations of the above.

Product overview

The 2100Q and 2100Q*is* portable turbidimeters measure turbidity from 0 to 1000 NTU (FNU). Primarily for field use, the portable meter operates on four AA batteries. Data can be stored and transferred to a printer, computer or USB storage device.

Figure 1 Product overview

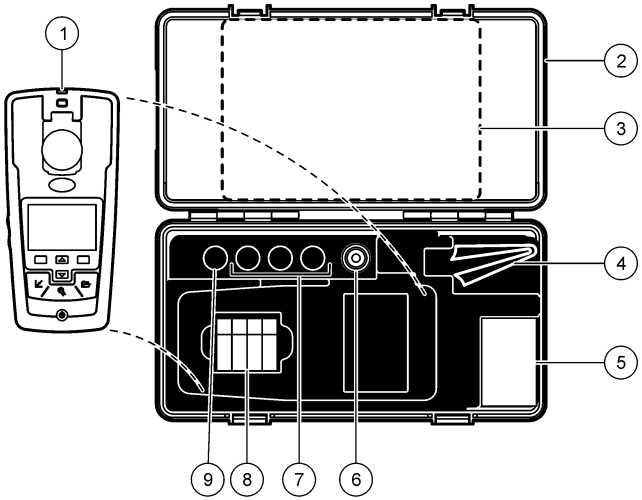


1	Power on or off	**5**	Alignment arrow
2	Backlight keys (+ and -)	**6**	Module
3	Sample cell holder with lid	**7**	Lamp compartment
4	Attachment for lanyard	**8**	Battery compartment

Product components

Refer to Figure 2 to make sure that all components have been received. If any of these items are missing or damaged, contact the manufacturer or a sales representative immediately.

Figure 2 2100Q and 2100Q*is* components



1 2100Q or 2100Q*is* turbidimeter	**6** Silicone oil
2 Carrying case	**7** 20, 100 and 800 NTU StablCal calibration standards
3 User manual, Quick reference guide and CD-ROM	**8** AA alkaline batteries (pk/4)
4 Oiling cloth	**9** StablCal 10 NTU verification standard
5 1" sample cell (10 mL) with cap (pk/6)	

Installation

⚠ CAUTION

Multiple hazards. Only qualified personnel must conduct the tasks described in this section of the document.

Install the battery

⚠ WARNING

Explosion hazard. An expired battery can cause hydrogen gas buildup inside the instrument. Replace the battery before it expires. Do not store the instrument for long periods with a battery installed.

⚠ WARNING

Potential fire hazard. Use only alkaline or nickel metal hydride batteries (NiMH) in the meter. Other battery types or incorrect installation can cause a fire. Never mix battery types in the meter.

NOTICE

The battery compartment is not waterproof. If the battery compartment becomes wet, remove and dry the batteries and dry the interior of the compartment. Check the battery contacts for corrosion and clean them if necessary.

NOTICE

When using nickel metal hydride (NiMH) batteries, the battery icon will not indicate a full charge after freshly charged batteries have been inserted (NiMH batteries are 1.2 V versus 1.5 V for alkaline batteries). Even though the icon does not indicate complete charge, 2300 mAH NiMH batteries will achieve 90% of instrument operation lifetime (before recharge) versus new alkaline batteries.

NOTICE

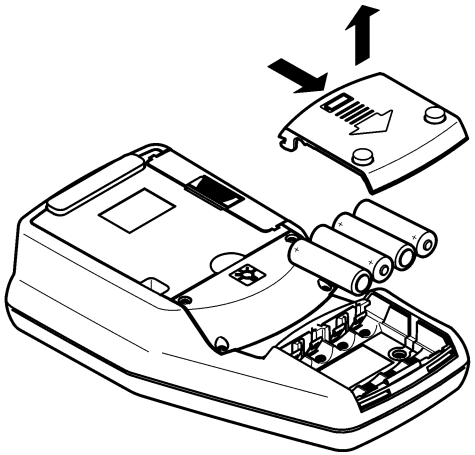
To avoid potential damage to the meter from battery leakage, remove the meter batteries prior to extended periods of non-use.

The meter can be powered with AA alkaline or rechargeable NiMH batteries. To conserve battery life, the meter will power off after 10 minutes of inactivity, the backlight powers off after 30 seconds. This time can be changed in the Power Management menu.
Note: *Rechargeable batteries will only be recharged with the USB/power module. Refer to the module documentation for further information.*

For battery installation refer to Figure 3.

1. Remove the battery cover.
2. Install 4 AA alkaline or 4 AA nickel metal hydride (NiMH) batteries. Make sure that the batteries are installed in the correct orientation.
3. Replace the battery cover.

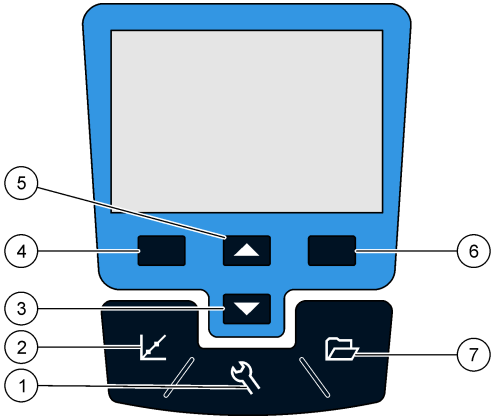
Figure 3 Battery installation



User interface and navigation

User interface

Figure 4 Keypad description

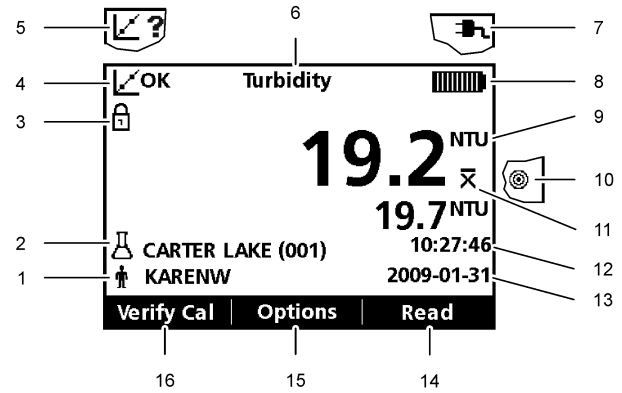


1 **SETTINGS** key: select menu options for setting up the meter	5 **UP** key: scroll through menus, enter numbers and letters
2 **CALIBRATION** key: shows calibration screen, start calibration, select cal options	6 **RIGHT** key (contextual): read turbidity sample, selects or confirms options, opens/jumps to sub-menus
3 **DOWN** key: scroll through menus, enter numbers and letters	7 **DATA MANAGEMENT** key: view, delete or transfer stored data
4 **LEFT** key (contextual): access for calibration verification, cancels or exits the current menu screen to the previous menu screen	

Display description

The measurement screen shows the turbidity, unit, calibration status, date and time, operator ID (if setup) and sample ID (if setup). Refer to Figure 5.

Figure 5 Single screen display



1 Operator identification	**9** NTU (Nephelometric Turbidity Unit) or FNU (Formazin Turbidity Unit)
2 Sample identification	**10** Reading mode: Rapidly Settling Turbidity (Target icon)
3 Stability or display lock indicator	**11** Reading mode: Signal Average (X-bar icon)
4 Calibration status indicator (Calibration OK=pass)	**12** Time
5 Calibration status indicator (Calibration ?=fail)	**13** Date
6 Parameter title	**14** Read (contextual: OK, Select)
7 AC power icon	**15** Options (contextual)
8 Battery icon	**16** Verification calibration

Navigation

The meter contains a Settings menu, Reading Options menu, Calibration Options menu and Calibration Verification Options menu to change various options. Use the **UP** and **DOWN** keys to highlight different options. Push the **RIGHT** key to select an option. There are two ways to change options:

1. Select an option from a list: Use the **UP** and **DOWN** keys to select an option. If check boxes are shown, more than one option can be selected. Push the **LEFT** key under Select.

   Note: *To deselect check boxes, push the **LEFT** key under Deselect.*
2. Enter an option value using the arrow keys:
   Push the **UP** and **DOWN** keys to enter or change a value.
3. Push the **RIGHT** key to advance to the next space.
4. Push the **RIGHT** key under **OK** to accept the value.

Startup

Turn the meter on and off

Push the **ON/OFF** key to turn on or turn off the meter. If the meter does not turn on, make sure that the batteries, or the module, are properly installed or that the AC power supply is properly connected to an electrical outlet.

Note: *The Auto-Shutoff option can also be used to turn off the meter. Refer to* Power management *on page 13.*

Change the language

There are three options to set the language:

- The display language is selected when the meter is powered on for the first time.
- The display language is selected when the power key is pushed and held.
- The language can be changed from the Settings menu.

1. Select a language from the list. Confirm with **OK**.
2. Push **Done** when the update is complete.

Change the date and time

The date and time can be changed from the Date & Time menu.

1. Push the **SETTINGS** key and select Date & Time.
2. Update the time and date information:

Option	Description
Format	Select one of the formats for the date and time: yyyy-mm-dd 24h yyyy-mm-dd 12h dd-mm-yyyy 24h dd-mm-yyyy 12h mm/dd/yyyy 24h mm/dd/yyyy 12h
Date	Enter the current date
Time	Enter the current time

The current date and time will be shown on the display.

After the date and time setup, the meter is ready to take a reading.

Standard operation

Use a sample ID

The sample ID tag is used to associate readings with a particular sample location. If assigned, stored data will include this ID.

1. Select **Sample ID** in the Settings menu.
2. Select, create or delete a sample ID:

Option	Description
Current ID	Select an ID from a list. The current ID will be associated with sample data until a different ID is selected.
Create a New Sample ID	Enter a name for a new sample ID.
Delete Sample ID	Delete an existing sample ID.

Use an operator ID

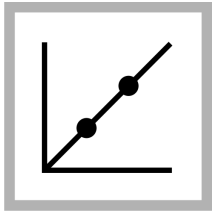
The operator ID tag associates readings with an individual operator. All stored data will include this ID.

1. Select **Operator ID** in the Settings menu.
2. Select, create or delete an operator ID:

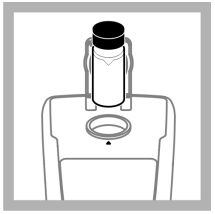
Option	Description
Current ID	Select an ID from a list. The current ID will be associated with sample data until a different ID is selected.
Create a New Operator ID	Enter a name for a new operator ID (maximum 10 names can be entered).
Delete Operator ID	Delete an existing operator ID.

Calibrate the turbidimeter with StablCal® Standards

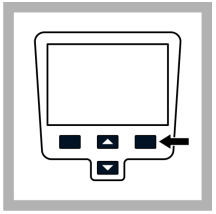
Note: *For best accuracy use the same sample cell or four matched sample cells for all readings during calibration. Insert the sample cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment.*



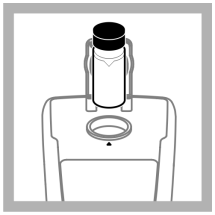
1. Push the **CALIBRATION** key to enter the Calibration mode. Follow the instructions on the display.
Note: *Gently invert each standard before inserting the standard.*



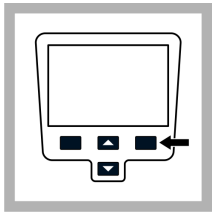
2. Insert the 20 NTU StablCal Standard and close the lid.
Note: *The standard to be inserted is bordered.*



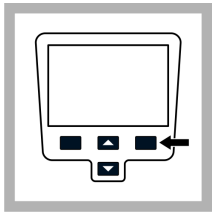
3. Push **Read**. The display shows Stabilizing and then shows the result.



4. Repeat Step 2 and 3 with the 100 NTU and 800 NTU StablCal Standard.
Note: *Push **Done** to complete a 2 point calibration.*



5. Push **Done** to review the calibration details.



6. Push **Store** to save the results.
After a calibration is complete, the meter automatically goes into the Verify Cal mode. Refer to Verification calibration.

Turbidity measurement

⚠WARNING

Potential explosion and fire hazard. This turbidimeter is designed for water based samples. Do not measure solvent or combustible based samples.

Readings can be taken with the Normal reading mode, Signal Average mode or in the Rapidly Settling Turbidity mode. Refer to Reading modes on page 17 for more information. For accurate turbidity readings use clean sample cells and remove air bubbles (degassing).

Measurement notes

Proper measurement techniques are important in minimizing the effects of instrument variation, stray light and air bubbles. Use the following measurement notes for proper measurements.

Instrument

- Make sure that the meter is placed on a level, stationary surface during the measurement.
  Note: *Do not hold the meter in the hand during measurement.*
- Always close the sample compartment lid during measurement, calibration and storage.

- Remove sample cell and batteries from the instrument if the instrument is stored for an extended time period (more than a month).
- Keep the sample compartment lid closed to prevent the entry of dust and dirt.

Sample cells

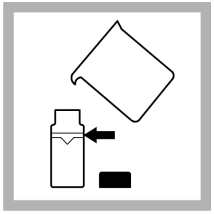
- Always cap the sample cell to prevent spillage of the sample into the instrument.
- Always use clean sample cells in good condition. Dirty, scratched or damaged cells can cause inaccurate readings.
- Make sure that cold samples do not “fog” the sample cell.
- Store sample cells filled with distilled or deionized water and cap tightly.

Measurement

- Measure samples immediately to prevent temperature changes and settling. Before a measurement is taken, always make sure that the sample is homogeneous throughout.
- Avoid sample dilution when possible.
- Avoid operation in direct sunlight.

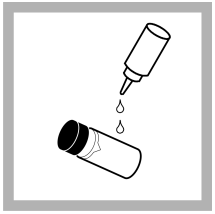
Turbidity measurement procedure

Note: *Before a measurement is taken, always make sure that the sample is homogeneous throughout.*



1. Collect a representative sample in a clean container. Fill a sample cell to the line (about 15 mL). Take care to handle the sample cell by the top. Cap the cell.

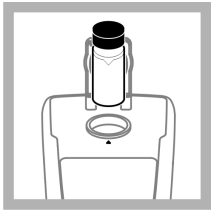
2. Wipe the cell with a soft, lint-free cloth to remove water spots and fingerprints.



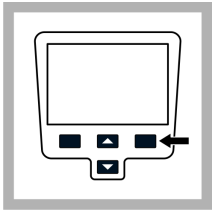
3. Apply a thin film of silicone oil. Wipe with a soft cloth to obtain an even film over the entire surface (Apply silicone oil to a sample cell).

4. Push the **Power** key to turn the meter on. Place the instrument on a flat, sturdy surface.

Note: *Do not hold the instrument while making measurements.*



5. Gently invert and then insert the sample cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment. Close the lid.



6. Push **Read**. The display shows Stabilizing then the turbidity in NTU (FNU). The result is shown and stored automatically. For additional information, refer to the CD for an expanded version of this manual.

Data management

About stored data

The following types of data are stored in the data log:

- Reading Log: stores automatically each time a sample reading is taken (500 records).
- Calibration Log: stores only when **Store** is selected at the end of a calibration (25 records).
- Verify Cal Log: stores only after **Done** is selected at the end of a verification calibration (250 records).

When the data log becomes full, the oldest data point is deleted when more data is added to the log.

View data log

The data log contains Reading Log, Calibration Log and Verify Cal log. All logs can be sorted by date.

1. Push the **DATA MANAGEMENT** key.
2. Select View Data Log to view the stored data.
3. Push **Select** to view additional information.

Option	Description
Reading Log	Reading Log—shows the date, time and reading mode and associated calibration data.
Calibration Log	Calibration Log—shows the date and time of calibration data and additional information about the calibration.
Verify Cal Log	Verify Cal Log—shows the calibration verification date and time and additional information about the verification.
All Logs by Date	The most recent data and additional information is shown. The icons show whether the data is from a reading, calibration or calibration verification and identifies the reading mode, if applicable.

Delete data log

There are two possibilities to delete stored readings in the Data Management menu:

1. Push the **DATA MANAGEMENT** key and select Delete Data Log.

Option	Description
Delete Last Reading	Only the last reading stored can be deleted until a new reading is taken and stored.
Delete All Logs	The entire Reading Log can be deleted at once.

Send stored data

Data can be stored and transferred to a printer, computer or USB storage device. The data will be formatted as an XML file. Install the USB/power module to the meter and to AC power. Refer to the module documentation for more information.

Advanced operation

Display contrast

1. Push the **SETTINGS** key and select Display Contrast.
2. Use the **UP** and **DOWN** key to adjust the contrast of the display and push OK.

Power management

Use power management to change the backlight option and the battery saving auto-shutoff option.
Note: *Power management is not active when the meter is connected to AC power.*

1. Push the **SETTINGS** key and select Power Management.
2. Select which display option to change.

Option	Description
Backlight	The display is illuminated. To maximize battery life, select a time period after which the backlight will automatically power off if no key is pushed: 10 s, 20 s, 30 s,1 min, 2 min, 5 min ***Note:*** *The Backlight keys (Product overview) will turn the backlight on and off.*
Auto-Shutoff	To maximize battery life, set a time period after which the meter will automatically power off if no key is pushed: 1 min, 2 min, 5 min, 10 min, 30 min, 1 h

Set the sound options

The meter can make an audible sound when a key is pushed, when a reading is complete or when the calibration reminder is due.

1. Push **SETTINGS** and select Sounds.
2. Select which events will produce an audible sound. Multiple items can be selected.

Option	Description
Key Press	The meter will make an audible sound whenever a key is pushed.
Reading complete	The meter will make an audible sound whenever a reading is completed.
Reminders	The meter will make an audible sound when a calibration is due.

Security options

The Security Options menu is used to protect the meter setup.

The Setup Date and Time, Delete Data Log, Restoring Factory Defaults and Restore Factory Cal screens are not accessible without a password.

Store the password in a safe and accessible place. If the specified password is forgotten and Security Options is turned on, the operator is locked out of the restricted menus. Contact technical support if the password is lost.

Turn security options on

The security options and the set password options are used together to prevent access to restricted menus.

1. Push the **SETTINGS** key and select Security Options.
2. Select Edit Password and use the **UP** and **DOWN** keys to set a password.
3. Select Security On to enable the password setting. The requirement for the password entry is controlled by setting Security Options on or off.
   Note: *Set the Security to Off to disable the password setting.*
4. Push the **ON/OFF** key to turn off and on the meter to activate the password settings.

View meter information

The instrument information menu shows specific information such as the meter name, model number, software version, serial number and available Operator and Sample IDs. 10 Operator IDs and 100 Sample IDs are available.

1. Push the **SETTINGS** key and select Meter Information.

Calibration

The portable turbidimeter is calibrated with Formazin Primary Standards at the factory. The meter should be calibrated upon receipt for best results. The manufacturer recommends calibration with a primary standard such as StablCal® Stabilized Standards or with formazin standards every three months.

Note: *Set* ***Cal Reminder Repeat*** *in the Calibration Options menu for periodical calibration. Verify the calibration once a week.*

Calibration options

The calibration options contain Calibration History, Calibration Curves, Cal Reminder Repeat and Restore Factory Calibration.

1. Push the **CALIBRATION** key and then the **UP**and **DOWN** key.

Option	Description
Calibration History	The calibration history shows a list of the times when the meter was calibrated. Select a date and time to view a summary of the calibration data.
Cal.Curve	Select one of the calibration curves for calibration: StablCal® RapidCal™ (0–40 NTU) StablCal® (0–1000 NTU) Formazin RapidCal™ (0–40 NTU) Formazin (0–1000 NTU) Degrees (0–100 mg/L) SDVB (0–1000 NTU) Custom (0–1000 NTU)
Cal Reminder Repeat	The meter will make an audible sound when calibration is due. Select one of the following options for time interval and push OK: Off, 1 d, 7 d, 30 d, 60 d, 90 d
Restore Factory Calibration	All user calibrations will be deleted. The original factory calibration is restored.

Calibration standard overview

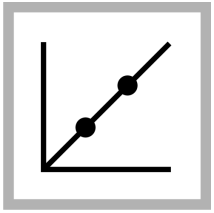
Refer to Calibration standard overview for the calibration standard overview.

Table 1 Calibration overview

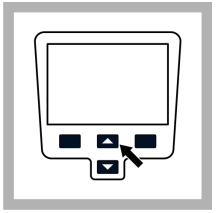
Type of calibration	Required standards			
StablCal® RapidCal™ (0–40 NTU)	–	20 NTU	–	–
StablCal® (0–1000 NTU)	–	20 NTU	100 NTU	800 NTU
Formazin RapidCal™ (0–40 NTU)	Typically deionized or distilled waterFootnote	20 NTU	–	–
Formazin (0–1000 NTU)	Typically deionized or distilled waterFootnote	20 NTU	100 NTU	800 NTU
Degrees (0–100 mg/L)	Typically deionized or distilled waterFootnote	20 NTU	100 NTU	–
SDVB (0–1000 NTU)	Typically deionized or distilled waterFootnote	20 NTU	100 NTU	800 NTU
Custom (0–1000 NTU)	Typically deionized or distilled waterFootnote	Select values		

[1] The water must have a turbidity <0.5 NTU to prepare the calibration standards.

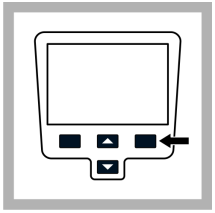
StablCal® RapidCal™ calibration



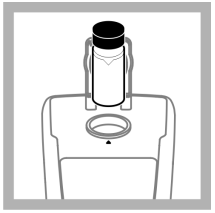
1. Push the **CALIBRATION** key to enter the Calibration mode. Follow the instructions on the display.
Note: *Gently invert each standard before inserting the standard.*



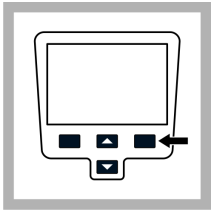
2. Push the **UP** and **DOWN** key to access Cal Options and then select Cal.Curve.



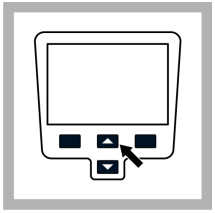
3. Select StablCal® RapidCal™ from the list and push **OK**.



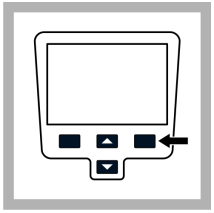
4. Insert the 20 NTU StablCal Standard and close the lid.
Note: *The standard to be inserted is bordered.*



5. Push **Read**. The display shows Stabilizing and then shows the result.



6. Push **Done** to review the calibration details.



7. Push **Store** to save the results.
After a calibration is complete, the meter automatically goes into the Verify Cal mode, refer to Verification calibration.

Verification options

The Verification Options contain: Set Verification Standard, Set Acceptance Criteria and Verification Reminder.

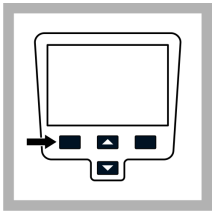
1. Push the **Left** key (Verify Cal) and then the **UP** and **DOWN** keys.

Option	Description
Set Verification Standard	To change the verification standard use the **UP** and **DOWN** keys to enter a new standard value. Range 0.50–20.0 NTU (Default setting: 10.00 NTU) Range 0–20 NTU for RapidCal™ (0–40 NTU) 0–800 NTU for calibration curves with a range from 0–1000 NTU
Set Acceptance Criteria	Enter the Acceptance Criteria for comparison against the initial calibration verification reading to determine passing or failing. Range 1–50% (Default setting: 10%)
Verification Reminder	Verification Reminder—The meter will make an audible sound when verification is due. Select one of the following options for time interval and push **OK**:Off, 30 min (Default setting), 2 h, 4 h, 8 h, 24 h Allow Defer—Push **Allow Defer** and select Yes or No to postpone the verification due time

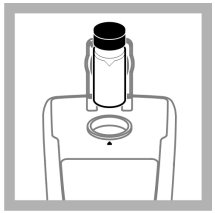
Calibration verification (Verify Cal)

The manufacturer recommends a calibration verification once a week. After a calibration is complete, the meter automatically goes into the Verify Cal mode.

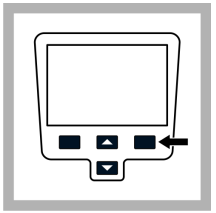
Make sure that the sample cell is clean. Oil the sample cell with silicone oil, refer to Apply silicone oil to a sample cell. Check the standard solution. Prepare a formazin standard at the same value and read the value.



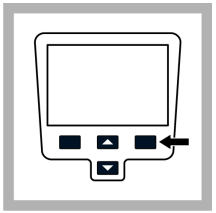
1. Push **Verify Cal** to enter the Verify menu.



2. Gently invert the standard. Insert the 10.0 NTU (or other defined value) Verification Standard and close the lid.



3. Push **Read**. The display shows Stabilizing and then shows the result and tolerance range.



4. Push **Done** to return to the reading display. Repeat the calibration verification if the verification failed.

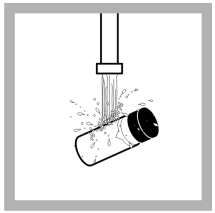
Reading modes

1. Push the **UP** or **DOWN** key to enter the Reading Options menu.
2. Select Reading Mode to select one of the following options:

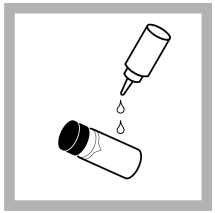
Option	Description
Normal (Default setting)	The normal mode reads and averages three readings. The result is shown after the reading.
Signal Average 	The Signal Average mode compensates for reading fluctuations caused by drifting of sample particles through the light path. The X-bar icon is shown on the display when signal averaging is on. The Signal Average mode measures 12 times and starts to show the average after three readings. The final result is the average of all 12 readings.
Rapidly Settling Turbidity™ (RST) 	The Rapidly Settling Turbidity (RST) mode calculates and continuously updates the turbidity reading of the sample to a confidence of 95%, based on the accumulated trend of the real time measured values. The RST mode is best used on samples that settle rapidly and continuously change in value. The reading is based on a correctly prepared sample that is homogeneous at the beginning of the reading. It is best applied to samples that are greater than 20 NTU. The sample must be mixed thoroughly by inversion immediately before inserting it into the meter. The target icon is shown on the display when the Rapidly Settling Turbidity is on. The Rapidly Settling Turbidity reads and calculates five readings while showing intermediate results.

Apply silicone oil to a sample cell

Sample cells and caps must be extremely clean and free from significant scratches. Apply a thin coating of silicone oil on the outside of the sample cells to mask minor imperfections and scratches that may contribute to light scattering.
Note: *Use only the provided silicone oil. This silicone oil has the same refractive index as the sample cell glass.*



1. Clean the inside and outside of the cells and caps by washing with a laboratory glass cleaning detergent. Follow with multiple rinses with distilled or demineralized water.

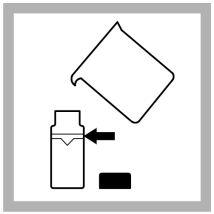


2. Apply a small bead of silicone oil from the top to the bottom of the cell.

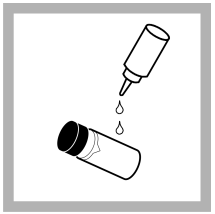
3. Use the provided oiling cloth to spread the oil uniformly. Wipe off the excess so that only a thin coat of oil is left. Make sure that the sample cell is almost dry with little or no visible oil.
Note: *Store the oiling cloth in a plastic storage bag to keep the cloth clean.*

Indexing a single cell

Precise measurements for very low turbidity samples require the use of a single cell for all measurements or optically matching the cells. Use one cell to provide the best precision and repeatability. When one cell is used, an index or orientation mark (other than the factory-placed diamond) can be placed on the cell so it is inserted into the instrument with the same orientation each time.
When using a single cell, make an index or orientation mark on the cell as follows:

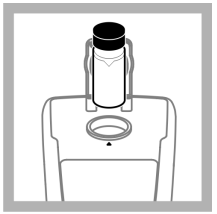


1. Fill the clean sample cell to the line with high quality water (< 0.5 NTU) and cap immediately. Let the sample cell degas for at least five minutes.

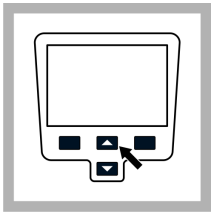


2. Wipe with lint-free cloth. Apply a thin film of silicone oil (Apply silicone oil to a sample cell).

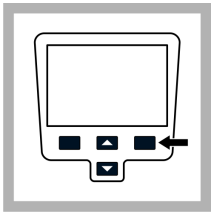
3. Push the **POWER** key to turn the meter on. Place the instrument on a flat, sturdy surface.
Note: *Do not hold the instrument while making measurements.*



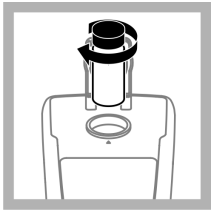
4. Insert the sample cell in the instrument cell compartment so the diamond or orientation mark always aligns with the raised orientation mark in front of the cell compartment. Close the lid.



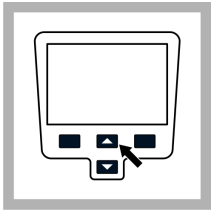
5. Push the **UP** and **DOWN** key to access the Reading Options and then select Indexing Sample Cell.
Note: *The instruments always stays in the last selected reading mode.*



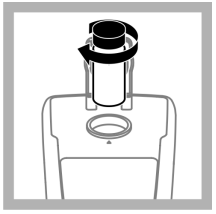
6. Push **Read**. The display shows Stabilizing then the turbidity in NTU. Record the cell position in the cell compartment and the reading result.



7. Remove the cell, rotate it slightly approximately ⅛ of a turn and insert it again into the cell compartment. Close the lid.



8. Push **Read**. Record the cell position in the cell compartment and the reading result.



9. Repeat step 6 until the lowest reading is shown. Place an orientation mark on the cell marking band near the top of the cell so the cell can be consistently inserted in the position that yields the lowest reading.

Maintenance

⚠ CAUTION

Multiple hazards. Only qualified personnel must conduct the tasks described in this section of the document.

Clean the meter

The meter is designed to be maintenance-free and does not require regular cleaning for normal operation. Exterior surfaces of the meter may be cleaned as necessary.

Note: *Do not clean the meter with solvents to avoid damaging the material.*

1. Clean the meter with a dust- and lint-free dry or slightly damp cloth. A mild soap solution can also be used for liposoluble contamination.

Store the sample cells

NOTICE

Do not air dry the sample cells.

Note: *Always store the sample cells with caps on to prevent the cells from drying.*

1. Fill the sample cells with distilled or demineralized water.
2. Cap and store the sample cells.
3. Wipe the outside of the sample cells dry with the a soft cloth.

Replace the battery

⚠ WARNING

Explosion hazard. An expired battery can cause hydrogen gas buildup inside the instrument. Replace the battery before it expires. Do not store the instrument for long periods with a battery installed.

⚠ WARNING

Potential fire hazard. Use only alkaline or nickel metal hydride batteries (NiMH) in the meter. Other battery types or incorrect installation can cause a fire. Never mix battery types in the meter.

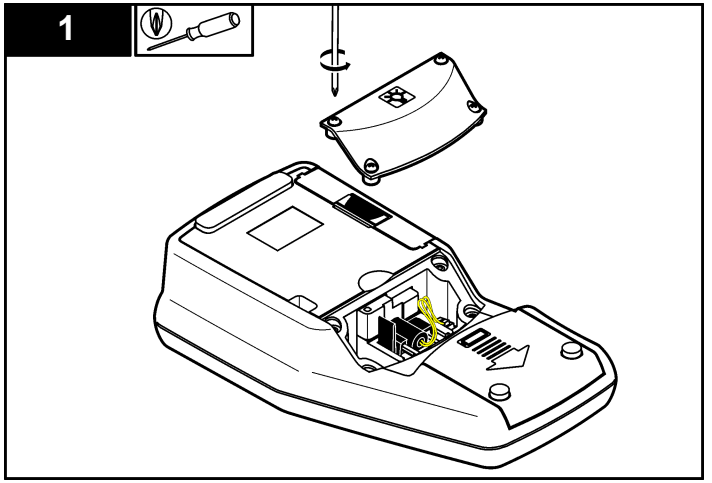
For battery replacement refer to Install the battery on page 6.

1. Remove the battery cover.
2. Remove the batteries.
3. Install 4 AA alkaline or 4 AA nickel metal hydride (NiMH) batteries. Make sure that the batteries are installed in the correct orientation.
4. Replace the battery cover.

Replace the lamp

⚠ CAUTION

Burn Hazard. Wait until lamp cools down. Contact with the hot lamp can cause burns.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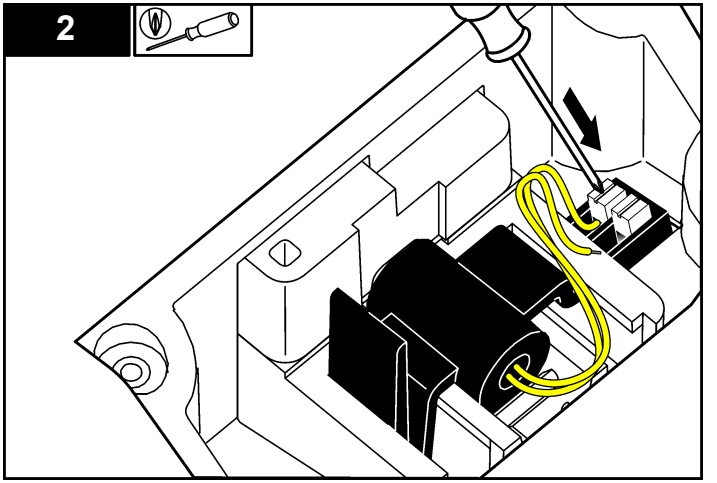
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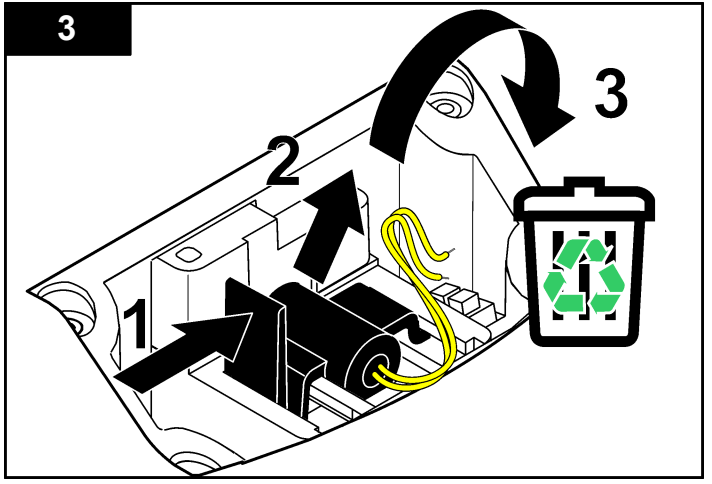
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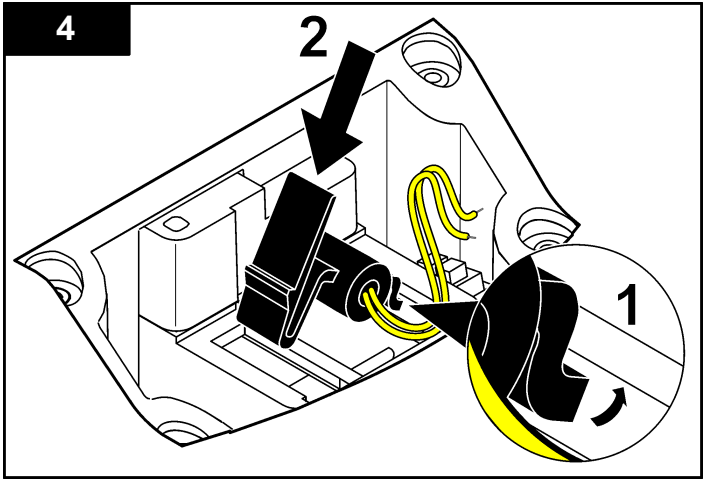
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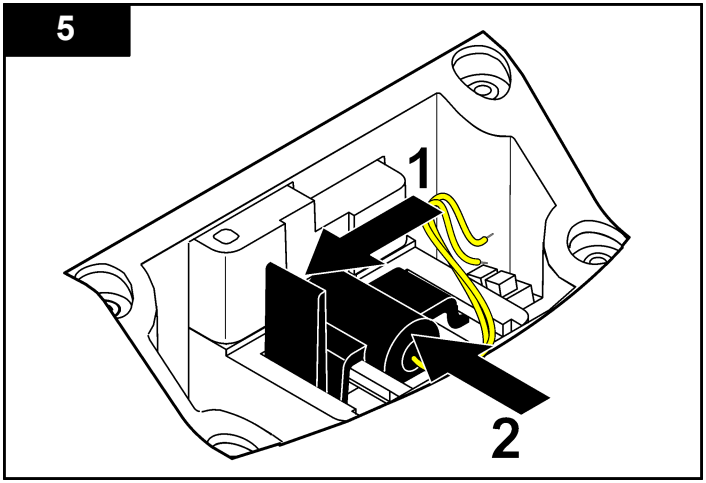
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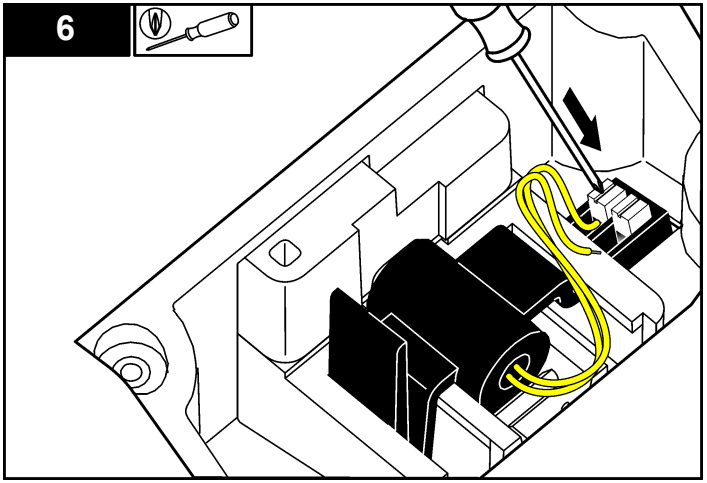
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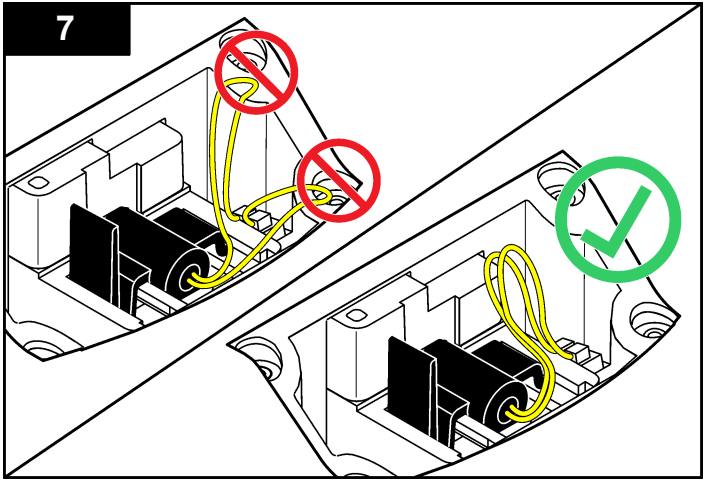
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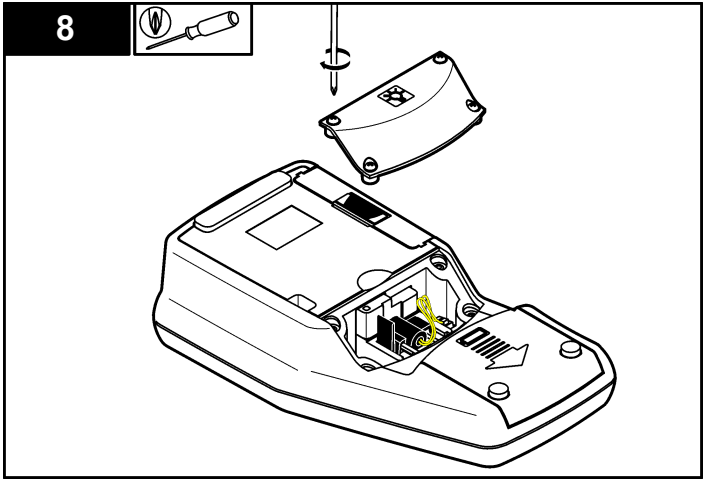
8

Troubleshooting

Refer to the following table for common problem messages or symptoms, possible causes and corrective actions.

Error/Warning	Description	Solution
Close lid and push Read.	The lid is open or lid detection failed.	Make sure that the lid is closed during reading and re-read.
Low Battery!	Battery is low.	• Insert new batteries • Connect USB/power module if rechargeable batteries are used
ADC Failure!	Hardware error causing reading to fail.	Repeat the reading.
Detector signal too low!	Insufficient light on the 180° detector.	• Check for obstructed light path. • Check the lamp.
Overrange!	Turbidity too high-caused probably by calibrating with RapidCal™ only.	• Calibrate the upper range. • Dilute the sample.
Underrange!	The measured absorbance is below the calibration range.	Repeat calibration
Please check the lamp!	Signals are too low on the 90° and 180° detector.	**2100Q:** The lamp is defective. Change the lamp (refer to Replace the lamp on page 19). **2100Q*is*:** Contact technical support.
Temperature too high! Switch off instrument.	Temperature has exceeded the meter limits (>60 °C or >140 °F).	Turn off the meter and let it cool down.
RST: Average value!	Solids are settling too slowly. The reading mode is not suitable for this sample.	Select Normal or Signal Average reading mode.
Confidence level is < 95%	The reading mode Rapidly Settling Turbidity did not meet the range of ≥ 95% confidence.	• Invert the sample several times so that the solids allocate. Repeat the reading again. • Switch to the Normal reading mode if the sample is stable and does not have settable solids.
Standard value out of range. Insert standard and push Read	Used incorrect standard value for the reading.	Insert the appropriate standard and read again.
ID already in use. Enter new ID	The Operator or Sample ID is unavailable as it is already assigned.	Create a new ID.
Error - Security Please set password before activating security	No password is created.	Create a new password.
Please enter at least one character.	Password must contain minimum of one character.	Create a password of at least one character.
Password incorrect. Please retry.	Incorrect password was entered.	Enter the appropriate password.

Error/Warning	Description	Solution
Please disconnect the USB cable from your computer.	Data storage does not respond while connected to the meter and the computer.	Disconnect the USB cable from the meter and try sending data again.
USB module memory full. Delete data and try again.	Data storage is full.	**1.** Connect USB/power module to the computer. **2.** Download the stored data to the computer. **3.** Delete Data Log on the module.
Delete Last Reading Failed!	Error in the data storage.	Turn the meter off and on. If the error message still occurs, contact technical support.
Delete Data Log failed!		
Can't read data set!		
Can't store data!		
Can't store to the Reading Log!		
Can't store to the Verify Cal Log!		
Error storing data!		
Error reading data!		

Replacement parts and accessories

Replacement parts

Description	Quantity	Item no.
StablCal ampule calibration kit	1	2971205
10 NTU verification standard	100 mL	2961701
Silicone Oil	15 mL	126936

Replacement parts (continued)

Description	Quantity	Item no.
Insert, molded bottom	1	2971507
Sample cell oiling cloth	1	4707600
1" glass sample cell (10 ml) w/cap (Turb)	pkg/6	2434706
Carrying case (includes insert)	1	2971500
Battery set, AA alkaline batteries	pkg/4	1938004
Lamp assy	1	4653900
Blank module	1	LZV797
Rubber foot set	1	LZV821
Lamp cover (includes screws)	1	LZV822
Battery cover (includes 2 feet)	1	LZV823
Module cover	1	LZV824
Connector cover for USB/power module	1	LZV825
Connector cover for power module	1	LZV826
Lid (includes magnet)	1	LZV827

Accessories

Description	Quantity	Item no.
USB/power module (includes: universal power supply, USB cable, instruction sheet)	1	LZV813.99.00002[1]
Power module (includes: universal power supply, instruction sheet)	1	LZV804.99.00002[1]
USB module with USB cable (2x)	1	LZV949.99.00002[1]
StablCal 0.1 NTU Standard	100 mL	2723342

Accessories (continued)

Description	Quantity	Item no.
StablCal 0.3 NTU Standard	100 mL	2697943
StablCal 0.5 NTU Standard	100 mL	2698042
StablCal calibration kit	100 mL	2971210
StablCal calibration kit	500 mL	2971200
Gelex secondary standards set	1	2464105
Deionized water	4 vials	27217
Filter	0.2 micron	2323810
Formazin	500 ml	246149
Formazin	1000 ml	246142
Sample degassing kit	1	4397500
Sample degassing and filtration kit	1	4397510
Battery, NiMH AA	pk/4	2971304

[1] Not available in all regions

V

HACH COMPANY World Headquarters
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Fax (970) 669-2932
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DR/890
COLORIMETER
PROCEDURES MANUAL

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TABLE OF CONTENTS

TABLE OF CONTENTS, continued

TABLE OF CONTENTS, continued

TABLE OF CONTENTS, continued

TABLE OF CONTENTS, continued

INTRODUCTION

This manual is divided into five sections:

Section 1 Chemical Analysis Information

This section applies to all the procedures. It provides background information and reference/review material for the technician or chemist. Commonly used techniques are explained in detail.

Section 2 Sample Pretreatment

This section provides a brief overview of sample pretreatment and two USEPA digestions. A brief discussion of the Hach Digesdahl Digestion Apparatus and the Hach Distillation Apparatus is included.

Section 3 Waste Management and Safety

Section 3 includes information on waste management, regulations, waste disposal and resources on waste management. The Safety portion covers reading an MSDS and general safety guidelines.

Section 4 Procedures

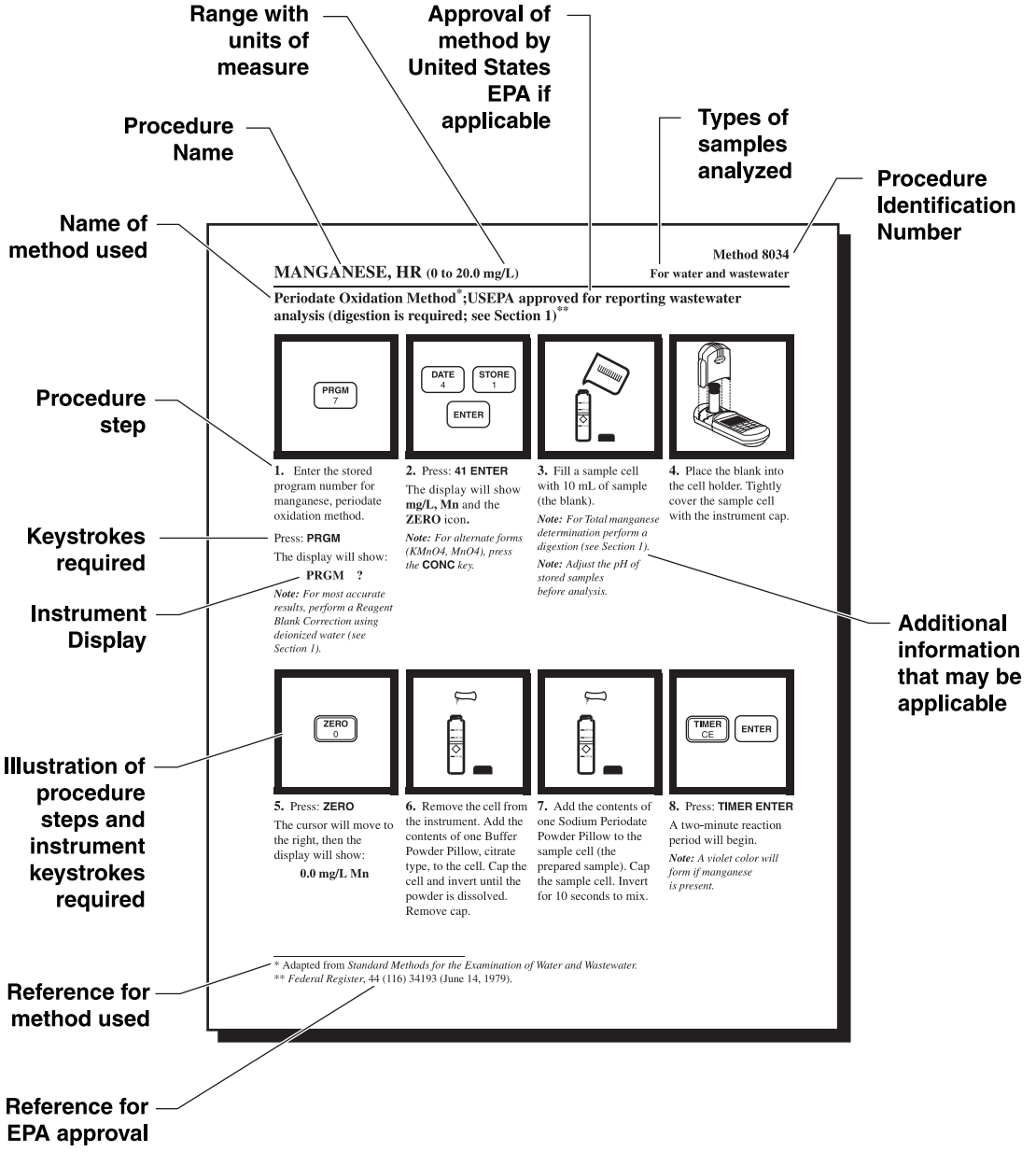
Section 4 contains step-by-step illustrated instructions for measuring parameters. The steps also include helpful notes. Each procedure contains information on sample collection, storage and preservation, accuracy checks, possible interferences, summary of method and a list of the reagents and apparatus necessary to run the test.

Section 5 Ordering Information

This section provides information needed for ordering, shipping, return of items and Hach trademarks.

Before attempting the analysis procedures the analyst should read the instrument manual to learn about the colorimeter's features and operation.

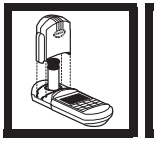
Sample Procedure Explained



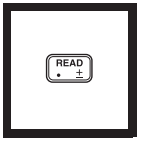
Specific sampling and storage information for this test

Confirm accuracy with these steps (in addition, may also be used to troubleshoot a test, improve technique, check reagents and to assure cleanliness of glassware)

MANGANESE, HR, continued



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L manganese will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-washed plastic bottles. Manganese may be lost by adsorption to glass container walls. Adjust the pH to less than 2 with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as manganese may be lost as a precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information. If only dissolved Mn is to be determined, filter before acid addition.

Accuracy Check

Standard Additions Method

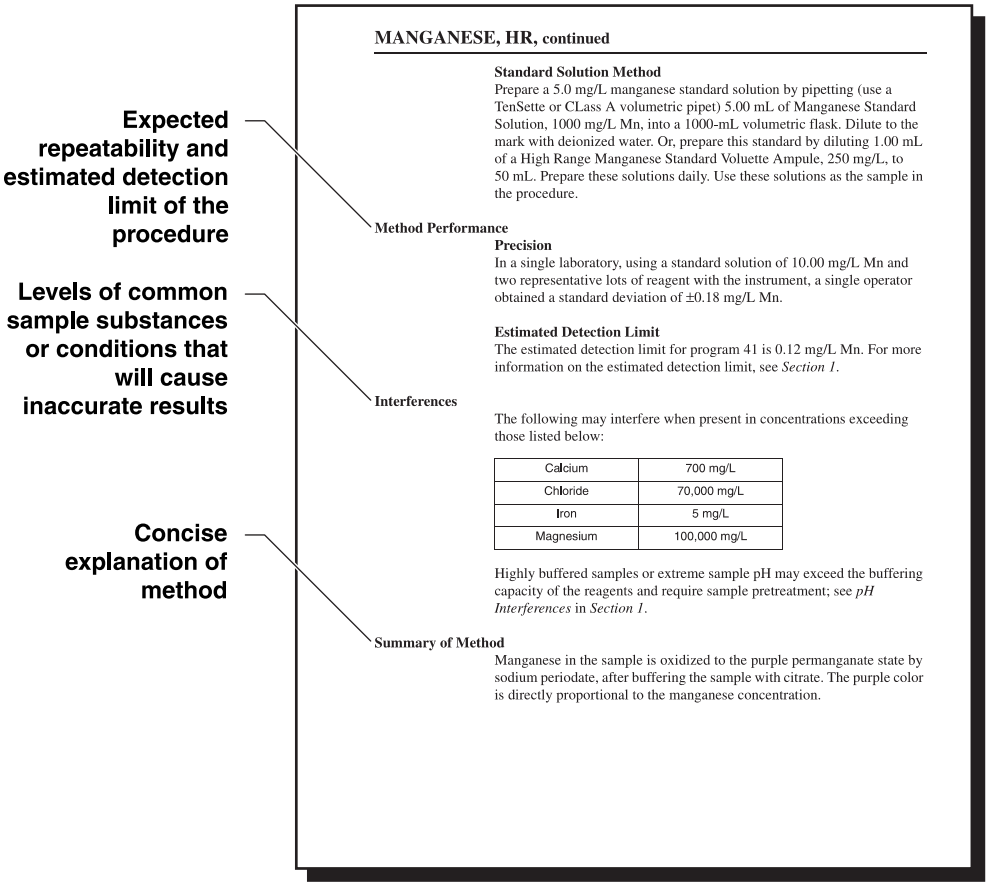
a) Snap the neck off a Manganese Voluette Ampule Standard Solution, 250 mg/L Mn.

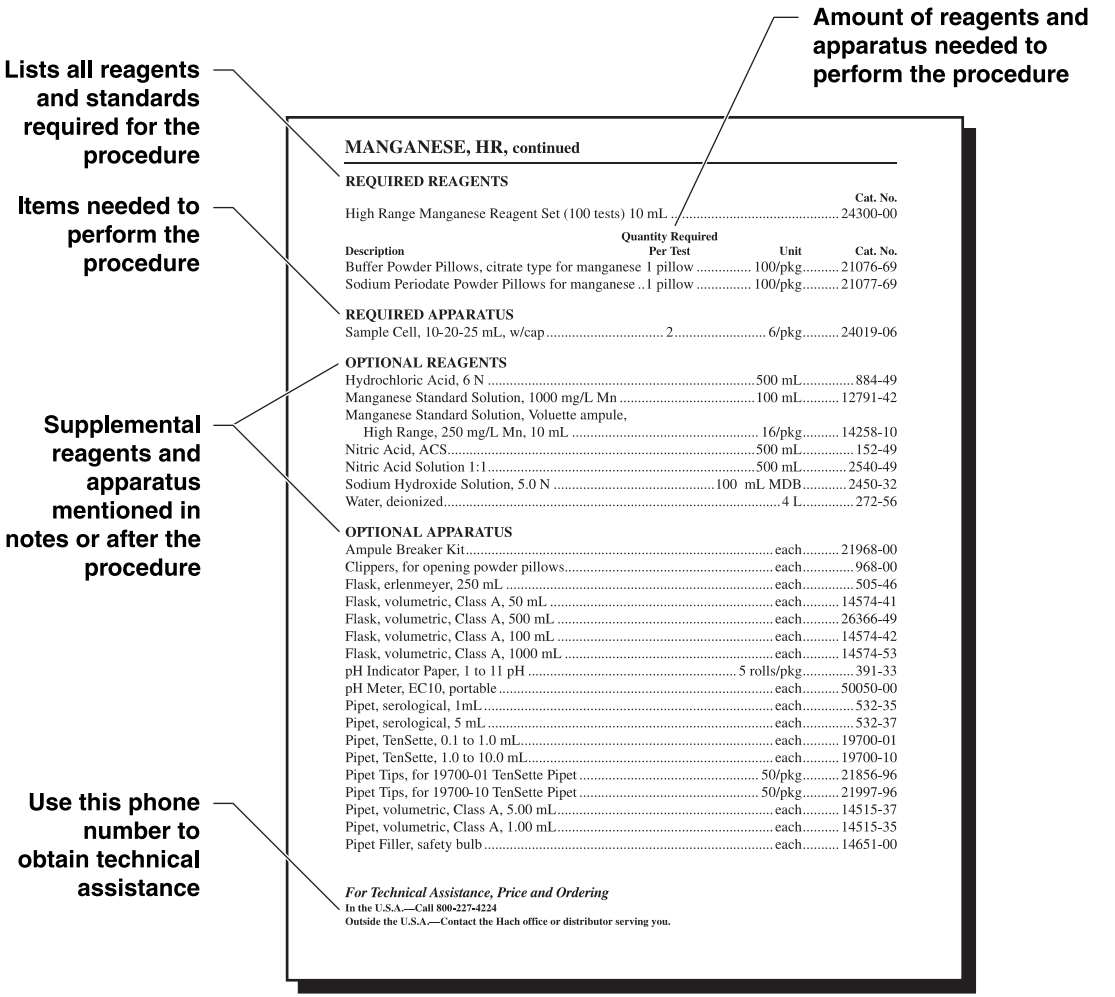
b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three 25-mL water samples. Swirl to mix.

c) Transfer only 10 mL of each solution to the 10-mL sample cells.

d) Analyze each standard addition sample as described in the procedure. The manganese concentration should increase 1.0 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.


Expected repeatability and estimated detection limit of the procedure
Levels of common sample substances or conditions that will cause inaccurate results
Concise explanation of method
MANGANESE, HR, continued
Standard Solution Method
Prepare a 5.0 mg/L manganese standard solution by pipetting (use a TenSette or CLass A volumetric pipet) 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Or, prepare this standard by diluting 1.00 mL of a High Range Manganese Standard Voluette Ampule, 250 mg/L, to 50 mL. Prepare these solutions daily. Use these solutions as the sample in the procedure.
Method Performance
Precision
In a single laboratory, using a standard solution of 10.00 mg/L Mn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.18 mg/L Mn.
Estimated Detection Limit
The estimated detection limit for program 41 is 0.12 mg/L Mn. For more information on the estimated detection limit, see Section 1.
Interferences
The following may interfere when present in concentrations exceeding those listed below:
Calcium 700 mg/L
Chloride 70,000 mg/L
Iron 5 mg/L
Magnesium 100,000 mg/L
Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see pH Interferences in Section 1.
Summary of Method
Manganese in the sample is oxidized to the purple permanganate state by sodium periodate, after buffering the sample with citrate. The purple color is directly proportional to the manganese concentration.



MANGANESE, HR, continued

REQUIRED REAGENTS

			Cat. No.
High Range Manganese Reagent Set (100 tests) 10 mL			24300-00

Description	Quantity Required Per Test	Unit	Cat. No.
Buffer Powder Pillows, citrate type for manganese	1 pillow	100/pkg	21076-69
Sodium Periodate Powder Pillows for manganese	1 pillow	100/pkg	21077-69

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Hydrochloric Acid, 6 N	500 mL	884-49
Manganese Standard Solution, 1000 mg/L Mn	100 mL	12791-42
Manganese Standard Solution, Voluette ampule, High Range, 250 mg/L Mn, 10 mL	16/pkg	14258-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution 1:1	500 mL	2540-49
Sodium Hydroxide Solution, 5.0 N	100 mL MDB	2450-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Clippers, for opening powder pillows	each	968-00
Flask, erlenmeyer, 250 mL	each	505-46
Flask, volumetric, Class A, 50 mL	each	14574-41
Flask, volumetric, Class A, 500 mL	each	26366-49
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 1000 mL	each	14574-53
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, serological, 1mL	each	532-35
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-10 TenSette Pipet	50/pkg	21997-96
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00

For Technical Assistance, Price and Ordering
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

SECTION 1 CHEMICAL ANALYSIS INFORMATION

Abbreviations

The following abbreviations are used throughout the text of the procedure section:

Abbrev-iation	Definition	Abbrev-iation	Definition
°C	degree(s) Celsius (Centigrade)	MDL	Method detection limit
°F	degree(s) Fahrenheit	MDB	marked dropping bottle
ACS	American Chemical Society reagent grade purity	mg/L	milligrams per liter (ppm)
APHA Standard Methods	*Standard Methods for the Examination of Water and Wastewater.*[1]	µg/L	micrograms per liter (ppb)
AV	AccuVac	mL	(milliliter)-approximately the same as a cubic centimeter (cc) or 1/1000 of a liter. Also known as a “cc”.
conc	concentrated	MR	medium range
CFR	Code of Federal Regulations	NIPDWR	National Interim Primary Drinking Water Regulations
DB	dropping bottle	NPDES	National Pollutant Discharge Elimination System
EDL	Estimated detection limit	PCB	Poly chlorinated biphenyl
FAU	Formazin Attenuation Units. Turbidity unit of measure based on a Formazin stock suspension.	SCDB	self-contained dropping bottle
g	grams	TNT	Test ‘N Tube™
gr/gal	grains per gallon (1 gr/gal = 17.12 mg/L)	TPH	Total petroleum hydrocarbons
HR	high range	TPTZ	(2,4,6-Tri-(2-Pyridyl)-1,3,5-Triazine)
L	Liter. Volume equal to one cubic decimeter (dm^3)	ULR	Ultra low range
LR	low range	USEPA	United States Environmental Protection Agency

[1] Published jointly by the American Public Health Association (APHA), the American Water Works Association (AWWA), and the Water Environment Federation (WEF). Order from Hach requesting Cat. No. 22708-00 or from the Publication Office of the American Public Health Association. This book is the standard reference work for water analysis. Many procedures contained in this manual are based on *Standard Methods.*

Converting Chemical Species

Species conversion factors for many commonly used substances are pre-programmed into the instrument (see *Table 1*). Conversions are method specific and are viewable after taking the reading by pressing **CONC**.

Table 1 Conversion Factors

To Convert From...	To...	Multiply By...
mg/L Al	mg/L Al_2O_3	1.8895
mg/L Ca-$CaCO_3$	mg/L Ca	0.4004
mg/L $CaCO_3$	mg/L Ca	0.4004
mg/L $CaCO_3$	mg/L Mg	0.2428
µg/L Carbohydrazide	µg/L Hydroquinone	1.92
µg/L Carbohydrazide	µg/L ISA	2.69
µg/L Carbohydrazide	µg/L MEKO	3.15
mg/L Cr^{6+}	mg/L CrO_4^{2-}	2.231
mg/L Cr^{6+}	mg/L Na_2CrO_4	3.115
mg/L Mg-$CaCO_3$	mg/L Mg	0.2428
mg/L Mn	mg/L $KMnO_4$	2.876
mg/L Mn	mg/L MnO_4^-	2.165
mg/L Mo^{6+}	mg/L MoO_4^{2-}	1.667
mg/L Mo^{6+}	mg/L Na_2MoO_4	2.146
mg/L N	mg/L NH_3	1.216
mg/L N	mg/L NO_3^-	4.427
mg/L Na_2CrO_4	mg/L Cr^{6+}	0.321
mg/L Na_2CrO_4	mg/L CrO_4^{2-}	0.72
mg/L NH_2Cl-N	mg/L Cl_2	5.0623
mg/L NH_2Cl-N	mg/L NH_2Cl	3.6750
mg/L NH_3-N	mg/L NH_3	1.216
mg/L NH_3-N	mg/L NH_4^+	1.288
mg/L NO_2^-	mg/L $NaNO_2$	1.5
mg/L NO_2^-	mg/L NO_2^--N	0.3045
mg/L NO_2^--N	mg/L $NaNO_2$	4.926
µg/L NO_2^--N	µg/L $NaNO_2$	4.926
mg/L NO_2^--N	mg/L NO_2^-	3.284
µg/L NO_2^--N	µg/L NO_2^-	3.284
mg/L NO_3^--N	mg/L NO_3^-	4.427
mg/L PO_4^{3-}	mg/L P	0.3261
µg/L PO_4^{3-}	µg/L P	0.3261
mg/L PO_4^{3-}	mg/L P_2O_5	0.7473
µg/L PO_4^{3-}	µg/L P_2O_5	0.7473
mg/L SiO_2	mg/L Si	0.4674
µg/L SiO_2	µg/L Si	0.4674

Hardness Conversion

Table 2 lists the factors for converting one unit of measure for hardness to another unit of measure. For example, to convert mg/L $CaCO_3$ to German parts/100,000 CaO, multiply the value in mg/L x 0.056.

Table 2 Hardness Conversion Factors

Units of Measure	mg/L $CaCO_3$	British gr/gal (Imperial) $CaCO_3$	American gr/gal (US) $CaCO_3$	French parts/ 100,000 $CaCO_3$	German Parts/ 100,000 CaO	meq/L[1]	g/L CaO	lbs./cu ft $CaCO_3$
mg/L $CaCO_3$	1.0	0.07	0.058	0.1	0.056	0.02	$5.6x10^{-4}$	$6.23x10^{-5}$
English gr/gal $CaCO_3$	14.3	1.0	0.83	1.43	0.83	0.286	$8.0x10^{-3}$	$8.9x10^{-4}$
US gr/gal $CaCO_3$	17.1	1.2	1.0	1.72	0.96	0.343	$9.66x10^{-3}$	$1.07x10^{-3}$
Fr. p/ 100,000 $CaCO_3$	10.0	0.7	0.58	1.0	0.56	0.2	$5.6x10^{-3}$	$6.23x10^{-4}$
Ger. p/ 100,000 CaO	17.9	1.25	1.04	1.79	1.0	0.358	$1x10^{-2}$	$1.12x10^{-3}$
meq/L	50.0	3.5	2.9	5.0	2.8	1.0	$2.8x10^{-2}$	$3.11x10^{-2}$
g/L CaO	1790.0	125.0	104.2	179.0	100.0	35.8	1.0	0.112
lbs./cu ft $CaCO_3$	16,100.0	1,123.0	935.0	1,610.0	900.0	321.0	9.0	1.0

[1] 'epm/L, or 'mval/L'
Note: 1 meq/L = 1N/1000

Dissolved Oxygen

Table 3 lists the mg/L dissolved oxygen in water at saturation for various temperatures and atmospheric pressures. The table was formulated in a laboratory using pure water. The values given are only approximations for estimating the oxygen content of a particular body of surface water.

Table 3 Dissolved Oxygen Saturation In Water

		Pressure in Millimeters and Inches Hg							
		mm							
		775	760	750	725	700	675	650	625
Temp		inches							
°F	°C	30.51	29.92	29.53	28.45	27.56	26.57	25.59	24.61
32.0	0	14.9	14.6	14.4	13.9	13.5	12.9	12.5	12.0
33.8	1	14.5	14.2	14.1	13.6	13.1	12.6	12.2	11.7
35.6	2	14.1	13.9	13.7	13.2	12.9	12.3	11.8	11.4
37.4	3	13.8	13.5	13.3	12.9	12.4	12.0	11.5	11.1
39.2	4	13.4	13.2	13.0	12.5	12.1	11.7	11.2	10.8
41.0	5	13.1	12.8	12.6	12.2	11.8	11.4	10.9	10.5
42.8	6	12.7	12.5	12.3	11.9	11.5	11.1	10.7	10.3
44.6	7	12.4	12.2	12.0	11.6	11.2	10.8	10.4	10.0
46.4	8	12.1	11.9	11.7	11.3	10.9	10.5	10.1	9.8
48.2	9	11.8	11.6	11.5	11.1	10.7	10.3	9.9	9.5
50.0	10	11.6	11.3	11.2	10.8	10.4	10.1	9.7	9.3
51.8	11	11.3	11.1	10.9	10.6	10.2	9.8	9.5	9.1
53.6	12	11.1	10.8	10.7	10.3	10.0	9.6	9.2	8.9
55.4	13	10.8	10.6	10.5	10.1	9.8	9.4	9.1	8.7
57.2	14	10.6	10.4	10.2	9.9	9.5	9.2	8.9	8.5
59.0	15	10.4	10.2	10.0	9.7	9.3	9.0	8.7	8.3
60.8	16	10.1	9.9	9.8	9.5	9.1	8.8	8.5	8.1
62.6	17	9.9	9.7	9.6	9.3	9.0	8.6	8.3	8.0
64.4	18	9.7	9.5	9.4	9.1	8.8	8.4	8.1	7.8
66.2	19	9.5	9.3	9.2	8.9	8.6	8.3	8.0	7.6
68.0	20	9.3	9.2	9.1	8.7	8.4	8.1	7.8	7.5
69.8	21	9.2	9.0	8.9	8.6	8.3	8.0	7.7	7.4
71.6	22	9.0	8.8	8.7	8.4	8.1	7.8	7.5	7.2
73.4	23	8.8	8.7	8.5	8.2	8.0	7.7	7.4	7.1

Table 3 Dissolved Oxygen Saturation In Water (continued)

		Pressure in Millimeters and Inches Hg							
		mm							
		775	760	750	725	700	675	650	625
Temp		inches							
°F	°C	30.51	29.92	29.53	28.45	27.56	26.57	25.59	24.61
75.2	24	8.7	8.5	8.4	8.1	7.8	7.5	7.2	7.0
77.0	25	8.5	8.4	8.3	8.0	7.7	7.4	7.1	6.8
78.8	26	8.4	8.2	8.1	7.8	7.6	7.3	7.0	6.7
80.6	27	8.2	8.1	8.0	7.7	7.4	7.1	6.9	6.6
82.4	28	8.1	7.9	7.8	7.6	7.3	7.0	6.7	6.5
84.2	29	7.9	7.8	7.7	7.4	7.2	6.9	6.6	6.4
86.0	30	7.8	7.7	7.6	7.3	7.0	6.8	6.5	6.2
87.8	31	7.7	7.5	7.4	7.2	6.9	6.7	6.4	6.1
89.6	32	7.6	7.4	7.3	7.0	6.8	6.6	6.3	6.0
91.4	33	7.4	7.3	7.2	6.9	6.7	6.4	6.2	5.9
93.2	34	7.3	7.2	7.1	6.8	6.6	6.3	6.1	5.8
95.0	35	7.2	7.1	7.0	6.7	6.5	6.2	6.0	5.7
96.8	36	7.1	7.0	6.9	6.6	6.4	6.1	5.9	5.6
98.6	37	7.0	6.8	6.7	6.5	6.3	6.0	5.8	5.6
100.4	38	6.9	6.7	6.6	6.4	6.2	5.9	5.7	5.5
102.2	39	6.8	6.6	6.5	6.3	6.1	5.8	5.6	5.4
104.0	40	6.7	6.5	6.4	6.2	6.0	5.7	5.5	5.3
105.8	41	6.6	6.4	6.3	6.1	5.9	5.6	5.4	5.2
107.6	42	6.5	6.3	6.2	6.0	5.8	5.6	5.3	5.1
109.4	43	6.4	6.2	6.1	5.9	5.7	5.5	5.2	5.0
111.2	44	6.3	6.1	6.0	5.8	5.6	5.4	5.2	4.9
113.0	45	6.2	6.0	5.9	5.7	5.5	5.3	5.1	4.8
114.8	46	6.1	5.9	5.9	5.6	5.4	5.2	5.4	4.8
116.6	47	6.0	5.9	5.8	5.6	5.3	5.1	4.8	4.7
118.4	48	5.9	5.8	5.7	5.5	5.3	5.0	4.8	4.6
120.2	49	5.8	5.7	5.6	5.4	5.2	5.0	4.7	4.5
122.0	50	5.7	5.6	5.5	5.3	5.1	4.9	4.7	4.4

Sample Collection, Preservation and Storage

Correct sampling and storage are critical for accurate testing. For greatest accuracy, thoroughly clean sampling devices and containers to prevent carryover from previous samples. Preserve the sample properly; each procedure has information about sample preservation.

- The least expensive containers are polypropylene or polyethylene.
- The best and most expensive containers are quartz or PTFE (polytetrafluoroethylene, Teflon).
- Avoid soft glass containers for metals in the microgram-per-liter range.
- Store samples for silver determination in light-absorbing containers, such as amber bottles.

Avoid contaminating the sample with metals from containers, deionized water or membrane filters. Thoroughly clean sample containers as described under Acid Washing Bottles.

Preservation slows the chemical and biological changes that continue after collection. These processes may change the amount of a chemical species available for analysis. Normally, analyze the samples as soon as possible after collection, especially when the analyte concentration is expected to be low. This also reduces the chance for error and minimizes labor.

Preservation methods include pH control, chemical addition, refrigeration and freezing. *Table 4* gives the recommended preservation for various substances. It also includes suggested types of containers and the maximum recommended holding times for properly preserved samples.

Preserve aluminum, cadmium, chromium, cobalt, copper, iron, lead, nickel, potassium, silver and zinc samples for at least 24 hours by adding one Nitric Acid Solution Pillow 1:1 (Cat. No. 2540-98) per liter of sample. Check the pH with pH indicator paper or a pH meter to assure the pH is 2 or less. Add additional pillows if necessary. Adjust the sample pH prior to analysis by adding an equal number of Sodium Carbonate Anhydrous Powder Pillows (Cat. No. 179-98). Or raise the pH to 4.5 with Sodium Hydroxide Standard Solution, 1 N or 5 N. Correct for the added volume of the preservatives; see *Correcting For Volume Additions.*

Table 4 Required Containers, Preservation Techniques and Holding Times[1]

Parameter No./Name	Container[2]	Preservation[3,4]	Maximum Holding Time[5]
Table 1A - Bacterial Tests:			
1-4. Coliform, fecal and total	P,G	Cool, 4°C, 0.008%, $Na_2S_2O_3$[6]	6 hours
5. Fecal streptococci	P,G	Cool, 4°C, 0.008%, $Na_2S_2O_3$	6 hours
Table 1B - Inorganic Tests:			
1. Acidity	P, G	Cool, 4°C	14 days
2. Alkalinity	P, G	Cool, 4°C	14 days
4. Ammonia	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days
9. Biochemical oxygen demand (BOD)	P, G	Cool, 4°C	48 hours
11. Bromide	P, G	None required	28 days
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours
15. Chemical oxygen demand	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days
16. Chloride	P, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze immediately
21. Color	P, G	Cool, 4°C	48 hours
23-24. Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH>12, 0.6 g ascorbic acid[6]	14 days[7]
25. Fluoride	P	None required	28 days
27. Hardness	P, G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately
31, 43. Kjeldahl and organic nitrogen	P, G	Cool 4°C, H_2SO_4 to pH<2	28 days
Metals:[8]			
18. Chromium VI	P, G	Cool, 4°C	24 hours
35. Mercury	P, G	HNO_3 to pH<2	6 months
3, 5-8, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75.[9] Metals, except boron, chromium VI and mercury	P, G	do	6 months
38. Nitrate	P, G	Cool, 4°C	48 hours
39. Nitrate-nitrite	P, G	Cool 4°C, H_2SO_4 to pH<2	28 days
40. Nitrite	P, G	Cool, 4°C	48 hours
41. Oil and grease	G	Cool, 4°C, HCl or H_2SO_4 to pH<2	28 days
42. Organic Carbon	P, G	Cool, 4°C, HCl or H_2SO4 or H_3PO_4 to pH<2	28 days
44. Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
46. Oxygen, dissolved probe	G Bottle and top	None required	Analyze immediately
47. Winkler	G Bottle and top	Fix on site and store in dark	8 hours

Table 4 Required Containers, Preservation Techniques and Holding Times[1] (continued)

Parameter No./Name	Container[2]	Preservation[3,4]	Maximum Holding Time[5]
48. Phenols	G only	Cool 4°C, H_2SO_4 to pH<2	28 days
49. Phosphorus, elemental	G	Cool, 4°C	48 hours
50. Phosphorus, total	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days
53. Residue, total	P, G	Cool, 4°C	7 days
54. Residue, filterable	P, G	Cool, 4°C	7 days
55. Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
56. Residue, Settleable	P, G	Cool, 4°C	48 hours
57. Residue, volatile	P, G	Cool, 4°C	7 days
61. Silica	P, PFTE or quartz	Cool, 4°C	28 days
64. Specific conductance	P, G	Cool, 4°C	28 days
65. Sulfate	P, G	Cool, 4°C	28 days
66. Sulfide	P, G	Cool 4°C, add zinc acetate plus sodium hydroxide to pH>9	7 days
67. Sulfite	P, G	none required	Analyze immediately
68. Surfactants	P, G	Cool, 4°C	48 hours
69. Temperature	P, G	None required	Analyze immediately
73. Turbidity	P, G	Cool, 4°C	48 hours

1 This table was taken from Table II published in the Federal Register, July 1, 1995, 40 CFR, Part 136.3, pages 643-645. Organic tests are not included.

2 Polyethylene (P) or glass (G).

3 Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

4 When any sample is to be shipped by common carrier or sent through United States Mails, it must comply with the Department of Transportation Hazardous Material Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

5 Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permitee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administer under §136.3(e). Some samples may not be stable for the maximum time period given in the table. A permitee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See §136.3(e) for details. The term “analyze immediately” usually means within 15 minutes or less after sample collection.

6 Should only be used in the presence of residual chlorine.

7 Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

8 Samples should be filtered immediately on-site before adding preservative for dissolved metals.

9 Numbers refer to parameter numbers in 40 CFR Part 136.3, Table 1B.

Collecting Water Samples

Obtain the best sample by careful collection. In general, collect samples near the center of the vessel or duct and below the surface. Use only clean containers (bottles, beakers). Rinse the container several times first with the water to be sampled.

Take samples as close as possible to the source of the supply. This lessens the influences of the distribution system on the sample. Let the water run long enough to flush the system. Fill sample containers slowly with a gentle stream to avoid turbulence and air bubbles. Collect water samples from wells after the pump has run long enough to deliver water representative of the ground water feeding the well.

It is hard to obtain a truly representative sample when collecting surface water samples. Obtain best results by testing several samples. Use samples taken at different times from several locations and depths. The results can be used to establish patterns for that particular body of water.

Generally, as little time as possible should elapse between collecting the sample and analyzing it.

Depending on the test, special precautions in handling the sample may be necessary. This prevents natural interferences such as organic growth or loss or gain of dissolved gases. Each procedure describes sample preservatives and storage techniques for samples that are held for testing.

Acid Washing Bottles

If a procedure suggests acid-washing, use the following instructions:

a) Clean the glassware or plasticware with laboratory detergent (phosphate-free detergent is recommended).

b) Rinse well with tap water.

c) Rinse with a 1:1 Hydrochloric Acid Solution or 1:1 Nitric Acid Solution.

d) Rinse well with deionized water at least four times. Up to 12-15 rinses may be necessary if chromium is being determined.

e) Air dry.

Use chromic acid or chromium-free substitutes to remove organic deposits from glass containers. Rinse containers thoroughly with water to remove traces of chromium.

Wash glassware for phosphate determinations with phosphate-free detergents and acid-wash with 1:1 HCl. Thoroughly rinse the glassware with deionized water. For ammonia and Kjeldahl nitrogen, rinse with ammonia-free water.

Correcting for Volume Additions

If you use a large volume of preservative, correct for the volume of preservative added. This accounts for dilution due to the acid added to preserve the sample and the base used to adjust the pH to the range of the procedure. This correction is made as follows:

1. Determine the volume of initial sample, the volume of acid and base added, and the total or final volume of the sample.

2. Divide the total volume by the initial volume of sample.

3. Multiply the test result by this factor.

Example:

A one-liter sample was preserved with 2 mL of nitric acid. It was neutralized with 5 mL of 5 N sodium hydroxide. The result of the analysis procedure was 10.00 mg/L. What is the volume correction factor and correct result?

1. Total Volume = 1000 mL + 2 mL + 5 mL = 1007 mL

2. $\frac{1007}{1000}$ = 1.007 = volume correction factor

3. 10.0 mg/L × 1.007 = 10.07 mg/L = correct result

Hach 1:1 Nitric Acid Pillows contain 2.5 mL of acid; correct for this volume. The addition of a Sodium Carbonate Power Pillow (neutralizes the 1:1 Nitric Acid Solution Pillow) does not need to be corrected for.

Boiling Aids

Boiling is necessary in some procedures. Using a boiling aid such as boiling chips (Cat. No. 14835-31) helps reduce bumping. Bumping is caused by the sudden, almost explosive conversion of water to steam as it is heated. Avoid bumping; it may cause injury or sample loss.

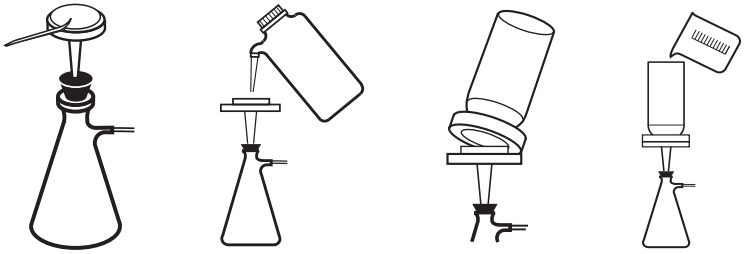
Make sure the boiling aids will not contaminate the sample. Do not use boiling aids (except glass beads) more than once. Loosely covering the sample during boiling will prevent splashing, reduce the chances of contamination and minimize sample loss.

Sample Filtration

Filtering separates particles from the aqueous sample. Filtration uses a medium, usually filter paper, to retain particles but pass solution. This is especially helpful when sample turbidity interferes with analysis. Two general methods of filtration are gravity and vacuum. Gravity filtration uses gravity to pull the sample though the filter paper. Vacuum filtration uses suction and gravity to move the sample through the filter. An aspirator or vacuum pump creates the suction. Vacuum filtration is faster than gravity filtration. Vacuum filter (see *Figure 1*) as follows:

1. Using tweezers, place a filter paper into the filter holder.
2. Place the filter holder assembly in the filtering flask. Wet the filter with deionized water to ensure adhesion to the holder. Empty the flask before filtering the sample.
3. Position the funnel housing on the filter holder assembly.
4. While applying a vacuum to the filtering flask, transfer the sample to the filtering apparatus.
5. Slowly release the vacuum from the filtering flask and transfer the solution from the filter flask to another container.

Figure 1 Vacuum Filtration



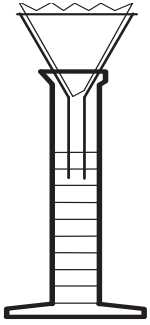
REQUIRED APPARATUS FOR VACUUM FILTRATION

Description	Unit	Cat. No.
Filter Discs, glass 47 mm, 1.5 µm	100/pkg	2530-00
Filter Holder, membrane	each	13529-00
Flask, filter, 500 mL	each	546-49
Pump, vacuum, hand operated	each	14283-00
OR		
Pump, vacuum, portable, 115 V	each	14697-00
Pump, vacuum, portable, 230 V	each	14697-02

Several procedures in this manual use gravity filtration. The only labware required is filter paper, a conical funnel and a receiving vessel. This labware is included under Optional Apparatus at the end of a procedure. Gravity filtration is better for retaining fine particles. For faster filtering, add solution until the filter paper cone is three-fourths filled. Never fill the cone completely. Gravity filter (see *Figure 2*) as follows:

1. Place a filter paper into the funnel.
2. Wet the filter with deionized water to ensure adhesion to the funnel. Allow all the deionized water to drain.
3. Place the funnel into an erlenmeyer flask or graduated cylinder.
4. Pour the sample into the funnel.

Figure 2 Gravity Filtration



REQUIRED APPARATUS FOR GRAVITY FILTRATION

Description	Unit	Cat No.
Cylinder, graduated, 100 mL	each	508-42
Funnel, poly, 65 mm	each	1083-67
Filter Paper, 12.5 cm	each	1894-57
Flask, erlenmeyer, 125 mL	each	505-43

Testing for metals requires acid and heat to pretreat the sample. Since these conditions destroy filter paper, vacuum filtration with glass fiber filter discs is recommended. Also, glass filter discs, unlike paper, do not retain colored species.

Temperature Considerations

For best results, perform most tests in this manual with sample temperatures between 20 °C (68 °F) and 25 °C (77 °F). If a test requires closer temperature control, notes in the procedure will indicate this.

Sample Dilution Techniques

Ten and 25 mL are the volumes used for most colorimetric tests. However, in some tests, the color developed in the sample may be too intense to be measured. Unexpected colors may develop in other tests. In both cases, dilute the sample to determine if interfering substances are present.

To dilute the sample easily, pipet the chosen sample portion into a clean graduated cylinder (or volumetric flask for more accurate work). Fill the cylinder (or flask) to the desired volume with deionized water. Mix well. Use the diluted sample when running the test.

To help with dilutions, *Table 5* shows the amount of sample used, the amount of deionized water used to bring the volume up to 25 mL and the multiplication factor.

The concentration of the sample is equal to the diluted sample reading multiplied by the multiplication factor.

More accurate dilutions can be done with a pipet and a 100-mL volumetric flask (see *Table 6* for more information). Pipet the sample and dilute to volume with deionized water. Swirl to mix.

Table 5 Sample Dilution Volumes

Sample Volume (mL)	mL Deionized Water Used to Bring the Volume to 25 mL	Multiplication Factor
25.0	0.0	1
12.5	12.5	2
10.0[1]	15.0	2.5
5.0[1]	20.0	5
2.5[1]	22.5	10
1.0[1]	24.0	25
0.250[1]	24.75	100

[1] For sample sizes of 10 mL or less, use a pipet to measure the sample into the graduated cylinder or volumetric flask.

Table 6 Multiplication Factors for Diluting to 100 mL

Sample Volume (mL)	Multiplication Factor
1	100
2	50
5	20
10	10
25	4
50	2

Sample Dilution and Interfering Substances

Sample dilution may influence the level at which a substance may interfere. The effect of the interferences decreases as the dilution increases. In other words, higher levels of an interfering substance can be present in the original sample if it is diluted before analysis.

An Example:

Copper does not interfere at or below 100 mg/L for a 25.00 mL sample in a procedure. If the sample volume is diluted with an equal volume of water, what is the level at which copper will not interfere?

$$\frac{\text{Total volume}}{\text{Sample volume}} = \text{Dilution factor}$$

$$\frac{25}{12.5} = 2$$

$$\text{Interference Level} \times \text{Dilution Factor} = \text{Interference level in sample}$$

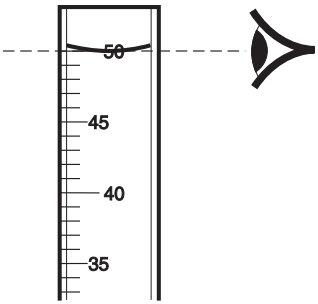
$$100 \times 2 = 200$$

The level at which copper will not interfere in the undiluted sample is at or below 200 mg/L.

Using Pipets and Graduated Cylinders

When small sample quantities are used, the accuracy of measurements is important. *Figure 3* illustrates the proper way of reading the sample level or the meniscus formed when the liquid wets the cylinder or pipet walls.

Figure 3 Reading the Meniscus



Rinse the pipet or cylinder two or three times with the sample to be tested before filling. Use a pipet filler or pipet bulb to draw the sample into the pipet. Never pipet chemical reagent solutions or samples by mouth. When filling a pipet, keep the tip of the pipet below the surface of the sample as the sample is drawn into the pipet.

Serological pipets have marks that indicate the volume of liquid delivered by the pipet. The marks may extend to the tip of the pipet or may be only on the straight portion of the tube. If the marks are only on the straight part of the tube, fill serological pipets to the zero mark and discharge the sample by draining the sample until the meniscus is level with the desired mark. If the serological pipet has marks extended to the tip of the pipet, fill the pipet to the desired volume and drain all the sample from the pipet. Then blow the sample out of the pipet tip for accurate measurements.

Volumetric (transfer) pipets have a bulb in the middle and a single ring above the bulb to indicate the volume of liquid when it is filled to the mark. To discharge a volumetric pipet, hold the pipet vertical until only a small amount of liquid remains (about ¾ inch), then hold the pipet at a slight angle against the container wall to drain. Do not attempt to discharge the solution remaining in the tip of the pipet after draining. Volumetric pipets are designed to retain a small amount of sample in the pipet tip.

If sample drops stay on the walls of the pipet, the pipet is dirty and is not delivering the correct amount of sample. Wash the pipet thoroughly with a laboratory detergent or cleaning solution and rinse several times with deionized water.

Using the TenSette Pipet

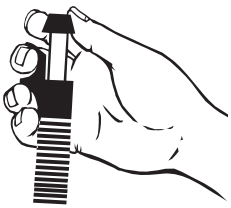
For best results use a new tip each time you pipet. After several uses, the pipet tip may retain some liquid, causing inaccurate delivery. Each pipet is supplied with 50 tips; order Hach replacement tips for best results.

Always use careful, even hand movements for best reproducibility. If the pipet does not operate smoothly, disassemble and coat the piston and retainer with high-quality stopcock grease. Also coat the metering turret lightly with grease. Refer to the TenSette Pipet manual.

For best pipetting accuracy, the solution and the room temperature should be between 20-25 °C.

Never lay the pipet down with the liquid in the tip. Solution could leak into the pipet and cause corrosion.

Operating the TenSette Pipet



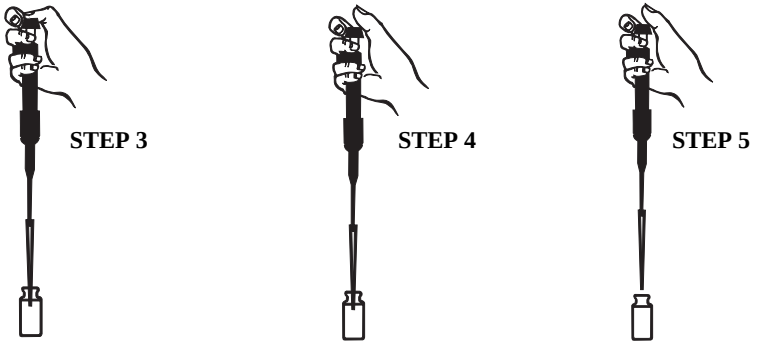
1. Attach a clean tip by holding the pipet body in one hand and gently pressing the large end of the pipet tip onto the tapered end of the pipet. Be sure a good seal is obtained.

2. Turn the turret cap to align the desired volume with the mark on the pipet body.

3. Using a smooth motion, press down on the turret cap until it reaches the stop. Immerse the tip about 5 mm (¼ inch) below the solution surface to avoid drawing air into the pipet. Do not insert the tip any deeper or the delivery volume may be affected.

4. While maintaining a constant pressure, allow the turret to return slowly to the extended position. A rapid return may affect the delivery volume.

5. With the turret up, take the tip out of the solution and move it to the receiving vessel. Do not press on the turret cap while moving the pipet.




STEP 6

6. Use the thumb and forefinger to twist the turret cap to the next higher volume position to ensure quantitative transfer of the sample. The “F” position provides full blowout.


STEP 7

7. With the tip in contact with the side of the receiving vessel, slowly and smoothly press down on the turret cap until it reaches the stop and the solution is completely discharged.

Mixing Water Samples

The following two methods may be helpful in tests that require mixing sample with chemicals (usually indicated by "invert to mix" instructions).

1. When mixing sample in a round sample cell or mixing cylinder, invert the cell or cylinder; see *Figure 4*. Hold the cell in a vertical position with the cap on top. Invert the cell so the cap is on the bottom. Return the cell to the original position. Do the same with the mixing cylinder.

2. Swirling is recommended when mixing samples in a graduated cylinder or a titration flask. Grip the cylinder (or flask) firmly with the tips of three fingers; see *Figure 5*. Hold the cylinder at a 45-degree angle and twist the wrist. This should move the cylinder in an approximately 12-inch circle, creating enough rotation to complete the mixing in a few turns.

These mixing procedures are the most gentle. Both methods are simple but take a bit of practice to obtain the best results.

Figure 4 Inverting a Sample Cell

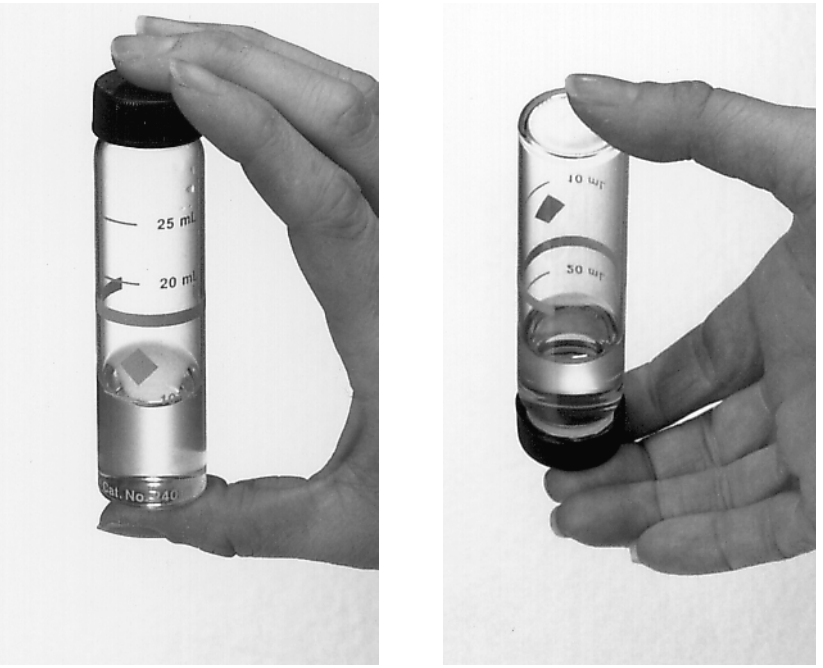
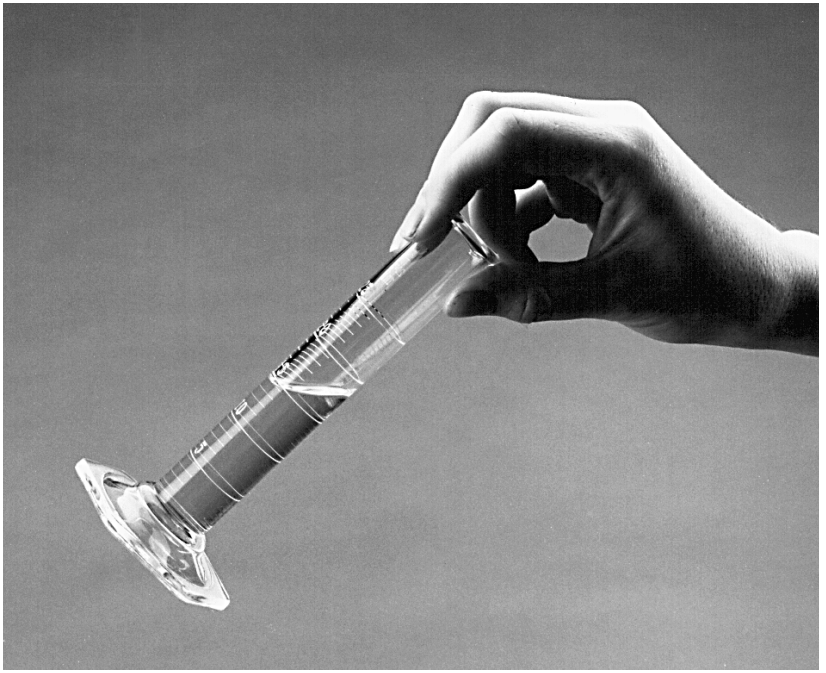


Figure 5 Swirling a Graduated Cylinder



Using Sample Cells

Orientation of Sample Cells

Two round sample cells are shipped with the DR/820, DR/850 and DR/890. They are marked with 10-, 20- and 25-mL fill lines which may be used to measure the sample volume unless the procedure instructs you to use other glassware to measure the sample volume.

To minimize variability of measurements using a particular cell, always place the cell into the cell holder with the same orientation. The cells are placed in the instrument with the fill marks facing the user.

In addition to proper orientation, the sides of the cells should be free of smudges, fingerprints, etc. to ensure accurate readings. Wipe the sides of the cells with a moist cloth followed by a dry soft cloth to clean the surface before taking measurements.

Care of Hach Sample Cells

Store sample cells in their boxes when not in use to protect them from scratching and breaking. It is good laboratory practice to empty and clean sample cells after analyses are complete--avoid leaving colored solutions in the cells for extended periods of time. Finish the cleaning procedure with a few rinses of deionized water and allow to dry. Individual procedures often recommend specific cleaning methods.

Cleaning Sample Cells

Most laboratory detergents can be used at recommended concentrations. Neutral detergents such as Neutracon are safer if regular cleaning is required, as in the case of protein residues.

If using a detergent, you can speed cleaning by increasing the temperature or using an ultrasonic bath.

Rinsing is more efficient when using deionized water.

Using the COD/TNT Adapter

Use care when seating a vial into the COD/ TNT adapter (for COD vials and Test ‘N Tubes). Place the vial into the adapter and press straight down on the top of the vial until it seats solidly. Do not move the vial from side to side; this can cause errors.

Volume Measurement Accuracy

The sample cells supplied with the instrument have fill marks to indicate 10, 20 or 25 mL. The fill marks are intended to measure the volume to be analyzed. Do not use these fill marks to perform sample dilutions.

If a sample must be diluted, use a pipet, graduated mixing cylinder and/or a volumetric flask for accurate measurement. When diluting, accuracy is important because a slight mistake in measuring a small sample will cause

a substantial error in the result. For instance, a 0.1-mL mistake in the dilution of a 1.0-mL final volume produces a 10% error in the test result.

Volumes for standard additions can be measured using the 25-mL mark, but it is not recommended for the 10-mL mark due to a potentially excessive relative error. An error of 0.5 mL in 25 mL is only 2%, while 0.5 mL error in 10 mL is 5%.

For 10 mL standard additions, follow this procedure:

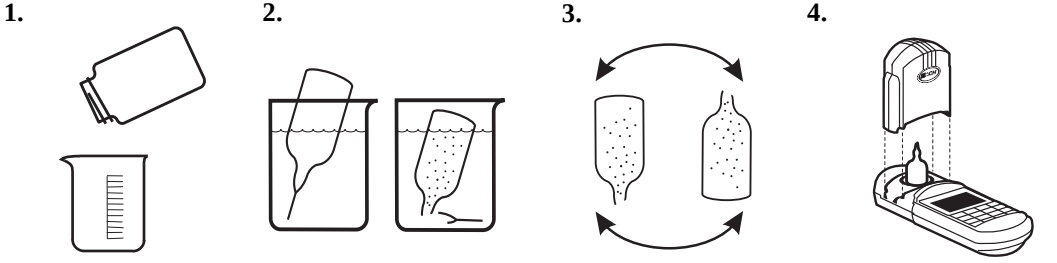
1. Transfer 10.0 mL of sample into a clean, dry sample cell (the unspiked sample).
2. Add the standard (spike) to a 25 mL portion of sample in a 25-mL mixing cylinder. Stopper and mix thoroughly.
3. Transfer 10 mL to another sample cell (use fill mark) for analysis.

Using AccuVac Ampuls

AccuVac ampuls contain pre-measured powder or liquid in optical-quality glass ampuls.

1. Collect the sample in a beaker or other open container.
2. Place the ampul tip well below the sample surface and break the tip off (see *Figure 6*) against the beaker wall. The break must be far enough below the surface to prevent air from being drawn in as the level of the sample lowers (the AccuVac Breaker may be used instead of breaking the ampul against the beaker side).
3. Invert the ampul several times to dissolve the reagent. Do not place your finger over the broken end; the liquid will stay in the ampul when inverted. Wipe the ampul with a towel to remove fingerprints, etc.
4. Insert the ampul into the instrument and read the results directly.

Figure 6 Using AccuVac Ampuls



Using Reagent Powder Pillows

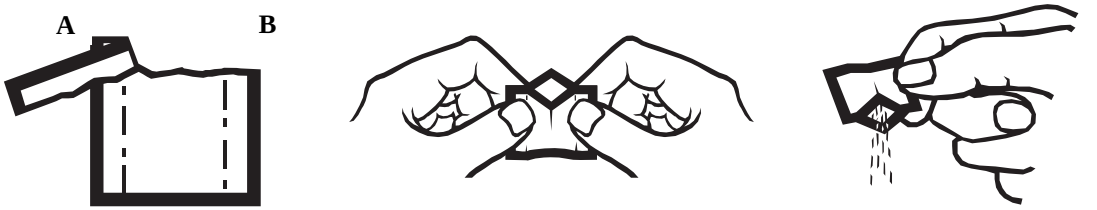
Hach uses dry powdered reagents when possible. This minimizes leakage and deterioration problems. Some powders are packaged in individual, pre-measured, polyethylene "powder pillows" or foil pillows called PermaChem® pillows. Each pillow contains enough reagent for one test. Open the poly powder pillows with nail clippers or scissors; see *Figure 7*.

Figure 7 Opening Powder Pillows

Using PermaChem Pillows

1. Tap the pillow on a hard surface to collect the powdered reagent in the bottom.
2. Tear (or cut) across the top of the pillow, from B to A, holding the pillow away from your face.
3. Using two hands, push both sides toward each other to form a spout.
4. Pour the pillow contents into the sample cell and continue the procedure according to the instructions. Tap the pillow to remove any powder from the corners.

1. Tear **2. Push** **3. Pour**



Reagent and Standard Stability

Hach always strives to make stable formulations and package them to provide maximum protection. Most chemicals and prepared reagents do not deteriorate after manufacture. However, the way they are stored and the packaging can affect how long the reagents are stable. Light, bacterial action, and absorption of moisture and gases from the atmosphere can affect shelf life. Some chemicals may react with the storage container or they may react with other chemicals.

Chemicals supplied with the colorimeter have an indefinite shelf life when stored under average room conditions, unless the packaging says something different. Product labels state any special storage conditions required. Otherwise, store reagents in a cool, dry, dark place for maximum life. It is always good practice to date chemicals when you receive them. Use older supplies first. If in doubt about the reagent shelf life, run a standard to check its effectiveness.

Interferences

Substances in the sample may interfere with a measurement. Hach mentions common interferences in the test procedures. The reagent formulations eliminate many interferences. You can remove others with sample pretreatments described in the procedure.

If you get an unusual answer, a color that you don't expect, or you notice an unusual odor or turbidity, the result may be wrong. Repeat the test on a sample diluted with deionized water; see *Sample Dilution Techniques*. Compare the result (corrected for the dilution) with the result of the original test. If these two are not close, the original result may be wrong and you should make an additional dilution to check the second test (first dilution). Repeat this process until you get the same corrected result twice in a row.

More information about interferences and methods to overcome them is contained in *Standard Additions* of this manual and the *General Introduction* section of APHA Standard Methods. Hach urges the analyst to obtain this book and refer to it when problems are encountered.

One of the greatest aids is knowing what is in the sample. You don't need to know exactly what is in each sample, but be aware of substances that are likely to interfere in the analysis method you use. When using a method, it may be helpful to determine if those interferences are present.

pH Interference

Many of the procedures in this manual only work within a certain pH range. Hach reagents contain buffers to adjust the pH of the typical sample to the correct pH range. However, the reagent buffer may not be strong enough for some samples. This occurs most often with highly buffered samples or samples with extreme sample pH.

The *Sampling and Storage* section of each procedure usually gives the proper pH range for the sample.

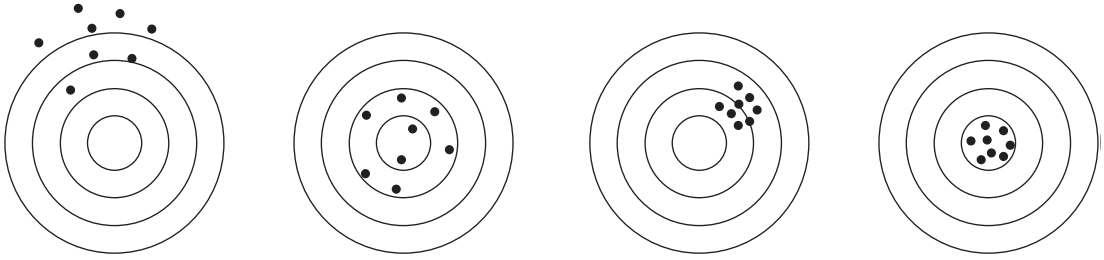
Adjust the sample to the proper pH range before testing. If this information is not given, follow these steps:

1. Measure the pH of your analyzed sample with a pH meter. For measuring Ag^+, K^+ or Cl^- , use pH paper.
2. Prepare a sample using deionized water. Add all reagents called for in the procedure. Timer sequences, etc., may be ignored. Mix well.
3. Measure the pH of the reagent blank with a pH meter.
4. Compare the pH values of your analyzed sample with the reagent blank.
5. If there is little difference in the values of your analyzed sample and the reagent blank, then pH interference is not the problem. Follow the *Accuracy Check* given in the procedure to help identify the problem.
6. If there is a large difference between the value of your analyzed sample and the reagent blank, adjust the sample pH to the value of the reagent blank. Adjust the sample pH to this same pH for all future samples from the same source before analysis. Use the appropriate acid, usually nitric acid, to lower the pH (do not use nitric acid for nitrate or nitrogen testing). Use the appropriate base, usually sodium hydroxide, to raise the pH. Adjust the final result for any dilution caused by adding acid or base; see *Correcting for Volume Additions*.
7. Analyze the sample as before.
8. Some purchased standards may be very acidic and will not work directly with Hach procedures. Adjust the pH of these standards as described above. Adjust the final concentration of the standard for the dilution. The Hach standard solutions suggested in the procedures are formulated so that no pH adjustment is necessary.

Accuracy and Precision

Accuracy is the nearness of a test result to the true value. Precision is how closely repeated measurements agree with each other. Although good precision suggests good accuracy, precise results can be inaccurate (see *Figure 8*). The following paragraphs describe how to improve accuracy and precision of analyses by using Standard Additions.

Figure 8 Precision and Accuracy Illustrated



Standard Additions

Standard Additions is a common technique for checking test results. Other names are “spiking” and “known additions.” The standard additions technique can test for interferences, bad reagents, faulty instruments, and incorrect procedures.

Perform Standard Additions by following the Standard Additions Method section in the procedure under *Accuracy Check*. Follow the detailed instructions given.

If you get about 100% recovery for each addition, everything is working right and your results are correct.

If you don’t get about 100% recovery for each addition, a problem exists. You can tell if you have an interference. Repeat the Standard Additions using deionized water as your sample. If you get about 100% recovery for each addition, you have an interference. If you didn’t get good recoveries with the deionized water, the following checklist may help to find the problem quickly:

1. Check to see that you are following the procedure exactly:

a) Are you using the proper reagents in the proper order? Are you using 10-mL reagents with a 10-mL sample or 25-mL reagents with a 25-mL sample?

b) Are you waiting the necessary time for color to develop?

c) Are you using the correct glassware?

d) Is the glassware clean?

e) Does the test need a specific sample temperature?

f) Is the sample's pH in the correct range?

Hach's written procedure should help you to answer these questions.

2. Check your reagents. Repeat the Standard Additions using new, fresh reagents. If your results are good, the original reagents were bad.

3. If nothing else is wrong, the standard is almost certainly bad. Repeat the Standard Additions with a new standard.

If the check list does not determine the problem, use the decision tree (*Figure 9*) and explanation of each branch, below, to identify the problem.

Branch A

Suppose a single standard addition to the sample did not give the correct concentration increase. A possible cause could be interferences. Other causes include defective reagents, incorrect technique, a defective instrument/apparatus or defective standard used for the standard addition.

If interferences are known or assumed to be absent, proceed to Branch B. If interferences are known to be present, proceed to Branch C.

Branch B

Perform multiple standard additions on a sample of deionized water as in the following example using iron as the analyte of interest:

1. Pour 25 mL of deionized water into a 25-mL sample cell.

2. Add 0.1 mL of a 50-mg/L iron standard solution to a second 25 mL sample of deionized water.

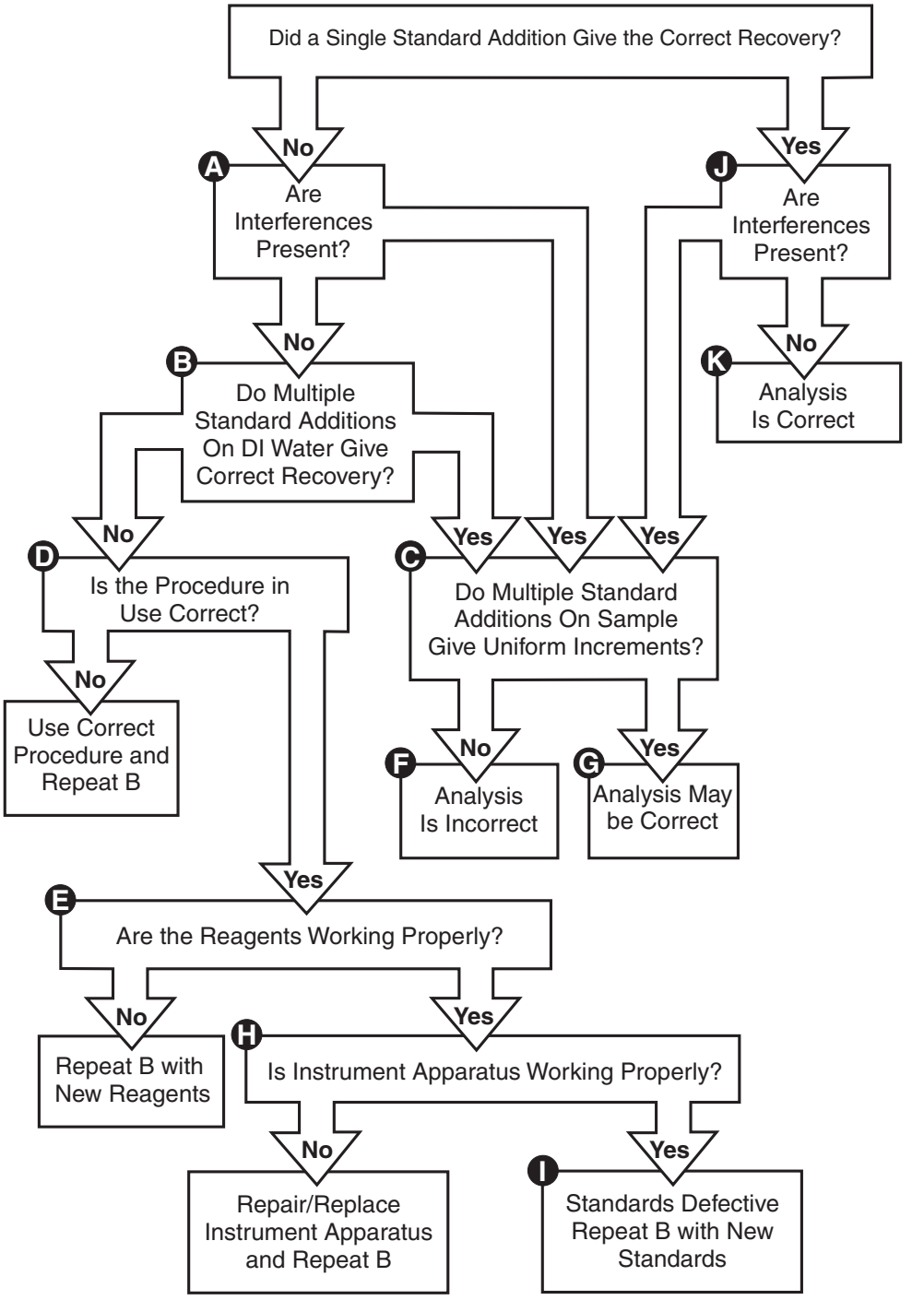
3. Add 0.2 mL of the same standard to a third 25 mL sample of deionized water.

4. Add 0.3 mL of the same standard to a fourth 25 mL sample of deionized water. Analyze all these samples for iron.

5. Tabulate the data as shown below:

mL of Standard Added	mg/L of Standard Added	mg/L of Iron Found
0	0	0
0.1	0.2	0.2
0.2	0.4	0.4
0.3	0.6	0.6

Figure 9 Standard Additions Decision Tree



The data show several points:

- The chemicals, instrument, procedure/technique and standards are working correctly because the iron added to the water sample was completely recovered in the same uniform steps that match the standard addition increments.
- Because iron added to the deionized water was recovered, but iron added to an actual sample was not recovered (Branch A), the sample contains an interference which prevents the test reagents from working properly.
- An iron analysis previously done on the actual sample using this method gave an inaccurate result.

If the results of multiple standard additions give the correct increment for each addition, proceed to Branch C.

If the results of multiple standard additions do not give the correct increment for each addition, go to Branch D.

Branch C

If interfering substances are present, the analysis may be incorrect. However, with multiple standard additions, it may be possible to arrive at an approximate result if the increases are uniform.

Suppose the sample result for iron was 1.0 mg/L. Because interferences may be present, a standard addition of 0.1 mL of a 50 mg/L iron standard to a 25 mL sample is made. The expected increase in the iron concentration is 0.2 mg/L, but the actual increase is 0.1 mg/L. Then 0.2 and 0.3 mL of the same standard are added to two more 25 mL samples and analyzed for iron.

If there is a uniform increase in concentration between each addition (i.e., 0.1 mg/L difference between each addition), use Branch G. If the increase in concentration is not uniform (i.e., 0.1, 0.08, 0.05), go to Branch F.

Branch D

Carefully check the instructions for the test. Make sure to use the correct reagents in the correct order. Be sure the glassware in use is what is required. Be sure time for color development and the sample temperature are as specified. If the procedure technique was incorrect, repeat Branch B. If the procedure was correctly followed, proceed to Branch E.

Branch E

Check the reagent performance. This may be done by obtaining a fresh lot of reagent or by using a known standard solution to run the test. Make sure the color development time given in the procedure is equal to the

time required for the reagent in question. If the reagent(s) is defective, repeat Branch B with new reagents. If the reagents are good, proceed with Branch H.

Branch F

Examples of non-uniform increments between standard additions are shown below.

Example A

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	1.0
0.1	0.2	1.10
0.2	0.4	1.18
0.3	0.6	1.23

Example B

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	0
0.1	0.2	0
0.2	0.4	0.2
0.3	0.6	0.4

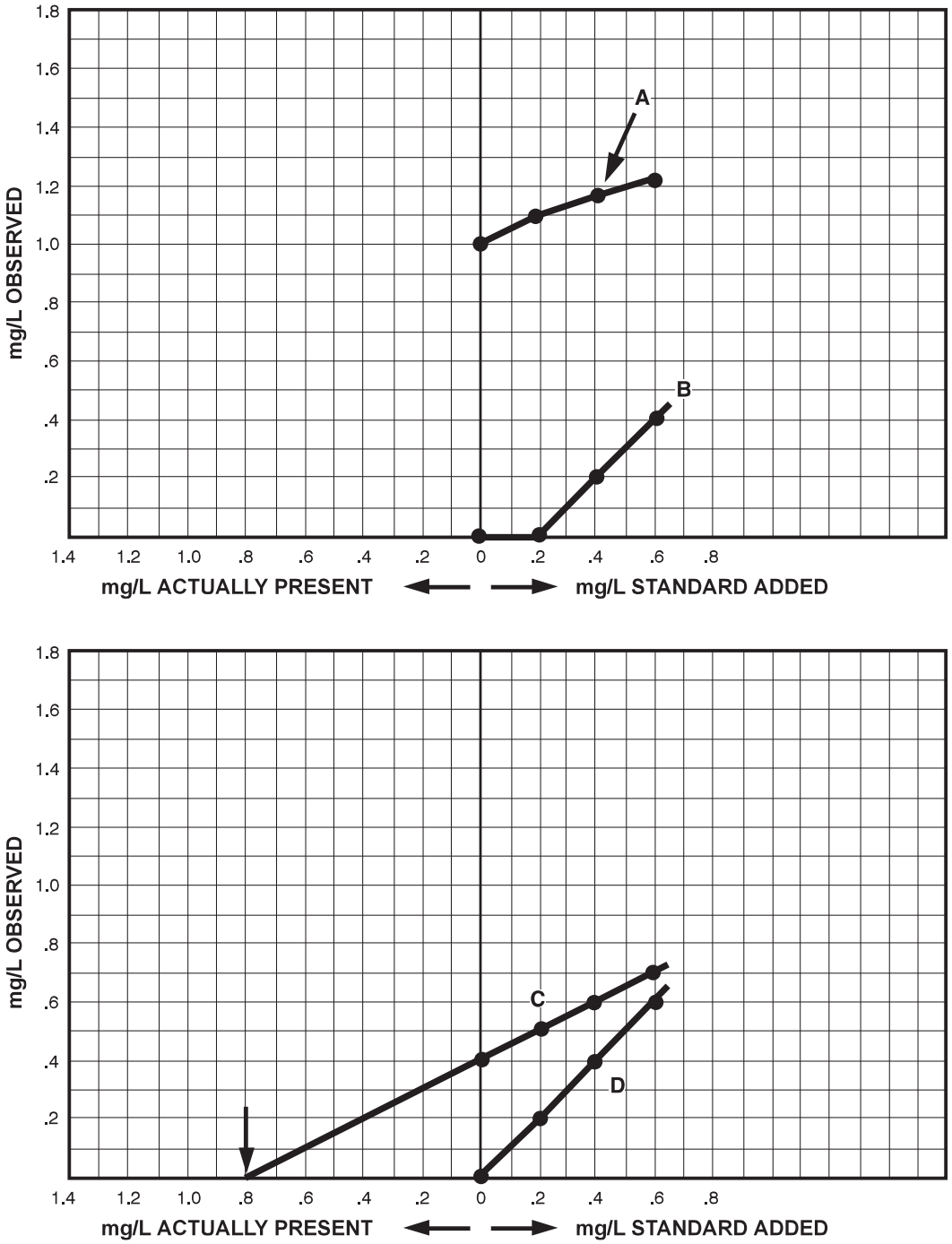
These examples show the effect of interferences on the standard addition. Data plotted on the graph in *Figure 10* for samples A and B show that the four data points do not lie on a straight line.

The plot for sample A illustrates an interference that becomes progressively worse as the concentration of the standard increases. This type of interference is uncommon and may be caused by an error or malfunction of the procedure, reagents or instrument. It is recommended Branch B be performed to verify the supposed interference.

The plot for sample B shows a common chemical interference which becomes less or even zero as the concentration of standard increases. The graph shows the first addition was consumed by the interference and the remaining additions gave the correct increment of 0.2 mg/L.

The apparent interference in Example B could be the result of an error made in the standard addition. Repeat the analysis to see if an error was made during standard addition. If not, the method is not appropriate for the sample matrix. When these two types of interferences occur, try to analyze the sample with a method which uses a different type of chemistry.

Figure 10 Multiple Standard Additions Graph



Branch G

Examples of uniform increments between standard additions are given below.

Example C

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	0.4
0.1	0.2	0.5
0.2	0.4	0.6
0.3	0.6	0.7

The plot for sample C illustrates a common interference with a uniform effect on the standard and the substances in the sample. The four data points form a straight line which may be extended back through the horizontal axis. The point where the line meets the axis can be used to determine the concentration of the substance you are measuring.

In this example, the first analysis gave 0.4 mg/L. After extrapolating the line to the horizontal axis, the graph shows the result should be much closer to the correct result: 0.8 mg/L.

Apparent interferences may also be caused by a defect in the instrument or standards. Before assuming the interference is chemical, check Branch B.

Example D

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	0
0.1	0.2	0.2
0.2	0.4	0.4
0.3	0.6	0.6

The plot for sample D illustrates a problem for the analyst. The increments are uniform and the recovery of the standard was complete. The result of the first analysis was 0 mg/L and the line extrapolates back through 0 mg/L. If interferences are known to be present, the interferences may be present in an amount equal to the substance in question, preventing the analyst from finding the substance. This would be an uncommon situation.

Branch H

Check operation of the instrument and/or apparatus used to perform the test. Check glassware used in the procedure and make sure it is extremely clean. Dirty pipets and graduated cylinders can cause contamination and will not deliver the correct volume. Check delivery of pipets by using deionized water and a balance; 0.2 mL = 0.2 grams.

If a defect is found in the instrument and/or apparatus, repeat Branch B after repair or replacement. If the instrument and apparatus are working, proceed with Branch I.

Branch I

After determining the procedure, reagents, instrument and/or apparatus are correct and working properly, you may conclude the only possible cause for standard additions not functioning correctly in deionized water is the standard used for performing standard additions. Obtain a new standard and repeat Branch B.

Branch J

If the standard additions gives the correct result, the analyst must then determine if an interfering substance(s) is present. If interfering substances are present, proceed to Branch C. If they are not present, the analysis is correct.

If you still cannot identify the problem, extra help is available. Please call our Technical Support Group at 800-227-4224 (U.S.A.) or 970-669-3050. A representative will be happy to help you.

Method Performance

Estimated Detection Limit

Ranges for chemical measurements have limits. The lower limit is important because it determines whether a measurement is different from zero. Many experts disagree about the definition of this detection limit, and determining it can be difficult. The Code of Federal Regulations (40 CFR, Part 136, Appendix B) provides a procedure to determine the "Method Detection Limit" or MDL. The MDL is the lowest concentration that is different from zero with a 99% level of confidence. A measurement below this MDL may be useful, but there is a greater chance that it is actually zero.

The MDL is not fixed; it varies for each reagent lot, instrument, analyst, sample type, etc. Therefore, a published MDL may be a useful guide, but is only accurate for a specific set of circumstances. Each analyst should determine a more accurate MDL for each specific sample matrix using the same equipment, reagents and standards that will routinely be used for measurements.

Hach provides a value called the Estimated Detection Limit (EDL) for all programs. It is the calculated lowest average concentration in a deionized water matrix that is different from zero with a 99% level of confidence. Specifically, it is the upper 99% confidence limit for zero concentration based on the calibration data used to prepare the pre-programmed calibration curve. **Do not use the EDL as the MDL**. The conditions for MDL determination must be exactly the same as the conditions used for analysis. The EDL may be useful to the analyst as a starting point in determining a MDL or as a way to compare methods. Measurements below the EDL may also be valuable because they can show a trend, indicate the presence of analyte and/or provide statistical data. However, these values have a large uncertainty.

Method Detection Limit (MDL)

This method is in accordance with the USEPA definition in 40 CFR, Part 136, Appendix B (see most current edition).

The USEPA defines the method detection limit (MDL) as the minimum concentration that can be determined with 99% confidence that the true concentration is greater than zero. Since the MDL will vary from analyst to analyst, it is important that analysts determine the MDL based on their unique operating conditions.

The procedure for determining MDL is based on replicate analyses at a concentration 1 to 5 times the estimated detection limit. The MDL value is calculated from the standard deviation of the replicate study results multiplied by the appropriate Student's *t* value for a 99% confidence interval. For this definition, the MDL does not account for variation in sample composition and can only be achieved under ideal conditions.

1. Estimate the detection limit. Use the Hach estimated detection limit (EDL) value stated in the *Method Performance* section of the analysis procedure.

2. Prepare a laboratory standard of the analyte in deionized water which is free of the analyte that is 1 to 5 times the estimated detection limit.

3. Analyze at least seven portions of the laboratory standard and record each result.

4. Calculate the average and standard deviation *(s)* of the results.

5. Compute the MDL using the appropriate Student's *t* value (see table below) and the standard deviation value:

MDL = Student's *t* x *s*

Number of Test Portions	Student's *t* Value
7	3.143
8	2.998
9	2.896
10	2.821

For example:
The EDL for measuring iron using the FerroZine method is 0.003 mg/L. An analyst accurately prepared 1 liter of a 0.010 mg/L (about 3x the EDL) laboratory standard by diluting a 10-mg/L iron standard in iron-free deionized water.

Eight portions of the standard were tested according to the FerroZine method with the following results:

Sample #	Result (mg/L)
1	0.009
2	0.010
3	0.009
4	0.010
5	0.008
6	0.011
7	0.010
8	0.009

Using a calculator program, the average concentration = 0.010 mg/L and the standard deviation (*s*) = 0.0009 mg/L

Based on the USEPA's definition, calculate the MDL as follows:

MDL for FerroZine method = 2.998 (Student's *t*) x 0.0009 (*s*)

MDL = 0.003 mg/L (agrees with initial estimate)

Note: *Occasionally, the calculated MDL may be very different than Hach's estimate of the detection limit. To test how reasonable the calculated MDL is, repeat the procedure using a standard near the calculated MDL. The average result calculated for the second MDL derivation should agree with the initial calculated MDL. Refer to 40 CFR, Part 136, Appendix B (7-1-94), pages 635-637 for detailed procedures to verify the MDL determination.*

Note: *Run a laboratory blank, containing deionized water without analyte, through the test procedure to confirm that the blank measurement is less than the calculated MDL. If the blank measurement is near the calculated MDL, repeat the MDL procedure using a separate blank for analysis for each standard solution portion analyzed. Subtract the average blank measurement from each standard and use the corrected standard values to calculate the average and standard deviation used in the MDL.*

Precision

Every measurement has some degree of uncertainty. Just as a ruler with markings of 0.1 mm leaves some doubt as to the exact length of a measurement, chemical measurements also have some degree of uncertainty. The quality of the entire chemical method determines the precision.

Uncertainty in chemical measurements may be due to systematic errors and/or random errors. A systematic error is a mistake that is always the same for every measurement made. For example, a blank can add to each measurement for a specific compound, giving consistently high results (a positive bias). Random errors are different for every test and add either positive or negative bias. Random errors may be caused by variation in analytical technique and cause response variation. Hach chemists work hard to eliminate systematic errors in Hach procedures using Hach reagents, but response variation occurs in all chemical measurements.

Estimating Precision

The method performance section in each procedure provides an estimate of the procedure's precision. The procedures use a "replicate analysis" estimate, based on real data.

In replicate analysis, a Hach chemist prepares a specific concentration of the analyte in a deionized water matrix. The standard is then analyzed seven individual times with the two reagent lots used in the calibration (14 total samples). A standard deviation of the two sets of seven values is calculated. The larger value is reported in the method. The reported value provides an estimate of the "scatter" of results at a particular point in the calibration curve.

It is important to stress that the estimates are based on a deionized water matrix. Precision on real samples with varying matrices can be quite different than these estimates.

Reagent Blank Correction

The Reagent Blank Correction subtracts the color absorbed when running the test with deionized water instead of sample. The blank value is subtracted from every result to correct for any background color due to reagents.

When using the Reagent Blank Correction feature, the blank correction should be entered before the Standard Adjust feature is used.

To enter a programmed correction for the reagent blank:

1. Run the test using deionized water with each new lot of reagents.
2. Press **READ** to obtain the blank value.
3. Press **SETUP**, scroll to **BLANK** and press **ENTER**. The display will show **BLANK?**.
4. Enter the blank value just read from the instrument.
5. Press **ENTER** to accept the value as the blank to be subtracted from each reading.
6. The display will show 0.00 mg/L (resolution and units vary) and the sample cell icon will be displayed, indicating that the reagent blank feature is enabled and the blank value will be subtracted from each reading. Repeat the reagent blank adjust for each new lot of reagents.

Note: *After entering a reagent blank adjust, the display may flash "limit" when zeroing if the sample used for zeroing has a lower absorbance value than the reagent blank.*

To disable the Reagent Blank adjust feature, press **SETUP**, scroll to **BLANK** and press **ENTER** twice. The concentration readings will be displayed without subtracting the blank. The sample cell icon will no longer appear in the display.

Do not use the Reagent Blank Adjust feature if the procedure uses a reagent blank for zeroing.

Standard Adjust (Adjusting the Standard Curve)

The colorimeter has Hach Programs permanently installed in memory. A program usually includes a pre-programmed calibration curve. Each curve is the result of an extensive calibration performed under ideal conditions and is normally adequate for most testing. Deviations from the curve can occur from using compromised testing reagents, defective sample cells, incorrect test procedure, incorrect technique, or other correctable causes. Interfering substances or other causes may be beyond the analyst's control.

In some situations, using the pre-programmed curve may not be convenient:

a) Running tests where frequent calibration curve checks are required.

b) Testing samples which give a consistent test interference.

Consider the following before adjusting the calibration curve:

1. Will future test results be improved by adjusting the curve?
2. Are interfering substances consistent in all the samples that you will test?

Any precision and test range information provided with the procedure may not apply to an adjusted curve calibration.

You can adjust many of the calibration curves by following the steps found in the test procedures. Working carefully is important. After the adjustment, it is wise to run standard solutions of several concentrations to make sure the adjusted curve is satisfactory. Perform standard additions on typical samples to help determine if the adjusted curve is acceptable.

Think of the standard adjust measurement as a two-step process. First, the instrument measures the sample using the pre-programmed calibration. Second, it multiplies this measurement by an adjustment factor. The factor is the same for all concentrations. The instrument will remember the factor indefinitely and will display the standard adjustment icon when it is used.

Adjust the calibration curve using the reading obtained with a Hach Standard Solution or carefully prepared standard made from a concentrated Hach Standard Solution. It is important to adjust the curve in the correct concentration range. For most purposes, Hach recommends adjusting the curve using a standard concentration that is 70 to 85% of the maximum concentration range of the test.

For example, the Hach pre-programmed method for fluoride has a range of 0-2.0 mg/L F. To adjust the calibration curve, use a standard with a concentration between 1.4-1.6 mg/L. Hach provides a 1.60 mg/L Fluoride Standard Solution (80% of the full range). This is a convenient standard to use for adjusting the calibration curve.

If the range of all your samples is known to be below a concentration that is less than 50% of the full range (50% of 2.0 is 1.0 mg/L), then adjust the standard curve with a standard that is within that range. For example, if all the samples contain 0.6-0.9 mg/L F, you may use a 1.00 mg/L fluoride standard to adjust the curve. You may use the 1.00 mg/L standard because it is closer to the sample range you are working with.

If you are using a Reagent Blank Correction, the blank correction should be entered before the standard curve is adjusted.

To adjust the standard curve:

1. Prepare the standard.
2. Use the standard as the sample in the procedure.
3. When the reading for the standard is obtained, press **SETUP**.
4. Use the arrow keys to scroll to the "**STD**" setup option.
5. Press **ENTER** to activate the standard adjust option.
6. Edit the standard concentration to match that of the standard used.
7. Press **ENTER**. A small plot of a line through a point will be displayed, indicating that the curve has been adjusted with the standard.

Note: *If the attempted correction is outside the allowable adjustment limit, the instrument will beep and flash Ø and the operation will not be allowed.*

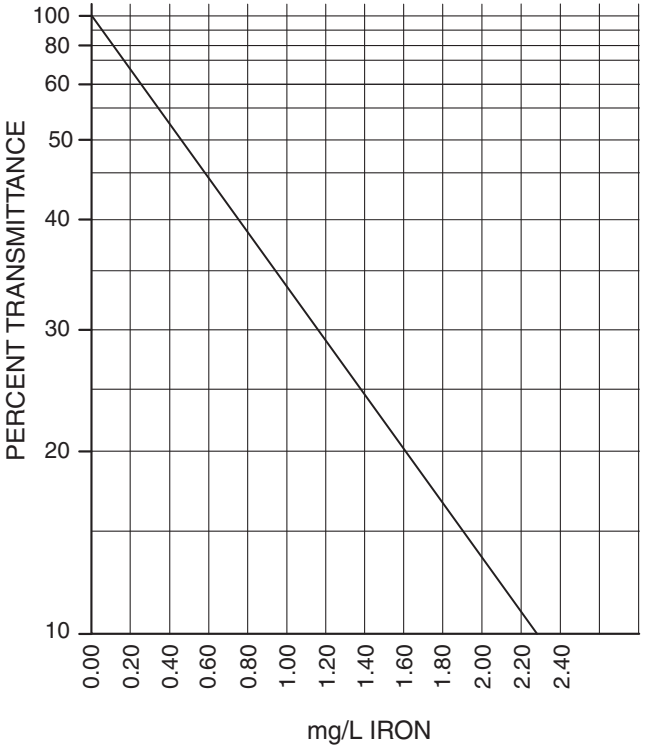
Preparing a User-Entered Calibration Curve

1. Prepare five or more standards of known concentration that cover the expected range of the test. Run tests as described in the procedure on each prepared standard. Pour the customary volume of each known solution into a separate clean sample cell of the type specified for your instrument.
2. Standardize (zero) the instrument using an untreated water sample or a reagent blank, whichever the procedure instructs you to use.
3. Measure and record the absorbance or %T of the known solutions. To use %T vs. concentration see *%T Versus Concentration Calibration*. To use absorbance vs. concentration, see *Absorbance Versus Concentration Calibration*. Or create a user-entered program by storing a custom calibration in the non-volatile memory of the instrument. Refer to the section on entering user-entered programs in the instrument manual.

%T Versus Concentration Calibration

If measuring %T, use semilogarithmic graph paper and plot %T (vertical scale) versus concentration (horizontal scale). In *Figure 11*, iron standard solutions of 0.1, 0.2, 0.4, 0.8, 1.2, 1.6, and 2.0 mg/L were measured on a spectrophotometer at 500 nm using half-inch test tubes. Results were plotted and the calibration table values were extrapolated from the curve (*Table 7*).

Figure 11 Logarithmic Calibration Curve



To convert %T readings to concentration, prepare a table such as *Table 7* and select the appropriate line from the "%T Tens" column and the appropriate column from the %T Units columns. The %T Ten value is the first number of the %T reading and the %T Units value is the second number of the %T reading. For example, if the instrument reading was 46%, the 40 line in the %T Tens column and the 6 column in the %T Units would be selected. The cell where these two intersect (0.78 mg/L) is the iron concentration of the sample.

Table 7 Calibration Table

%T Tens	%T Units									
	0	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
0										
10	2.30	2.21	2.12	2.04	1.97	1.90	1.83	1.77	1.72	1.66
20	1.61	1.56	1.51	1.47	1.43	1.39	1.35	1.31	1.27	1.24
30	1.20	1.17	1.14	1.11	1.08	1.04	1.02	.99	.97	.94
40	.92	.89	.87	.84	.82.	.80	.78	.76	.73	.71
50	.69	.67	.65	.64	.62	.60	.58	.56	.55	.53
60	.51	.49	.48	.46	.45	.43	.42	.40	.39	.37
70	.36	.34	.33	.32	.30	.29	.28	.26	.25	.24
80	.22	.21	.20	.19	.17	.16	.15	.14	.13	.12
90	.11	.09	.08	.07	.06	.05	.04	.03	.02	.01

Absorbance Versus Concentration Calibration

To read concentration values directly from the instrument, create a user-entered program. See the instrument manual for more information.

If absorbance values are measured, plot the results on linear graph paper. Plot the absorbance value on the vertical axis and the concentration on the horizontal axis.

Plot increasing absorbance values from bottom to top. Plot increasing concentration values from left to right. Values of 0.000 absorbance units and 0 concentration will begin at the bottom left corner of the graph. A calibration table can be extrapolated from the curve or the concentration values can be read directly from the graph for determining an equation for the line using the slope and the y-intercept.

USEPA Approved and Accepted Definitions

The United States Environmental Protection Agency (USEPA) establishes limits for maximum contamination levels of certain constituents in water. It also requires that specific methodology be used to analyze for these constituents. These methods originate from several sources. The USEPA has developed some of these methods. In other cases, the USEPA has evaluated and approved methods developed by manufacturers, professional groups and public agencies such as:

- American Public Health Association

- American Water Works Association
- Water Environmental Federation
- American Society for Testing and Materials
- United States Geological Survey
- Associates of Official Analytical Chemists

All USEPA approved methods are cited in the *Federal Register* and compiled in the Code of Federal Regulations. USEPA approved methods may be used for reporting results to the USEPA and other regulatory agencies.

USEPA Accepted

Hach has developed several procedures that are equivalent to USEPA approved methods. Even though minor modifications exist, the USEPA has reviewed and accepted certain procedures for reporting purposes. These methods are not published in the *Federal Register,* but are referenced to the equivalent USEPA method in the procedure.

Digestion

Several procedures require sample digestion. Digestion uses chemicals and heat to break down a substance into components that can be analyzed. This section has three different digestion procedures.

The Hach Digesdahl® system is a process that yields a digest suitable for the determination of metals, total phosphorus and total kjeldahl nitrogen (TKN). It is rapid, convenient and the method of choice for digesting most samples analyzed by Hach methods.

For USEPA reporting purposes, USEPA-approved digestions are required. USEPA presents two digestions (mild and vigorous) for metals analysis. These are much more inconvenient and time consuming compared to the Hach Digesdahl system. Other digestion procedures are required for phosphorus and TKN.

EPA Mild Digestion with Hot Plate for Metals Analysis Only

1. Acidify the entire sample at the time of collection with concentrated nitric acid by adding 5 mL of acid per liter (or quart) of sample.

2. Transfer 100 mL of well-mixed sample to a beaker or flask. Add 5 mL of distilled 1:1 hydrochloric acid (HCl).

3. Heat using a steam bath or hot plate until the volume has been reduced to 15-20 mL. Make certain the sample does not boil.

4. After this treatment, the sample may be filtered to remove any insoluble material.

5. Adjust the digested sample to pH 4 by drop-wise addition of 5.0 N Sodium Hydroxide Standard Solution. Mix thoroughly and check the pH after each addition.

6. Quantitatively transfer the sample with deionized water to a 100-mL volumetric flask and dilute to volume with deionized water. Continue with the procedure. This mild digestion may not suffice for all sample types. A reagent blank also should be carried through the digestion and measurement procedures.

EPA Vigorous Digestion with Hot Plate for Metals Analysis Only

A vigorous digestion can be followed to ensure all organo-metallic bonds are broken.

1. Acidify the entire sample with redistilled 1:1 Nitric Acid Solution to a pH of less than two. Do not filter the sample before digestion.

2. Transfer an appropriate sample volume (see *Table 8*) into a beaker and add 3 mL of concentrated redistilled nitric acid.

3. Place the beaker on a hot plate and evaporate to near dryness, making certain the sample does not boil.

4. Cool the beaker and add another 3 mL of the concentrated redistilled nitric acid.

5. Cover the beaker with a watch glass and return it to the hot plate. Increase the temperature of the hot plate so that a gentle reflux occurs. Add additional acid, if necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change color or appearance with continued refluxing).

6. Again, evaporate to near dryness (do not bake) and cool the beaker. If any residue or precipitate results from the evaporation, add redistilled 1:1 hydrochloric acid (5 mL per 100 mL of final volume). See *Table 8*.

7. Warm the beaker. Add 5 mL of 5.0 N sodium hydroxide and quantitatively transfer the sample with deionized water to a volumetric flask. See *Table 8* below for the suggested final volume.

8. Adjust the sample to pH 4 by drop-wise addition of 5.0 N Sodium Hydroxide Standard Solution; mix thoroughly and check the pH after each addition. Dilute to volume with deionized water. Multiply the result by the correction factor in *Table 8*. A reagent blank also should be carried through the digestion and measurement procedures.

Table 8 Vigorous Digestion Volumes

Expected Metal Concentration	Suggested Sample Vol. for Digestion	Suggested Volume of 1:1 HCl	Suggested Final Volume After Digestion	Correction Factor
1 mg/L	50 mL	10 mL	200 mL	4
10 mg/L	5 mL	10 mL	200 mL	40
100 mg/L	1 mL	25 mL	500 mL	500

General Digesdahl Digestion (Not USEPA accepted)

Many samples may be digested using the Digesdahl Digestion Apparatus (Cat. No. 23130). It is designed to digest many types of samples such as oils, wastewater, sludges, feeds, grains, plating baths, food, and soils. In this procedure the sample is oxidized by a mixture of sulfuric acid and hydrogen peroxide. Digestion of a dry sample requires less than ten minutes, while liquid samples require about 1 minute/mL. The digestion is done in a special flat-bottomed 100-mL volumetric flask. Aliquots (sample portions) are taken for analysis using colorimetric methods.

Procedures for digestion and using the Digesdahl Digestion Apparatus are based on the type and form of the sample, and are found in the Digesdahl Digestion Apparatus Instruction Manual, which is included with each Digesdahl Digestion Apparatus.

Distillation

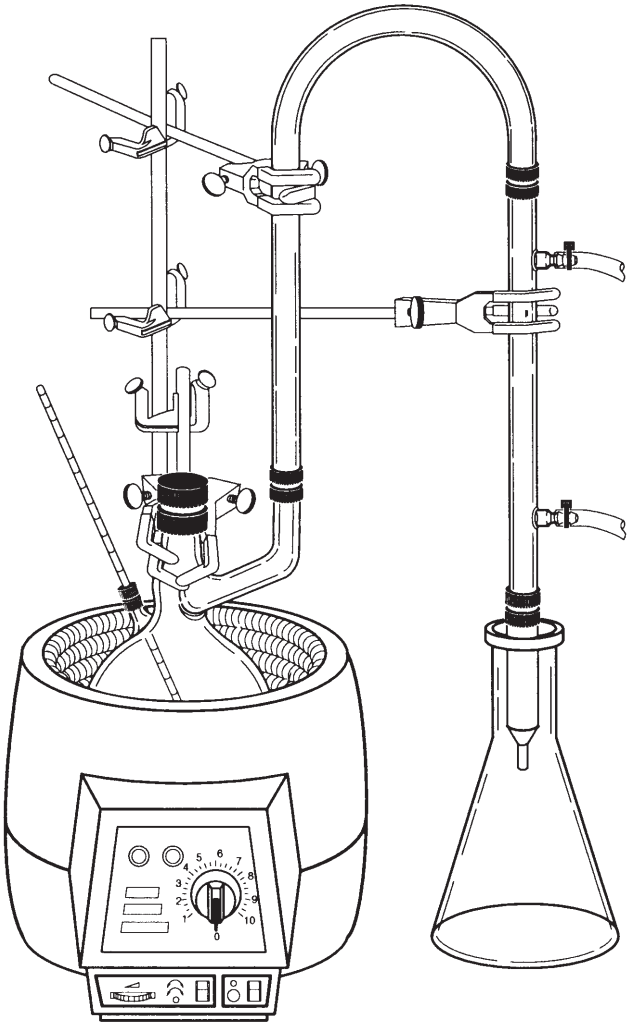
Distillation is an effective way of separating chemical components for analysis. The Hach Distillation Apparatus (see *Figure 12*) is adapted easily for many test needs and is suitable for water and wastewater samples. Sample distillations are easy and safe to perform.

Applications for the General Purpose Distillation Apparatus include:

- fluoride
- albuminoid nitrogen
- ammonia nitrogen
- phenols
- selenium
- volatile acids

Arsenic and cyanide require special glassware sets in addition to the General Purpose Set (the Arsenic Distillation Apparatus and the Cyanide Distillation Apparatus). All connecting glassware is manufactured with threaded connectors for ease and safety. The General Purpose Heater provides efficient heating and the Support Apparatus anchors the glassware.

Figure 12 General Purpose Distillation Apparatus with Heater and Support Apparatus



SECTION 3 WASTE MANAGEMENT AND SAFETY

Waste Management

This section provides guidelines for laboratory waste management. It should assist you in complying with USEPA regulations governing waste management. It summarizes basic requirements, but does not contain all USEPA regulations. It does not relieve people from complying with all regulations contained in the Code of Federal Regulations. Regulations change regularly and additional state and local laws may apply to your waste. Each waste generator is responsible for knowing and obeying the laws that apply to them.

Waste Minimization

Waste minimization is the foundation of good waste management. Minimizing waste greatly reduces the disposal problems and expense. If possible, try to generate less waste rather than recycle or re-use it. For laboratories, ways to reduce waste include:

- Use the smallest sample size possible.
- Choose methods that use non-hazardous or “less” hazardous reagents when possible.
- Buy chemicals in small quantities which will be used before they expire. This eliminates disposal of outdated materials.
- Clean glassware and laboratory apparatus with non-hazardous soaps when possible, rather than solvents or acids which may be hazardous.

Regulatory Overview

Federal waste disposal regulations were issued in accordance with the Resource Conservation and Recovery Act (RCRA). They are given in Title 40 Code of Federal Regulations (CFR) part 260. The Act controls all forms of solid waste disposal and encourages recycling and alternative energy sources. The major emphasis is controlling hazardous waste disposal. The regulations create a system to identify wastes and track waste generation, transport, and ultimate disposal. Each facility involved in managing hazardous waste must be registered with the USEPA. This includes the generator, transporters, and treatment, storage, and disposal facilities (TSDF).

Under federal regulations, there are three categories of generators with increasingly more strict regulation for larger quantity generators. The categories are based on the amount of hazardous waste generated in any given month.

The categories are as follows:

- Conditionally Exempt Small Quantity Generator - less than 100 kg (220 lb.) per month
- Small Quantity Generator - between 100 kg (220 lb.) and 1,000 kg (2,200 lb.) per month
- Large Quantity Generator - greater than 1,000 kg (2,200 lb.) per month

Note: *If a laboratory generates acutely hazardous waste (as defined on 40 CFR 261) or accumulates more than a certain amount of waste, the facility may be moved into a larger generator status. Check with your environmental compliance manager or state and local officials to determine which category your facility is in.*

Hazardous Waste Definition

For regulatory purposes, a "hazardous waste" is a material which is subject to special laws by the USEPA under 40 CFR 261. In addition, many states or local authorities regulate additional materials as hazardous waste. Be aware that many very toxic compounds are not regulated by this definition of hazardous waste. However, improper management or disposal of these compounds may lead to legal problems under other laws such as CERCLA (Superfund) or common law torts.

The 40 CFR 261 defines a hazardous waste as a solid waste which is not excluded from regulation and meets any of the following criteria:

- It is a discarded commercial chemical product, off-specification species, container residue, or spill residue of materials specifically listed in 40 CFR 261.33;
- It is a waste from a specific source listed in 40 CFR 261.32;
- It is a waste from a non-specific source listed in 40 CFR 261.31; or
- It displays any of the following characteristics of hazardous waste defined in 40 CFR 261.20-24:
    - ignitability
    - corrosivity
    - reactivity
    - toxicity

There are many exceptions to these regulations, and each generator should review the regulations and determine if they are excluded from the regulations.

Characteristic Hazardous Waste Codes

Hazardous wastes are categorized by specific codes assigned in 40 CFR 261.20-261.33. These codes will help you identify hazardous waste. The generator is responsible for making the actual waste code determination.

Selected characteristic waste codes for chemicals which may be generated using Hach methods for water analysis are given in the following table. A complete list of waste codes is found in 40 CFR 261.24.

USEPA Code	Characteristic	CAS No.	Regulatory Level (mg/L)
D001	Ignitability	na	na
D002	Corrosivity	na	na
D003	Reactivity	na	na
D004	Arsenic	6440-38-2	5.0
D005	Barium	6440-39-3	100.0
D018	Benzene	71-43-2	0.5
D006	Cadmium	7440-43-9	1.0
D022	Chloroform	67-66-3	6.0
D007	Chromium	7440-47-3	5.0
D008	Lead	7439-92-1	5.0
D009	Mercury	7439-97-6	0.2
D010	Selenium	7782-49-2	1.0
D011	Silver	7440-22-4	5.0

How to Determine if Waste is Hazardous

Federal laws do not require you to test a material to decide if it is a hazardous waste. You may apply product knowledge to decide if a material is hazardous. Often, information on a material safety data sheet (MSDS) is enough to decide. If the product is specifically listed in the regulation, it is a hazardous waste.

You also need to decide if it has any characteristics of a hazardous waste. Physical information on the MSDS may help you decide. If the flash point is below 60 °F (15 °C) or is classified by DOT as an oxidizer, the material may be ignitable. If the pH of the material is ≤2 or ≥12.5, the material may be corrosive. If the material is unstable, reacts violently with water, or may generate toxic gases, vapors, or fumes when mixed with water, it may be reactive.

Use the chemical composition data to decide if a material is toxic. This decision is based on the concentration of certain contaminants (heavy metals and a number of organic compounds). If the waste is a liquid, compare the concentration of the contaminants in the liquid to the concentrations listed in 40 CFR 261.24. If the waste is a solid, analyze the sample by the Toxicity Characteristic Leachability Procedure (TCLP) and compare the results to the concentration listed in the 40 CFR 261.24. Levels above the threshold amount listed in the table are hazardous.

See "Sections of the MSDS" on page 63. describing the MSDS for help in finding information for making hazardous waste determinations.

Examples of Hazardous Waste

A number of chemicals used in and final solutions created from Hach procedures are hazardous wastes when they are disposed. In addition, substances in the sample matrix may be a hazardous waste. Sometimes, reagents which would be hazardous are neutralized or changed during the analytical procedure. In that case, the final solutions are not regulated. Finally, many reagents and final solutions may be non-regulated. The generator must either use their knowledge of the materials used or conduct analytical tests to determine if the final material is a hazardous waste.

Examples of tests using Hach reagents that generate hazardous waste include those containing mercury or mercury compounds such as COD tests or Nessler's reagent. Conversely, a test using Hach reagents such as ManVer 2 Hardness Indicator Powder Pillows and EDTA Titration Cartridges do not produce a hazardous waste unless the sample contains a hazardous substance.

Hazardous Waste Disposal

Hazardous waste must be managed and disposed of according to federal, state, and local regulations. The waste generator is responsible for making hazardous waste determinations. Analysts should check with the facility's environmental compliance people for specific instructions.

Hazardous wastes should be handled by treatment, storage, and disposal facilities (TSDF) that have USEPA permits. In some cases, the generator may treat the hazardous waste. In most cases, a permit from the USEPA is required to treat hazardous waste. Laboratories are not exempt from these regulations. If your facility is a "Conditionally Exempt Small Quantity Generator," special rules may apply. Check 40 CFR 261 to determine if have to comply with all the laws.

The most common allowed treatment is elementary neutralization. This refers to neutralizing wastes that are hazardous only because they are corrosive or are listed only for that reason. Neutralize acidic solutions by adding a base such as sodium hydroxide; neutralize basic solutions by

adding an acid such as hydrochloric acid. Slowly add the neutralizing agent while stirring. Monitor the pH. When it is at or near 7, the material is neutralized and may be flushed down the drain. Many wastes generated from Hach procedures may be treated in this manner.

Other chemical or physical treatments such as cyanide destruction or evaporation may require a permit. Check with your environmental department or local regulators to determine which rules apply to your work facility.

Laboratory chemicals may be mixed and disposed of with other hazardous wastes generated at your facility. They may also be accumulated in accordance with 40 CFR 262.34 satellite accumulation rules. After collection they may be disposed of in a "labpack." A number of environmental and hazardous waste companies offer labpacking services. They will inventory, sort, pack, and arrange proper disposal for hazardous waste. Find companies offering these services in the Yellow Pages under "Waste Disposal - Hazardous" or contact state and local regulators for assistance.

Management of Specific Wastes

Hach has several documents to assist customers in managing waste generated from our products. You can obtain the following documents by calling 1-800-227-4224 or 970-669-3050 and requesting the literature codes given:

Literature Code	Title
1321	Waste Reduction: A Primer
9323	Mercury Waste Disposal Firms
9325	COD Waste Management
9326	COD Heavy Metal Total Concentrations

Special Considerations for Cyanide-Containing Materials

Several procedures in this manual use reagents that contain cyanide compounds. These materials are regulated as reactive (D003) waste by the Federal RCRA. Waste disposal instructions provided with each procedure tell you how to collect these materials for proper disposal. It is imperative that these materials be handled safely to prevent the release of hydrogen cyanide gas (an extremely toxic material with the smell of bitter almonds). Most cyanide compounds are stable and can be safely stored for disposal in highly alkaline solutions (pH >11) such as 2 N sodium hydroxide. Never mix these wastes with other laboratory wastes that may contain lower pH materials such as acids or even water.

If a cyanide-containing compound is spilled, you must be careful not to be exposed to hydrogen cyanide gas. Take the following steps to destroy the cyanide compounds in an emergency:

a) Use a fume hood, supplied air or self-contained breathing apparatus.

b) While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and either calcium hypochlorite or sodium hypochlorite (household bleach).

c) Add an excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.

d) Neutralize the solution and flush it down the drain with a large amount of water. If the solution contains other regulated materials such as chloroform or heavy metals, it may still need to be collected for hazardous waste disposal. Never flush hazardous wastes down the drain.

Resources

Many sources of information on proper waste management are available. The USEPA has a hotline number for questions about the Resource Conservation and Recovery Act (RCRA). The RCRA Hotline number is 1-800-424-9346. You may also get a copy of the appropriate regulations. Federal hazardous waste regulations are found in 40 CFR 260- 99. Obtain this book from the U.S. Government Printing Office or a number of other vendors. Other documents which may be helpful to the laboratory hazardous waste manager include:

1. Task Force on Laboratory Waste Management. *Laboratory Waste Management, A Guidebook*; American Chemical Society, Department of Government Relations and Science Policy: Washington, DC 1994.

2. Task Force on Laboratory Waste Management. *Waste Management Manual for Laboratory Personnel*; American Chemical Society, Department of Government Relations and Science Policy: Washington, DC 1990.

3. Task Force on Laboratory Waste Management. *Less is Better*; 2nd ed.; American Chemical Society, Department of Government Relations and Science Policy: Washington, DC 1993.

4. Committee on Chemical Safety. *Safety in Academic Chemistry Laboratories*, 5th ed.; American Chemical Society: Washington, DC, 1990.

5. Armour, Margaret-Ann. *Hazardous Laboratory Chemicals Disposal Guide*; CRC Press: Boca Raton, FL, 1991.

6. *Environmental Health and Safety Manager's Handbook*; Government Institutes, Inc.: Rockville, MD, 1988.

7. Lunn, G.; Sansone, E.B. *Destruction of Hazardous Chemicals in the Laboratory*; John Wiley and Sons: New York, 1990.

8. National Research Council. *Prudent Practices for Disposal of Chemicals from Laboratories*; National Academy Press: Washington, DC, 1983.

9. National Research Council. *Prudent Practices for Handling Hazardous Chemicals in Laboratories*; National Academy Press: Washington, DC, 1981.

10. Environmental Protection Agency, Office of Solid Waste and Emergency Response. *The RCRA Orientation Manual*; U.S. Government Printing Office: Washington, DC, 1991.

11. Environmental Protection Agency, Office of Solid Waste and Emergency Response. *Understanding the Small Quantity Generator Hazardous Waste Rules: A Handbook for Small Business*; U.S. Government Printing Office: Washington, DC, 1986.

Material Safety Data Sheets

Material safety data sheets (MSDS) describe the hazards of chemical products. This section describes the information provided on a Hach MSDS and how to locate important information for safety and waste disposal. The information provided on the MSDS applies to the product as sold by Hach. The properties of any mixtures obtained by using this product will be different.

How to Obtain an MSDS

Hach ships an MSDS to each customer with the first order of any chemical product. A new MSDS may be sent when the information on the data sheet is updated. Please review all new MSDS's for new information. If you need another copy of an MSDS, simply call 1-800-227-4227.

Sections of the MSDS

Each MSDS has ten sections. The sections and the information found in them are described below.

Header Information

The Hach catalog number, MSDS date, change number, company address and telephone number, and emergency telephone numbers are listed at the top of the MSDS.

1 Product Identification

This section contains:

- Hach product name
- Chemical Abstract Services (CAS) number
- Chemical name
- Chemical formula, if appropriate
- Chemical family to which the material belongs

2 Ingredients

This section lists each component in the product. It contains the following information for each component:

- PCT: Percent by weight of this component
- CAS NO.: Chemical Abstract Services (CAS) registry number for this component
- SARA: Superfund Amendments and Reauthorization Act, better known as the "Community Right to Know Law" tells you if the component is listed in SARA 313. If the component is listed and you use more than the amount listed, you must report this to the USEPA every year.
- TLV: Threshold Limit Value. The maximum airborne concentration for an 8 hour exposure that is recommended by the American Conference of Governmental Industrial Hygienists (ACGIH).
- PEL: Permissible Exposure Limit. The maximum airborne concentration for an 8 hour exposure that is regulated by the Occupational Safety and Health Administration (OSHA).
- HAZARD: Physical and health hazards of the component are explained.

3 Physical Data

The physical properties of the product are given in this section. They include the physical state, color, odor, solubility, boiling point, melting point, specific gravity, pH, vapor density, evaporation rate, corrosivity, stability, and storage precautions.

4 Fire, Explosion Hazard And Reactivity Data

This section contains the flash point and flammable limits of the material. It also includes how to fight fires if the material catches on fire. Key terms in this section include:

- Flashpoint: The temperature at which a liquid will give off enough flammable vapor to ignite.
- Flammability and ignitability are usually defined by the flash point.
- Lower Flammable Limit (LFL or LEL): The lowest concentration that will produce a fire or flash when an ignition source is present.
- Upper Flammable Limit (UFL or UEL): The vapor concentration in air above which the concentration is too rich to burn.
- NFPA Codes: The National Fire Protection Association (NFPA) has a system to rate the degree of hazards presented by a chemical. These codes are usually placed in a colored diamond. The codes range from 0 for minimal hazard to 4 for extreme hazard. They are grouped into the following hazards: health (blue), flammability (red), reactivity (yellow), and special hazards (white).

5 Health Hazard Data

This section describes different ways the chemical can enter your body (ingestion, inhalation, skin contact). It also gives acute (immediate) and chronic (long-term) health effects. If the material causes cancer or genetic damage, it is identified in this section.

6 Precautionary Measures

This section contains special precautions for the material. These may include special storage instructions, handling instructions, conditions to avoid, and protective equipment required to use this material safely.

7 First Aid

First aid instructions for exposures to the chemical are given in this section. Be sure to read this section before inducing vomiting in a victim. Some chemicals are better treated by not inducing vomiting. Seek prompt medical attention for all chemical exposures.

8 Spill And Disposal Procedures

This section tells about safe work practices for cleaning up and disposing of spilled material. Please refer to the Waste Management section of this manual. Final determination of proper and legal disposal options is the responsibility of the waste generator. Be sure you know the federal, state, and local laws that apply to your facility.

9 Transportation Data

Domestic and International shipping information is provided in this section. It gives shipping name, hazard class, and ID number of the product.

10 References

This section lists the reference materials used to write the MSDS.

Following the Reference section, the product is listed as having SARA 313 chemicals or California Proposition 65 List Chemicals, if applicable. Also found here is any special information about the product.

Safety

Safety is the responsibility of each person performing analytical procedures. Because many of the procedures in this methods manual use potentially hazardous chemicals and equipment, it is important to prevent accidents by practicing good laboratory techniques. The following guidelines apply to water analysis. These guidelines do not cover every aspect of safety, but they are important for preventing injuries.

Material Safety Data Sheet

A material safety data sheet (MSDS) comes with the first shipment of all products. The MSDS provides environmental and safety information about the products. Always read the MSDS before using a new product.

Reading Labels Carefully

Read each reagent label carefully. Pay particular attention to the precautions given. Never remove or block the label on a reagent container while it contains reagent. Do not put a different reagent into a labeled container without changing the label. When preparing a reagent or standard solution, label the container clearly. If a label is hard to read, re-label promptly according to your facility's hazard communication program.

Warning labels also appear on some of the apparatus used with the test procedures. The protective shields with the COD Reactor and the Digesdahl Digestion Apparatus point out potential hazards. Be sure these shields are in place during use and observe the precautions on the label.

Protective Equipment

Use the right protective equipment for the chemicals and procedures. The MSDS contains this information. Protective equipment may include:

- Eye protection such as safety glasses or goggles to protect from flying objects or chemical splashes.
- Gloves to protect skin from toxic or corrosive materials, sharp objects, very hot or very cold materials, or broken glass. Use tongs or finger cots when transferring hot apparatus.

- Laboratory coats or splash aprons to protect skin and clothing from splashes.
- Footwear to protect feet from spills. Open toed shoes should not be worn in chemistry settings.
- Respirators may be needed to protect you from breathing toxic vapors if adequate ventilation, such as fume hoods, are not available.
- Use fume hoods as directed by the procedure or as recommended in the MSDS.
- For many procedures, adequate ventilation is enough. Be sure there is enough fresh air and air exhaust to protect against unnecessary exposure to chemicals.

First Aid Equipment and Supplies

Most first aid instructions for chemical splashes in eyes or on skin call for thorough flushing with water. Laboratories should have eyewash and shower stations. For field work, carry a portable eyewash unit. Laboratories should also have appropriate fire extinguishers and fume hoods.

General Safety Rules

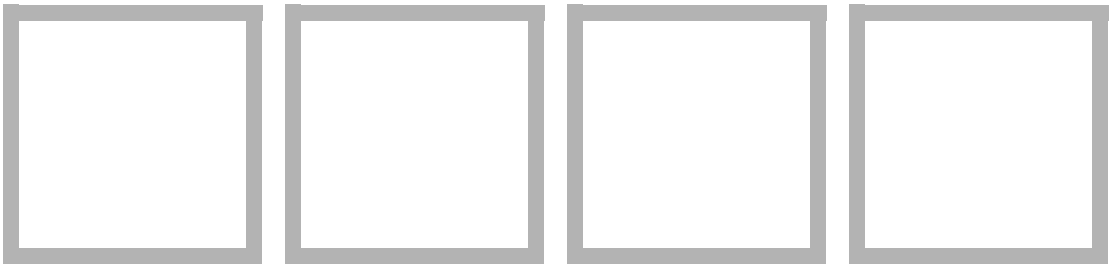
Follow these rules to make work with toxic and hazardous chemicals safer:

1. **Never** pipet by mouth. Always use a mechanical pipet or pipet bulb to avoid ingesting chemicals.
2. Follow test procedures carefully and observe all precautionary measures. Read the entire procedure carefully before beginning.
3. Wipe up all spills promptly. Get proper training and have the right response equipment to clean up spills. See your safety director for more information.
4. **Do not** smoke, eat, or drink in an area where toxic or irritating chemicals are used.
5. Use reagents and equipment only as directed in the test procedure.
6. **Do not** use damaged labware and broken equipment.
7. Minimize all chemical exposures. **Do not** breathe vapors or let chemicals touch your skin. Wash your hands after using chemicals.
8. Keep work areas **neat** and **clean**.

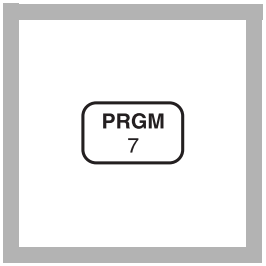
9. **Do not** block exits or emergency equipment.

OSHA Chemical Hygiene Plan

The Occupational Safety and Health Administration (OSHA) enforces laws about the control exposure to hazardous chemicals in laboratories. These regulations are in Title 29 CFR 1910.1450. They apply to all employers who use hazardous chemicals. They require employers to develop and use a written Chemical Hygiene Plan and appoint a qualified person as the Chemical Hygiene Officer.



Aluminon Method*



1. Enter the stored program number for aluminum (Al).

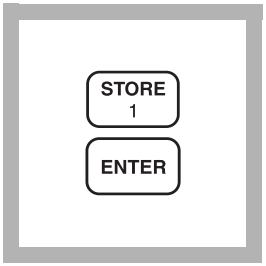
Press: **PRGM**

The display will show:

PRGM ?

Note: *Adjust the pH of stored samples before analysis.*

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

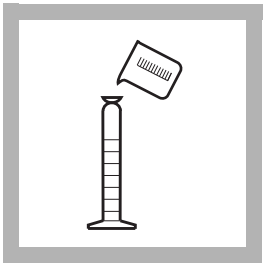


2. Press: **1 ENTER**

The display will show **mg/L, Al** and the **ZERO** icon**.**

Note: *Total aluminum determination requires a digestion prior to analysis (see Section 2).*

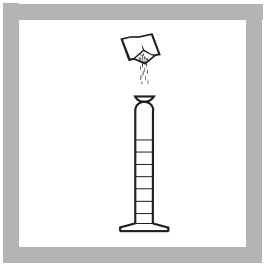
Note: *For alternate form (Al_2O_3), press* **CONC**.



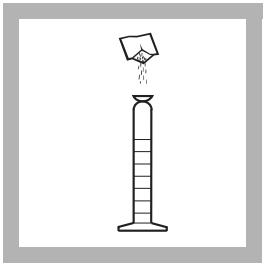
3. Fill a 50-mL graduated mixing cylinder to the 50-mL mark with sample.

Note: *Rinse cylinder with 1:1 Hydrochloric Acid and deionized water before use to avoid errors due to contaminants absorbed on the glass.*

Note: *Sample temperature must be 20-25 °C (68-77 °F) for accurate results.*



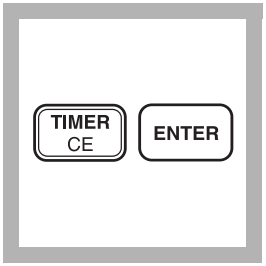
4. Add the contents of one Ascorbic Acid Powder Pillow. Stopper. Invert several times to dissolve powder.



5. Add the contents of one AluVer® 3 Aluminum Reagent Powder Pillow. Stopper.

Note: *A red-orange color develops if aluminum is present.*

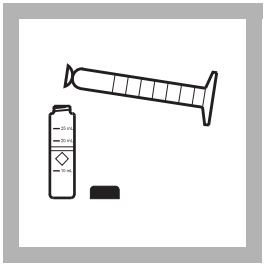
Note: *Inconsistent results will occur if any powder is undissolved.*



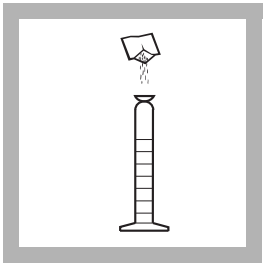
6. Press:

TIMER ENTER

A three-minute reaction period will begin. Invert the cylinder repeatedly for the three minutes.

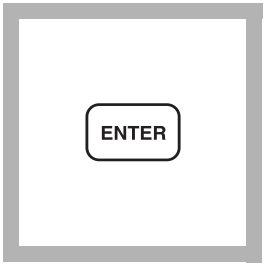


7. Pour 25 mL of mixture into a 25-mL sample cell (the prepared sample).



8. Add the contents of one Bleaching 3 Reagent Powder Pillow to the remaining 25 mL in the mixing graduated cylinder (the blank). Stopper the cylinder.

* Adapted from *Standard Methods for the Examination of Water and Wastewater*

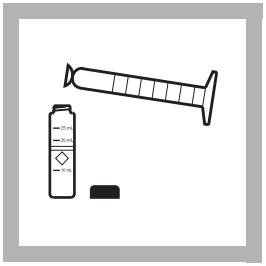


9. The display will show: **00:30 Timer 2**

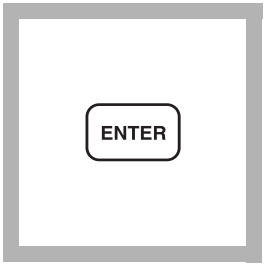
Press: **ENTER**

A thirty-second reaction period will begin. Vigorously shake the cylinder for the 30-second period.

Note: *This solution should turn a light to medium orange upon bleaching. It will not become colorless.*



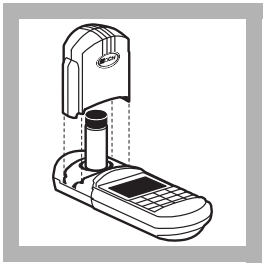
10. Pour the 25 mL of mixture in the cylinder into a second 25-mL sample cell (the blank).



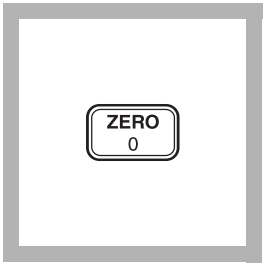
11. The display will show: **15:00 TIMER 3**

Press: **ENTER**

A 15-minute reaction period will begin.



12. Within three minutes after the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

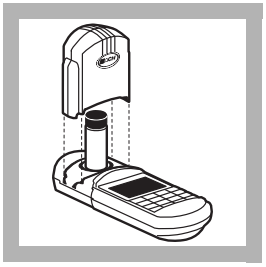


13. Press: **ZERO**

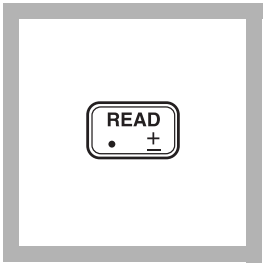
The cursor will move to the right, then the display will show:

0.000 mg/L Al

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



14. Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



15. Press: **READ**

The cursor will move to the right, then the result in mg/L aluminum will be displayed.

Note: *Clean the graduated cylinder and sample cells with soap and brush immediately following the test.*

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in a clean glass or plastic container. Preserve the sample by adjusting the pH to 2 or less with nitric acid (about 1.5 mL per liter). Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the pH to 3.5–4.5 with 5.0 N Sodium Hydroxide. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1* for more information.

Accuracy Check

Standard Additions Method

a) Snap the neck off an Aluminum Voluette Ampule Standard Solution, 50 mg/L as Al.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to three 50-mL samples. Swirl gently to mix. Also prepare a sample without any standard added (the unspiked sample).

c) Analyze each sample as described above. The aluminum concentration should increase 0.1 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

Standard Solution Method

Prepare a 0.40-mg/L aluminum standard solution by pipetting 1.00 mL of Aluminum Standard Solution, 100 mg/L as Al^{3+}, into a 250-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution immediately before use. Perform the aluminum procedure as described above. The mg/L Al reading should be 0.40 mg/L Al.

Or, using the TenSette Pipet, add 0.8 mL of solution from an Aluminum Voluette Ampule Standard Solution (50 mg/L as Al) into a 100-mL volumetric flask. Dilute to volume with deionized water. Prepare this standard immediately before testing and use as the sample.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.40 mg/L Al and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.013 mg/L Al.

Estimated Detection Limit

The estimated detection limit for program #1 is 0.013 mg/L Al. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Acidity	Acidity interferes at greater than 300 mg/L as $CaCO_3$. Treat samples with greater than 300 mg/L acidity as $CaCO_3$ as follows: **1.** Add one drop of m-Nitrophenol Indicator Solution to the sample taken in Step 3. **2.** Add one drop of 5.0 N Sodium Hydroxide Standard Solution. Stopper the cylinder. Invert to mix. Repeat as often as necessary until the color changes from colorless to yellow. **3.** Add one drop of 5.25 N Sulfuric Acid Standard Solution to change the solution from yellow back to colorless. Continue with the test.
Alkalinity	1000 mg/L as $CaCO_3$. Eliminate interferences from higher alkalinity concentrations using the following pretreatment: **1.** Add one drop of m-Nitrophenol Indicator Solution to the sample taken in Step 3. A yellow color indicates excessive alkalinity. **2.** Add one drop of 5.25 N Sulfuric Acid Standard Solution. Stopper the cylinder. Invert to mix. If the yellow color persists, repeat until the sample becomes colorless. Continue with the test.
Calcium	Does not interfere.
Fluoride	Interferes at all levels. See graph below.
Iron	Greater than 20 mg/L.
Phosphate	Greater than 50 mg/L.
Polyphosphate	Polyphosphate interferes at all levels by causing negative errors and must not be present. Before running the test, polyphosphate must be converted to orthophosphate by acid hydrolysis as described under the phosphorus procedures.

Fluoride interferes at all levels by complexing with aluminum. The actual aluminum concentration can be determined using the Fluoride Interference Graph when the fluoride concentration is known. To use the fluoride interference graph:

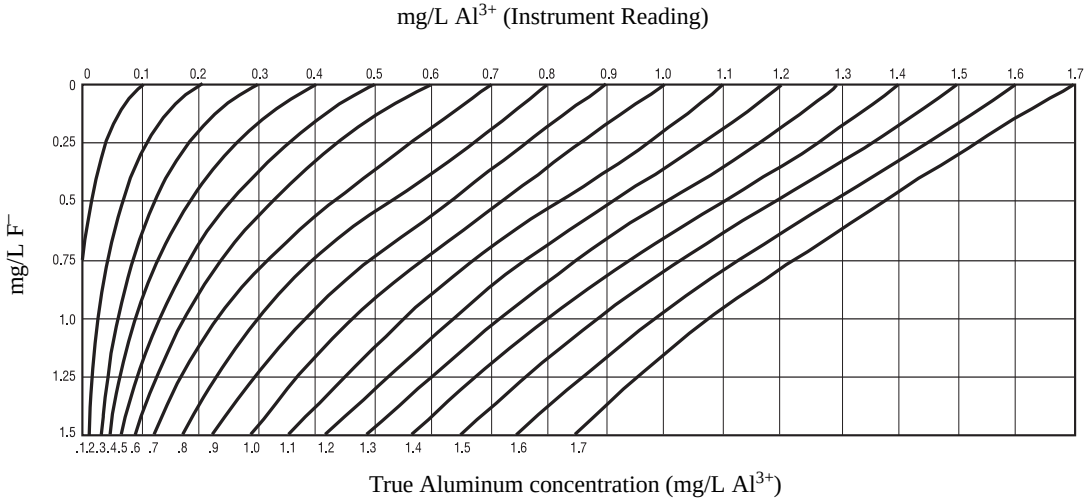
1. Select the vertical grid line along the top of the graph that represents the aluminum reading obtained in Step 15.

2. Locate the point of the vertical line (instrument reading) where it intersects with the horizontal grid line that indicates how much fluoride is present in the sample.

3. Extrapolate the true aluminum concentration by following the curved lines on either side of the intersect point down to the true aluminum concentration.

For example, if the aluminum test result was 0.7 mg/L Al^{3+} and the fluoride present in the sample was 1.0 mg/L F^-, the point where the 0.7 grid line intersects with the 1.0 mg/L F^- grid line falls between the 1.2 and 1.3 mg/L Al curves. In this case, the true aluminum content would be 1.27 mg/L.

Fluoride Interference Graph



Summary of Method

Aluminon indicator combines with aluminum in the sample to form a red-orange color. The intensity of color is proportional to the aluminum concentration. Ascorbic acid is added to remove iron interference. The AluVer 3 Aluminum Reagent, packaged in powder form shows exceptional stability and is applicable for fresh water samples.

REQUIRED REAGENTS

	Cat. No.
Aluminum Reagent Set (100 Tests)	22420-00
Includes: (1) 14290-99, (1) 14577-99, (1) 14294-49	

Description	Quantity Required Per Test	Unit	Cat. No.
AluVer 3 Aluminum Reagent Powder Pillow	1 pillow	100/pkg	14290-99
Ascorbic Acid Powder Pillow	1 pillow	100/pkg	14577-99
Bleaching 3 Reagent Powder Pillow	1 pillow	100/pkg	14294-49

REQUIRED APPARATUS

Cylinders, graduated mixing, 50 mL	1	each	1896-41
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Aluminum Standard Solution, 100 mg/L	100 mL	14174-42
Aluminum Standard Solution, Voluette ampule, 50 mg/L as Al, 10 mL	16/pkg	14792-10
Hydrochloric Acid Solution, 6N (1:1)	500 mL	884-49
m-Nitrophenol Indicator Solution, 10 g/L	100 mL	2476-32
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sodium Hydroxide Standard Solution, 5.0 N	50 mL SCDB	2450-26
Sulfuric Acid Standard Solution, 5.25 N	100 mL MDB	2449-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Brush	each	690-00
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 250 mL	each	14574-46
Fluoride Combination Electrode	each	51928-00
Fluoride ISA Powder Pillows	25/pkg	2589-99
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH/ISE Meter, *sens**ion**™2*, portable	each	51725-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, Volumetric, Class A, 1.00 mL	each	14515-35
Thermometer, –20 to 110 °C, non-mercury	each	26357-02

For Technical Assistance, Price and Ordering

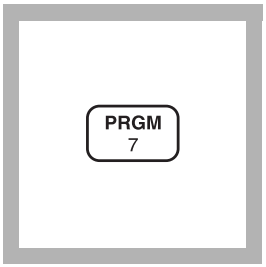
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

BENZOTRIAZOLE (0 to16.0 mg/L) or TOLYLTRIAZOLE (0 to 16.0 mg/L)

UV Photolysis Method*

For cooling or boiler water



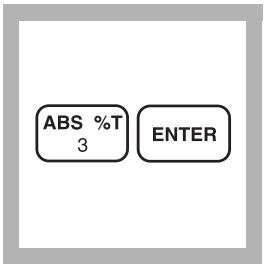
1. Enter the stored program number for benzotriazole (Benzo) or tolyltriazole (Toly).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



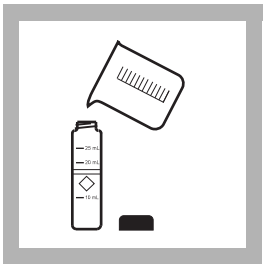
2. Press: **3 ENTER** for either triazole test.

The display will show **mg/L, BENZO,** and the **ZERO** icon

or

the display will show **mg/L, TOLY,** and the **ZERO** icon.

Press the **CONC** key to choose the desired triazole.



3. Fill a sample cell with 25 mL of sample.

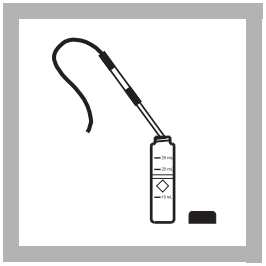
Note: *Sample temperature should be between 20-25 °C (68-77 °F).*

Note: *If sample contains nitrite or borax (sodium borate), adjust the pH to between 4 and 6 with 1 N sulfuric acid.*

4. Add the contents of one Triazole Reagent Powder Pillow. Swirl to dissolve completely.

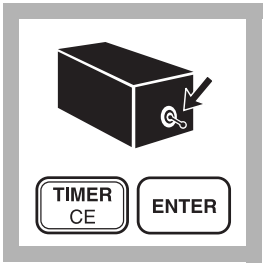
Note: *If the sample contains more than 500 mg/L hardness (as $CaCO_3$), add 10 drops of Rochelle Salt Solution.*

* Adapted from Harp, D., Proceedings 45th International Water Conference, 299 (October 22-24, 1984)



5. Insert the ultraviolet lamp into the sample cell.

Note: *UV safety goggles should be worn while the lamp is on.*

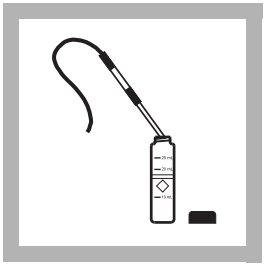


6. Turn the UV lamp ON and press:

TIMER ENTER

A five-minute reaction period will begin.

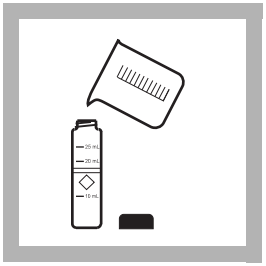
Note: *A yellow color will form if triazole is present.*



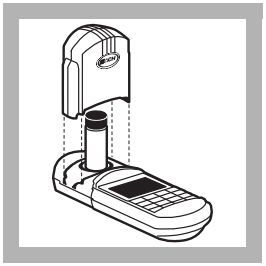
7. When the timer beeps, turn the lamp off and remove it from the cell (the prepared sample). Swirl the cell to mix thoroughly.

Note: *Low results will occur if photolysis (lamp ON) takes place for more or less than five minutes.*

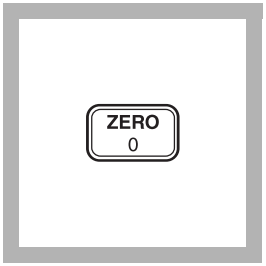
Note: *Avoid handling the quartz surface of the lamp. Rinse the lamp and wipe with a soft, clean tissue between tests.*



8. Fill another sample cell with 25 mL of sample (the blank).



9. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **ZERO**

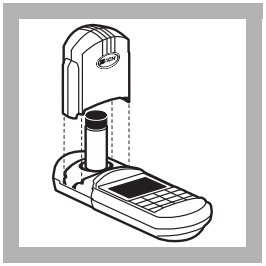
The cursor will move to the right, then the display will show:

0.0 mg/L Benzo

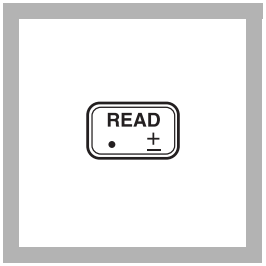
or

0.0 mg/L Toly

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L benzotriazole or tolyltriazole will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling And Storage

The most reliable results are obtained when samples are analyzed as soon as possible after collection.

Accuracy Check

Standard Additions Method

a) Use the TenSette pipet to add 0.1, 0.2 and 0.3 mL of 500-mg/L Benzotriazole Standard Solution to three 25-mL samples. Perform the test according to the above procedure.

Note: *The test will not distinguish between benzotriazole and tolyltriazole.*

b) Each addition of 0.1 mL of standard solution should increase the benzotriazole reading by 2 mg/L over the reading of an unspiked sample.

c) If these increases are not obtained see *Standard Additions* in *Section 1* for more information.

UV Lamp Check

To verify the ultraviolet lamp (normal life equals 5000 hours) is working properly, perform the following test:

a) Prepare a 5.0 mg/L benzotriazole standard solution by pipetting 10.0 mL of Benzotriazole Standard Solution, 500 mg/L benzotriazole, into a 1000-mL volumetric flask. Dilute to volume.

b) Analyze according to the above procedure. If the result is significantly below 5.0 mg/L, replace the lamp.

Method Performance

Precision

In a single laboratory using a standard solution of 9.0 mg/L triazole and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.21 mg/L benzotriazole and ±0.20 mg/L tolyltriazole.

Estimated Detection Limit

The estimated detection limit for program 3 is 0.7 mg/L benzotriazole or tolyltriazole. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Acrylates (as methyl acrylate)	50 mg/L
Alum	400 mg/L
Borate (as sodium tetraborate)	4000 mg/L
Chlorine (as Cl_2)	20 mg/L
Chromium (as chromate)	12 mg/L
Copper	10 mg/L
Hardness	500 mg/L as $CaCO_3$
Iron	20 mg/L
Lignosulfonates	40 mg/L
Magnesium	300 mg/L as $CaCO_3$
Molybdenum (as molybdate)	200 mg/L
Nitrite	4000 mg/L
Phosphonates (AMP or HEDP)	100 mg/L
Sulfate	200 mg/L
Zinc	80 mg/L

Strong oxidizing or reducing agents present in the sample will interfere directly with the test.

Summary of Method

Benzotriazole or tolyltriazole, used in many applications as corrosion inhibitors for copper and copper alloys, are determined by a proprietary catalytic ultraviolet (UV) photolysis procedure requiring less than 10 minutes to perform.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Triazole Reagent Powder Pillows	1 pillow	100/pkg	21412-99

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
Select one based on available voltage:			
Lamp, UV, with power supply, 115 V, 60 Hz, with goggles	1	each	20828-00
Lamp, UV, with power supply, 230 V, 50 Hz, with goggles	1	each	20828-02

OPTIONAL REAGENTS

Benzotriazole Standard Solution, 500 mg/L		100 mL	21413-42
Rochelle Salt Solution		29 mL* DB	1725-33
Sulfuric Acid Standard Solution, 1.00 N		100 mL MDB	1270-32
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Flask, volumetric, Class A, 1000 mL		each	14574-53
Lamp, UV (lamp only)		each	26710-00
pH Paper, 1 to 11 pH		5 rolls/pkg	391-33
pH Meter, *sens**ion***™***1***, portable with electrode		each	51700-10
Pipet Filler, safety bulb		each	14651-00
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
Pipet, volumetric, 10.0 mL, Class A		each	14515-38
Safety Goggles, UV		each	21134-00
Stopwatch		each	14645-00
Thermometer, –20 to 110 °C, non-mercury		each	26357-02
Timer, interval, 1 second to 99 hours		each	23480-00

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

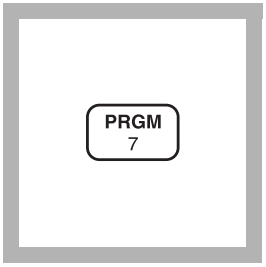
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes.

BROMINE (0 to 4.50 mg/L)

DPD Method* (Powder Pillows or AccuVac Ampuls)

Using Powder Pillows



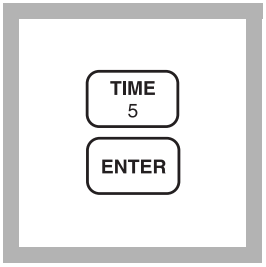
1. Enter the stored program number for bromine (Br_2)-powder pillows.

Press: **PRGM**

The display will show:

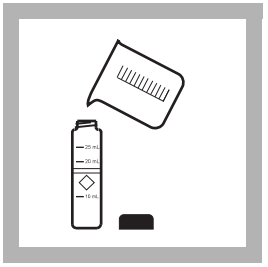
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



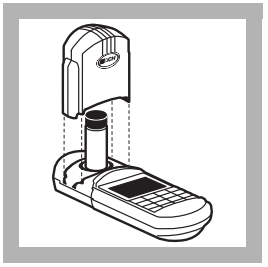
2. Press: **5 ENTER**

The display will show **mg/L, Br2** and the **ZERO** icon**.**



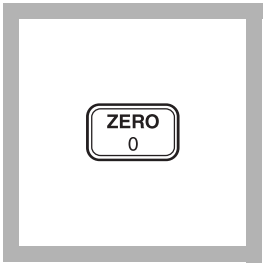
3. Fill a sample cell with 10 mL of sample (the blank).

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

* Adapted from *Standard Methods for the Examination of Water and Wastewater*

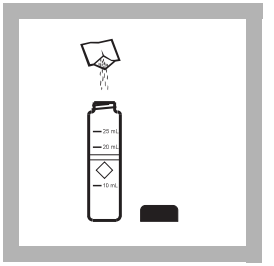


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

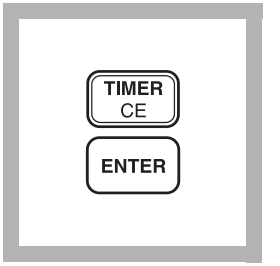
0.00 mg/L Br2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



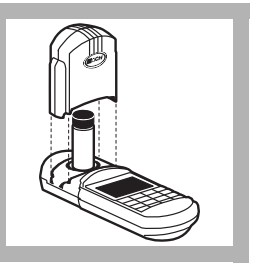
6. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and swirl vigorously to dissolve the powder.

Note: *It is not necessary that all the powder dissolves. A pink color will develop if bromine is present.*

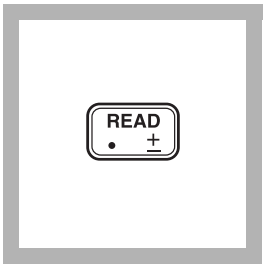


7. Press:**TIMER ENTER**

A three-minute reaction period will begin.



8. When the timer beeps, place the sample into the cell holder. Tightly cover the sample cell with the instrument cap.



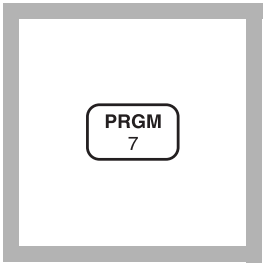
9. Press: **READ**

The cursor will move to the right, then the result in mg/L bromine will be displayed.

Note: *If samples temporarily turn yellow after reagent addition, or the display flashes "limit", it is due to high bromine levels. Dilute fresh samples and repeat the test. A slight loss of bromine may occur during dilution. Multiply results by the dilution factor; see Section 1.*

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Using AccuVac Ampuls



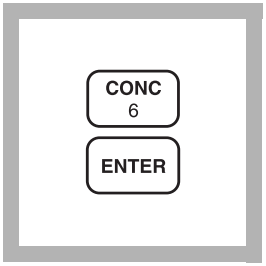
1. Enter the stored program number for bromine (Br_2) AccuVac Ampuls.

Press: **PRGM**

The display will show:

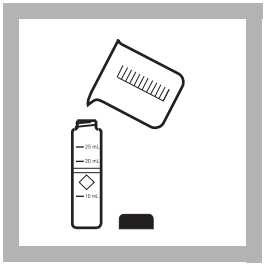
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



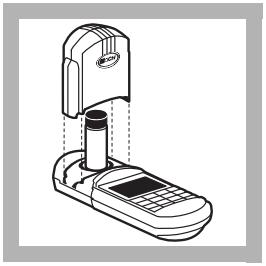
2. Press: **6 ENTER**

The display will show **mg/L, Br2** and the **ZERO** icon**.**

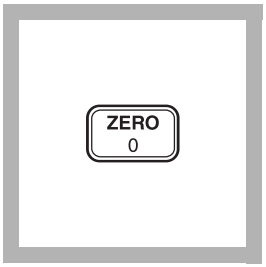


3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



4. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

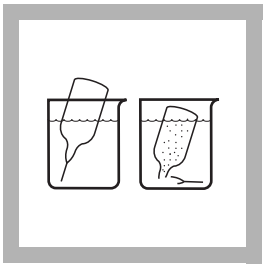


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

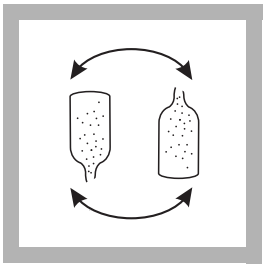
0.00 mg/L Br2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



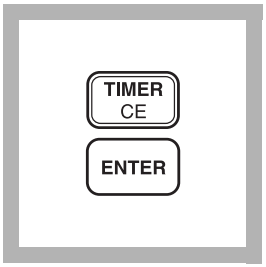
6. Fill one DPD Total Chlorine Reagent AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*



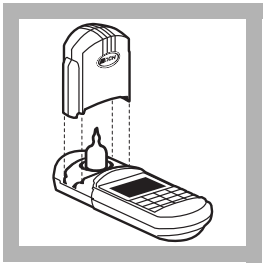
7. Quickly invert the ampule several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will form if bromine is present.*

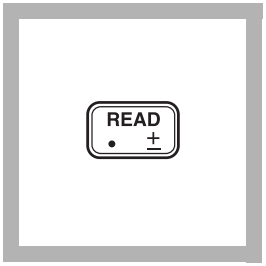


8. Press: **TIMER ENTER**

A three-minute reaction period will begin.



9. After the timer beeps, place the AccuVac ampul into the cell holder. Tightly cover the ampule with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L bromine will be displayed.

Note: *If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high bromine levels. Dilute a fresh sample and repeat the test. A slight loss of bromine may occur during dilution. Multiply the result by the dilution factor; see Section 1.*

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Analyze samples for bromine **immediately** after collection.

Avoid plastic containers since these may have a large bromine demand. **Pretreat glass** sample containers to remove any bromine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for bromine is introduced when a representative sample is not obtained. If sampling from a tap, let the sample flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark.

Perform the bromine analysis immediately after collection.

Accuracy Check

Standard Additions Method (using powder pillows)

a) Snap the top off a LR Chlorine PourRite® Ampule Standard Solution.

b) Use a TenSette Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.

c) Re-zero the instrument using the original sample (the blank).

d) Place the spiked sample in the cell holder and press **READ**. Record the result.

e) Calculate the equivalent concentration of mg/L bromine added to the sample:

$$\text{mg/L Bromine added} = \frac{0.1\ (\text{vol. standard added}) \times \text{Label value (mg/L Chlorine)} \times 2.25}{10.1\ (\text{sample + standard volume})}$$

f) The spiked sample result (step d) should reflect the analyzed sample result + the calculated mg/L Br_2 added (step e).

g) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method (using AccuVac Ampuls)

a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.

b) Use a graduated cylinder to measure 25 mL of sample into each of two beakers.

c) Use a TenSette Pipet to add 0.2 mL of the standard to one of the beakers (this is the spiked sample). Swirl to mix.

d) Fill a DPD Total Chlorine AccuVac completely from each beaker.

e) Analyze the spiked and unspiked sample as described in the procedure.

f) Calculate the equivalent concentration of mg/L bromine added to the sample:

$$\text{mg/L Bromine added} = \frac{0.2\ (\text{vol. standard added}) \times \text{Label value (mg/L Chlorine)} \times 2.25}{25.2\ (\text{sample + standard volume})}$$

g) The spiked sample result should reflect the analyzed sample result + the calculated mg/L Br_2 added (step f).

h) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 2.34 mg/L bromine and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.02 mg/L bromine.

In a single laboratory using a standard solution of 2.31 mg/L bromine and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation ± 0.02 mg/L bromine.

Estimated Detection Limit

The estimated detection limit for program 5 is 0.04 mg/L Br_2 and 0.03 mg/L Br_2 for program 6. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Chlorine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$
Iodine	Interferes at all levels
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium , Oxidized (Cr^{6+})	**1.** Adjust sample pH to 6-7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait 1 minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct bromine concentration.
Monochloramine	Interferes at all levels
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See *Interferences, Section 1*.

Summary of Method

Bromine reacts with DPD (N,N-diethyl-p-phenylenediamine) to form a magenta color which is proportional to the total bromine concentration.

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interference will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS (USING POWDER PILLOWS)

Description	Quantity Required Per Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows	1 pillow	100/pkg	21056-69

REQUIRED REAGENTS (USING ACCUVAC AMPULS)

DPD Total Chlorine Reagent AccuVac Ampuls	1 ampule	25/pkg	25030-25

REQUIRED APPARATUS (USING POWDER PILLOWS)

Sample Cells, 10-20-25-mL, w/ cap		6/pkg	24019-06

REQUIRED APPARATUS (USING ACCUVAC AMPULS)

Beaker, 50 mL	1	each	500-41

OPTIONAL REAGENTS

Chlorine Standard Solution, PourRite ampule, 25-30 mg/L, 2 mL		20/pkg	26300-20
DPD Total Chlorine Reagent, SwifTest		250 Tests	28024-00
Potassium Iodide Solution, 30 g/L		100 mL* MDB	343-32
Sodium Arsenite, 5 g/L		100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1.000 N		100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1 N		100 mL* MDB	1270-32
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

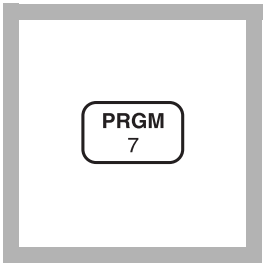
AccuVac Snapper Kit		each	24052-00
PourRite Ampule Breaker		each	24846-00
Cylinder, graduated, 25 mL		each	508-40
pH Meter, *sens**ion***™*1*, portable		each	51700-00
pH Indicator Paper, 1 to 11 pH units		5 rolls/pkg	391-33
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

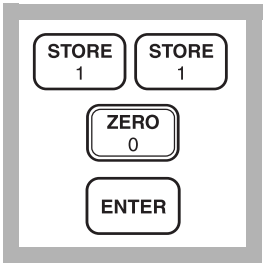
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes



1. Enter the user program number for monochloramine.

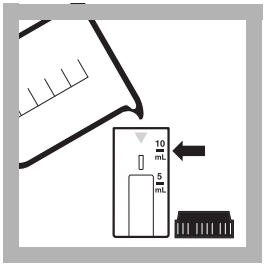
Press: **PRGM**
The display will show:
PRGM?



2. Press:
110 ENTER

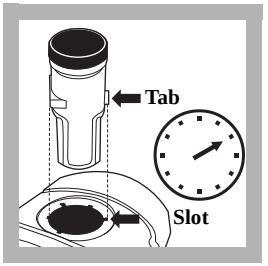
The display will show
mg/L Cl_2
then: **ZERO**

Note: *For alternate forms, press the* **CONC** *key.*



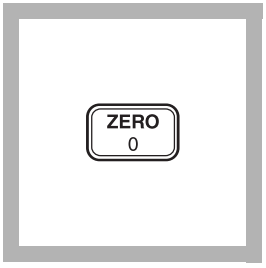
3. Fill the 10-mL/1-cm cell to the 10-mL line with sample.

Note: *For the most accurate results, determine a reagent blank for each new lot of reagent by running the test using deionized water instead of sample.*



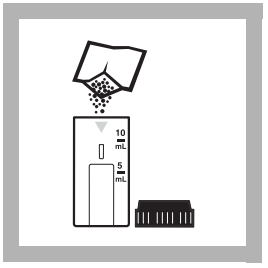
4. Place the cell into the instrument. Tightly cover the sample cell with the instrument cap.

Note: *Place the cell into the cell holder as illustrated. The cell's tab should be at the 2 o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.*

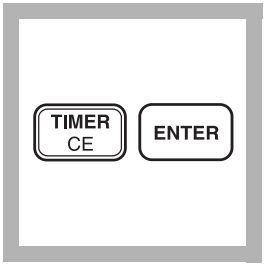


5. Press: **ZERO**

The cursor will move to the right, then the display will show:
0.00 mg/L Cl_2



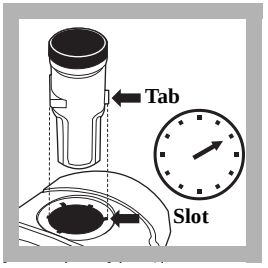
6. Remove the cell from the cell holder and add the contents of one pillow of Monochlor–F to the sample. Cap and shake the cell about 20 seconds to dissolve.



7. Press:
TIMER ENTER

A 5-minute reaction period will begin.

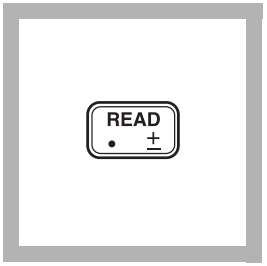
Note: *The color development time depends on the sample temperature. Refer to Table 3 for the actual time required.*



8. After the timer beeps, place the cell into the instrument. Tightly cover the sample cell with the instrument cap.

Note: *Place the cell into the cell holder as illustrated. The cell's tab should be at the 2-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.*

* Patent pending



9. Press: **READ**

The cursor will move to the right, then the result in mg/L monochloramine (as Cl_2 or chosen units) will be displayed.

Sampling and Storage

Analyze samples for monochloramine immediately after collection. If sampling with the sample cell, rinse the sample cell several times with the sample, then carefully fill to the 10-mL mark. If sampling from a tap, let the water flow for at least 5 minutes. Let the container overflow with the sample several times, then cap the container so there is no headspace (air) above the sample.

Accuracy Check

1. Prepare the following monochloramine standard fresh before use.

2. Add the contents of one Buffer Powder Pillow, pH 8.3 to about 50-mL of organic-free water in a clean 100-mL Class A volumetric flask. Swirl to dissolve the powder.

3. Using a Class A volumetric pipet, transfer 2.00 mL of Nitrogen, Ammonia Standard Solution, 100 mg/L as NH_3–N into the flask.

4. Dilute to volume with organic-free water, cap and mix thoroughly. This is a 2.00 mg/L buffered ammonia standard.

5. Pipet 50.00 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a stir bar.

6. Obtain a recent lot of Chlorine Solution Ampules, 50–70 mg/L, and note the actual free chlorine concentration for this lot.

7. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

8. Open an ampule and, using a glass Mohr pipet, add the calculated amount of Chlorine Solution slowly to the ammonia standard, while mixing at medium speed on a stir-plate.

9. Allow the monochloramine solution to mix for 1 minute after all Chlorine Solution is added.

10. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water, cap, and mix thoroughly. This is a nominal 4.5 mg/L (as Cl_2) monochloramine standard.

Use this standard within 1 hour of preparation.

Method Performance

Precision

In a single laboratory, using a monochloramine standard solution of 2.10 mg/L Cl_2 and representative lots of reagent, a single operator obtained a standard deviation of ±0.12 mg/L Cl_2.

Estimated Detection Limit

The estimated detection limit for Method 10171 is 0.05 mg/L Cl_2. For more information on the estimated detection limit, see *Section 1* of the *Procedure Manual*.

Interferences

The following have been tested for interference and found *not* to interfere up to the indicated levels:

Table 9 Non-interfering Substances

Substance	Maximum Level Tested
Alanine	1 mg/L N
Aluminum	10 mg/L
Bromide	100 mg/L Br^-

Table 9 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Bromine	15 mg/L Br_2
Calcium	1000 mg/L $CaCO_3$
Chloride	18,000 mg/L
Chlorine Dioxide	5 mg/L ClO_2
Chromium (III)	5 mg/L
Copper	10 mg/L
Cyanide	10 mg/L CN^-
Free chlorine	10 mg/L Cl_2
Glycine	1 mg/L N
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Lead	10 mg/L
Nitrate	100 mg/L as N
Nitrite	50 mg/L N
Phosphate	100 mg/L PO_4^{3-}
Silica	100 mg/L SiO_2
Silver	10 mg/L
Sulfate	2600 mg/L
Sulfite	50 mg/L SO_3^{2-}
Tyrosine	1 mg/L N
Urea	10 mg/L N
Zinc	5 mg/L

Table 10 Interfering Substances

Interfering Substance and its effect		Interference Level	Recommended Treatment
Magnesium	+	Above 400 mg/L $CaCO_3$	Add 5 drops Rochelle Salt Solution prior to testing.
Manganese (+7)	–	Above 3 mg/L	
Ozone	–	Above 1 mg/L	Usually doesn't coexist with monochloramine.
Sulfide	+	Turns a "rust" color if present.	Usually doesn't coexist with monochloramine.
Thiocyanate	–	Above 0.5 mg/L	

Summary of Method

In the presence of a cyanoferrate catalyst, monochloramine in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate couples with excess substituted phenol to form a green-colored indophenol, which is proportional to the amount of monochloramine present in the sample.

Sample Temperature		Minutes
° C	° F	
5	40	10
7	42	9
9	48	8
10	50	8
12	54	7
14	58	7
16	61	6
18	68	4
20	73	3
23	75	2.5
25	77	2
>25	>77	2

Instrument Setup

This procedure will add the current method as a new Hach program to your DR/850 or DR/890.

1. Turn on the instrument by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key until the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.
6. Enter each of the numbers in the right column, each followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time, you may use the arrow keys to scroll back to review or change a number already entered.

Line Number	Entry	Line Number	Entry
1	110	29	108
2	42	30	78
3	74	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	63
8	0	36	57
9	0	37	199
10	0	38	104
11	0	39	62
12	64	40	74
13	176	41	61
14	120	42	45
15	106	43	1
16	0	44	204
17	0	45	0
18	0	46	5
19	0	47	10
20	67	48	1
21	108	49	44
22	50	50	0
23	0	51	0
24	0	52	0
25	78	53	0
26	72	54	3
27	50	55	0
28	67	56	255

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Monochlor F Reagent Pillows	1	50/pkg	28022-46

REQUIRED APPARATUS

Sample Cell, 10-mL/1-cm	1	2/pkg	48643-02
Clippers, shears	1	each	23694-00

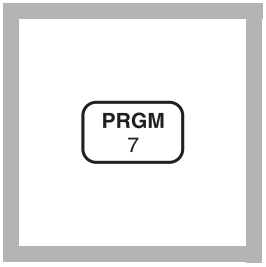
OPTIONAL REAGENTS

Rochelle Salt Solution		29-mL DB	1725-33
Organic-Free Water		500-mL	26415-49
Buffer Powder Pillows, pH 8.3		25/pkg	898-68
Nitrogen, Ammonia Standard Solution, 100 mg/L as NH_3-N		500-mL	24065-49
Chlorine Solution Voluette Ampule, 50–75 mg/L		16/pkg	14268-10

OPTIONAL APPARATUS

Beaker, 100-mL		each	500-42H
Flask, Volumetric, Class A, 100-mL		each	14574-42
Pipet, Mohr, Glass, 10-mL		each	20934-38
Pipet, Volumetric, Class A, 2.00 mL		each	14515-36
Pipet, Volumetric, Class A, 50.00 mL		each	14515-41
Stir Bar, Octagonal		each	20953-52
Stirrer, Magnetic, 110 V, 4” x 4”		each	28812-00

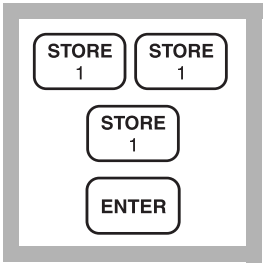
Indophenol Method* **For chlorinated drinking water and chlorinated wastewater**



1. Enter the user program number for Chloramine, HR.

Press: **PRGM**
The display will show:
PRGM?

Note: *For most accurate results, perform a Reagent Blank Correction (Section 1 of the DR/800 Instrument Manual).*



2. Press:
111 ENTER

The display will show:
mg/L Cl_2

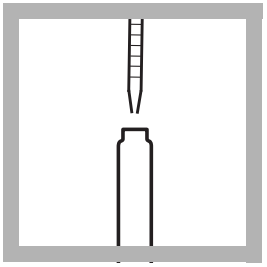
and then

Zero

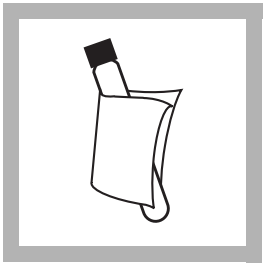
Note: *For alternate forms, press the* **CONC** *key.*

3. Insert the COD/TNT Vial Adapter into the cell holder by rotating the adapter until it drops in place. Push down to fully insert it.

Note: *For better performance, adiffuser band covers the light path holes on the adapter. Do not remove the band.*

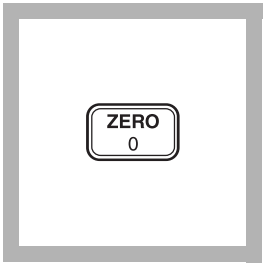


4. Remove the cap from one HR Monochloramine Diluent vial. Use a glass pipet to add 2.0 mL of sample to the vial. Re-cap and invert several times to mix.



5. Wipe the outside of the vial clean.

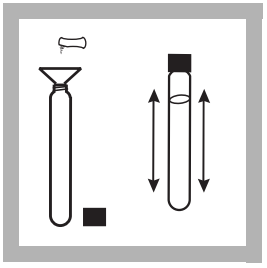
Place the vial into the adapter. Cover the sample vial tightly with the instrument cap.



6. Press: **ZERO**

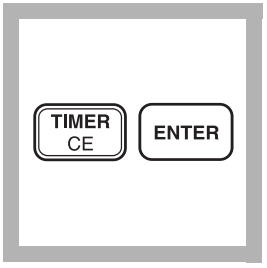
The cursor will move to the right and the display will show:

0.0 mg/L Cl_2.



7. Remove the vial from the cell holder, uncap, and add the contents of one Monochlor–F pillow to the sample. Cap and shake the vial about 20 seconds to dissolve.

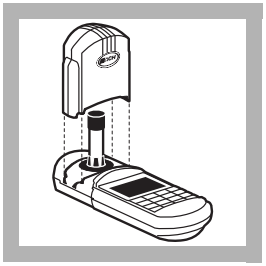
Note: *Use the microfunnel as an aid in adding reagent powder to the vial.*



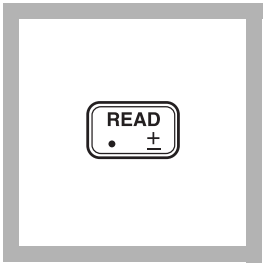
8. Press:
TIMER ENTER

A five-minute reaction period will begin.

* U.S. Patent 6,315,950



9. After the timer beeps, wipe the prepared vial and place it into the instrument. Cover the sample vial tightly with the instrument cap.



10. Press: **READ**.

The cursor will move to the right , then the results in mg/L monochloramine (as Cl_2) will be displayed.

Sampling and Storage

Analyze samples for monochloramine immediately after collection. Rinse the sample container several times with the sample water allowing it to overflow each time. If sampling from a tap, let the water flow for at least 5 minutes. Cap the container so that there is no head space (air) above the sample.

Accuracy Check

Prepare the following monochloramine standard fresh before use:

1. Using a clean 100-mL Class A volumetric flask, add the contents of one Buffer Powder Pillow, pH 8.3, to approximately 50 mL of organic-free water. Swirl to dissolve the powder.

2. Use a Class A volumetric pipet to transfer 2.00 mL of Nitrogen Ammonia Standard Solution, 100-mg/L as NH_3–N, into a flask.

3. Dilute to volume with organic-free water. Cap and mix thoroughly. This is the 2.00-mg/L buffered ammonia standard.

4. Pipet 50.00 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a magnetic stir bar and place the beaker on a stir plate.

5. Note the free chlorine concentration for the Chlorine Solution Ampules, 50–70 mg/L. Use ampules from a recent lot.

6. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

7. Turn the stir plate on to medium speed.

8. Open an ampule. Use a glass Mohr pipet to add the calculated amount of Chlorine Solution slowly to the ammonia standard while it is mixing.

9. Allow the monochloramine solution to mix for 1 minute after all the Chlorine Solution is added.

10. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water. Cap and mix thoroughly. This is a nominal 4.5-mg/L (as Cl_2) monochloramine standard.

Use this solution within 1 hour of preparation.

Method Performance

Precision

In a single laboratory, using a standard solution of 3.5 mg/L monochloramine as chlorine and two representative lots of reagent, a single operator obtained a standard deviation of ± 0.2 mg/L Cl_2.

Estimated Detection Limit

The estimated detection limit (EDL) for Method 10172 is 0.2 mg/L Cl_2. For more information on the EDL, see *Section 1* of the DR/800 Procedure Manual.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 11 Non-interfering Substances

Substance	Maximum Level Tested
Alanine	1 mg/L N
Aluminum	10 mg/L
Bromide	100 mg/L Br^-
Bromine	15 mg/L Br_2
Calcium	1000 mg/L as $CaCO_3$

Table 11 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Chloride	18,000 mg/L
Chlorine Dioxide	5 mg/L ClO_2
Chromium (III)	5 mg/L
Copper	10 mg/L
Cyanide	10 mg/L CN^-
Free Chlorine	10 mg/L Cl_2
Glycine	1 mg/L N
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	1000 mg/L as $CaCO_3$
Manganese (VII)	10 mg/L
Lead	10 mg/L
Nitrate	100 mg/L N
Nitrite	50 mg/L N
Phosphate	100 mg/L PO_4
Silica	100 mg/L SiO_2
Silver	10 mg/L
Sulfate	2600 mg/L
Sulfite	50 mg/L SO_3^{2-}
Tyrosine	1 mg/L as N
Urea	10 mg/L as N
Zinc	5 mg/L

Table 12 Interfering Substances

Interfering Substance and its effect		Interference Level	Recommended Treatment
Ozone	–	Above 1 mg/L	Usually doesn't coexist with monochloramine
Sulfide	+	Turns a "rust" color if present.	Usually doesn't coexist with monochloramine
Thiocyanate	–	Above 0.5 mg/L	

Summary of Method

The sample is first diluted in a Test 'N Tube™. In the presence of a cyanoferrate catalyst, monochloramine (NH_2Cl) in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate compound couples with excess substituted phenol to form a green indophenol. Color intensity is proportional to the amount of monochloramine present in the sample.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet (MSDS) for information specific to the reagent used.

Instrument Setup

This procedure will add the current method as a new Hach program to your DR/850 or DR/890 instrument.

1. Turn the instrument on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key until the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Key in "8138", then press **ENTER**.
6. Key the number in the "Enter" column corresponding to line number 1 on the display. Press **ENTER**. Repeat for lines 2–56 on the display.

Table 13

Line number on display	Enter	Line number on display	Enter
1	111	29	108
2	42	30	78
3	73	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	63
8	0	36	58
9	0	37	61
10	0	38	112

Table 13 (Continued)

11	0	39	62
12	65	40	74
13	116	41	61
14	49	42	112
15	248	43	0
16	0	44	110
17	0	45	0
18	0	46	0
19	0	47	10
20	67	48	1
21	108	49	44
22	50	50	0
23	0	51	0
24	0	52	0
25	78	53	0
26	72	54	153
27	50	55	0
28	67	56	255

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
HR Monochloramine Test 'N Tubes, 50 tests			28051-45
Includes:			
HR Monochloramine Diluent Vials	50		*
Funnel, micro	1	each	25843-35
Monochlor F Reagent Pillows	1	50/pkg	28022-46

REQUIRED APPARATUS

COD/TNT Vial Adapter, DR/800	1	each	48464-00
Pipet, Mohr, glass, 2.00-mL	1	each	20936-36
Test Tube Rack	1	each	18641-00

OPTIONAL REAGENTS

Organic-free Water	500-mL		26415-49
Buffer Powder Pillows, pH 8.3	25/pkg		898-68
Nitrogen, Ammonia Standard Solution, 100-mg/L as NH_3–N	500-mL		24065-49
Chlorine Solution Voluette® Ampule, 50–75 mg/L, 10-mL	16/pkg		14268-10

OPTIONAL APPARATUS

Beaker, 100-mL		each	500-42H
Clippers (medium powder pillows)		each	968-00
Clippers (shears)		each	23694-00
Flask, Volumetric, Class A, 100-mL		each	14574-42
Pipet, Mohr, Glass, 10-mL		each	20934-38
Pipet, Volumetric, Class A, 2.00-mL		each	14515-36
Pipet, Volumetric, Class A, 50.00-mL		each	14515-41
Stir Bar, Octagonal		each	20953-52
Stirrer, Magnetic, 110 V, 4" x 4"		each	23436-00

* Not sold separately.

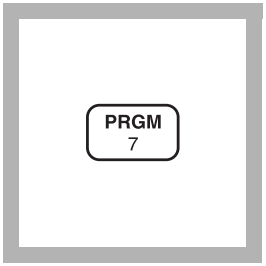
CHLORINE DIOXIDE (0 to 5.00 mg/L) Method 10126

DPD Method* **For water**

USEPA accepted for reporting for drinking water analysis

Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*

Using Powder Pillows

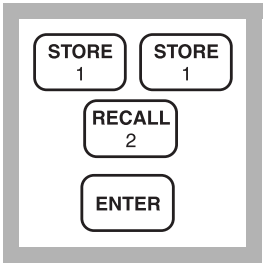


1. Enter the stored program number for chlorine dioxide (ClO_2) powder pillows.

Press: **PRGM**

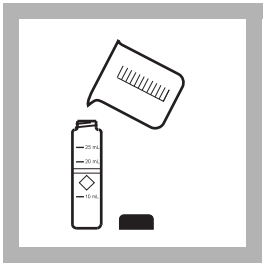
The display will show:

PRGM ?



2. Press: **112 ENTER**

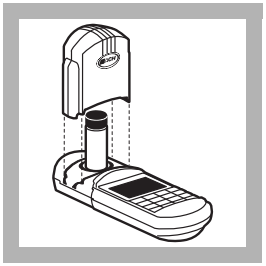
The display will show **mg/L, ClO2**, and the **ZERO** icon.



3. Fill a sample cell with 10 mL of sample (the blank).

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

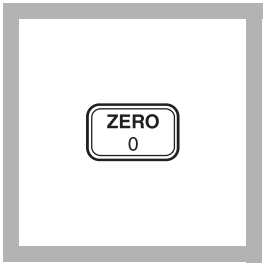
Note: *Wipe off any liquid or fingerprints before inserting the sample cell into the instrument.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *For best results, run a reagent blank using deionized water as the sample. Subtract the blank value from the sample reading to obtain the final result. See* Reagent Blank Correction *in Section 1 of the DR/800 Procedure Manual.*

* Procedure is equivalent to *Standard Method 4500, $Cl0_2P$*

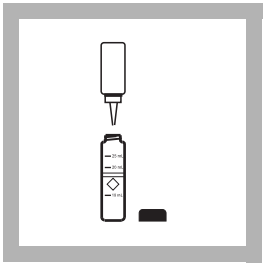


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L ClO2

Note: *If Reagent Blank Correction is on, the display may flash* **"limit"**. *See Section 1 of the DR/800 Procedures Manual.*

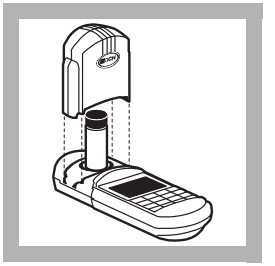


6. Add four drops of Glycine Reagent to the sample cell. Swirl to mix.

7. Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and swirl to mix.

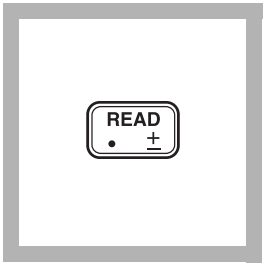
Note: *A pink color will develop if free chlorine dioxide is present.*

Note: *Perform step 9 within one minute of reagent addition.*



8. Allow 30 seconds for undissolved powder to settle. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Wipe off any liquid or fingerprints before inserting the sample cell into the instrument.*

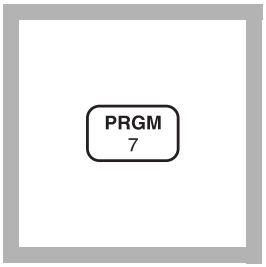


9. Press: **READ**

The cursor will move to the right, then the result in mg/L chlorine dioxide will be displayed.

Note: *If the sample temporarily turns yellow after reagent addition, or the display flashes* **"limit"**, *it is due to high chlorine dioxide levels. Dilute a fresh sample with chlorine dioxide-free water and repeat the test. A slight loss of chlorine dioxide may occur during dilution. Multiply the result by the dilution factor.*

Using AccuVac® Ampuls

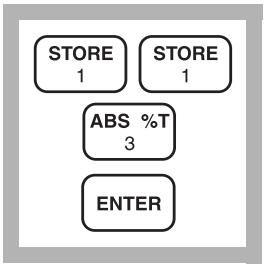


1. Enter the stored program number for chlorine dioxide (ClO_2) AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?



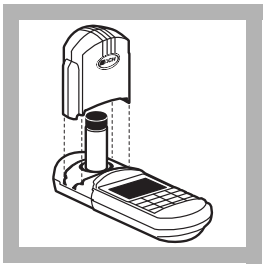
2. Press: **113 ENTER**

The display will show **mg/L, ClO2** and the **ZERO** icon.

3. Fill a sample cell with at least 10 mL of sample (the blank). Fill a 50-mL beaker with 40 mL of sample. Using the correct sample volume is important.

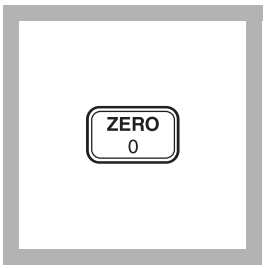
Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

Note: *Wipe off any liquid or fingerprints before inserting the sample cell into the instrument.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *For best results, run a reagent blank using deionized water as the sample. Subtract the blank value from the sample reading to obtain the final result. See* Reagent Blank Correction *in Section 1 of the DR/800 Procedure Manual.*

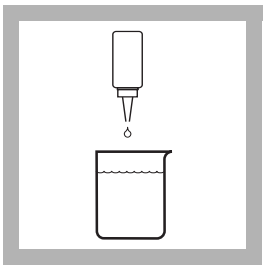


5. Press: **ZERO**

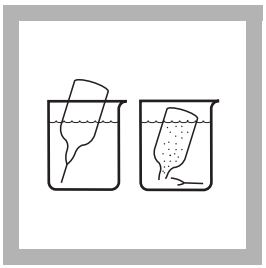
The cursor will move to the right, then the display will show:

0.00 mg/L ClO2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1 of the DR/800 Procedures Manual.*



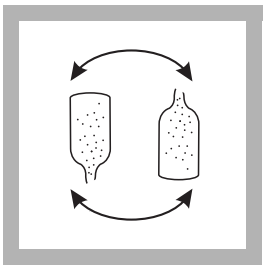
6. Add 16 drops of Glycine Reagent to the sample in the beaker. Swirl to mix.



7. Fill a DPD Free Chlorine Reagent AccuVac Ampul with sample.

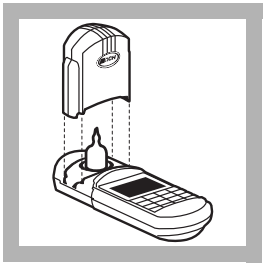
Note: *Keep the tip immersed while the ampul fills completely.*

Note: *Perform step 10 within one minute of reagent addition.*

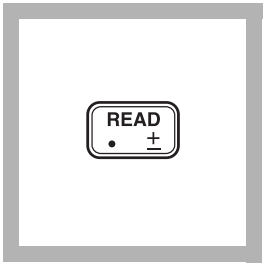


8. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will form if chlorine dioxide is present.*



9. Allow 30 seconds for undissolved powder to settle. Place the AccuVac Ampul into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L chlorine dioxide will be displayed.

Note: *If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high chlorine dioxide levels. Dilute a fresh sample with chlorine dioxide-free water and repeat the test. A slight loss of chlorine dioxide may occur during dilution. Multiply the result by the dilution factor.*

Sampling and Storage

Analyze samples for chlorine dioxide **immediately** after collection. Chlorine dioxide is a strong oxidizing agent, and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of chlorine dioxide in water.

Avoid plastic containers since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine dioxide demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for chlorine dioxide is introduced when a representative sample is not obtained. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the analysis immediately.

Accuracy Check

Because chlorine dioxide is difficult and hazardous to produce, check the DPD and glycine reagents by using chlorine standards. Proceed as follows:

1. Prepare a 1-mg/L free chlorine standard.

Method 1

a. Obtain Free Chlorine Standards, (Cat. No. 14268-10).

b. Determine the concentration of the standard from the certificate of analysis shipped with the standard (50-75 mg/L). Calculate the volume of standard needed as follows:

mL standard needed = 100 ÷ standard concentration

c. Pipet the volume of standard needed into a 100-mL volumetric flask. Dilute to the line with chlorine demand-free deionized water. Invert to mix.

Method 2

a. Dilute 1 drop of commercial 5% chlorine bleach in 1 liter of chlorine demand-free deionized water. Use this as the standard.

2. Verify the standard's concentration using the Hach Free Chlorine Method, #8021.

3. Perform the chlorine dioxide test on the standard without adding glycine (*step 6*).

4. The chlorine dioxide reading should be about 2.45 times greater than the chlorine result. If so, this verifies the DPD and the instrument are functioning properly.

5. Repeat the chlorine dioxide test on the chlorine standard, including the glycine addition (*step 6*). The reading should be less than 0.10 mg/L. This verifies that the glycine is eliminating free chlorine interference.

Method Performance

Precision

Program	Standard	95% Confidence Limits
112	0.24 mg/L	0.22–0.26 mg/L ClO_2
112	4.79 mg/L	4.67–4.91 mg/L ClO_2
113	0.26 mg/L	0.21–0.27 mg/L ClO_2
113	4.83 mg/L	4.71–4.97 mg/L ClO_2

For more information on determining precision data and method detection limits, see *Section 1* of the *DR/800 Procedures Manual*.

Estimated Detection Limit (EDL)

Program	EDL
112	0.04 mg/L ClO_2
113	0.04 mg/L ClO_2

For more information on derivation and use of Hach's estimated detection limit, see *Section 1* of the *DR/800 Procedures Manual*.

Interferences

A substance interferes if it changes the final reading by 0.1 mg/L ClO_2 or more.

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (see *Section 1, Correction For Volume Additions, in the DR/800 Procedures Manual).*
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (see *Section 1, Correction For Volume Additions, in the DR/800 Procedures Manual).*
Bromine, Br_2	Interferes at all levels.
Chlorine, Cl_2	May interfere at levels greater than 6 mg/L. Additional glycine may be able to compensate for this interference.
Chloramines, organic	May interfere.
Flocculating agents	High levels of most flocculating agents can be tolerated. This tolerance is decreased if chlorine is present. See the information about metals in this table. In the presence of 0.6 mg/L Cl_2, $Al(SO_4)_3$ (< 500 mg/L) and $FeCl_2$ (<200 mg/L) may be tolerated.
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$.

Interfering Substance	Interference Levels and Treatments
Iodine, I_2	Interferes at all levels.
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	Oxidized manganese interferes at all levels. Oxidized chromium interferes at levels greater than 2 mg/L. To remove the interferences: **1.** Adjust sample pH to 6–7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result of this test from the original analysis to obtain the correct chlorine dioxide concentration.
Metals	Various metals may interfere by combining with the glycine needed to remove the chlorine interference. Metal interference is limited except when chlorine is present. In the presence of 0.6 mg/L Cl_2, both copper (>10 mg/L) and nickel (>50 mg/L) interfere. Other metals may also interfere, depending on their ability to prevent glycine from reacting with any Cl_2 in the sample. It may be necessary to add more glycine to overcome this interference.
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L ClO_2 increase in the reading.
Ozone	Interferes at levels greater than 1.5 mg/L.
Peroxides	May interfere.
Extreme sample pH	Adjust to pH 6–7. See *Section 1, pH Interferences, in the DR/800 Procedures Manual.*
Highly buffered samples	Adjust to pH 6–7. *See Section 1, pH Interferences, in the DR/800 Procedures Manual.*

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004).

Summary of Method

Chlorine dioxide reacts with DPD (N,N-diethyl-p-phenylenediamine) Indicator Reagent (to the extent of one-fifth of its total available chlorine content corresponding to reduction of chlorine dioxide to chlorite) to form a pink color. The color intensity is proportional to the ClO_2 in the sample. Chlorine interference is eliminated by adding glycine, which converts free chlorine to chloroaminoacetic acid, but has no effect on chlorine dioxide at the test pH.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required per test	Unit	Cat. No.
Chlorine Dioxide DPD/Glycine Reagent Set (100 tests)			27709-00
Includes one of each:			
DPD Free Chlorine Reagent Powder Pillows, 10 mL	1 pillow	100/pkg	21055-69
Glycine Reagent	4 drops	29 mL	27621-33

REQUIRED REAGENTS (Using AccuVac® Ampuls)

Chlorine Dioxide DPD/Glycine AccuVac® Ampul Reagent Set (25 tests)			27710-00
Includes one of each:			
DPD Free Chlorine Reagent AccuVac® Ampuls	1	25/pkg	25020-25
Glycine Reagent	16 drops	29 mL	27621-33

OPTIONAL REAGENTS

Chlorine Standard Solution, Voluette™ ampule, 50-75 mg/L, 10 mL		16/pkg	14268-10
DPD Free Chlorine Reagent, SwifTest™		250 tests	28023-00
Potassium Iodide Solution, 30 g/L		100 mL* MDB	343-32
Sodium Arsenite, 5 g/L		100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1.000 N		100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N		100 mL* MDB	1270-32
Water, deionized		4 L	272-56
Water, sterile, chlorine dioxide-free		500 mL	26415-49

OPTIONAL APPARATUS

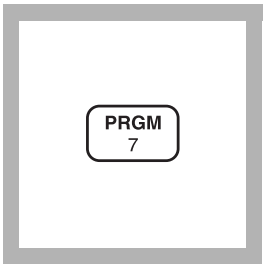
AccuVac® Snapper Kit		each	24052-00
Cylinder, graduated, 25 mL		each	508-40
pH Meter, *sens**ion**™1*, portable, with electrode		each	51700-10
pH Paper, 1 to 11 pH units		5 rolls/pkg	391-33
Pipet, TenSette®, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette® Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette® Pipet		1000/pkg	21856-28
PourRite™ Ampule Breaker		each	24846-00

For Technical Assistance, Price and Ordering
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Marked Dropper Bottle - contact Hach for larger sizes.

CHLORINE DIOXIDE, Mid Range (0 to 50.0 mg/L) For water and wastewater

Direct Reading Method

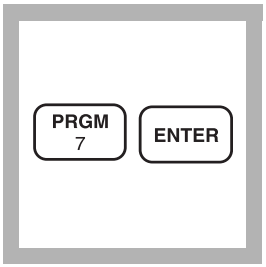


1. Enter the stored program number for mid-range chlorine dioxide (ClO_2).

Press: **PRGM**

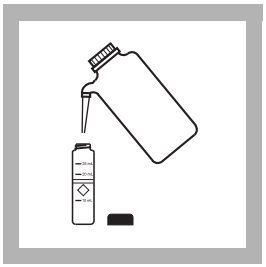
The display will show:

PRGM ?



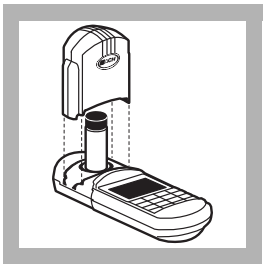
2. Press: **7 ENTER**

The display will show **mg/L, ClO2** and the **ZERO** icon**.**

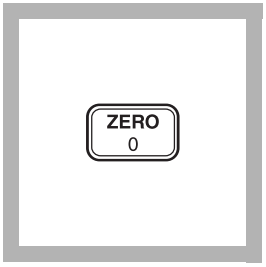


3. Fill a sample cell (the blank) with 10 mL of deionized water.

Note: *Analyze samples immediately after collection.*



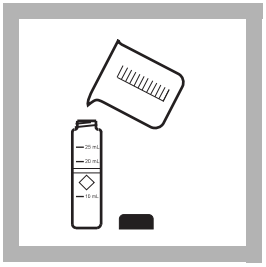
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



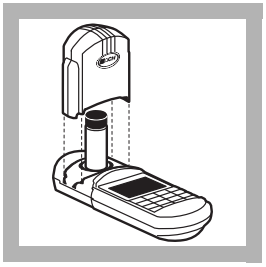
5. Press: **ZERO**

The cursor will move to the right, then the display will show:

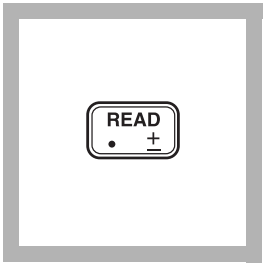
0.0 mg/L ClO2



6. Fill another sample cell with 10 mL of sample (the prepared sample).



7. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **READ**

The cursor will move to the right, then the result in mg/L chlorine dioxide will be displayed.

Note: *If the display flashes "limit" it is due to high ClO_2 levels. A slight loss of chlorine dioxide may occur during dilution. Dilute a fresh sample and repeat the test. Multiply the result by the dilution factor; see Section 1.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Chlorine dioxide is very volatile and unstable; analyze samples immediately upon collection.

Accuracy Check

Standard Solution Method

Preparing chlorine dioxide standards is difficult and dangerous. In addition, **these standards are both explosive and volatile!** Only a trained chemist should prepare the standards using appropriate safety equipment and precautions. Hach does not recommend independent standard preparation of chlorine dioxide standards. If independent standard preparation is required, please refer to the instructions in *Standard Methods for the Examination of Water and Wastewater*, 19th ed., under the headings "Stock chlorine dioxide solution" and "Standard chlorine dioxide solution" (pg. 4-54).

Method Performance

Precision

In a single laboratory, using a standard solution of 25.0 mg/L ClO_2, a single operator obtained a standard deviation of ±0.3 mg/L ClO_2. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program 7 is 7.3 mg/L ClO_2. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

Chlorine dioxide, a yellow gas, can be measured directly in a water solution. This method uses a wavelength of 420 nm to increase the range of the test.

REQUIRED REAGENTS AND APPARATUS

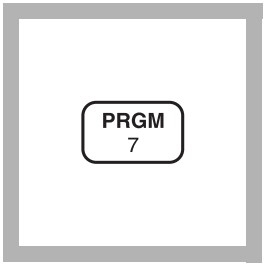
Description	Quantity Required Per Test	Unit	Cat. No.
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06
Water, deionized	10 mL	4 L	272-56

Outside the U.S.A.—Contact the Hach office or distributor serving you.

DPD Method

USEPA accepted for reporting drinking water analyses*
For testing higher levels of free chlorine (hypochlorous acid and hypochlorite) in drinking water, cooling water, and industrial process waters

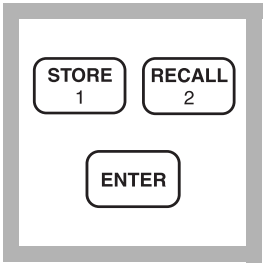
Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



1. Enter the user program number for Chlorine, UHR.

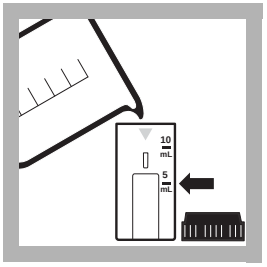
Press: **PRGM**
The display will show:
PRGM?

Note: *If the chlorine is typically less than 2.0 mg/L, use method 8021, program number 9.*

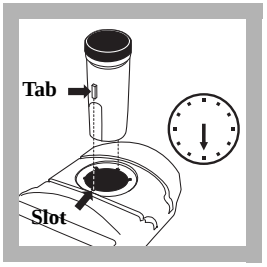


2. Press:
12 ENTER

The display will show
mg/L Cl_2
then: **ZERO**



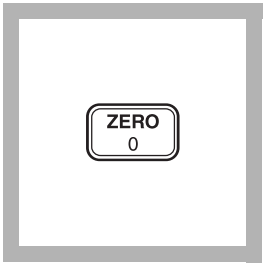
3. Fill the 10-mL/1-cm cell to the 5-mL line with sample.



4. Place the cell into the instrument. Cover the sample cell tightly with the instrument cap.

Note: *Place the cell into the cell holder as illustrated. The sample cell tab should be at the 6 o'clock position and completely seated in the cell holder slot.*

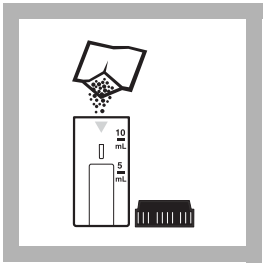
* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-C1-G for drinking water.



5. Press: **ZERO**

The cursor will move to the right, then the display will show:

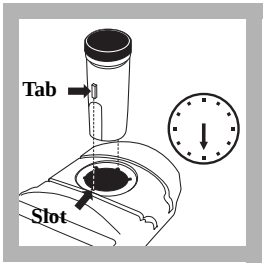
0.0 mg/L Cl_2



6. Remove the sample cell from the cell holder and add the contents of one 25-mL DPD Free Chlorine Reagent pillow to the sample. Cap and shake the sample cell about 20 seconds to dissolve.

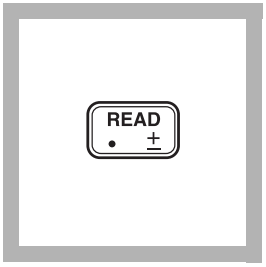
Proceed **immediately** to *step 7*.

Note: *A pink color will develop if chlorine is present.*



7. Place the sample cell into the instrument. Cover the sample cell tightly with the instrument cap.

Note: *Place the sample cell into the cell holder as illustrated. The sample cell tab should be at the 6-o'clock position and completely seated in the cell holder slot.*



8. Within one minute after reagent addition, press: **READ**.

The cursor will move to the right. The result in mg/L chlorine (as Cl_2) will be displayed.

Note: *See "Interferences" on page 120 for samples with high monochloramine concentrations.*

Sampling and Storage

Analyze samples for chlorine immediately after collection. Free chlorine is a strong oxidizing agent and reacts rapidly with various compounds. Many factors such as sunlight, pH, temperature, and sample composition will influence decomposition of free chlorine in water.

- Avoid plastic containers which may have a large chlorine demand.
- Pretreat glass sample containers to remove chlorine demand by soaking in a dilute bleach solution (1 mL of commercial bleach to 1 liter of deionized water) for at least one hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.
- Use separate, dedicated sample cells for free and total chlorine determinations. If trace iodide from the total chlorine reagent is carried over to the free chlorine test, monochloramine could interfere.

- A common error in testing for chlorine is failure to obtain a representative sample. If sampling from a tap, let the water flow for at least five minutes to ensure a representative sample. Let the sample container overflow with sample several times. Cap the container so there is no air above the sample.
- If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 5-mL mark. Proceed with the chlorine test immediately.

Accuracy Check

1. Fill three mixing cylinders (Cat. No. 20886-38) with 5-mL of sample.
2. Snap the neck of a HR Chlorine Ampule Standard, 50–75 mg/L Cl_2. Using the TenSette® Pipet, add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each cylinder and mix thoroughly.
3. Analyze each standard addition sample as described in the procedure. Record each result.
4. Calculate the concentration of mg/L chlorine added to each sample.

$$\text{mg/L chlorine added} = \frac{\text{volume of standard added} \times \text{label value of } Cl_2 \text{ standard ampule}}{\text{sample volume} + \text{volume of standard added}}$$

The spiked sample results should reflect the analyzed sample result plus the calculated mg/L Cl_2 added to each sample. If these increases do not occur, see Standard Additions in Section 1 of a DR/800 Procedure Manual for more information.

Method Performance

Precision

In a single laboratory, using a chlorine standard solution of 5.05 mg/L Cl_2 and representative lots of reagent, a single operator obtained a standard deviation of ± 0.05 mg/L Cl_2.

Estimated Detection Limit

The estimated detection limit for Method 10069 is 0.1 mg/L Cl_2. For more information on the estimated detection limit, see Section 1 of the DR/800 Procedure Manual.

Interferences

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. **1.** Neutralize to pH 6–7 with 1 N Sodium Hydroxide. **2.** Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested. **3.** Correct for volume addition.
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. **1.** Neutralize to pH 6–7 with 1 N Sulfuric Acid. **2.** Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested. **3.** Correct for volume addition.
Bromine, Br_2	Interferes at all levels
Chlorine Dioxide, ClO_2	Interferes at all levels
Chloramines, organic	May interfere
Iodine, I_2	Interferes at all levels
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7. **2.** Add 2 drops Potassium Iodide (30 g/L) to a 5-mL sample. **3.** Mix and wait 1 minute. **4.** Add 2 drops of Sodium Arsenite (5 g/L) and mix. **5.** Analyze the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.

<table>
<tr><th>Interfering Substance</th><th>Interference Levels and Treatments</th></tr>
<tr><td>Monochloramine</td><td>For conventional free chlorine disinfection (beyond the breakpoint), monochloramine concentrations are very low. If monochloramine is present in the sample, its interference in the free chlorine test varies with the sample temperature, the relative amount of monochloramine to free chlorine, and the time required to do the analysis. Approximate interference levels of monochloramine in the free chlorine test are listed below (as mg/L Cl_2).
<table>
<tr><th>NH_2Cl</th><th colspan="4">Sample Temperature °C (°F)</th></tr>
<tr><th>(as Cl_2)</th><th>5 (40)</th><th>10 (50)</th><th>20 (68)</th><th>30(83)</th></tr>
<tr><td>1.2</td><td>0.2</td><td>0.2</td><td>0.3</td><td>0.3</td></tr>
<tr><td>2.5</td><td>0.4</td><td>0.5</td><td>0.6</td><td>0.6</td></tr>
<tr><td>3.5</td><td>0.5</td><td>0.6</td><td>0.7</td><td>0.8</td></tr>
</table></td></tr>
<tr><td>Ozone</td><td>Interferes at all levels</td></tr>
<tr><td>Peroxides</td><td>May interfere</td></tr>
<tr><td>Extreme sample pH or highly buffered samples</td><td>Adjust the sample pH to 6–7 with Sulfuric Acid or Sodium Hydroxide</td></tr>
</table>

Summary of Method

The range of analysis using the DPD method for free chlorine can be extended by adding more indicator in proportion to sample volume. Thus, a larger fill powder pillow of DPD Free Chlorine Reagent is added to a 5-mL sample portion.

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) reacts immediately with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color which is proportional in intensity to the chlorine concentration.

Instrument Setup

The following procedure will add this method as a new Hach program to a DR/800 instrument.

1. Turn on the instrument by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the **DOWN** arrow key until the prompt line shows USER.
4. Press the **ENTER** key.
5. Enter “8138”, followed by **ENTER**.

6. Key the number in the "Enter" column corresponding to line number 1 on the display. Press **ENTER**. Repeat for lines 2–56 on the display.

Line Number	Enter	Line Number	Enter
1	12	29	0
2	24	30	0
3	73	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	62	36	0
9	55	37	0
10	23	38	0
11	88	39	0
12	64	40	0
13	113	41	0
14	242	42	0
15	18	43	0
16	0	44	110
17	0	45	0
18	0	46	0
19	0	47	10
20	67	48	0
21	108	49	180
22	50	50	0
23	0	51	0
24	0	52	0
25	0	53	0
26	0	54	236
27	0	55	0
28	0	56	255

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
DPD Free Chlorine Reagent Powder Pillows, 25-mL	1	100/pkg	14070-99

REQUIRED APPARATUS

Sample Cell, 10-mL/1-cm	1	2/pkg	48643-02

OPTIONAL REAGENTS

Chlorine Standard Solution, 2-mL Voluette® Ampule, 50–75 mg/L		20/pkg	14268-20
Potassium Iodide Solution, 30-g/L		100 mL MDB	343-32
Sodium Arsenite Solution, 5-g/L		100 mL MDB	1047-32
Sodium Hydroxide Standard Solution, 1.00 N		100 mL MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N		100 mL MDB	1270-32
Water, deionized		4 L	272-56

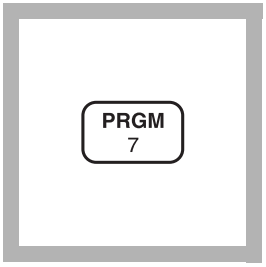
OPTIONAL APPARATUS

Ampule Breaker Kit		each	24846-00
Cylinder, graduated, 10-mL, mixing		each	20886-38
pH Meter, sens***ion***™1, portable, with electrode		each	51700-10
Pipet, TenSette®, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28

CHLORINE, TOTAL, Ultra-High Range (0.0–10.0 mg/L Cl_2) Method 10070

DPD Method **USEPA accepted for reporting water and wastewater analyses***
For testing higher levels of total chlorine (free and combined) in drinking water, cooling water, industrial process waters, or treated wastewater

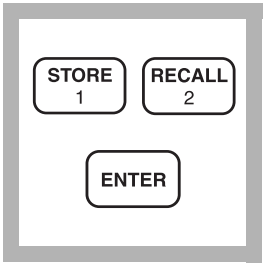
Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



1. Enter the user program number for Chlorine, UHR.

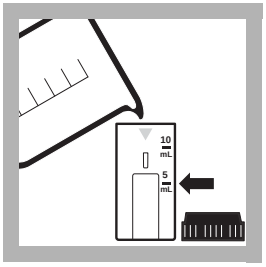
Press: **PRGM**
The display will show:
PRGM?

Note: *If the chlorine is typically less than 2.0 mg/L, use method 8167, program number 9.*

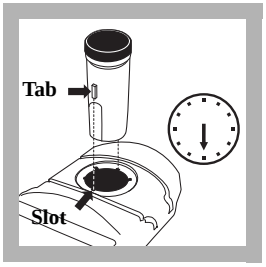


2. Press:
12 ENTER

The display will show
mg/L Cl_2
then: **ZERO**



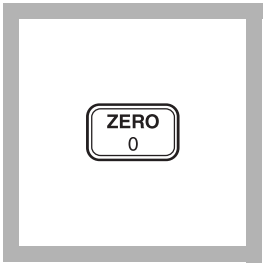
3. Fill the 10-mL/1-cm cell to the 5-mL line with sample.



4. Place the sample cell into the instrument. Cover the sample cell tightly with the instrument cap.

Note: *Place the cell into the cell holder as illustrated. The sample cell tab should be at the 6 o'clock position and completely seated in the cell holder slot.*

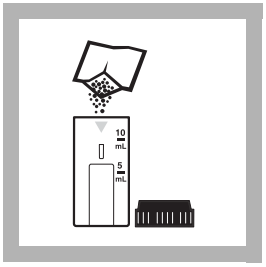
* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-C1-G for drinking water.



5. Press: **ZERO**

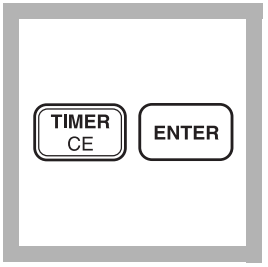
The cursor will move to the right, then the display will show:

0.0 mg/L Cl_2



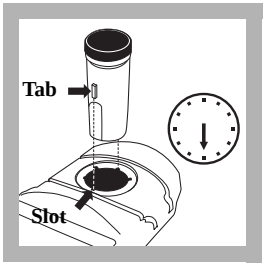
6. Remove the sample cell from the cell holder and add the contents of one 25- mL DPD Total Chlorine Reagent pillow to the sample. Cap and shake the sample cell about 20 seconds to dissolve.

Note: *A pink color will develop if chlorine is present.*



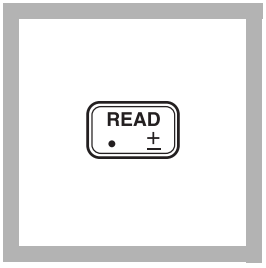
7. Press:
TIMER ENTER

A 3-minute reaction period will begin.



8. Within 3 minutes after the timer beeps, place the sample cell into the instrument. Cover the sample cell tightly with the instrument cap.

Note: *Place the cell into the cell holder as illustrated. The sample cell tab should be at the 6-o'clock position and completely seated in the cell holder slot.*



9. Press: **READ**

The cursor will move to the right. The result in mg/L chlorine (as Cl_2) will be displayed.

Sampling and Storage

Analyze samples for chlorine immediately after collection. Free and combined chlorine are strong oxidizing agents and react rapidly with various compounds. Many factors such as sunlight, pH, temperature, and sample composition will influence decomposition of chlorine in water.

- Avoid plastic containers which may have a large chlorine demand.
- Pretreat glass sample containers to remove chlorine demand by soaking in a dilute bleach solution (1 mL of commercial bleach to 1 liter of deionized water) for at least one hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.
- Use separate, dedicated sample cells for free and total chlorine determinations. If trace iodide from the total chlorine reagent is carried over to the free chlorine test, monochloramine could interfere.
- A common error in testing for chlorine is failure to obtain a representative sample. If sampling from a tap, let the water flow for at least five minutes to ensure a representative sample. Let the sample container overflow with sample several times. Cap the container so there is no air above the sample.
- If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 5-mL mark. Proceed with the chlorine test immediately.

Accuracy Check

1. Fill three mixing cylinders (Cat. No. 20886-38) with 5-mL of sample.

2. Snap the neck of a HR Chlorine Ampule Standard, 50–75 mg/L Cl_2. Using the TenSette® Pipet, add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each cylinder and mix thoroughly.

3. Analyze each standard addition sample as described in the procedure. Record each result.

4. Calculate the concentration of mg/L chlorine added to each sample.

$$\text{mg/L chlorine added} = \frac{\text{volume of standard added} \times \text{label value of } Cl_2 \text{ standard ampule}}{\text{sample volume + volume of standard added}}$$

The spiked sample results should reflect the analyzed sample result plus the calculated mg/L Cl_2 added to each sample. If these increases do not occur, see Standard Additions in Section 1 of a DR/800 Procedure Manual for more information.

Method Performance

Precision

In a single laboratory, using a chlorine standard solution of 5.05 mg/L Cl_2 and representative lots of reagent, a single operator obtained a standard deviation of ± 0.05 mg/L Cl_2.

Estimated Detection Limit

The estimated detection limit for Method 10070 is 0.05 mg/L Cl_2. For more information on the estimated detection limit, see Section 1 of a DR/800 Procedure Manual.

Interferences

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. **1.** Neutralize to pH 6–7 with 1 N Sodium Hydroxide. **2.** Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested. **3.** Correct for volume addition.
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. **1.** Neutralize to pH 6–7 with 1 N Sulfuric Acid. **2.** Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested. **3.** Correct for volume addition.
Bromine, Br_2	Interferes at all levels
Chlorine Dioxide, ClO_2	Interferes at all levels
Chloramines, organic	May interfere
Iodine, I_2	Interferes at all levels

Interfering Substance	Interference Levels and Treatments
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7. **2.** Add 2 drops Potassium Iodide (30 g/L) to a 5-mL sample. **3.** Mix and wait 1 minute. **4.** Add 2 drops of Sodium Arsenite (5 g/L) and mix. **5.** Analyze the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH or highly buffered samples	Adjust the sample pH to 6–7 with Sulfuric Acid or Sodium Hydroxide

Summary of Method

The range of analysis using the DPD method for total chlorine can be extended by adding more indicator in proportion to sample volume. Thus, a larger fill powder pillow of DPD Total Chlorine Reagent is added to a 5-mL sample portion.

The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N,N-diethyl-p-phenylenediamine) along with free chlorine present in the sample to form a pink color which is proportional in intensity to the total chlorine concentration.

Instrument Setup

The following procedure will add this method as a new Hach program to a DR/800 instrument.

1. Turn on the instrument by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the **DOWN** arrow key until the prompt line shows USER.
4. Press the **ENTER** key.
5. Enter “8138”, followed by **ENTER**.
6. Key the number in the “Enter” column corresponding to line number 1 on the display. Press **ENTER**. Repeat for lines 2–56 on the display.

Line Number	Enter	Line Number	Enter
1	12	29	0
2	24	30	0
3	73	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	62	36	0
9	55	37	0
10	23	38	0
11	88	39	0
12	64	40	0
13	113	41	0
14	242	42	0
15	18	43	0
16	0	44	110
17	0	45	0
18	0	46	0
19	0	47	10
20	67	48	0
21	108	49	180
22	50	50	0
23	0	51	0
24	0	52	0
25	0	53	0
26	0	54	236
27	0	55	0
28	0	56	255

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows, 25-mL	1	100/pkg	14064-99

REQUIRED APPARATUS

Sample Cell, 10-mL/1-cm	1	2/pkg	48643-02

OPTIONAL REAGENTS

Chlorine Standard Solution, 2-mL Voluette® Ampule, 50–75 mg/L		20/pkg	14268-20
Potassium Iodide Solution, 30-g/L		100 mL MDB	343-32
Sodium Arsenite Solution, 5-g/L		100 mL MDB	1047-32
Sodium Hydroxide Standard Solution, 1.00 N		100 mL MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N		100 mL MDB	1270-32
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

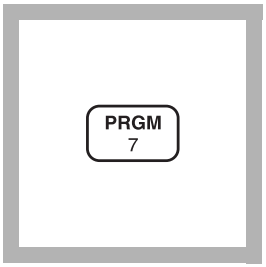
Ampule Breaker Kit		each	24846-00
Cylinder, graduated, 10-mL, mixing		each	20886-38
pH Meter, sens***ion***™1, portable, with electrode		each	51700-10
Pipet, TenSette®, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28

CHLORINE, FREE (0 to 2.00 mg/L)

DPD Method (Powder Pillows or AccuVac Ampuls)USEPA accepted for reporting wastewater and drinking water analyses*

Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*

Using Powder Pillows



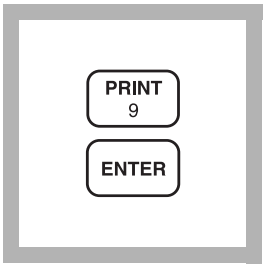
1. Enter the stored program number for free and total chlorine (Cl_2) powder pillows.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



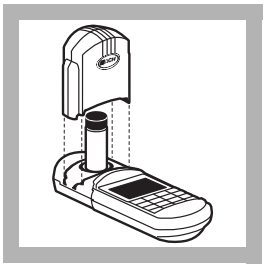
2. Press: **9 ENTER**

The display will show **mg/L, Cl2** and the **ZERO** icon.

3. Fill a sample cell with 10 mL of sample (the blank).

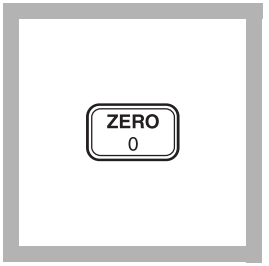
Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

Note: *The SwifTest Dispenser for Free Chlorine can be used in place of the powder pillows in step 7.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-Cl G for drinking water.

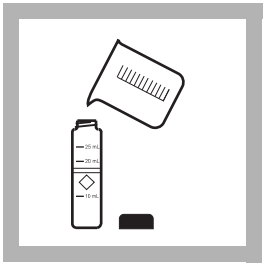


5. Press: **ZERO**

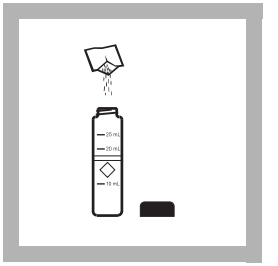
The cursor will move to the right, then the display will show:

0.00 mg/L Cl2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

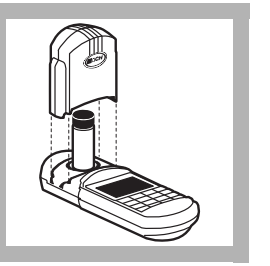


6. Fill another cell with 10 mL of sample.



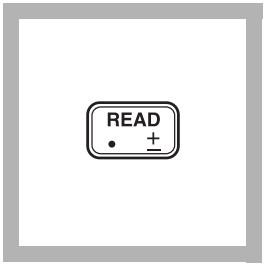
7. Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and swirl vigorously to dissolve the powder.

Note: *A pink color will develop if free chlorine is present.*



8. Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Perform Step 9 within one minute of reagent addition.*



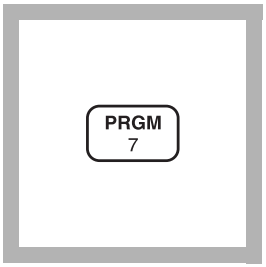
9. Press: **READ**

The cursor will move to the right, then the result in mg/L chlorine will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Note: *If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the dilution factor; see Section 1. Or, use the High Range Free Chlorine test, program #8.*

Using AccuVac Ampuls



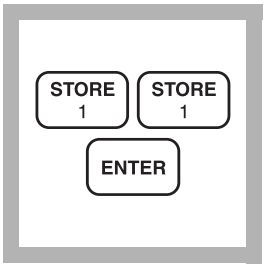
1. Enter the stored program number for free and total chlorine (Cl_2)-AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

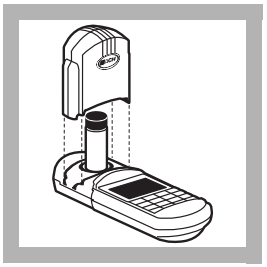


2. Press: **11 ENTER**

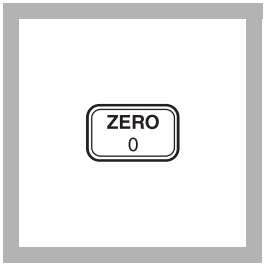
The display will show **mg/L, Cl2** and the **ZERO** icon.

3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

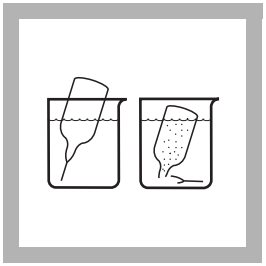


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

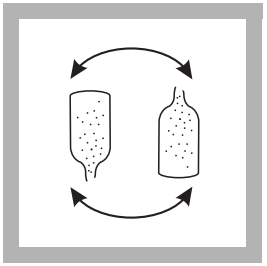
0.00 mg/L Cl2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



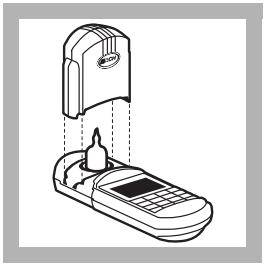
6. Fill a DPD Free Chlorine Reagent AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampule fills completely.*



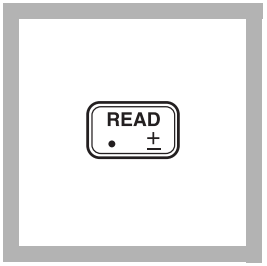
7. Quickly invert the ampule several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will form if chlorine is present.*



8. Immediately place the AccuVac Ampul into the cell holder. Tightly cover the ampule with the instrument cap.

Note: *Perform step 9 within one minute of reagent addition.*



9. Press: **READ**

The cursor will move to the right, then the result in mg/L chlorine will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Note: *If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the dilution factor; see Section 1.*

Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free chlorine is a strong oxidizing agent, and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of free chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over into the free chlorine determination, monochloramine will interfere. It is best to use separate, dedicated sample cells for free and total chlorine determinations.

A common error in testing for chlorine is introduced when a representative sample is not obtained. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the analysis immediately.

Accuracy Check

Standard Additions Method (using powder pillows)

a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.

b) Use a TenSette Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.

c) Re-zero the instrument using the original sample (the blank).

d) Place the spiked sample in the cell holder and press **READ**. Record the results.

e) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L Chlorine added} = \frac{0.1(\text{vol. standard added}) \times \text{Label value (mg/L } Cl_2)}{10.1(\text{sample + standard volume})}$$

f) The spiked sample result (step d) should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step e).

g) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method (using AccuVac Ampuls)

a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.

b) Use a graduated cylinder to measure 25 mL of sample into each of two beakers.

c) Use a TenSette Pipet to add 0.2 mL of the standard to one of the beakers (this is the spiked sample). Swirl to mix.

d) Fill a DPD Free Chlorine AccuVac completely from each

beaker.

e) Analyze the spiked and unspiked sample as described in the procedure.

f) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L Chlorine added} = \frac{0.2(\text{vol. standard added}) \times \text{Label value (mg/L } Cl_2)}{25.2(\text{sample + standard volume})}$$

g) The spiked sample result should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step f).

h) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 1.00 mg/L chlorine and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of ±0.01 mg/L chlorine.

In a single laboratory using a standard solution of 1.00 mg/L chlorine and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.01 mg/L chlorine.

Estimated Detection Limit (EDL)

The estimated detection limit for programs 9 and 11 is 0.02 mg/L Cl_2. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$
Iodine	Interferes at all levels
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium , Oxidized (Cr^{6+})	**1.** Adjust sample pH to 6-7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L increase in the reading.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See *Interferences, Section 1*.

Summary of Method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a magenta color which is proportional to the chlorine concentration.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
DPD Free Chlorine Powder Pillows, 10 mL	1 pillow	100/pkg	21055-69
Sample Cell, 10, 20, 25 mL, w/ cap	2	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

DPD Free Chlorine Reagent AccuVac Ampuls	1 ampul	25/pkg	25020-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Chlorine Standard Solution, PourRite ampule, 25-30 mg/L, 2 mL	20/pkg	26300-20
DPD Free Chlorine Reagent, SwifTest	250 tests	28023-00
Potassium Iodide Solution, 30 g/L	100 mL* MDB	343-32
Sodium Arsenite, 5 g/L	100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1.000 N	100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N	100 mL* MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Cylinder, graduated, 25 mL	each	508-40
pH Meter, *sens**ion***™***1***, portable, with electrode	each	51700-10
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96Pipet
Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
PourRite Ampule Breaker	each	24846-00

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Marked Dropper Bottle - contact Hach for larger sizes.

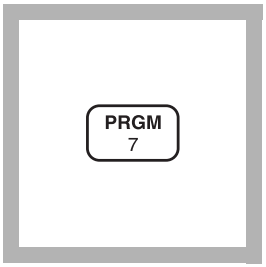
CHLORINE, TOTAL (0 to 2.00 mg/L)

DPD Method (Powder Pillows or AccuVac Ampuls)

USEPA accepted for reporting water and wastewater analyses*

Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*

Using Powder Pillows



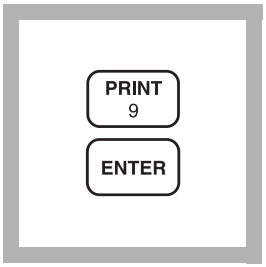
1. Enter the stored program number for total chlorine (Cl_2) powder pillows.

Press: **PRGM**

The display will show:

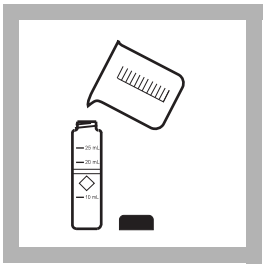
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



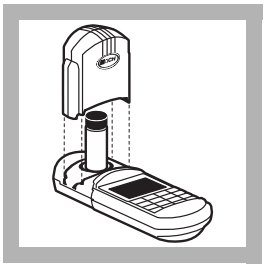
2. Press: **9 ENTER**

The display will show **mg/L, Cl2** and the **ZERO** icon**.**



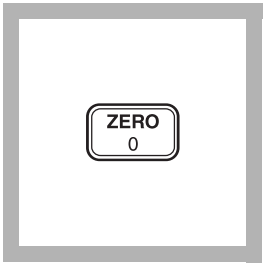
3. Fill a sample cell with 10 mL of sample (the blank).

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-Cl G for drinking water.

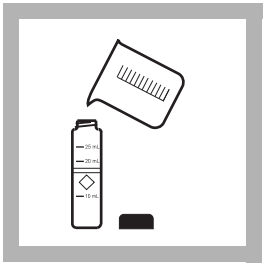


5. Press: **ZERO**

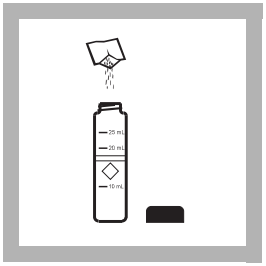
The cursor will move to the right, then the display will show:

0.00 mg/L Cl2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

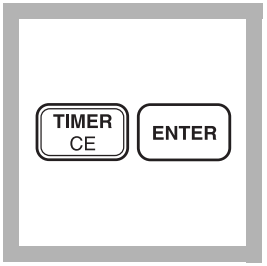


6. Fill a second cell to the 10-mL mark with sample.



7. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Cap and swirl the sample cell vigorously to dissolve the powder.

Note: *It is not necessary that all the powder dissolves.*

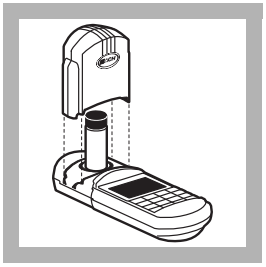


8. Press:

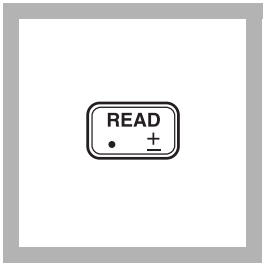
TIMER ENTER

A three-minute reaction period will begin. A pink color will develop if chlorine is present.

Note: *The SwifTest Dispenser for Total Chlorine can be used in place of the powder pillows in step 7.*



9. After the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



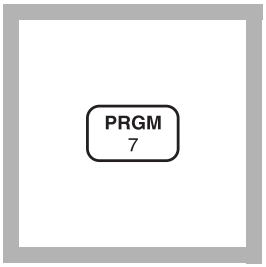
10. Press: **READ**

The cursor will move to the right, then the result in mg/L total chlorine will be displayed.

Note: *It the sample temporarily turns yellow after sample addition, or the display flashes "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the dilution factor; see Section 1. Or use the High Range Total Chlorine test, program #8.*

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Using AccuVac Ampuls



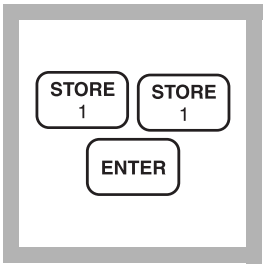
1. Enter the stored program number for total chlorine (Cl_2) AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

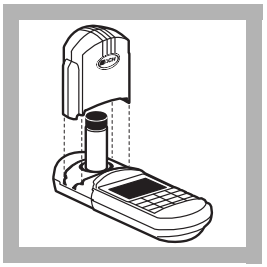


2. Press: **11 ENTER**

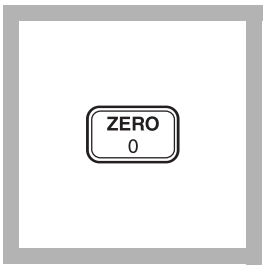
The display will show **mg/L, Cl2** and the **ZERO** icon**.**

3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

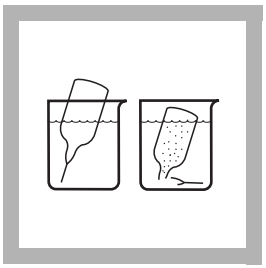


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

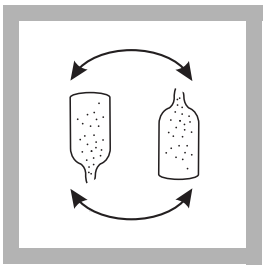
0.00 mg/L Cl2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



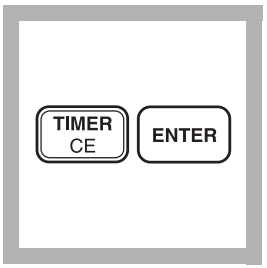
6. Fill a DPD Total Chlorine Reagent AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampule fills completely.*



7. Quickly invert the ampule several times to mix. Wipe off any liquid or fingerprints.

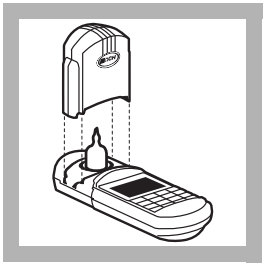
Note: *A pink color will form if chlorine is present.*



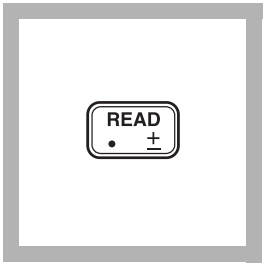
8. Press:

TIMER ENTER

A three-minute reaction period will begin.



9. When the timer beeps, place the AccuVac Ampul into the cell holder. Tightly cover the ampule with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L total chlorine will be displayed.

Note: *If the sample temporarily turns yellow after sample addition, or the display shows "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the appropriate dilution factor; see Section 1.*

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free chlorine is a strong oxidizing agent, and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence decomposition of chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over into the free chlorine determination, monochloramine will interfere. It is best to use separate, dedicated sample cells for free and total chlorine determinations.

A common error in testing for chlorine is introduced when a representative sample is not obtained. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the chlorine analysis immediately.

Accuracy Check

Standard Additions Method (using powder pillows)

a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.

b) Use a TenSette Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.

c) Re-zero the instrument using the original sample (the blank).

d) Place the spiked sample into the cell holder and press **READ**. Record the results.

e) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L chlorine added} = \frac{0.1\ (\text{vol. standard added}) \times \text{Label value (mg/L } Cl_2)}{10.1(\text{sample + standard volume})}$$

f) The spiked sample result (step d) should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step e).

g) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method (using AccuVac Ampuls)

a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.

b) Use a graduated cylinder to measure 25 mL of sample into

each of two beakers.

c) Use a TenSette Pipet to add 0.2 mL of the standard to one of the beakers (this is the spiked sample). Swirl to mix.

d) Fill a DPD Total Chlorine AccuVac completely from each beaker.

e) Analyze the spiked and unspiked sample as described in
the procedure.

f) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L chlorine added} = \frac{0.2\ (\text{vol. standard added}) \times \text{Label value (mg/L Chlorine)}}{25.2\ (\text{sample + standard volume})}$$

g) The spiked sample result should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step f).

h) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Method Performance

Precision

In a single laboratory, using a standard solution of 1.00 mg/L chlorine and two lots of reagents with the instrument, a single operator obtained standard deviations of ±0.01 mg/L chlorine.

In a single laboratory, using a standard solution of 1.00 mg/L chlorine and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.01 mg/L chlorine.

Estimated Detection Limit (EDL)

The estimated detection limit for programs 9 and 11 is 0.02 mg/L Cl_2. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$
Iodine	Interferes at all levels
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium , Oxidized (Cr^{6+})	**1.** Adjust sample pH to 6-7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See *Interferences, Section 1*.

Summary of Method

Chlorine can be present in water as free available chlorine and as combined available chlorine. Both forms can exist in the same water and be determined together as the total available chlorine. Free chlorine is present as hypochlorous acid and/or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride and other chloro derivatives.

The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N, N-diethyl-p-phenylenediamine)

along with free chlorine present in the sample to form a red color which is proportional to the total chlorine concentration. To determine the concentration of combined chlorine, run free chlorine and total chlorine tests. Subtract the results of the free chlorine test from the results of the total chlorine test to obtain combined chlorine.

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS & APPARATUS (USING POWDER PILLOWS)

Description	Qty/Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows	1 pillow	100/pkg	21056-69
Sample Cell, 10-20-25 mL, w/caps	2	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (USING ACCUVAC AMPULS)

DPD Total Chlorine Reagent AccuVac Ampuls	1 ampul	25/pkg	25030-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Chlorine Standard Solution, PourRite ampule, 25-30 mg/L Cl_2	20/pkg	26300-20
DPD Total Chlorine Reagent, SwifTest	250 tests	28024-00
Potassium Iodide Solution, 30 g/L	100 mL* MDB	343-32
Sodium Arsenite, 5 g/L	100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1 N	100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1 N	100 mL* MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
PourRite Ampule Breaker	each	24846-00
Cylinder, graduated, 25 mL	each	508-40
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable	each	51700-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28

For Technical Assistance, Price and Ordering

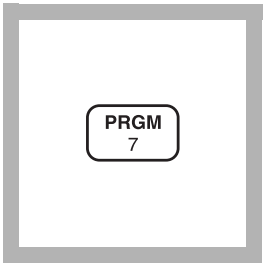
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Marked Dropper Bottle - contact Hach for larger sizes.

CHLORINE, FREE (0 to 5.00 mg/L)

DPD Test 'N Tube™ Method*

Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



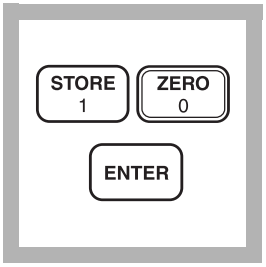
1. Enter the stored program number for Test 'N Tube free chlorine (Cl_2).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

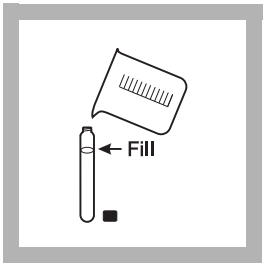


2. Press: **10 ENTER**

The display will show **mg/L, Cl2** and the **ZERO** icon**.**

3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down fully to insert it.

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*

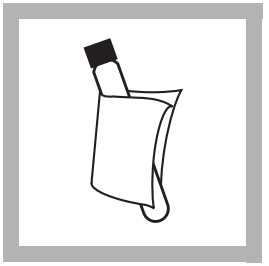


4. Fill an empty Test 'N Tube vial with sample (the blank).

Note: *Fill to the top of the Hach logo "oval" mark.*

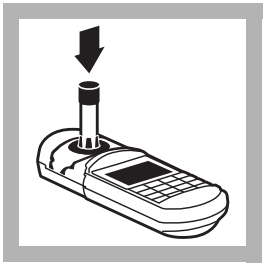
Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater*



5. Wipe the outside of the blank vial with a towel.

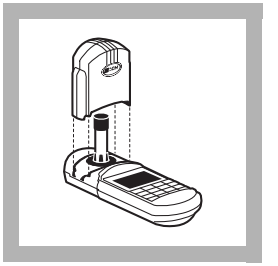
Note: *Wiping with a damp cloth followed by a dry one removes fingerprints and other marks.*



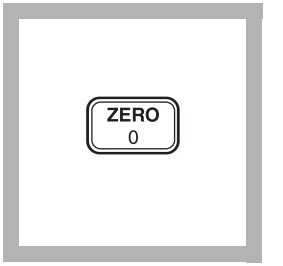
6. Place the blank in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



7. Cover the vial tightly with the instrument cap.

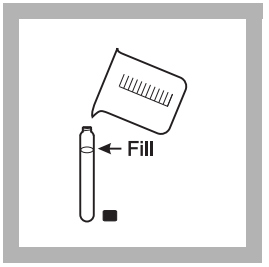


8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Cl2

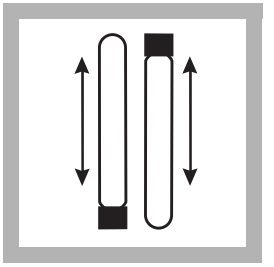
Note: *If Reagent Blank Correction is on, the display may show "limit". See Section 1.*



9. Remove the cap from a Free Chlorine DPD-TNT tube. Add 10 mL of sample.

Note: *Fill to the top of the Hach logo "oval" mark.*

Note: *A pink color will develop if chlorine is present.*



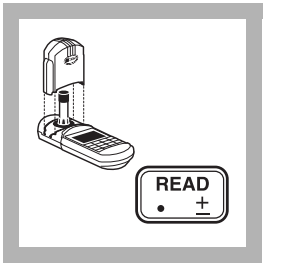
10. Cap and invert at least 10 times to dissolve the powder. This is the prepared sample.

Note: *Use slow, deliberate inversion for complete recovery. Ten inversions should take at least 30 seconds. One inversion equals turning the vial upside down, then returning it to an upright position.*

11. Within 30 seconds after mixing, wipe the prepared sample vial with a towel, then place it in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Cover the vial tightly with the instrument cap.

Press: **READ**

The cursor will move to the right, then the result in mg/L free chlorine will be displayed.

Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free chlorine is a strong oxidizing agent and is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of free chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for chlorine is obtaining an unrepresentative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. Perform the analysis immediately.

Accuracy Check

Standard Additions Method

a) Snap the top off a HR Chlorine PourRite™ Ampule Standard Solution.

b) Use a TenSette® Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.

c) Analyze the spiked sample, beginning at Step 8 of the procedure.

d) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L chlorine added} = \frac{0.1(\text{vol. standard added}) \times \text{Label value}(\text{mg/L } Cl_2)}{10.1(\text{sample + standard volume})}$$

e) The spiked sample result (step c) should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step d).

f) If this increase does not occur, see *Standard Additions, Section 1* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 2.53 mg/L chlorine and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of ±0.14 mg/L chlorine.

Estimated Detection Limit (EDL)

The estimated detection limit for program 10 is 0.03 mg/L Cl_2. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions* in the *DR/800 Series Procedures Manual*).
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1 Correcting for Volume Additions*).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$
Iodine	Interferes at all levels
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium , oxidized (Cr^{6+})	**1.** Adjust sample pH to 6-7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.

<table>
<tr><th>Interfering Substance</th><th>Interference Level and Treatment</th></tr>
<tr><td>Monochloramine</td><td>For conventional free chlorine disinfection (beyond the breakpoint), typical monochloramine concentrations are very low. If monochloramine is present in the sample, its interference in the free chlorine test depends on the sample temperature, relative amount of monochloramine to free chlorine, and the time required to do the analysis. Typical interference level of monochloramine in the free chlorine test are listed below (as mg/L Cl_2).
<table>
<tr><th>NH_2Cl as Cl_2</th><th colspan="4">Sample Temp. °C (°F)</th></tr>
<tr><td></td><td>5 (40)</td><td>10 (50)</td><td>20 (68)</td><td>30 (83)</td></tr>
<tr><td>1.2 mg/L</td><td>+0.15</td><td>+0.19</td><td>+0.30</td><td>+0.29</td></tr>
<tr><td>2.5 mg/L</td><td>0.35</td><td>0.38</td><td>0.55</td><td>0.61</td></tr>
<tr><td>3.5 mg/L</td><td>0.38</td><td>0.56</td><td>0.69</td><td>0.73</td></tr>
</table>
</td></tr>
<tr><td>Ozone</td><td>Interferes at all levels</td></tr>
<tr><td>Peroxides</td><td>May interfere</td></tr>
<tr><td>Extreme sample pH and highly buffered samples</td><td>Adjust to pH 6-7. See Interferences, Section 1.</td></tr>
</table>

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

Summary of Method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD
(N,N-diethyl-p-phenylenediamine) indicator to form a magenta color which is proportional to the chlorine concentration.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Test 'N Tube DPD Free Chlorine Reagent	1 vial	50/pkg	21055-45
Test 'N Tube Vials	1 vial	6/pkg	22758-06

REQUIRED APPARATUS

Caps, white	1 cap	6/pkg	22411-06
COD/TNT Adapter	1	each	48464-00

OPTIONAL REAGENTS

Chlorine Standard Solution, PourRite ampule, 50-75 mg/L, 2 mL		20/pkg	14268-20
Potassium Iodide Solution, 30 g/L		100 mL* MDB	343-32
Sodium Arsenite, 5 g/L		100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1.000 N		100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N		100 mL* MDB	1270-32

OPTIONAL APPARATUS

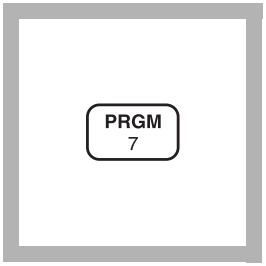
Beaker, 50 mL		each	500-41H
pH Meter, *sens**ion**™1*, portable, with electrode		each	51700-10
pH Paper, pH 1 to 11 pH		5 rolls/pkg	391-33
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite Ampule Breaker		each	24846-00
Test Tube Rack		each	18641-00

* Marked Dropper Bottle - contact Hach for larger sizes.

CHLORINE, TOTAL (0 to 5.00 mg/L)

DPD Test 'N Tube™ Method*

Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



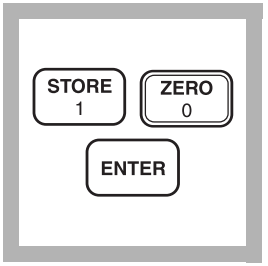
1. Enter the stored program number for Test 'N Tube total chlorine (Cl_2).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

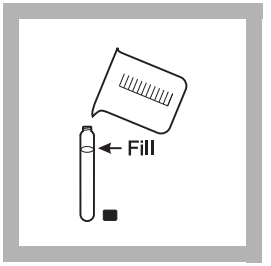


2. Press: **10 ENTER**

The display will show **mg/L, Cl2** and the **ZERO** icon**.**

3. Insert the COD/TNT Vial Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*

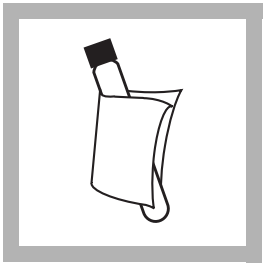


4. Fill an empty Test 'N Tube vial with sample (the blank).

Note: *Fill to the top of the Hach logo "oval" mark.*

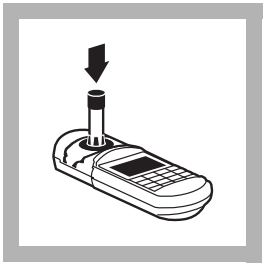
Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



5. Wipe the outside of the blank vial with a towel.

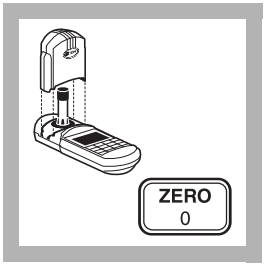
Note: *Wiping with a damp cloth followed by a dry one removes fingerprints and other marks.*



6. Place the blank in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



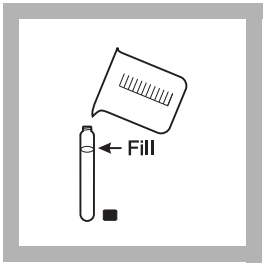
7. Cover the vial tightly with the instrument cap.

Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Cl2

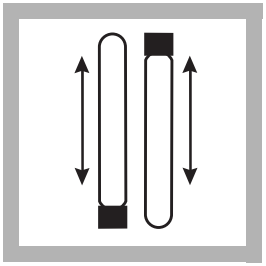
Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



8. Remove the cap from a Total Chlorine DPD-TNT tube. Add 10 mL of sample.

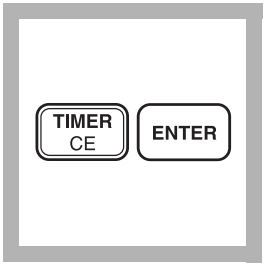
Note: *Fill to the top of the Hach logo "oval" mark.*

Note: *A pink color will develop if chlorine is present.*



9. Cap and invert at least 10 times to dissolve the powder. This is the prepared sample.

Note: *Use slow, deliberate inversion for complete recovery. Ten inversions should take at least 30 seconds. One inversion equals turning the vial upside down, then returning it to an upright position.*

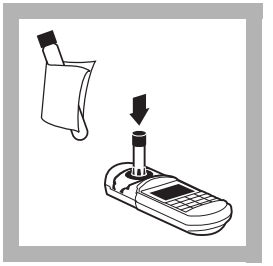


10. Press:

TIMER ENTER

A three-minute reaction period will begin.

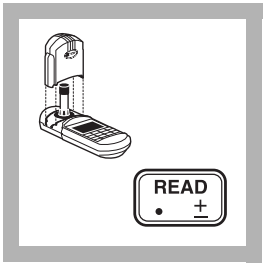
Note: *A pink color will develop if chlorine is present.*



11. When the timer beeps, wipe the prepared sample vial with a towel, then place it in the vial adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Cover the vial tightly with the instrument cap.

Press: **READ**

The cursor will move to the right, then the result in mg/L total chlorine will be displayed.

Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free and combined chlorine are strong oxidizing agents and are unstable in natural waters. They react rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence decomposition of chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

A common error in testing for chlorine is obtaining an unrepresentative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. Perform the analysis immediately.

Accuracy Check

Standard Additions Method

a) Snap the top off a High Range Chlorine PourRite™ Ampule Standard Solution.

b) Use a TenSette® Pipet to add 0.1 mL of the standard to 10 mL of sample (this is the spiked sample). Swirl to mix.

c) Analyze the spiked sample, beginning at Step 8 of the procedure.

d) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L chlorine added} = \frac{0.1\ (\text{vol. standard added}) \times \text{Label value (mg/L } Cl_2)}{10.1(\text{sample + standard volume})}$$

e) The spiked sample result (step c) should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step d).

f) If this increase does not occur, see *Standard Additions, Section 1* for more information.

Method Performance

Precision

In a single laboratory, using a standard solution of 2.53 mg/L chlorine and two representative lots of reagents with the instrument, a single operator obtained standard deviations of ±0.14 mg/L chlorine.

Estimated Detection Limit (EDL)

The estimated detection limit for programs 10 is 0.03 mg/L Cl_2. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Correcting for Volume Additions* in *Section 1*).
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Correcting for Volume Additions* in *Section 1*).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$
Iodine	Interferes at all levels
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium , oxidized (Cr^{6+})	**1.** Adjust sample pH to 6-7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See *Interferences* in *Section 1*.

Summary of Method

Chlorine can be present in water as free available chlorine and as combined available chlorine. Both forms can exist in the same water and be determined together as the total available chlorine. Free chlorine is present as hypochlorous acid and/or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride and other chloro derivatives.

The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N, N-diethyl-p-phenylenediamine) along with free chlorine present in the sample to form a red color which is proportional to the total chlorine concentration. To determine the concentration of combined chlorine, run free chlorine and total chlorine tests. Subtract the results of the free chlorine test from the results of the total chlorine test to obtain combined chlorine.

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004).

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Test 'N Tube DPD Total Chlorine Reagent	1 vial	25/pkg	21056-25
Test 'N Tube Vials	1 vial	6/pkg	22758-06

REQUIRED APPARATUS

COD/TNT Adapter, DR/800	1	each	48464-00

OPTIONAL REAGENTS

Chlorine Standard Solution, 2-mL PourRite ampule, 50-75 mg/L	20/pkg		14268-20
Potassium Iodide Solution, 30 g/L	100 mL*	MDB	343-32
Sodium Arsenite Solution, 5 g/L	100 mL*	MDB	1047-32
Sodium Hydroxide Standard Solution, 1.00 N	100 mL*	MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N	100 mL*	MDB	1270-32

OPTIONAL APPARATUS

Beaker, 50 mL	each		500-41H
PourRite Ampule Breaker	each		24846-00
pH Indicator Paper, pH 1 to 11	5 rolls/pkg		391-33
pH Meter, *sens**ion***™***1**, portable, with electrode	each		51700-10
Pipet, TenSette, 0.1 to 1.0 mL	each		19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg		21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg		21856-28
Test Tube Rack	each		18641-00

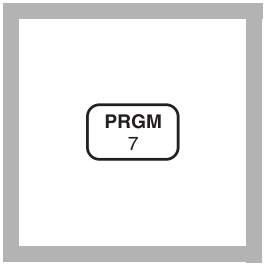
* Marked Dropper Bottle - contact Hach for larger sizes.

CHROMIUM, HEXAVALENT (0 to 0.60 mg/L Cr^{6+}) For water and wastewater

1,5-Diphenylcarbohydrazide Method* (Powder Pillows or AccuVac Ampuls)

USEPA accepted for wastewater analyses**

Using Powder Pillows

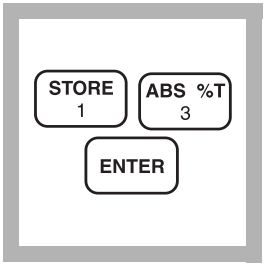


1. Enter the stored program number for hexavalent chromium (Cr^{6+})- powder pillows.

Press: **PRGM**

The display will show:

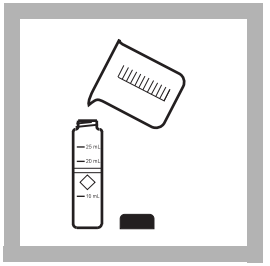
PRGM ?



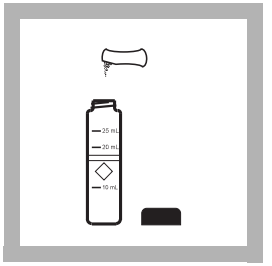
2. Press: **13 ENTER**

The display will show **mg/L, Cr6** and the **ZERO** icon**.**

Note: *For alternate forms (CrO_4, Cr_2O_7), press the* **CONC** *key.*

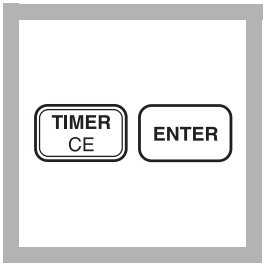


3. Fill a sample cell with 10 mL of sample.



4. Add the contents of one ChromaVer 3 Reagent Powder Pillow to the cell (the prepared sample). Cap the cell and invert several times to mix.

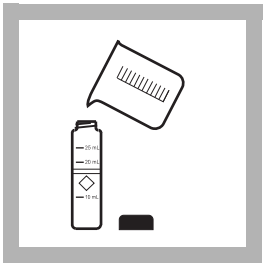
Note: *A purple color will form if Cr^{6+} is present.*



5. Press:

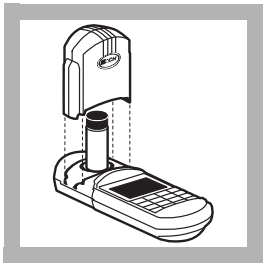
TIMER ENTER

A five-minute reaction period will begin.

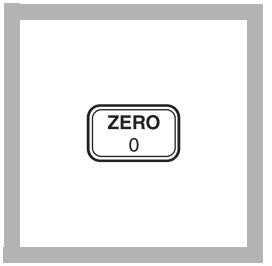


6. Fill another sample cell with 10 mL of sample (the blank).

Note: *For turbid samples, add the contents of one Acid Reagent Powder Pillow. This ensures turbidity dissolved by the acid in the ChromaVer 3 Chromium Reagent is also dissolved in the blank.*



7. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Cr6

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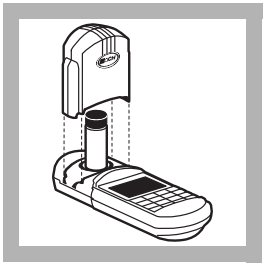
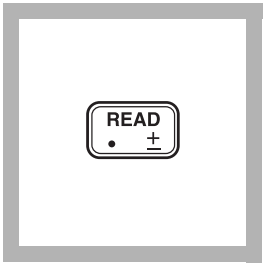
* Adapted from *Standard Methods for the Examination of Water and Wastewater*

** Procedure is equivalent to USGS method I-1230-85 for wastewater.



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

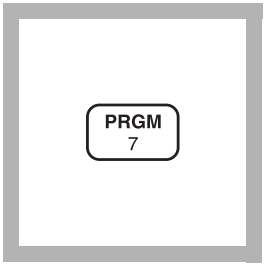


10. Press: **READ**

The cursor will move to the right, then the result in mg/L hexavalent chromium will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Using Accuvac Ampuls

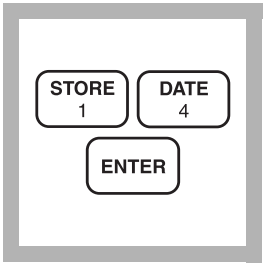


1. Enter the stored program number for hexavalent chromium (Cr^{6+})- AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?



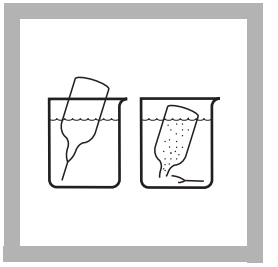
2. Press: **14 ENTER**

The display will show **mg/L, Cr6** and the **ZERO** icon**.**

Note: *For alternate forms (CrO_4, Cr_2O_7), press the* **CONC** *key.*

3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

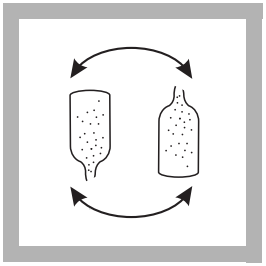
Note: *For turbid samples, add the contents of one Acid Reagent Powder Pillow to 10 mL of the blank. This ensures turbidity dissolved by the acid in the ChromaVer 3 Chromium Reagent is also dissolved in the blank.*



4. Fill a ChromaVer 3 Reagent AccuVac Ampul (the prepared sample) with sample.

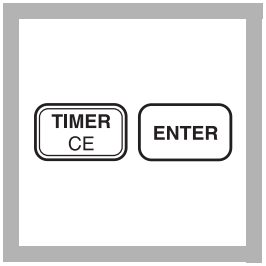
Note: *Keep the tip immersed while the ampul fills completely.*

Note: *ChromaVer 3 should be white to tan in color. Replace if it is brown or green.*



5. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

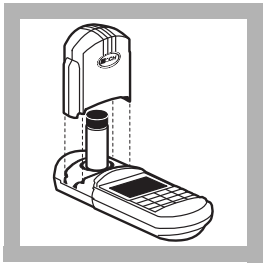
Note: *A purple color will form if hexavalent chromium is present.*



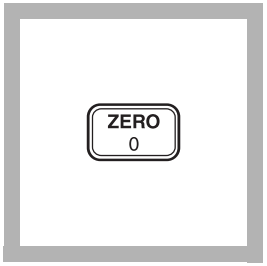
6. Press:

TIMER ENTER

A five-minute reaction period will begin.



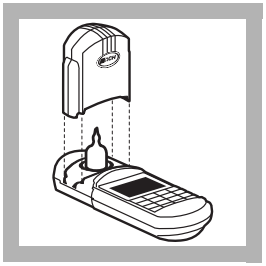
7. When the timer beeps place the blank into the cell holder.



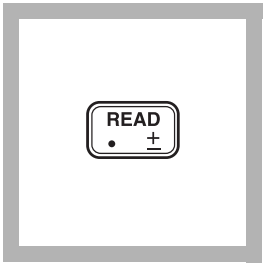
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Cr6



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L hexavalent chromium will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in a cleaned glass or plastic container. Store at 4 °C (39 °F) up to 24 hours. Samples must be analyzed within 24 hours.

Accuracy Check

Standard Additions Method (powder pillows)

a) Snap the neck off a Hexavalent Chromium PourRite Standard Ampule, 5 mg/L Cr^{6+}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 10-mL samples, respectively. Swirl to mix.

c) Analyze each sample as described above. The chromium concentration should increase 0.05 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method (AccuVac Ampuls)

a) Snap the neck off a Hexavalent Chromium Voluette Standard Ampule, 12.5 mg/L Cr^{6+}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 25-mL samples in beakers. Swirl gently to mix.

c) Analyze each sample as described above. The chromium concentration should increase 0.05 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 0.50-mg/L Cr^{6+} solution by pipetting 10.00 mL of Hexavalent Chromium Standard Solution, 50.0 mg/L Cr^{6+}, into a 1000-mL volumetric flask and diluting to the mark with deionized water. Invert repeatedly to mix. Prepare this solution daily. Perform the chromium procedure as described above, using this solution in place of the sample.

Method Performance

Precision

In a single laboratory using a standard solution of 0.6 mg/L Cr^{6+} and two representative lots of powder pillow reagent with the instrument, a single operator obtained a standard deviation of ±0.008 mg/L Cr^{6+}.

In a single laboratory using a standard solution of 0.6 mg/L Cr^{6+} and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.005 mg/L Cr^{6+}.

Estimated Detection Limit (EDL)

The EDL for program 13 (powder pillows) and program 14 (AccuVac Ampuls) is 0.01 mg/L Cr^{6+}. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

The following substances do not interfere in the test, up to the following concentration:

Substance	Concentration
Mercurous & Mercuric Ions	Interferes slightly
Iron	1 mg/L
Vanadium	1 mg/L. At higher levels vanadium interference can be overcome by waiting ten minutes before reading.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interference* in *Section 1*.

Summary of Method

Hexavalent chromium is determined by the 1,5-diphenylcarbohydrazide method using a single dry powder formulation called ChromaVer 3 Chromium Reagent. This reagent contains an acidic buffer combined with 1,5-diphenylcarbohydrazide, which reacts to give a purple color which is proportional to the amount of hexavalent chromium present.

REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
ChromaVer 3 Chromium Reagent Powder Pillows..	1 pillow	100/pkg	12710-99
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

REQUIRED REAGENTS AND APPARATUS (Using AccuVac Ampuls)

ChromaVer 3 AccuVac Ampuls	1 ampul	25/pkg	25050-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Description	Unit	Cat. No
Acid Reagent Powder Pillows	100/pkg	2126-99
Chromium, Hexavalent, Standard Solution, 50 mg/L Cr^{6+}	100 mL	810-42
Chromium, Hexavalent, Standard Solution, Voluette Ampule, 12.5 mg/L Cr^{6+}, 10 mL	16/pkg	14256-10
Chromium, Hexavalent, Standard Solution, PourRite Ampule, 5 mg/L Cr^{6+}, 2 mL	20/pkg	26056-20
Water, deionized	4 L	272-56

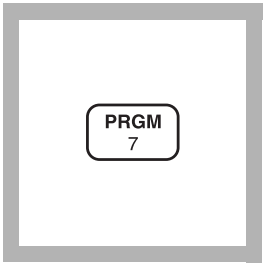
OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit	each	21968-00
Flask, volumetric, Class A, 1000 mL	each	14574-53
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 5.00 mL, Class A	each	14515-37
Pipet Filler, safety bulb	each	14651-00
PourRite Ampule Breaker, 2 mL	each	24846-00

In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

CHROMIUM, TOTAL (0 to 0.60 mg/L)

Alkaline Hypobromite Oxidation Method* **



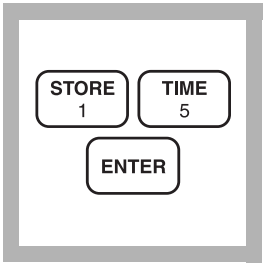
1. Enter the stored program number for total chromium (Cr).

Press: **PRGM**

The display will show:

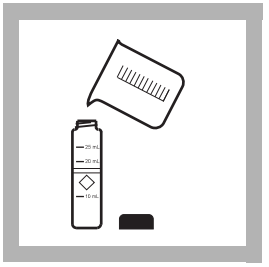
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



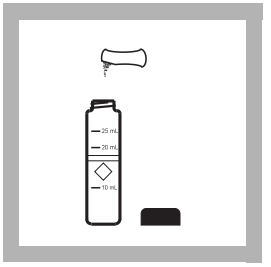
2. Press: **15 ENTER**

The display will show **mg/L, Cr** and the **ZERO** icon**.**

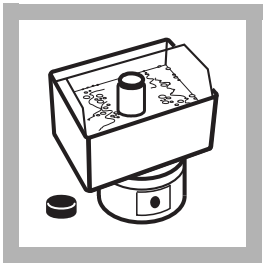


3. Fill a clean sample cell with 25 mL of sample.

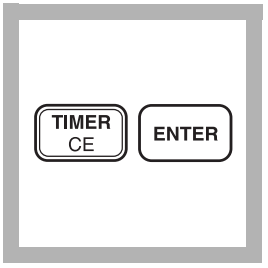
Note: *Adjust the pH of stored samples before analysis.*



4. Add the contents of one Chromium 1 Reagent Powder Pillow (the prepared sample). Cap the cell and invert repeatedly to mix. Remove the cap.



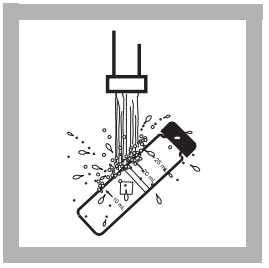
5. Place the prepared sample into a boiling water bath.



6. Press:

TIMER ENTER

A five-minute reaction period will begin.



7. After the beeper beeps, remove the prepared sample. Cap the cell. Use running tap water to cool the cell to 25 °C.

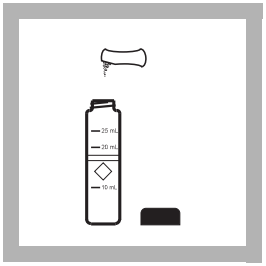
Note: *Use finger cots to handle the hot sample cell.*

8. Add the contents of one Chromium 2 Reagent Powder Pillow. Cap the cell and invert repeatedly to mix. Remove the cap.

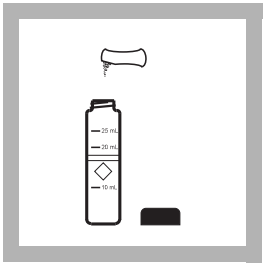
---

* Adapted from *Standard Methods for the Examination of Water and Wastewater*

** Procedure is equivalent to Standard Method 3500-Cr D for wastewater.



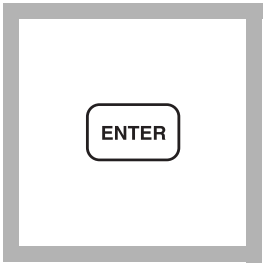
9. Add the contents of one Acid Reagent Powder Pillow. Cap the cell and invert repeatedly to mix. Remove the cap.



10. Add the contents of one ChromaVer 3 Chromium Reagent Powder Pillow. Cap the cell and invert repeatedly to mix.

Note: *A purple color will form if chromium is present.*

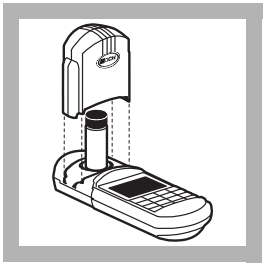
Note: *ChromaVer 3 is white to tan in color. Replace brown or green powder. Undissolved powder does not affect accuracy.*



11. The display will show: **05:00 TIMER 2**

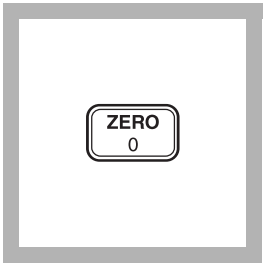
Press: **ENTER**

A five-minute reaction period will begin.



12. After the timer beeps, fill another sample cell with 25 mL of sample (the blank). Place it into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *For turbid samples, treat the blank as a sample, adding all reagents except the ChromaVer 3.*

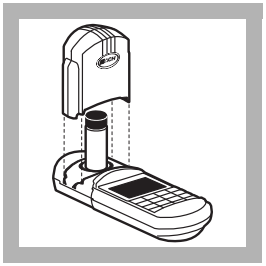


13. Press: **ZERO**

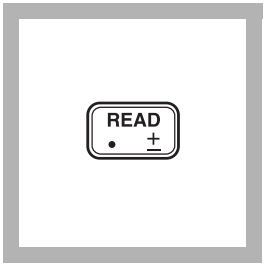
The cursor will move to the right, then the display will show:

0.00 mg/L Cr

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



14. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



15. Press: **READ**

The cursor will move to the right, then the result in mg/L total chromium (Cr) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-washed glass or plastic containers. To preserve samples, adjust the pH to 2 or lower with nitric acid (about 2 mL per liter). Store preserved samples at room temperature up to six months. Adjust the pH to about 4 with 5.0 N Sodium Hydroxide before analysis. Correct the test results for volume additions (see *Section 1*).

Accuracy Check

Standard Additions Method

a) Fill three sample cells with 25 mL of sample.

b) Snap the top off a Trivalent Chromium Standard Ampule, 12.5 mg/L as Cr^{3+}.

c) Use the TenSette pipet to add 0.1, 0.2, and 0.3 mL of standard to the three sample cells. Cap and invert repeatedly to mix .

d) Analyze each sample as described above. The chromium concentration should increase 0.05 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur see *Standard Additions* (*Section 1*).

Standard Solution Method

Prepare a 0.5 mg/L trivalent chromium standard by diluting 1.00 mL of Trivalent Chromium Standard Solution, 50 mg/L as Cr^{3+}, to 100 mL with deionized water. Mix thoroughly. Prepare this solution daily. Perform the chromium procedure as described above. The mg/L Cr reading should be 0.5 mg/L.

Method Performance

Precision

In a single laboratory using a standard solution of 0.4 mg/L trivalent chromium and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.004 mg/L chromium.

Estimated Detection Limit

The estimated detection limit for program 15 is 0.01 mg/L Cr. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Suggested Treatment
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interferences* in *Section 1*.
Large amounts of organic material	May inhibit complete oxidation of trivalent chromium. If high levels of organic material are present, see *Digestion* in *Section 2* for instruction on sample digestion. Perform the analysis as described on the digested sample.

Summary of Method

Trivalent chromium in the sample is oxidized to the hexavalent form by hypobromite ion under alkaline conditions. The sample is acidified. The total chromium content is determined by the 1,5-diphenylcarbohydrazide method. Determine trivalent chromium by subtracting the results of a separate hexavalent chromium test from the results of the total chromium test.

REQUIRED REAGENTS

	Cat. No.
Total Chromium Reagent Set (100 Tests)	22425-00
Includes: (1) 2126-99, (1) 12066-99, (1) 2043-99, (1) 2044-99	

Description	Quantity Required Per Test	Unit	Cat. No.
Acid Reagent Powder Pillows	1 pillow	100/pkg	2126-99
ChromaVer 3 Chromium Reagent Powder Pillows	1 pillow	100/pkg	12066-99
Chromium 1 Reagent Powder Pillows	1 pillow	100/pkg	2043-99
Chromium 2 Reagent Powder Pillows	1 pillow	100/pkg	2044-99

REQUIRED APPARATUS

Hot plate, 4" diameter, 120 V	1	each	12067-01
OR			
Hot plate, 4" diameter, 240 V	1	each	12067-02
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06
Water bath and rack	1	each	1955-55

OPTIONAL REAGENTS

Chromium, trivalent, Standard Solution, 50 mg/L Cr^{3+}		100 mL	14151-42
Chromium, trivalent, Standard Solution, PourRite ampule, 12.5 mg/L Cr^{3+}, 10 mL		16/pkg	14257-10
Nitric Acid, ACS		500 mL	152-49
Nitric Acid Solution 1:1		500 mL	2540-49
Sodium Hydroxide Standard Solution 5.0 N		50 mL* DB	2450-26
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Cylinder, graduated, polypropylene, 25 mL		each	1081-40
Finger Cots		2/pkg	14647-02
pH Paper, 1 to 11 pH units		5 rolls/pkg	391-33
pH Meter, sens*ion 1*, with electrode		each	51700-10
Pipet, serological, 2 mL		each	532-36
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet, volumetric, Class A, 1.00 mL		each	14515-35
Pipet Filler, safety bulb		each	14651-00
Ampule Breaker, 10-mL		each	21968-00

For Technical Assistance, Price and Ordering

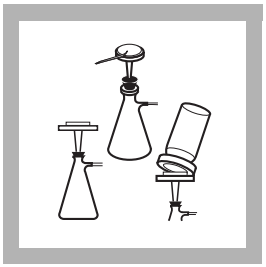
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes.

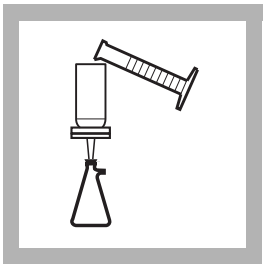
COLOR, TRUE AND APPARENT (0 to 500 units)

APHA Platinum-Cobalt Standard Method* For water, wastewater and seawater

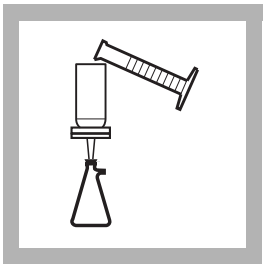


1. Assemble the filtering apparatus (membrane filter, filter holder, filter flask, and aspirator).

Note: *To test for apparent color, do not filter; begin at Step 4 and skip Step 7.*



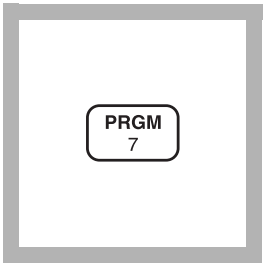
2. Rinse the filter by pouring about 50 mL of deionized water through the filter. Discard the rinse water.



3. Pour another 50 mL of deionized water through the filter. Keep this for Step 4.

4. Fill a sample cell (the blank) with 25 mL of filtered deionized water. Discard the excess.

Note: *For apparent color use unfiltered deionized water.*

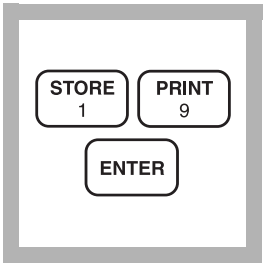


5. Enter the stored program number for APHA color.

Press: **PRGM**

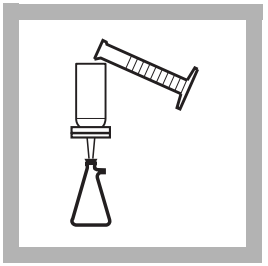
The display will show:

PRGM ?



6. Press: **19 ENTER**

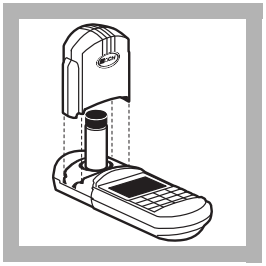
The display will show **PtCo** and the **ZERO** icon**.**



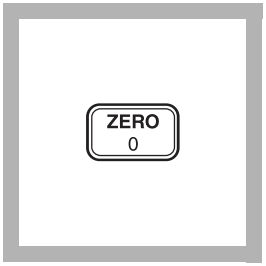
7. Pour about 50 mL of sample through the filter.

8. Fill a second sample cell (the prepared sample) with 25 mL of the filtered sample.

* Adapted from *Standard Methods for the Examination of Water and Wastewater*



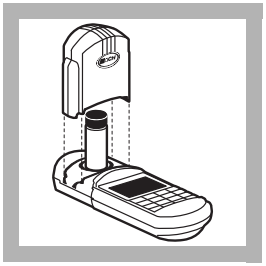
9. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



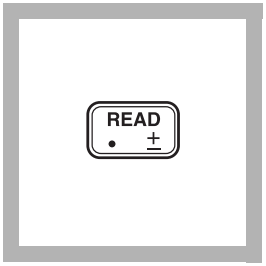
10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L Pt Co



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in Platinum-Cobalt color units (Pt-Co) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze the sample as soon as possible after collection for best results. If prompt analysis is impossible, fill bottles completely and cap tightly. Avoid excessive agitation or prolonged contact with air. Samples can be stored for 48 hours by cooling to 4 °C (39 °F). Warm to room temperature before running the test.

Accuracy Check

Standard Solution Method

A 500 Platinum-Cobalt Units Color Standard solution is available for checking test accuracy. A 250 Platinum-Cobalt Units Standard can be made by pipetting 50.0 mL of the 500 Platinum-Cobalt Units Standard into a 100-mL volumetric flask and diluting to volume with deionized water.

Method Performance

Precision

In a single laboratory, using a standard solution of 250 Pt-Co color units and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±10 Pt-Co color units. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program 19 is 25 Pt-Co color units. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

Color may be expressed as “apparent” or “true” color. The apparent color includes color from dissolved materials plus that from suspended matter. By filtering or centrifuging out the suspended materials, the true color can be determined. The procedure describes true color analysis. If apparent color is desired, it can be determined by measuring an unfiltered water sample. The stored program is used for both forms of color.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Units	Cat. No.
Water, deionized	50 mL	4 L	272-56

REQUIRED APPARATUS

Aspirator, vacuum	1	each	2131-00
Filter Holder, 47 mm, 300 mL graduated	1	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	1	100/pkg	13530-00
Flask, filtering, 500 mL	1	each	546-49
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
Stopper, No. 7, one hole	1	6/pkg	2119-07

OPTIONAL REAGENTS

Color Standard Solution, 500 platinum-cobalt units		1 L	1414-53

OPTIONAL APPARATUS

Cylinder, graduated, 50-mL, glass		each	508-41
Flask, volumetric, Class A, 100 mL		each	14574-42
Pipet, volumetric, Class A, 50 mL		each	14515-41
Thermometer, –20 to 110 °C, non-mercury		each	26357-02

For Technical Assistance, Price and Ordering

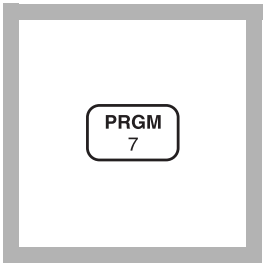
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

COPPER (0 to 5.00 mg/L)

For water, wastewater and seawater*

Bicinchoninate Method** (Powder Pillows or AccuVac Ampuls); USEPA approved for reporting wastewater analysis (digestion needed; See Section 2)*** Using Powder Pillows



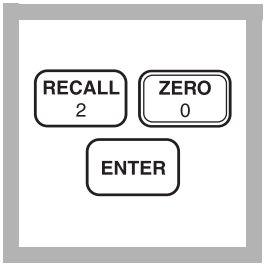
1. Enter the stored program number for bicinchoninate copper (Cu)- powder pillows.

Press: **PRGM**

The display will show:

PRGM ?

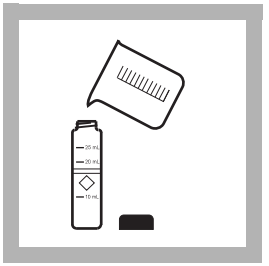
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **20 ENTER**

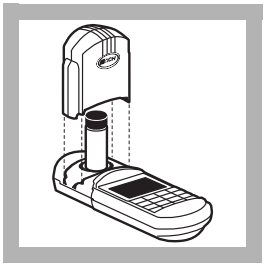
The display will show **mg/L, Cu** and the **ZERO** icon**.**

Note: *Determination of total copper needs a prior digestion (see Digestion in Section 2).*



3. Fill a sample cell with 10 mL of sample (the blank).

Note: *Adjust the pH of acid-preserved samples to 4-6 with 8 N KOH before analysis. Do not exceed pH 6 or copper may precipitate.*

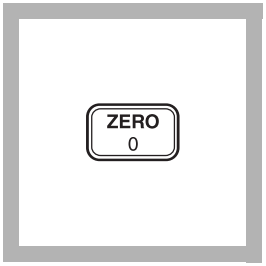


4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

* Pretreatment required; see *Interferences (Using Powder Pillows)*

** Adapted from Nakano, S., *Yakugaku Zasshi*, 82 486-491 (1962) [*Chemical Abstracts*, 58 3390e (1963)]

*** Powder Pillows only: *Federal Register*, 45 (105) 36166 (May 29, 1980)

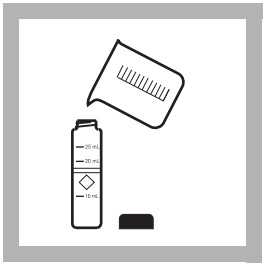


5. Press: **ZERO**

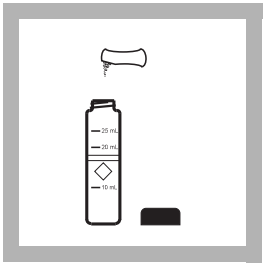
The cursor will move to the right, then the display will show:

0.00 mg/L Cu

Note: *If Reagent Blank Correction is on, the display may flash "limit".*

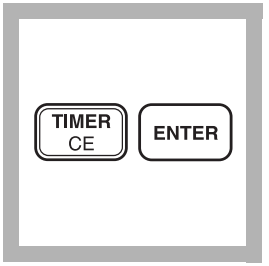


6. Fill another sample cell with 10 mL of the sample.



7. Add the contents of one CuVer 1 Copper Reagent Powder Pillow to the sample cell (the prepared sample). Swirl the cell to mix.

Note: *If copper is present, A purple color will develop.*

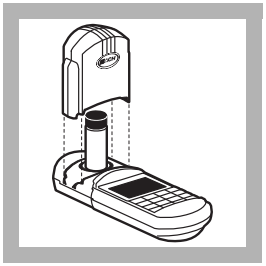


8. Press:

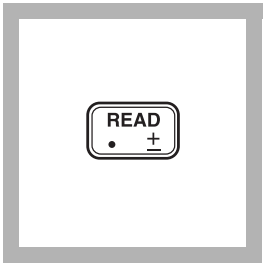
TIMER ENTER

A two-minute reaction period will begin.

Note: *Accuracy is not affected by undissolved powder.*



9. Within 30 minutes after the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



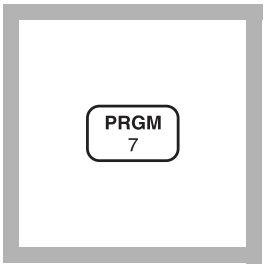
10. Press: **READ**

The cursor will move to the right, then the result in mg/L copper will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Using AccuVac Ampuls

Method 8026



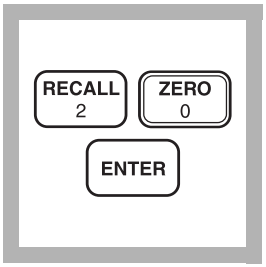
1. Enter the stored program number for bicinchoninate copper (Cu)- AccuVac ampuls.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



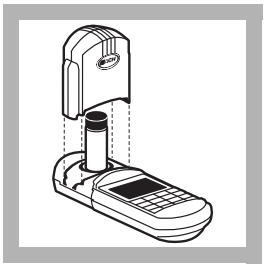
2. Press: **20 ENTER**

The display will show **mg/L, Cu** and the **ZERO** icon**.**

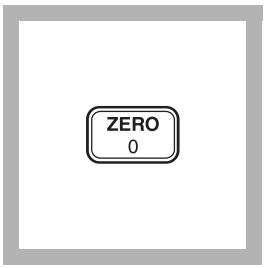
Note: *Determination of total copper needs a prior digestion (see Digestion in Section 2).*

Note: *Adjust the pH of stored samples before analysis.*

3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

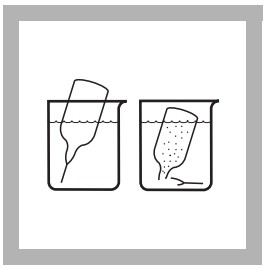


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

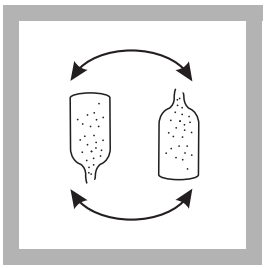
0.00 mg/L Cu

Note: *If Reagent Blank Correction is on, the display may flash "limit".*



6. Fill a CuVer 2 Copper Reagent AccuVac Ampul with sample.

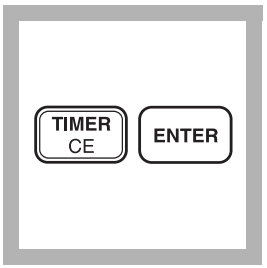
Note: *Keep the tip immersed while the ampul fills completely.*



7. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A purple color will form if copper is present.*

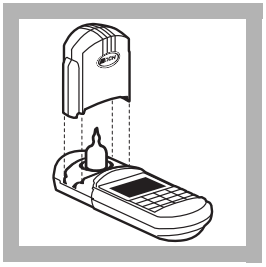
Note: *Accuracy is not affected by undissolved powder*



8. Press:

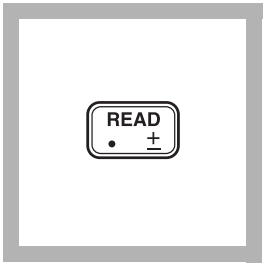
TIMER ENTER

A two-minute reaction period will begin.



9. After the timer beeps, place the AccuVac ampul in the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Step 10 must be completed within 30 minutes after the timer beeps.*



10. Press: **READ**

The cursor will move to the right, then the result in mg/L copper (Cu) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see in Section 1).*

Sampling and Storage

Collect samples in acid-cleaned glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Before analysis, adjust the pH to 4 to 6 with 8 N potassium hydroxide. Do not exceed pH 6, as copper may precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information. If only dissolved copper is to be determined, filter the sample before acid addition using the labware listed under *Optional Apparatus*.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Copper Voluette Ampule Standard, 75 mg/L as Cu.

c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to the mixing cylinders. Stopper and mix thoroughly.

d) For analysis with AccuVac Ampuls, transfer the solutions to dry, clean 50-mL beakers to fill the ampules. For analysis with powder pillows, transfer only 10 mL of the solution to 10-mL sample cells.

e) Analyze each sample as described in the procedure. The copper concentration should increase about 0.3 mg/L for each 0.1 mL of standard added.

f) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 1.00 mg/L copper standard by pipetting 1.00 mL of Copper Standard Solution, 100 mg/L as Cu, into 100-mL volumetric flask. Dilute to volume with deionized water and mix well. Prepare this solution daily. Using this solution as the sample, perform the copper procedure as described above.

Method Performance

Precision

In a single laboratory, using a standard solution of 2.25 mg/L Cu

and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.02 mg/L Cu.

In a single laboratory, using a standard solution of 2.25 mg/L Cu and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.02 mg/L Cu.

Estimated Detection Limit (EDL)
The EDL for program 20 (Powder Pillows and AccuVac Ampuls) is 0.02 mg/L Cu. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatments for Powder Pillows

Interfering Substance	Interference Level and Treatment
Acidity	If the sample is extremely acidic (pH 2 or less) a precipitate may form. Add 8 N Potassium Hydroxide Standard Solution drop-wise while swirling to dissolve the turbidity. Continue with Step 3.
Aluminum, Al^{3+}	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in Step 4. Results obtained will include total dissolved copper (free and complexed).
Cyanide, CN^-	Prevents full color development. Add 0.2 mL of formaldehyde to the 10-mL sample. Wait 4 minutes before taking the reading. Multiply the test results by 1.02 to correct for sample dilution by the formaldehyde.
Hardness	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in Step 4. Results obtained will include total dissolved copper (free and complexed).
Iron, Fe^{3+}	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in Step 4. Results obtained will include total dissolved copper (free and complexed).
Silver, Ag^+	If a turbidity remains and the precipitate turns black, silver interference is likely. Add 10 drops of saturated Potassium Chloride Solution to 75 mL of sample, followed by filtering through a fine or highly retentive filter. Use the filtered sample in the procedure.

To differentiate free copper from that complexed to EDTA or other complexing agents, use a Free Copper Reagent Powder

Pillow in place of the CuVer 1 pillow in Step 4. Results in Step 10 will be free copper only. Add a Hydrosulfite Reagent Powder Pillow to the same sample and re-read the result. This result will include the total dissolved copper (free and complexed).

Interfering Substances and Suggested Treatments for AccuVac Ampuls

Interfering Substance	Interference Level and Treatment
Acidity	If the sample is extremely acidic (pH 2 or less) a precipitate may form. Add 8 N Potassium Hydroxide Standard Solution drop-wise until sample pH is above 4. Continue with Step 3.
Aluminum, Al^{3+}	Reagents accommodate high levels.
Cyanide, CN^-	Prevents full color development. Add 1.0 mL of formaldehyde to a 50-mL sample. Wait 4 minutes before taking the reading. Multiply the test results by 1.02 to correct for sample dilution by the formaldehyde.
Hardness	Reagents accommodate high levels
Iron, Fe^{3+}	Reagents accommodate high levels
Silver, Ag^+	If a turbidity remains and the precipitate turns black, silver interference is likely. Add 10 drops of saturated Potassium Chloride Solution to 75 mL of sample, followed by filtering through a fine or highly retentive filter. Use the filtered sample in the procedure.

Unlike CuVer 1 Reagent, CuVer 2 Reagent reacts directly with copper which is complexed by chelants such as EDTA. If free copper is to be determined separately from complexed copper, see the Powder Pillow Interference section above.

Summary of Method

Copper in the sample reacts with a salt of bicinchoninic acid contained in CuVer 1 or 2 Copper Reagent to form a purple colored complex in proportion to the copper concentration. This method includes procedures for both powder pillow and AccuVac reagents.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
CuVer 1 Copper Reagent Powder Pillows	1 pillow	100/pkg	21058-69
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

CuVer 2 Copper Reagent AccuVac Ampuls	1 ampul	25/pkg	25040-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Copper Standard Solution, 100 mg/L		100 mL	128-42
Copper Standard Solution, Voluette Ampule, 75 mg/L Cu, 10 mL		16/pkg	14247-10
CuVer 2 Reagent Powder Pillows		100/pkg	21882-99
Formaldehyde, 37%, ACS		100 mL* MDB	2059-32
Free Copper Reagent Powder Pillows		100/pkg	21186-69
Hydrochloric Acid Solution, 6.0 N		500 mL	884-49
Hydrosulfite Reagent Powder Pillows		100/pkg	21188-69
Metals Drinking Water Standard, LR for Cu, Fe, Mn		500 mL	28337-49
Metals Drinking Water Standard, HR for Cu, Fe, Mn		500 mL	28336-49
Nitric Acid, ACS		500 mL	152-49
Nitric Acid Solution, 1:1		500 mL	2540-49
Potassium Chloride Solution, saturated		100 mL	765-42
Potassium Hydroxide Standard Solution, 8.0 N		100 mL* MDB	282-32H
Sodium Hydroxide Standard Solution, 5.0 N		100 mL* MDB	2450-32
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit	each	21968-00
Cylinder, graduated, mixing, 25 mL	each	20886-40
Cylinder, graduated, polypropylene, 25 mL	each	1081-40
Cylinder, graduated, 100 mL	each	508-42
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Filter Pump	each	2131-00
Flask, volumetric, 100 mL, Class A	each	14547-42
Funnel, polypropylene, 65 mm	each	1083-67
Hot Plate, 4" diameter, 120 V	each	12067-01
Hot Plate, 4" diameter, 240 V	each	12067-02
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, sens***ion 1***, with electrode	each	51700-10
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01

* Contact Hach for larger sizes.

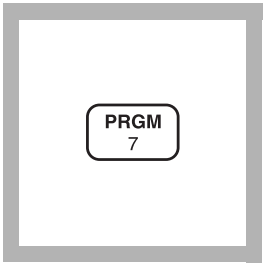
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

Porphyrin Method*

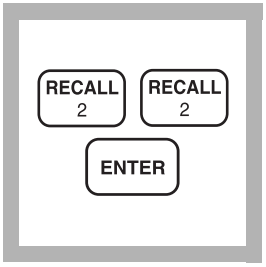


1. Enter the stored program number for copper (Cu), porphyrin method.

Press: **PRGM**

The display will show:

PRGM ?



2. Press: **22 ENTER**

The display will show **µg/L, Cu** and the **ZERO** icon**.**

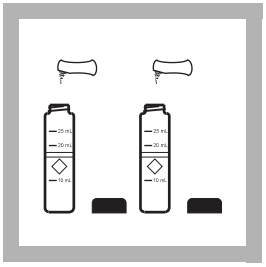
Note: *Total copper determination needs a prior digestion; use either the Digesdahl or vigorous digestion (Section 2).*

3. Fill two sample cells with 10 mL of sample.

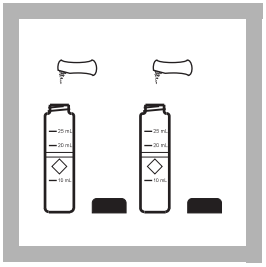
Note: *Wash all glassware with detergent. Rinse with tap water. Rinse again with Nitric Acid Solution, 1:1. Rinse a third time with copper-free, deionized water.*

4. Add the contents of one Copper Masking Reagent Powder Pillow to one of the sample cells (the blank). Swirl to dissolve.

Note: *The other sample cell is the prepared sample.*

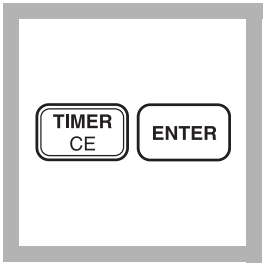


5. Add the contents of one Porphyrin 1 Reagent Powder Pillow to each sample cell. Swirl to dissolve the powder.



6. Add the contents of one Porphyrin 2 Reagent Powder Pillow to each sample cell. Swirl to dissolve the powder.

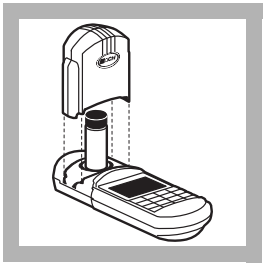
Note: *The yellow color will turn blue momentarily. If any copper is present, the yellow color will return.*



7. Press:

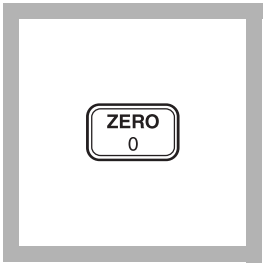
TIMER ENTER

A three-minute reaction period will begin.



8. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

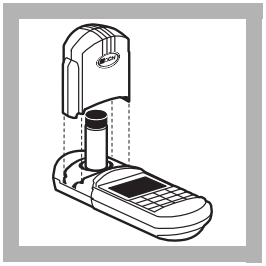
* Adapted from Ishii and Koh, *Bunseki Kagaku*, 28 473 (1979)



9. Press: **ZERO**

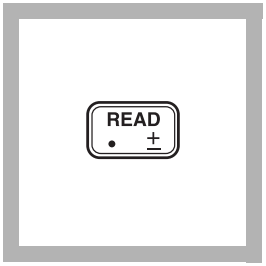
The cursor will move to the right, then the display will show:

0.0 µg/L Cu



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *If samples with high levels of metal are analyzed, a slight metallic deposit or yellow buildup may appear on the sample cell wall. Remove by rinsing with nitric acid. Dilute a fresh sample and repeat the test. Multiply the result by the dilution factor; see Section 1.*



11. Press: **READ** .

The cursor will move to the right, then the result in µg/L copper (Cu) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in acid-washed plastic bottles. To preserve, adjust the pH to 2 or less with nitric acid (about 5 mL per liter). Store preserved samples up to six months at room temperature.

Before testing, adjust the pH of the sample to between 2 and 6. If the sample is too acidic, adjust the pH with 5.0 N Sodium Hydroxide Standard Solution. Correct test results for volume additions; see *Correction for Volume Additions* in *Section 1* for more information.

Accuracy Check

Standard Additions Method

a) Fill six (3 pairs) 25-mL graduated mixing cylinders with 25 mL of sample. Properly mark each pair of cylinders as "sample" and "blank".

b) Using a TenSette Pipet, add 0.1 mL of Copper Standard Solution, 10.0 mg/L Cu, to two of the cylinders. Add 0.2 mL of standard to two more of the cylinders. Add 0.3 mL of standard to the other two cylinders, making a total of six samples (2 for each volume of standard).

c) Analyze the samples as described above. The copper concentration reading should increase by 40 µg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To assure the accuracy of the test, prepare a 100 µg/L copper standard:

a) Pipet 1.00 mL of Copper Standard Solution, 10.0 mg/L Cu, into a 100-mL volumetric flask.

b) Dilute to volume with copper-free, reagent-grade water.

c) Use this standard in place of the sample in the procedure. The reading should be 100 µg/L Cu.

d) Prepare this solution daily.

Method Performance

Precision

In a single laboratory, using a standard solution of 100 µg/L copper and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±3.4 µg/L copper.

Estimated Detection Limit

The estimated detection limit for program 22 is 5.4 µg/L Cu. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Substance	Concentration	Substance	Concentration
Aluminum	60 mg/L	Magnesium	10,000 mg/L
Cadmium	10 mg/L	Manganese	140 mg/L
Calcium	15,000 mg/L	Mercury	3 mg/L
Chloride	90,000 mg/L	Molybdenum	11 mg/L
Chromium (Cr^{6+})	110 mg/L	Nickel	60 mg/L
Cobalt	100 mg/L	Potassium	60,000 mg/L
Fluoride	30,000 mg/L	Sodium	90,000 mg/L
Iron (Fe^{2+})	6 mg/L	Zinc	9 mg/L
Lead	3 mg/L		

Chelating agents, such as EDTA, interfere at all levels unless either the Digesdahl or vigorous digestion (*Section 2*) is performed.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment: see pH Interferences in *Section 1*.

Summary of Method

The porphyrin method is very sensitive to trace amounts of free copper. Due to the sensitivity of the method, a masking agent is used to prepare a “blank” for each sample. The method is free from most interferences and does not require any sample extraction or preconcentration. Interferences from other metals are eliminated by the copper masking reagent. The porphyrin indicator forms an intense, yellow-colored complex proportional to any free copper present in the sample. Total copper may be determined if a digestion is performed prior to analysis.

REQUIRED REAGENTS

	Cat. No.
Copper Reagent Set, 10-mL samples (100 tests) Includes: (1) 26034-49, (2) 26035-49, (2) 26036-49	26033-00

Description	Quantity Required Per Test	Unit	Cat. No.
Copper Masking Reagent Powder Pillows	1 pillow	100/pkg	26034-49
Porphyrin 1 Reagent Powder Pillows	2 pillows	100/pkg	26035-49
Porphyrin 2 Reagent Powder Pillows	2 pillows	100/pkg	26036-49

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ caps	2	6/pkg	24019-06

OPTIONAL REAGENTS

Copper Standard Solution, 10 mg/L Cu	100 mL	128-42
Hydrochloric Acid Solution, 1:1 (6 N)	500 mL	884-49
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 5 N	1 L	2450-53
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Beaker, 100 mL	each	500-42H
Cylinder, mixing, graduated, 25 mL	each	20886-40
Flask, volumetric, Class A, 100 mL	each	14574-42
Hot Plate, 7 x 7 inches, 120 V	each	23441-00
Hot Plate, 7 x 7 inches, 240 V	each	23441-02
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable, with electrode	each	51700-10
Pipet, Mohr, 5 mL	each	20934-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet, volumetric, 1.0 mL, Class A	each	14515-35
Pipet Filler, safety bulb	each	14651-00
Watch Glass, Pyrex®, 100 mL	each	578-70

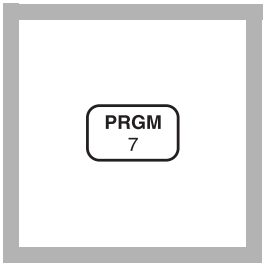
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

CYANIDE (0 to 0.240 mg/L)

Pyridine-Pyrazalone Method*



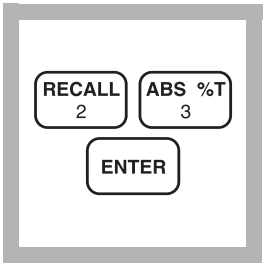
1. Enter the stored program number for cyanide (CN).

Press: **PRGM**

The display will show:

PRGM ?

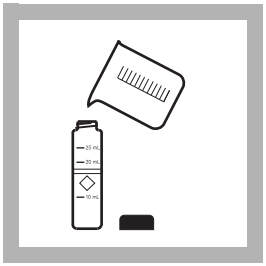
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **23 ENTER**

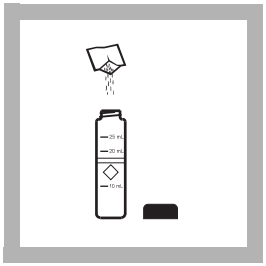
The display will show **mg/L, CN** and the **ZERO** icon**.**

Note: *Adjust the pH of stored samples before analysis.*



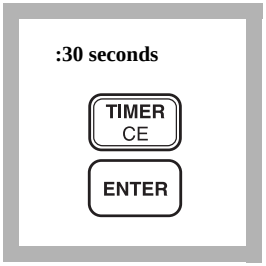
3. Fill a sample cell with 10-mL of sample.

Note: *Samples at less than 23 °C require a longer reaction time and samples at greater than 25 °C give low test results. Sample temperature must be 23-25 °C.*



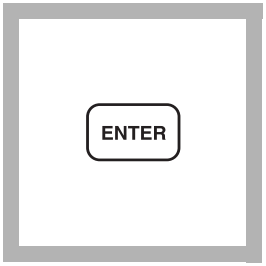
4. Add the contents of one CyaniVer 3 Cyanide Reagent Powder Pillow. Cap the sample cell.

* Adapted from Epstein, Joseph, *Anal. Chem*. 19 (4), 272 (1947)



5. Press: **TIMER ENTER**

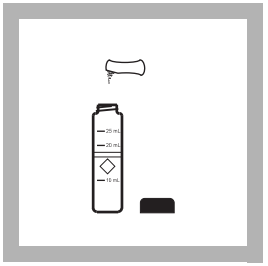
A 30-second reaction period will begin. Shake the sample cell for the 30 seconds.



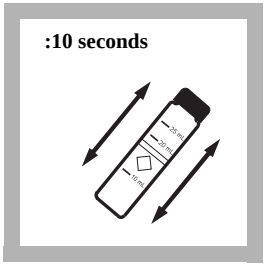
6. After the first timer beeps, the display will show: **0:30 TIMER 2**

Press **ENTER.**

A 30-second reaction period will begin. Let the sample cell sit undisturbed for this 30-second period.



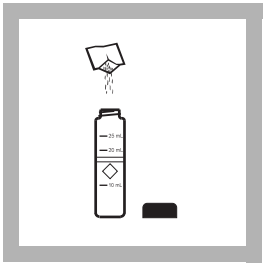
7. After the timer beeps, add the contents of one CyaniVer 4 Cyanide Reagent Powder Pillow. Cap the sample cell.



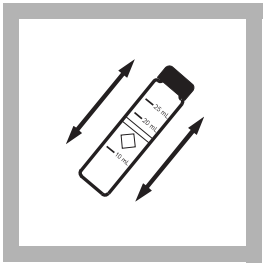
8. Shake the sample cell for ten seconds. Immediately proceed with Step 9.

Note: *Delaying the addition of the CyaniVer 5 Cyanide Reagent Powder for more than 30 seconds after the addition of the CyaniVer 4 Cyanide Reagent Powder will give lower test results.*

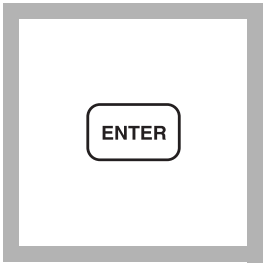
Note: *Accuracy is not affected by undissolved CyaniVer 4 Cyanide Reagent Powder.*



9. Add the contents of one CyaniVer 5 Cyanide Reagent Powder Pillow. Cap the cell.



10. Shake vigorously to completely dissolve the CyaniVer 5 Cyanide Reagent Powder (the prepared sample).



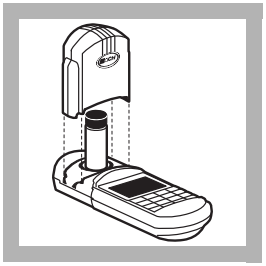
11. The display will show: **30:00 Timer 3**

Press: **ENTER**

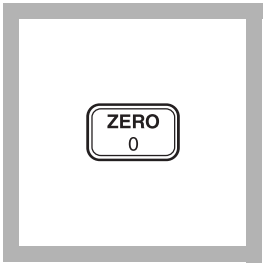
A 30-minute reaction period will begin.

Note: *If cyanide is present, a pink color will develop which then turns blue after a few minutes.*

12. Fill another 10-mL sample cell (the blank) with 10 mL of sample.



13. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

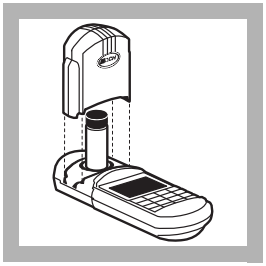


14. Press: **ZERO**

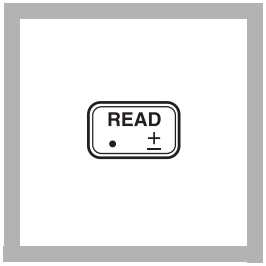
The cursor will move to the right, then the display will show:

0.000 mg/L CN

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



15. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



16. Press: **READ**

The cursor will move to the right, then the result in mg/L cyanide (CN) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in glass or plastic bottles and analyze as soon as possible.

The presence of oxidizing agents, sulfides and fatty acids can cause cyanide loss during sample storage. Samples containing these substances must be pretreated as described in the following procedures before preservation with sodium hydroxide. If the sample contains sulfide and is not pretreated, it must be analyzed within 24 hours.

Preserve the sample by adding 4.0 mL of 5.0 N Sodium Hydroxide Standard Solution to each liter (or quart) of sample, using a glass serological pipet and pipet filler. Check the sample pH. Four mL of sodium hydroxide are usually enough to raise the pH of most water and wastewater samples to 12. Add more 5.0 N sodium hydroxide if necessary. Store the samples at 4 °C (39 °F) or less. Samples preserved in this manner can be stored for 14 days.

Before testing, samples preserved with 5.0 N sodium hydroxide or samples that are highly alkaline due to chlorination treatment processes or distillation procedures should be adjusted to approximately pH 7 with 2.5 N Hydrochloric Acid Standard

Solution. If significant amounts of preservative are used, correct for the volume added; see *Correction for Volume Additions* in *Section 1* for more information.

Oxidizing Agents

Oxidizing agents such as chlorine decompose cyanides during storage. To test for their presence and eliminate their effect, pretreat the sample as follows:

a) Take a 25-mL portion of the sample and add one drop of m-Nitrophenol Indicator Solution, 10 g/L. Swirl to mix.

b) Add 2.5 N Hydrochloric Acid Standard Solution dropwise until the color changes from yellow to colorless. Swirl the sample thoroughly after the addition of each drop.

c) Add two drops of Potassium Iodide Solution, 30 g/L, and two drops of Starch Indicator Solution, to the sample. Swirl to mix. The solution will turn blue if oxidizing agents are present.

d) If Step c suggests the presence of oxidizing agents, add two level 1-g measuring spoonfuls of ascorbic acid per liter of sample.

e) Withdraw a 25-mL portion of sample treated with ascorbic acid and repeat Steps a to c. If the sample turns blue, repeat Steps d and e.

f) If the 25-mL sample remains colorless, adjust the remaining sample to pH 12 for storage with 5 N Sodium Hydroxide Standard Solution (usually 4 mL/L).

g) Perform the procedure given under Interferences, Reducing Agents, to eliminate the effect of excess ascorbic acid, before following the cyanide procedure.

Sulfides

Sulfides quickly convert cyanide to thiocyanate (SCN). To test for the presence of sulfide and eliminate its effect, pretreat the sample as follows:

a) Place a drop of sample on a disc of hydrogen sulfide test paper that has been wetted with pH 4 Buffer Solution.

b) If the test paper darkens, add a 1-g measuring spoon of lead acetate to the sample. Repeat Step a. (Purchase lead acetate from a local supplier.)

c) If the test paper continues to turn dark, keep adding lead acetate until the sample tests negative for sulfide.

d) Filter the black lead sulfide precipitate using the apparatus listed under Optional Apparatus. Preserve the sample for storage with 5 N Sodium Hydroxide Standard Solution or neutralize to a pH of 7 for analysis.

Fatty Acids

Caution—perform this operation in a hood as quickly as possible.

When distilled, fatty acids will pass over with cyanide and form soaps under the alkaline conditions of the absorber. If the presence of fatty acid is suspected, do not preserve samples with sodium hydroxide until the following pretreatment is performed. The effect of fatty acids can be minimized as follows:

a) Acidify 500 mL of sample to pH 6 or 7 with Acetic Acid Solution. (Prepare a 1:10 dilution of Acetate Acid concentration in water.)

b) Pour the sample into a 1000-mL separatory funnel and add 50 mL of hexane.

c) Stopper the funnel and shake for one minute. Allow the layers to separate.

d) Drain off the sample (lower) layer into a 600-mL beaker. If the sample is to be stored, add 5 N Sodium Hydroxide Standard Solution to raise the pH to above 12.

Accuracy Check

Standard Solution Method

Caution—Cyanides and their solutions, and the hydrogen cyanide liberated by acids, are very poisonous. Both the solutions and the gas can be absorbed through the skin.

Prepare a 100 mg/L cyanide stock solution weekly by dissolving 0.2503 grams of potassium cyanide in deionized water and diluting to 1000 mL.

Immediately before use, prepare a 0.10 mg/L cyanide working solution by diluting 1.00 mL of the 100 mg/L stock solution to 1000 mL using deionized water. Use this prepared standard in place of sample in Step 3. Results should be 0.10 mg/L CN^-.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.19 mg/L CN^- and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.017 mg/L CN^-.

Estimated Detection Limit (EDL)

The estimated detection limit for program 23 is 0.008 mg/L CN. For more information on the estimated detection limit, see *Section 1*.

Interferences

Turbidity

Large amounts of turbidity will interfere and cause high readings. If the water sample is highly turbid, it should first be filtered before use in Steps 3 and 12. Filter using the labware listed under Optional Apparatus. The test results should then be recorded as soluble cyanide.

Oxidizing and Reducing Agents

Large amounts of chlorine in the sample will cause a milky white precipitate after the addition of the CyaniVer 5 Reagent. If chlorine or other oxidizing agents are known to be present, or if reducing agents (such as sulfide or sulfur dioxide) are known to be present, use adequate ventilation and pretreat the sample before testing as follows:

Oxidizing Agents

a) Adjust a 25-mL portion of the alkaline sample to between pH 7 and 9 with 2.5 N Hydrochloric Acid Standard Solution. Count the number of drops of acid added.

b) Add two drops of Potassium Iodide Solution and two drops of Starch Indicator Solution to the sample. Swirl to mix. The sample will turn blue if oxidizing agents are present.

c) Add Sodium Arsenite Solution drop-wise until the sample turns colorless. Swirl the sample thoroughly after each drop. Count the number of drops.

d) Take another 25-mL sample and add the total number of drops of Hydrochloric Acid Standard Solution counted in Step a.

e) Subtract one drop from the amount of Sodium Arsenite Solution added in Step c. Add this amount to the sample and mix thoroughly.

f) Using 10 mL of this sample, continue with Step 3 of the cyanide procedure.

Reducing Agents

a) Adjust a 25-mL portion of the alkaline sample to between pH 7 and 9 with 2.5 N Hydrochloric Acid Standard Solution. Count the number of drops added.

b) Add four drops of Potassium Iodide Solution and four drops of Starch Indicator Solution to the sample. Swirl to mix. The sample should be colorless.

c) Add Bromine Water drop-wise until a blue color appears. Count the number of drops, and swirl the sample after the addition of each drop.

d) Take another 25 mL sample and add the total number of drops of Hydrochloric Acid Standard Solution counted in Step a.

e) Add the total number of drops of Bromine Water counted in Step c to the sample and mix thoroughly.

f) Using 10 mL of this sample, continue with Step 3 of the cyanide procedure.

Metals

Nickel or cobalt in concentrations up to 1 mg/L do not interfere. Eliminate the interference from up to 20 mg/L copper and 5 mg/L iron by adding the contents of one HexaVer Chelating Reagent Powder Pillow to the sample and then mixing before adding the CyaniVer 3 Cyanide Reagent Powder Pillow in Step 4. Prepare a reagent blank of deionized water and reagents to zero the instrument in Step 13.

Acid Distillation

For USEPA reporting purposes, samples must be distilled.

All samples should be treated by acid distillation except when experience has shown that there is no difference in results obtained with or without distillation. With most compounds, a one-hour reflux is adequate.

If thiocyanate is present in the original sample, a distillation step is absolutely necessary as thiocyanate causes a positive interference. High concentrations of thiocyanate can yield a substantial quantity of sulfide in the distillate. The “rotten egg” smell of hydrogen sulfide will accompany the distillate when sulfide is present. The sulfide must be removed from the distillate prior to testing.

If cyanide is not present, the amount of thiocyanate can be determined. The sample is not distilled and the final reading is multiplied by 2.2. The result is mg/L thiocyanate.

The distillate can be tested and treated for sulfide after the last step of the distillation procedure by using the following lead acetate
treatment procedure.

a) Place a drop of the distillate (already diluted to 250 mL) on a disc of hydrogen sulfide test paper that has been wetted with pH 4.0 Buffer Solution.

b) If the test paper darkens, add 2.5 N Hydrochloric Acid Standard Solution drop-wise to the distillate until a neutral pH is obtained.

c) Add a 1-g measuring spoon of lead acetate to the distillate and mix. Repeat Step a.

d) If the test paper continues to turn dark, keep adding lead acetate until the distillate tests negative for sulfide.

e) Filter the black lead sulfide precipitate through filter paper and funnel. This sample should now be neutralized to pH 7 and analyzed for cyanide without delay.

Distillation Procedures

A detailed procedure for the distillation of cyanide samples is included with the Hach Distillation Apparatus. Three detailed procedures, Free Cyanides, Cyanides Amenable to Chlorination, and Total Cyanides, are included with the four- and ten-position Midi-Dist Distillation System. See the Optional Apparatus listing.

Summary of Method

The pyridine-pyrazolone method gives an intense blue color with free cyanide. A sample distillation is required to determine cyanide from transition and heavy metal cyanide complexes.

REQUIRED REAGENTS

	Cat. No.
Cyanide Reagent Set (100 Tests), 10 mL samples	24302-00
Includes: (1) 21068-69, (1) 21069-69, (1) 21070-69	

Description	Quantity Required Per Test	Unit	Cat. No.
CyaniVer 3 Cyanide Reagent Powder Pillows	1 pillow	100/pkg	21068-69
CyaniVer 4 Cyanide Reagent Powder Pillows	1 pillow	100/pkg	21069-69
CyaniVer 5 Cyanide Reagent Powder Pillows	1 pillow	100/pkg	21070-69

REQUIRED APPARATUS

Sample Cell, 10-20-25, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Acetic Acid, Glacial	500 mL	100-49
Ascorbic Acid	100 g	6138-26
Bromine Water	25 mL	2211-20
Buffer Solution, pH 4.0	500 mL	12223-49
Hexanes, ACS	500 mL	14478-49
HexaVer Chelating Reagent Powder Pillows	100/pkg	243-99
Hydrochloric Acid Standard Solution, 2.5 N	100 mL MDB	1418-32
Magnesium Chloride Solution	1 L	14762-53
m-Nitrophenol Indicator	100 mL MDB	2476-32
Potassium Iodide Solution, 30 g/L	100 mL MDB	343-32
Sodium Arsenite Solution, APHA	100 mL MDB	1047-32
Potassium Cyanide, ACS	28 g	767-14
Sodium Hydroxide Standard Solution, 0.25 N	1 L	14763-53
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53

OPTIONAL REAGENTS (continued)

Description	Unit	Cat. No.
Starch Indicator Solution	10 mL MDB	349-32
Sulfuric Acid Standard Solution, 19.2 N	500 mL	2038-49
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Beaker, glass, 600 mL	each	500-52
Bottle, wash, 500 mL	each	620-11
Cylinder, graduated, 50 mL	each	508-41
Cylinder, graduated, 250 mL	each	508-46
Distillation Apparatus, cyanide accessories	each	22658-00
Distillation Apparatus, general purpose accessories	each	22653-00
Distillation Apparatus Heater and Support Apparatus, 115 Vac, 60 Hz	each	22744-00
Distillation Apparatus Heater and Support Apparatus, 230 Vac, 50 Hz	each	22744-02
Dropper, plastic	each	6080-00
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, volumetric, Class A, 1000 mL	each	14574-53
Flask, volumetric, Class A, 250 mL	each	14574-46
Funnel, poly, 65 mm	each	1083-67
Funnel, separatory, 500 mL	each	520-49
Hydrogen Sulfide Test Papers	100/pkg	25377-33
pH Meter, *sens**ion***™*1*, portable	each	51700-10
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00
Scoop, double ended	each	12257-00
Spoon, measuring, 1.0 g	each	510-00
Support Ring, 4 inch	each	580-01
Support Stand	each	563-00
Thermometer, –20 to 110 °C, non-mercury	each	26357-02

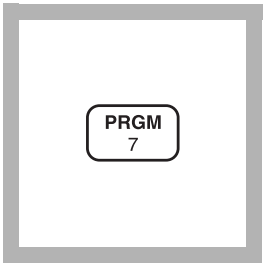
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

CYANURIC ACID (7 to 55 mg/L)

Turbidimetric Method



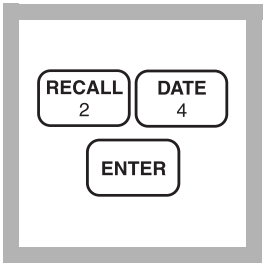
1. Enter the stored program number for cyanuric acid.

Press: **PRGM**

The display will show:

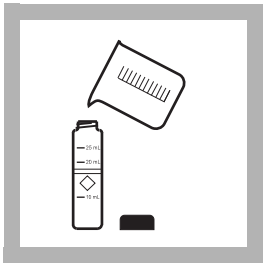
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



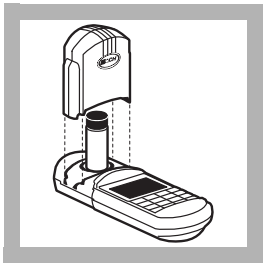
2. Press: **24 ENTER**

The display will show **mg/L, CYACD** and the **ZERO** icon.

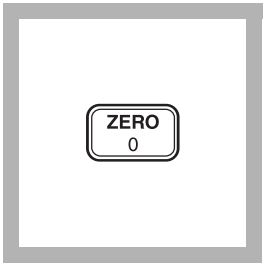


3. Fill a sample cell with 25 mL of sample (the blank).

Note: *Filtering is required for highly turbid samples.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

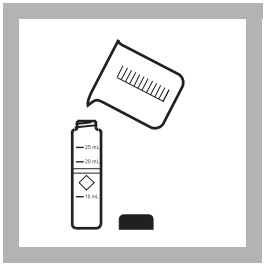


5. Press: **ZERO**

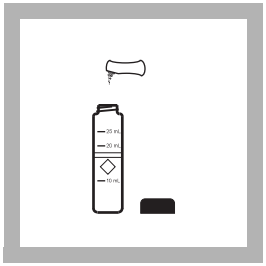
The cursor will move to the right, then the display will show:

0 mg/L CYACD

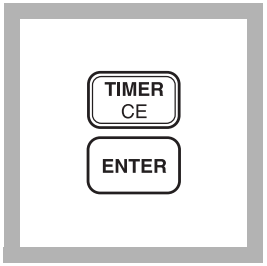
Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



6. Fill another cell with 25 mL of sample.



7. Add the contents of one Cyanuric Acid 2 Reagent Powder Pillow (the prepared sample). Swirl to mix.

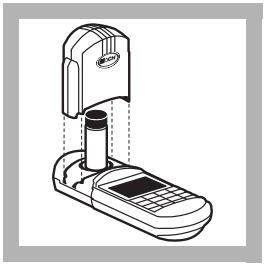


8. Press **TIMER ENTER**

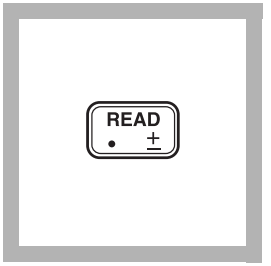
A three-minute reaction period will begin.

Note: *A white turbidity will form if cyanuric acid is present.*

Note: *Accuracy is not affected by undissolved powder.*



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L cyanuric acid will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Note: *Clean sample cells with soap, water and a brush soon after each test to prevent a white film from forming.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Samples must be analyzed within 24 hours.

Accuracy Check

Standard Solution Method

a) Dissolve 1.000 gram of cyanuric acid in 1000 mL of deionized water to make a 1000 mg/L solution. It takes several hours for the cyanuric acid to dissolve. This solution is stable for several weeks.

b) Dilute 2.00 mL of the 1000 mg/L solution to 100 mL with deionized water to make a 20 mg/L solution. Prepare fresh daily.

c) Testing the 20 mg/L solution should give test results of about 20 mg/L cyanuric acid.

Method Performance

Precision

In a single laboratory, using a standard solution of 25.0 mg/L cyanuric acid and two lots of reagent with the instrument, a single

operator obtained a standard deviation of ±1.2 mg/L cyanuric acid.

Estimated Detection Limit

The estimated detection limit for program 24 is 7.0 mg/L cyanuric acid. For more information on the estimated detection limit, see *Section 1*.

Interferences

Turbidity will interfere. Filter turbid samples before running the test.

Summary of Method

The test for cyanuric acid uses the turbidimetric method. Cyanuric Acid 2 Reagent precipitates any cyanuric acid present and holds it in suspension. The amount of turbidity caused by the suspended particles is directly proportional to the amount of cyanuric acid present. Due to the nature of the precipitation reaction, low levels of cyanuric acid (less than 7 mg/L) are not detected by this method.

REQUIRED REAGENTS AND APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Cyanuric Acid 2 Reagent Powder Pillow	1 pillow	50/pkg	2460-66
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Cyanuric Acid		25 g	7129-24
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Balance, Acculab UI Series		each	26947-00
Filter Paper, folded 12.5 cm		100/pkg	1894-57
Flask, volumetric, Class A, 100 mL		each	14574-42
Flask, volumetric, Class A, 1000 mL		each	14574-53
Funnel, poly, 65 mm		each	1083-67
Pipet, Bulb		each	14651-00
Pipet, volumetric, Class A, 2.00 mL		each	14515-36

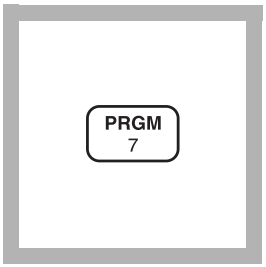
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

DEHA (0 to 500 µg/L)

Iron Reduction Method for Oxygen Scavengers*



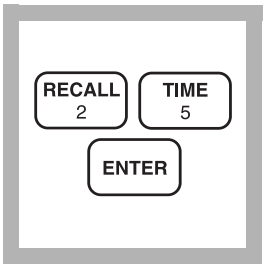
1. Enter the stored program number for diethylhydroxylamine (DEHA).

Press: **PRGM**

The display will show:

PRGM ?

Note: *To determine other oxygen scavengers, multiply the result by the appropriate factor. See* Other Oxygen Scavengers *following these steps.*

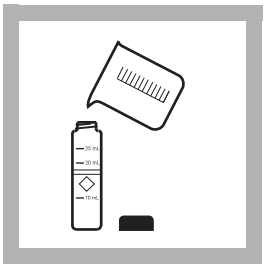


2. Press: **25 ENTER**

The display will show **µg/L, DEHA** and the **ZERO** icon**.**

Note: *To prevent contamination from iron deposits, rinse sampling containers and sample cells with 1:1 Hydrochloric Acid Solution. Follow with several rinsings of deionized water.*

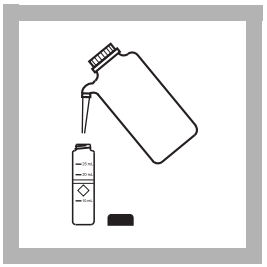
Note: *Samples must be analyzed immediately.*



3. Fill a sample cell with 25 mL of sample (the prepared sample).

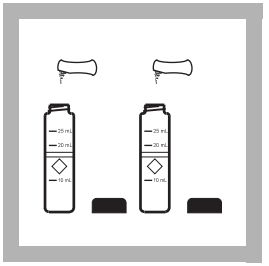
Note: *The sample temperature should be 25 ±3°C (77 ±5 °F).*

Note: *When testing for compounds that react quickly with oxygen at room temperature, stopper the cell containing the sample in Steps 5–11.*

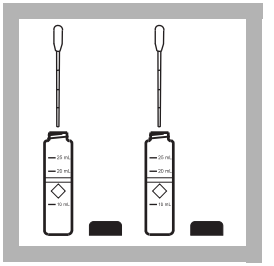


4. Fill a second sample cell with 25 mL of deionized water (the blank).

* Adapted from Ishii and Koh, *Buneseki Kagaku,* 28 473 (1979)

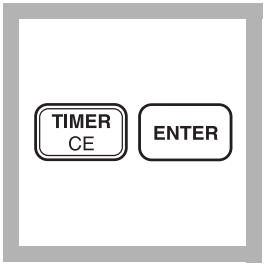


5. Add the contents of one DEHA Reagent 1 Powder Pillow to each sample cell. Cap. Swirl to mix.



6. Add exactly 0.5 mL of DEHA Reagent 2 Solution to each sample cell. Cap and swirl to mix. Place both sample cells in the dark.

Note: *A purple color will slowly develop if DEHA is present.*



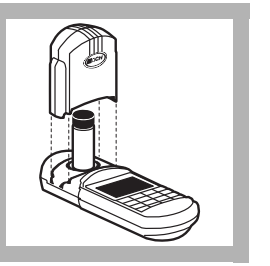
7. Immediately, press:

TIMER ENTER

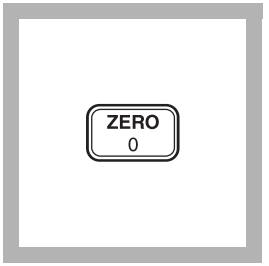
A 10-minute reaction period will begin. For hydroquinone, allow only a two-minute reaction period.

Note: *Both sample cells must remain in the dark for the entire reaction period.*

Note: *Temperature and reaction time affect results.*



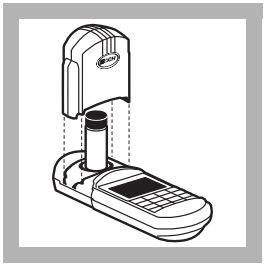
8. Immediately after the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



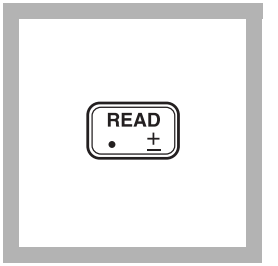
9. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 µg/L DEHA



10. Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in µg/L DEHA will be displayed.

Note: *If the display flashes "limit" it is due to high DEHA levels. Dilute a fresh sample with deoxygenated deionized water and repeat the test. Multiply the result by the dilution factor; see Section 1.*

Ferrous Iron Adjustment

Note: *Repeat the above procedure, but do not add DEHA Reagent 2 (Step 6) to determine the ferrous iron content in the sample. Then press SETUP, scroll to "BLANK" and press* **ENTER**. *The display will show; "BLANK?" Enter the blank value just read. Press* **ENTER** *to accept the value as the blank to be subtracted from each reading.*

Sampling and Storage

Most oxygen scavengers will react quickly with atmospheric oxygen. Collect samples in acid-rinsed plastic or glass containers, allowing the sample to overflow. Cap the container so there is no head space above the sample. Rinse each sample cell several times with sample, then carefully fill to the fill mark. Analyze the sample immediately.

Other Oxygen Scavengers

To determine other oxygen scavengers, perform the test as directed above, then multiply the DEHA result by the appropriate factor below:

Oxygen Scavenger	Factor
Erythorbic Acid (Iso-ascorbic acid)	3.5
Hydroquinone	2.5
Methylethylketoxime (MEKO)	4.1
Carbohydrazide	1.3

Method Performance

Precision

In a single laboratory, using a standard solution of 242 µg/L DEHA and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±6.2 µg/L DEHA.

Estimated Detection Limit

The estimated detection limit for program 25 is 9 µg/L DEHA. For more information on the estimated detection limit, see *Section 1*.

Interferences

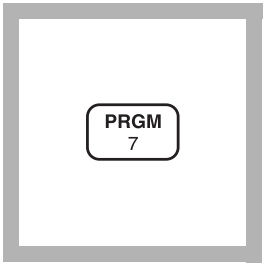
Substances which reduce ferric iron will interfere. Substances which complex iron strongly may also interfere. Light interferes with the color development. The following may also interfere when present in concentrations exceeding those listed below:

Borate (as $Na_2B_4O_7$)	500 mg/L	Molybdenum	80 mg/L
Cobalt	0.025 mg/L	Nickel	0.8 mg/L
Copper	8.0 mg/L	Phosphate	10 mg/L
Hardness (as $CaCO_3$)	1000 mg/L	Phosphonates	10 mg/L
Lignosulfonates	0.05 mg/L	Sulfate	1000 mg/L
Manganese	0.8 mg/L	Zinc	50 mg/L

FLUORIDE (0 to 2.00 mg/L F^-)

SPADNS Method* (Reagent Solution or AccuVac Ampuls)

Using SPADNS Reagent Solution

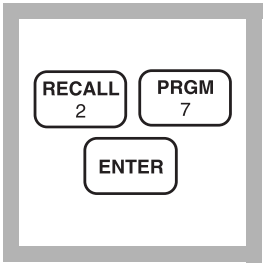


1. Enter the stored program number for fluoride (F) powder pillows.

Press: **PRGM**

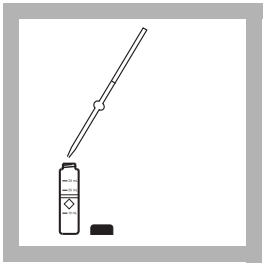
The display will show:

PRGM ?

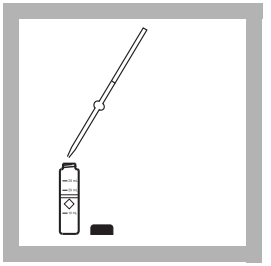


2. Press: **27 ENTER**

The display will show **mg/L, F** and the **ZERO** icon.

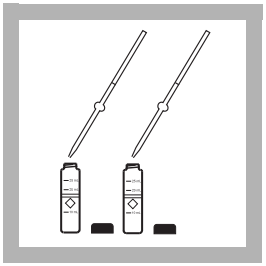


3. Pipet 10.0 mL of sample into a dry 10-mL sample cell (the prepared sample).



4. Measure 10.0 mL of deionized water into a second dry sample cell (the blank).

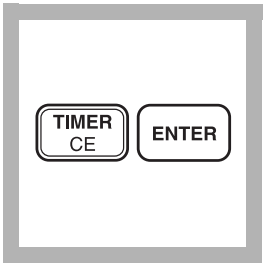
Note: *The sample and blank should be at the same temperature (±1 °C). Temperature adjustments may be made before or after reagent addition.*



5. Pipet 2.00 mL of SPADNS Reagent into each cell. Swirl to mix.

Note: *SPADNS Reagent is toxic and corrosive; use care while measuring. Use a pipet filler.*

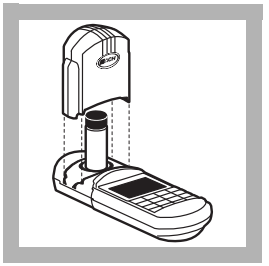
Note: *The SPADNS Reagent must be measured accurately.*



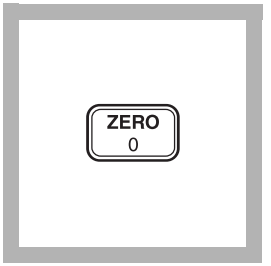
6. Press:

TIMER ENTER

A one minute reaction period will begin.



7. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L F

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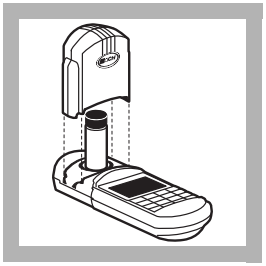
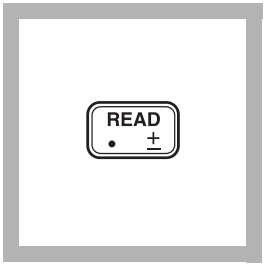
* Adapted from *Standard Methods for the Examination of Water and Wastewater. The procedure for this instrument uses an alternate wavelength outside the accepted 550-580 nm range. The reagents used are the same as those in the USEPA accepted method.*



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

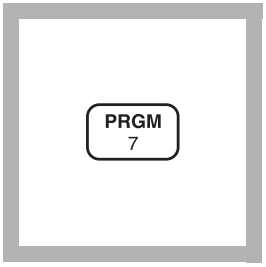


10. Press: **READ**

The cursor will move to the right, then the result in mg/L fluoride will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

Using AccuVac Ampuls

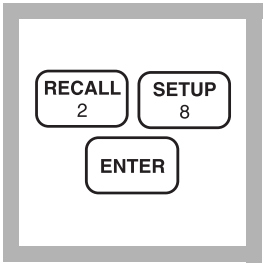


1. Enter the stored program number for fluoride (F^-)- AccuVac Ampuls.

Press: **PRGM**

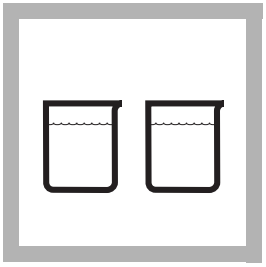
The display will show:

PRGM ?

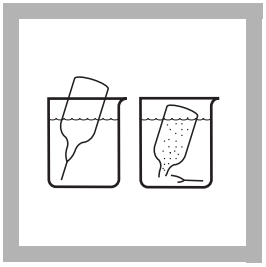


2. Press: **28 ENTER**

The display will show **mg/L, F** and the **ZERO** icon**.**

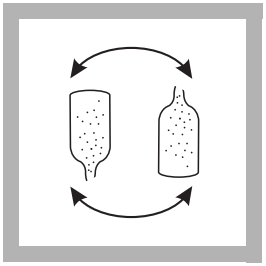


3. Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second beaker.



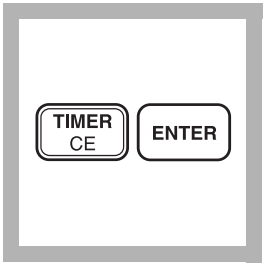
4. Fill a SPADNS Fluoride Reagent AccuVac Ampul with sample by breaking the tip on the bottom of the beaker. Fill a second AccuVac Ampul with deionized water (the blank).

Note: *Keep the tip immersed while the ampule fills completely.*



5. Quickly invert the ampules several times to mix. Wipe off any liquid or fingerprints.

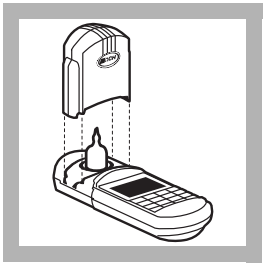
Note: *Do not place finger over the broken tip- the liquid will remain in the ampul.*



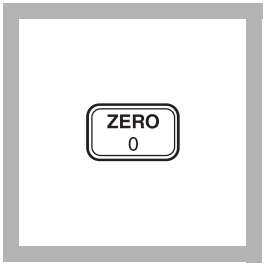
6. Press:

TIMER ENTER

A one-minute reaction period will begin.



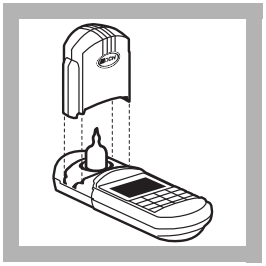
7. After the timer beeps place the blank into the cell holder. Tightly cover the ampule with the instrument cap.



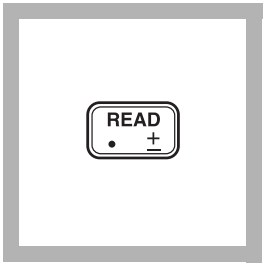
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L F



9. Place the AccuVac Ampul containing the sample into the instrument. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L fluoride will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

Sampling and Storage

Collect samples in plastic bottles. Samples may be stored up to 28 days.

Accuracy Check

Standard Solution Method

A variety of standard solutions covering the entire range of the test are available from Hach. Use these in place of sample to verify technique. Minor variations between lots of reagent become measurable above
1.5 mg/L. While results in this region are usable for most purposes, better accuracy may be obtained by diluting a fresh sample 1:1 with deionized water and retesting. Multiply the result by 2.

Standard Adjust

To adjust the calibration curve using the reading obtained with a 1.80-mg/L Standard Solution, press **SETUP** and use the arrow keys to scroll to the "STD" setup option. Press **ENTER** to activate the option. Then enter **1.80** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment* in *Section 1* for more information.

Method Performance

Precision

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS Reagent with the instrument, a single operator obtained standard deviations of ±0.035 mg/L fluoride.

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS AccuVac Reagent with the instrument, a single operator obtained standard deviations of ±0.040 mg/L fluoride.

Estimated Detection Limit (EDL)

The EDL for programs 27 and 28 is 0.05 mg/L F^-. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

This test is sensitive to small amounts of interference. Glassware must be very clean. Repeating the test with the same glassware is recommended to ensure that results are accurate.

The following substances interfere to the extent shown:

Substance	Concentration	Error
Alkalinity (as $CaCO_3$)	5000 mg/L	-0.1 mg/L F^-
Aluminum	0.1 mg/L	-0.1 mg/L F^-
Chloride	7000 mg/L	+0.1 mg/L F^-
Iron, ferric	10 mg/L	-0.1 mg/L F^-
Phosphate, ortho	16 mg/L	+0.1 mg/L F^-
Sodium Hexametaphosphate	1.0 mg/L	+0.1 mg/L F^-
Sulfate	200 mg/L	+0.1 mg/L F^-

SPADNS Reagent contains enough arsenite to eliminate interference up to 5 mg/L chlorine. For higher chlorine levels, add one drop of Sodium Arsenite Solution to 25 mL of sample for each 2 mg/L of chlorine.

To check for interferences from aluminum, read the concentration one minute after reagent addition, then again after 15 minutes. An appreciable increase in concentration suggests aluminum interference. Waiting two hours before making the final reading will eliminate the effect of up to 3.0 mg/L aluminum.

Most interferences can be eliminated by distilling the sample from an acid solution as described below:

a) Set up the distillation apparatus for the general purpose distillation. See the Hach Distillation Apparatus Manual. Turn on the water and make certain it is flowing through the condenser.

b) Measure 100 mL of sample into the distillation flask. Add a magnetic stirring bar and turn on the heater power switch.
Turn the stir control to 5.

c) Cautiously measure 150 mL of StillVer Distillation Solution (2:1 Sulfuric Acid) into the flask. If high levels of chloride are present, add 5 mg silver sulfate for each mg/L chloride present.

d) Turn the heat control to setting 10, with the thermometer in place. The yellow pilot lamp shows when the heater is on.

e) When the temperature reaches 180 °C (about one hour), turn the still off.

f) Dilute the collected distillate to 100 mL, if necessary. Analyze the distillate by the above method.

Summary of Method

The SPADNS Method for fluoride determination involves the reaction of fluoride with a red zirconium-dye solution. The fluoride combines with part of the zirconium to form a colorless complex, thus bleaching the red color in an amount proportional to the fluoride concentration. Seawater and wastewater samples require distillation. See Optional Apparatus for Distillation Apparatus listing.

Pollution Prevention and Waste Management

SPADNS Reagent contains sodium arsenite. Final solutions will contain sodium arsenite (D004) in sufficient concentration to be regulated as hazardous waste for Federal RCRA. See *Section 3* for more information on disposal of these materials.

REQUIRED REAGENTS (Using Solution)

Description	Quantity Required Per Test	Unit	Cat. No.
SPADNS Reagent for Fluoride	4 mL	500 mL	444-49
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS (Using Solution)

Pipet Filler safety bulb	1	each	14651-00
Pipet, volumetric, Class A, 10.00 mL	1	each	14515-38
Pipet, volumetric, Class A, 2.00 mL	1	each	14515-36
Sample Cell, 10-20-25 mL w/ cap	2	6/pkg	24019-06
Thermometer, –20 to 110°C, non-mercury	1	each	26357-02

REQUIRED REAGENTS (Using AccuVac Ampuls)

SPADNS Fluoride Reagent AccuVac Ampuls	2 ampuls	25/pkg	25060-25
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	2	each	500-41H

OPTIONAL REAGENTS

Drinking Water Inorganics Standard for F^-, NO_3^-, PO_4^{3-}, and SO_4^{2-}	500 mL		28330-49
Fluoride Standard Solution, 0.2 mg/L F^-	500 mL		405-02
Fluoride Standard Solution, 0.5 mg/L F^-	500 mL		405-05
Fluoride Standard Solution, 0.8 mg/L F^-	500 mL		405-08
Fluoride Standard Solution, 1.0 mg/L F^-	1000 mL		291-53
Fluoride Standard Solution, 1.0 mg/L F^-	500 mL		291-49
Fluoride Standard Solution, 1.2 mg/L F^-	500 mL		405-12
Fluoride Standard Solution, 1.5 mg/L F^-	500 mL		405-15
Fluoride Standard Solution, 2.0 mg/L F^-	500 mL		405-20
Silver Sulfate, ACS	113 g		334-14
Sodium Arsenite Solution	100 mL MDB		1047-32
StillVer Distillation Solution	500 mL		446-49

OPTIONAL APPARATUS

AccuVac Snapper Kit		each	24052-00
Cylinder, graduated, 100 mL		each	508-42
Cylinder, graduated, 250 mL		each	508-46
Distillation Heater and Support Apparatus Set, 115 V, 50/60 Hz		each	22744-00
Distillation Heater and Support Apparatus Set, 230 V, 50/60 Hz		each	22744-02
Distillation Apparatus General Purpose Accessories		each	22653-00
pH Meter, *sens**ion***™***1,** portable, with electrode		each	51700-10
Pipet, TenSette, 1.0 to 10.0 mL		each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet		50/pkg	21997-96
Stopper		6/pkg	1731-06

For Technical Assistance, Price and Ordering

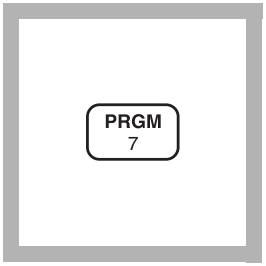
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

FLUORIDE (0 to 2.00 mg/L F^-)

SPADNS 2 Method* (Reagent Solution or AccuVac Ampuls)

Using SPADNS 2 Reagent Solution

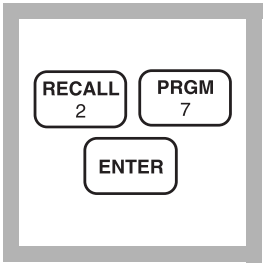


1. Enter the stored program number for fluoride (F^-) powder pillows.

Press: **PRGM**

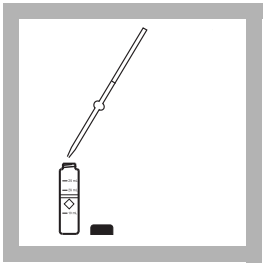
The display will show:

PRGM ?

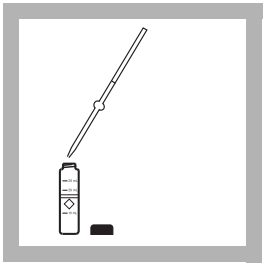


2. Press: **27 ENTER**

The display will show **mg/L, F** and the **ZERO** icon.

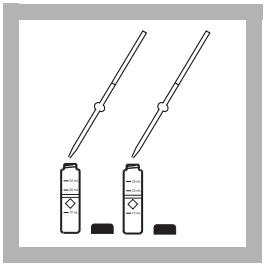


3. Pipet 10.0 mL of sample into a dry 10-mL sample cell (the prepared sample).



4. Measure 10.0 mL of deionized water into a second dry sample cell (the blank).

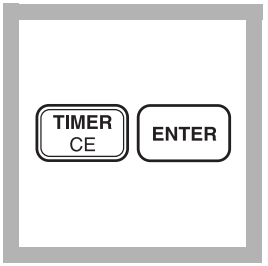
Note: *The sample and blank should be at the same temperature (±1 °C). Temperature adjustments may be made before or after reagent addition.*



5. Pipet 2.00 mL of SPADNS 2 Reagent into each cell. Swirl to mix.

Note: *SPADNS 2 Reagent is corrosive; use care while measuring. Use a pipet filler.*

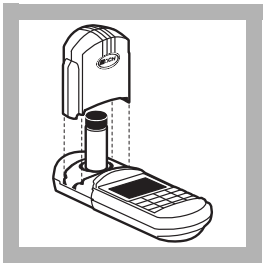
Note: *The SPADNS 2 Reagent must be measured accurately.*



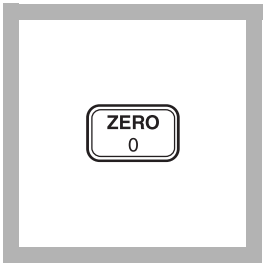
6. Press:

TIMER ENTER

A one minute reaction period will begin.



7. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



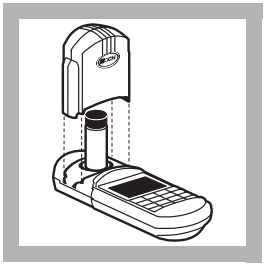
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

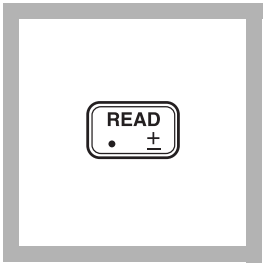
0.00 mg/L F

---

* Adapted from *Standard Methods for the Examination of Water and Wastewater. Per USEPA Rules and Regulations at 40 CFR 136.6, Method Modifications and Analytical Requirements, Hach Method 10225 (SPADNS 2) for the determination of fluoride in water is equivalent to the EPA Reference Method SM 4500-F D. Equivalency data is available upon request.*



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

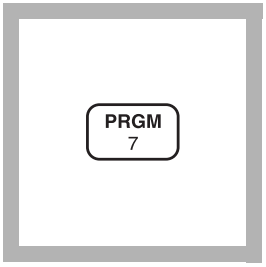


10. Press: **READ**

The cursor will move to the right, then the result in mg/L fluoride will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

Using AccuVac Ampuls

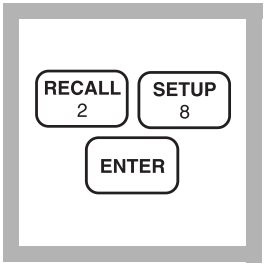


1. Enter the stored program number for fluoride (F^-) AccuVac Ampuls.

Press: **PRGM**

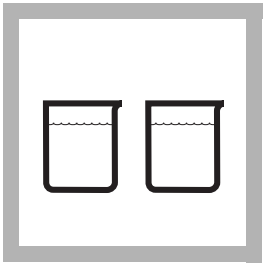
The display will show:

PRGM ?

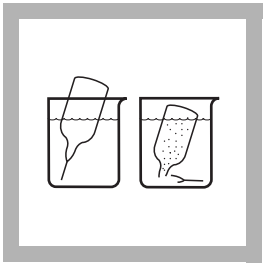


2. Press: **28 ENTER**

The display will show **mg/L, F** and the **ZERO** icon**.**

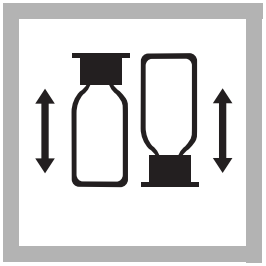


3. Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second beaker.

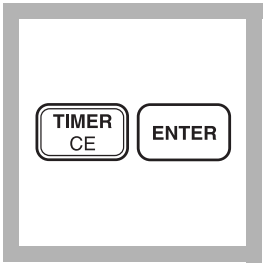


4. Fill an SPADNS 2 Fluoride Reagent AccuVac Ampul with sample by breaking the tip on the bottom of the beaker. Fill a second AccuVac Ampul with deionized water (the blank).

Note: *Keep the tip immersed while the ampule fills completely.*



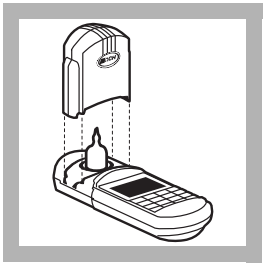
5. Cap and quickly invert the ampules several times to mix. Wipe off any liquid or fingerprints.



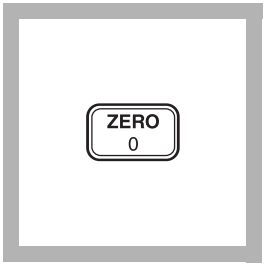
6. Press:

TIMER ENTER

A one-minute reaction period will begin.



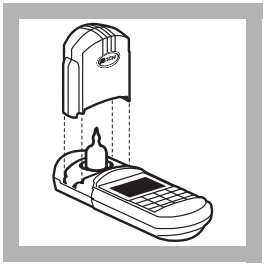
7. After the timer beeps place the blank into the cell holder. Tightly cover the ampule with the instrument cap.



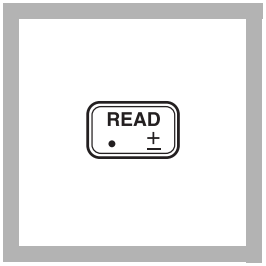
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L F



9. Place the AccuVac Ampul containing the sample into the instrument. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L fluoride will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

Sampling and Storage

Collect samples in plastic bottles. Samples may be stored up to 28 days.

Accuracy Check

Standard Solution Method

A variety of standard solutions covering the entire range of the test are available from Hach. Use these in place of sample to verify technique. Minor variations between lots of reagent become measurable above
1.5 mg/L. While results in this region are usable for most purposes, better accuracy may be obtained by diluting a fresh sample 1:1 with deionized water and retesting. Multiply the result by 2.

Standard Adjust

To adjust the calibration curve using the reading obtained with a 1.80-mg/L Standard Solution, press **SETUP** and use the arrow keys to scroll to the "STD" setup option. Press **ENTER** to activate the option. Then enter **1.80** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment* in *Section 1* for more information.

Method Performance

Precision

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS 2 Reagent with the instrument, a single operator obtained standard deviations of ±0.035 mg/L fluoride.

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS 2 AccuVac Reagent with the instrument, a single operator obtained standard deviations of ±0.040 mg/L fluoride.

Estimated Detection Limit (EDL)

The EDL for programs 27 and 28 is 0.05 mg/L F^-. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

This test is sensitive to small amounts of interference. Glassware must be very clean. Repeating the test with the same glassware is recommended to ensure that results are accurate.

The following substances interfere to the extent shown:

Substance	Concentration	Error
Alkalinity (as $CaCO_3$)	5000 mg/L	-0.1 mg/L F^-
Aluminum	0.1 mg/L	-0.1 mg/L F^-
Chloride	7000 mg/L	+0.1 mg/L F^-
Iron, ferric	10 mg/L	-0.1 mg/L F^-
Phosphate, ortho	16 mg/L	+0.1 mg/L F^-
Sodium Hexametaphosphate	1.0 mg/L	+0.1 mg/L F^-
Sulfate	200 mg/L	+0.1 mg/L F^-

SPADNS 2 Reagent contains enough non-toxic reducing agent to eliminate interference up to 5 mg/L chlorine. For higher chlorine levels, dilute sample with deionized water by a factor that will lower chlorine concentration to below 5 mg/L. Perform the procedure, and multiply results by this factor to obtain mg/L Fluoride.

To check for interferences from aluminum, read the concentration one minute after reagent addition, then again after 15 minutes. An appreciable increase in concentration suggests aluminum interference. Waiting two hours before making the final reading will eliminate the effect of up to 3.0 mg/L aluminum.

Most interferences can be eliminated by distilling the sample from an acid solution as described below:

a) Set up the distillation apparatus for the general purpose distillation. See the Hach Distillation Apparatus Manual. Turn on the water and make certain it is flowing through the condenser.

b) Measure 100 mL of sample into the distillation flask. Add a magnetic stirring bar and turn on the heater power switch.
Turn the stir control to 5.

c) Cautiously measure 150 mL of StillVer Distillation Solution (2:1 Sulfuric Acid) into the flask. If high levels of chloride are present, add 5 mg silver sulfate for each

mg/L chloride present.

d) Turn the heat control to setting 10, with the thermometer in place. The yellow pilot lamp shows when the heater is on.

e) When the temperature reaches 180 °C (about one hour), turn the still off.

f) Dilute the collected distillate to 100 mL, if necessary. Analyze the distillate by the above method.

Summary of Method

The SPADNS 2 Method for fluoride determination involves the reaction of fluoride with a red zirconium-dye solution. The fluoride combines with part of the zirconium to form a colorless complex, thus bleaching the red color in an amount proportional to the fluoride concentration. Seawater and wastewater samples require distillation. See Optional Apparatus for Distillation Apparatus listing.

Pollution Prevention and Waste Management

SPADNS 2 Reagent contains a non-toxic proprietary reducing agent in place of sodium arsenite.

REQUIRED REAGENTS (Using Solution)

Description	Quantity Required Per Test	Unit	Cat. No.
SPADNS 2 Reagent for Fluoride	4 mL	500 mL	29475-49
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS (Using Solution)

Pipet Filler safety bulb	1	each	14651-00
Pipet, volumetric, Class A, 10.00 mL	1	each	14515-38
Pipet, volumetric, Class A, 2.00 mL	1	each	14515-36
Sample Cell, 10-20-25 mL w/ cap	2	6/pkg	24019-06
Thermometer, –20 to 110°C, non-mercury	1	each	26357-02

REQUIRED REAGENTS (Using AccuVac Ampuls)

SPADNS 2 Fluoride Reagent AccuVac Ampuls	2 ampuls	25/pkg	25270-25
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	2	each	500-41H

OPTIONAL REAGENTS

Drinking Water Inorganics Standard for F^-, NO_3^-, PO_4^{3-}, and SO_4^{2-}	500 mL	28330-49
Fluoride Standard Solution, 0.2 mg/L F^-	500 mL	405-02
Fluoride Standard Solution, 0.5 mg/L F^-	500 mL	405-05
Fluoride Standard Solution, 0.8 mg/L F^-	500 mL	405-08
Fluoride Standard Solution, 1.0 mg/L F^-	1000 mL	291-53
Fluoride Standard Solution, 1.0 mg/L F^-	500 mL	291-49
Fluoride Standard Solution, 1.2 mg/L F^-	500 mL	405-12
Fluoride Standard Solution, 1.5 mg/L F^-	500 mL	405-15
Fluoride Standard Solution, 2.0 mg/L F^-	500 mL	405-20
Silver Sulfate, ACS	113 g	334-14
StillVer Distillation Solution	500 mL	446-49

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Cylinder, graduated, 100 mL	each	508-42
Cylinder, graduated, 250 mL	each	508-46
Distillation Heater and Support Apparatus Set, 115 V, 50/60 Hz	each	22744-00
Distillation Heater and Support Apparatus Set, 230 V, 50/60 Hz	each	22744-02
Distillation Apparatus General Purpose Accessories	each	22653-00
pH Meter, *sens**ion***™***1,** portable, with electrode	each	51700-10
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet	50/pkg	21997-96
Stopper	6/pkg	1731-06

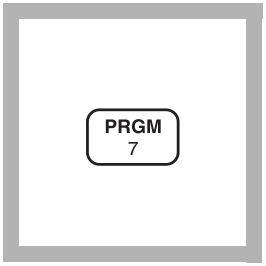
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

HARDNESS (0 to 4.00 mg/L Ca and Mg as $CaCO_3$) For water, wastewater, seawater

Calcium and Magnesium; Calmagite Colorimetric Method



1. Enter the stored program number for magnesium hardness (as $CaCO_3$).

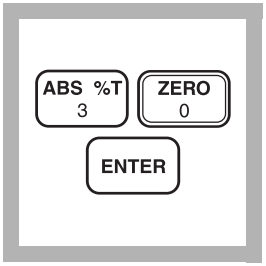
Press: **PRGM**

The display will show:

PRGM ?

Note: *Adjust the pH of stored samples before analysis.*

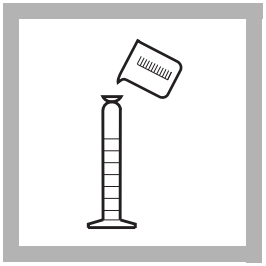
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **30 ENTER**

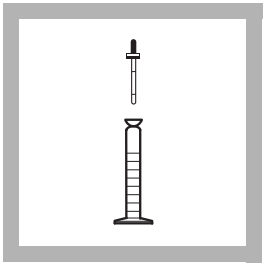
The display will show **mg/L, CaCO3** and the **ZERO** icon**.**

Note: *For alternate forms (Mg, $MgCO_3$), press the* **CONC** *key.*

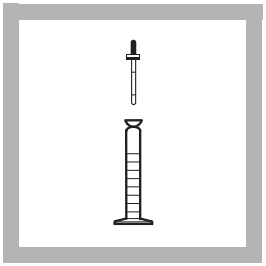


3. Pour 100 mL of sample into a 100-mL graduated mixing cylinder.

Note: *The sample temperature should be 21-29 °C (70-84 °F).*

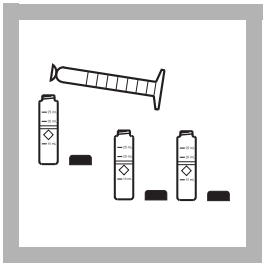


4. Add 1.0 mL of Calcium and Magnesium Indicator Solution using a 1.0-mL measuring dropper. Stopper. Invert several times to mix.



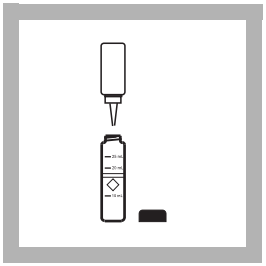
5. Add 1.0 mL of Alkali Solution for Calcium and Magnesium Test using a 1.0-mL measuring dropper. Stopper. Invert several times to mix.

Note: *If the sample turns read after adding Alkali Solution, dilute sample 1:1 and repeat analysis.*

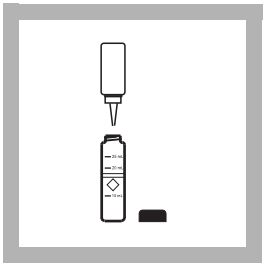


6. Pour 10 mL of the solution into each of three sample cells.

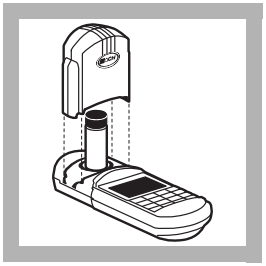
Note: *The test will detect any calcium or magnesium contamination in the mixing cylinder, measuring droppers or sample cells. To test cleanliness, repeat the test multiple times until you obtain consistent results.*



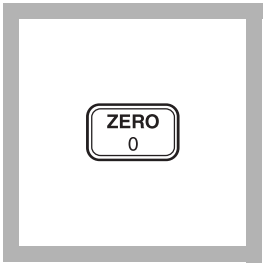
7. Add one drop of 1 M EDTA Solution to one cell (the blank). Swirl to mix.



8. Add one drop of EGTA Solution to another cell (the prepared sample). Swirl to mix.



9. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

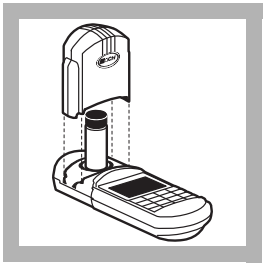


10. Press: **ZERO**

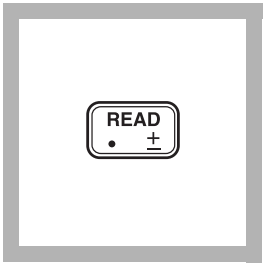
The cursor will move to the right, then the display will show:

0.00 mg/L CaCO3

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

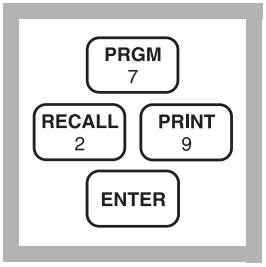


11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L magnesium hardness (as $CaCO_3$) will be displayed.



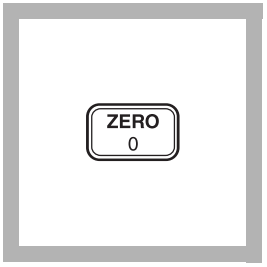
13. Without removing the cell, press:

PRGM 29 ENTER

The display will show:

PRGM ?

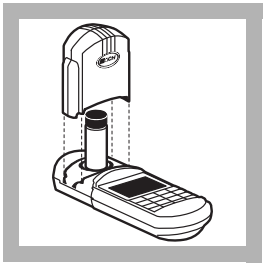
Note: *For alternate forms (Ca) press the* **CONC** *key.*



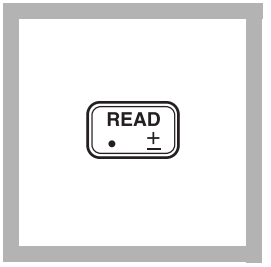
14. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L CaCO3



15. Place the third sample cell into the cell holder.



16. Press: **READ**

The cursor will move to the right, then the result in mg/L calcium hardness (as $CaCO_3$) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Note: *mg/L total hardness = mg/L Ca as $CaCO_3$ + mg/L Mg as $CaCO_3$.*

Sampling and Storage

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 5 mL per liter). Preserved samples can be stored up to six months. Adjust the sample pH to

between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution just before analysis. Correct the test results for volume additions; see *Correction for Volume Additions* in *Section 1* for more information.

Accuracy Check

Using a 2.00 mg/L (as $CaCO_3$) standard solution as sample, perform the hardness procedure described above. The results should be 2.00 mg/L calcium (as $CaCO_3$).

Method Performance

Precision

In a single laboratory using a standard solution of 2.00 mg/L Mg as $CaCO_3$ and 1.88 mg/L Ca as $CaCO_3$ with the instrument, a single operator obtained a standard deviation of ± 0.09 mg/L Mg as $CaCO_3$ and ± 0.08 mg/L Ca as $CaCO_3$.

Estimated Detection Limit

The estimated detection limit for program 30 is 0.13 mg/L magnesium hardness and 0.08 mg/L calcium hardness. For more information on the estimated detection limit, see *Section 1*.

Interferences

For the most accurate hardness test result, the test should be rerun on a diluted sample if the calcium is over 1.0 or the magnesium is over
0.25 mg/L as $CaCO_3$. No retesting is needed if either is below those respective concentrations.

The following cause a detectable error in test results.

Interfering Substance	Level at Which Substance Interferes
Cr^{3+}	0.25 mg/L
Cu^{2+}	0.75 mg/L
EDTA, chelated	0.2 mg/L as $CaCO_3$
Fe^{2+}	1.4 mg/L
Fe^{3+}	2.0 mg/L
Mn^{2+}	0.20 mg/L
Zn^{2+}	0.050 mg/L

Traces of EDTA or EGTA remaining in sample cells from previous tests will give erroneous results. Rinse cells thoroughly before use.

Summary of Method

The colorimetric method for measuring hardness supplements the conventional titrimetric method because it can measure very low levels of calcium and magnesium. Also some interfering metals (those listed above) in the titrimetric method are inconsequential in the colorimetric method when diluting the sample to bring it within the range of this test.

The indicator dye, calmagite, forms a purplish-blue color in a strongly alkaline solution and changes to red when it reacts with free calcium or magnesium. Calcium is chelated with EGTA to destroy any red color due to calcium and then the sample is chelated with EDTA to destroy the red color due to both calcium and magnesium. Measuring the red color in the different stages of chelation gives results as the calcium and magnesium hardness concentrations.

REQUIRED REAGENTS

	Cat. No.
Hardness Reagent Set (100 Tests)	23199-00
Includes: (1) 22417-32, (1) 22418-32, (1) 22419-26, (1) 22297-26	

Description	Quantity Required Per Test	Unit	Cat. No.
Alkali Solution for Calcium and Magnesium Test	1 mL	100 mL MDB	22417-32
Calcium and Magnesium Indicator Solution	1 mL	100 mL MDB	22418-32
EDTA Solution, 1 M	1 drop	50 mL	22419-26
EGTA Solution	1 drop	50 mL	22297-26

REQUIRED APPARATUS

Cylinder, 100-mL mixing	1	each	1896-42
Dropper, measuring, 0.5 and 1.0 mL	2	20/pkg	21247-20
Sample Cell, 10-20-25 mL, w/cap	3	6/pkg	24019-06

OPTIONAL REAGENTS

Calcium Standard Solution, 2.0 mg/L as $CaCO_3$		946 mL	20581-16
Nitric Acid, ACS		500 mL	152-49
Nitric Acid Solution, 1:1		500 mL	2540-49
Sodium Hydroxide Standard Solution 5.0 N		100 mL MDB	2450-32

OPTIONAL APPARATUS

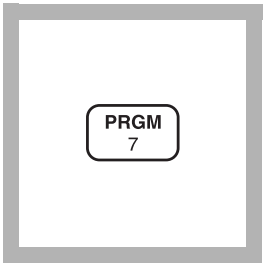
pH Meter, *sens**ion***™*1*, portable, with electrode		each	51700-10
Thermometer, –20 to 110 °C		each	26357-02

HYDRAZINE (0 to 500 µg/L)

For boiler water/feedwater, water and seawater

p-Dimethylaminobenzaldehyde Method*

Using Reagent Solution



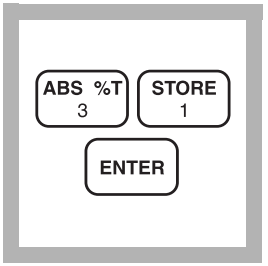
1. Enter the stored program number for hydrazine (N_2H_4).

Press: **PRGM**

The display will show:

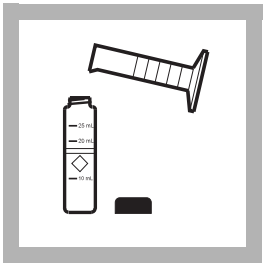
PRGM ?

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

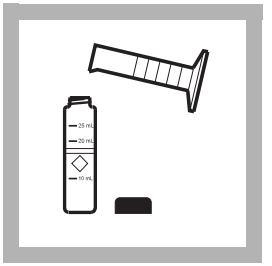


2. Press: **31 ENTER**

The display will show **µg/L, N2H4** and the **ZERO** icon**.**

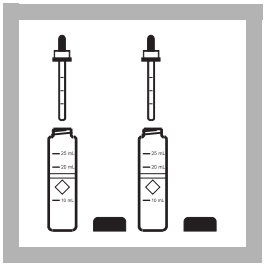


3. Pour 10.0 mL of deionized water into a sample cell (the blank) using a graduated cylinder.

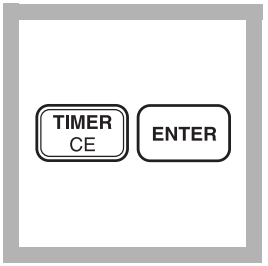


4. Pour 10.0 mL of sample into a second sample cell (the sample) using a graduated cylinder.

Note: *The sample temperature should be 21 ± 4 °C (70 ± 7 °F).*



5. Add 0.5 mL of HydraVer 2 Hydrazine Reagent to each sample cell. Cap. Invert to mix.



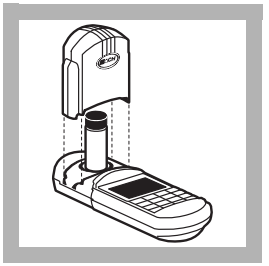
6. Press:

TIMER ENTER

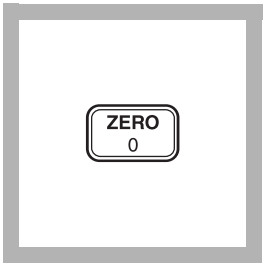
A 12-minute reaction period will begin.

Note: *Complete Steps 7-9 within 3 minutes.*

Note: *A yellow color will form if hydrazine is present. The blank will be a faint yellow color due to the HydraVer 2 reagent.*



7. Immediately after the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

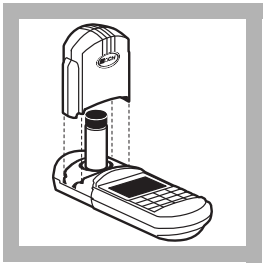


8. Press: **ZERO**

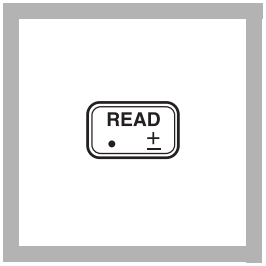
The cursor will move to the right, then the display will show:

0 µg/L N2H4

* Adapted from ASTM Manual of Industrial Water, D1385-78, 376 (1979)



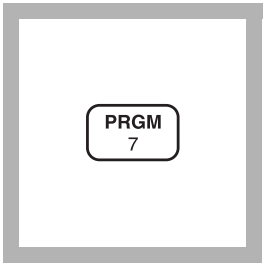
9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in µg/L hydrazine will be displayed.

Using AccuVac Ampuls



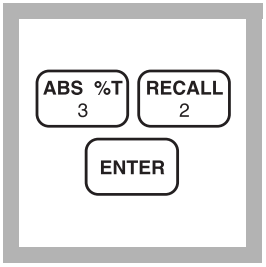
1. Enter the stored program number for hydrazine (N_2H_4)-AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

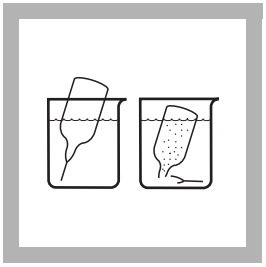
Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



2. Press: **32 ENTER**

The display will show **µg/L, N2H4** and the **ZERO** icon.

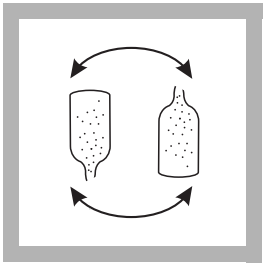
3. Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second 50-mL beaker.



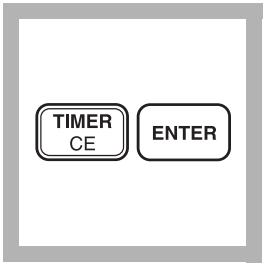
4. Fill a Hydrazine AccuVac Ampul with sample. Fill a second Hydrazine AccuVac Ampul with deionized water (the blank).

Note: *Keep the tip immersed while the ampul fills completely.*

Note: *The sample temperature should be 21 ± 4 °C (70 ± 7 °F).*



5. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.



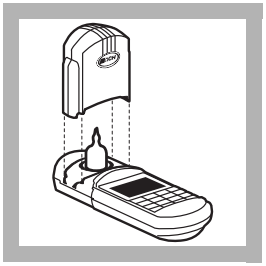
6. Press:

TIMER ENTER

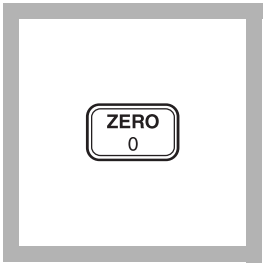
A 12-minute reaction period will begin.

Note: *Complete Steps 7-9 during this period.*

Note: *A yellow color will develop if hydrazine is present. The blank will be a faint yellow color due to the reagent.*



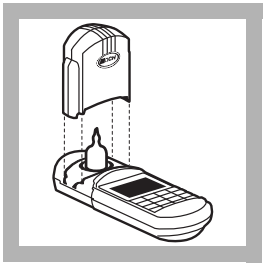
7. Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



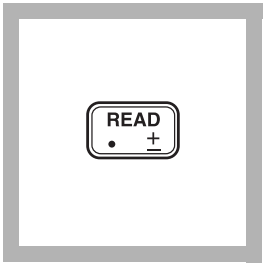
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 µg/L N2H4



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Immediately after the timer beeps, press **READ.**

The cursor will move to the right, then the result in µg/L hydrazine will be displayed.

Sampling and Storage

Collect samples in glass or plastic containers. Fill the containers completely and cap them tightly. Avoid excessive agitation or exposure to air. Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

Accuracy Check

Standard Solution Method

To assure the accuracy of the test, prepare the following solutions:

a) Prepare a 25 mg/L hydrazine stock solution by dissolving 0.1016 g of hydrazine sulfate in 1000 mL of oxygen-free deionized water. Use Class A glassware. Prepare this stock solution daily.

b) Prepare a 100 µg/L hydrazine working solution by diluting 4.00 mL of the 25 mg/L stock solution to 1000 mL with deionized oxygen-free water. Prepare just before analysis.

c) Use the working solution in place of the sample in Step 4. The result should be 100 µg/L hydrazine.

Method Performance

Precision

In a single laboratory using a standard solution of 250 µg/L hydrazine (N_2H_4) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±9 µg/L hydrazine.

In a single laboratory using a standard solution of 250 µg/L hydrazine (N_2H_4) and two lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±3 µg/L hydrazine.

Estimated Detection Limit

The estimated detection limit for program 31 is 16 µg/L N_2H_4, and the estimated detection limit for program 32 is 10 µg/L N_2H_4. For more information on the estimated detection limit, see *Section 1*.

Interferences

For highly colored or turbid samples, prepare a blank by oxidizing the hydrazine in a portion of the sample. This can be accomplished with a
1:1 mixture of deionized water and household bleach. Add two drops of this mixture to 40 mL of sample contained in a graduated mixing cylinder and invert to mix. Use this solution in Step 3, in place of deionized water, to prepare the blank.

Ammonia has no effects up to 10 mg/L ammonia. At 20 mg/L, a

positive interference occurs.

Morpholine does not interfere up to 10 mg/L.

Summary of Method

Hydrazine reacts with the p-dimethylaminobenzaldehyde from the HydraVer 2 Reagent to form a yellow color which is proportional to the hydrazine concentration.

REQUIRED REAGENTS (Using Reagent Solution)

Description	Quantity Required Per Test	Unit	Cat. No.
HydraVer 2 Hydrazine Reagent	1 mL	100 mL MDB	1790-32
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS (Using Reagent Solution)

Cylinder, graduated, 25 mL	1	each	508-40
Sample Cells, 10-, 20- and 25 mL, w/ caps	2	6/pkg	24019-06

REQUIRED REAGENTS (Using AccuVac Ampuls)

Hydrazine Reagent AccuVac Ampul	2	25/pkg	25240-25
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	2	each	500-41H

OPTIONAL REAGENTS

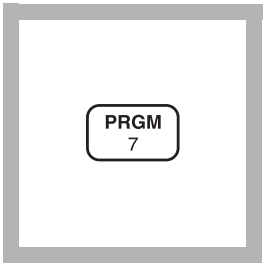
Hydrazine Sulfate, ACS		100 g	742-26

OPTIONAL APPARATUS

AccuVac Snapper Kit		each	24052-00
Balance, Analytical, 115 V, 0.1 mg		each	28014-01
Balance, Analytical, 220 V, 0.1 mg		each	28014-02
Cylinder, graduated, mixing, 25 mL		each	1896-40
Flask, volumetric, 100 mL, Class A		each	14574-42
Flask, volumetric, 1000 mL, Class A		each	14574-53
Pipet, serological, 1 mL		each	9190-02
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet, volumetric, Class A, 1.00 mL		each	14515-35
Pipet, volumetric, Class A, 4.00 mL		each	14515-04
Pipet Filler, safety bulb		each	14651-00
Thermometer, –20 to 110 °C, non-mercury		each	26357-02
Weighing Boat, 67/46 mm, 8.9 cm sq.		500/pkg	21790-00

1,10 Phenanthroline Method* (Powder Pillows or AccuVac Ampuls)

Using Powder Pillows



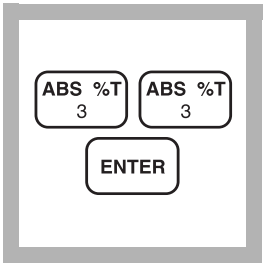
1. Enter the stored program number for Ferrous iron (Fe^{2+})-powder pillows.

Press: **PRGM**

The display will show:

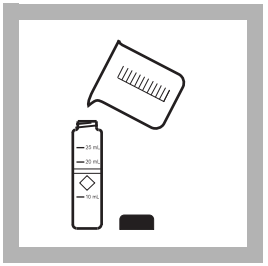
PRGM ?

Note: *Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not determined.*

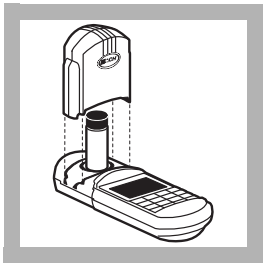


2. Press: **33 ENTER**

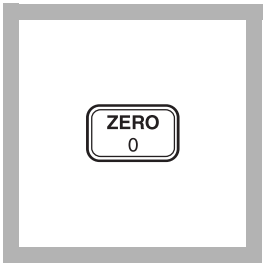
The display will show **mg/L, Fe** and the **ZERO** icon.



3. Fill a sample cell with 25 mL of sample (the blank).



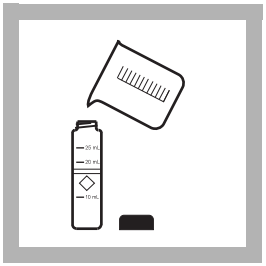
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

The cursor will move to the right, then the display will show:

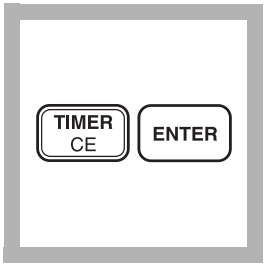
0.00 mg/L Fe



6. Fill another sample cell with 25 mL of sample.

7. Add the contents of one Ferrous Iron Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert to mix.

Note: *Undissolved powder does not affect accuracy.*



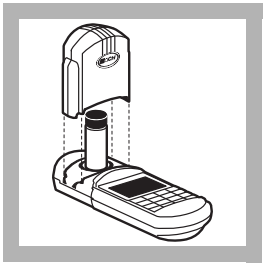
8. Press:

TIMER ENTER

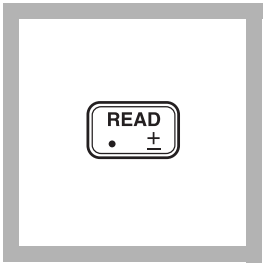
A three-minute reaction period will begin.

Note: *An orange color will form if ferrous iron is present.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

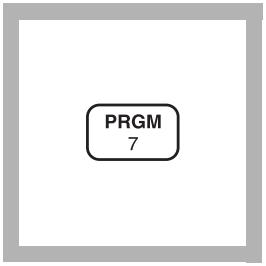


10. Press: **READ**

The cursor will move to the right, then the result in mg/L ferrous iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Using AccuVac Ampuls



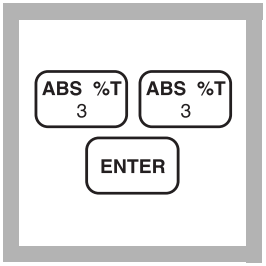
1. Enter the stored program number for ferrous iron (Fe^{2+}) AccuVac ampuls.

Press: **PRGM**

The display will show:

PRGM ?

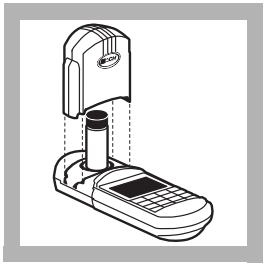
Note: *Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric, which is not determined.*



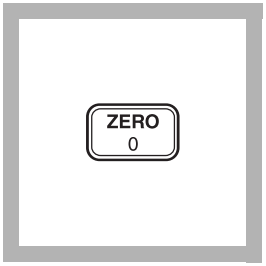
2. Press: **33 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon.

3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.



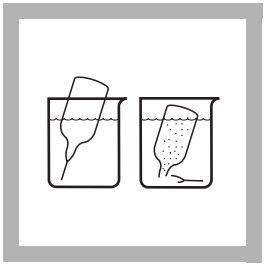
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

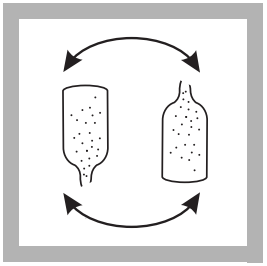
The cursor will move to the right, then the display will show:

0.00 mg/L Fe



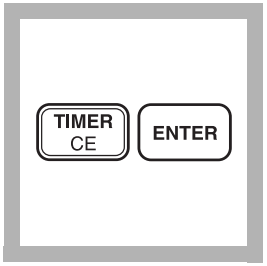
6. Fill a Ferrous Iron AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*



7. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *Undissolved powder does not affect accuracy.*

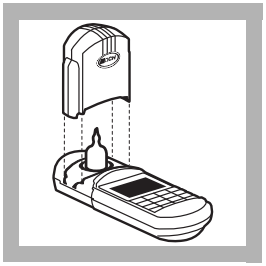


8. Press:

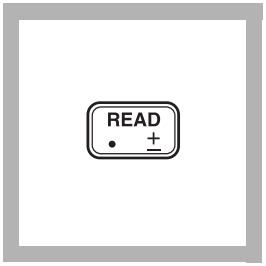
TIMER ENTER

A three-minute reaction period will begin.

Note: *An orange color will form if ferrous iron is present.*



9. Place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L ferrous iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Ferrous iron must be analyzed immediately and cannot be stored. Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not measured.

Accuracy Check

Standard Solution Method

Prepare a ferrous iron stock solution (100 mg/L Fe^{2+}) by dissolving 0.7022 grams of ferrous ammonium sulfate, hexahydrate, in deionized water. Dilute to 1 liter. Prepare immediately before use. Dilute 1.00 mL of this solution to 100 mL with deionized water to make a 1.00 mg/L standard solution. Prepare immediately before use.

Run the test using the 1.00 mg/L Fe^{2+} Standard Solution by following either the powder pillow or AccuVac procedure. Results should be between 0.90 mg/L and 1.10 mg/L Fe^{2+}.

Method Performance

Precision

In a single laboratory using an iron standard solution of 2.00 mg/L Fe^{2+} and two representative lots of powder pillow reagents with the instrument, a single operator obtained a standard deviation of ±0.017 mg/L Fe^{2+}.

In a single laboratory using a standard solution of 2.00 mg/L Fe^{2+} and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of ±0.009 mg/L Fe^{2+}.

Estimated Detection Limit

The estimated detection limit for program 33 (powder pillows and AccuVac Ampuls) is 0.03 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

The 1,10-phenanthroline indicator in Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react. The ferric iron (Fe^{3+}) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test.

REQUIRED REAGENTS & APPARATUS (USING POWDER PILLOWS)

Description	Quantity Required Per Test	Units	Cat. No.
Ferrous Iron Reagent Powder Pillows	1 pillow	100/pkg	1037-69
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (USING ACCUVAC AMPULS)

Ferrous Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25140-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Ferrous Ammonium Sulfate, hexahydrate, ACS		113 g	11256-14
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit		each	24052-00
Balance, analytical, 115 V, 0.1 mg		each	28014-01
Balance, analytical, 230 V, 0.1 mg		each	28014-02
Clippers, for opening powder pillows		each	968-00
Flask, volumetric, 100 mL, Class A		each	14574-42
Flask, volumetric, 1000 mL, Class A		each	14574-53
Pipet, volumetric, Class A, 1.00 mL		each	14515-35
Pipet Filler, safety bulb		each	14651-00
Weighing Boat, 67/46 mm, 8.9 cm square		500/pkg	21790-00

For Technical Assistance, Price and Ordering

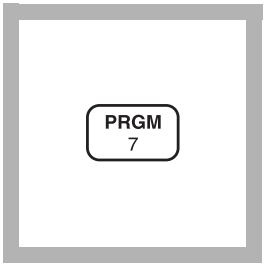
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

IRON, TOTAL (0 to 3.00 mg/L)

FerroVer Method (Powder Pillows or AccuVac Ampuls)

USEPA approved for reporting wastewater analysis (digestion is required; see Section 2*)



1. Enter the stored program number for iron (Fe) powder pillows.

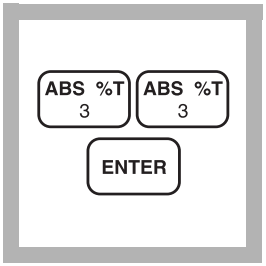
Press: **PRGM**

The display will show:

PRGM ?

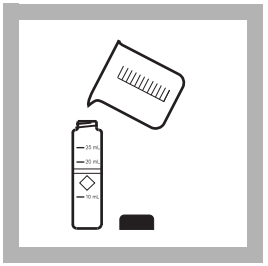
Note: *Determination of total iron requires a digestion prior to analysis (see Section 2).*

Note: *Adjust pH of stored samples before analysis.*



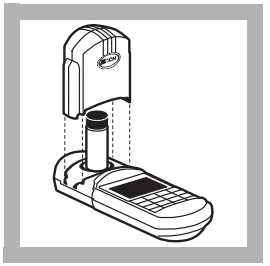
2. Press: **33 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

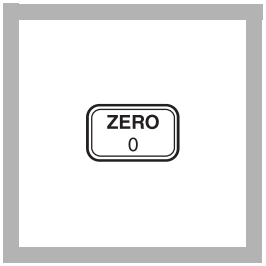


3. Fill a clean sample cell with 10 mL of sample (the blank).

Note: *For turbid samples, treat the blank with one 0.1-gram scoop of RoVer Rust Remover. Swirl to mix.*



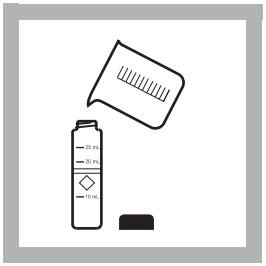
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



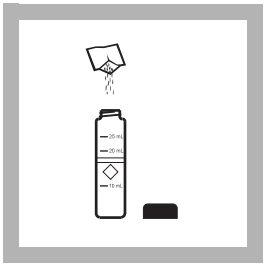
5. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Fe

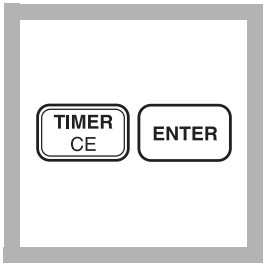


6. Fill another sample cell with 10 mL of sample.



7. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert to dissolve the reagent powder.

Note: *Accuracy is not affected by undissolved powder.*



8. Press:

TIMER ENTER

A three-minute reaction period will begin.

Note: *An orange color will form if iron is present.*

Note: *Samples containing visible rust should be allowed to react at least five minutes.*

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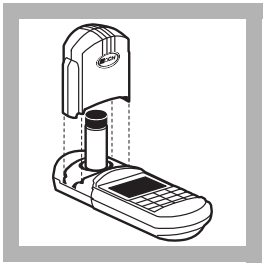
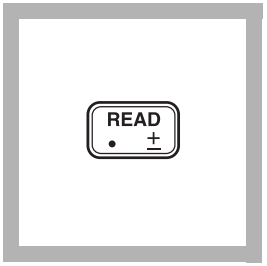
* *Federal Register*, 45 (126) 43459 (June 27, 1980). See also 40 CFR, part 136.3, Table IB.



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

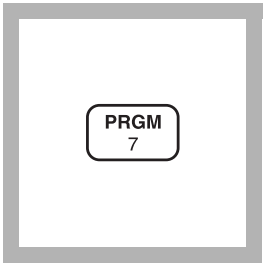


10. Press: **READ**

The cursor will move to the right, then the result in mg/L iron (Fe) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Using AccuVac Ampuls



1. Enter the stored program number for iron (Fe), AccuVac ampuls.

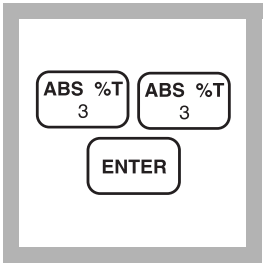
Press: **PRGM**

The display will show:

PRGM ?

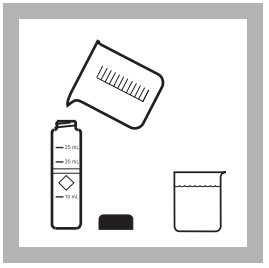
Note: *Adjust pH of stored samples before analysis.*

Note: *Determination of total iron requires a digestion prior to analysis (see Section 2).*



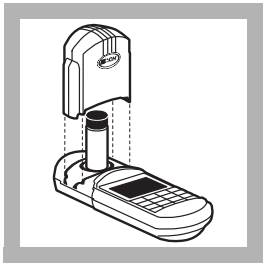
2. Press: **33 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

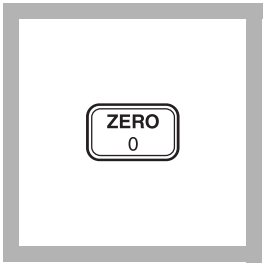


3. Fill a sample cell (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.

Note: *For turbid samples, treat the blank with one 0.1 g scoop of RoVer Rust Remover. Swirl to mix.*



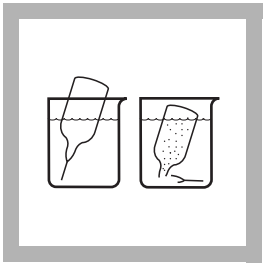
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

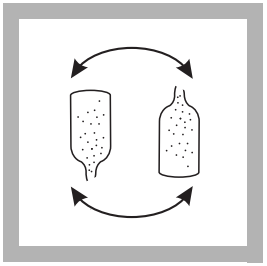
The cursor will move to the right, then the display will show:

0.00 mg/L Fe



6. Fill a FerroVer AccuVac Ampul with sample.

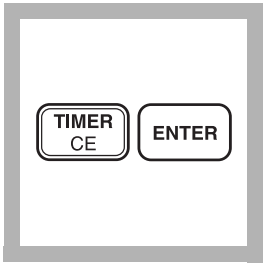
Note: *Keep the tip immersed while the ampul fills completely.*



7. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *An orange color will form if iron is present.*

Note: *Accuracy is not affected by undissolved powder.*

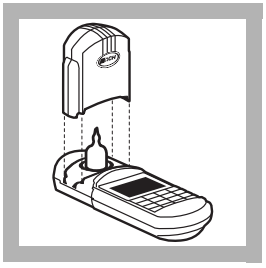


8. Press:

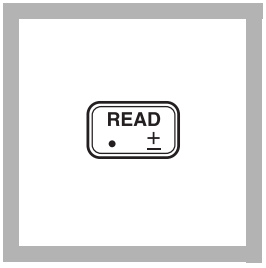
TIMER ENTER

A three-minute reaction period will begin.

Note: *Samples containing visible rust should be allowed to react at least five minutes.*



9. Place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L iron (Fe) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the sample immediately. To preserve samples, adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1* for more information. If only dissolved iron is to be determined, filter the sample before adding the acid.

Accuracy Check

Standard Additions Method

a) Snap the neck off a 50 mg/L Iron PourRite Ampule Standard Solution.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to three 25-mL samples and mix thoroughly.

c) For analysis using AccuVac Ampuls, transfer solutions to dry, clean 50-mL beakers to facilitate filling of the ampuls. For analysis with powder pillows, transfer only 10 mL of solution to the 10-mL sample cells.

d) Analyze each standard addition sample as described above. The iron concentration should increase 0.2 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

Standard Solution Method

Prepare a 1.0-mg/L iron standard by diluting 1.00 mL of Iron Standard Solution, 100 mg/L Fe, to 100 mL with deionized water. Or, dilute 1.00 mL of an Iron PourRite Ampule Standard Solution (50 mg/L) to 50 mL in a volumetric flask. Prepare this solution daily.

Run the test following the procedure for powder pillows or AccuVac Ampuls. Results should be between 0.90 mg/L and 1.10 mg/L Fe.

Method Performance

Precision

In a single laboratory, using a standard solution of 2.00 mg/L Fe and two representative lots of powder pillow reagents with the instrument, a single operator obtained a standard deviation of ±0.017 mg/L.

In a single laboratory, using a standard solution of 2.00 mg/L Fe and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of ±0.009 mg/L Fe.

Estimated Detection Limit (EDL)

The EDL for program 33 is 0.03 mg/L Fe. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatments

Interfering Substance	Interference Level and Treatment
Calcium, Ca^{2+}	No effect at less than 10,000 mg/L as $CaCO_3$
Chloride, Cl^-	No effect at less than 185,000 mg/L.
Copper, Cu^{2+}	No effect. Masking agent is contained in FerroVer Iron Reagent.
High Iron Levels	Inhibits color development. Dilute sample and retest to verify results.
Iron Oxide	Requires mild, vigorous or Digesdahl digestion (see Section 2). After digestion, adjust sample to pH 3-5 with sodium hydroxide, then analyze.
Magnesium	No effect at 100,000 mg/L as $CaCO_3$.
Molybdate, Molybdenum	No effect at 25 mg/L as Mo.
High Sulfide Levels, S^{2-}	**1.** Treat in fume hood or well-ventilated area. Add 5 mL HCl to 100 mL sample in a 250-mL Erlenmeyer flask. Boil 20 minutes. **2.** Cool. Adjust pH to 3-5 with NaOH. Re-adjust volume to 100 mL with deionized water. **3.** Analyze.

Interfering Substance	Interference Level and Treatment
Turbidity	**1.** Add 0.1 g scoop of RoVer Rust Remover to the blank in Step 3. Swirl to mix. **2.** Zero the instrument with this blank. **3.** If sample remains turbid, add three 0.2 g scoops of RoVer to a 75-mL sample. Let stand 5 minutes. **4.** Filter through a glass filter or centrifuge. **5.** Use filtered sample in Steps 3 and 6.
Sample pH (extreme)	Adjust pH to 3-5. See *Interferences* in *Section 1*.
Highly Buffered Samples	Adjust pH to 3-5. See *Interferences* in *Section 1*.

Summary of Method

FerroVer Iron Reagent reacts with all soluble iron and most insoluble forms of iron in the sample to produce soluble ferrous iron. This reacts with 1,10-phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat No.
FerroVer Iron Reagent Powder Pillows	1 pillow	100/pkg	21057-69
Sample cell, 10-20-25 mL, with screw cap	1	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

FerroVer Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25070-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Ammonium Hydroxide, ACS	500 mL	106-49
Drinking Water Standard, Metals, LR (Cu, Fe, Mn)	500 mL	28337-49
Drinking Water Standard, Metals, HR (Cu, Fe, Mn)	500 mL	28336-49
Hydrochloric Acid Standard Solution, 6 N	500 mL	884-49
Hydrochloric Acid, ACS	500 mL	134-49
Iron Standard Solution, 100 mg/L	100 mL	14175-42
Iron Ampule Standard, 50 mg/L	20/pkg	14254-20
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
RoVer Rust Remover	454 g	300-01
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each		24052-00
Ampule Breaker, PourRite Ampules	each		24846-00
Clippers, Shears $7^{1}/_{4}$ "	each		23694-00
Cylinder, graduated, poly, 25 mL	each		1081-40
Cylinder, graduated, poly, 100 mL	each		1081-42
Digesdahl Digestion Apparatus, 115 V	each		23130-20
Digesdahl Digestion Apparatus, 230 V	each		23130-21
Filter Discs, glass, 47 mm	100/pkg		2530-00
Filter Holder, membrane	each		2340-00
Filter Pump	each		2131-00
Flask, Erlenmeyer, 250 mL	each		505-46
Flask, filtering, 500 mL	each		546-49
Flask, volumetric, Class A, 50 mL	each		14574-41
Flask, volumetric, Class A, 100 mL	each		14574-42
Hot Plate, 4" diameter, 120 VAC	each		12067-01
Hot Plate, 4" diameter, 240 VAC	each		12067-02
pH Meter, *sens**ion***™***1***, portable, with electrode	each		51700-10
pH Indicator Paper, 1 to 11 pH	each		391-33
Pipet Filler, safety bulb	each		14651-00
Pipet, serological, 2 mL	each		532-36
Pipet, serological, 5 mL	each		532-37
Pipet, TenSette, 0.1 to 1.0 mL	each		19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg		21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg		21856-28
Pipet, volumetric, Class A, 1.00 mL	each		14515-35
Spoon, measuring, 0.1 g	each		511-00

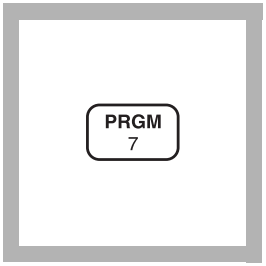
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

IRON (0 to 1.300 mg/L)

FerroZine Method*



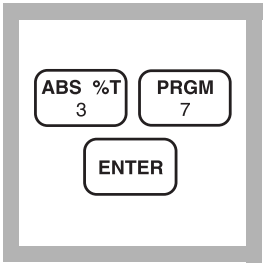
1. Enter the stored program number for iron (Fe).

Press: **PRGM**

The display will show:

PRGM ?

Note: *Adjust the pH of stored samples before analysis.*



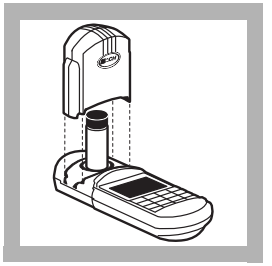
2. Press: **37 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

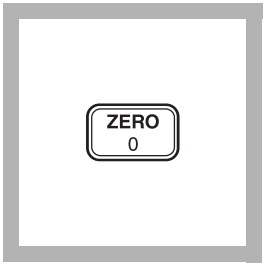
Note: *Total iron determinations need a prior digestion; use any of the three procedures given in Digestion (Section 2).*

3. Fill a sample cell with 25-mL of sample (the blank).

Note: *Rinse glassware with a 1:1 Hydrochloric Acid Solution and deionized water before use to avoid errors due to iron deposits on the glass.*



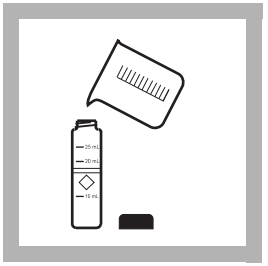
4. Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

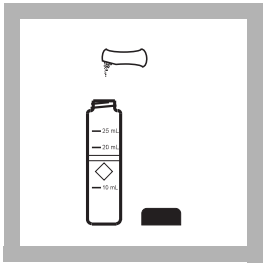
The cursor will move to the right, then the display will show:

0.000 mg/L Fe



6. Fill another sample cell with 25 mL of sample.

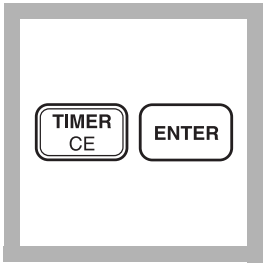
Note: *If the sample contains rust, see Interferences below.*



7. Add the contents of one FerroZine Iron Reagent Solution Pillow to the cell (the prepared sample). Cap and invert to mix.

Note: *Do not allow the clippers to come into contact with the contents of the pillow.*

Note: *If preferred , use 0.5 mL of FerroZine Iron Reagent Solution in place of the solution pillow.*



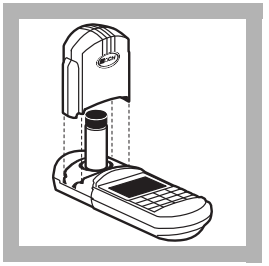
8. Press:

TIMER ENTER

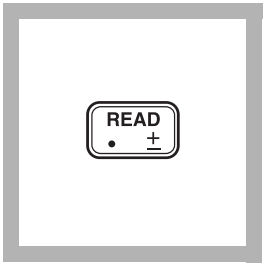
A five-minute reaction period will begin.

Note: *A violet color will develop if iron is present.*

* Adapted from Stookey, L.L., Anal. Chem., 42 (7) 779 (1970)



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-washed glass or plastic bottles. To preserve samples, adjust the sample pH to 2 or less with nitric acid (about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature. If only dissolved iron is to be reported, filter sample immediately after collection and before the addition of nitric acid.

Before testing, adjust the sample pH to 3–5 with ammonium hydroxide, ACS. Do not exceed pH 5 as iron may precipitate. Correct test results for volume additions; see *Correction for Volume Additions* in *Section 1* for more detailed information.

Accuracy Check

Standard Additions Method

a) Snap the neck off an Iron Voluette Ampule Standard, 25 mg/L Fe.

b) Use the TenSette Pipet to add 0.1 mL of standard to the prepared sample measured in Step 10.

c) Swirl to mix and allow another five-minute reaction period, then measure the iron concentration as in Step 10.

d) Add two additional 0.1-mL standard increments, taking a

concentration reading after allowing the five-minute reaction period for each increment.

e) Each 0.1 mL of standard added should cause a 0.1 mg/L increase in the concentration reading.

f) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

a) Pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a
250-mL volumetric flask.

b) Dilute to volume with deionized water. This solution should be prepared daily. Analyze the working solution according to the above procedure.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.80 mg/L iron and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.004 mg/L iron.

Estimated Detection Limit

The estimated detection limit for program 37 is 0.011 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Strong chelants, EDTA	Interfere at all levels. Use the FerroVer or TPTZ methods to test these samples. Use the TPTZ method for low iron concentrations.
Cobalt	May give slightly high results
Copper	May give slightly high results
Hydroxides	Boil the sample, with the FerroZine Iron Reagent from Step 7 added to it for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with Step 8. Return the sample volume to 25 mL with deionized water. OR Use any of the digestions in *Section 2*.
Magnetite (black iron oxide) or Ferrites	**1.** Fill a 25-mL graduated cylinder with 25 mL of sample. **2.** Transfer this sample into a 125-mL erlenmeyer flask. **3.** Add the contents of one FerroZine Iron Reagent Solution Pillow and swirl to mix. **4.** Place the flask on a hot plate or over a flame and bring to a boil. **5.** Continue boiling gently for 20 to 30 minutes. ***Note:*** *Do not allow to boil dry.* ***Note:*** *A purple color will develop if iron is present.* **6.** Return the boiled sample to the 25-mL graduated cylinder. Rinse the erlenmeyer flask with small amounts of deionized water and empty into the graduated cylinder. **7.** Return the sample volume to the 25-mL mark with deionized water. **8.** Pour this solution into a sample cell. Swirl to mix. **9.** Proceed with Step 9. OR Use any of the digestions in *Section 2*.
Rust	Boil the sample, with the FerroZine Iron Reagent from Step 7 for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with Step 8. Return the volume to 25 mL with deionized water. OR Use any of the digestions in *Section 2*.

Summary of Method

The FerroZine Iron Reagent forms a purple colored complex with trace amounts of iron in samples that are buffered to a pH of 3.5. This method is applicable for determining trace levels of iron in chemical reagents and glycols and can be used to analyze samples containing magnetite (black iron oxide) or ferrites after treatment as described in Interferences.

REQUIRED REAGENTS AND APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
FerroZine Iron Reagent Solution Pillows	1 pillow	50/pkg	2301-66
Clippers, for opening pillows	1	each	968-00
Sample Cell, 10-20-25, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Ammonium Hydroxide, ACS	500 mL	106-49	Drinking
Water Standard, Metals, LR (Cu, Fe, Mn)	500 mL		28337-49
Hydrochloric Acid Solution, 1:1 (6N)	500 mL		884-49
FerroZine Iron Reagent Solution	500 mL		2301-49
Iron Standard Solution, 100 mg/L Fe	100 mL		14175-42
Iron Standard Solution, Voluette Ampule, 25 mg/L Fe, 10 mL	16/pkg		14253-10
Nitric Acid, ACS	500 mL		152-49
Nitric Acid Solution, 1:1	500 mL		2540-49
Water, deionized	4 L		272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Clippers, shears, 7¼-inch	each	20658-00
Cylinder, graduated, 25 mL	each	508-40
Dropper, calibrated, 0.5-mL & 1.0-mL mark	6/pkg	23185-06
Flask, erlenmeyer, 125 mL	each	505-43
Flask, volumetric, 250 mL, Class A	each	14574-46
Hot plate, 3 ½" diameter, 120 V	each	12067-01
Hot plate, 3 ½" diameter, 240 V	each	12067-02
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, serological, 2 mL	each	532-36
pH Meter, *sens**ion***™***1***, portable, with electrode	each	51700-10
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96 Pipet
Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, 1.00 mL, Class A	each	14515-35
Thermometer, –20 to 110 °C, non-mercury	each	26357-02
Water Bath, with sample cell rack	each	1955-55

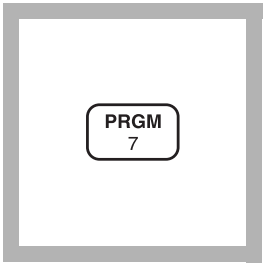
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

IRON, TOTAL (0 to 1.80 mg/L) For cooling water with molybdenum-based treatment

FerroMo™ Method*



1. Enter the stored program number for iron (Fe).

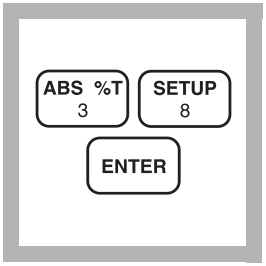
Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

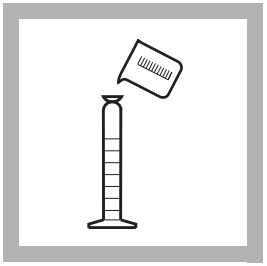
Note: *Adjust the pH of stored samples before analysis.*



2. Press: **38 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

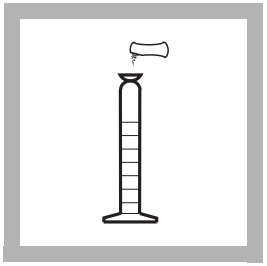
Note: *Determination of total iron requires digestion; see Section 2.*



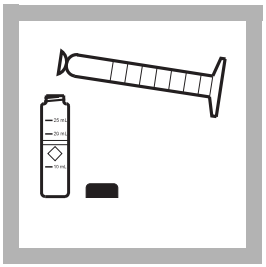
3. Fill a 50-mL graduated mixing cylinder with 50 mL of sample.

Note: *Sample pH is important in the test; see Interferences.*

Note: *Rinse glassware with 1:1 Hydrochloric Acid Solution. Rinse again with deionized water. This removes iron deposits which can cause slightly high results.*

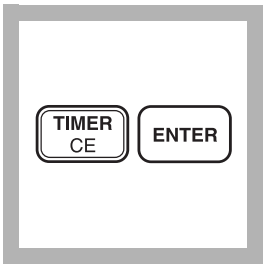


4. Add the contents of one FerroMo Iron Reagent 1 Powder Pillow to the graduated cylinder. Stopper and invert several times to mix. Remove the stopper. This is the prepared sample.



5. Transfer 25 mL of the prepared sample to a sample cell.

6. Add the contents of one FerroMo Iron Reagent 2 Powder Pillow to the sample cell. Cap the cell and shake for 30 seconds. This is the prepared sample.



7. Press:

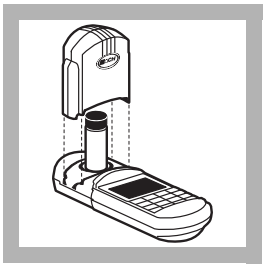
TIMER ENTER

A three-minute reaction period will begin.

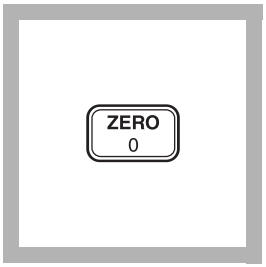
Note: *A blue color will develop if iron is present.*

8. Fill a second sample cell with 25 mL of the prepared sample from Step 4 (the blank).

* Adapted from G. Frederic Smith Chemical Company, *The Iron Reagents*, 3rd ed. (1980).



9. Insert the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

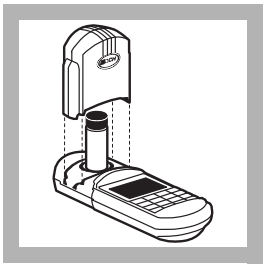


10. Press: **ZERO**

The cursor will move to the right, then the display will show:

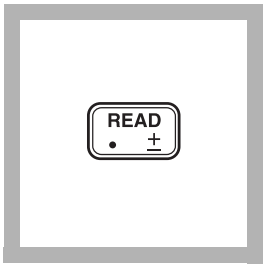
0.00 mg/L Fe

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



11. After the timer beeps, place the prepared sample in the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *For samples containing high levels of molybdate (≥100 mg/L), read the sample immediately after zeroing the blank.*



12. Press: **READ**

The cursor will move to the right, then the result in mg/L iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-cleaned plastic or glass bottles. If prompt analysis is impossible, preserve the sample by adjusting to pH 2 or less with hydrochloric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. If reporting only dissolved iron, filter the sample immediately after collection and before adding the acid.

Before analysis, adjust the sample pH to between 3 and 4 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 5 as iron may precipitate. Correct the test result for volume; see *Correction for Volume Additions* in *Section 1*.

Accuracy Check

Standard Additions Method

a) Snap the top off an Iron PourRite Ampule Standard Solution, 25 mg/L Fe.

b) Use the TenSette Pipet to add 0.2, 0.4 and 0.6 mL of standard to three 50-mL samples. Swirl gently to mix.

c) Analyze each sample as described above. The iron concentration should increase by 0.1 mg/L for each 0.2

mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more Information.

Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

a) Pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a
250-mL volumetric flask.

b) Dilute to volume with deionized water. Prepare this solution daily. Analyze this working solution according to the above procedure. Results should be between 0.36 and 0.44 mg/L Fe.

Method Performance

Precision

In a single laboratory, using a standard solution of 1.00 mg/L Fe and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of ±0.006 mg/L Fe.

Estimated Detection Limit

The estimated detection limit for program 38 is 0.03 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

Interferences

A sample pH of less than 3 or greater than 4 after reagent addition may inhibit color formation, cause the developed color to fade, or result in turbidity. Adjust the sample pH before reagent addition to between 3 and 5 using a pH meter or pH paper. Drop by drop, add an appropriate amount of acid (1.0 N Sulfuric Acid Solution) or base (1.0 N Sodium Hydroxide Standard Solution). Make volume corrections if significant amounts of acid or base are used (see *Correction for Volume Additions* in *Section 1*).

Summary of Method

FerroMo Iron Reagent 1 contains a reducing agent combined with a masking agent. The masking agent eliminates interference from high levels of molybdate. The reducing agent converts precipitated or suspended iron (rust) to the ferrous state. FerroMo Iron Reagent 2 contains the indicator combined with a buffering

agent. The indicator reacts with the ferrous iron in the sample, buffered between pH 3-4, resulting in a deep blue-purple color.

REQUIRED REAGENTS

	Cat. No.
FerroMo Reagent Set (100 tests)	25448-00
Includes: (4) 25437-68, (2) 25438-66	

Description	Quantity Required Per Test	Unit	Cat. No
FerroMo Iron Reagent 1 Powder Pillows	1 pillow	25/pkg	25437-68
FerroMo Iron Reagent 2 Powder Pillows	1 pillow	50/pkg	25438-66

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, mixing, 50 mL	1	each	1896-41
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	884-49
Hydrochloric Acid, ACS	500 mL	134-49
Iron Standard Solution, 100 mg/L Fe	100 mL	14175-42
Iron Standard Solution, PourRite Ampule, 25 mg/L Fe, 2 mL	20/pkg	24629-20
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045-32
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sulfuric Acid Standard Solution, 1.0 N	100 mL MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	24846-00
Flask, volumetric, Class A, 250 mL	each	14574-46
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *Sens**ion**™1*, portable, with electrode	each	51700-10
Pipet Filler, safety bulb	each	14651-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 1.00 mL	each	14515-35

For Technical Assistance, Price and Ordering

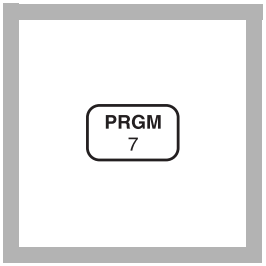
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

IRON, TOTAL (0 to 1.80 mg/L)

TPTZ Method* (Powder Pillows or AccuVac Ampuls)

Using Powder Pillows



1. Enter the stored program number for iron (Fe)- powder pillows.

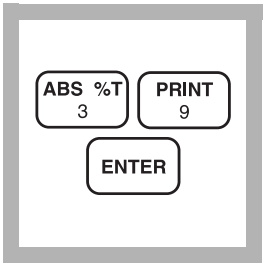
Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

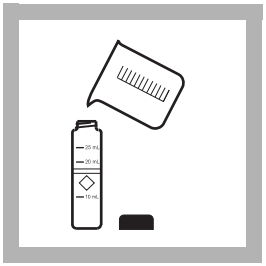
Note: *Adjust the pH of stored samples before analysis.*



2. Press: **39 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

Note: *Total iron determination needs a prior digestion. Use any of the procedures in Digestion (Section 2).*



3. Fill a sample cell with 10 mL of sample.

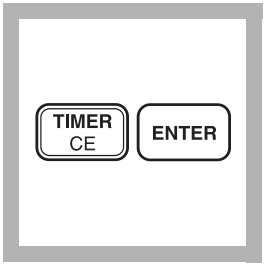
Note: *Sample pH is important in this test; see Interferences.*

Note: *Rinse glassware with a 1:1 hydrochloric acid and deionized water before use to avoid errors due to iron deposits on the glass.*

4. Add the contents of one TPTZ Iron Reagent Powder Pillow (the prepared sample). Cap and shake the cell for 30 seconds.

Note: *A blue color will develop if iron is present.*

* Adapted from G. Frederic Smith Chemical Co., *The Iron Reagents*, 3rd ed. (1980).

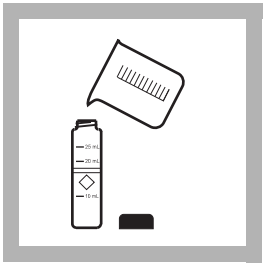


5. Press:

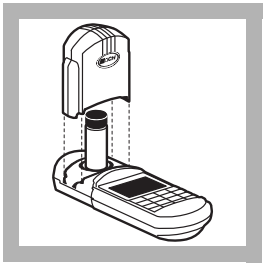
TIMER ENTER

A three-minute reaction period will begin.

Note: *Continue with Steps 6 to 8 while the timer is running.*

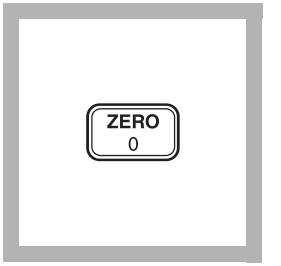


6. Fill a second sample cell with 10 mL of sample (the blank).



7. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Press* **EXIT** *to zero the instrument while the timer is running.*

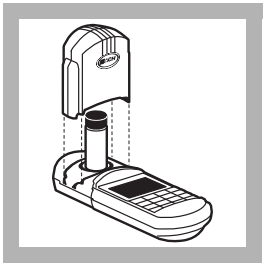


Press: **ZERO**

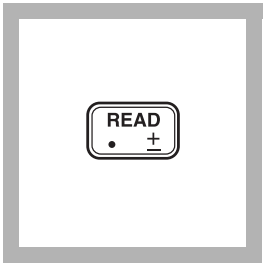
The cursor will move to the right, then the display will show:

0.00 mg/L Fe

If Reagent Blank Correction is on, the display may flash "limit". See Section 1.



8. After the timer beeps, place the prepared sample in the cell holder. Tightly cover the sample cell with the instrument cap.

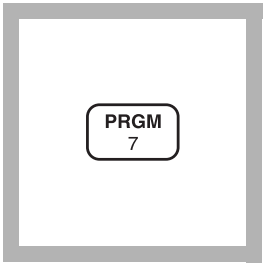


9. Press: **READ**

The cursor will move to the right, then the result in mg/L iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Using AccuVac Ampuls



1. Enter the stored program number for iron (Fe)- AccuVac Ampuls.

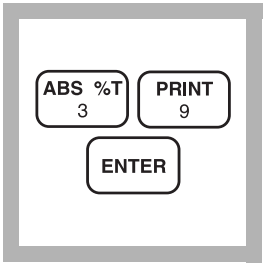
Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

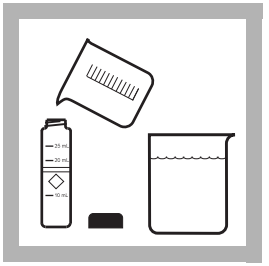
Note: *Adjust the pH of stored samples before analysis.*



2. Press: **39 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

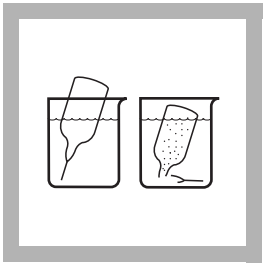
Note: *Total iron determination needs a prior digestion. Use any of the three procedures in Digestion (Section 2).*



3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

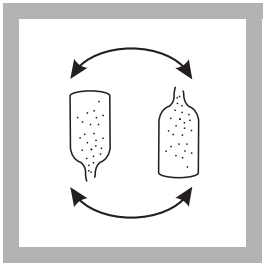
Note: *Sample pH is important in this test; see Interferences.*

Note: *Rinse glassware with a 1:1 hydrochloric acid and deionized water before use to avoid errors due to iron deposits on the glass.*



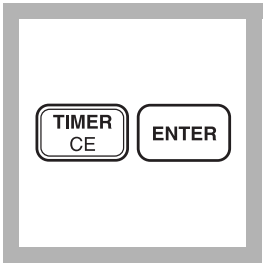
4. Fill a TPTZ Iron AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*



5. Invert the ampul (the prepared sample) repeatedly to mix. Wipe off any liquid or fingerprints.

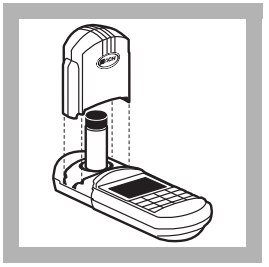
Note: *A blue color will develop if iron is present.*



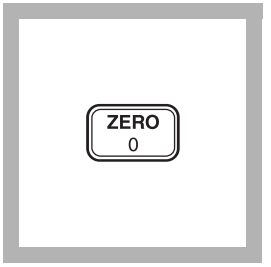
6. Press:

TIMER ENTER

A three-minute reaction period will begin.



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



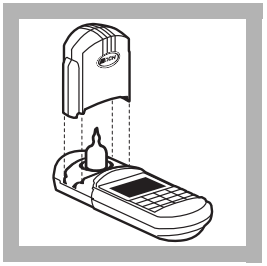
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

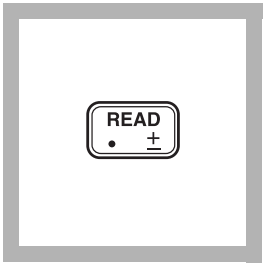
0.00 mg/L Fe

Note: *Press* **EXIT** *to zero the instrument while the timer is running.*

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



9. When the timer beeps, place the prepared sample into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-washed glass or plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 2 mL per liter). Store samples preserved in this manner up to six months at room temperature. If reporting only dissolved iron, filter sample immediately after collection and before addition of nitric acid.

Before testing, adjust the pH of the stored sample to between 3 and 4 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 5 as iron may precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1*.

Accuracy Check

Standard Additions Method (Powder Pillows)

a) Snap the neck off a PourRite Iron Ampule Standard, 25 mg/L Fe.

b) Use the TenSette Pipet to add 0.1 mL of standard to the prepared sample measured in Step 10. Swirl to mix.

c) Measure the iron concentration as in Step 10. The measurement does not require the three-minute waiting period.

d) Add two additional 0.1-mL aliquots of standard, measuring the concentration after each addition. The iron concentration should increase by 0.25 mg/L for each 0.1-mL addition of standard.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method (AccuVac Ampuls)

a) Use a graduated cylinder to measure 25.0 mL of sample into each of three 50-mL beakers.

b) Snap the neck off an Iron Ampule Standard, 25 mg/L Fe.

c) Using a TenSette Pipet, add 0.1, 0.2 and 0.3 mL of standard, respectively, to the 50-mL beakers. Swirl to mix.

d) Fill a TPTZ AccuVac Ampul from each beaker.

e) Measure the concentration of each ampul according to the procedure. The iron concentration should increase by 0.1 mg/L for each 0.1 mL addition of standard.

f) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

a) Using Class A glassware, pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a 250-mL volumetric flask.

b) Dilute to volume with deionized water. Stopper and invert repeatedly to mix. Prepare this solution daily.

Method Performance

Precision

In a single laboratory using a standard solution of 1.00 mg/L Fe and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.017 mg/L Fe.

In a single laboratory using a standard solution of 1.00 mg/L Fe and one representative lot of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.022 mg/L Fe.

Estimated Detection Limit

The estimated detection limit for program 39 is 0.04 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Cadmium	Greater than 4.0 mg/L
Chromium ($^{3+}$)	Greater than 0.25 mg/L
Chromium ($^{6+}$)	Greater than 1.2 mg/L
Cobalt	Greater than 0.05 mg/L
Color or turbidity	If the sample is turbid, add one 0.1-g scoop of RoVer Rust Remover to the blank in Step 6 (Step 3 for AccuVac procedure). Swirl to mix.
Copper	Greater than 0.6 mg/L
Cyanide	Greater than 2.8 mg/L
Manganese	Greater than 50.0 mg/L
Mercury	Greater than 0.4 mg/L
Molybdenum	Greater than 4.0 mg/L
Nickel	Greater than 1.0 mg/L
Nitrite Ion	Greater than 0.8 mg/L
pH	A sample pH of < 3 or >4 after the addition of reagent may inhibit color formation, cause the developed color to fade quickly or result in turbidity. Adjust the sample pH to 3–5 before adding reagent using a pH meter or pH paper and adding (dropwise) an appropriate amount of iron-free acid or base (i.e., 1.0 N Sulfuric Acid Standard Solution or 1.0 N Sodium Hydroxide Standard Solution). Make a volume correction if significant volumes of acid or base are used.

Summary of Method

The TPTZ Iron Reagent forms a deep blue-purple color with ferrous iron. The indicator is combined with a reducing agent which converts precipitated or suspended iron, such as rust, to the ferrous state. The amount of ferric iron present can be determined as the difference between the results of a ferrous iron test and the concentration of total iron.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
TPTZ Iron Reagent Powder Pillows,	1 pillow	100/pkg	26087-99
Sample Cell, 10-20-25 mL, w/cap	1	6/pkg	24019-06

REQUIRED REAGENTS (Using AccuVac Ampuls)

TPTZ Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25100-25

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41H
Sample Cell, 10-20-25 mL, w/cap	1	6/pkg	24019-06

OPTIONAL REAGENTS

Drinking Water Standard, Metals, LR (Cu, Fe, Mn)	500 mL	28337-49
Drinking Water Standard, Metals, HR (Cu, Fe, Mn)	500 mL	28336-49
Hydrochloric Acid Solution, 1:1, 6.0 N	500 mL	884-49
Iron Standard Solution, 100 mg/L Fe	100 mL	14175-42
Iron Standard Solution, Ampule, 25 mg/L Fe, 2 mL	20/pkg	24629-20
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
RoVer Rust Remover	454 g	300-01
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045-32
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sulfuric Acid Standard Solution	100 mL MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit	each	24052-00
Ampule Breaker, Ampules	each	24846-00
Cylinder, graduated, 25 mL	each	1081-40
Dropper, graduated, 0.5 and 1.0 mL marks	20/pkg	21247-20
Flask, volumetric, Class A, 250 mL	each	14574-46
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *sens**ion**™1*, portable, with electrode	each	51700-10
Pipet Filler, safety bulb	each	14651-00
Pipet, serological, 2 mL	each	532-36
Pipet TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 1.00 mL	each	14515-35

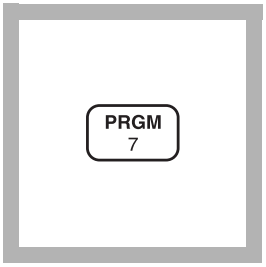
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

MANGANESE, High Range (0 to 20.0 mg/L)

Periodate Oxidation Method* USEPA approved for reporting wastewater analysis (digestion is required; see Section 2)**

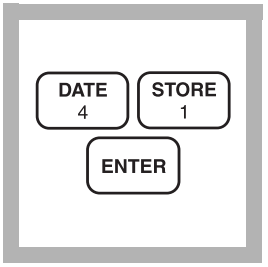


1. Enter the stored program number for manganese, periodate oxidation method.

Press: **PRGM**

The display will show:

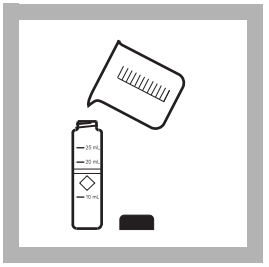
PRGM ?



2. Press: **41 ENTER**

The display will show **mg/L, Mn** and the **ZERO** icon**.**

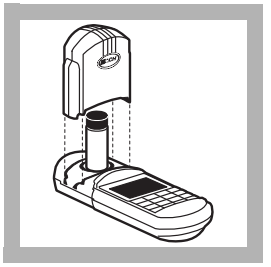
Note: *For alternate forms ($KMnO_4$, MnO_4), press the* **CONC** *key.*



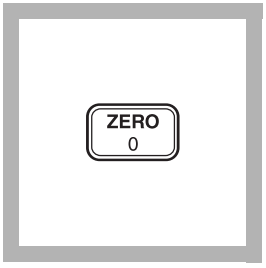
3. Fill a sample cell with 10 mL of sample (the blank).

Note: *For total manganese determination perform a digestion (see Section 2).*

Note: *Adjust the pH of stored samples before analysis.*



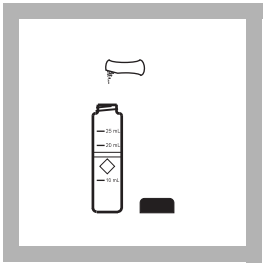
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



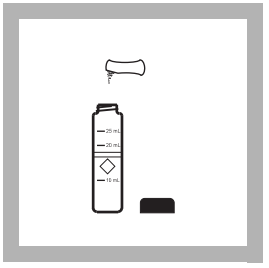
5. Press: **ZERO**

The cursor will move to the right, then the display will show:

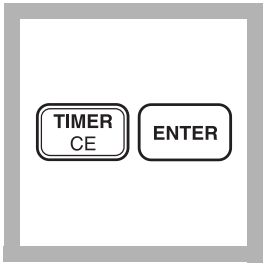
0.0 mg/L Mn



6. Remove the cell from the instrument. Add the contents of one Buffer Powder Pillow, citrate type, to the cell. Cap the cell and invert until the powder is dissolved. Remove cap.



7. Add the contents of one Sodium Periodate Powder Pillow to the sample cell (the prepared sample). Cap the sample cell. Invert for 10 seconds to mix.



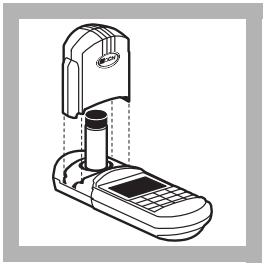
8. Press:**TIMER ENTER**

A two-minute reaction period will begin.

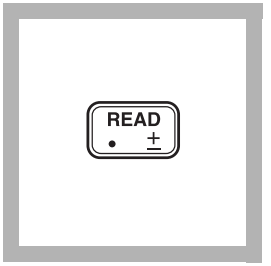
Note: *A violet color will form if manganese is present.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

** *Federal Register,* 44 (116) 34193 (June 14, 1979).



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L manganese will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-washed plastic bottles. Manganese may be lost by adsorption to glass container walls. Adjust the pH to less than 2 with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as manganese may be lost as a precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information. If only dissolved Mn is to be determined, filter before acid addition.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Manganese Voluette Ampule Standard Solution, 250 mg/L Mn.

b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three 25-mL water samples. Swirl to mix.

c) Transfer only 10 mL of each solution to the 10-mL sample cells.

d) Analyze each standard addition sample as described in the procedure. The manganese concentration should increase 1.0 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

Standard Solution Method

Prepare a 5.0 mg/L manganese standard solution by pipetting (use a TenSette or Class A volumetric pipet) 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Or, prepare this standard by diluting 1.00 mL of a High Range Manganese Standard Voluette Ampule, 250 mg/L, to 50 mL. Prepare these solutions daily. Use these solutions as the sample in the procedure.

Method Performance

Precision

In a single laboratory, using a standard solution of 10.00 mg/L Mn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.18 mg/L Mn.

Estimated Detection Limit

The estimated detection limit for program 41 is 0.2 mg/L Mn. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Calcium	700 mg/L
Chloride	70,000 mg/L
Iron	5 mg/L
Magnesium	100,000 mg/L

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interferences* in *Section 1*.

Summary of Method

Manganese in the sample is oxidized to the purple permanganate state by sodium periodate, after buffering the sample with citrate. The purple color is directly proportional to the manganese concentration.

REQUIRED REAGENTS

	Cat. No.
High Range Manganese Reagent Set (100 tests) 10 mL	24300-00
Includes: (1) 21076-69, (1) 21077-69	

Description	Quantity Required Per Test	Unit	Cat. No.
Buffer Powder Pillows, citrate type for Manganese	1 pillow	100/pkg	21076-69
Sodium Periodate Powder Pillows for Manganese	1 pillow	100/pkg	21077-69

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Drinking Water Standard, Metals, HR (Cu, Fe, Mn)	500 mL	28336-49
Hydrochloric Acid, 6 N	500 mL	884-49
Manganese Standard Solution, 1000 mg/L Mn	100 mL	12791-42
Manganese Standard Solution, Voluette ampule, High Range, 250 mg/L Mn, 10 mL	16/pkg	14258-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution 1:1	500 mL	2540-49
Sodium Hydroxide Solution, 5.0 N	100 mL MDB	2450-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Flask, Erlenmeyer, 250 mL	each	505-46
Flask, volumetric, Class A, 50 mL	each	14574-41
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 1000 mL	each	14574-53
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable, with electrode	each	51700-10
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96Pipet
Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet Tips, for 19700-10 TenSette Pipet	50/pkg	21997-96
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00

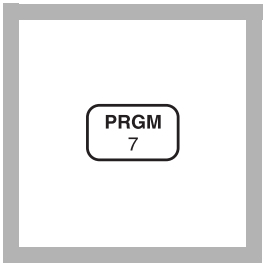
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

MANGANESE, Low Range (0 to 0.700 mg/L)

PAN Method*

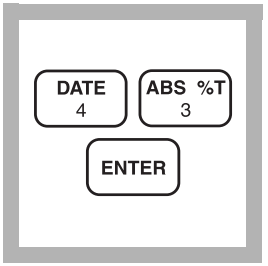


1. Enter the stored program number for low range manganese.

Press: **PRGM**

The display will show:

PRGM ?

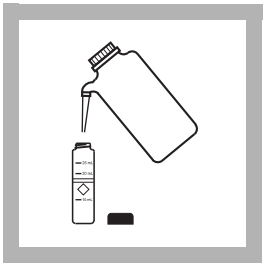


2. Press: **43 ENTER**

The display will show **mg/L, Mn** and the **ZERO** icon**.**

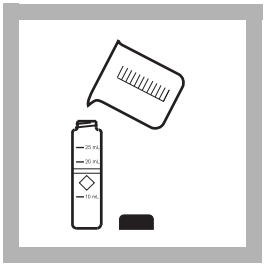
Note: *For alternate forms (MnO_4, $KMnO_4$), press the* **CONC** *key.*

Note: *Total manganese determination requires a prior digestion; see Digestion (Section 2).*

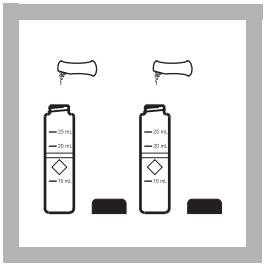


3. Fill a sample cell with 10 mL of deionized water (the blank).

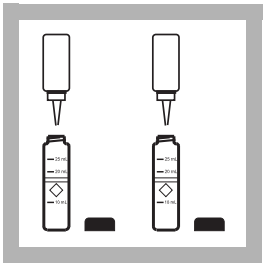
Note: *Rinse all glassware with 1:1 Nitric Acid Solution. Rinse again with deionized water.*



4. Fill another sample cell with 10 mL of sample (the prepared sample).



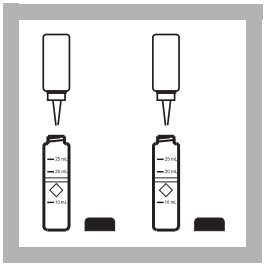
5. Add the contents of one Ascorbic Acid Powder Pillow to each cell. Swirl to mix.



6. Add 12 drops of Alkaline-Cyanide Reagent Solution to each cell. Swirl to mix.

Note: *A cloudy solution may form in some samples after reagent addition. The turbidity should dissipate after Step 8.*

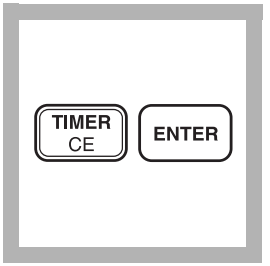
Note: *A Tensette Pipet may be used to dispense 0.4 mL of the Alkaline Cyanide Reagent.*



7. Add 12 drops of PAN Indicator Solution, 0.1%, to each sample cell. Swirl to mix.

Note: *An orange color will develop in the sample if manganese is present.*

Note: *A Tensette Pipet may be used to dispense 0.4 mL of the PAN Indicator Solution.*

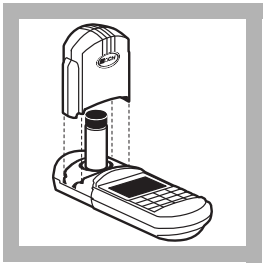


8. Press:

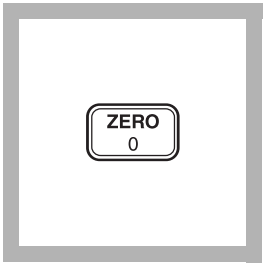
TIMER ENTER

A two-minute reaction period will begin.

* Adapted from Goto, K., et al., Talanta, 24, 752-3 (1977).



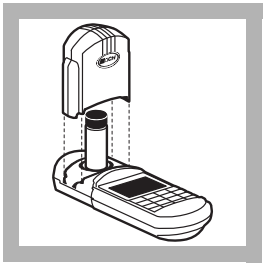
9. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



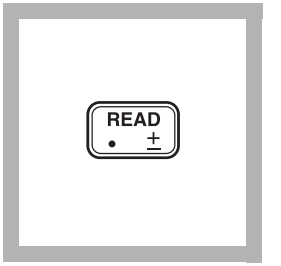
10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.000 mg/L Mn



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L manganese will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Note: *See Waste Management below for proper disposal of cyanide wastes.*

Sampling and Storage

Collect samples in a clean glass or plastic container. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the pH to 4.0-5.0 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1*.

Accuracy Check

Standard Additions Method

Note: *Volume accuracy is very important when performing standard additions with 10-mL volumes. The fill mark on the 10-mL sample cell is not intended to measure standard addition volumes.*

a) Fill three 10-mL graduated mixing cylinders with 10.0 mL of sample.

b) Snap the neck off a Manganese Voluette Ampule Standard,
10 mg/L Mn.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper and mix each thoroughly.

d) Analyze each sample as described in the procedure. The manganese concentration should increase 0.1 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Note: *An alternative to the above procedure is to pipet 10.0 mL of sample into dry sample cells before performing standard additions. A volumetric pipet or a TenSette Pipet can be used to deliver the sample volume.*

Standard Solution Method

Prepare a 0.5 mg/L manganese standard solution as follows:

a) Pipet 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask.

b) Dilute to the mark with deionized water. Prepare this solution daily.

c) Pipet 10.00 mL of the solution from Step b into a 100-mL volumetric flask.

d) Dilute to the mark with deionized water. This second dilution is equivalent to 0.5 mg/L Mn.

Method Performance

Precision

In a single laboratory using a standard solution of 0.5 mg/L Mn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.013 mg/L Mn.

Estimated Detection Limit

The estimated detection limit for program 43 is 0.020 mg/L Mn. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following do not interfere up to the indicated concentrations:

Substance	Suggested Treatment For Levels That Interfere
Aluminum	20 mg/L
Cadmium	10 mg/L
Cobalt	20 mg/L
Copper	50 mg/L
Hardness	300 mg/L.
Iron	If the sample contains more than 5 mg/L iron, allow 10 minutes for complete color development. Instead of performing Step 8, set the timer for 10 minutes by pressing **TIMER** twice. Then press **1000**. Press **ENTER** to start the timer.
Lead	0.5 mg/L
Magnesium	For samples containing hardness greater than 300 mg/L $CaCO_3$, add four drops of Rochelle Salt Solution to the sample after addition of the Ascorbic Acid Powder Pillow.
Nickel	40 mg/L
Zinc	15 mg/L

Waste Management

The alkaline cyanide solution contains cyanide. Cyanide solutions should be collected for disposal as reactive (D003) waste. Store all cyanide solutions in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. In case of a spill, clean up the area as outlined below:

1. Use a fume hood or self-contained breathing apparatus.
2. While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochlorite (household bleach).
3. Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.
4. Flush the solution down the drain with a large excess of water.

Summary of Method

The PAN method is a highly sensitive and rapid procedure for detecting low levels of manganese. An ascorbic acid reagent is used initially to reduce all oxidized forms of manganese to Mn^{2+}. An alkaline-cyanide reagent is added to mask any potential

interferences. PAN Indicator is then added to combine with the Mn^{2+} to form an orange-colored complex.

REQUIRED REAGENTS

	Cat. No.
Manganese Reagent Set (50 tests)	26517-00
Includes: (1) 21223-26, (1) 14577-99, (1) 21224-26	

Description	Quantity Required Per Test	Unit	Cat. No.
Alkaline-Cyanide Reagent	30 drops	50 mL SCDB	21223-26
Ascorbic Acid Powder Pillows	2 pillows	100/pkg	14577-99
PAN Indicator Solution, 0.1%	42 drops	50 mL SCDB	21224-26
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508-40
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Drinking Water Standard, Metals, LR (Cu, Fe, Mn)	500 mL	28337-49
Hydrochloric Acid Solution, 1:1 (6 N)	500 mL	884-49
Manganese Standard Solution, 1000 mg/L Mn	100 mL	12791-42
Manganese Standard Sol'n, Ampule, 25 mg/L Mn, 2 mL	20/pkg.	21128-20
Nitric Acid Solution, 1:1	500 mL	2540-49
Rochelle Salt Solution.	29 mL DB	1725-33
Sodium Hydroxide Solution, 50%	500 mL	2180-49
Nitric Acid, ACS	500 mL	152-49

OPTIONAL APPARATUS

Ampule Breaker, Ampule	each	24846-00
Beaker, glass, 1000 mL	each	500-53
Cylinder, graduated, mixing, 10 mL	each	20886-38
Dropper, plastic, calibrated, 1.0 mL	20/pkg	21247-20
Flask, volumetric, Class A, 1000 mL	each	14574-53
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 10.0 mL	each	14515-38
Pipet, volumetric, Class A, 5.0 mL	each	14515-37
Pipet Filler, safety bulb	each	14651-00

For Technical Assistance, Price and Ordering

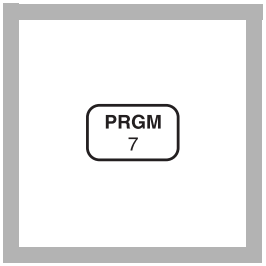
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

MOLYBDENUM, MOLYBDATE, Low Range (0 to 3.00 mg/L)

Ternary Complex Method

For boiler and cooling tower waters



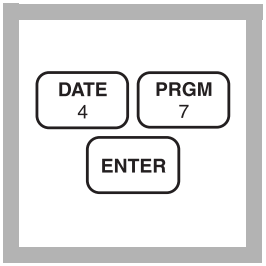
1. Enter the stored program number for molybdate molybdenum.

Press: **PRGM**

The display will show:

PRGM ?

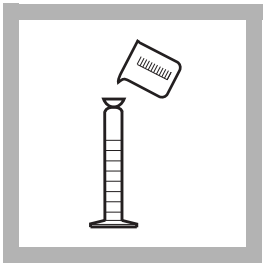
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **47 ENTER**

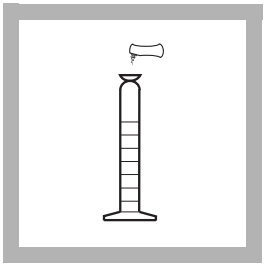
The display will show **mg/L, Mo6** and the **ZERO** icon**.**

Note: *For alternate forms (MoO_4), press the* **CONC** *key.*



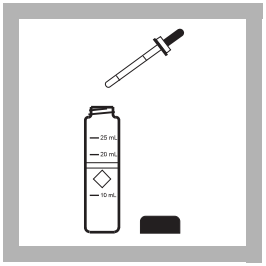
3. Fill a 25-mL mixing graduated cylinder with 20 mL of the sample.

Note: *Filter turbid samples using the labware listed under Optional Apparatus.*



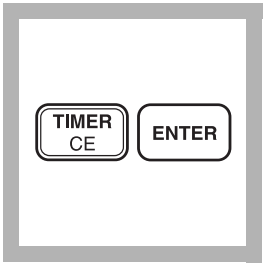
4. Add the contents of one Molybdenum 1 Reagent Powder Pillow to the graduated cylinder. Stopper. Invert the graduated cylinder several times to dissolve the reagents.

5. Pour 10 mL of the solution into a sample cell.



6. Add 0.5 mL of Molybdenum 2 Reagent to the sample cell. Swirl to mix. This is the prepared sample.

Note: *Molybdenum will cause a green color to form.*

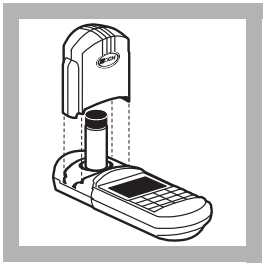


7. Press:

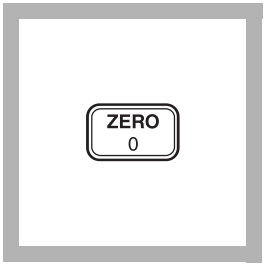
TIMER ENTER

A two-minute reaction period will begin.

8. Fill a second sample cell with 10 mL of solution from the graduated cylinder (the blank).



9. Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

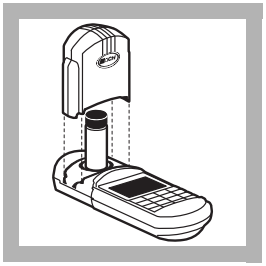


10. Press: **ZERO**

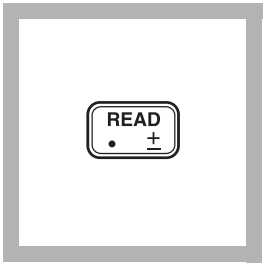
The cursor will move to the right, then the display will show:

0.00 mg/L Mo6

Note: *If Reagent Blank Correction is on, the display may flash "limit" (see Section 1).*



11. Place the developed sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L molybdate molybdenum will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in glass or plastic bottles.

Accuracy Check

Standard Addition Method

a) Add 25 mL of sample to three 25-mL mixing cylinders.

b) Snap the neck off a Molybdenum PourRite Ampule Standard Solution, 75 mg/L Mo^{6+}.

c) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to three 25-mL samples. Mix thoroughly.

d) Analyze 20 mL of each spiked sample as described in the procedure. The molybdenum concentration reading should increase by 0.3 mg/L for each 0.1 mL addition of standard.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 2.0-mg/L molybdenum standard solution by pipetting 10 mL of a 10-mg/L Molybdenum Standard Solution into a 50-

mL graduated mixing cylinder. Dilute to the mark with deionized water and mix thoroughly. Analyze 20 mL of this solution according to the procedure.

Method Performance

Precision

In a single laboratory using standard solutions of 2.00 mg/L Mo^{6+} and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.009 mg/L Mo^{6+}.

Estimated Detection Limit

The estimated detection limit for program 47 is 0.07 mg/L Mo^{6+}. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interference studies were conducted by preparing a molybdenum standard solution (2 mg/L Mo^{6+}) as well as a solution of the potential interfering ion. When the standard solution concentration changed by ±5% with a given ion concentration, the ion was considered an interference.

Table 1 Negative Interferences

Ion	Level above which it interferes (mg/L)
Iron	200
Copper	98
Chromium (Cr^{6+})	4.5[1]
Chloride	1,400
AMP (Phosphonate)	15
Phosphonohydroxyacetic Acid	32
Bisulfate	3,300
Nitrite	350
Aluminum	2
Acrylates	790
Alum	7
Lignin Sulfonate	105
Orthophosphate	4,500
Bicarbonate	5,650
EDTA	1,500
Borate	5,250
Ethylene Glycol	2% (by volume)
Sulfite	6,500
Diethanoldithiocarbamate	32

Table 1 Negative Interferences (continued)

Ion	Level above which it interferes (mg/L)
Positive Interferences	
Carbonate	1,325
Silica	600
Benzotriazole	210
Morpholine	6

[1] Read molybdenum concentration immediately after the completion of the two-minute reaction period.

Table 2 No Interference

Ion	Highest Concentration Tested (mg/L)
Zinc	400
Calcium	720
Magnesium	8,000
Manganese	1,600
Chlorine	7.5
PBTC (phosphonate)	500
Sulfate	12,800
Bisulfite	9,600
Nickel	250

Phosphonate HEDP at concentrations up to 30 mg/L will increase the apparent molybdenum concentration reading by approximately 10% (positive interference). For these samples, multiply the value obtained in step 12 by 0.9 to obtain the actual molybdenum concentration. As the concentration of HEDP increases above 30 mg/L, a decrease in the molybdenum concentration reading occurs (negative interference).

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagent and require pretreatment. Adjust the sample pH to 3-5 (use a pH meter or pH paper) by adding drops of an acid or base such as 1.0 N Sulfuric Acid Standard Solution, or 1.0 N Sodium Hydroxide Standard Solution. If a significant volume of acid or base is used, correct the result by dividing the total volume (sample + acid + base) by the original volume and multiplying the test result by this factor.

Large interferences are caused by some biocides used in cooling tower samples. Hach recommends testing the ternary complex procedure on molybdenum standards containing the specific biocides in use to determine if the ternary complex method will work with these samples.

After many samples have been analyzed, the sample cells may show a slight blue color. Rinse with Hydrochloric Acid Solution, 1:1, to eliminate the build-up.

Summary of Method

The ternary complex method for molybdenum determination is a method in which molybdate molybdenum reacts with an indicator and sensitizing agent to give a stable blue complex.

REQUIRED REAGENTS

Molybdenum Reagent Set, 20 mL sample (100 tests) 24494-00
Includes: (1) 23524-49, (1) 23525-12

Description	Quantity Required Per Test	Unit	Cat. No.
Molybdenum 1 Reagent for 20 mL sample size	1 pillow	100/pkg	23524-49
Molybdenum 2 Reagent Solution	0.5 mL	.50 mL MDB	23525-12

REQUIRED APPARATUS

Cylinder, mixing, graduated, 25 mL	1	each	1896-40
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Hydrochloric Acid Solution, 1:1, 6.0 N	500 mL	884-49
Molybdenum Standard Solution, Ampule 75 mg/L Mo^{6+}, 2 mL	20/pkg	25575-20
Molybdenum Standard Solution, 10 mg/L Mo^{6+}	100 mL	14187-42
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Cylinder, mixing, graduated, 50 mL		each	1896-41
Filter Paper, folded, 12.5 cm		100/pkg	1894-57
Funnel, poly, 65 mm		each	1083-67
pH Paper, 1-11 pH units		5 rolls/pkg	391-33
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet, volumetric, 10.00 mL, Class A		each	14515-38
Pipet Filler, safety bulb		each	14651-00
PourRite Ampule Breaker		each	24846-00

For Technical Assistance, Price and Ordering

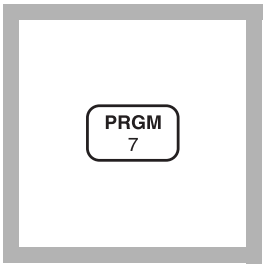
In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

MOLYBDENUM, MOLYBDATE, High Range (0 to 40.0 mg/L)

Mercaptoacetic Acid Method* — For water and wastewater

Using Powder Pillows



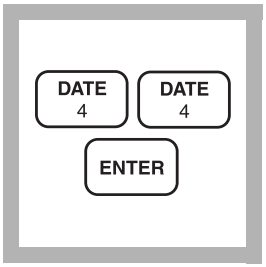
1. Enter the stored program number for high range molybdenum-powder pillows

Press: **PRGM**

The display will show:

PRGM ?

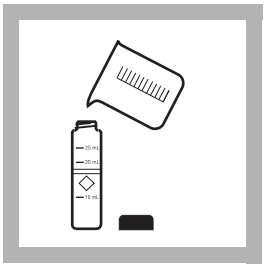
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **44 ENTER**

The display will show **mg/L, Mo6** and the **ZERO** icon**.**

Note: *For alternate form (MoO_4), press the* **CONC** *key.*

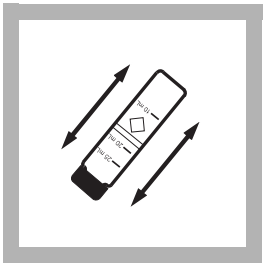


3. Fill a sample cell with 10 mL of sample.

Note: *Filter turbid samples.*

Note: *Adjust pH of stored samples before analysis.*

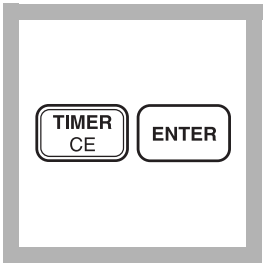
4. Add the contents of one MolyVer 1 Reagent Powder Pillow. Cap the cell and invert several times to mix.



5. Add the contents of one MolyVer 2 Reagent Powder Pillow. Cap the cell and invert several times to mix.

6. Add the contents of one MolyVer 3 Reagent Powder Pillow. Cap the cell and invert several times to mix. This is the prepared sample.

Note: *Accuracy is not affected by undissolved powder.*

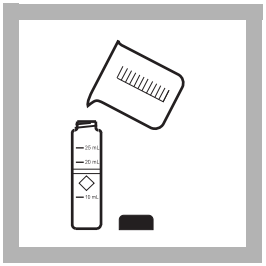


7. Press:

TIMER ENTER

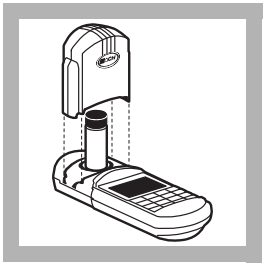
A five-minute reaction period will begin.

Note: *Molybdenum will cause a yellow color to form.*

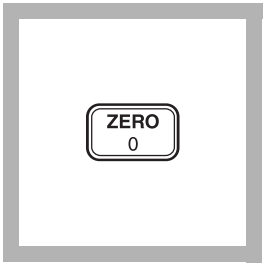


8. After the timer beeps, fill a second sample cell with 10 mL of sample (the blank).

* Adapted from Analytical Chemistry, 25(9) 1363 (1953).



9. Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

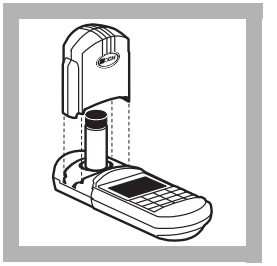


10. Press: **ZERO**

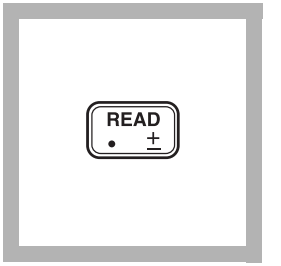
The cursor will move to the right, then the display will show:

0.0 mg/L Mo6

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



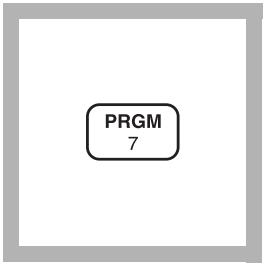
12. Press: **READ**

The cursor will move to the right, then the result in mg/L molybdenum (or alternate form) will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagents is highly recommended. See Accuracy Check.*

Using AccuVac Ampuls

Method 10046



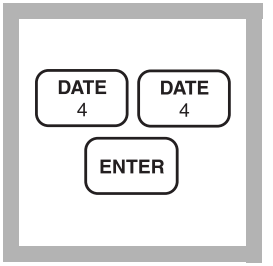
1. Enter the stored program number for high range molybdenum using AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

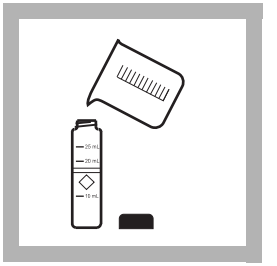
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **44 ENTER**

The display will show **mg/L, Mo6** and the **ZERO** icon**.**

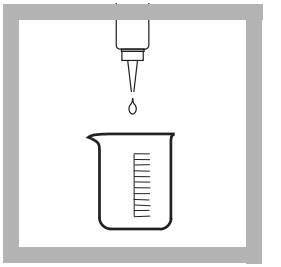
Note: *For alternate form (MoO_4), press the* **CONC** *key.*



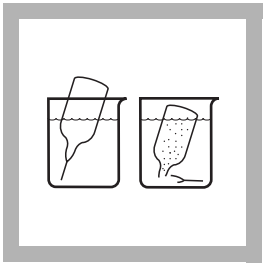
3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Filter turbid samples.*

Note: *Adjust the pH of stored samples before analysis.*

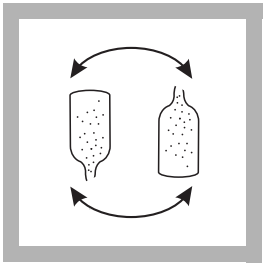


4. Add 4 drops of 0.4 M CDTA Solution to the beaker. Swirl to mix.



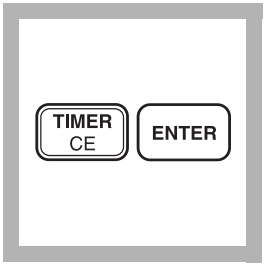
5. Fill a MolyVer 6 Reagent AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills.*



6. Invert the ampul repeatedly to mix. Wipe off any liquid or fingerprints.

Note: *Undissolved reagent will not affect the result.*

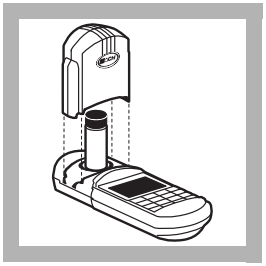


7. Press:

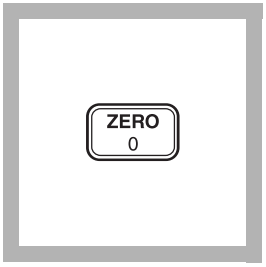
TIMER ENTER

A five-minute reaction period will begin.

Note: *If molybdenum is present a yellow color will develop.*



8. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

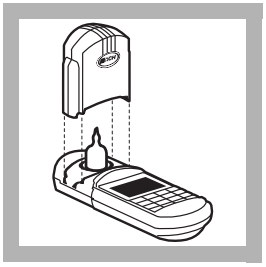


9. Press: **ZERO**

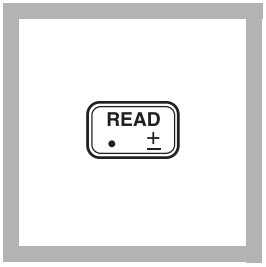
The cursor will move to the right, then the display will show:

0.0 mg/L Mo6

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



10. Place the AccuVac Ampul in the cell holder. Tightly cover the ampul with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L molybdenum will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic bottles. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to 6 months at room temperature. Adjust the pH to 7 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Volume Additions* (*Section 1*) for more information.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Molybdenum Voluette Ampule Standard Solution, 500 mg/L Mo^{6+}.

c) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper each and mix thoroughly.

d) For analysis with AccuVac Ampuls, transfer solutions to dry, clean 50-mL beakers. For analysis with powder pillows, transfer only
10 mL of solution to the sample cells.

e) Analyze each standard addition sample as described in the procedure. The molybdenum concentration reading should increase 2.0 mg/L for each 0.1 mL of standard added.

f) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

Standard Solution Method

To assure the accuracy of the test, use a Molybdenum Standard Solution, 10.0 mg/L Mo^{6+}. Follow the procedure for powder pillows or AccuVac Ampuls. Results should be between 9.0 and 11.0 mg/L Mo^{6+}.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustmen*t for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 20.0 mg/L Mo^{6+} and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of ±0.3 mg/L Mo^{6+}.

In a single laboratory using a standard solution of 20.0 mg/L Mo^{6+} and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.1 mg/L Mo^{6+}.

Estimated Detection Limit

The estimated detection limit for program 44 is 0.2 mg/L Mo^{6+}. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 50 mg/L
Chromium	Greater than 1000 mg/L
Copper	Samples containing 10 mg/L copper or more will exhibit an increasing positive interference upon standing. Read these samples as soon as possible after the five-minute reaction period is complete.
Iron	Greater than 50 mg/L
Nickel	Greater than 50 mg/L
Nitrite	Interference from up to 2000 mg/L as NO_2^- can be eliminated by adding one Sulfamic Acid Powder Pillow to the sample.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see *Section 1, pH Interferences*.

Summary of Method

Powder Pillows

MolyVer 1 and 2 Reagents are added to buffer and condition the sample. MolyVer 1 contains a buffer to control the pH in addition to a chelating agent to mask interferences. MolyVer 3 provides the mercaptoacetic acid, which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration.

AccuVac Ampuls

The CDTA Solution masks metal interferences. The MolyVer 6 reagent provides the mercaptoacetic acid, which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration.

REQUIRED REAGENTS (for Powder Pillows)

	Cat. No.
Molybdenum Reagent Set, 10 mL (100 tests)	26041-00
Includes: (1) 26042-99, (1) 26043-99, (1) 26044-99	

Description	Quantity Required Per Test	Unit	Cat. No.
MolyVer 1 Reagent Powder Pillows	1 pillow	100/pkg	26042-99
MolyVer 2 Reagent Powder Pillows	1 pillow	100/pkg	26043-99
MolyVer 3 Reagent Powder Pillows	1 pillow	100/pkg	26044-99

REQUIRED REAGENTS (for AccuVac Ampuls)

MolyVer 6 Molybdenum AccuVac Reagent Set (25 tests)			25220-98
Includes: (1) 25220-25, (1) 26154-36			
CDTA Solution 0.4M	4 drops	15 mL SCDB	26154-36
MolyVer 6 Reagent AccuVac Ampuls	1 ampul	25/pkg	25220-25

REQUIRED APPARATUS (for Powder Pillows)

Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

REQUIRED APPARATUS (for AccuVac Ampuls)

Beaker, 50 mL	2	each	500-41H
Sample Cell, 10-20-25 mL, w/cap	1	6/pkg	24019-06

OPTIONAL REAGENTS

Molybdenum Standard Solution, 10 mg/L Mo^{6+}	100 mL	14187-42
Molybdenum Standard Solution, Voluette Ampule, 500 mg/L Mo^{6+}, 10 mL	16/pkg	14265-10
Nitric Acid, ACS	500 mL	152-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sulfamic Acid Powder Pillows	100/pkg	1055-99
Water, deionized	4 L	272-56

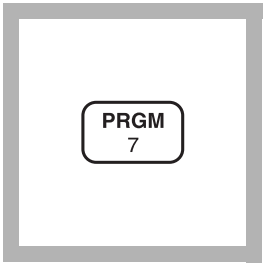
OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit	each	21968-00
Cylinder, graduated, mixing, 25 mL	each	20886-40
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, Erlenmeyer, 250 mL	each	505-46
Funnel, poly, 65 mm	each	1083-67
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28

Indophenol Method* (0–4.50 mg/L Cl_2 and 0–0.50 mg/L NH_3–N)

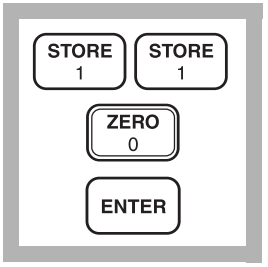
For finished chloraminated drinking water

Note: *For the most accurate chloramine results, determine a reagent blank for each new lot of reagent using deionized water instead of sample. Subtract the blank value from the final chloramine result.*



1. Enter the user program number for monochloramine.

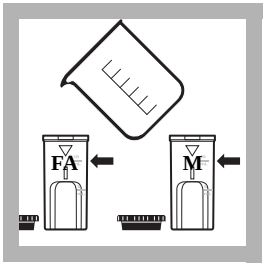
Press: **PRGM**
The display will show:
PRGM?



2. Press:
110 ENTER

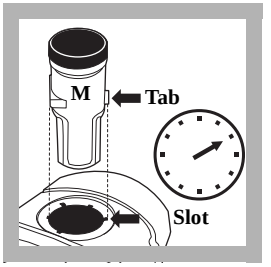
The display will show
mg/L Cl_2
and the zero icon.

Note: *For alternate forms, press the* **CONC** *key.*



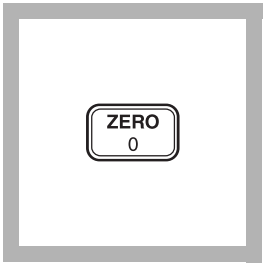
3. Fill two cells with 10 mL of sample

Label one cell "Free Ammonia" and one cell "Monochloramine".



4. Place the Monochloramine cell into the instrument so that the cell tab is at the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

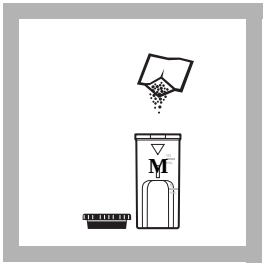
Tightly cover the sample cell with the instrument cap.



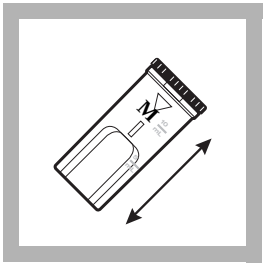
5. Press: **ZERO**

The cursor will move to the right, then the display will show:
0.00 mg/L Cl_2

Remove the cell from the instrument.

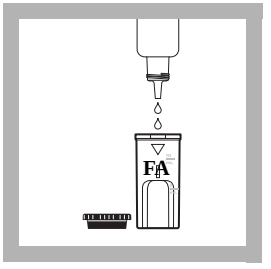


6. Add the contents of one pillow of Monochlor F to the cell for the Monochloramine measurement.



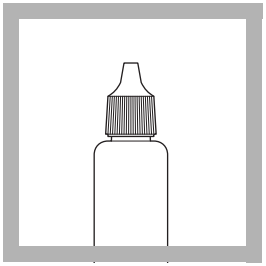
7. Cap the cell and shake for 20 seconds to dissolve the reagent.

A green color will form if monochloramine is present.

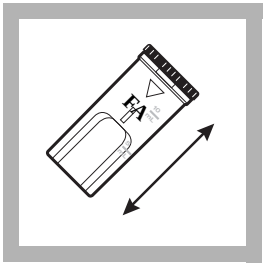


8. Add one drop of Free Ammonia Reagent Solution to the cell for Free Ammonia measurement.

* U.S. Patent 6,315,950

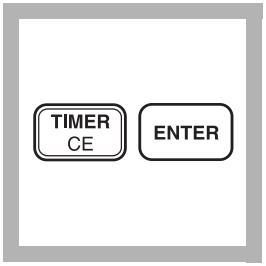


9. Cap the reagent bottle to maintain reagent performance and stability.



10. Cap the cell and mix.

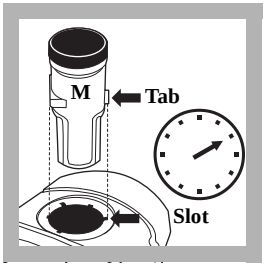
Note: *If the sample becomes cloudy by the end of the reaction period, pretreat the sample and retest. See Interferences on page 296.*



11. Press:
TIMER ENTER

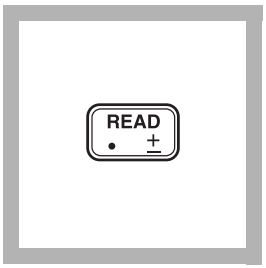
A five-minute reaction period will begin.

Note: *The color development time depends on the sample temperature. See Table 1. For accurate results allow the full reaction period to occur.*



12. When the timer expires, place the Monochloramine cell into the instrument so that the cell tab is in the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

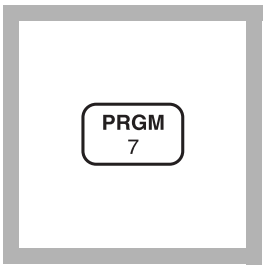
Tightly cover the sample cell with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L Monochloramine (as Cl_2 or chosen units) will be displayed.

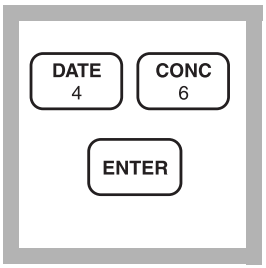
Leave the cell in the instrument.



14. Enter the stored program number for Free Ammonia.

Press: **PRGM**

The display will show PRGM?



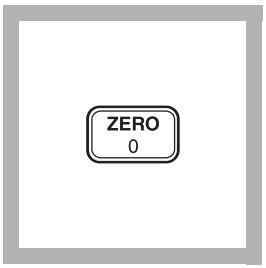
15. Press:
46 ENTER

The display will show

NH_3–N

and the zero icon.

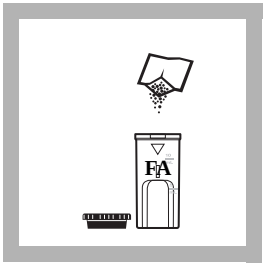
Note: *For alternate forms, press the* **CONC** *key.*



16. With the Monochloramine sample still in the cell holder, press **ZERO**.

The cursor will move to the right, then the display will show: 0.00 mg/L NH_3–N.

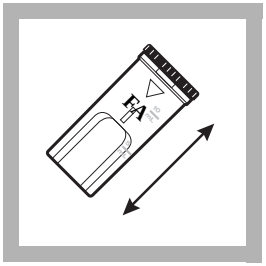
Remove the cell from the instrument.



17. Add the contents of one pillow of Monochlor F to the cell for the Free Ammonia measurement.

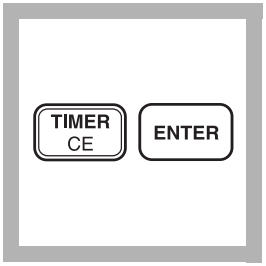
Cap and shake the cell about 20 seconds to dissolve the reagent.

Note: *The reaction period indicated in step 11 must be complete before the addition of Monochlor F to the cell for free ammonia measurement.*



18. Cap and shake the cell about 20 seconds to dissolve the reagent.

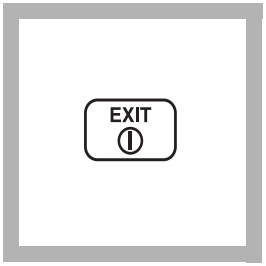
A green color will form if ammonia or monochloramine is present.



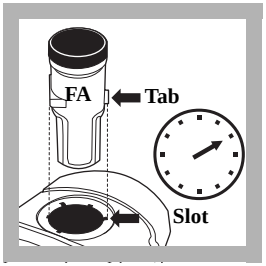
19. Press: **TIMER ENTER**

A five-minute reaction period will begin.

Note: *The color development time depends on the sample temperature. See Table 1.*

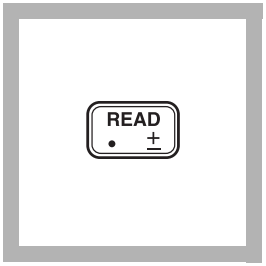


20. After the timer has expired, press: **EXIT**



21. Place the Free Ammonia cell into the instrument so that the cell tab is at the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

Tightly cover the sample cell with the instrument cap.



22. Press: **READ**

The cursor will move to the right, then the result in mg/L free ammonia as nitrogen (NH_3–N) or chosen units will be displayed.

Sampling and Storage

Collect samples in clean glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

Color Development Time

Test results are strongly influenced by sample temperature. **Both reaction periods in the procedure are the same and depend on the temperature of the sample.** The reaction periods indicated in the procedure are for a sample temperature of 18–20 °C (68–73 °F). Adjust both reaction periods according to Table 3.

Table 3 Reaction Period

Sample Temperature		Reaction Periods (Minutes)
° C	° F	
5	40	10
7	42	9
9	48	8
10	50	8
12	54	7
14	58	7
16	61	6
18	68	5
20	73	5
23	75	2.5
25	77	2
>25	>77	2

Interferences

This method is intended for finished, chloraminated drinking water samples that have a measurable combined (total) chlorine disinfectant residual. Samples where the disinfectant residual has disappeared and samples which exhibit a chlorine demand may produce low ammonia test results. Blanks and ammonia standards analyzed without a disinfectant residual must be prepared using high quality, reagent grade water.

The following do not interfere in free ammonia determination when at or below the stated concentration.

Substance	Level Tested
Aluminum	0.2 mg/L Al
Chloride	1200 mg/L Cl
Copper	1 mg/L Cu
Iron	0.3 mg/L Fe
Manganese	0.05 mg/L Mn
Nitrate	10 mg/L NO_3–N
Nitrite	1 mg/L NO_2–N
Phosphate	2 mg/L –PO_4
Silica	100 mg/L SiO_2
Sulfate	1600 ppm as $CaCO_3$
Zinc	5 ppm Zn

Samples containing high levels of both Total Hardness and Alkalinity may become turbid (cloudy) after the addition of the Free Ammonia Reagent Solution. If this occurs by the end of the first reaction period, the sample for Free Ammonia measurement must be pretreated as follows:

Note: *The sample for Monochloramine measurement does not need pretreatment.*

1. Measure 10 mL of sample into the cell for Free Ammonia measurement.
2. Add the contents of one Hardness Treatment Reagent Powder Pillow (Cat. No. 28823-46) to the sample.
3. Cap the cell and invert until the reagent is dissolved.
4. Remove the cap.

Continue with the analysis at step 2 using the pretreated sample as the Free Ammonia cell.

Accuracy Check (Monochloramine, Program 110)

1. Prepare the following monochloramine standard fresh before use.
2. Add the contents of one Buffer Powder Pillow, pH 8.3 to about 50-mL of organic-free water in a clean 100-mL Class A volumetric flask. Swirl to dissolve the powder.
3. Using a Class A volumetric pipet, transfer 2.00 mL of Nitrogen, Ammonia Standard Solution, 100 mg/L as NH_3–N into the flask.

4. Dilute to volume with organic-free water, cap and mix thoroughly. This is a 2.00 mg/L buffered ammonia standard.

5. Pipet 50.0 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a stir bar.

6. Obtain a recent lot of Chlorine Solution Ampules, 50–70 mg/L, and note the actual free chlorine concentration for this lot.

7. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

8. Open an ampule and, using a glass Mohr pipet, add the calculated amount of Chlorine Solution slowly to the ammonia standard, while mixing at medium speed on a stir plate.

9. Allow the monochloramine solution to mix for 1 minute after all Chlorine Solution is added.

Important Note: *Because of the strong buffer used in the preparation of this standard, it cannot be used for accuracy verification of the Free Ammonia test.*

10. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water, cap, and mix thoroughly. This is a nominal 4.5 mg/L (as Cl_2) monochloramine standard.

Use this standard within 1 hour of preparation.

Accuracy Check (Free Ammonia Test, Program 46)

Dilution water is required when testing a diluted sample and preparing standard solutions. Dilution water must be free of ammonia, chlorine and chlorine demand. A convenient source is a recirculating, deionizer system with carbon filtration which produces 18 megaohm-cm water.

Standard Additions Method

1. Measure 50 mL of sample into three 50-mL mixing cylinders.

2. Use the TenSette Pipet to add 0.3, 0.6, and 1.0 mL of Ammonium Nitrogen Standard, 10 mg/L as NH_3-N to the three samples. Mix well.

3. Analyze each spiked sample, following all steps of the Monochloramine and Free Ammonia procedure. The ammonia nitrogen concentration should increase 0.02 mg/L for each 0.1 mL of standard added.

4. If these increases do not occur, see *Standard Additions (Section 1 of the DR/890 Procedures Manual)* for more information.

Standard Solution Method

Prepare a 0.20 mg/L ammonia nitrogen standard by diluting 2.00 mL of the Ammonia Nitrogen Standard Solution, 10 mg/L, to 100 mL with dilution water. Or, using the TenSette Pipet, prepare a 0.20 mg/L ammonia nitrogen standard by diluting 0.4 mL of a Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as NH_3–N, to 100 mL with dilution water. Analyze the standard solution, following all steps of the Monochloramine and Free Ammonia procedure.

Method Performance

Monochloramine Test

Precision

In a single laboratory, using a monochloramine standard solution of 2.10 mg/L Cl_2 and representative lots of reagent, a single operator obtained a standard deviation of ± 0.12 mg/L Cl_2.

Estimated Detection Limit

The estimated detection limit for Method 10171 is 0.05 mg/L Cl_2.

Free Ammonia Test

Precision

In a single laboratory using a solution containing 1.80 mg/L Cl_2 plus 0.20 mg/L ammonia nitrogen (NH_3–N) and two representative lots of reagent with the DR/890, a single operator obtained a standard deviation of ± 0.01 mg/L N for seven replicates.

Estimated Detection Limit

The estimated detection limit for program 46 is 0.02 mg/L N.

For more information on the estimated detection limit, see *Section 1* of the *DR/850 or DR/890 Procedure Manual*.

Summary of Method

Monochloramine (NH_2Cl) and "free ammonia" (NH_3 and NH_4^+) can exist in the same water sample. Added hypochlorite combines with free ammonia to form more monochloramine. In the presence of a cyanoferrate catalyst, monochloramine in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate couples with excess substituted phenol to form a green-colored indophenol, which is proportional to the amount of monochloramine present in the sample. Free ammonia is determined by comparing the color intensities, with and without added hypochlorite.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet (MSDS) for information specific to the reagent used.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Free Ammonia Reagent Set (50 tests)			
Includes: (1) 28022-99, (1) 28773-36			28797-00
Free Ammonia Reagent Solution	1 drop	4 mL SCDB	28773-36
Monochlor F Reagent Pillows	2 pillows	100/pkg	28022-99

REQUIRED APPARATUS

Sample Cell, 1-cm/10-mL, with cap	2	2/pkg	48643-02

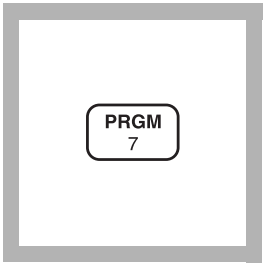
OPTIONAL REAGENTS

Buffer, pH 8.3, Powder Pillows	25/pkg	898-68
Chlorine Solution, Voluette® Ampule	16/pkg	14268-10
Hardness Treatment Reagent Pillows (1 per test)	50/pkg	28823-46
Nitrogen Ammonia Standard Solution, 10 mg/L as NH_3–N	500 mL	153-49
Nitrogen Ammonia Standard Ampule, 50 mg/L as NH_3–N, 10 mL	16/pkg	14791-10
Nitrogen Ammonia Standard Solution, 100 mg/L as NH_3–N	500 mL	24065-10

OPTIONAL APPARATUS

Description	Per Test	Unit	Cat. No.
Ampule Breaker Kit		each	21968-00
Beaker, 100 mL, Polypropylene		each	1080-42
Beaker, 100 mL, Glass		each	500-42H
Cylinder, 50 mL, mixing		each	20886-41
Flask, Volumetric, Class A, 100 mL		each	14574-42
Pipet Filler, Safety Bulb		each	14651-00
Pipet, TenSette®, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet, Mohr, Glass, 10 mL		each	20934-38
Pipet, Volumetric, Class A, 2.0 mL		each	14515-36
Pipet, Volumetric, Class A, 50.00 mL		each	14515-41
Scissors		each	28831-00
Stir Bar, Octagonal		each	20953-53
Stirrer, Magnetic		each	23436-00
Thermometer, –10 to 110 °C		each	1877-01
Wipers, Disposable Kimwipes®, 30 x 30 cm, 280/box		box	20970-01

PAN Method*

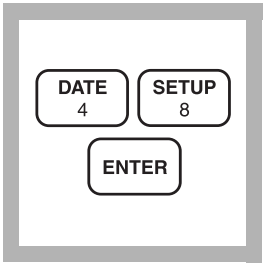


1. Enter the stored program number for nickel (Ni), PAN method.

Press: **PRGM**

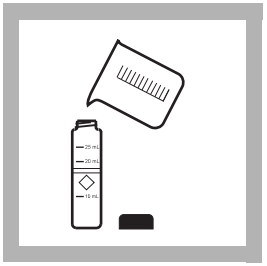
The display will show:

PRGM ?



2. Press: **48 ENTER**

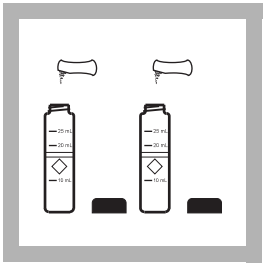
The display will show **mg/L, Ni** and the **ZERO** icon**.**



3. Fill a sample cell with 25 mL of sample (the prepared sample).

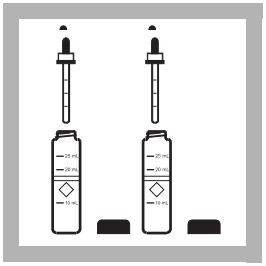
Note: *If sample is less than 10 °C (50 °F), warm to room temperature before analysis. Adjust the pH of stored samples.*

4. Fill a second sample cell with 25 mL of deionized water (the blank).



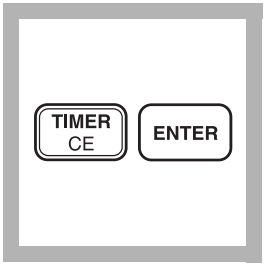
5. Add the contents of one Phthalate-Phosphate Reagent Powder Pillow to each cell. Cap. Invert several times to mix.

Note: *If sample contains iron (Fe^{3+}), all the powder must be dissolved completely before continuing with Step 6.*



6. Add 1.0 mL of 0.3% PAN Indicator Solution to each cell. Cap. Invert several times to mix.

Note: *Use the plastic dropper provided.*

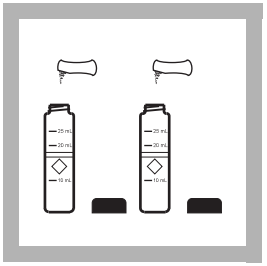


7. Press:

TIMER ENTER

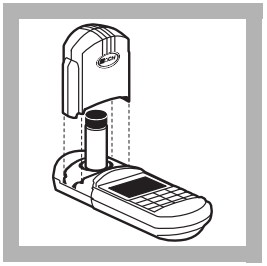
A 15-minute reaction period will begin.

Note: *The sample solution color may vary from yellowish-orange to dark red. The blank should be yellow.*

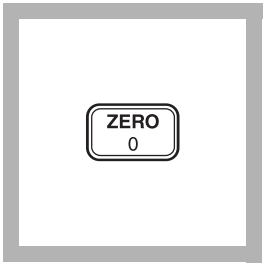


8. After the timer beeps, add the contents of one EDTA Reagent Powder Pillow to each cell. Cap. Invert several times to dissolve the reagent.

* Adapted from Watanabe, H., Talanta, 21 295 (1974)



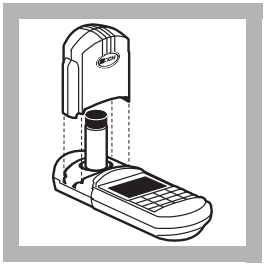
9. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



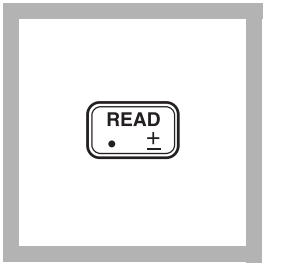
10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.000 mg/L Ni



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L nickel will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 5 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the sample pH to between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution just before analysis. Do not exceed pH 8 as this may cause some loss of nickel as a precipitate. Correct test results for volume additions, see *Correcting for Volume Additions*, (*Section 1*) for more information.

Accuracy Check

Standard Solution Method

Prepare a 0.5 mg/L nickel standard solution by diluting 10.0 mL of a 5 mg/L working stock solution to 100 mL in a 100-mL volumetric flask. The working stock solution should be prepared daily by diluting 5.00 mL of Nickel Standard Solution, 1000 mg/L as Ni, to 1000 mL with deionized water.

Or, using the TenSette Pipet, add 0.2 mL of a Nickel Voluette Ampule Standard Solution, 300 mg/L Ni, into a 100-mL volumetric flask. Dilute to volume with deionized water. This is a 0.6 mg/L standard solution.

Method Performance

Precision

In a single laboratory using a standard solution of 0.50 mg/L nickel and two representative lots of reagent with the instrument,

a single operator obtained a standard deviation of ±0.008 mg/L nickel.

Estimated Detection Limit

The estimated detection limit for program 48 is 0.013 mg/L Ni. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Interfering Substance	Interference Level
Al^{3+}	32 mg/L
Ca^{2+}	1000 mg/L as ($CaCO_3$)
Cd^{2+}	20 mg/L
Cl^-	8000 mg/L
Co	Causes a positive interference at all levels.
Cr^{3+}	20 mg/L
Cr^{6+}	40 mg/L
Cu^{2+}	15 mg/L
F^-	20 mg/L
Fe^{3+}	10 mg/L
Fe^{2+}	Interferes directly and must not be present.
K^+	500 mg/L
Mg^{2+}	400 mg/L
Mn^{2+}	25 mg/L
Mo^{6+}	60 mg/L
Na^+	5000 mg/L
Pb^{2+}	20 mg/L
Zn^{2+}	30 mg/L

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and required sample pretreatment; see *pH Interferences (Section 1)*.

Chelating agents, such as EDTA, interfere. Use either the Digesdahl or vigorous digestion (*Section 2*) to eliminate this interference.

Summary of Method

After buffering the sample and masking any Fe^{3+} with pyrophosphate, the nickel is reacted with 1-(2-Pyridylazo)-2-Naphthol indicator.

The indicator forms complexes with most metals present. After color development, EDTA is added to destroy all metal-PAN complexes except nickel and cobalt.

REQUIRED REAGENTS

	Cat. No.
Nickel Reagent Set, 25 mL sample (100 tests)	22426-00
Includes: (2) 7005-99, (4) 21501-66, (2) 21502-32	

Description	Quantity Required Per Test	Unit	Cat. No.
EDTA Reagent Powder Pillows	2 pillows	100/pkg	7005-99
Phthalate-Phosphate Reagent Powder Pillows	2 pillows	50/pkg	21501-66
P.A.N. Indicator Solution, 0.3%	2 mL	100 mL MDB	21502-32
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, mixing, 25 mL	1	each	20886-40
Sample Cell, 10-20-25, w/caps	2	6/pkg	24019-06

OPTIONAL REAGENTS

Nickel Standard Solution, 1000 mg/L Ni		100 mL	14176-42
Nickel Standard Solution, Voluette Ampule, 300 mg/L Ni, 10 mL		16/pkg	14266-10
Nitric Acid, ACS		500 mL	152-49
Nitric Acid Solution, 1:1		500 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N		100 mL MDB	2450-32

OPTIONAL APPARATUS

Ampule Breaker Kit		each	21968-00
Flask, volumetric, Class A, 100 mL		each	14574-42
Flask, volumetric, Class A, 1000 mL		each	14574-53
pH Paper, 1 to 11 pH units		5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable, with electrode		each	51700-10
Pipet, serological, 1 mL		each	9190-02
Pipet, serological, 5 mL		each	532-37
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
Pipet, volumetric, Class A, 5.0 mL		each	14515-37
Pipet, volumetric, Class A, 10.0 mL		each	14515-38
Pipet Filler, safety bulb		each	14651-00
Thermometer, –20 to 110 °C, non-mercury		each	26357-02

For Technical Assistance, Price and Ordering

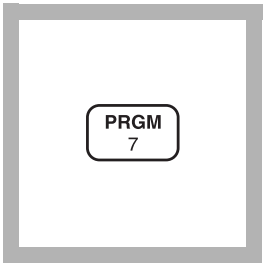
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITRATE, High Range (0 to 30.0 mg/L NO_3^--N) For water, wastewater, and seawater*

Cadmium Reduction Method (Using Powder Pillows or AccuVac Ampuls)

Using Powder Pillows



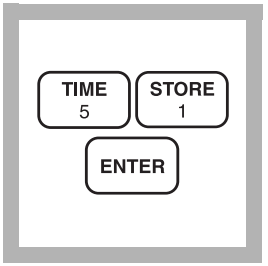
1. Enter the stored program number for high range nitrate nitrogen (NO_3^-–N) powder pillows.

Press: **PRGM**

The display will show:

PRGM ?

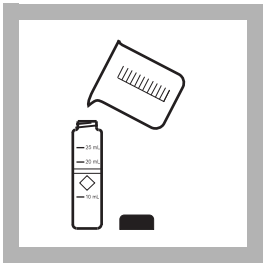
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **51 ENTER**

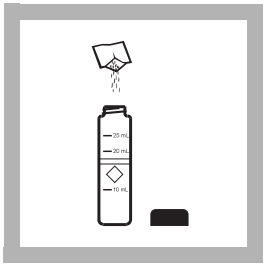
The display will show **mg/L, NO3-N** and the **ZERO** icon**.**

Note: *For alternate forms (NO_3), press the* **CONC** *key.*



3. Fill a sample cell with 10 mL of sample.

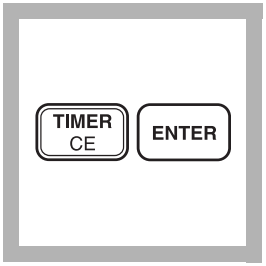
Note: *Adjust the pH of stored samples before analysis.*



4. Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow to the sample cell (the prepared sample). Cap the sample cell.

Note: *It is important to remove all of the powder from the foil pillow. Tap the pillow until no more powder pours out.*

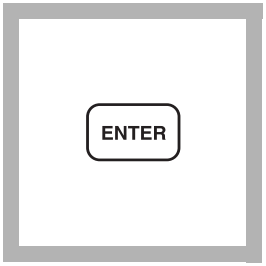
* Seawater requires a manual calibration; see Interferences.



5. Press:
TIMER ENTER

A one-minute reaction period will begin. Shake the sample cell vigorously until the timer beeps.

Note: *It is important to shake the cell vigorously. Shaking time and technique influence color development. For most accurate results, do successive tests on a standard solution and adjust the shaking time to obtain the correct result.*



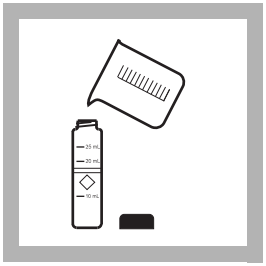
6. After the timer beeps, the display will show:
5:00 TIMER 2

Press: **ENTER**

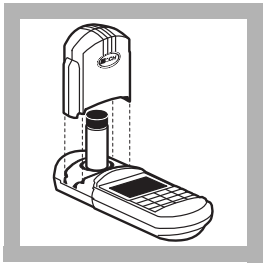
A five-minute reaction period will begin.

Note: *A deposit will remain after the reagent dissolves and will not affect test results.*

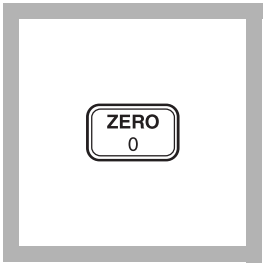
Note: *An amber color will develop if nitrate nitrogen is present.*



7. Fill another cell with 10 mL of sample (the blank). Wipe off any fingerprints or liquid.



8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

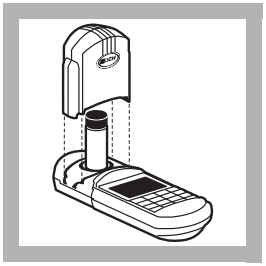


9. When the timer beeps, press **ZERO.**

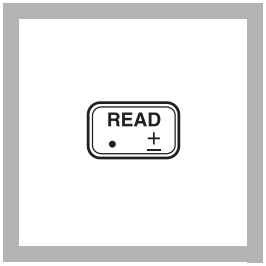
The cursor will move to the right, then the display will show:

0.0 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



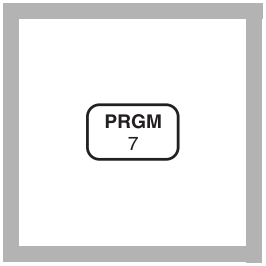
11. Press: **READ**

The cursor will move to the right, then the result in mg/L NO_3-N (or alternate form) will be displayed.

Note: *Use of the Standard Adjust feature for each new lot of reagent is highly recommended. See* Accuracy Check.

Note: *Rinse the sample cell immediately after use to remove all cadmium particles. Save the spent sample for proper hazardous waste disposal for cadmium.*

Using AccuVac Ampuls



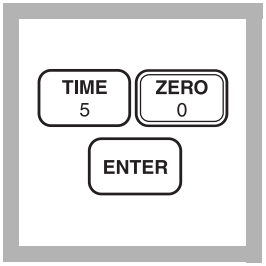
1. Enter the stored program number for high range nitrate nitrogen (NO_3^-–N) AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

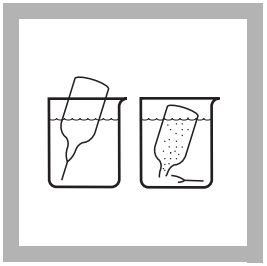
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **50 ENTER**

The display will show **mg/L, NO3-N** and the **ZERO** icon.

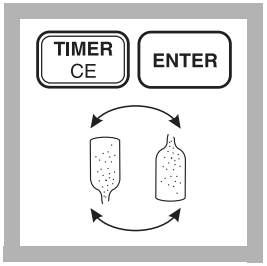
Note: *For alternate forms (NO_3), press the* **CONC** *key.*



3. Collect at least 40 mL of sample in a 50-mL beaker. Fill a NitraVer 5 Nitrate AccuVac Ampul with sample. Place a stopper over the tip of the ampul.

Note: *Keep the tip immersed while the ampul fills. The ampul will not fill completely.*

Note: *Adjust the pH of stored samples before analysis.*

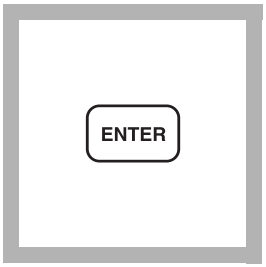


4. Press:

TIMER ENTER

A one-minute mixing period will begin. Invert the ampul repeatedly back and forth until the timer beeps. Wipe off any liquid or fingerprints.

Note: *Mixing time and technique influence color development. For most accurate results, do successive tests on a standard solution and adjust the mixing time to obtain the correct result.*



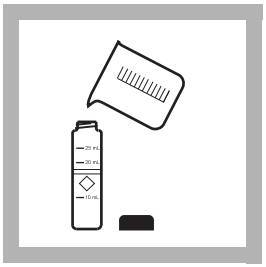
5. The display will show: **5:00 TIMER 2**

Press: **ENTER**

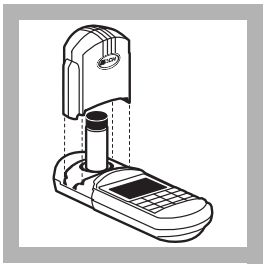
A five-minute reaction period will begin.

Note: *A deposit will remain after the reagent dissolves and will not affect results.*

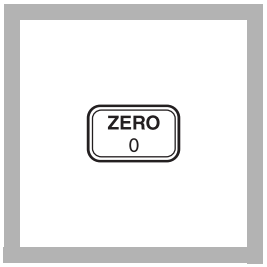
Note: *An amber color will develop if nitrate nitrogen is present.*



6. Fill a sample cell with at least 10 mL of sample (the blank).



7. When the timer beeps, place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

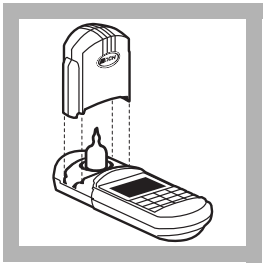


8. Press: **ZERO**

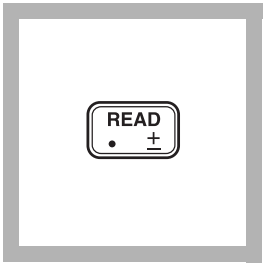
The cursor will move to the right, then the display will show:

0.0 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



9. Place the AccuVac Ampul into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L NO_3-N (or alternate form) will be displayed.

Note: *Use of the Standard Adjust feature for each new lot of reagent is highly recommended. See* Accuracy Check.

Note: *See Pollution Prevention and Waste Management for proper disposal of cadmium.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions* (*Section 1*) for more information.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL mixing cylinders with 25 mL of sample.

b) Snap the neck off a Nitrate Nitrogen Ampule Standard, 500 mg/L nitrate nitrogen.

c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of Nitrate Nitrogen Standard Solution to the three samples. Stopper and mix thoroughly.

d) For AccuVac analysis, transfer the solutions to clean, dry 50-mL beakers. For analysis with powder pillows, transfer only 10 mL of solution to clean, dry sample cells.

e) Analyze each sample as described above. The nitrate nitrogen (NO_3^--N) concentration should increase 2.0 mg/L for each 0.1 mL of standard added.

f) If these increases do not occur, see *Standard Additions* (*Section 1*) for more information.

Standard Solution Method

Use a Hach Nitrate-Nitrogen Standard Solution, 10.0 mg/L NO_3^--N, listed under Optional Reagents as the sample and perform the procedure as described above.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the curve adjustment. See *Section 1, Standard Curve Adjustmen*t for more information. If you are using a reagent blank correction, the blank correction should be entered before the Standard Adjust value is entered.

Method Performance

Precision

In a single laboratory using standard solutions of 25.0 mg/L nitrate nitrogen (NO_3^--N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of $\pm$0.3 mg/L nitrate nitrogen for program #50 and ±1.7 mg/L nitrate nitrogen for program # 51.

Estimated Detection Limit

The estimated detection limit for program 50 is 0.5 mg/L NO_3^--N and 0.8 mg/L NO_3^--N for program 51. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	All levels
Nitrite	All levels Compensate for nitrite interference as follows: Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. Add one drop of 30-g/L Phenol Solution to destroy the color. Proceed with Step 4. Report the results as total nitrate and nitrite.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels.

Summary Of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to gentisic acid to form an amber-colored product.

Pollution Prevention and Waste Management

NitraVer 5 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous wastes by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
NitraVer 5 Nitrate Reagent Powder Pillows	1 pillow	100/pkg	21061-69
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

REQUIRED REAGENTS (Using AccuVac Ampuls)

NitraVer 5 Nitrate Reagent AccuVac Ampul	1 ampul	25/pkg	25110-25

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41H
Stopper	1	6/pkg	1731-06

OPTIONAL REAGENTS

Bromine Water 30 g/L		29 mL*	2211-20
Nitrate Nitrogen Standard Solution, 10.0 mg/L as (NO_3^--N)		500 mL	307-49
Nitrate Nitrogen Standard Solution, 1000 mg/L as (NO_3^--N)		500 mL	12792-49
Nitrate Nitrogen Standard Solution, PourRite ampule, 500 mg/L as NO_3^--N, 2 mL		20/pkg	14260-20
Phenol Solution		29 mL	2112-20
Sodium Hydroxide Standard Solution, 5.0 N		50 mL*	2450-26
Sulfuric Acid, ACS		500 mL*	979-49
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit		each	24052-00
Cylinder, graduated, mixing, 25 mL		each	1896-40
Dropper, for 29-mL bottle		each	2258-00
pH Indicator Paper, 1 to 11 pH		5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable, with electrode		each	51700-10
Pipet Filler, safety bulb		each	14651-00
Pipet, serological, 2 mL		each	532-36
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite Ampule Breaker		each	24846-00
Thermometer, –20 to 110 °C, non-mercury		each	26357-02

For Technical Assistance, Price and Ordering

In the U.S.A. call 800-227-4224

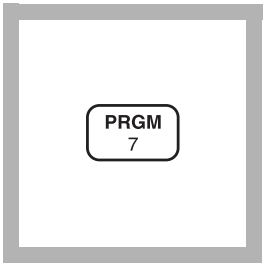
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes.

NITRATE, Mid Range (0 to 5.0 mg/L NO_3^--N) For water, wastewater and seawater*

Cadmium Reduction Method (Using Powder Pillows or AccuVac Ampuls)

Using Powder Pillows



1. Enter the stored program number for medium range nitrate nitrogen using powder pillows.

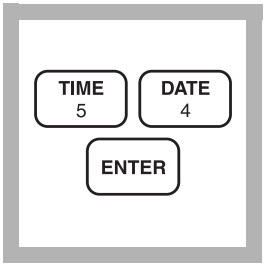
Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

Note: *Adjust the pH of stored samples before analysis.*

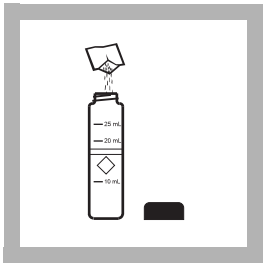


2. Press: **54 ENTER**

The display will show **mg/L, NO3-N** and the **ZERO** icon**.**

Note: *For alternate form (NO_3), press the* **CONC** *key.*

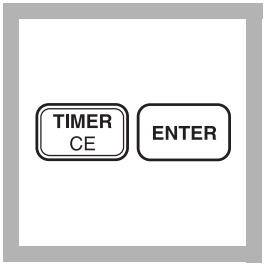
3. Fill two sample cells with 10 mL of sample each. One cell will be the prepared sample, the other is the blank. Set the blank aside.



4. Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow to one cell (the prepared sample). Cap the cell.

Note: *It is necessary to remove all the powder from the foil pouch by tapping repeatedly until no more powder comes out.*

* Seawater requires a manual calibration; see *Interferences*.

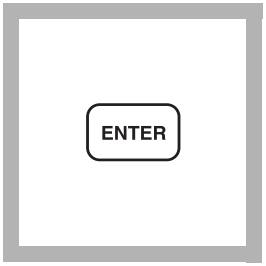


5. Press:

TIMER ENTER

A one-minute reaction period will begin. Shake the sample vigorously until the timer beeps.

Note: *Shaking time and technique influence color development. Low results usually occur if shaking is not vigorous enough. For most accurate results, do successive tests on a standard solution and adjust the shaking time by ±1 minute to obtain the correct result. See the Accuracy Check section for more information.*



6. After the timer beeps, the display will show:
5:00 TIMER 2

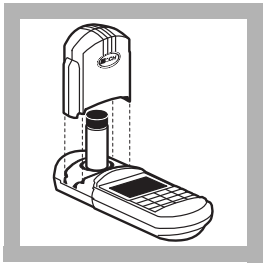
Press: **ENTER**

A five-minute reaction period will begin.

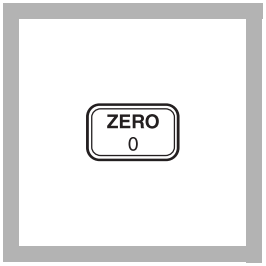
Note: *A cadmium deposit will remain after the NitraVer 5 Nitrate Reagent Powder dissolves and will not affect test results.*

Note: *An amber color will develop if nitrate nitrogen is present.*

7. After the timer beeps, wipe off any liquid or fingerprints.



8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

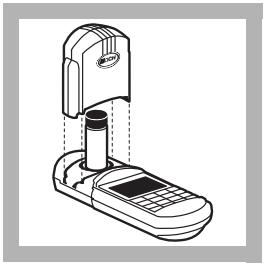


9. Press: **ZERO**

The cursor will move to the right, then the display will show:

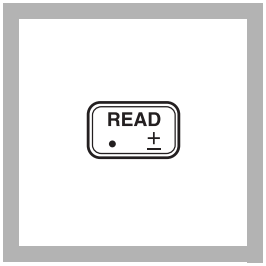
0.0 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit".*



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Read the sample within two minutes after the timer beeps.*



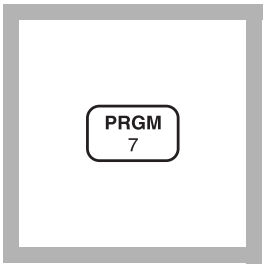
11. Press: **READ**

The cursor will move to the right, then the result in mg/L NO_3-N (or NO_3) will be displayed.

Note: *Use of the standard adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Note: *Rinse the sample cell immediately after use to remove all the cadmium particles. See Pollution Prevention and Waste Management following these steps for disposal of cadmium particles.*

Using AccuVac Ampuls



1. Enter the stored program number for medium range nitrate nitrogen using AccuVac Ampuls.

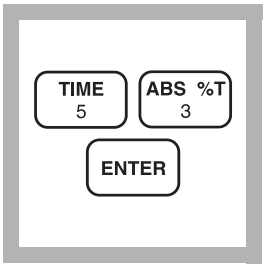
Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

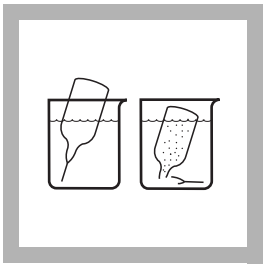
Note: *Adjust the pH of stored samples before analysis.*



2. Press: **53 ENTER**

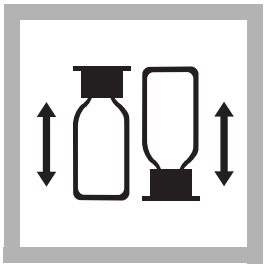
The display will show **mg/L, NO3-N** and the **ZERO** icon**.**

Note: *For alternate form (NO_3), press the* **CONC** *key.*



3. Collect at least 40 mL of sample in a 50-mL beaker. Fill a NitraVer 5 Nitrate AccuVac Ampul with sample. Place a stopper over the tip of the ampul.

Note: *Keep the tip immersed while the ampul fills. The ampul will not fill completely to allow room for mixing.*

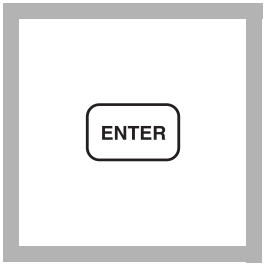


4. Press:

TIMER ENTER

A one-minute mixing period will begin. Invert the ampul repeatedly back and forth until the timer beeps. Wipe off any liquid or fingerprints after mixing.

Note: *Mixing speed and technique influence color development. For most accurate results, do successive tests on a standard solution and increase or decrease the mixing time to obtain the correct result. See Accuracy Check for more information.*



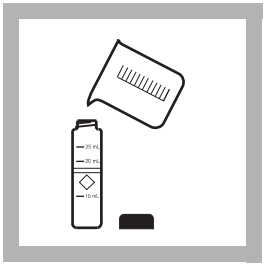
5. After the timer beeps, the display will show:
05:00 Timer 2

Press: **ENTER**

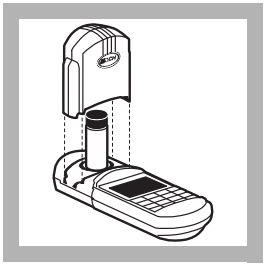
A five-minute reaction period will begin.

Note: *A cadmium deposit will remain after the NitraVer 5 Nitrate Reagent Powder dissolves and will not affect results.*

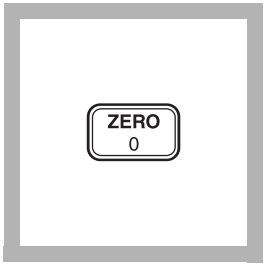
Note: *An amber color will develop if nitrate nitrogen is present.*



6. Fill a sample cell with at least 10 mL of sample (the blank).



7. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

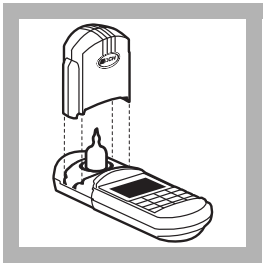


8. Press: **ZERO**

The cursor will move to the right, then the display will show:

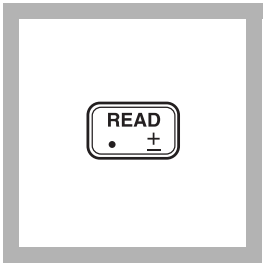
0.0 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



9. Place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Read the sample within two minutes after the timer beeps.*



10. Press: **READ**

The cursor will move to the right, then the result in mg/L NO_3-N (or NO_3) will be displayed.

Note: *Use of the standard adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions*, (*Section 1)* for more information.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Nitrate Nitrogen Ampule Standard Solution, 100 mg/L NO_3^--N.

c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the standard to the three samples. Stopper and mix well.

d) For analysis with AccuVac Ampuls, transfer the solutions to dry, clean 50 mL beakers. For analysis with powder pillows, transfer only 10 mL of the solution to dry, clean sample cells.

e) Analyze each sample as described above. The nitrate nitrogen (NO_3^--N) concentration should increase 0.4 mg/L for each 0.1 mL of standard added.

f) If these increases do not occur, see *Standard Additions* (*Section 1*) for more information.

Standard Solution Method

A 1.0 mg/L Nitrate Nitrogen Standard Solution is available from Hach. Use this standard in place of sample in the above procedure.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 1.00-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **1.0** to edit the

standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment . See *Section 1*, *Standard Curve Adjustmen*t for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 3.0 mg/L nitrate nitrogen (NO_3^--N) and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of ±0.2 mg/L nitrate nitrogen.

In a single laboratory using a standard solution of 3.0 mg/L NO_3^--N and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.1 mg/L nitrate nitrogen.

Estimated Detection Limit

The estimated detection limit for programs 53 and 54 is 0.2 mg/L NO_3-N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatments

Interfering Substance	Interference Levels and Treatments
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	All levels
Nitrite	All levels interfere. Compensate for nitrite interference as follows: **1.** Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. **2.** Add one drop of 30-g/L Phenol Solution to destroy the color. **3.** Proceed with Step 3. Report the results as total nitrate and nitrite.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels.

Summary of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to gentisic acid to form an amber-colored product.

Pollution Prevention and Waste Management

NitraVer 5 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Qty/ Test	Unit	Cat. No.
NitraVer 5 Nitrate Reagent Powder Pillows	1 pillow	100/pkg	21061-69
Sample Cell, 10-20-25 mL, w/ caps	2	6/pkg	24019-06

REQUIRED REAGENTS (Using AccuVac Ampuls)

NitraVer 5 Nitrate Reagent AccuVac Ampul	1 ampul	25/pkg	25110-25

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41
Stopper	1	6/pkg	1731-06

OPTIONAL REAGENTS

Bromine Water 30 g/L		29 mL*	2211-20
Drinking Water Standard, Inorganics, (Fe^{-}, NO_3^{-}, SO_4^{2-}, PO_4^{3-})		500 mL	28330-49
Nitrate Nitrogen Standard Solution, 1.0 mg/L as NO_3^{-}-N		500 mL	2046-49
Nitrate Nitrogen Standard Solution, 100 mg/L as NO_3^{-}-N		500 mL	1947-49
Nitrate Nitrogen Standard Solution, PourRite Ampule, 100 mg/L as NO_3^{-}-N, 2 mL		20/pkg	1947-20
Phenol Solution, 30 g/L		29 mL	2112-20
Sodium Hydroxide Standard Solution, 5.0 N		50 mL SCDB*	2450-26
Sulfuric Acid, ACS		500 mL*	979-49
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

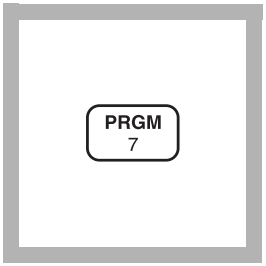
AccuVac Snapper Kit		each	24052-00
Cylinder, graduated, mixing, 25 mL		each	20886-40
Dropper, for 1-oz bottle		each	2258-00
pH Paper, 1 to 11 pH units		5 rolls/pkg	391-33
pH Meter, *sens**ion***™***1**, portable, with electrode		each	51700-10
Pipet Filler, safety bulb		each	14651-00
Pipet, serological, 2 mL		each	532-36
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite Ampule Breaker		each	24846-00

* Contact Hach for larger sizes.

NITRATE, Low Range (0 to 0.50 mg/L NO_3^--N)

For water, wastewater and seawater*

Cadmium Reduction Method



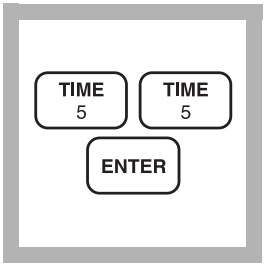
1. Enter the stored program number for low range nitrate nitrogen (NO_3^--N).

Press: **PRGM**

The display will show:

PRGM ?

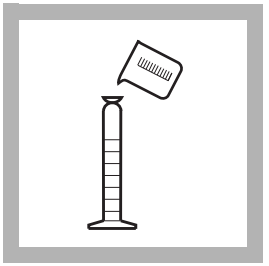
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **55 ENTER**

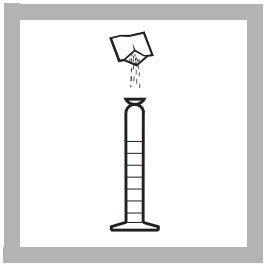
The display will show **mg/L, NO3-N** and the **ZERO** icon**.**

Note: *For alternate forms (NO_3), press the* **CONC** *key.*



3. Fill a 25-mL graduated mixing cylinder to the 15-mL mark with sample.

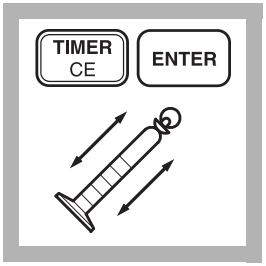
Note: *Adjust the pH of stored samples before analysis.*



4. Add the contents of one NitraVer 6 Nitrate Reagent Powder Pillow to the cylinder. Stopper.

Note: *It is necessary to remove* ***all*** *the powder from the foil pillow. Tap the pillow until no more powder pours out. Be sure to remove powder from the corners of the pillow.*

* Seawater requires a manual calibration; see Interferences.

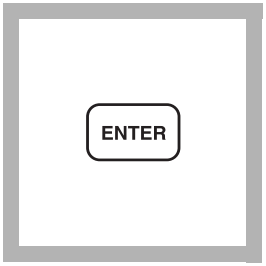


5. Press:

TIMER ENTER

A 3-minute reaction period will begin. Shake the cylinder vigorously throughout this three minute period.

Note: *Shaking time and technique influence color development. For most accurate results, analyze a standard solution several times and adjust the shaking time to obtain the correct result.*

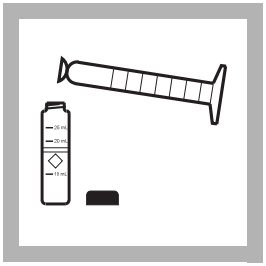


6. When the timer beeps, the display will show: **2:00 TIMER 2**

Press: **ENTER**

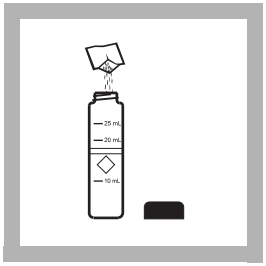
A 2-minute reaction period will begin.

Note: *A deposit will remain after the powder dissolves and will not affect results.*



7. When the timer beeps, pour 10 mL of the sample into a sample cell.

Note: *Do not transfer any cadmium particles.*



8. Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow to the sample cell (the prepared sample). Cap the cell and shake gently for 30 seconds.

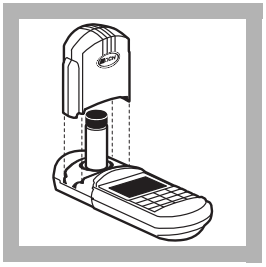
Note: *A pink color will form if nitrate is present.*

9. The display will show: **15:00 TIMER 3**

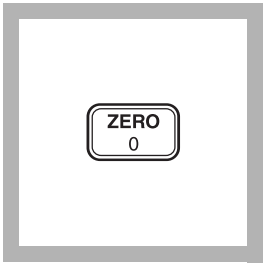
Press: **ENTER**

A 15-minute reaction period will begin.

Fill another sample cell (the blank) with 10 mL of sample.



10. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

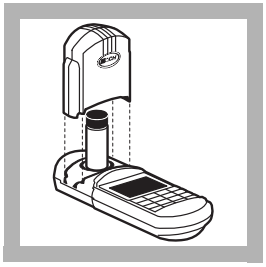


11. Press: **ZERO**

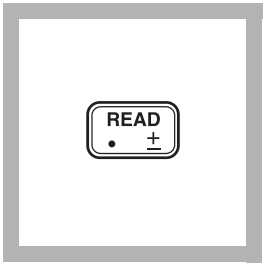
The cursor will move to the right, then the display will show:

0.00 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



12. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L NO_3^--N (or alternate form) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Note: *Rinse the sample cell and cylinder immediately after use to remove all cadmium particles.*

Note: *See Pollution Prevention and Waste Management for proper disposal of cadmium.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution. Do not use mercury compounds as preservatives. Correct the test result for volume additions; see *Correction for Volume Additions* (*Section 1*) for more information.

Accuracy Check

Standard additions Method

a) Fill three 25-mL graduated mixing cylinders with 15 mL of sample.

b) Snap the neck off a Nitrate Nitrogen Ampule Standard Solution, 12.0 mg/L NO_3^--N.

c) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of the standard to the three samples. Stopper and mix well.

d) Analyze each sample as described above. The nitrate nitrogen concentration should increase 0.08 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

Standard Solution Method

Prepare a 0.20 mg/L nitrate nitrogen standard by diluting 2.00 mL of a
10.0 mg/L Nitrate Nitrogen Standard Solution to 100.0 mL with deionized water. Use this standard in place of sample in Step 3.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 0.20-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **0.20** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the curve adjustment. If you are using a reagent blank correction, the blank correction should be entered before the Standard Adjust feature is entered. See *Section 1*, *Standard Curve Adjustmen*t for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 0.25 mg/L nitrate nitrogen (NO_3^--N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of $\pm$0.03 mg/L nitrate nitrogen.

Estimated Detection Limit

The estimated detection limit for program 55 is 0.01 mg/L NO_3^--N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Calcium	100 mg/L
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	All levels
Nitrite	All levels: This method measures both the nitrate and nitrite in the sample. If nitrite is present, the nitrite nitrogen test Program 60 should be done on the sample. Pretreat the nitrate nitrogen sample with the following pretreatment. Then subtract the amount of nitrite found from the results of the LR nitrate nitrogen test using the pretreated sample. **1.** Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. Mix after each drop. **2.** Add one drop of 30-g/L Phenol Solution to destroy the yellow color. **3.** Proceed with the LR Nitrate procedure.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels

Summary of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to chromotropic acid to form a pink-colored product.

Pollution Prevention and Waste Management

NitaVer 6 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous wastes by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS

Low Range Nitrate Reagent Set (100 tests) 24298-00
Includes: (1) 21071-69, (1) 21072-49

Description	Quantity Required Per Test	Unit	Cat. No.
NitriVer 3 Nitrite Reagent Powder Pillows	1 pillow	100/pkg	21071-69
NitraVer 6 Nitrate Reagent Powder Pillows	1 pillow	100/pkg	21072-49

REQUIRED APPARATUS

Cylinder, graduated, mixing, 25 mL	1	each	1896-40
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Bromine Water, 30 g/L	29 mL*	2211-20
Nitrate Nitrogen Standard Solution, 10.0 mg/L as NO_3^--N	500 mL	307-49
Nitrate Nitrogen Standard Solution, Voluette ampule, 12 mg/L as NO_3^--N, 10 mL	16/pkg	14333-10
Phenol Solution, 30 g/L	29 mL	2112-20
Pretreatment Kit, contains: (1) 2112-20, (1) 2211-20	each	2268-00
Sodium Hydroxide Standard Solution, 5.0 N	50 mL* SCDB	2450-26
Sulfuric Acid, ACS	500 mL*	979-49
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker	each	21968-00
Dropper, for 29-mL bottle	each	2258-00
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH	5-roll/pkg	391-33
pH Meter, *sens**ion**™1*, portable, with electrode	each	51700-10
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet Filler, safety bulb	each	14651-00
Thermometer, –20 to 110 °C	each	26357-02

Nitrate at these levels can also be determined directly using the Nitrate Ion Selective Electrode (Cat. No. 23488-00).

For Technical Assistance, Price and Ordering

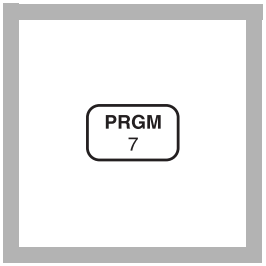
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes

NITRATE, High Range, Test 'N Tube (0 to 30.0 mg/L NO_3^--N)

Chromotropic Acid Method

For water and wastewater



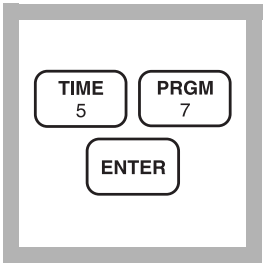
1. Enter the stored program number for Test 'N Tube nitrate nitrogen (NO_3^--N).

Press: **PRGM**

The display will show:

PRGM ?

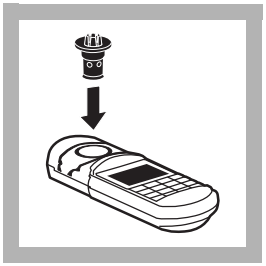
Note: *If samples cannot be analyzed immediately, see Sampling and Storage on page 331.*



2. Press: **57 ENTER**

The display will show **mg/L, NO3-N** and the **ZERO** icon**.**

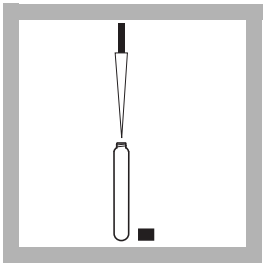
Note: *For alternate forms (NO_3) press the* **CONC** *key.*



3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

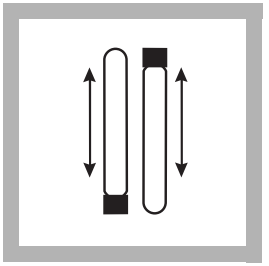
Note: *For proof of accuracy, use a 20 mg/L NO_3^--N standard in place of the sample.*

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



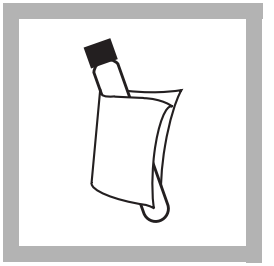
4. Remove the cap from a Nitrate Pretreatment Solution Vial and add 1 mL of sample (the blank).

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



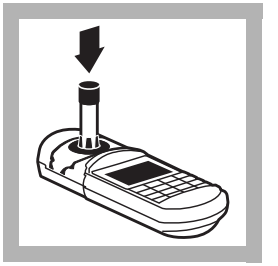
5. Cap the tube and invert 10 times to mix.

Note: *This test is technique-sensitive. Low results may occur if these instructions are not followed. Hold the vial vertical with the cap up. Invert the vial so the cap points down. Wait for all of the solution to flow to the cap end. Pause. Return the vial to the upright position. Wait for all the solution to flow to the vial bottom. This process equals 1 inversion. Do this 10 times.*



6. Clean the outside of the vial with a towel.

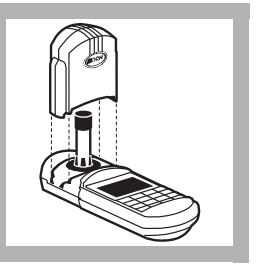
Note: *Wipe with a damp towel and follow with a dry one to remove fingerprints and other marks.*



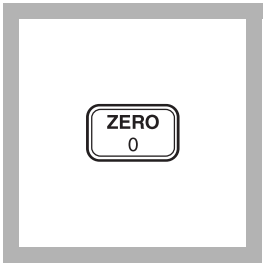
7. Place the blank in the vial adapter with the Hach logo facing the front of the instrument.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



8. Cover the vial tightly with the instrument cap.

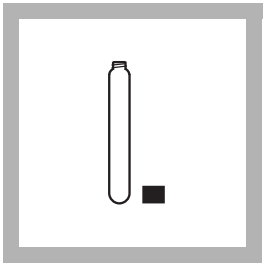


9. Press: **ZERO**

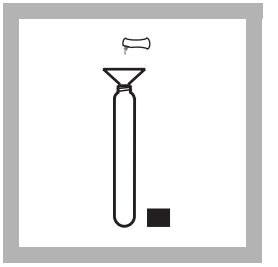
The cursor will move to the right, then the display will show:

0.0 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



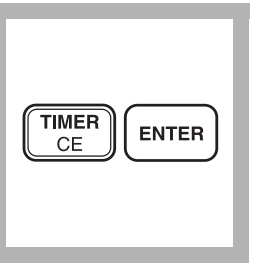
10. Remove the vial from the instrument. Remove the cap from the vial.



11. Using a funnel, add the contents of one NitraVer X Reagent B Powder Pillow to the vial. Cap. Invert 10 times to mix (this will be the prepared sample).

Note: *See Step 5 for inversion instructions*

Note: *Some solid matter will not dissolve.*



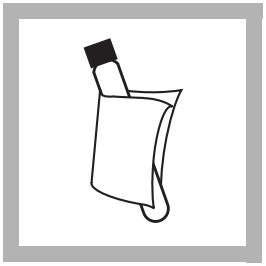
12. Press:

TIMER ENTER

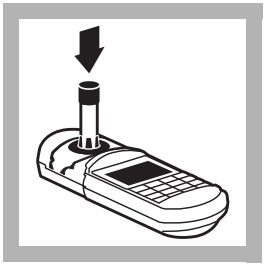
A five-minute reaction period will begin. Do not invert the vial again.

Note: *A yellow color will develop if nitrate nitrogen is present.*

Note: *Complete Steps 13-16 within five minutes after the timer beeps.*



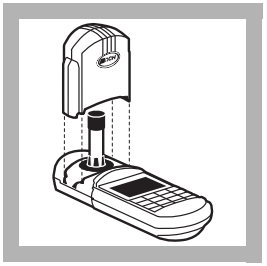
13. After the timer beeps, clean the outside of the vial with a damp towel and follow with a dry one to remove fingerprints and other marks.



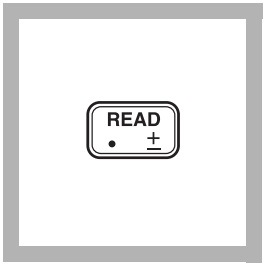
14. Place the prepared sample in the adapter with the Hach logo facing the front of the instrument.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



15. Cover the vial tightly with the instrument cap.



16. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrate nitrogen (NO_3-N) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods (up to 14 days), adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a fresh High Range Nitrate Nitrogen Voluette Ampule Standard, 500 mg/L NO_3^--N.

c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to the three mixing cylinders, respectively. Mix each thoroughly.

d) Analyze each sample as described in the procedure; use a 1-mL aliquot of the spiked sample in each test. The nitrogen concentration should increase 2.0 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* (*Section 1*) for more information.

Standard Solution Method

To test accuracy, prepare a 20.0 mg/L nitrate nitrogen standard solution by pipetting 2.00 mL of a High Range Nitrate Nitrogen Voluette Ampule Standard Solution, 500 mg/L NO_3^--N, into a 50 mL Class A volumetric flask. Dilute to the line with deionized water. Substitute this standard for the sample and perform the test as described in the procedure.

Method Performance

Precision

In a single laboratory, using a standard solution of 25.0 mg/L nitrate nitrogen (NO_3^--N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ± 0.5 mg/L NO_3-N.

Estimated Detection Limit

The estimated detection limit for program 57 is 0.3 mg/L NO_3-N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level
Barium	A negative interference at concentrations greater than 1 mg/L.
Chloride	Does not interfere below 1000 mg/L.
Hardness	Does not interfere.
Nitrite	A positive interference at concentrations greater than 12 mg/L. Remove nitrite interference up to 100 mg/L by adding 400 mg of urea (one full 0.5 g Hach measuring spoon) to 10 mL of sample. Swirl to dissolve. Proceed with the nitrate test as usual.

Summary of Method

Nitrate in the sample reacts with chromotropic acid under strongly acidic conditions to yield a yellow product with a maximum absorbance at 410 nm.

REQUIRED REAGENTS

	Cat. No.
NitraVer X Nitrate, High Range Test 'N Tube Reagent Set (50 tests)	26053-45
Includes: (1) 26055-46, (1) 272-42, *(50) Nitrate Pretreatment Solution Vials	

Description	Quantity Required Per Test	Unit	Cat. No.
Nitrate Pretreatment Solution Vials	1	50/pkg	*
NitraVer X Reagent B Powder Pillows	1	50/pkg	26055-46

REQUIRED APPARATUS

COD Vial Adapter	1	each	48464-00
Funnel, micro	1	each	25843-35
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	varies	50/pkg	21856-96
Test Tube Rack	1-3	each	18641-00

OPTIONAL REAGENTS

Nitrate-Nitrogen Standard Solution, Voluette Ampules, 500 mg/L N	16/pkg	14260-10
Sodium Hydroxide Standard Solution, 5.0 N	50 mL	2450-26
Sulfuric Acid, ACS, concentrated	500 mL	979-49
Urea, ACS	100 g	11237-26
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Cylinder, graduated, mixing, 25-mL (3 required)	each	26363-40
Flask, volumetric, Class A, 50 mL	each	14574-41
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
Pipet, volumetric, Class A, 2 mL	each	14515-36
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Spoon, measuring, 0.5 g	each	907-00

For Technical Assistance, Price and Ordering

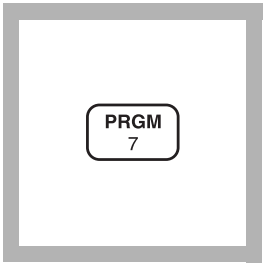
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Not available separately.

NITRITE, High Range (0 to 150 mg/L NO_2^-)

Ferrous Sulfate Method*



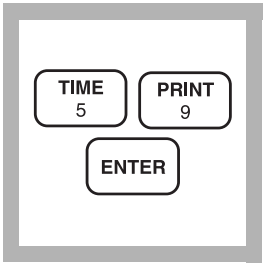
1. Enter the stored program number for high range nitrite (NO_2^-).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

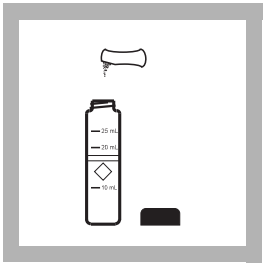


2. Press: **59 ENTER**

The display will show **mg/L, NO2** and the **ZERO** icon**.**

Note: *For alternate forms (NO_2-N, $NaNO_2$), press the* **CONC** *key.*

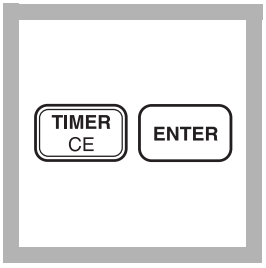
3. Fill a sample cell with 10 mL of sample.



4. Add the contents of one NitriVer 2 Nitrite Reagent Powder Pillow. Cap the cell and invert 5-7 times to mix (the prepared sample).

Note: *A greenish-brown color will develop if nitrite is present.*

Note: *Avoid excessive mixing or low results may occur. Accuracy is not affected by undissolved powder.*

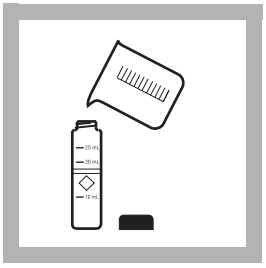


5. Press:

TIMER ENTER

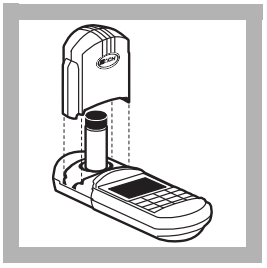
A ten-minute reaction period will begin.

Do not move or disturb the sample cell during this reaction period.

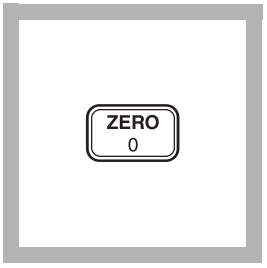


6. Fill another sample cell with 10 mL of sample (the blank). Clean the outside of the cells with a towel.

Note: *Wiping with a damp towel, followed by a dry one, removes fingerprints and other marks.*



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



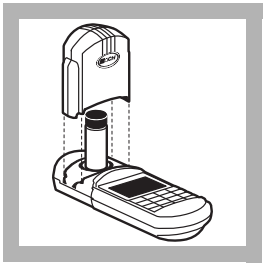
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L NO2

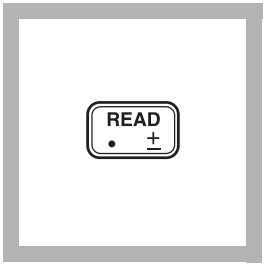
Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

* Adapted from McAlpine, R. and Soule, B., *Qualitative Chemical Analysis*, New York, 476,575 (1933)



9. After the timer beeps, gently invert the prepared sample twice. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Avoid excessive mixing or low results may occur.*



10. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrite will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. If prompt analysis is impossible, store at 4 °C (39 °F) or lower if the sample is to be analyzed within 48 hours. Warm to room temperature before running the test. Do not use acid preservatives. Remove suspended solids by filtration.

Accuracy Check

Standard Solution Method

Dissolve 0.150 grams of fresh sodium nitrite and dilute to 1000 mL with deionized water to prepare a 100 mg/L nitrite standard solution. Prepare this solution daily.

Alternatively, make a dilution of a fresh Hach Nitrite Standard Solution, 821 mg/L NO_2^- (250 mg/L NO_2^--N) using Class A glassware. Dilute 10 mL of this standard to 100 mL with deionized water to give an
82 mg/L nitrite standard. Prepare this solution just before use. Using this solution as the sample, perform the nitrite procedure as described above.

Method Performance

Precision

In a single laboratory using a standard solution of 123 mg/L nitrite and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of ±1 mg/L nitrite.

Estimated Detection Limit

The estimated detection limit for program 59 is 2 mg/L NO_2^-. For more information on the estimated detection limit, see *Section 1*.

Interferences

This test does not measure nitrates nor is it applicable to glycol based samples. Dilute glycol based samples and follow the Low Range Nitrite Procedure.

Summary of Method

The method uses ferrous sulfate in an acidic medium to reduce nitrite to nitrous oxide. Ferrous ions combine with the nitrous oxide to form a greenish-brown complex in direct proportion to the nitrite present.

REQUIRED REAGENTS AND APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
NitriVer 2 Nitrite Reagent Powder Pillows	1 pillow	100/pkg	21075-69
Sample cell, 10-20-25, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Nitrite Standard Solution, 821 mg/L NO_2 (250 mg/L NO_2^--N)		500 mL	23402-49
Sodium Nitrite, ACS		454 g	2452-01
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Balance, analytical, 110 V, Acculab UI Series, 120 g		each	26947-00
Flask, volumetric, 1000 mL		each	14547-53
Flask, volumetric, 100 mL, Class A		each	14574-42
Pipet, volumetric, 10.00 mL, Class A		each	14515-38
Pipet Filler, safety bulb		each	14651-00

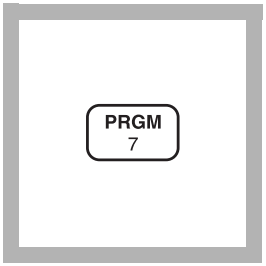
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITRITE, Low Range (0 to 0.350 mg/L NO_2^--N) For water, wastewater, seawater

Diazotization Method* (Powder Pillows or AccuVac Ampuls);
USEPA approved for reporting wastewater and drinking water analyses.



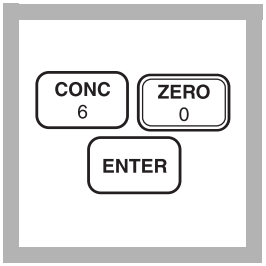
1. Enter the stored program number for nitrite nitrogen (NO_2^--N), powder pillows.

Press: **PRGM**

The display will show:

PRGM ?

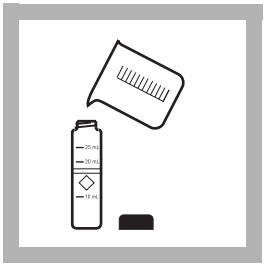
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



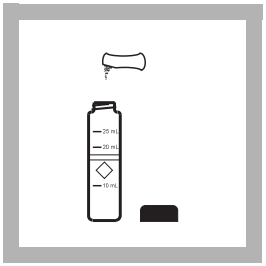
2. Press: **60 ENTER**

The display will show **mg/L, NO2-N** and the **ZERO** icon.

Note: *For alternate forms (NO_2^-, $NaNO_2$), press the* **CONC** *key.*



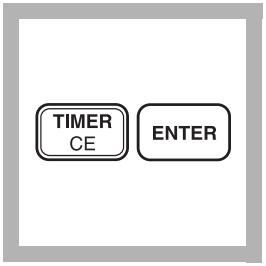
3. Fill a sample cell with 10 mL of sample.



4. Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow to the sample cell. Cap the cell and shake to dissolve.

Note: *Accuracy is not affected by undissolved powder.*

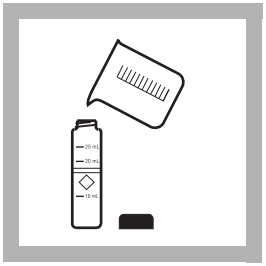
* *Federal Register*, 44(85) 25505 (May 1, 1979)



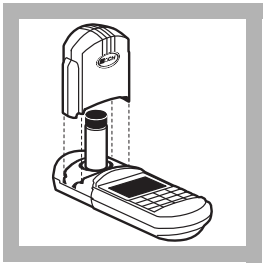
5. Press:
TIMER ENTER

A 15-minute reaction period will begin.

Note: *A pink color will develop if nitrite is present.*

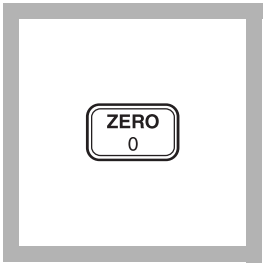


6. When the timer beeps, fill an empty sample cell with 10 mL of sample (the blank).



7. Wipe the outside of the sample cell with a towel. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Wiping with a damp cloth, followed by a dry pne, removes fingerprints and other marks.*

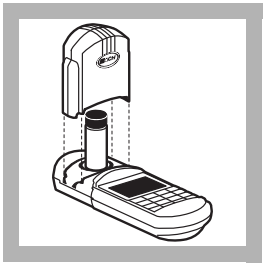


8. Press: **ZERO**

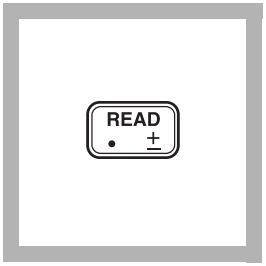
The cursor will move to the right, then the display will show:

0.000 mg/L NO2-N

Note: *If Reagent Blank Correction is on, the display may flash "limit." See Section 1.*



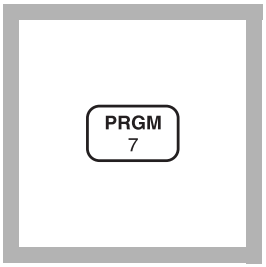
9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrite nitrogen (or an alternate form) will be displayed.

Using AccuVac Ampuls



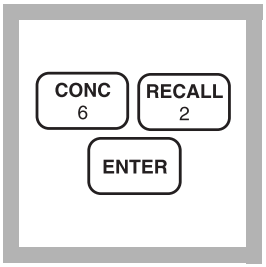
1. Enter the stored program number for nitrite nitrogen (NO_2^--N), AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

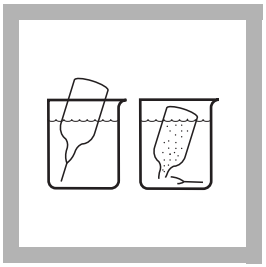
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **62 ENTER**

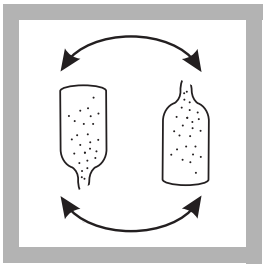
The display will show **mg/L, NO2-N** and the **ZERO** icon**.**

Note: *For alternate forms (NO_2^-, $NaNO_2$), press the* **CONC** *key.*



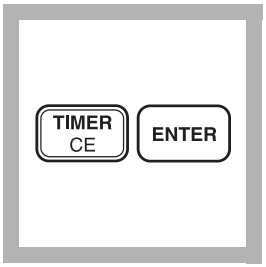
3. Collect at least 40 mL of sample in a 50-mL beaker. Fill a NitriVer 3 Nitrite AccuVac Ampul with the sample.

Note: *Keep the tip immersed while the ampul fills completely.*



4. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

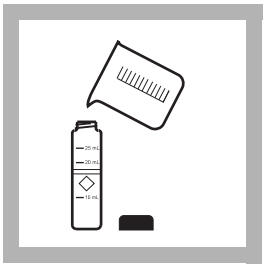
Note: *Accuracy is not affected by undissolved powder.*



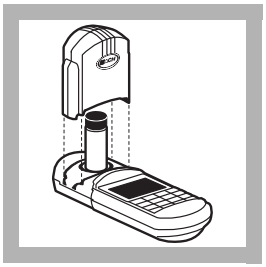
5. Press:
TIMER ENTER

A 15-minute reaction period will begin.

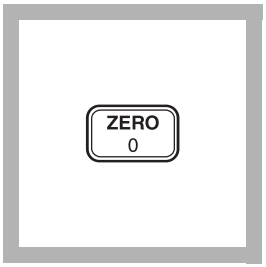
Note: *A pink color will develop if nitrite is present.*



6. When the timer beeps, fill a sample cell with at least 10 mL of sample (the blank).



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

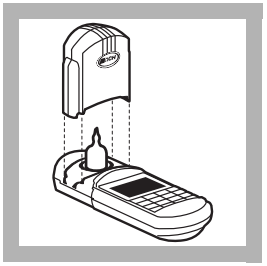


8. Press: **ZERO**

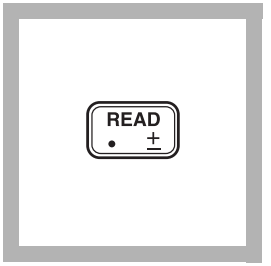
The cursor will move to the right, then the display will show:

0.000 mg/L NO2-N

Note: *If Reagent Blank Correction is on, the display may flash "limit." See Section 1.*



9. Place the AccuVac Ampul into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrite nitrogen will be displayed.

Sampling and Storage

Collect samples in clean plastic or glass bottles.

Store at 4 °C (39 °F) or lower and analyze within 48 hours. Warm to room temperature before running the test.

Do not use acid preservatives.

Remove the suspended solids by filtration.

Accuracy Check

Standard Solution Method

Pipet 5.00 mL of a fresh 250 mg/L NO_2^--N standard into a 250.0 mL volumetric flask. Dilute to the mark with deionized water. This makes a 5.00-mg/L intermediate standard. To prepare a 0.100-mg/L NO_2^--N standard solution, dilute 10.00 mL of the 5.00-mg/L intermediate standard to 500 mL in a volumetric flask. Prepare this solution immediately before use.

Run the test using the 0.100 mg/L NO_2^--N standard in place of the sample. Results should be between 0.090 and 0.110 mg/L NO_2^--N.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.250 mg/L nitrite nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.001 mg/L NO_2^--N for the powder pillow method and ±0.003 mg/L NO_2^--N for the
AccuVac method.

Estimated Detection Limit

The estimated detection limit for programs 60 and 62 is 0.005 mg/L NO_2^--N. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels
Antiminous ions	Interfere by causing precipitation
Auric ions	Interfere by causing precipitation
Bismuth ions	Interfere by causing precipitation
Chloroplatinate ions	Interfere by causing precipitation
Cupric ions	Cause low results
Ferric ions	Interfere by causing precipitation
Ferrous ions	Cause low results
Lead ions	Interfere by causing precipitation
Mercurous ions	Interfere by causing precipitation
Metavanadate ions	Interfere by causing precipitation
Nitrate	Very high levels of nitrate (>100 mg/L nitrate as N) appear to undergo a slight amount of reduction to nitrite, either spontaneously or during the course of the test. A small amount of nitrite will be found at these levels.
Silver ions	Interfere by causing precipitation
Strong oxidizing and reducing substances	Interfere at all levels

Summary of Method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink colored complex directly proportional to the amount of nitrite present.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
NitriVer 3 Nitrite Reagent Powder Pillows	1 pillow	100/pkg	21071-69
or			
NitriVer 3 Nitrite Reagent AccuVac Ampuls	1 ampul	25/pkg	25120-25

REQUIRED APPARATUS

Beaker, 50 mL (for AccuVac procedure)	1	each	500-41H
or			
Sample Cells, 10-20-25 mL (powder pillow procedure)	2	6/pkg	24019-06

OPTIONAL REAGENTS

Nitrite Standard Solution, 250 mg/L as NO_2^--N		500 mL	23402-49
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit	each	24052-00
Flask, volumetric, 250 mL	each	14574-46
Flask, volumetric, 500 mL	each	14574-49
Pipet, serological, 10 mL	each	532-38
Pipet, TenSette, 1 to 10 mL	each	19700-01
Pipet Tips for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 10.00 mL	each	14515-38
Pipet Filler, safety bulb	each	14651-00
Thermometer, –20 to 110 °C	each	26357-02

For Technical Assistance, Price and Ordering

In the U.S.A. call 800-227-4224

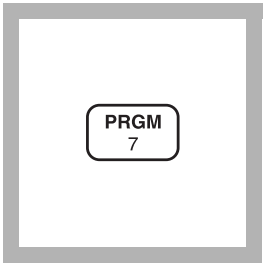
Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITRITE, Low Range, Test 'N Tube (0–0.500 mg/L NO_2^--N)

Diazotization Method

For water, wastewater, and seawater

USEPA approved for wastewater analysis*



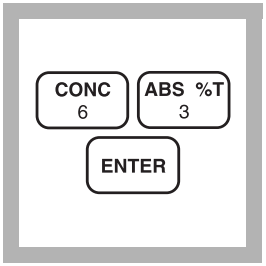
1. Enter the stored program number for nitrite nitrogen (NO_2^--N), Test 'N Tube.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



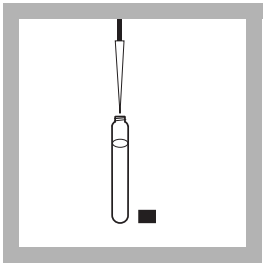
2. Press: **63 ENTER**

The display will show **mg/L, NO2-N** and the **ZERO** icon.

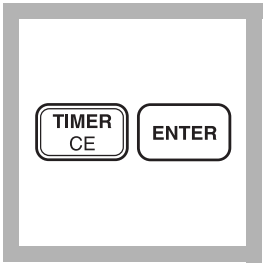
Note: *For alternate forms (NO_2^-, $NaNO_2$), press the* **CONC** *key.*

3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert.

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



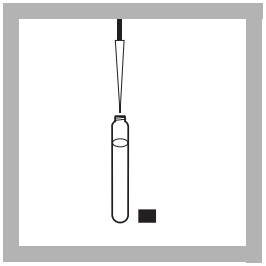
4. Fill a Test 'N Tube NitriVer® 3 Nitrite vial with 5 mL of sample. Cap and shake to dissolve powder. This is the prepared sample.



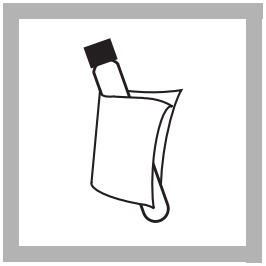
5. Press:
TIMER ENTER

A 20-minute reaction period will begin.

Note: *A pink color will develop if nitrite is present.*

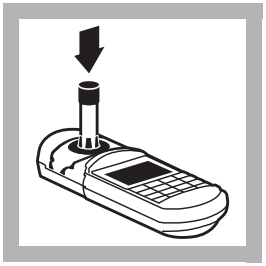


6. When the timer beeps, fill an empty Test 'N Tube vial with 5 mL of sample (the blank).



7. Clean the outside of the vials with a towel.

Note: *Wipe with a damp towel and follow with a dry one to remove fingerprints and other marks.*

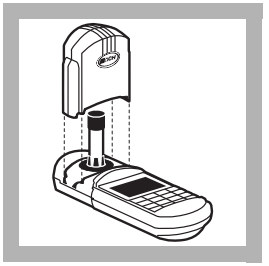


8. Place the blank in the vial adapter.

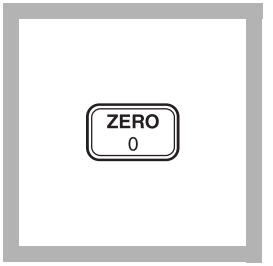
Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*

* *Federal Register,* 44(85) 25505 (May 1, 1979).



9. Cover the sample cell tightly with the instrument cap.

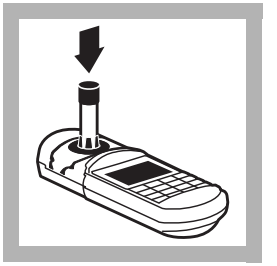


10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.000 mg/L NO2-N

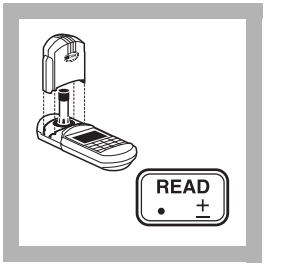
Note: *If the reagent blank correction is on, the display may flash "limit." See Section 1.*



11. Place the prepared sample in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Tightly cover the sample cell with the instrument cap.

Press: **READ**

The cursor will move to the right, then the result in mg/L nitrite nitrogen (or an alternate form) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles.

Store at 4 °C (39 °F) or lower and analyze within 48 hours. Warm to room temperature before running the test.

Do not use acid preservatives.

Remove suspended solids by filtration.

Accuracy Check

Standard Solution Method

Pipet 5.00 mL of a fresh Hach standard, 250 mg/L as NO_2^--N into a Class A 250-mL volumetric flask. Dilute to the line with deionized water to make a 5.00-mg/L intermediate standard. Pipet 10.00 mL of the 5.0-mg/L intermediate standard into a Class A 500-mL volumetric flask. Dilute to the line with deionized water to make a 0.100 mg/L NO_2^--N standard solution. Prepare immediately before use.

Run the test using the 0.100 mg/L NO_2^--N standard in place of the sample. Results should be between 0.090 and 0.110 mg/L NO_2^--N.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.250 mg/L nitrite nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.004 mg/L NO_2^--N.

Estimated Detection Limit

The estimated detection limit for program 63 is 0.006 mg/L NO_2^--N. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels
Antiminous ions	Interfere by causing precipitation
Auric ions	Interfere by causing precipitation
Bismuth ions	Interfere by causing precipitation
Chloroplatinate ions	Interfere by causing precipitation
Cupric ions	Cause low results
Ferric ions	Interfere by causing precipitation
Ferrous ions	Cause low results
Lead ions	Interfere by causing precipitation
Mercurous ions	Interfere by causing precipitation
Metavanadate ions	Interfere by causing precipitation
Nitrate	Very high levels of nitrate (>100 mg/L nitrate as N) appear to undergo a slight amount of reduction to nitrite, either spontaneously or during the course of the test. A small amount of nitrite will be found at these levels.
Silver ions	Interfere by causing precipitation
Strong oxidizing and reducing substances	Interfere at all levels

Summary of Method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink-colored complex directly proportional to the amount of nitrite present.

REQUIRED REAGENTS

Description	Cat. No.
NitriVer® 3 Nitrite, Low Range Test 'N Tube Reagent Set (50 tests)	26083-45
Includes:	
(50) NitriVer® 3 Nitrite Test 'N Tube Vials	*
Vials, 6 x 100 mm, 6/pkg	22758-06
Caps, for 22758-06 vials, 6/pkg	22411-06
Deionized water, 100-mL	272-42

REQUIRED APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
COD/TNT Adapter	1	each	48464-00
Test Tube Rack	1-3	each	18641-00
Pipet, TenSette, 1 to 10 mL	1	each	19700-10
Pipet Tips for 19700-10 TenSette Pipet	1	50/pkg	21997-96

OPTIONAL REAGENTS

Nitrite Standard Solution, 250 mg/L as NO_2^--N	500	mL	23402-49
Water, deionized	4	L	272-56

OPTIONAL APPARATUS

Flask, volumetric, 250 mL		each	14574-46
Flask, volumetric, 500 mL		each	14574-49
Pipet, volumetric, Class A, 10.00 mL		each	14515-38

For Technical Assistance, Price and Ordering

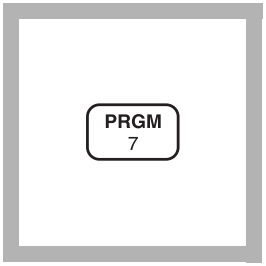
In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Not available separately.

NITROGEN, AMMONIA (0 to 0.50 mg/L NH_3-N) For water, wastewater, seawater

Salicylate Method*

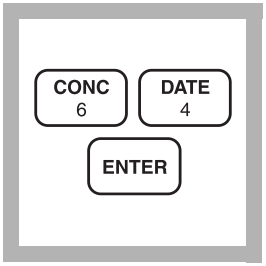


1. Enter the stored program number for ammonia nitrogen (NH_3-N).

Press: **PRGM**

The display will show:

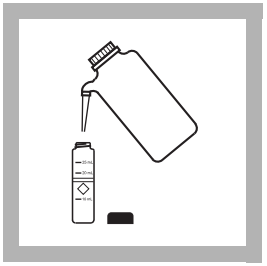
PRGM ?



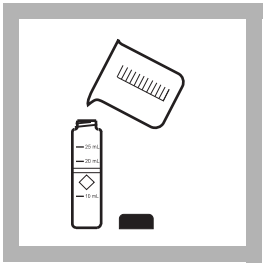
2. Press: 64 ENTER

The display will show **mg/L, NH3-N** and the **ZERO** icon**.**

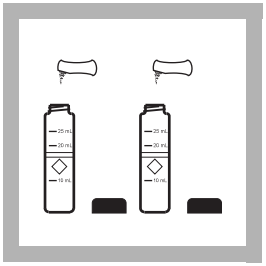
Note: *For alternate forms (NH_3, NH_4), press the* **CONC** *key.*



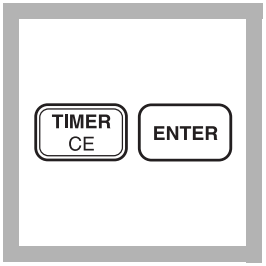
3. Fill a sample cell with 10 mL of deionized water (the blank).



4. Fill a second sample cell with 10 mL of the sample.



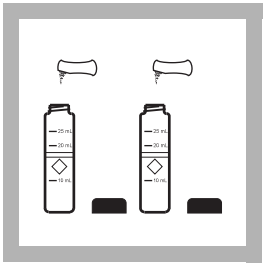
5. Add the contents of one Ammonia Salicylate Reagent Powder Pillow to each sample cell. Cap both cells and shake to dissolve.



6. Press:

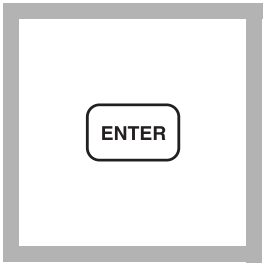
TIMER ENTER

A three-minute reaction period will begin.



7. After the timer beeps add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each sample cell. Cap the cells and shake to dissolve the reagent.

Note: *A green color will develop if ammonia nitrogen is present.*

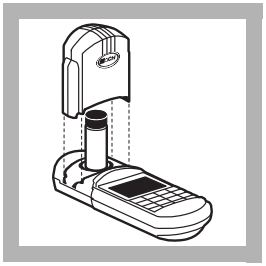


8. The display will show: **15:00 TIMER 2**

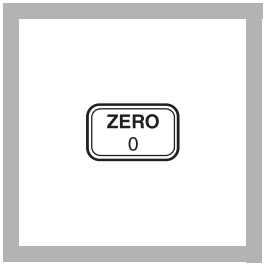
Press: **ENTER**

A 15-minute reaction period will begin.

* Adapted from Clin. Chim. Acta., 14 403 (1966)



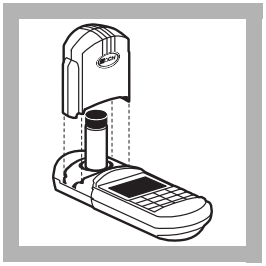
9. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



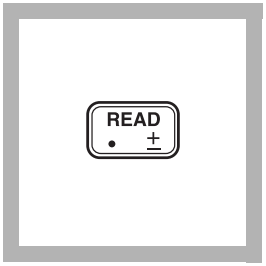
10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L NH3-N



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L ammonia nitrogen will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

If chlorine is known to be present, the sample must be treated immediately with sodium thiosulfate. Add one drop of Sodium Thiosulfate Standard Solution, 0.1 N, for each 0.3 mg of chlorine present in a one liter sample.

To preserve the sample, adjust the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter). Store samples at 4 °C or less. Samples preserved in this manner can be stored up to 28 days. Just before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution. Correct the test result for volume additions; see *Correction for Volume Additions*, in *Section 1* for more detailed information.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL mixing cylinders with 20 mL of sample.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of Ammonium Nitrogen Standard, 10 mg/L as NH_3-N to the three samples. Stopper the cylinders and mix well.

c) Analyze a 10-mL portion of sample as described above. The ammonia nitrogen concentration should increase 0.05 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

Standard Solution Method

Prepare a 0.40 mg/L ammonia nitrogen standard by diluting 4.00 mL of the Ammonia Nitrogen Standard Solution, 10 mg/L, to 100 mL with deionized water. Or, using the TenSette Pipet, prepare a 0.40 mg/L ammonia nitrogen standard by diluting 0.8 mL of a Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as NH_3-N, to 100 mL with
deionized water.

Method Performance

Precision

In a single laboratory using a standard solution of 0.40 mg/L ammonia nitrogen (NH_3-N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of $\pm$0.02 mg/L ammonia nitrogen.

Estimated Detection Limit

The estimated detection limit for program 64 is 0.02 mg/L NH_3-N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatments.

Interfering Substance	Interference Level and Treatments
Calcium	Greater than 1000 mg/L as $CaCO_3$
Glycine, hydrazine	Less common. Will cause intensified colors in the prepared sample.
Iron	All levels. Correct for iron interference as follows: **1.** Determine the amount of iron present in the sample using one of the Total Iron procedures. **2.** Prepare a deionized water sample containing the same iron concentration as the original sample. Run the procedure on this solution to determine the interference due to iron. Subtract this value from the result in Step 12 obtained on the original sample.
Magnesium	Greater than 6000 mg/L as $CaCO_3$
Nitrate	Greater than 100 mg/L as NO_3^--N
Nitrite	Greater than 12 mg/L as NO_2^--N
Phosphate	Greater than 100 mg/L as PO_4^{3-}-P
Sulfate	Greater than 300 mg/L as SO_4^{2-}
Sulfide	Sulfide will intensify the color. Eliminate sulfide interference as follows: **1.** Measure about 350 mL of sample in a 500-mL erlenmeyer flask. **2.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix. **3.** Filter the sample through a folded filter paper. **4.** Use the filtered solution in Step 3.
Turbidity, sample color	Turbidity and sample color will give erroneous high values. Samples with severe interferences require distillation. Albuminoid nitrogen samples also require distillation. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set. See the Optional Apparatus list.

Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

REQUIRED REAGENTS AND APPARATUS

			Cat. No.
Ammonia Nitrogen Reagent Set for 10-mL samples (100 tests)			26680-00
Includes: (2) 26531-99, (2) 26532-99			
Description	**Quantity Required Per Test**	**Unit**	**Cat. No.**
Ammonia Cyanurate Reagent Powder Pillows	2 pillows	100/pkg	26531-99
Ammonia Salicylate Reagent Powder Pillows	2 pillows	100/pkg	26532-99
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Ammonia Nitrogen Standard Solution, 10 mg/L as NH_3-N	500 mL	153-49
Ammonia Nitrogen, PourRite Ampules, 50 mg/L as NH_3-N, 2 mL	20/pkg	14791-20
Cylinder, graduated, mixing, 25 mL	each	20886-40
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045-32
Sodium Hydroxide Standard Solution, 5.0 N	50 mL SCDB	2450-26
Sodium Thiosulfate Standard Solution, 0.1 N	100 mL MDB	323-32
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	2418-99
Sulfuric Acid, concentrated, ACS	500 mL	979-49
Sulfuric Acid Standard Solution, 1.0 N	100 mL MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Cylinder, graduated, polypropylene, 500 mL	each	1081-49
Distillation Heater and Support Apparatus, 115 V	each	22744-00
Distillation Heater and Support Apparatus, 230 V	each	22744-02
Distillation Set, General Purpose	each	22653-00
Filter Paper, folded, 12.5 cm	100	1894-57
Flask, Erlenmeyer, polypropylene, 500 mL	each	1082-49
Flask, volumetric, Class A, 100 mL	each	14574-42
Funnel, poly, 65 mm	each	1083-67
pH Meter, *sens**ion**™1*, portable, with electrode	each	51700-10
Pipet Filler, safety bulb	each	14651-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 2.0 mL	each	14515-36
PourRite Ampule Breaker Kit	each	24846-00
Thermometer, –20 to 110 °C	each	26357-02

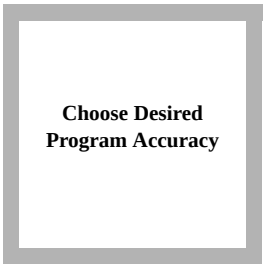
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

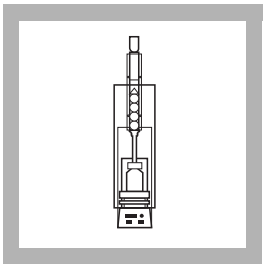
Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITROGEN, TOTAL KJELDAHL (0 to 150 mg/L)

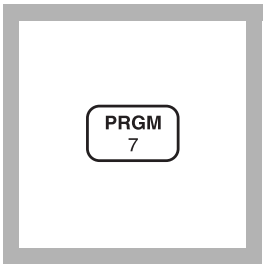
Nessler Method* (digestion required) **For water, wastewater and sludge**



1. A User-Entered Calibration is necessary to obtain the most accurate results. See the User Calibration section following these steps.



2. Digest the sample as described in the Digesdahl Apparatus Instruction manual. Digest an equal amount of deionized water as the blank.

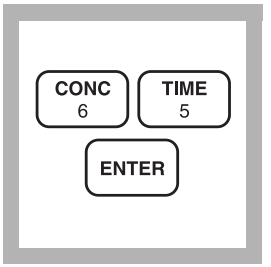


3. Enter the stored program number for total Kjeldahl nitrogen.

Press: **PRGM**

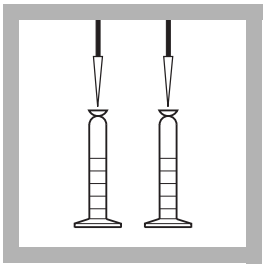
The display will show:

PRGM ?

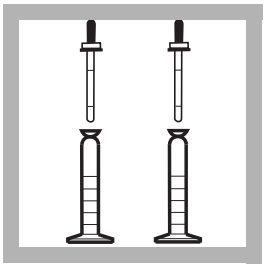


4. Press: **65 ENTER**

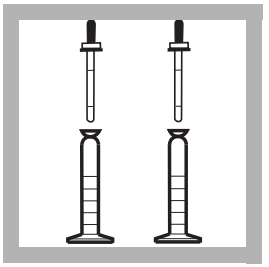
The display will show **mg/L, TKN** and the **ZERO** icon**.**



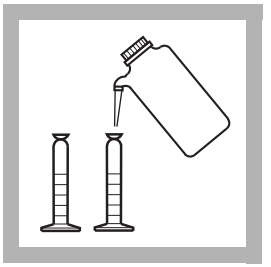
5. Select the appropriate analysis volume of the digested sample given in *Table 1* on page *357*. Pipet the analysis volume from the sample and the digested blank into separate 25-mL mixing graduated cylinders.



6. Add one drop of TKN Indicator to each cylinder. Add 8.0 N KOH dropwise to each cylinder, mixing after each addition. Continue until the first apparent blue color is visible.



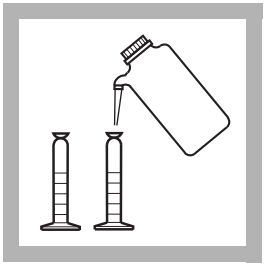
7. Add 1.0 N KOH to each cylinder, one drop at a time, mixing after each addition. Continue until the first permanent blue color appears.



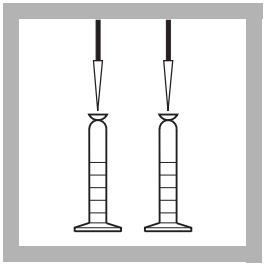
8. Fill both mixing cylinders to the 20-mL mark with deionized water. Add 3 drops of Mineral Stabilizer to each cylinder. Invert several times to mix. Add 3 drops of Polyvinyl Alcohol Dispersing Agent to each cylinder. Invert several times to mix.

Note: *Hold the dropping bottles upright while dispensing.*

* Adapted from: Hach et al., *Journal of Association of Official Analytical Chemists,* 70 (5) 783-787 (1987); Hach et al., *Journal of Agricultural and Food Chemistry,* 33 (6) 1117-1123 (1985); *Standard Methods for the Examination of Water and Wastewater.*

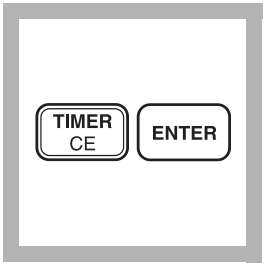


9. Fill both cylinders to the 25-mL mark with deionized water.



10. Pipet 1 mL of Nesslers Reagent to each cylinder. Stopper, invert repeatedly. The solution should not be hazy.

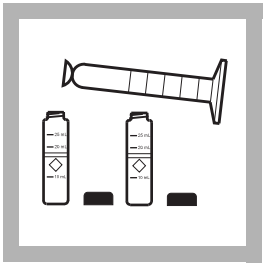
Note: *Any haze (turbidity) will cause incorrect results.*



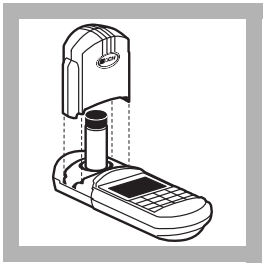
11. Press:

TIMER ENTER

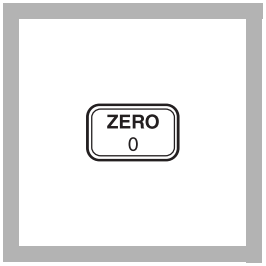
A two-minute reaction period will begin.



12. When the timer beeps, pour the contents of each cylinder into a separate labeled sample cell.



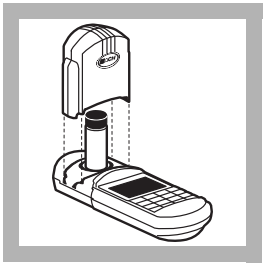
13. Place the blank into a cell holder. Tightly cover the sample cell with the instrument cap.



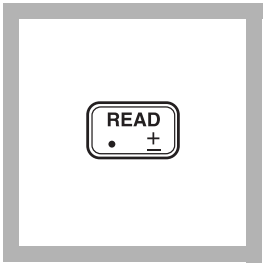
14. Press: **ZERO**

The cursor will move to the right, then the display will show:

0. mg/L TKN



15. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



16. Press: **READ**

The cursor will move to the right, then the result in mg/L total Kjeldahl nitrogen will be displayed.

Note: *Standard Adjust may be performed using a prepared ammonia standard (see Standard Adjust in Section 1).*

$$\text{ppm TKN} = \frac{75 \times A}{B \times C}$$

17. Use the formula shown to calculate the final TKN value.

Where:

A = mg/L displayed

B = g (or mL of water) sample taken for digest

C = mL analysis volume of digested sample (step 5).

Note: *For water samples ppm TKN = mg/L TKN.*

Note: *For maximum accuracy, the reagent blank value may be determined by repeating procedure using reagents only. Subtract the reagent blank value from the reading on the display.*

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Table 1 Analysis Volumes Based on Concentration

AQUEOUS SAMPLES (Solutions of suspensions in water- less than 1% solids)	
Expected Nitrogen Concentration (mg/L)	**Analysis Volume (mL)**
0.5-28	10.00
2-112	5.00
11-560	2.00
45-2250	1.00
425-22500	0.50
DRY SAMPLES	
Expected Nitrogen Concentration (mg/L)	**Analysis Volume (mL)**
42-2200	10.0
106-5600	5.00
350-18000	2.00
1000-56000	1.00
4200-220000	0.50
OILS AND FATS	
Expected Nitrogen Concentration (mg/L)	**Analysis Volume (mL)**
85-4500	10.0
210-11000	5.00
2100-11000	1.00

Sampling and Storage

Collect samples in a cleaned glass or plastic container. Adjust the pH to 2 or less with sulfuric acid (about 2 mL per liter) and cool to 4 °C. Preserved samples can be stored up to 28 days.

Accuracy Check

Kjeldahl Nitrogen Standard Method

This procedure checks digestion efficiency and indicates that amount of bound nitrogen that is freed during digestion. The methods and standards available to check digestion technique are found in the Accuracy Check section following the procedures in the Digesdahl Digestion Apparatus Instruction Manual. Using the digested Kjeldahl standard, perform the above TKN analysis on the colorimeter. The TKN value should come within about ±3% of the value of the prepared Kjeldahl standard.

Standard Solution Method (to check calibration accuracy only)

Add one drop of TKN Indicator to each of two 25-mL graduated mixing cylinders. Fill one cylinder to the 20-mL mark with deionized water. Fill the other cylinder to the 20-mL mark with a 1.0 mg/L Ammonia Nitrogen Solution. Add 3 drops of Mineral Stabilizer to each cylinder. Invert several times to mix. Add 3 drops of Polyvinyl Alcohol Dispersing agent to each cylinder. Perform the TKN procedure as described in Steps 9 to 16. This display should show 26-27 mg/L TKN.

User Calibration

For most accurate results, use a user-calibrated program. The Standard Adjust feature should not be used with a user-entered calibration; it will hinder performance.

A one-time setup of a program for TKN is recommended for each new lot of reagents. A new calibration may be performed for each lot of Nessler Reagent by following these instructions:

Standard Preparation

Use the following standards to make a calibration curve. See *Preparing a User-Entered Calibration Curve* on page *49*, for more information and instructions. Prepare standards representing concentrations of 20, 60, 80, 100, 140 and 160 mg/L NH_3-N as follows:

a) Using volumetric pipets, transfer 5.0, 15.0, 20.0, 25.0, 35.0, and 40.0 mL of 100 mg/L NH_3-N standard solution into six separate 100-mL volumetric flasks. Dilute to volume with deionized water, stopper, and invert to mix.

b) Begin at step 4 of the procedure using a 3-mL aliquot for the sample volume. Also prepare a blank solution by substituting a 3 mL aliquot of deionized water for sample in Step 4.

Note: *Standard solutions are prepared as if a 25-mL volume was used for the digestion. Actual concentrations prepared in Step 1 are 5, 15, 20, 25, 35, and*
40 mg/L NH_3-N. These represent original concentrations of 20, 60, 80, 100, 140, and 160 mg/L NH_3-N, based on the 25 to 100 mL dilution in the digestion.

User Entered Calibration Settings For TKN
Program # = 101 to 105
Wavelength = 420 nm
Resolution = 0 mg/L

Method Performance

Precision

In a single laboratory using a standard solution of 64 mg/L TKN and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±1.0 mg/L TKN.

Estimated Detection Limit

The estimated detection limit for program 65 is 2 mg/L TKN. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

"Total Kjeldahl Nitrogen" (also called crude protein) refers to the combination of ammonia and organic nitrogen. Organically-bound in the trinegative state, it is converted into ammonium salts by the action of sulfuric acid and hydrogen peroxide. The ammonia is then analyzed by a modified nessler method test. The Mineral Stabilizer complexes calcium and magnesium. The Polyvinyl Alcohol Dispersing Agent aids the color formation in the reaction of Nessler Reagent with ammonium ions. A yellow color forms, proportional to the ammonia concentration.

Pollution Prevention And Waste Management

Nessler reagent contains mercuric iodide. Both the sample and blank will contain mercury (D009) at concentrations regulated as a hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. See Section *3* for more information on proper disposal of these materials.

REQUIRED REAGENTS

Total Kjeldahl Nitrogen Reagent Set 24953-00
Includes: (1) 21196-49, (1) 23766-26, (1) 21194-49, (1) 23765-26, (1) 282-32H, (1) 23144-26, (1) 979-49, (1) 22519-26

Description	Quantity Required Per Test	Unit	Cat. No.
Hydrogen Peroxide, 50%	20 mL	490 mL	21196-49
Mineral Stabilizer	6 drops	50 mL SCDB	23766-26
Nesslers Reagent	2 mL	500 mL	21194-49
Polyvinyl Alcohol Dispersing Agent	6 drops	50 mL SCDB	23765-26
Potassium Hydroxide Standard Solution, 8.0 N	varies	100 mL MDB	282-32H
Potassium Hydroxide Standard Solution, 1.0 N	varies	50 mL SCDB	23144-26
Sulfuric Acid, ACS	6 mL	500 mL	979-49
TKN Indicator Solution	2 drops	50 mL SCDB	22519-26
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Boiling Chips, silicon carbide	2-3	500 g	20557-34
Cylinder, graduated, mixing, tall-form, 25 mL	2	each	20886-40
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	2	50/pkg	21856-96
Safety Shield, for Digesdahl	1	each	50040-00
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

Select one based on available voltage:

Digesdahl Digestion Apparatus, 115 V	1	each	23130-20
Digesdahl Digestion Apparatus, 230 V	1	each	23130-21

OPTIONAL REAGENTS

Ammonia Nitrogen Standard Solution, 1 mg/L NH_3-N		500 mL	1891-49
Ammonia Nitrogen Standard Solution, Voluette Ampule, 150 mg/L NH_3-N, 10 mL		16/pkg	21284-10
Ammonia Nitrogen Standard Solution, 100 mg/L NH_3-N		500 mL	24065-49
Nitrogen Standard, Primary		3/set	22778-00

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Ampule Breaker Kit	each	21968-00
Balance, AccuLab Pocket Pro 250B	each	27969-00
Bottle, glass dispenser, 118 mL	each	591-00
Bottle, plastic wash, 1000 mL	each	620-16
Cylinder, graduated, 50 mL	each	508-41
Flask, volumetric, 100 mL, Class A	each	14574-42
Mini Grinder, 120 V	each	20991-00
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 0.50 mL	each	14515-34
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 10.00 mL	each	14515-38
Pipet, volumetric, Class A, 15.00 mL	each	14515-39
Pipet, volumetric, Class A, 20.00 mL	each	14515-20
Pipet, volumetric, Class A, 25.00 mL	each	14515-40
Safety Glasses	each	18421-00

For Technical Assistance, Price and Ordering

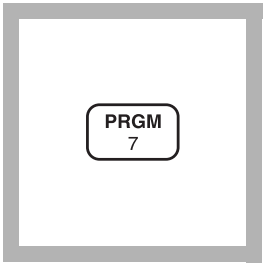
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITROGEN, AMMONIA, Low Range, Test 'N Tube (0 to 2.50 mg/L NH_3-N)

Salicylate Method*

For water, wastewater, and seawater

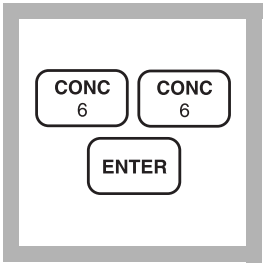


1. Enter the stored program number for low range nitrogen, ammonia Test 'N Tube.

Press: **PRGM**

The display will show:

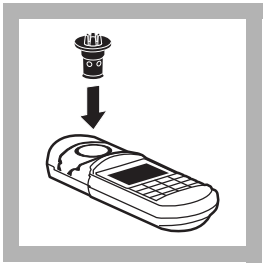
PRGM ?



2. Press: **66 ENTER**

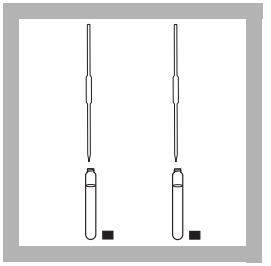
The display will show **mg/L, NH3-N** and the **ZERO** icon**.**

Note: *For alternate forms (NH_3), press the* **CONC** *key.*



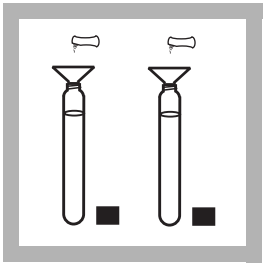
3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*

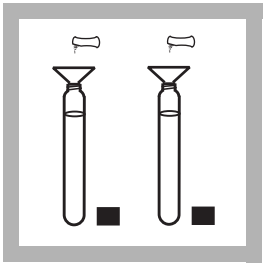


4. Remove the caps from 2 AmVer Diluent Reagent vials. Add 2 mL of sample to one vial (the sample). Add 2 mL of deionized water to the other vial (the blank).

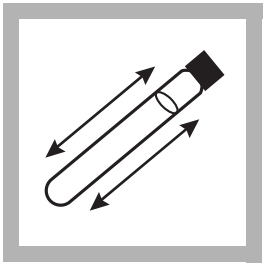
Note: *Adjust the pH of stored samples before analysis. See Interferences on page 365.*



5. Using a funnel, add the contents of one Ammonia Salicylate Reagent Powder Pillow for 5 mL sample to each vial.

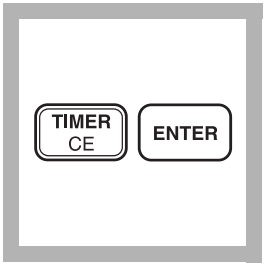


6. Using a funnel, add the contents of one Ammonia Cyanurate Reagent Powder Pillow for 5 mL sample to each vial.



7. Cap the vials tightly and shake thoroughly to dissolve the powder.

Note: *A green color will develop if ammonia is present.*



8. Press:

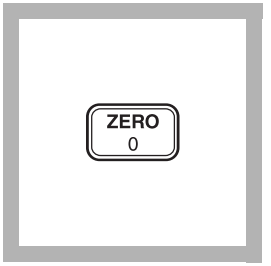
TIMER ENTER

A 20-minute reaction period will begin.

* Adapted from *Clin. Chim. Acta*, 14 403 (1966).

9. Wipe the outside of the vials with a towel. After the timer beeps, place the blank into the adapter. Tightly cover the vial with the instrument cap.

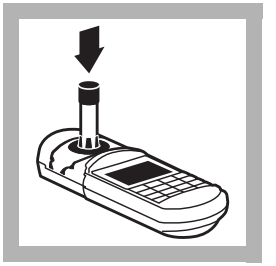
Note: *Wipe with a damp cloth followed by a dry one to remove fingerprints and other marks.*



10. Press: **ZERO**

The cursor will move to the right, then the display will show:

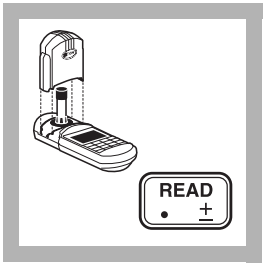
0.00 mg/L NH3-N



11. Place the prepared sample in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Tightly cover the sample cell with the instrument cap.

Press: **READ**

The cursor will move to the right, then the result in mg/L ammonia nitrogen will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust (Adjusting the Standard Curve) on page 47).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add one drop of 0.1 N sodium thiosulfate for each 0.3 mg/L Cl_2 in a one liter sample. Preserve the sample by reducing the pH to 2 or less with hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize with 5.0 N sodium hydroxide. Correct the test result for volume additions. See *Correcting for Volume Additions on page 22* for more information.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Nitrogen, Ammonia Ampule Standard Solution, 50 mg/L NH_3-N.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25 mL samples. Mix thoroughly.

c) Analyze each sample as described above. The nitrogen concentration should increase 0.20 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions, Section 1*, for more information.

Standard Solution Method

To check accuracy, use a 1.0 mg/L Nitrogen, Ammonia Standard Solution listed under Optional Reagents. Or, dilute 1 mL of solution from a
50 mg/L Ampule Standard for Nitrogen, Ammonia to 50 mL with deionized water using a 50-mL volumetric flask.

Method Performance

Precision

In a single laboratory, using a standard solution of 1.0 mg/L ammonia nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of $\pm$0.02 mg/L NH_3-N.

Estimated Detection Limit

The estimated detection limit for program 66 is 0.08 mg/L NH_3-N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level and Treatment
Calcium	2500 mg/L as $CaCO_3$
Iron	**1.** Determine the amount of iron present in the sample following one of the total iron procedures. **2.** Add the same iron concentration to the deionized water in step 4. The interference will then be successfully blanked out.
Magnesium	5000 mg/L as $CaCO_3$
Nitrite	30 mg/L as NO_2^--N
Nitrate	250 mg/L as NO_3^--N
Orthophosphate	250 mg/L as PO_4^{3-}-P
pH	Acidic or basic samples should be adjusted to about pH 7. Use 1 N Sodium Hydroxide Standard Solution for acidic samples and 1 N Hydrochloric Acid Standard Solution for basic samples.
Sulfate	300 mg/L as SO_4^{2-}

Interfering Substance	Interference Level and Treatment
Sulfide	**1.** Measure about 350 mL of sample in a 500 mL erlenmeyer flask. **2.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix. **3.** Filter the sample through a folded filter paper. **4.** Use the filtered solution in step 4.
Other	Less common interferences such as **hydrazine** and **glycine** will cause intensified colors in the prepared sample. **Turbidity** and **color** will give erroneous high values. Samples with severe interferences require distillation. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set. See Optional Apparatus at the end of this procedure.

Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

Pollution Prevention And Waste Management

The ammonia salicylate reagent contains sodium nitroferricyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Collect cyanide solutions for disposal as reactive (D001) waste. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. See *Section 3* for further information in proper disposal of these materials.

REQUIRED REAGENTS

	Cat. No.
AmVer Reagent Set for Nitrogen, Ammonia, Low Range TNT (25 tests) Includes: (1) 23952-66, (1) 23954-66, (1) 272-42, *(50) AmVer Low Range Vials	26045-45

Description	Quantity Required Per Test	Unit	Cat. No.
AmVer Diluent Reagent, Low Range Test 'N Tube	2 vials	50/pkg	*
Salicylate Reagent Powder Pillows, 5 mL sample	2 pillows	50/pkg	23952-66
Cyanurate Reagent Powder Pillows, 5 mL sample	2 pillows	50/pkg	23954-66

* Not available separately.

REQUIRED APPARATUS

Vial Adapter, COD	1	each	48464-00
Test Tube Rack	1-3	each	18641-00
Pipet, TenSette, 0-10 mL	1	each	19700-10
Pipet Tips for 19700-10	2	50/pkg	21997-96
Funnel, micro (for reagent addition)	1	each	25843-35

OPTIONAL REAGENTS

Nitrogen, Ammonia Standard Solution, 1.0 mg/L NH_3-N		500 mL	1891-49
Nitrogen, Ammonia Standard Solution, 10 mL Voluette ampules, 50 mg/L NH_3-N		16/pkg	14791-10
Nitrogen, Ammonia Standard Solution, 2 mL PourRite ampules, 50 mg/L NH_3-N		20/pkg	14791-20
Hydrochloric Acid, ACS		500 mL	134-49
Sodium Hydroxide Standard Solution, 5.0 N		50 mL SCDB	2450-26
Sodium Hydroxide, 1.000 N		100 mL MDB	1045-32
Sodium Thiosulfate Standard Solution, 0.1 N		100 mL MDB	323-32
Sulfide Inhibitor Reagent Powder Pillows		100/pkg	2418-99
Sulfuric Acid, 1.00 N		100 mL MDB	1270-32
Wastewater Effluent Standard, Inorganics (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28332-49
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit		each	21968-00
Cylinder, graduated, mixing, 25 mL, Class A		each	508-40
Distillation Apparatus Set		each	22653-00
Heater and Support Apparatus (for distillation), 115 Vac		each	22744-00
Heater and Support Apparatus (for distillation), 230 Vac		each	22744-02
Filter Paper, folded		100/box	1894-57
Flask, Erlenmeyer, 500 mL		each	505-49
Flask, volumetric, 50 mL, Class A		each	14547-41
Funnel, analytical (for filtering)		each	1083-68
Jack, laboratory (use with distillation apparatus)		each	22743-00
pH Indicator Paper, 1 to 11 pH		5 rolls/pkg	391-33
Ampule Breaker Kit, PourRite		each	24846-00
Thermometer, –20 to 110 °C, non-mercury		each	26357-02
Thermometer, –10 to 260 °C, non-mercury		each	26357-01

For Technical Assistance, Price and Ordering

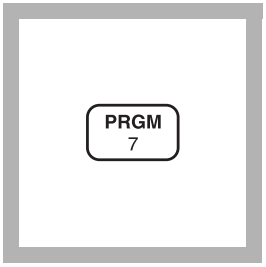
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITROGEN, AMMONIA, High Range, Test 'N Tube

(0 to 50 mg/L NH_3-N) **For water, wastewater, and seawater**

Salicylate Method*

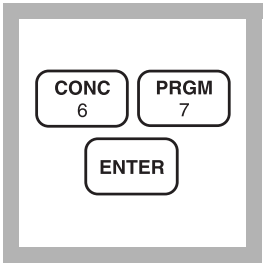


1. Enter the stored program number for nitrogen, ammonia, high range Test 'N Tube (NH_3-N) method.

Press: **PRGM**

The display will show:

PRGM ?

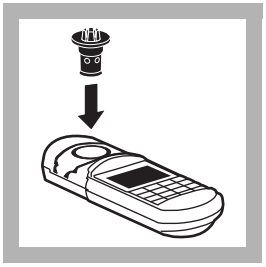


2. Press: **67 ENTER**

The display will show **mg/L, NH3-N** and the **ZERO** icon**.**

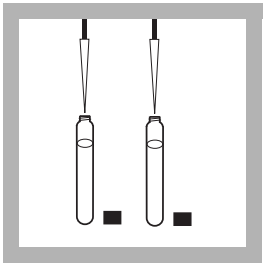
Note: *For alternate forms (NH_3), press the* **CONC** *key.*

Note: *For proof of accuracy, use a 10-mg/L nitrogen, ammonia standard in place of the sample.*

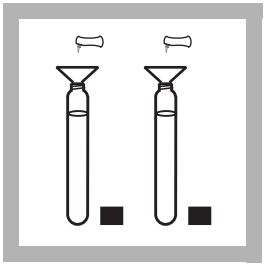


3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

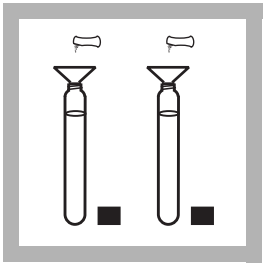
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



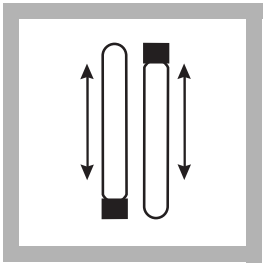
4. Remove the caps from 2 AmVer Diluent Reagent High Range Vials. Add 0.1 mL of sample to one vial (the sample). Add 0.1 mL of deionized water to the other (the blank).



5. Add the contents of 1 Ammonia Salicylate Reagent Powder Pillow for 5 mL Sample to each vial.

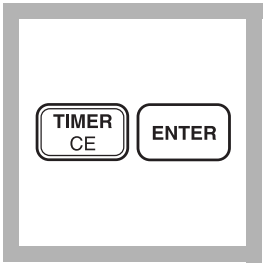


6. Add the contents of 1 Ammonia Cyanurate Reagent Powder Pillow for 5 mL Sample to each vial.



7. Cap the vials tightly and shake thoroughly to dissolve the powder.

Note: *A green color will develop if ammonia is present.*

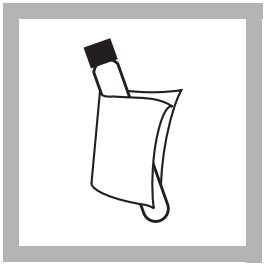


8. Press:

TIMER ENTER

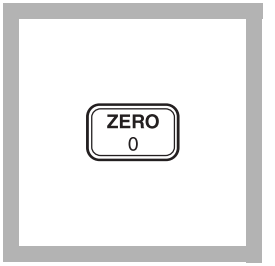
A 20-minute reaction period will begin.

* Adapted from *Clin. Chim. Acta*, **14** 403 (1966).



9. Clean the outside of the vial with a towel. After the timer beeps, place the blank into the vial adapter. Tightly cover the vial with the instrument cap.

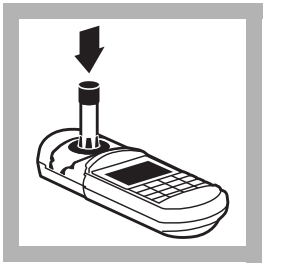
Note: *Wipe with a damp cloth and follow with a dry one to remove fingerprints and other marks.*



10. Press: **ZERO**

The cursor will move to the right, then the display will show:

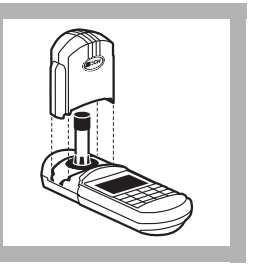
0 mg/L NH3-N



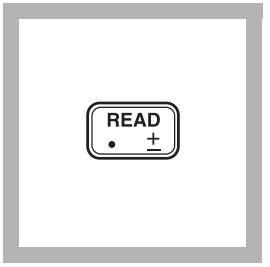
11. Place the prepared sample in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Tightly cover the vial with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L NH_3-N will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add one drop of 0.1 N sodium thiosulfate for each 0.3 mg/L Cl_2 in a one liter sample. Preserve the sample by reducing the pH to 2 or less with hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize with 5.0 N sodium hydroxide. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method

a) Snap the top off an Ammonia PourRite Ampule Standard, 150 mg/L NH_3-N.

b) Use the TenSette Pipet to add 0.2, 0.4 and 0.6 mL of standard to three 25-mL samples. Swirl to mix.

c) Analyze each sample as described above. The ammonia concentration should increase approximately 1.2 mg/L NH_3-N for each 0.2 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To check accuracy, use a 10 or 50 mg/L Nitrogen, Ammonia Standard Solution or use a Nitrogen, Ammonia Voluette Ampule Standard, 50 mg/L.

Method Performance

Precision

In a single laboratory, using a standard solution of 50 mg/L ammonia nitrogen (NH_3-N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of $\pm$5 mg/L NH_3-N.

Estimated Detection Limit

The estimated detection limit for program 67 is 1 mg/L NH_3-N. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following ions may interfere when present in concentrations exceeding those listed below.

In some lab environments, airborne cross contamination of the blank is possible. Complete preparation of the blank before opening or handling any samples or standards to avoid transfer of ammonia. If sample or standard containers have already been open, move to a separate area of the lab to prepare the blank.

Substance	Concentration and Suggested Treatments
Acidic or basic samples	Adjust to approximately pH 7. Use 1 N Sodium Hydroxide Standard Solution for acidic samples and 1 N Hydrochloric Acid Standard Solution for basic samples.
Calcium	50,000 mg/L as $CaCO_3$
Glycine, hydrazine	Will cause intensified colors in the prepared sample.
Magnesium	300,000 mg/L as $CaCO_3$
Iron	Eliminate iron interference as follows: **1.** Determine the amount of iron present in the sample using one of the total iron procedures. **2.** Add the same iron concentration to the deionized water in step 4. **3.** The interference will then be successfully blanked out.
Nitrite	600 mg/L as NO_2^--N
Nitrate	5,000 mg/L as NO_3^--N
Orthophosphate	5,000 mg/L as PO_4^{3-}-P
Sulfate	6,000 mg/L as SO_4^{2-}
Sulfide	Sulfide will intensify the color. Eliminate sulfide interference as follows: **1.** Measure about 350 mL of sample in a 500 mL Erlenmeyer flask. **2.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix. **3.** Filter the sample through folded filter paper. Use the filtered solution in step 4.
Turbidity and color	Give erroneous high values. Samples with severe interferences require distillation. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set.

Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a green-colored solution.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheets* for information specific to the reagents used. For additional information, refer to *Section 3*.

Pollution Prevention And Waste Management

The ammonia salicylate reagent contains sodium nitroferricyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Collect cyanide solutions for disposal as reactive (D001) waste. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. See *Section 3* for further information in proper disposal of these materials.

REQUIRED REAGENTS

AmVer™ Reagent Set for Nitrogen, Ammonia, High Range, TNT (25 tests) 26069-45
Includes: (1) 23952-66, (1) 23954-66, (1) 272-42, *(50) AmVer HR Vials

Description	Quantity Required Per Test	Unit	Cat. No.
AmVer™ HR Reagent Test 'N Tube™ Vials	2 vials	50/pkg	*
Ammonia Salicylate Reagent Powder Pillows	2 pillows	50/pkg	23952-66
Ammonia Cyanurate Reagent Powder Pillows	2 pillows	50/pkg	23954-66

REQUIRED APPARATUS

COD/TNT Adapter	1	each	48464-00
Pipet, TenSette®, 0-1 mL	1	each	19700-01
Pipet Tips for 19700-01	varies	50/pkg	21856-96
Test Tube Rack	1-3	each	18641-00
Funnel, micro (for reagent addition)	1	each	25843-35

* Not available separately.

OPTIONAL REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Nitrogen, Ammonia Standard Solution, 50 mg/L NH_3-N		500 mL	14791-50
Nitrogen, Ammonia Standard Solution, 10 mg/L NH_3-N		500 mL	153-49
Ammonia Standard Solution, PourRite™ ampules, 150 mg/L NH_3-N, 2 mL		20/pkg	21284-20
Hydrochloric Acid, ACS		500 mL	134-49
Sodium Hydroxide Standard Solution, 5.0 N		50 mL	2450-26
Sodium Hydroxide Standard Solution, 1.0 N		100 mL	1045-32
Sodium Thiosulfate Standard Solution, 0.1 N		100 mL	323-32
Sulfide Inhibitor Powder Pillows		100/pkg	2418-99
Sulfuric Acid, 1.00 N		100 mL MDB	1270-32
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3, PO_4, COD, SO_4, TOC)		500 mL	28331-49
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Cylinder, 25 mL, graduated, mixing		each	20886-40
Distillation Apparatus Set, general purpose		each	22653-00
Heater and Support Apparatus (for distillation), 115 VAC		each	22744-00
Heater and Support Apparatus (for distillation), 230 VAC		each	22744-02
Filter Paper, folded		100/pkg	1894-57
Flask, Erlenmeyer, 500 mL		each	505-49
Funnel, analytical (for filtering)		each	1083-68
Jack, laboratory (use with distillation apparatus)		each	22743-00
pH Indicator Paper, 1 to 11 pH		5 rolls/pkg	391-33
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite™ Ampule Breaker		each	24846-00
Sample Cell, 10-20-25 mL, w/cap		6/pkg	24019-06

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

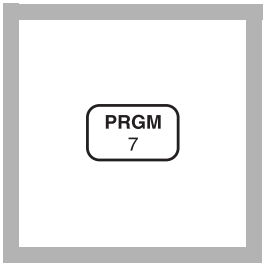
Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITROGEN, Total Inorganic, Test 'N Tube™ (0 to 25.0 mg/L N)

Titanium Trichloride Reduction Method

For water, wastewater, and seawater

Requires Centrifuge

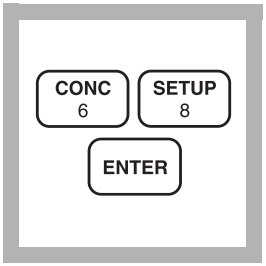


1. Enter the stored program number for Test 'N Tube Total Inorganic Nitrogen.

Press: **PRGM**

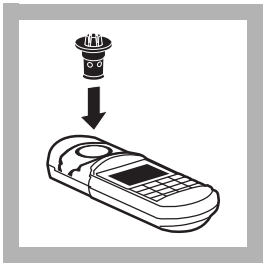
The display will show:

PRGM ?



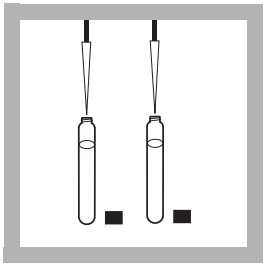
2. Press: **68 ENTER**

The display will show **mg/L, N** and the **ZERO** icon**.**

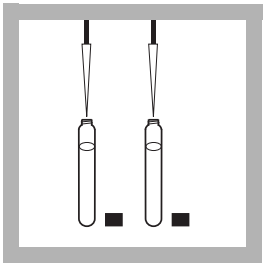


3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert.

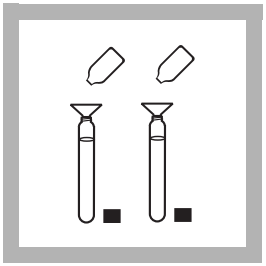
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



4. Pipet 1 mL of Total Inorganic Nitrogen Pretreatment Base Concentrate into each of 2 Total Inorganic Nitrogen Pretreatment Diluent Vials.



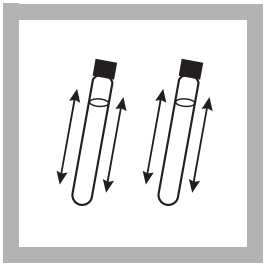
5. Pipet 1 mL of sample into 1 TIN Diluent Vial (the sample). Pipet 1 mL of deionized water into the other vial (the blank). Cap the vials and shake for 30 seconds to mix.



6. Snap the necks off two Total Inorganic Nitrogen Reductant ampules and pour the contents of one into the TIN Diluent Vial containing sample. Repeat for the second vial, the blank.

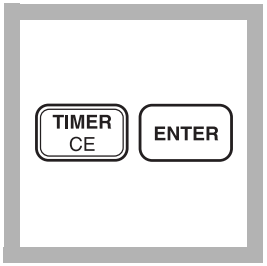
Note: *For safety, wear gloves while breaking the ampules.*

Note: *A black precipitate will form immediately.*



7. Cap the vials. Shake gently for 30 seconds to mix the reagents. Allow the vials to sit for at least one minute.

Note: *The precipitate should remain black after shaking. Excessive shaking will cause a white precipitate and low results.*

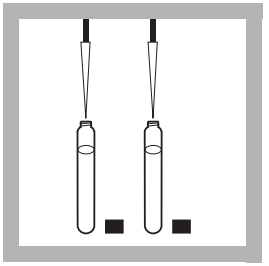


8. Centrifuge the vials for 3 minutes or until the solids settle to the bottom of the vial.

Press: **TIMER ENTER**

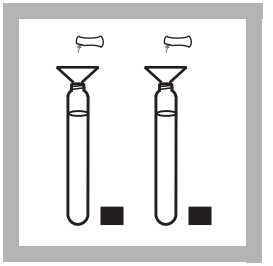
immediately after starting the centrifuge.

Note: *The precipitate will settle without using a centrifuge, but it may take up to 30 minutes.*

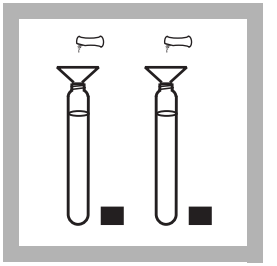


9. Remove the caps from 2 AmVer Diluent Reagent Test 'N Tubes for Low Range Ammonia Nitrogen. Using a pipet, add 2 mL of centrifuged sample into 1 vial. Add 2 mL of centrifuged blank to the other vial. Label the vials appropriately.

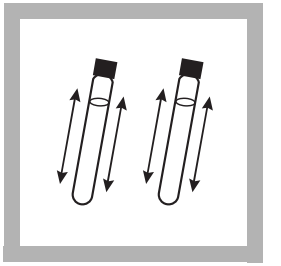
Note: *Pipet carefully to avoid disturbing the sediment.*



10. Using a funnel, add the contents of one Ammonia Salicylate Reagent Powder Pillow to each vial.

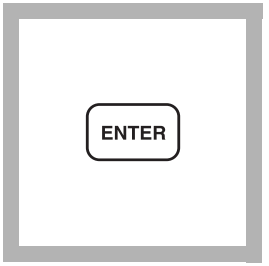


11. Using a funnel, add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each vial.



12. Cap the vials tightly and shake thoroughly to dissolve the powder.

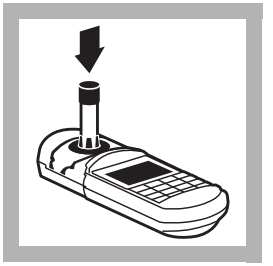
Note: *A green color will develop if inorganic nitrogen is present.*



13. The display will show: **15:00 TIMER 2**

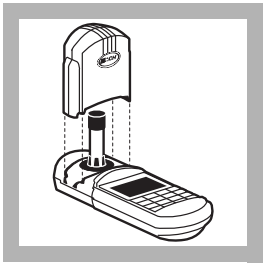
Press: **ENTER**

A 15-minute reaction period will begin.

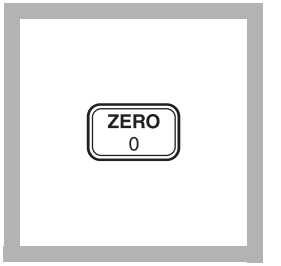


14. After the timer beeps, clean the outside of the vials with a towel. Place the blank in the adapter. Push straight down on the top of the vial until it seats solidly into the adapter. Do not move the vial from side to side as this can cause errors.

Note: *Wipe with a damp cloth and follow with a dry one to remove fingerprints and other marks.*



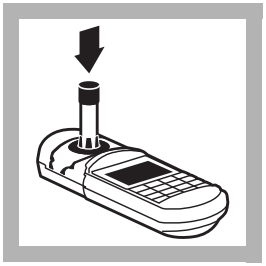
15. Tightly cover the sample cell with the instrument cap.



16. Press: **ZERO**

The cursor will move to the right, then the display will show:

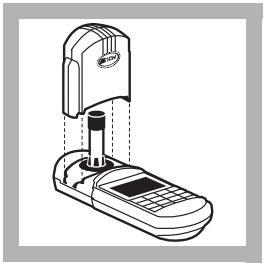
0.0 mg/L N



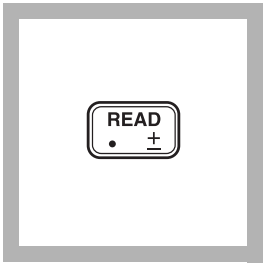
17. Place the prepared sample in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



18. Tightly cover the sample cell with the instrument cap.



19. Press: **READ**

The cursor will move to the right, then the result in mg/L total inorganic nitrogen will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling And Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

If chlorine is known to be present, add 1 drop of 0.1 N sodium thiosulfate for each 0.3 mg/L Cl_2 in a 1 liter sample.

Preserve the sample by reducing the pH to 2 or less with concentrated hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N Sodium Hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1*.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a fresh High Range Nitrate Nitrogen PourRite Ampule Standard, 500 mg/L NO_3^--N.

c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to 3 25-mL mixing cylinders. Mix thoroughly.

d) Analyze each sample as described in the procedure; use a 1-mL aliquot of the prepared sample in Step 5. The nitrogen concentration should increase about 1.8 to 1.9 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To check accuracy, use a 10.0 mg/L Nitrate Nitrogen Standard Solution listed under Optional Reagents. Alternatively, a 20.0 mg/L nitrate nitrogen standard can be prepared by diluting 2 mL of solution from a PourRite Ampule Standard for High Range Nitrate Nitrogen, 500 mg/L NO_3^--N, to 50 mL with deionized water. Substitute this standard for the sample and perform the test as described. The recovery of the standards should be about 90-95%.

Method Performance

Precision/Accuracy

The total inorganic nitrogen test provides an estimate of the total nitrite, nitrate, and ammonia nitrogen load in water or wastewater samples. This test is most applicable for monitoring an industrial process stream or a wastewater treatment stream where it is important to track the inorganic nitrogen load as it passes through the treatment process. The test exhibits different recoveries of each of the three nitrogen species, as summarized below. This test is not recommended for quantifying only one of the three species. In that case, use a specific procedure for each particular analyte.

Ammonia Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NH_3^-N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 21.3 mg/L with a standard deviation of
± 0.77 mg/L N (replicate number = 7 per reagent lot).

Nitrate Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NO_3^-N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 18.9 mg/L with a standard deviation of
± 0.55 mg/L N (replicate number = 7 per reagent lot).

Nitrite Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NO_2^-N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 14.6 mg/L with a standard deviation of
± 0.77 mg/L N (replicate number = 7 per reagent lot).

Estimated Detection Limit
The estimated detection limit for program 68 is 0.7 mg/L N. For more information on the estimated detection limit, *see Section 1*.

Interferences

The following ions may interfere when present in concentrations exceeding those listed below:

Species	Level	Effect
Calcium	1000 mg/L as $CaCO_3$	Positive
Manganese (IV)	3 mg/L	Negative
Magnesium	1000 mg/L as $CaCO_3$	Positive
Sulfide	3 mg/L	Negative
Sulfate	250 mg/L	Negative

The following do not interfere below the levels listed:

Species	Level
Al^{3+}	8 mg/L
Ba^{2+}	40 mg/L
Cu^{2+}	40 mg/L
Fe^{3+}	8 mg/L
Zn^{2+}	80 mg/L
F^-	40 mg/L
PO_4^{3-}-P	8 mg/L
SiO_2	80 mg/L
EDTA	80 mg/L

Summary of Method

Titanium (III) ions reduce nitrate and nitrite to ammonia in a basic environment. After centrifugation to remove solids, the ammonia is combined with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

REQUIRED REAGENTS

Total Inorganic Nitrogen Pretreatment Reagent Set ($TiCl_3$ Reduction) (25 tests)..... 26049-45
Includes: (1) 26051-50, (1) 2040-59, *(50) TIN Pretreatment Diluent Vials

AmVer™ Reagent Set for Nitrogen, Ammonia, Low Range (25 tests)..... 26045-45
Includes: (1) 23952-66, (1) 23954-66 , (1) 272-42, *(50) AmVer™ Diluent LR Vials

Description	Quantity Required Per Test	Unit	Cat. No.
Total Inorganic Nitrogen Pretreatment Diluent Vials	2 vials	50/pkg	*
Total Inorganic Nitrogen Reductant Ampules	2 ampules	50/pkg	26051-50
Total Inorganic Nitrogen Pretreatment Base Concentrate	2 mL	50 mL	2040-59
AmVer™ Diluent Reagent, Low Range Vials	2 vials	50/pkg	*
Ammonia Salicylate Reagent Powder Pillows for 5-mL sample	2 pillows	50/pkg	23952-66
Ammonia Cyanurate Reagent Powder Pillows for 5-mL sample	2 pillows	50/pkg	23954-66

REQUIRED APPARATUS

Centrifuge, 115V	1	each	26765-00
Centrifuge, 230V	1	each	26765-02
COD/TNT Vial Adapter	1	each	48464-00
Funnel, micro	1	each	25843-35
Pipet, TenSette®, 0.1 to 1.0	1	each	19700-01
Pipet Tips for 19700-01	2	50/pkg	21856-96
Test Tube Rack	1	each	18641-00

OPTIONAL REAGENTS

Hydrochloric Acid, ACS	500 mL	134-49
Nitrate Nitrogen Standard Solution, 10 mg/L NO_3^--N	500 mL	307-49
Nitrate Nitrogen Standard Solution, PourRite Ampules, 500 mg/L NO_3^--N, 2 mL	20/pkg	14260-20
Sodium Hydroxide Standard Solution, 5.0 N	50 mL SCDB	2450-26
Sodium Thiosulfate Standard Solution, 0.1 N	100 mL MDB	323-32
Wastewater Effluent Standard, Inorganics (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49
Water, deionized	4 L	272-56

* These items are not sold separately. Please order the complete set (cat. no. 26049-45 or 26045-45).

OPTIONAL APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Cylinder, graduated, mixing, 25 mL		each	20886-40
Flask, volumetric, Class A, 50.0 mL		each	14574-41
pH Indicator Paper, 1 to 11 pH		5 rolls/pkg	391-33
Pipet, volumetric, Class A, 2.0 mL		each	14515-36
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite Ampule Breaker		each	24846-00

For Technical Assistance, Price and Ordering

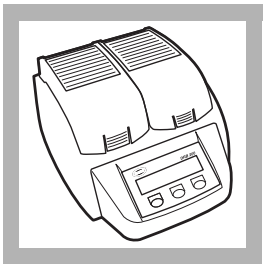
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITROGEN, TOTAL, Test 'N Tube (0.0 to 25.0 mg/L N)

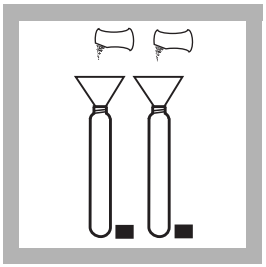
TNT Persulfate Digestion Method

For water and wastewater



1. Turn on the DRB 200 Reactor. Heat to 103-106 °C (optimum temperature is 105 °C).

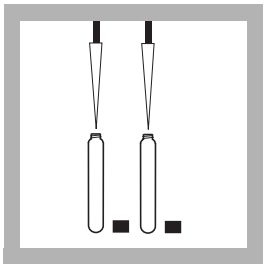
Note: *For proof of accuracy, run a 20 mg/L NH_3-N standard through digestion and analysis.*



2. Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to each of two Total Nitrogen Hydroxide Reagent vials.

Note: *Wipe off any reagent that may get on the lid or the tube threads.*

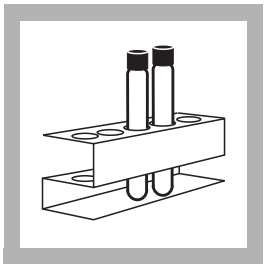
Note: *One reagent blank is sufficient for each set of samples.*



3. Add 2 mL of sample to one vial. Add 2 mL of organic-free water to another vial (the reagent blank). Cap both vials and shake vigorously (about 30 seconds). Place the vials in the Reactor. Heat for 30 minutes.

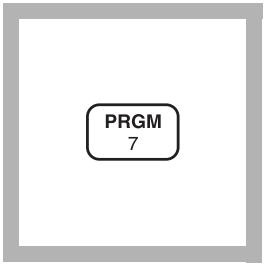
Note: *The reagent may not dissolve completely after shaking.*

Note: *Alternate water must be free of all nitrogen-containing species.*



4. Using finger cots or gloves, remove the hot vials from the reactor and allow to cool to room temperature.

Note: *It is very important to remove the vials from the Reactor after exactly 30 minutes.*

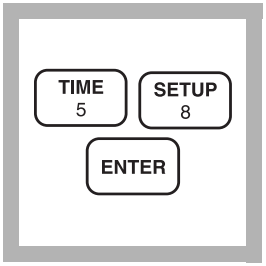


5. Enter the stored program number for Test ‘N Tube Total Nitrogen.

Press: **PRGM**

The display will show:

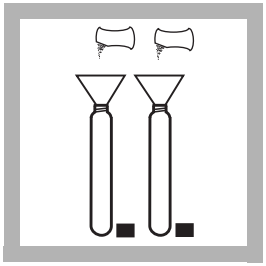
PRGM ?



6. Press: **58 ENTER**

The display will show **mg/L, N** and the **ZERO** icon**.**

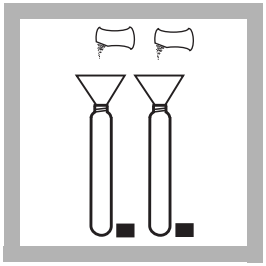
Note: *For alternate forms (NH_3, NO_3), press the* **CONC** *key.*



7. Remove the caps from the digested vials and add the contents of one TN Reagent A Powder Pillow to each vial. Cap the vials and shake for 15 seconds.

Press: **TIMER ENTER** after shaking.

A three-minute reaction period will begin.



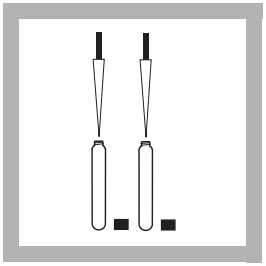
8. After the timer beeps, remove the caps from the vials and add one TN Reagent B Powder Pillow to each vial. Cap the vials and shake for 15 seconds. The display will show:

02:00 Timer 2

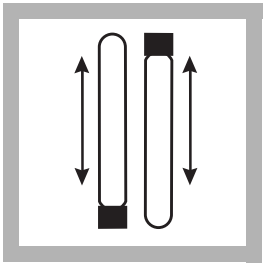
Press **ENTER** after shaking.

A two-minute reaction period will begin.

Note: *The reagent will not completely dissolve. The solution will begin to turn yellow.*

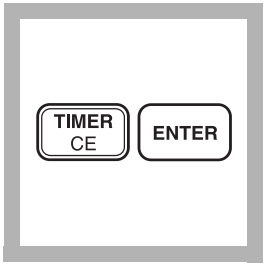


9. After the timer beeps, remove the caps from two TN Reagent C Vials. Add 2 mL of digested, treated sample to one vial. Add 2 mL of the digested, treated reagent blank to the second TN Reagent C Vial.



10. Cap and invert 10 times to mix. Use slow, deliberate inversions for complete recovery. The vials will be warm.

Note: *Follow these instructions for inversion or low results may occur. Hold the vial vertical with the cap up. Invert the vial and wait for all of the solution to flow to the cap end. Pause. Return the vial to the upright position and wait for all of the solution to flow to the vial bottom. This is one inversion (10 inversions = 30 seconds).*

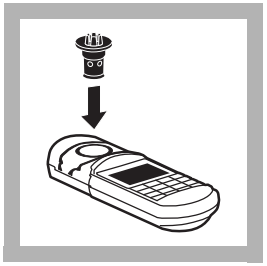


11. The display will show: **05:00 Timer 3**

Press: **ENTER**

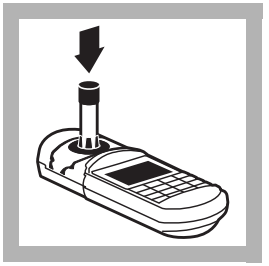
A five-minute reaction period will begin.

Note: *The yellow color will intensify.*



12. During the reaction period, insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

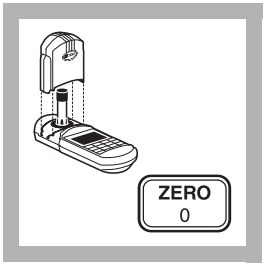
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



13. After the timer beeps, wipe the TN Reagent C vial containing the reagent blank. Place the vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



14. Tightly cover the vial with the instrument cap.

Press: **ZERO**

The cursor will move to the right, then the display will show:

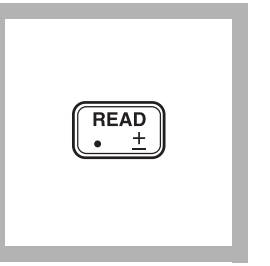
0.0 mg/L N

Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*

Note: *The reagent blank is stable when stored in the dark; see* Blanks For Colorimetric Measurement *following these steps.*

15. Wipe the TN Reagent C vial containing the sample and place it into the adapter. Tightly cover the vial with the instrument cap.

Note: *Multiple samples may be read after zeroing on one reagent blank.*



16. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrogen (N) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Note: *If the display flashes "limit", dilute the sample and repeat the digestion and the colorimetric finish. The digestion must be repeated for accurate results; diluting and repeating the color finish does not yield complete results. Multiply the result by the dilution factor; see Section 1.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated sulfuric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1*.

Accuracy Check

This method generally yields 95-100% recovery on organic nitrogen standards. For proof of accuracy Hach offers a set of three Primary Standards for Kjeldahl Nitrogen.

1. Prepare one or more of the following three solutions. Each preparation is for an equivalent 25 mg/L N standard. Use water that is free of all organic and nitrogen-containing species.

   a) Weigh 0.3379 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   b) Weigh 0.4416 g of Glycine p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   c) Weigh 0.5274 g of Nicotinic p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

2. Analyze each of these solutions using the test procedure above. Calculate the percent recovery for each using this formula:

$$\% \text{ recovery} = \frac{\text{measured concentration}}{25} \times 100$$

The percent recovery should be:

Compound	Lowest Expected % Recovery
Ammonia-PTSA	95%
Glycine-PTSA	95%
Nicotinic-PTSA	95%

Hach analysts have found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

Standard Solution Method

Substitute 2 mL of a 20 mg/L ammonia nitrogen standard solution for the sample. To prepare a 20-mg/L standard, use a 20-mL Class A pipet to transfer 20 mL of a 100-mg/L Ammonia Nitrogen Standard (see *Optional Reagents*) to a 100-mL Class A volumetric flask. Dilute to the line with organic-free water. A single analyst should obtain less than 5% variation on replicates. Comparison of the user-obtained value with the standard concentration is an indication of test performance for this user.

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off an Ammonia Nitrogen Voluette Ampule Standard Solution, 160 mg/L as NH_3-N.

c) Use the TenSette Pipet to add 0.3 mL, 0.6 mL, and 0.9 mL of standard, respectively, to the three mixing cylinders.

d) Stopper each cylinder and mix thoroughly.

e) Add 2 mL of each prepared solution, respectively, to three TN Hydroxide Reagent Sample Digestion Vials.

f) Analyze each standard addition sample as described in the procedure. The nitrogen concentration should increase 2 mg/L for each 0.3 mL of standard added.

g) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

Blanks for Colorimetric Measurement

The reagent blank may be used up to 7 days for measurements using the same lots of reagents. Store the reagent blank in the dark at room temperature (18-25 °C). If a small amount of white floc appears prior to the end of one week, discard the reagent blank and prepare a new one.

Method Performance

Precision

A Hach chemist analyzed two independent nutrient standards. The lowest average percent recovery was 95% with a standard deviation of ±2%.

In a single laboratory, using a standard solution of 15.0 mg/L N and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than ±0.5 mg/L N. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program 58 is 2 mg/L N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering substances that resulted in a concentration change of ±10%:

Substance	Level and Effect
Bromide	>60 ppm; positive interference
Chloride	>1000 ppm; positive interference

The substances in the following table have been tested and found **not** to interfere up to the indicated levels (in mg/L):

Substance	Maximum Level Tested (mg/L)
Barium	2.6
Calcium	300
Chromium (3+)	0.5
Iron	2
Lead	6.6 ppb
Magnesium	500
Organic Carbon	150
pH	13 pH units
Phosphorus	100
Silica	150
Silver	0.9
Tin	1.5

Hach chemists tested this chemistry on standard nitrogen solutions prepared from the following compounds and obtained ≥95% recovery:

- Ammonium chloride
- Ammonium sulfate
- Ammonium acetate
- Urea
- Glycine

Ammonium chloride or nicotinic-PTSA spikes in domestic influent, effluent and the ASTM standard specification for substitute wastewater (D 5905-96) also resulted in ≥95% recovery.

Large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

Summary of Method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum near 420 nm.

REQUIRED REAGENTS

Description	Cat. No.
Test 'N Tube Total Nitrogen Reagent Set (50 vials)	26722-45
Includes:	
TN Reagent C Vials, Acid Solution*, 50/pkg	26721-45
TN Hydroxide Reagent Sample Digestion Vials*, 50/pkg	26717-45

Description	Quantity Required Per Test	Unit	Cat. No.
TN Persulfate Reagent Powder Pillows	2 pillows	100/pkg	26718-49
TN Reagent A, Bisulfite Powder Pillows	2 pillows	100/pkg	26719-49
TN Reagent B, Indicator Powder Pillows	2 pillows	100/pkg	26720-49

REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
COD/TNT Adapter	1	each	48464-00
Funnel, micro	1	each	25843-35
Pipet, TenSette, 1.0-10.0 mL	1	each	19700-10
Pipet Tips for 19700-10	1	50/pkg	21997-96
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips for 19700-01	2	50/pkg	21856-96
Test Tube Cooling Rack	1-3	each	18641-00

* Not available separately.

OPTIONAL REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Nitrogen,Ammonia, 100 mg/L NH_3-N	500 mL	24065-49	
Nitrogen, Ammonia, Voluette Ampule, 160 mg/L NH_3–N, 10 mL		16/pkg	21091-10
Sulfuric Acid, ACS		500 mL	979-49
Primary Standards for Kjeldahl Nitrogen		set of 3	22778-00
Ammonium p-Toluenesulfonate		25 g	22779-24
Glycine p-Toluenesulfonate		25 g	22780-24
Nicotinic Acid p-Toluenesulfonate		25 g	22781-24
Sodium Hydroxide Standard Solution, 5.0 N		50 mL MDB	2450-26
Wastewater Effluent Standard, Inorganics (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28332-49
Water, organic-free		500 mL	26415-49

OPTIONAL APPARATUS

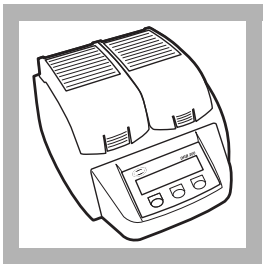
Ampule Breaker Kit	each	21968-00
Balance, analytical, 115 VAC	each	28014-01
Balance, analytical, 230 VAC	each	28014-02
Cots, finger	2/pkg	14647-02
Cylinder, graduated, mixing, 25 mL (3 required)	each	26363-40
Flask, volumetric, Class A, 1000 mL (3 required)	each	14574-53
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, volumetric, Class A, 20 mL	each	14515-20
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33

DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm	LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm	LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm	LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm	LTV082.52.30001

NITROGEN, TOTAL, HR, Test 'N Tube™ (10.0 to 150.0 mg/L N)

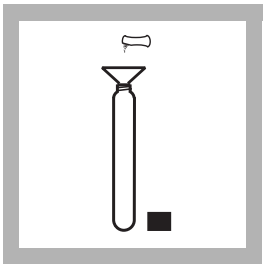
TNT Persulfate Digestion Method

For water and wastewater



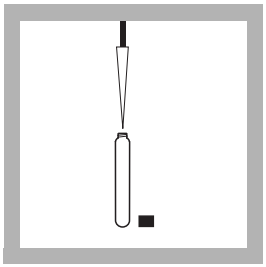
1. Turn on the DRB 200 Reactor. Heat to 103-106 °C (optimum temperature is 105 °C).

Note: *For proof of accuracy, run a 125 mg/L NH_3-N standard through digestion and analysis.*



2. Prepare a reagent blank: Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to one HR Total Nitrogen Hydroxide Digestion Vial.

Note: *Wipe off any reagent that gets on the lid or the tube threads.*



3. Add 0.5 mL of organic-free water to the vial. Cap the vial and shake vigorously for about 30 seconds.

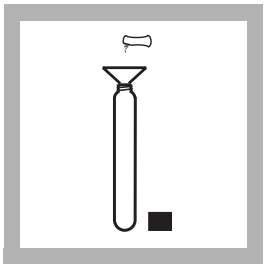
Process this reagent blank exactly the same as the sample, including digestion and color finish. Proceed to *step 6.*

Note: *Alternate water must be free of all nitrogen-containing species.*

Note: *The persulfate reagent may not dissolve completely after shaking.*

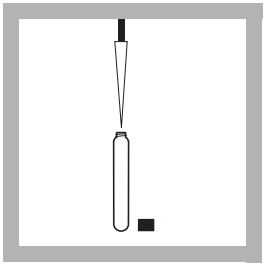
Note: *One reagent blank is sufficient for each set of samples using the same lots of reagents.*

Note: *The reagent blank is stable for as long as seven days when stored in the dark; see* Blanks for Colorimetric Measurement *following this procedure.*



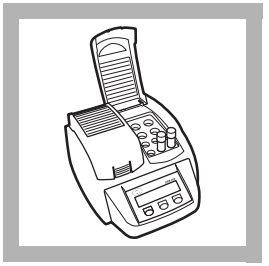
4. Prepare a sample: Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to one HR Total Nitrogen Hydroxide Digestion Vial.

Note: *Wipe off any reagent that gets on the lid or the tube threads.*

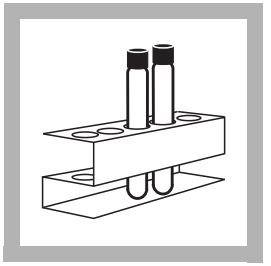


5. Add 0.5 mL of sample to the vial. Cap the vial and shake vigorously for about 30 seconds.

Note: *The persulfate reagent may not dissolve completely after shaking.*

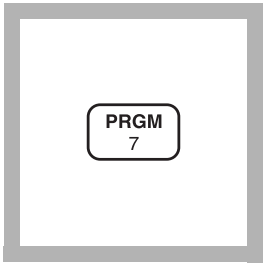


6. Place the vials in the Reactor. Heat for 30 minutes.



7. Using finger cots or gloves, remove the hot vials from the reactor and allow to cool to room temperature.

Note: *It is very important to remove the vials from the Reactor after exactly 30 minutes.*

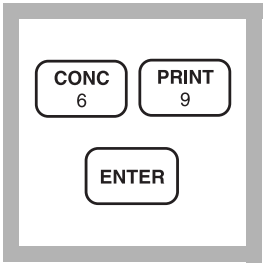


8. Enter the stored program number for Test 'N Tube HR Total Nitrogen.

Press: **PRGM**

The display will show:

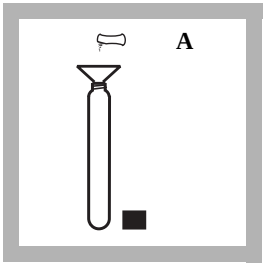
PRGM ?



9. **Press: 69 ENTER**

The display will show **mg/L, N** and the **ZERO** icon**.**

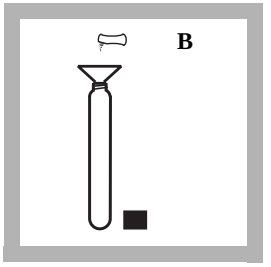
Note: *For alternate forms (NH_3, NO_3), press the* **CONC** *key.*



10. Add the contents of one Total Nitrogen Reagent A Powder Pillow to the vial containing the digested blank or sample. Cap the vial and shake for 15 seconds.

Press: **TIMER ENTER** after shaking.

A three-minute reaction period will begin.



11. After the timer beeps, add one Total Nitrogen Reagent B Powder Pillow to the vial. Cap the vial and shake for 15 seconds.

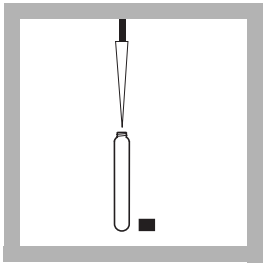
The display will show:

02:00 Timer 2

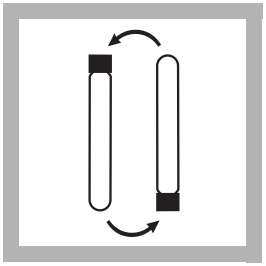
Press **ENTER** after shaking.

A two-minute reaction period will begin.

Note: *The reagent will not completely dissolve. The solution will begin to turn yellow.*

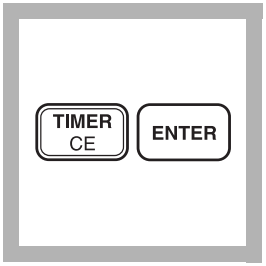


12. After the timer beeps, remove the cap from one Total Nitrogen Reagent C Vial. Add 2 mL of digested, treated sample (or reagent blank) to the vial. The vial will be warm.



13. Cap and invert slowly 10 times to mix. The vial will be warm.

Note: *Proper mixing is important for complete recovery. Hold the vial vertical with the cap up. Invert the vial and wait for all of the solution to flow to the cap end. Pause. Return the vial to the upright position and wait for all of the solution to flow to the vial bottom. This is one inversion (10 inversions = 30 seconds).*



14. The display will show: **05:00 Timer 3**

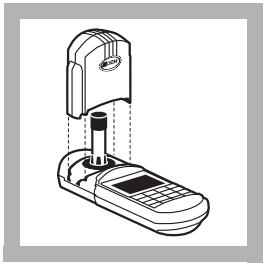
Press: **ENTER**

A five-minute reaction period will begin. Do not invert the vial again.

Note: *The yellow color will intensify.*

15. Insert the COD/ TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



16. When the timer beeps, wipe the outside of the Total Nitrogen Reagent C vial containing the reagent blank.

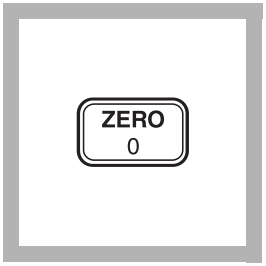
Place the vial into the adapter with the Hach logo facing the front of the instrument.

Push straight down on the top of the vial until it seats solidly into the adapter.

Tightly cover the vial with the instrument cap.

Note: *Do not move the vial from side to side during insertion, as this can cause errors.*

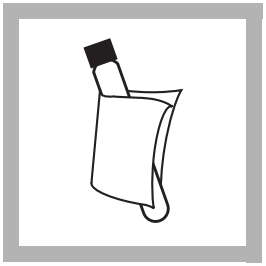
Note: *Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.*



17. Press: **ZERO**

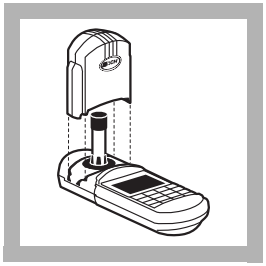
The cursor will move to the right, then the display will show:

0 mg/L N



18. Wipe the Total Nitrogen Reagent C vial containing the sample.

Note: *Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.*



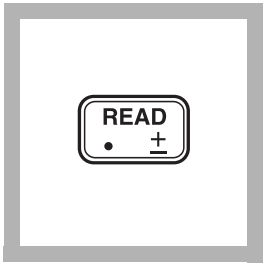
19. Place the vial into the adapter with the Hach logo facing the front of the instrument.

Push straight down on the top of the vial until it seats solidly into the adapter.

Tightly cover the vial with the instrument cap.

Note: *Do not move the vial from side to side during insertion, as this can cause errors.*

Note: *Multiple samples may be read after zeroing on one reagent blank.*



20. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrogen (N) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see* Standard Adjust *in* Section1 *of the Procedures Manual).*

Note: *If the display flashes* **Limit***, dilute the sample and repeat the digestion and the colorimetric finish. The digestion must be repeated for accurate results; diluting and repeating the color finish does not yield complete results. Multiply the result by the dilution factor; see SECTION 1.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated sulfuric acid (at least 2 mL/L). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1*.

Accuracy Check

This method generally yields 95-100% recovery on organic nitrogen standards. For proof of accuracy Hach offers a set of three Primary Standards for Kjeldahl Nitrogen.

1. Prepare one or more of the following three solutions. Each preparation is for an equivalent 120 mg/L N standard. Use water that is free of all organic and nitrogen-containing species.

   a) Weigh 1.6208 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   b) Weigh 2.1179 g of Glycine p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   c) Weigh 2.5295 g of Nicotinic p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

2. Analyze each of these solutions using the test procedure above. Calculate the percent recovery for each using this formula:

$$\% \text{ recovery} = \frac{\text{measured concentration}}{120} \times 100$$

The percent recovery should be:

Compound	Lowest Expected % Recovery
Ammonia-PTSA	95%
Glycine-PTSA	95%
Nicotinic-PTSA	95%

Hach analysts have found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

Standard Solution Method

For proof of accuracy, substitute 0.5 mL of a 125 mg/L ammonia nitrogen standard solution for the sample in the procedure. To prepare a 125-mg/L standard, use a 25-mL Class A pipet to transfer 25.00 mL of a 1000-mg/L Ammonia Nitrogen Standard

(see *OPTIONAL REAGENTS* on page *400*) to a 200-mL Class A volumetric flask. Dilute to the line with organic-free water.

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off an Ammonia Nitrogen Voluette™ Ampule Standard Solution, 1000 mg/L as NH_3-N.

c) Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to the three mixing cylinders.

d) Stopper each cylinder and mix thoroughly.

e) Add 0.5 mL of each prepared solution, respectively, to three HR Total Nitrogen Hydroxide Digestion Vials.

f) Analyze each standard addition sample as described in the procedure. The nitrogen concentration should increase 4 mg/L N for each 0.1 mL of standard added.

g) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

Blanks for Colorimetric Measurement

The reagent blank may be used repeatedly for measurements using the same lots of reagents. Store the reagent blank in the dark at room temperature (18–25 °C) for a maximum of seven days. If a small amount of white floc appears prior to the end of one week, discard the reagent blank and prepare a new one.

Method Performance

Precision

In a single laboratory, using a standard solution of 125 mg/L N and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than 3 mg/L N. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program 69 is 7 mg/L N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering substances that resulted in a concentration change of ±10%:

Substance	Level and Effect
Bromide	> 240 ppm; positive interference
Chloride	≥3000 ppm; positive interference

The substances in the following table have been tested and found **not** to interfere up to the indicated levels:

Substance	Maximum Level Tested (mg/L)
Barium	10.4
Calcium	1200
Chromium (3+)	2
Iron	8
Lead	26.4 ppb
Magnesium	2000
Organic Carbon	600
pH	13 pH units
Phosphorus	400
Silica	600
Silver	3.6
Tin	6.0

The large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

Summary of Method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum near 420 nm.

REQUIRED REAGENTS

Test 'N Tube HR Total Nitrogen Reagent Set (50 vials) 27141-00
Includes: (1) 26718-46, (1) 26719-46, (1) 26720-46, *(50) Hydroxide Digestion Vials, *(50) Acid Solution Vials

Description	Quantity Required Per Test	Unit	Cat. No.
HR Total Nitrogen Hydroxide Digestion Vials	1 vial	50/pkg	*
Total Nitrogen Persulfate Reagent Powder Pillows	1 pillow	50/pkg	26718-46
Total Nitrogen Reagent A, Bisulfite Powder Pillows	1 pillow	50/pkg	26719-46
Total Nitrogen Reagent B, Indicator Powder Pillows	1 pillow	50/pkg	26720-46
Total Nitrogen Reagent C Vials, Acid Solution	1 vial	50/pkg	*

REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
COD/TNT Adapter	1	each	48464-00
Funnel, micro	1	each	25843-35
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips for 19700-01	2	50/pkg	21856-96
Test Tube Rack, for cooling vials	1-3	each	18641-00

OPTIONAL REAGENTS

Nitrogen, Ammonia, 1000 mg/L NH_3-N	1 L	23541-53
Nitrogen, Ammonia, Voluette Ampule, 1000 mg/L NH_3-N, 10 mL	16/pkg	23541-10
Sulfuric Acid, ACS	500 mL	979-49
Primary Standards for Kjeldahl Nitrogen	set of 3	22778-00
Ammonium p-Toluenesulfonate	25 g	22779-24
Glycine p-Toluenesulfonate	25 g	22780-24
Nicotinic Acid p-Toluenesulfonate	25 g	22781-24
Sodium Hydroxide Standard Solution, 5.0 N	50 mL MDB	2450-26
Wastewater Influent Standard, Inorganics (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49
Water, organic-free	500 mL	26415-49

* These items are not sold separately. Please order the complete set (Cat. No. 27141-00) as a replacement.

OPTIONAL APPARATUS

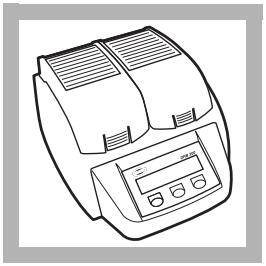
Description	Unit	Cat. No.
Ampule Breaker Kit	each	21968-00
Balance, analytical, 115 Vac	each	28014-01
Balance, analytical, 230 Vac	each	28014-02
Cots, finger	2/pkg	14647-02
Cylinder, graduated, mixing, 25 mL	3 each	26363-40
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm		LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm		LTV082.52.30001
Flask, volumetric, Class A, 1000 mL	3 each	14574-53
Flask, volumetric, Class A, 200 mL	each	14574-45
Pipet, volumetric, Class A, 25 mL	2 each	14515-40
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224. Out side the U.S.A— Contact the Hach office or distributor serving you.
Outside the U.S.A.—Contact the Hach office or distributor serving you.

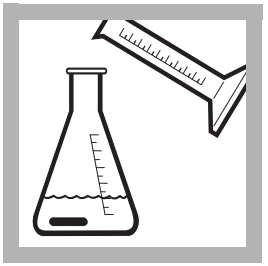
ORGANIC CARBON, TOTAL, Low Range (0.0–20.0 mg/L C)

Direct Method* **For water, drinking water, and wastewater**

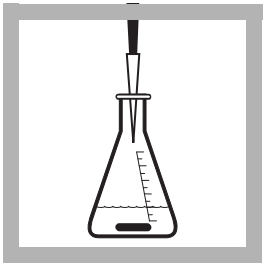


1. Turn on the DRB 200 reactor. Heat to 103-105 °C.

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*

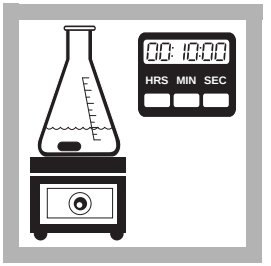


2. Use a graduated cylinder to add 10 mL of sample to a 50-mL erlenmeyer flask containing a stir bar.

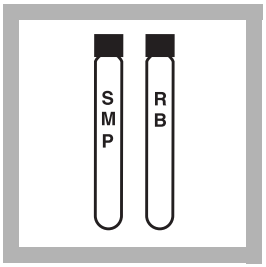


3. Add 0.4 mL of Buffer Solution, pH 2.0.

Note: *Use pH paper to make sure the sample pH is 2.*

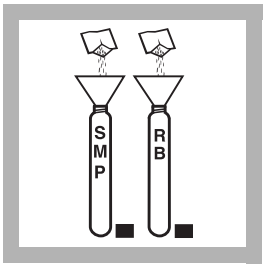


4. Place the flask on a stir plate and stir at a moderate speed for 10 minutes.

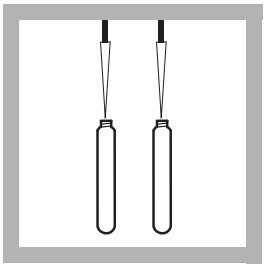


5. Label two Low Range Acid Digestion vials: **sample** and **reagent blank**.

Note: *A reagent blank is required for each series of samples.*



6. Using a funnel, add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).

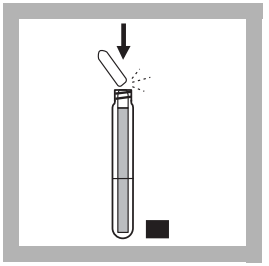


7. Use a TenSette® Pipet to add 3.0 mL of **organic-free water** to the **reagent blank** vial and 3.0 mL of **prepared sample** to the **sample** vial. Swirl to mix.

8. Rinse two blue Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

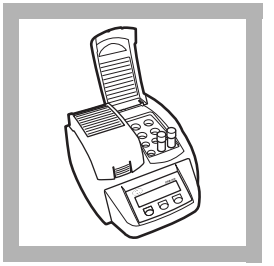
Note: Do not touch the ampules on the sides after wiping. Pick them up by the top.

* Patent pending

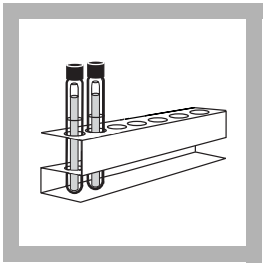


9. Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

Note: Do not invert or tilt the vial after inserting the ampule to prevent the Indicator Reagent from mixing with the contents of the acid digestion vial.

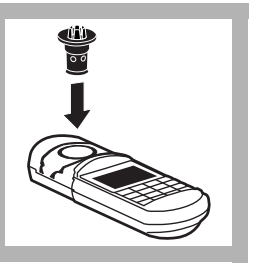


10. Cap the vial assemblies tightly and place them in the reactor for 2 hours at 103-105 °C.



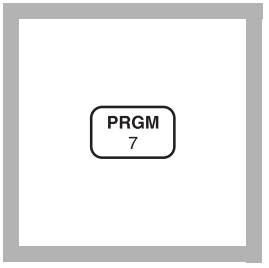
11. Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

Allow the vials to cool for **one hour** for accurate results.



12. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

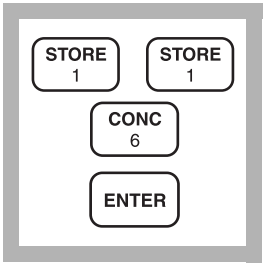
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



13. Enter the stored program number for Low Range TOC.

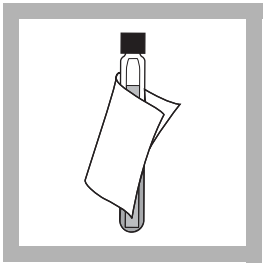
Press: **PRGM**

The display will show:
PRGM?



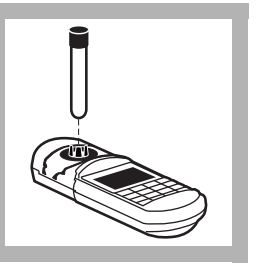
14. Press: **116 ENTER**

The display will show **mg/L** and the **ZERO** icon.



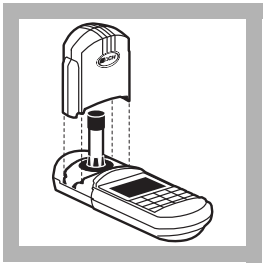
15. Wipe the reagent blank vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

Note: *The liquid in the reagent blank vial should be dark blue.*

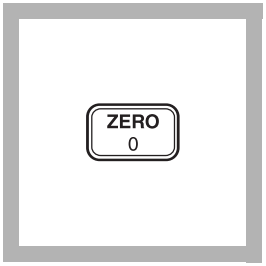


16. Place the **reagent blank** vial assembly in the adapter.

Push straight down on the top of the vial until it seats solidly in the adapter.



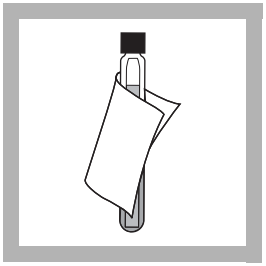
17. Tightly cover the vial assembly with the instrument cap.



18. Press: **ZERO**

The cursor will move to the right, then the display will show:

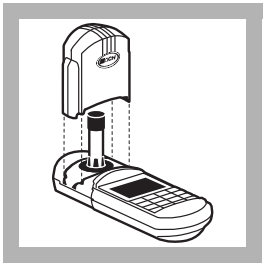
0.0 mg/L C



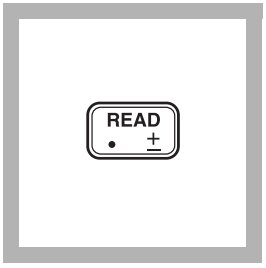
19. Wipe the sample vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

20. Place the **sample** vial assembly in the adapter.

Push straight down on the top of the vial assembly until it seats solidly in the adapter.



21. Tightly cover the vial assembly with the instrument cap.



22. Press: **READ**

The cursor will move to the right, then the result in mg/L C will be displayed.

Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle with minimum headspace before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

Accuracy Check

Standard Solutions Method

a. Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.

Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27915-05).

b. Prepare a 10.0 mg/L C standard by transferring 1.00 mL of the stock standard to a 100-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh daily.

Standard Additions Method

a. Prepare a 150 mg/L C standard by transferring 15.00 mL of 1000 mg/L C stock solution to a 100-mL Class A volumetric flask. Dilute to volume with organic-free water. Mix.

b. Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 150 mg/L C standard to each of three Acid Digestion vials.

c. Add the contents of one TOC Persulfate powder pillow to each vial.

d. Add 3.0 mL of sample to each vial. Swirl to mix.

e. Proceed with the procedure starting at *step 8*.

f. The mg/L C concentration should increase by 5.0 mg/L for each 0.1 mL increment.

Method Performance

Precision

In a single laboratory, using a standard solution of 9.0 mg/L C and one lot of reagents, a single operator obtained a standard deviation of ±0.5 mg/L C.

Estimated Detection Limit

The estimated detection limit for Method 10129 is 0.3 mg/L C.

Sensitivity

At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 0.2 mg/L C.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br^-
Bromine	25 mg/L Br_2
Calcium	2000 mg/L as $CaCO_3$
Chloride	500 mg/L
Chlorine	10 mg/L Cl_2
Chlorine Dioxide	6 mg/L ClO_2
Copper	10 mg/L
Cyanide	10 mg/L CN^-
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as $CaCO_3$
Manganese (VII)	1 mg/L
Monochloramine	14 mg/L NH_2Cl as Cl_2
Nitrite	500 mg/L NO_2^-
Ozone	2 mg/L O_3
Phosphate	3390 mg/L PO_4^{2-}

Table 1 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Silica	100 mg/L SiO_2
Sulfate	5000 mg/L SO_4^{2-}
Sulfide	20 mg/L S^{2-}
Sulfite	50 mg/L SO_3^{2-}
Zinc	5 mg/L

If the sample contains greater than 600 mg/L $CaCO_3$ alkalinity, lower the sample pH to less than 7 before testing by adding sulfuric acid solution.

Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 50 NTU have been tested without interference.

Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

REQUIRED REAGENTS

Description	Qty/Test	Unit	Cat. No.
Total Organic Carbon Direct Method Low Range Test 'N Tube Reagent Set	50	vials	27603-45
Includes:			
Acid Digestion Solution Vials, Low Range TOC	1	50/pkg	*
Buffer Solution, Sulfate	0.4 mL	25 mL	452-33
Funnel, micro	1	each	25843-35
Indicator Ampules, Low Range TOC	1	10/pkg	*
TOC Persulfate Powder Pillows	1	50/pkg	*
Water, organic-free**	3.0 mL	500 mL	26415-49

* These items are not sold separately.

** This item must be purchased separately.

REQUIRED APPARATUS

Description	Qty/Test	Unit	Cat. No.
Cylinder, graduated, 10-mL	1	each	508-38
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Flask, Erlenmeyer, 50-mL	1	each	505-41
Magnetic Stirrer, 115 V, 4" x 4"	1	each	28812-00
Test Tube Rack	1-3	each	18641-00
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet, TenSette®, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for 19700-01 TenSette® Pipet	2	50/pkg	21856-96
Pipet Tips, for 19700-10 TenSette® Pipet	2	50/pkg	21997-96
Stir Bar, Magnetic	1	each	45315-00
Wipes, Disposable, Kimwipes	1	280/pkg	20970-00

OPTIONAL REAGENTS

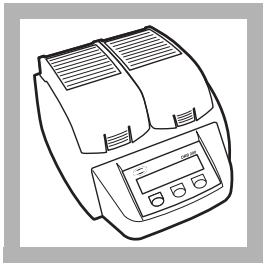
Potassium Acid Phthalate		500 g	315-34
Sulfuric Acid Reagent Solution, 5.25 N		100 mL MDB	2449-32
TOC Standard Solution Ampules (KHP Standard, 1000 mg/L C)		5/pkg	27915-05
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28332-49

OPTIONAL APPARATUS

Analytical Balance		each	28014-01
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm			LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm			LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm			LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm			LTV082.52.30001
Flask, volumetric, 100-mL		each	14574-42
Pipet, Class A, 200-mL		each	14515-35
Pipet, Class A, 15.00-mL		each	14515-39

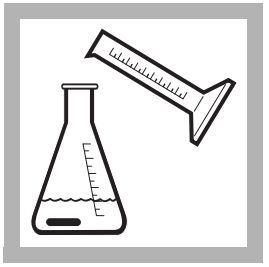
ORGANIC CARBON, TOTAL, Mid Range

Direct Method* **For wastewater and industrial waters**

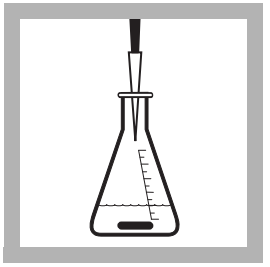


1. Turn on the DRB 200 reactor. Heat to 103–105 °C.

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*

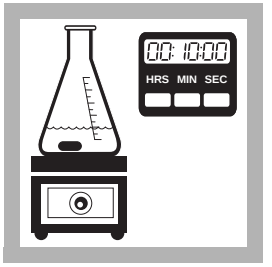


2. Use a graduated cylinder to add 10 mL of sample to a 50-mL erlenmeyer flask containing a stir bar.

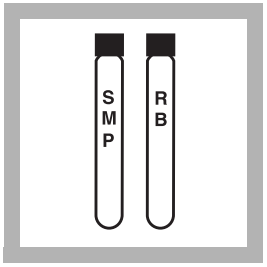


3. Add 0.4 mL of Buffer Solution, pH 2.0.

Note: *Use pH paper to make sure the sample pH is 2.*

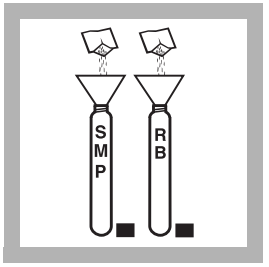


4. Place the flask on a stir plate and stir at a moderate speed for 10 minutes.

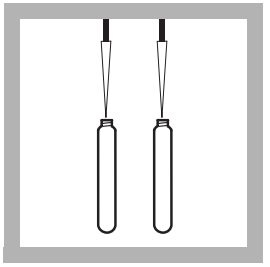


5. Label two Mid Range Acid Digestion vials: **sample** and **reagent blank**.

Note: *A reagent blank is required for each series of samples.*



6. Using a funnel, add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).

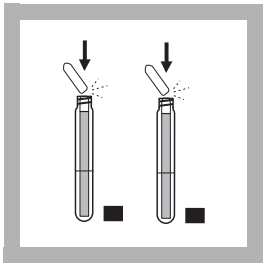


7. Use a TenSette® Pipet to add 1.0 mL of **organic-free water** to the **reagent blank** vial and1.0 mL of **prepared sample** to the **sample** vial. Do not cap the vial; swirl gently to mix.

8. Rinse two blue Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

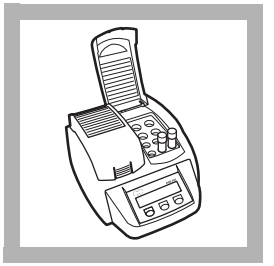
Note: Do not touch the ampules on the sides after wiping. Pick them up by the top.

* Patent pending

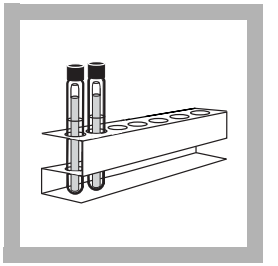


9. Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

Note: Do not invert or tilt the vial after inserting the ampule to prevent the Indicator Reagent from mixing with the contents of the acid digestion vial.

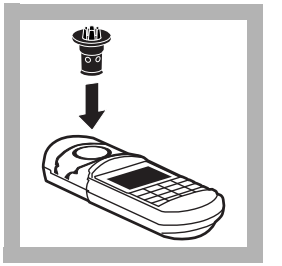


10. Cap the vial assemblies tightly and place them in the reactor for 2 hours at 103–105 °C.



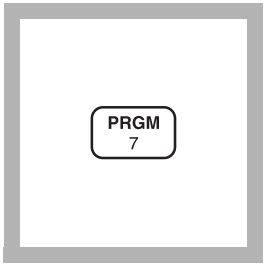
11. Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

Allow the vials to cool for **one hour** for accurate results.



12. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

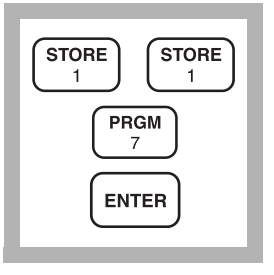
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



13. Enter the stored program number for Mid Range TOC.

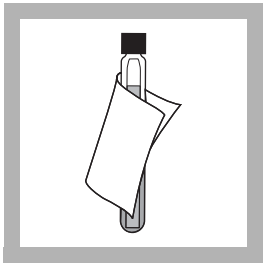
Press: **PRGM**

The display will show:
PRGM?



14. Press: **117 ENTER**

The display will show **mg/L** and the **ZERO** icon.

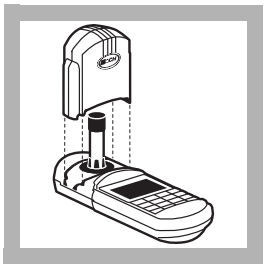


15. Wipe the reagent blank vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

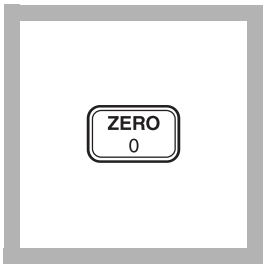
Note: *The liquid in the reagent blank vial should be dark blue.*

16. Place the **reagent blank** vial assembly in the adapter.

Push straight down on the top of the vial until it seats solidly in the adapter.



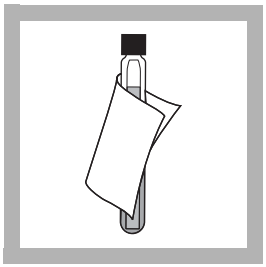
17. Tightly cover the vial assembly with the instrument cap.



18. Press: **ZERO**

The cursor will move to the right, then the display will show:

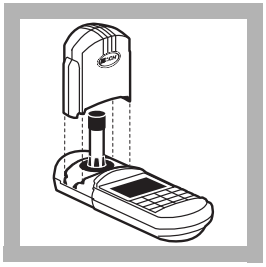
0 mg/L C



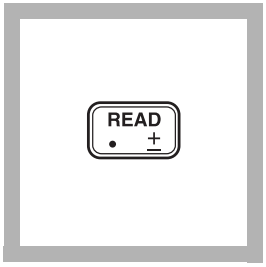
19. Wipe the sample vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

20. Place the **sample** vial assembly in the adapter.

Push straight down on the top of the vial assembly until it seats solidly in the adapter.



21. Tightly cover the vial assembly with the instrument cap.



22. Press: **READ**

The cursor will move to the right, then the result in mg/L C will be displayed.

Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle with minimum headspace before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

Accuracy Check

Standard Solutions Method

a. Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature. Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27915-05).

b. Prepare a 100 mg/L C standard by transferring 5.00 mL of the stock standard to a 50-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh weekly.

Standard Additions Method

a. Prepare a 300 mg/L C standard by transferring 15.00 mL of 1000 mg/L C stock solution to a 50-mL Class A volumetric flask. Dilute to volume with Organic-Free Water. Mix.

b. Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 300 mg/L C standard to each of three Acid Digestion vials.

c. Add the contents of one TOC Persulfate powder pillow to each vial.

d. Add 1.0 mL of sample to each vial. Swirl to mix.

e. Proceed with the procedure starting at *step 8*.

f. The mg/L C concentration should increase by 30 mg/L for each 0.1 mL increment.

Method Performance

Precision

mg/L C	95% Confidence Limits
15	± 5 mg/L C
50	± 6 mg/L
75	± 7 mg/L
115	± 4 mg/L
150	± 6 mg/L

Estimated Detection Limit

Use Method Number 10173 to test TOC levels below 15 mg/L C.

Sensitivity

At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 1.9 mg/L C.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br
Bromine	25 mg/L Br_2
Calcium	2000 mg/L as $CaCO_3$
Chloride	1500 mg/L
Chlorine	10 mg/L Cl_2
Chlorine Dioxide	6 mg/L ClO_2
Copper	10 mg/L
Cyanide	10 mg/L CN
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as $CaCO_3$
Manganese (VII)	1 mg/L

Table 1 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Monochloramine	14 mg/L NH_2Cl as Cl_2
Nitrite	500 mg/L NO_2^-
Ozone	2 mg/L O_3
Phosphate	3390 mg/L PO_4^{3-}
Silica	100 mg/L SiO_2
Sulfate	5000 mg/L SO_4^{2-}
Sulfide	20 mg/L S^{2-}
Sulfite	50 mg/L SO_3^{2-}
Zinc	5 mg/L

Note: *If the sample contains greater than 1000 mg/L $CaCO_3$ alkalinity, lower the sample pH to less than 7 before testing by adding sulfuric acid solution.*

Note: *Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 50 NTU have been tested without interference.*

Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

Instrument Setup

This procedure will add the current method as a new Hach program to your DR/850 or DR/890.

1. Turn the instrument on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key until the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.
6. Enter each of the numbers in the right column, followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time you may use the arrow keys to scroll back to review or change a number already entered.

Line Number	Entry	Line Number	Entry
1	117	29	0
2	42	30	0
3	72	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	66	36	0
9	36	37	0
10	92	38	0
11	40	39	0
12	195	40	0
13	89	41	0
14	74	42	0
15	61	43	0
16	0	44	165
17	0	45	128
18	0	46	0
19	0	47	10
20	67	48	0
21	0	49	0
22	0	50	0
23	0	51	0
24	0	52	0
25	0	53	0
26	0	54	25
27	0	55	0
28	0	56	255

REQUIRED REAGENTS

Total Organic Carbon Direct Method Mid Range
Test 'N Tube Reagent Set ..... 50 vials 28159-45

Includes:

Description	Quantity Required Per Test	Unit	Cat. No.
Acid Digestion Solution Vials, Mid Range TOC	1	50/pkg	*
Buffer Solution, Sulfate	0.4 mL	25 mL	452-33
Funnel, micro	1	each	25843-35
Indicator Ampules, Mid/High Range TOC	1	50/pkg	*
TOC Persulfate Powder Pillows	1	50/pkg	*
Water, organic-free**	1.0 mL	500 mL	26415-49

REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Cylinder, graduated, 10-mL	1	each	508-38
Flask, Erlenmeyer, 50-mL	1	each	505-41
Magnetic Stirrer, 115 V, 4" x 4"	1	each	28812-00
Test Tube Rack	1–3	each	18641-00
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette® Pipet	2	50/pkg	21856-96
Stir Bar, Magnetic	1	each	45315-00
Wipes, Disposable, Kimwipes	1	280/pkg	20970-00

OPTIONAL REAGENTS

Description	Per Test	Unit	Cat. No.
TOC Standard Solution (KHP Standard, 1000 mg/L C)		5/pkg	27915-05
Potassium Acid Phthalate		500 g	315-34
Sulfuric Acid Reagent Solution, 5.25 N		100 mL MDB	2449-32

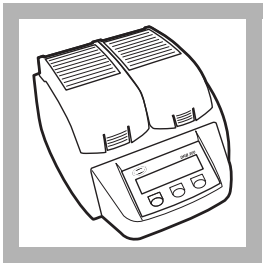
OPTIONAL APPARATUS

Analytical Balance		each	28014-01
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm			LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm			LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm			LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm			LTV082.52.30001
Flask, volumetric, 100-mL		each	14574-42
Pipet, Class A, 10.00-mL		each	14515-38
Pipet, Class A, 15.00-mL		each	14515-39
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28

* These items are not sold separately.
** This item must be purchased separately.

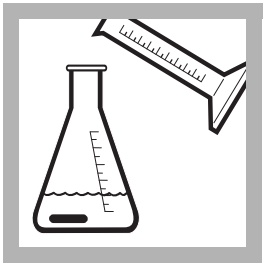
ORGANIC CARBON, TOTAL, High Range (20–700 mg/L C)

Direct Method* **For wastewater and industrial waters**

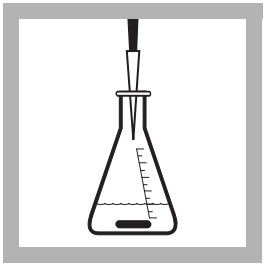


1. Turn on the DRB 200 reactor. Heat to 103-105 °C.

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*

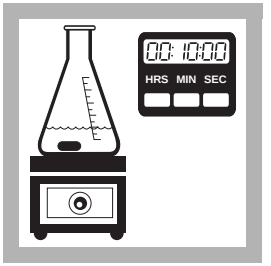


2. Use a graduated cylinder to add 10 mL of sample to a 50-mL erlenmeyer flask containing a stir bar.

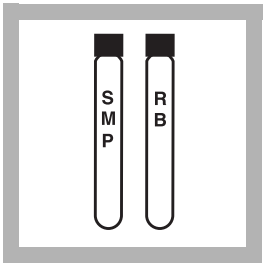


3. Add 0.4 mL of Buffer Solution, pH 2.0.

Note: *Use pH paper to make sure the sample pH is 2.*

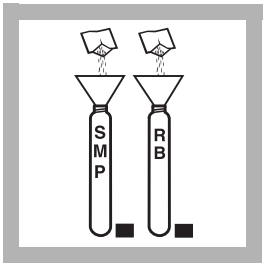


4. Place the flask on a stir plate and stir at a moderate speed for 10 minutes.

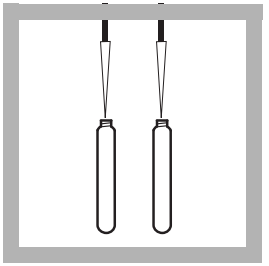


5. Label two High Range Acid Digestion vials: **sample** and **reagent blank**.

Note: *A reagent blank is required for each series of samples.*



6. Using a funnel, add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).

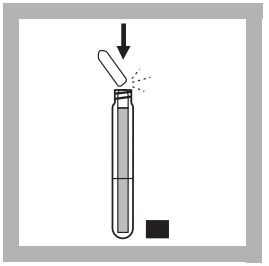


7. Use a TenSette® Pipet to add 0.3 mL of **organic-free water** to the **reagent blank** vial and 0.3 mL of **prepared sample** to the **sample** vial. Swirl to mix.

8. Rinse two blue Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

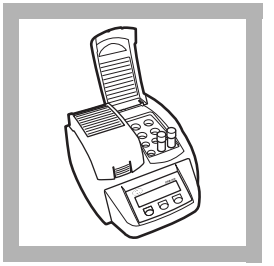
Note: Do not touch the ampules on the sides after wiping. Pick them up by the top.

* Patent pending

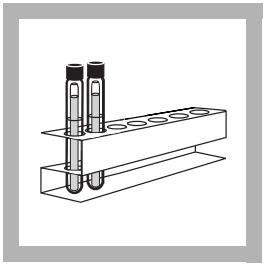


9. Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

Note: Do not invert or tilt the vial after inserting the ampule to prevent the Indicator Reagent from mixing with the contents of the acid digestion vial.

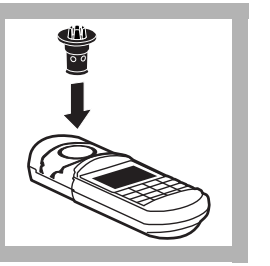


10. Cap the vial assemblies tightly and place them in the reactor for 2 hours at 103–105 °C.



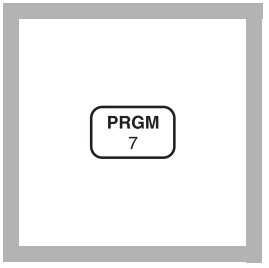
11. Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

Allow the vials to cool for **one hour** for accurate results.



12. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

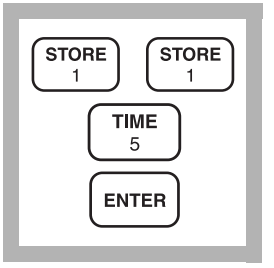
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



13. Enter the stored program number for High Range TOC.

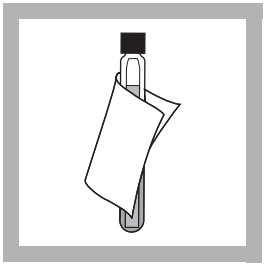
Press: **PRGM**

The display will show:
PRGM?



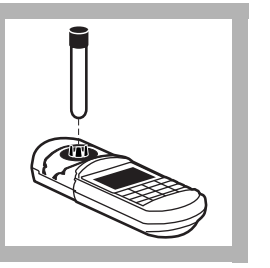
14. Press: **115 ENTER**

The display will show **mg/L** and the **ZERO** icon.



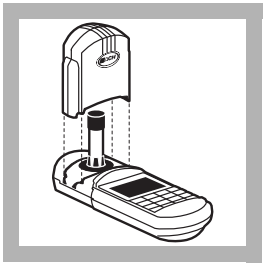
15. Wipe the reagent blank vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

Note: *The liquid in the reagent blank vial should be dark blue.*

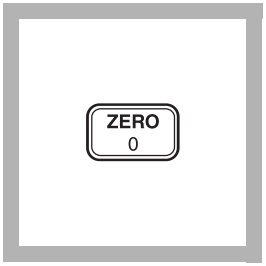


16. Place the **reagent blank** vial assembly in the adapter.

Push straight down on the top of the vial until it seats solidly in the adapter.



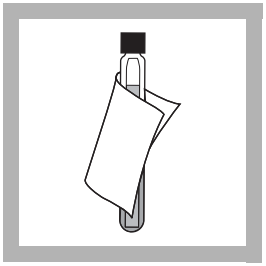
17. Tightly cover the vial assembly with the instrument cap.



18. Press: **ZERO**

The cursor will move to the right, then the display will show:

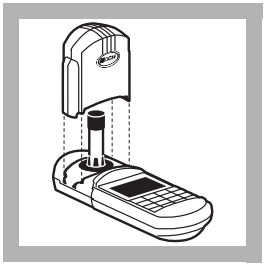
0 mg/L C



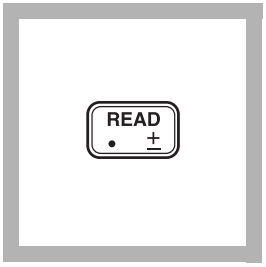
19. Wipe the sample vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

20. Place the **sample** vial assembly in the adapter.

Push straight down on the top of the vial assembly until it seats solidly in the adapter.



21. Tightly cover the vial assembly with the instrument cap.



22. Press: **READ**

The cursor will move to the right, then the result in mg/L C will be displayed.

Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle with minimum headspace before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

Accuracy Check

Standard Solutions Method

a. Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.
Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27915-05).

b. Prepare a 300 mg/L C standard by transferring 15.00 mL of the stock standard to a 50-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh weekly.

Standard Additions Method

a. Prepare a 300 mg/L C standard by transferring 18.00 mL of 1000 mg/L C stock solution to a 50-mL Class A volumetric flask. Dilute to volume with Organic-Free Water. Mix.

b. Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 300 mg/L C standard to each of three Acid Digestion vials.

c. Add the contents of one TOC Persulfate powder pillow to each vial.

d. Add 0.3 mL of sample to each vial. Swirl to mix.

e. Proceed with the procedure starting at *step 8*.

f. The mg/L C concentration should increase by 100 mg/L for each 0.1 mL increment.

Method Performance

Precision
In a single laboratory, using a standard solution of 360 mg/L C and one lot of reagents, a single operator obtained a standard deviation of ±8 mg/L C.

Estimated Detection Limit
Use Method Number 10129 to test TOC levels below 20 mg/L C.

Sensitivity
At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 6 mg/L C.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br
Bromine	25 mg/L Br_2
Calcium	2000 mg/L as $CaCO_3$
Chloride	5000 mg/L
Chlorine	10 mg/L Cl_2
Chlorine Dioxide	6 mg/L ClO_2
Copper	10 mg/L
Cyanide	10 mg/L CN
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as $CaCO_3$
Manganese (VII)	1 mg/L
Monochloramine	14 mg/L NH_2Cl as Cl_2
Nitrite	500 mg/L NO_2^-
Ozone	2 mg/L O_3
Phosphate	3390 mg/L PO_4^{3-}

Table 1 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Silica	100 mg/L SiO_2
Sulfate	5000 mg/L SO_4^{2-}
Sulfide	20 mg/L S^{2-}
Sulfite	50 mg/L SO_3^{2-}
Zinc	5 mg/L

If the sample contains greater than 600 mg/L $CaCO_3$ alkalinity, lower the sample pH to less than 7 before testing by adding sulfuric acid solution.

Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 900 NTU have been tested without interference.

Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

REQUIRED REAGENTS

Total Organic Carbon Direct Method High Range
Test 'N Tube Reagent Set ..... 50 vials 27604-45

Includes:

Description	Qty/Test	Unit	Cat. No.
Acid Digestion Solution Vials, High Range TOC	1	50/pkg	*
Buffer Solution, Sulfate	0.4 mL	25 mL	452-33
Funnel, micro	1	each	25843-35
Indicator Ampules, High Range TOC	1	10/pkg	*
TOC Persulfate Powder Pillows	1	50/pkg	*
Water, organic-free**	0.3 mL	500 mL	26415-49

* These items are not sold separately.

REQUIRED APPARATUS

Cylinder, graduated, 10-mL	1	each	508-38
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Flask, Erlenmeyer, 50-mL	1	each	505-41
Magnetic Stirrer, 115 V, 4" x 4"	1	each	28812-00
Safety Shield, laboratory bench	1	each	50030-00
Test Tube Rack	1-3	each	18641-00
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet, TenSette®, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for 19700-01 TenSette® Pipet	2	50/pkg	21856-96
Pipet Tips, for 19700-10 TenSette® Pipet	2	50/pkg	21997-96
Stir Bar, Magnetic	1	each	45315-00
Wipes, Disposable, Kimwipes	1	280/pkg	20970-00

OPTIONAL REAGENTS

Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules		16/pkg	28335-10
Potassium Acid Phthalate		500 g	315-34
Sulfuric Acid Reagent Solution, 5.25 N		100 mL MDB	2449-32
TOC Standard Solution Ampules (KHP Standard, 1000 mg/L C)		5/pkg	27915-05
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28331-49

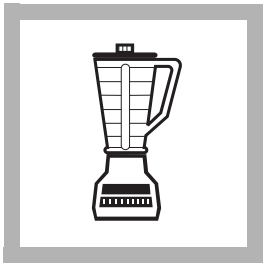
OPTIONAL APPARATUS

Analytical Balance		each	28014-01
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm			LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm			LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm			LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm			LTV082.52.30001
Flask, volumetric, 1000-mL		each	14574-53
Flask, volumetric, 100-mL		each	14574-42
Pipet, Class A, 10.00-mL		each	14515-38
Pipet, Class A, 15.00-mL		each	14515-39

** This item must be purchased separately.

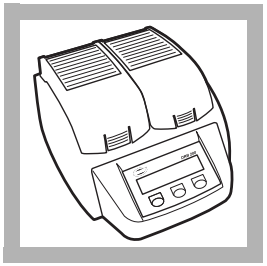
OXYGEN DEMAND, CHEMICAL

Reactor Digestion Method* USEPA approved for reporting wastewater analysis**
Digestion



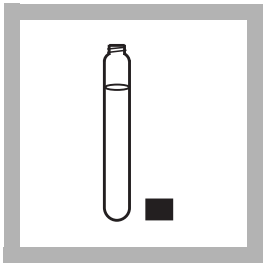
1. Homogenize 500 mL of sample for 2 minutes in a blender.

Note: For the 0-15,000 mg/L range, *homogenize 100 mL of sample. Pour the blended sample into a 250-mL beaker. Stir with a magnetic stirrer while withdrawing a sample aliquot. This improves accuracy and reproducibility.*



2. Turn on the DRB 200 Reactor. Preheat to 150 °C.

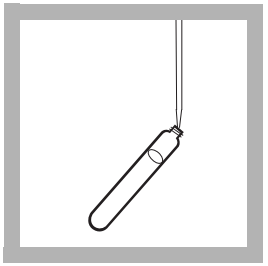
Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*



3. Remove the cap of a COD Digestion Reagent Vial for the appropriate range:

Sample Conc. Range (mg/L)	COD Digestion Reagent Vial Type
0 to 150	Low Range
0 to 1500	High Range
0 to 15,000	High Range Plus

Note: *The reagent mixture is light-sensitive. Keep unused vials in the opaque shipping container, in a refrigerator if possible. The light striking the vials during the test will not affect results.*



4. Hold the vial at a 45-degree angle. Pipet 2.00 mL (0.2 mL for the 0 to 15,000 mg/L range) of sample into the vial.

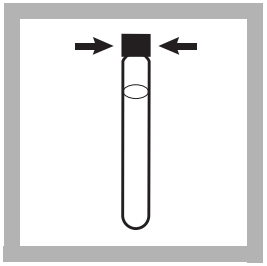
Note: For the 0-15,000 mg/L range, *pipet only 0.20 mL of sample, not 2.00 mL of sample, using a TenSette Pipet. For greater accuracy analyze a minimum of three replicates and average the results.*

Note: *Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Do not run tests with vials which have been spilled. If spills occur, wash with running water.*

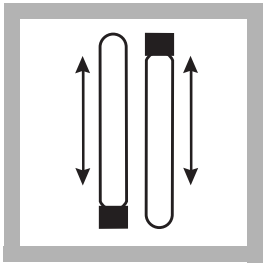
Caution: Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately or accidentally misused. Please read all warnings and the safety section of this manual. Wear appropriate eye protection and clothing. If contact occurs, flush the affected area with running water. Follow all instructions carefully.

* Jirka, A.M.; Carter, M.J. *Analytical Chemistry,* 1975, 47(8). 1397.

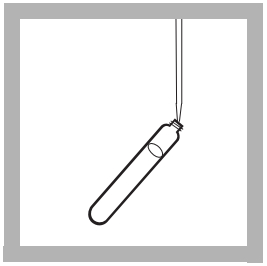
** *Federal Register,* April 21, 1980, 45(78), 26811-26812. The 0-15,000 mg/L range is **not** USEPA approved.



5. Replace the vial cap tightly. Rinse the outside of the COD vial with deionized water and wipe the vial clean with a paper towel.

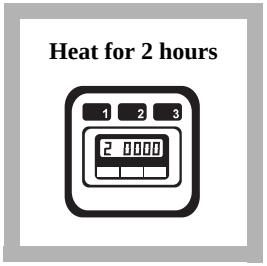


6. Hold the vial by the cap and over a sink. Invert gently several times to mix the contents. Place the vial in the preheated DRB 200 Reactor.
Note: *The vial will become very hot during mixing.*



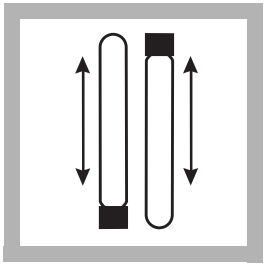
7. Prepare a blank by repeating Steps 3 to 6, substituting 2.00 mL (0.2 mL for the 0 to 15,000 mg/L range) deionized water for the sample.
Note: *Be sure the pipet is clean.*

Note: *One blank must be run with each set of samples. Run samples and blanks with vials from the same lot number (lot # is on the container label).*



8. Heat the vials for 2 hours.
Note: *Many samples are digested completely in less than two hours. If desired, measure the concentration (while still hot) at 15 minute intervals until the reading remains unchanged. Cool vials to room temperature for final measurement.*

9. Turn the reactor off. Wait about 20 minutes for the vials to cool to 120 °C or less.

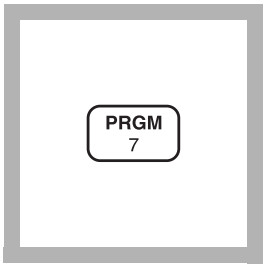


10. Invert each vial several times while still warm. Place the vials into a rack. Wait until the vials have cooled to room temperature.
Note: *If a pure green color appears in the reacted sample, measure the COD and, if necessary, repeat the test with a diluted sample.*

11. Use one of the following analytical techniques to measure the COD:

- Colorimetric method, 0-150 mg/L COD
- Colorimetric method, 0-1,500 mg/L COD
- Colorimetric method, 0-15,000 mg/L COD

Colorimetric Determination, 0 to 150 mg/L COD

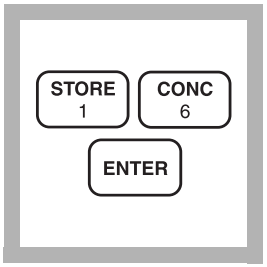


1. Enter the stored program number for chemical oxygen demand (COD), low range.

Press: **PRGM**

The display will show:

PRGM ?



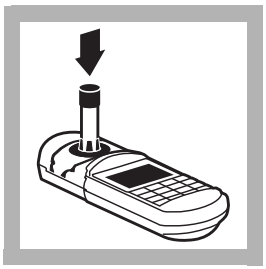
2. Press: **16 ENTER**

The display will show **mg/L, COD** and the **ZERO** icon**.**

Note: *For alternate form (O_2), press the* **CONC** *key.*

3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*

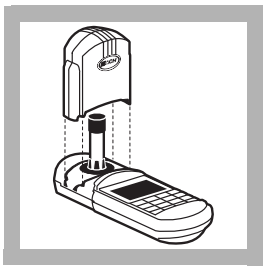
4. Clean the outside of the blank with a towel.
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



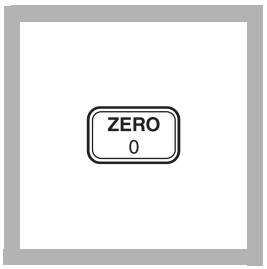
5. Place the blank in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



6. Tightly cover the vial with the instrument cap.
Note: *The blank is stable when stored in the dark. See Blanks for Colorimetric Determination following these procedures.*

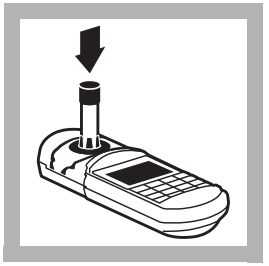


7. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L COD

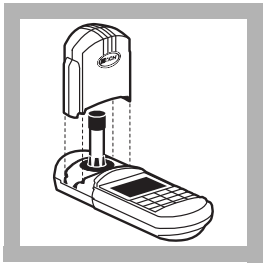
8. Clean the outside of the sample vial with a towel.



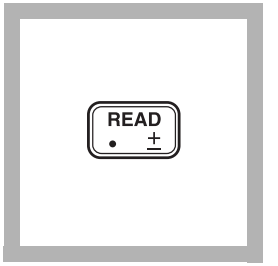
9. Place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



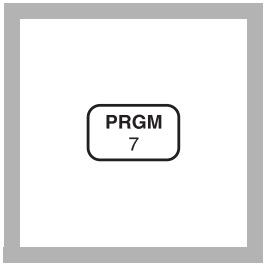
10. Tightly cover the vial with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

Colorimetric Determination, 0 to 1,500 and 0 to 15,000 mg/L COD

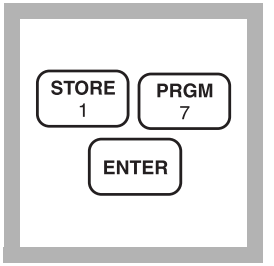


1. Enter the stored program number for chemical oxygen demand, high range.

Press: **PRGM**

The display will show:

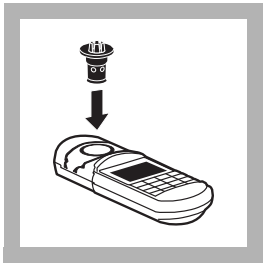
PRGM ?



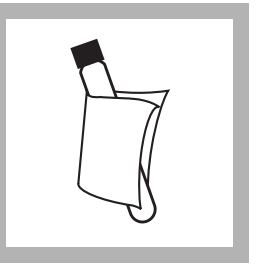
2. Press: **17 ENTER**

The display will show **mg/L, COD** and the **ZERO** icon**.**

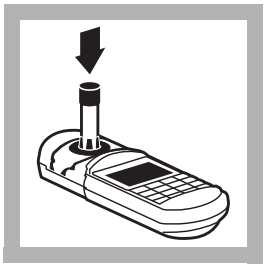
Note: *For alternate form (O_2), press the* **CONC** *key.*



3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.



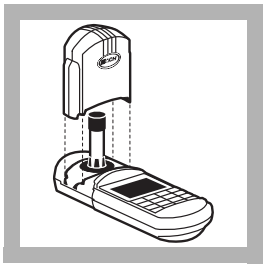
4. Clean the outside of the blank with a towel.
Note: *Wiping with a damp towel followed by a dry one will remove fingerprints or other marks.*



5. Place the blank in the adapter.

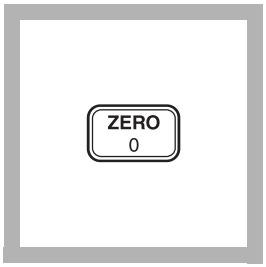
Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



6. Tightly cover the sample cell with the instrument cap.

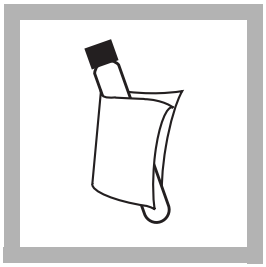
The blank is stable when stored in the dark. See Blanks for Colorimetric Determination following these procedures.



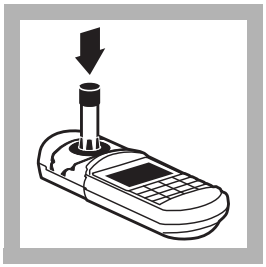
7. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L COD



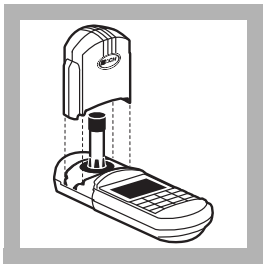
8. Clean the outside of the sample vial with a towel.



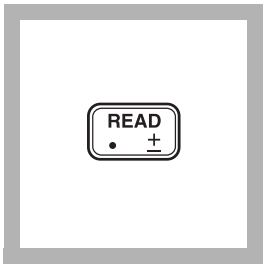
9. Place the sample in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



10. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

Note: *When using High Range Plus COD Digestion Reagent Vials, multiply the reading by 10.*

Note: *For most accurate results with samples near 1,500 or15,000 mg/L COD, repeat the analysis with a diluted sample.*

Sampling and Storage

Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days. Correct results for volume additions; see *Correction for Volume Additions* (Section 1) for more information.

Accuracy Check

Standard Solution Method

Check the accuracy of the 0 to 150 mg/L range with a 100 mg/L standard. Prepare by dissolving 85 mg of dried (120 °C, overnight) potassium acid phthalate (KHP) in 1 liter of deionized water. Use 2.0 mL as the sample volume. The expected result will be 100 mg/L COD. As an alternative, dilute 10 mL of 1000-mg/L COD Standard Solution to 100 mL to make a 100-mg/L standard.

Check the accuracy of the 0 to 1,500 mg/L range by using either a 300 mg/L or 1000 mg/L COD Standard Solution. Alternatively, prepare a 500 mg/L standard by dissolving 425 mg of dried (120 °C, overnight) KHP. Dilute to 1 liter with deionized water. Use 2.0 mL of one of these solutions as the sample volume.

Check the accuracy of the 0 to 15,000 mg/L range by using a 10,000 mg/L COD standard solution. Prepare the 10,000 mg/L solution by dissolving 8.500 g of dried (120 °C, overnight) KHP in 1 liter of deionized water. Use 0.2 mL of this solution as the sample volume; the expected result will be 10,000 mg/L COD.

Method Performance

Precision

Program #16: In a single laboratory, using a standard solution of 100 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±2 mg/L COD.

Program #17: In a single laboratory, using a standard solution of 1000 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ± 16 mg/L COD. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit (EDL)

The EDL for program 16 is 4 mg/L COD. The EDL for program 17 is 30 mg/L COD. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Alternate reagents

Mercury-free COD2 Reagents can provide a mercury-free testing option for non-reporting purposes. For process control applications, COD2 reagents will eliminate mercury waste and save on disposal costs. These reagents are fully compatible with test procedures and calibration curves programmed into the DR/2400 spectrophotometer. Determine chloride and ammonia for accurate results.

Note: *Mercury-free COD2 reagents are not approved for USEPA reporting. Request a copy of the COD Reagent Vial Information Brochure, Lit. No. 1356, for more information about specific applications.*

Interferences

Chloride is the primary interference when determining COD concentration. Each COD vial contains mercuric sulfate that will eliminate chloride interference up to the level specified in column 1 in *Table 1*. Samples with higher chloride concentrations should be diluted. Dilute the sample enough to reduce the chloride concentration to the level given in column 2.

If sample dilution will cause the COD concentration to be too low for accurate determination, add 0.50 g of mercuric sulfate ($HgSO_4$) to each COD vial before the sample is added. The additional mercuric sulfate will raise the maximum chloride concentration allowable to the level given in column 3.

Table 1

	Column 1	**Column 2**	**Column 3**
Vial Type Used	Maximum Cl^- concentration in sample (mg/L)	Maximum Cl^- concentration of diluted samples (mg/L)	Maximum Cl^- concentration in sample when 0.50 $HgSO_4$ added (mg/L)
Low Range	2000	1000	8000
High Range	2000	1000	4000
High Range Plus	20,000	10,000	40,000

Blanks for Colorimetric Determination

The blank may be used repeatedly for measurements using the same lot of vials. Store it in the dark. Monitor decomposition by measuring the absorbance at the appropriate wavelength (420 or 610 nm). Zero the instrument in the absorbance mode, using a vial containing 5 mL of deionized water and measure the absorbance of the blank. Record the value. Prepare a new blank when the absorbance has changed by about 0.01 absorbance units.

Summary of Method

The mg/L COD results are defined as the mg of O_2 consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion ($Cr_2O_7^{2-}$) to green chromic ion (Cr^{3+}). When the 0-150 mg/L colorimetric method is used, the amount of Cr^{6+} remaining is determined. When the 0-1,500 mg/L or 0-15,000 mg/L colorimetric method is used, the amount of Cr^{3+} produced is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex the chloride interference.

Pollution Prevention and Waste Management

Final samples will contain mercury (D009), silver (D011), and chromium (D007) at concentration levels regulated by the Federal RCRA. Please see *Section 3* for further information on proper disposal of these materials.

REQUIRED REAGENTS

Description	Qty/Test	Unit	Cat. No.
Select the appropriate COD Digestion Reagent Vial:			
Low Range, 0 to 150 mg/L COD	1 to 2 vials	25/pkg	21258-25
High Range, 0 to 1,500 mg/L COD	1 to 2 vials	25/pkg	21259-25
High Range Plus, 0 to 15,000 mg/L COD	1 to 2 vials	25/pkg	24159-25
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Blender, Osterizer, 120 V, 14 speed	1	each	26160-00
Blender, Osterizer, 240 V, 14 speed	1	each	26160-02
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001

REQUIRED APPARATUS (continued)

Description	Qty/Test	Unit	Cat. No.
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
COD/TNT Adapter	1	each	48464-00
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	1	50/pkg	21856-96
Pipet, volumetric, Class A, 2.00 mL	1	each	14515-36
Pipet Filler, safety bulb	1	each	14651-00
Test Tube Rack	1 to 2 racks	each	18641-00

ALTERNATE REAGENTS*

COD2, LR, 0 to 150 mg/L COD	1–2 vials	25/pkg	25650-25
COD2, HR, 0 to 1500 mg/L COD	1–2 vials	25/pkg	25651-25
COD2, HR, 0 to 1500 mg/L COD	1–2 vials	150/pkg	25651-15
COD2, HR, 0 to 15,000 mg/L COD	1–2 vials	25/pkg	28343-25

OPTIONAL REAGENTS

Description	Unit	Cat. No.
COD Digestion Reagent Vials, 0 to 150 mg/L COD	150/pkg	21258-15
COD Digestion Reagent Vials, 0 to 1,500 mg/L COD	150/pkg	21259-15
COD Standard Solution, 300 mg/L	200 mL	12186-29
COD Standard Solution, 1000 mg/L	200 mL	22539-29
Mercuric Sulfate	28.3 grams	1915-20
Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules	16/pkg	28335-10
Potassium Acid Phthalate, ACS	500 g	315-34
Potassium Dichromate Standard Solution, 0.25 N	1000 mL*	1809-53
Sulfuric Acid, ACS	500 mL**	979-49
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49

OPTIONAL APPARATUS

Balance, analytical, 115 V	each	28014-01
Balance, analytical, 230 V	each	28014-02
Beaker, 250 mL	each	500-46H
Cylinder, graduated, 5 mL	each	508-37
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm		LTV082.53.30001

* Mercury-free COD2 reagents are not approved for USEPA reporting. Request a copy of the COD Reagent Vial Information Brochure, Lit. No. 1356, for more information about specific applications.

** Contact Hach for larger sizes.

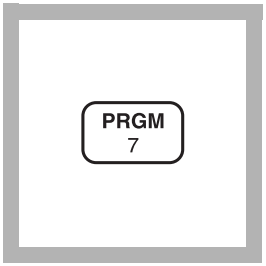
OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm		LTV082.52.30001
Electromagnetic Stirrer, 120 V, with electrode stand	each	45300-01
Electromagnetic Stirrer, 230 V, with electrode stand	each	45300-02
Flask, volumetric, Class A, 1000 mL	each	14574-53
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
Pipet, serological, 5 mL	each	532-37
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 10 mL	each	14515-38
Spoon, measuring, 0.5 g	each	907-00
Stir Bar, 22.2 x 4.76 mm (7/8" x 3/16")	each	45315-00
Stir Bar Retriever	each	15232-00
Timer	each	26304-00

For Technical Assistance, Price and Ordering
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

OXYGEN DEMAND, CHEMICAL (20 to 1,000 mg/L) For water and wastewater

Manganese III Digestion Method* (without chloride removal)



1. Enter the stored program number for Manganese III COD.

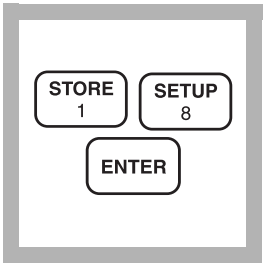
Press: **PRGM**

The display will show:

PRGM ?

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

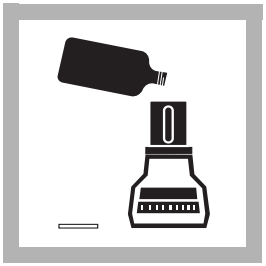
Note: *Preheat the COD Reactor to 150 °C for use later in the procedure.*



2. Press: **18 ENTER**

The display will show **mg/L, COD** and the **ZERO** icon**.**

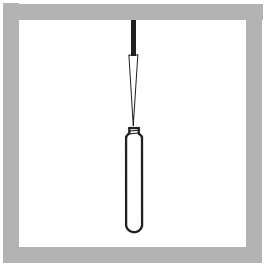
Note: *For alternate forms (O_2), press the* **CONC** *key.*



3. Homogenize 100 mL of sample for 30 seconds in a blender.

Note: *Blending promotes even distribution of solids and improves accuracy and reproducibility.*

Note: *Continue mixing the sample while pipetting if suspended solids are present.*



4. If chloride is not present in significant amounts†, pipet 0.50 mL of homogenized sample into a Mn III COD vial. Cap and invert several times to mix.

Note: *If the sample COD value is not between 20-1000 mg/L dilute the sample with deionized water to obtain a range of 20-1000 mg/L COD. Multiply the final result by the dilution factor.*

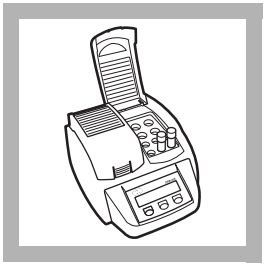
† To determine if chloride will interfere, run the sample with and without the chloride removal procedure and compare the results.

Caution: Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and the safety section of this manual. Wear appropriate eye protection and appropriate clothing. If contact occurs, flush the affected area with running water. Follow all instructions carefully.

* U.S. Patent 5,556,787

5. Prepare a blank (see note) by substituting 0.50 mL of deionized water for the sample. Continue with step 9 of this procedure.

Note: *The reagent blank is stable and can be reused. Verify reagent blank quality by measuring the absorbance of the blank vs. a clean COD vial filled with deionized water. The absorbance range should be about 1.36-1.43.*

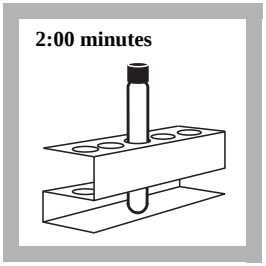


6. Place the vials in the DRB 200 Reactor that is preheated to 150 °C. Digest for 1 hour.

Note: *Boiling sample in the vials during digestion indicates the vial is not properly sealed; test results will be invalid.*

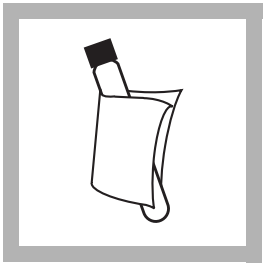
Note: *Samples can be digested up to 4 hours to oxidize more resistant organics. The prepared blank must be treated in the same manner.*

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*



7. Remove the vials and place them in a cooling rack for two minutes to air cool. Then cool the vials to room temperature in a cool water bath or running tap water. This usually takes about three minutes.

Note: *Occasionally a vial will develop a colorless upper layer and a purple lower layer. Invert the vial several times to mix and proceed. This will not affect test results.*

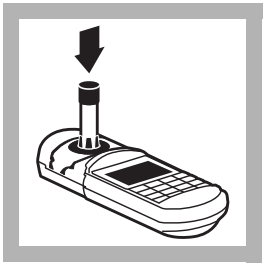


8. Remove the vials from the water and wipe with a clean, dry paper towel.

Invert the vials several times to mix.

9. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

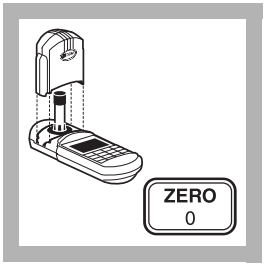
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



10. Place the blank in the sample cell adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



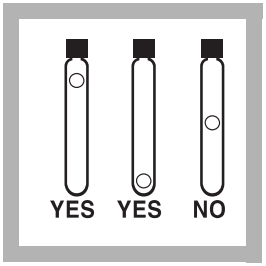
11. Tightly cover the sample cell with the instrument cap.

Note: *Clean the COD vial with a towel to remove fingerprints or other marks.*

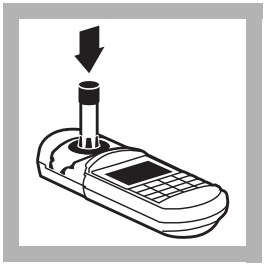
Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L COD



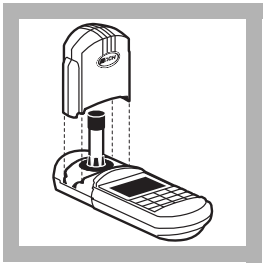
12. If the chloride removal was done, make sure the filter disc is not suspended in the middle of the vial; it can interfere with the instrument reading. Move it with gentle swirling or by lightly tapping the vial on the table top.



13. Place the sample in the adapter.

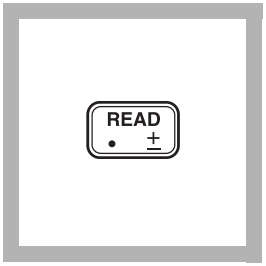
Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



14. Tightly cover the sample cell with the instrument cap.

Note: *Clean the COD vial with a towel to remove fingerprints or other marks.*



15. Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Note: *Adjust the result for any sample dilution in Steps 4 or 6.*

Sampling and Storage

Collect samples in clean glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with concentrated sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C may be stored up to 28 days. Correct results for volume additions; see *Correcting for Volume Additions* (*Section 1*) for more information.

Accuracy Check

Standard Solution Method

Prepare an 800 mg/L COD standard solution by adding 0.6808 g of
dried (103 °C, overnight) potassium acid phthalate (KHP) to 1 liter of deionized water. Use 0.50 mL of this solution (0.60 mL for the chloride removal procedure) as the sample volume. The result should be
800 ±26 mg/L COD.

An 800 mg/L COD solution can also be purchased directly from Hach (see *Optional Reagents*).

Method Performance (for Manganic III COD without the chloride removal procedure)

Precision

In a single laboratory, using a standard solution of 800 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±23 mg/L COD.

Estimated Detection Limit (EDL)

The EDL for program 18 is 14 mg/L COD. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Inorganic materials may also be oxidized by trivalent manganese and constitute a positive interference when present in significant amounts. Chloride is the most common interference and is removed by sample pretreatment with the Chloride Removal Cartridge. If chloride is known to be absent or present in insignificant levels, the pretreatment can be omitted. A simple way to determine if chloride will affect test results is to run

routine samples with and without the chloride removal, then compare results. Other inorganic interferences (i.e., nitrite, ferrous iron, sulfide) are not usually present in significant amounts. If necessary, these interferences can be corrected for after determining their concentrations with separate methods and adjusting the final COD test
results accordingly.

Ammonia nitrogen is known to interfere in the presence of chloride; it does not interfere if chloride is absent.

Summary of Method

Chemical oxygen demand (COD) is defined as "... a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant" (APHA Standard Methods, 19th ed., 1995). Trivalent manganese is a strong, non-carcinogenic chemical oxidant that changes quantitatively from purple to colorless when it reacts with organic matter. It typically oxidizes about 80% of the organic compounds. Studies have shown that the reactions are highly reproducible and test results correlate closely to Biochemical Oxygen Demand (BOD) values and hexavalent chromium COD tests. None of the oxygen demand tests provide 100% oxidation of all
organic compounds.

A calibration is provided which is based on the oxidation of Potassium Acid Phthalate (KHP). A different response may be seen in analyzing various wastewaters. The KHP calibration is adequate for most applications. The highest degree of accuracy is obtained when test results are correlated to a standard reference method such as BOD or one of the chromium COD methods. Special waste streams or classes will require a separate calibration to obtain a direct mg/L COD reading or to generate a correction factor for the precalibrated KHP response. The sample digestion time can be extended up to 4 hours for samples which are difficult to oxidize.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Manganese III COD Reagent Vials, 20-1000 mg/L	1	25/pkg	26234-25
Sulfuric Acid, concentrated	1 mL	4 Kg	979-09
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Adapter, COD/TNT	1	each	48464-00
Blender, Osterizer, 120 Vac, 14-speed	1	each	26747-00
Blender Container, 118 mL	1	2/pkg	26748-00
Cap, with inert Teflon liner, for mixing bottle	varies	12/pkg	24018-12
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Forceps, extra fine point	1	each	26696-00
Mixing Bottle, glass, for sample + acid	1	each	24276-06
Pipet, TenSette, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette	2	250/pkg	21997-25
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette	2	1000/pkg	21856-28
Test Tube Rack, stainless steel	1	each	18641-00

OPTIONAL REAGENTS

COD Standard Solution, 800 mg/L COD		200 mL	26726-29
Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules		16/pkg	28335-10
Potassium Acid Phthalate		500 g	315-34
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28332-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28331-49

OPTIONAL APPARATUS

Dispenser for sulfuric acid		each	25631-37
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm			LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm			LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm			LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm			LTV082.52.30001

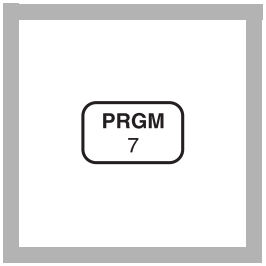
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

OXYGEN DEMAND, CHEMICAL (20 to 1,000 mg/L) For water and wastewater

Manganese III Digestion Method* (with chloride removal)



1. Enter the stored program number for Manganese III COD.

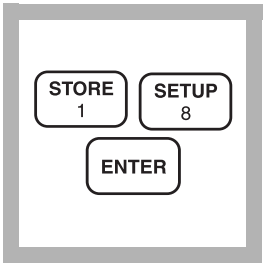
Press: **PRGM**

The display will show:

PRGM ?

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

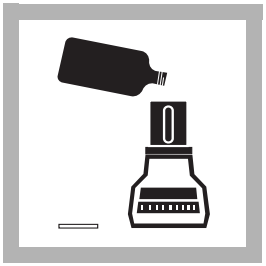
Note: *Preheat the COD Reactor to 150 °C for use later in the procedure.*



2. Press: **18 ENTER**

The display will show **mg/L, COD** and the **ZERO** icon**.**

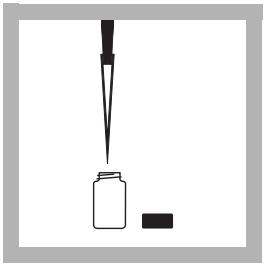
Note: *For alternate forms (O_2), press the* **CONC** *key.*



3. Homogenize 100 mL of sample for 30 seconds in a blender.

Note: *Blending promotes even distribution of solids and improves accuracy and reproducibility.*

Note: *Continue mixing the sample while pipetting if suspended solids are present.*



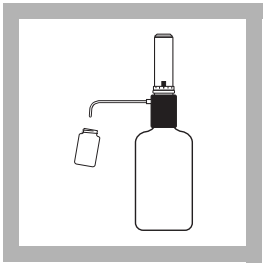
Chloride Removal Procedure

4. Using a TenSette Pipet or a pipet and safety bulb, pipet 9.0 mL of homogenized sample into an empty glass mixing cell. If the sample COD exceeds 1000 mg/L, dilute the sample as described in *Table 1*.

Note: *If suspended solids are present, continue mixing the sample while pipetting.*

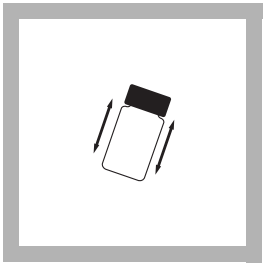
Caution: Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and the safety section of this manual. Wear appropriate eye protection and appropriate clothing. If contact occurs, flush the affected area with running water. Follow all instructions carefully.

* U.S. Patent 5,556,787



5. Using an automatic dispenser or TenSette Pipet, add 1.0 mL of concentrated sulfuric acid to the mixing cell.

Note: *Mixing concentrated sulfuric acid and water is not additive. Adding 1.0 mL of concentrated sulfuric acid to 9.0 mL of sample does not result in a final volume of 10.0 mL. This factor is built into the calibration curve.*



6. Cap the cell tightly and invert it several times. The solution will become hot. Cool to room temperature before proceeding.

Note: *Acidified samples are stable for several months when refrigerated at 4 °C.*

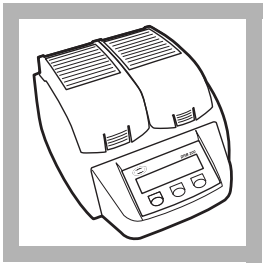
PREPARE BLANK

7. Prepare a blank (see note) by repeating Steps 4-6, substituting 9.0 mL of deionized water for the sample.

Note: *The reagent blank is stable and can be reused. Verify reagent blank quality by measuring the absorbance of the blank vs. a clean COD vial filled with deionized water. The absorbance range, when using chloride removal, should be about 1.31-1.36.*

Note: *Use a clean pipet or rinse it thoroughly.*

Note: *One blank must be run with each lot of reagents. Run all samples and blanks with the same lot of vials (lot number is on the container label).*



8. If not already on, turn on the DRB 200 Reactor and heat to 150 °C.

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*

Table 1 Dilution Table (for use with Chloride Removal Procedure Only)

Sample (mL)	Deionized Water (mL)	Range (mg/L COD)	Multiplication Factor
6.0	3.0	30-1500	1.5
3.0	6.0	60-3000	3
1.0	8.0	180-9000	9
0.5	8.5	360-18000	18

All dilutions require that the ratio of sample to sulfuric acid remain at 9:1. For other dilutions that are not listed in Table 1, simply add the sample volume + deionized water and divide by the sample volume to obtain the multiplication factor.

Example:

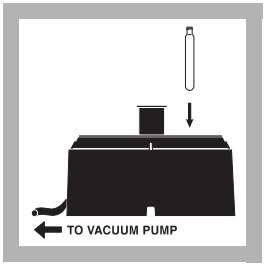
Dilute the sample to a range of 90-4500 mg/L COD

Sample Volume (2.0 mL) + Deionized water (7.0 mL) = Total Volume (9.0 mL)

$$\text{Multiplication Factor} = \frac{\text{Total Volume}}{\text{Sample Volume}} = \frac{9.0\ \text{mL}}{2.0\ \text{mL}} = 4.5$$

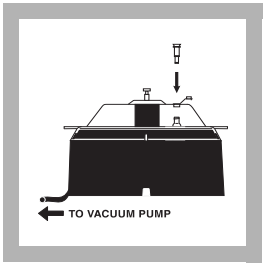
Standard test range is 20-1000 mg/L COD. Example Test Range = 4.5 (20) to 4.5 (1000) = 90-4500 mg/L COD

It is best to use 0.5 mL or more of sample for diluting. If sample values exceed 18,000 mg/L COD, use a separate sample dilution before the sample chloride removal procedure.

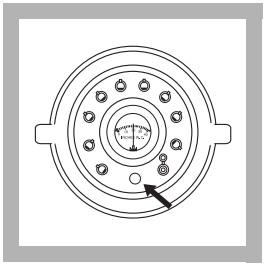


9. Label each Mn III COD vial and remove the cap. Place the vial in one of the numbered holes in the Vacuum Pretreatment Device (VPD)* base.

Note: *The VPD must be attached to a vacuum pump (not an aspirator-type vacuum) that can create a vacuum of 20 to 25 inches of mercury.*

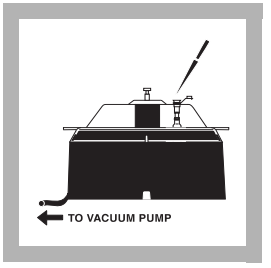


10. Place the VPD top on the base. Insert a fresh Chloride Removal Cartridge (CRC)** directly above each Mn III COD Reagent Vial. Plug any open holes in the VPD top using the stoppers provided.



11. Turn the vacuum pump on and adjust the vacuum regulator valve on top of the VPD until the internal gauge reads 20 inches of water.

Note: *The optimum setting allows the sample to flow through the CRC in about 30 to 45 seconds.*

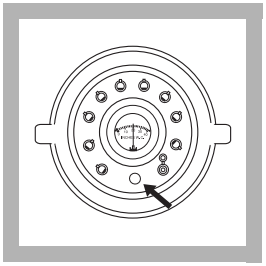


12. Pipet 0.60 mL of acidified sample (made in Steps 4-6) into the CRC. Pipet 0.60 mL of acidified blank into another CRC.

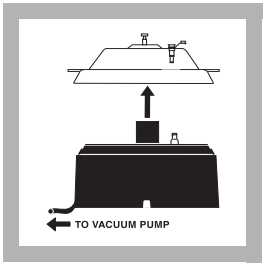
Note: *If the sample does not flow through the CRC, increase the vacuum until flow starts, then reduce the vacuum to 20 inches of water. Proceed as usual.*

* Patent Pending.

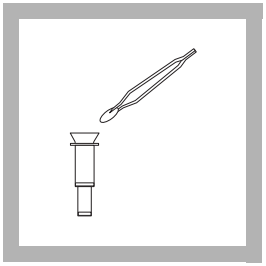
** U.S. patents 5,667,754 and 5,683,914.



13. Close the vacuum regulator valve completely to achieve full vacuum. After one minute under full vacuum, slide the VPD back and forth several times to dislodge any drops clinging to the cartridge.



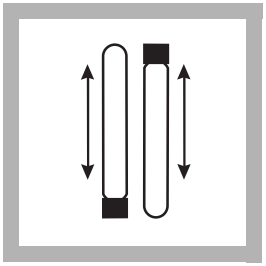
14. Open the VPD regulator valve to release the vacuum. Turn the pump off. Remove the VPD top and set it beside the base.



15. Use forceps to remove the filter from the top of each CRC. Place each filter in the corresponding Mn III COD Vial (use the numbers on the VPD as a guide).

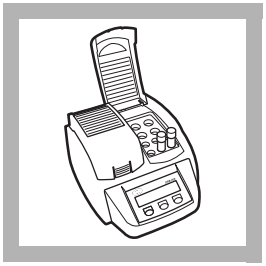
Note: *If the sample does not contain suspended solids, it is not necessary to transfer the filter to the digestion vial.*

Note: *To avoid cross contamination, clean forceps tips between samples by wiping with a clean towel or rinsing with deionized water.*



16. Remove the Mn III COD vial from the vacuum chamber and replace the original cap. Screw the cap on tightly. Invert several times to mix.

Note: *Dispose of the used Chloride Removal Cartridge. Do not reuse it.*

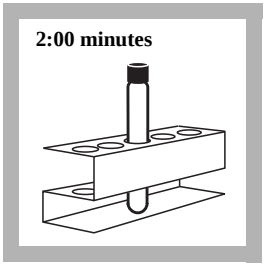


17. Place the vials in the DRB 200 Reactor that is preheated to 150 °C. Digest for 1 hour.

Note: *Boiling sample in the vials during digestion indicates the vial is not properly sealed; test results will be invalid.*

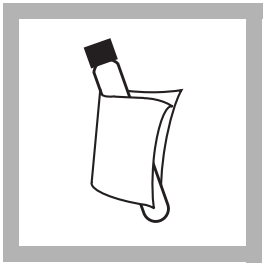
Note: *Samples can be digested up to 4 hours to oxidize more resistant organics. The prepared blank must be treated in the same manner.*

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*



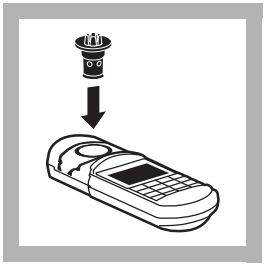
18. Remove the vials and place them in a cooling rack for two minutes to air cool. Then cool the vials to room temperature in a cool water bath or running tap water. This usually takes about three minutes.

Note: *Occasionally a vial will develop a colorless upper layer and a purple lower layer. Invert the vial several times to mix and proceed. This will not affect test results.*



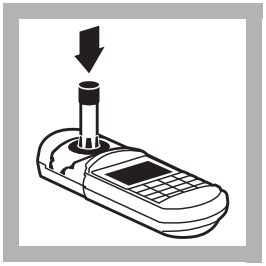
19. Remove the vials from the water and wipe with a clean, dry paper towel.

Invert the vials several times to mix.



20. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

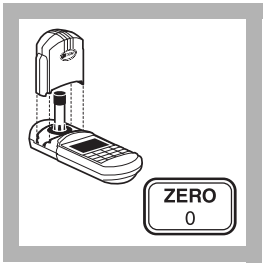
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



21. Place the blank in the sample cell adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



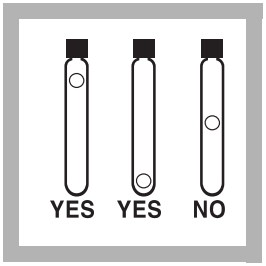
22. Tightly cover the sample cell with the instrument cap.

Note: *Clean the COD vial with a towel to remove fingerprints or other marks.*

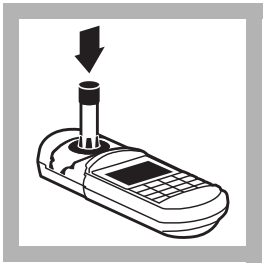
Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L COD



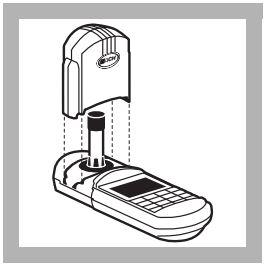
23. If the chloride removal was done, make sure the filter disc is not suspended in the middle of the vial; it can interfere with the instrument reading. Move it with gentle swirling or by lightly tapping the vial on the table top.



24. Place the sample in the adapter.

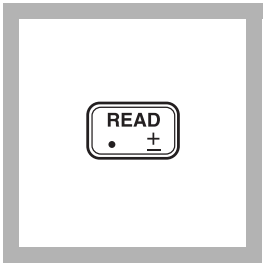
Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



25. Tightly cover the sample cell with the instrument cap.

Note: *Clean the COD vial with a towel to remove fingerprints or other marks.*



26. Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Note: *Adjust the result for any sample dilution.*

Sampling and Storage

Collect samples in clean glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with concentrated sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C may be stored up to 28 days. Correct results for volume additions; see *Correcting for Volume Additions* (*Section 1*) for more information.

Accuracy Check

Standard Solution Method

Prepare an 800 mg/L COD standard solution by adding 0.6808 g of
dried (103 °C, overnight) potassium acid phthalate (KHP) to 1 liter of deionized water. Use 0.50 mL of this solution (0.60 mL for the chloride removal procedure) as the sample volume. The result should be
800 ±26 mg/L COD.

An 800 mg/L COD solution can also be purchased directly from Hach (see *Optional Reagents*).

Method Performance (for Manganic III COD without the chloride removal procedure)

Precision

In a single laboratory, using a standard solution of 800 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±23 mg/L COD.

Estimated Detection Limit (EDL)

The EDL for program 18 is 14 mg/L COD. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Inorganic materials may also be oxidized by trivalent manganese and constitute a positive interference when present in significant amounts. Chloride is the most common interference and is removed by sample pretreatment with the Chloride Removal Cartridge. If chloride is known to be absent or present in insignificant levels, the pretreatment can be omitted. A simple way to determine if chloride will affect test results is to run

routine samples with and without the chloride removal, then compare results. Other inorganic interferences (i.e., nitrite, ferrous iron, sulfide) are not usually present in significant amounts. If necessary, these interferences can be corrected for after determining their concentrations with separate methods and adjusting the final COD test
results accordingly.

Ammonia nitrogen is known to interfere in the presence of chloride; it does not interfere if chloride is absent.

Summary of Method

Chemical oxygen demand (COD) is defined as "... a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant" (APHA Standard Methods, 19th ed., 1995). Trivalent manganese is a strong, non-carcinogenic chemical oxidant that changes quantitatively from purple to colorless when it reacts with organic matter. It typically oxidizes about 80% of the organic compounds. Studies have shown that the reactions are highly reproducible and test results correlate closely to Biochemical Oxygen Demand (BOD) values and hexavalent chromium COD tests. None of the oxygen demand tests provide 100% oxidation of all
organic compounds.

A calibration is provided which is based on the oxidation of Potassium Acid Phthalate (KHP). A different response may be seen in analyzing various wastewaters. The KHP calibration is adequate for most applications. The highest degree of accuracy is obtained when test results are correlated to a standard reference method such as BOD or one of the chromium COD methods. Special waste streams or classes will require a separate calibration to obtain a direct mg/L COD reading or to generate a correction factor for the precalibrated KHP response. The sample digestion time can be extended up to 4 hours for samples which are difficult to oxidize.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Chloride Removal Cartridges (CRC)	1	25/pkg	26618-25
Manganese III COD Reagent Vials, 20-1000 mg/L	1	25/pkg	26234-25
Sulfuric Acid, concentrated	1 mL	4 Kg	979-09
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Adapter, COD/TNT	1	each	48464-00
Blender, Osterizer, 120 Vac, 14-speed	1	each	26747-00
Blender Container, 118 mL	1	2/pkg	26748-00
Cap, with inert Teflon liner, for mixing bottle	varies	12/pkg	24018-12
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Forceps, extra fine point	1	each	26696-00
Mixing Bottle, glass, for sample + acid	1	each	24276-06
Pipet, TenSette, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette	2	250/pkg	21997-25
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette	2	1000/pkg	21856-28
Test Tube Rack, stainless steel	1	each	18641-00
Vacuum Pretreatment Device (VPD)	1	each	49000-00
Vacuum Pump, 115 V	1	each	14697-00
Vacuum Pump, 230V	1	each	14697-02

OPTIONAL REAGENTS

COD Standard Solution, 800 mg/L COD	200 mL		26726-29
Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules	16/pkg		28335-10
Potassium Acid Phthalate	500 g		315-34
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL		28332-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL		28331-49

OPTIONAL APPARATUS

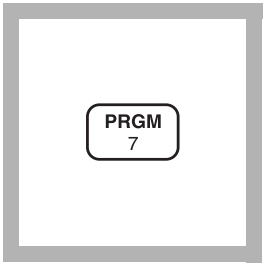
Dispenser for sulfuric acid		each	25631-37
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm			LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm			LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm			LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm			LTV082.52.30001

For Technical Assistance, Price and Ordering
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

OXYGEN, DISSOLVED, High Range (0 to 15.0 mg/L O_2)

HRDO Method

For water and wastewater

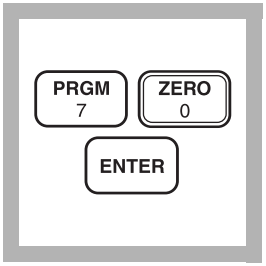


1. Enter the stored program number for dissolved oxygen, high range.

Press: **PRGM**

The display will show:

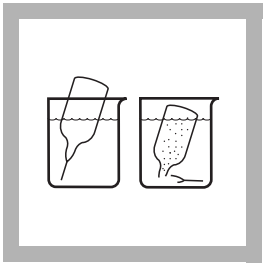
PRGM ?



2. Press: **70 ENTER**

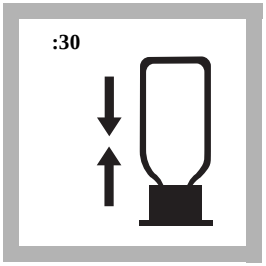
The display will show **mg/L, O2** and the **ZERO** icon.

3. Fill a sample cell (the blank) with at least 10 mL of sample. Fill a blue ampul cap with sample. Collect at least 40 mL of sample in a 50-mL beaker.



4. Fill a High Range Dissolved Oxygen AccuVac Ampul with sample.

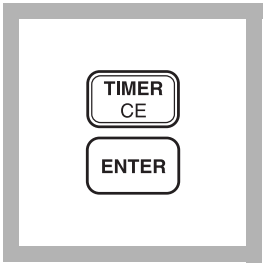
Note: *Keep the tip immersed while the ampul fills completely.*



5. Without inverting the ampul, immediately place the ampul cap that has been filled with sample securely over the tip of the ampul. Shake for about 30 seconds.

Note: *Accuracy is not affected by undissolved powder.*

Note: *The cap prevents contamination with atmospheric oxygen.*

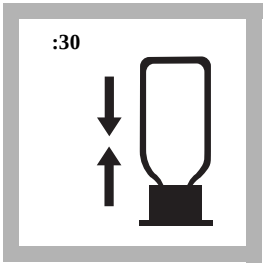


6. Press:

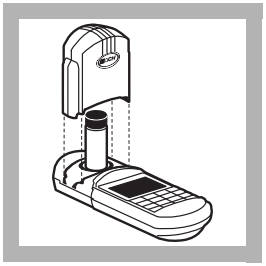
TIMER ENTER

A 2-minute reaction period will begin.

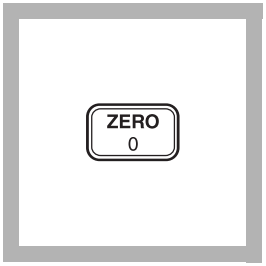
Note: *The two-minute period allows oxygen which was degassed during aspiration to redissolve in the sample and react.*



7. When the timer beeps, shake the ampul for 30 seconds.



8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

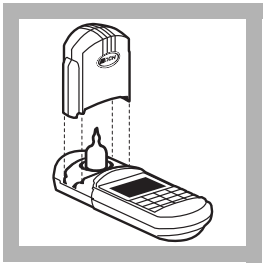


9. Press: **ZERO**

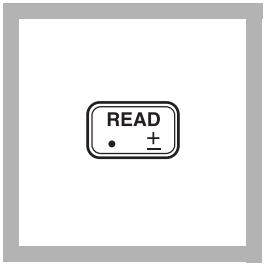
The cursor will move to the right, then the display will show:

0.0 mg/L O2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



10. Place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap. Wait approximately 30 seconds for the air bubbles to disperse from the light path.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L O_2 will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

The main consideration in sampling with the High Range Dissolved Oxygen AccuVac Ampul is to prevent the sample from becoming contaminated with atmospheric oxygen. This is accomplished by capping the ampul with an ampul cap in the interval between breaking open the ampul and reading the absorbance. If the ampul is securely capped, it should be safe from contamination for several hours. The absorbance will decrease by approximately 3% during the first hour and will not change significantly afterwards.

Sampling and sample handling are important considerations in obtaining meaningful results. The dissolved oxygen content of the water being tested can be expected to change with depth, turbulence, temperature, sludge deposits, light, microbial action, mixing, travel time and other factors. A single dissolved oxygen test rarely reflects the accurate over-all condition of a body of water. Several samples taken at different times, locations and depths are recommended for most reliable results. Samples must be tested immediately upon collection although only a small error results if the absorbance reading is taken several hours later.

Accuracy Check

The results of this procedure may be compared with the results of a dissolved oxygen meter (Cat. No. 51815-01).

Method Performance

Precision

In a single laboratory, using a standard solution of 8.0 mg/L O_2 determined by the Winkler method and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of
±0.41 mg/L O_2.

Estimated Detection Limit

The estimated detection limit for program 70 is 0.10 mg/L O_2. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Cr^{3+}	Greater than 10 mg/L
Cu^{2+}	Greater than 10 mg/L
Fe^{2+}	Greater than 10 mg/L
Mg^{2+}	Magnesium is commonly present in seawater and causes a negative interference. If the sample contains more than 50% seawater, the oxygen concentration obtained by this method will be 25% less than the true oxygen concentration. If the sample contains less than 50% seawater, the interference will be less than 5%.
Mn^{2+}	Greater than 10 mg/L
Ni^{2+}	Greater than 10 mg/L
NO_2^-	Greater than 10 mg/L

Summary of Method

The High Range Dissolved Oxygen AccuVac Ampul contains reagent vacuum sealed in a 12-mL ampul. When the AccuVac ampul is broken open in a sample containing dissolved oxygen, a yellow color forms, which turns purple as the oxygen reacts with the reagent. The color developed is proportional to the concentration of dissolved oxygen.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
High Range Dissolved Oxygen AccuVac Ampuls, with 2 reusable ampul caps	1 ampul	25/pkg	25150-25

REQUIRED APPARATUS

Beaker, 50 mL	1	each	500-41H
Caps, ampul, blue	varies	25/pkg	1731-25
Sample Cell, 10-20-25 mL, w/ cap	1	6/pkg	24019-06

OPTIONAL REAGENTS AND APPARATUS

AccuVac Dissolved Oxygen Sampler	each	24051-00
AccuVac Snapper Kit	each	24052-00
AccuVac Drainer	each	41036-00
BOD bottle and stopper, 300 mL	each	621-00
Dissolved Oxygen Meter, Portable HQ 10	each	51815-01
Dissolved Oxygen Reagent Set (Buret Method)	100 tests	23514-00
Dissolved Oxygen Reagent Set (Digital Titrator Method)	50 tests	22722-00

Dissolved oxygen may also be determined by titrimetric methods. Request Publication 8042 for additional information.

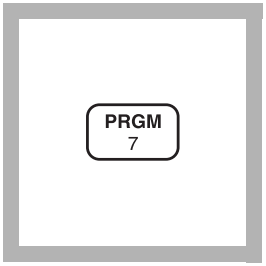
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

OXYGEN, DISSOLVED, Low Range (0 to 1000 µg/L O_2) For boiler feedwater

Indigo Carmine Method (Using AccuVac Ampuls)



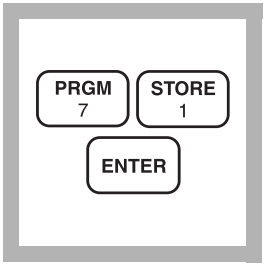
1. Enter the stored program number for low range dissolved oxygen (O_2).

Press: **PRGM**

The display will show:

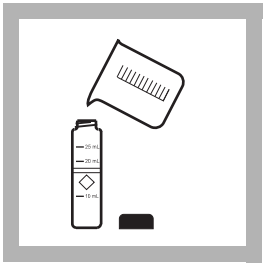
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



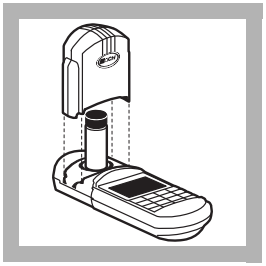
2. Press: **71 ENTER**

The display will show **µg/L, O2** and the **ZERO** icon**.**

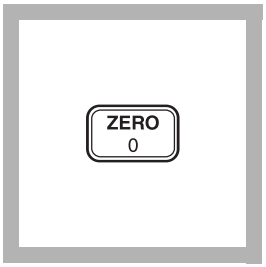


3. Fill a sample cell with at least 10 mL of sample (the blank).

Note: *Samples must be analyzed immediately and cannot be stored.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

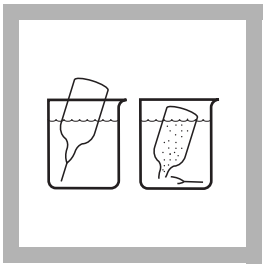


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

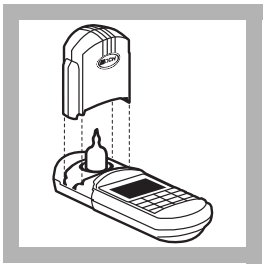
0 µg/L O2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



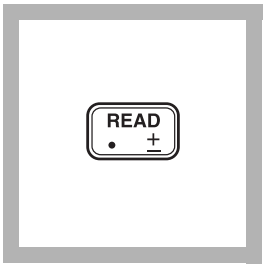
6. Collect at least 40 mL of sample in a 50-mL beaker. Fill a Low Range Dissolved Oxygen AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*



7. Immediately place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.

Note: *The ampuls will contain a small piece of wire to maintain reagent quality. The solution color will be yellow.*



8. Press: **READ**

The cursor will move to the right, then the result in µg/L dissolved oxygen will be displayed.

Note: *Use the initial reading. The reading is stable for 30 seconds. After 30 seconds, the ampul solution will absorb oxygen from the air.*

Sampling and Storage

The main consideration in this procedure is to prevent contaminating the sample with atmospheric oxygen. Sampling from a stream of water that is hard plumbed to the sample source is ideal. Use a funnel to maintain a continual flow of sample and yet collect enough sample to immerse the ampul. It is important not to introduce air in place of the sample. Rubber tubing, if used, will introduce unacceptable amounts of oxygen into the sample unless the length of tubing is minimized and the flow rate is maximized. Flush the sampling system with sample for at least 5 minutes.

Accuracy Check

The reagent blank for this test can be checked by following these steps:

a) Fill a 50-mL beaker with sample and add approximately 50 mg sodium hydrosulfite.

b) Immerse the tip of a Low Range Dissolved Oxygen AccuVac Ampul in the sample and break the tip. Keep the tip immersed while the ampul fills completely.

c) Determine the dissolved oxygen concentration according to the preceding procedure. The result should be 0 ±1 µg/L.

Method Performance

Precision

In a single laboratory, using a standard solution of 500 µg/L O_2 and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±2 µg/L O_2. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program #71 is 10 µg/L O_2. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Hydrazine	100,000 fold excess will begin to reduce the oxidized form of the indicator solution.
Sodium hydrosulfite	Reduces the oxidized form of the indicator solution and will cause a significant interference.

Excess amounts of sodium thioglycolate, sodium ascorbate, sodium ascorbate + sodium sulfite, sodium ascorbate + cupric sulfate, sodium nitrite, sodium sulfite, sodium thiosulfate, and hydroquinone do not cause significant interference.

Summary of Method

When the vacuum-sealed AccuVac ampul is broken open in a sample containing dissolved oxygen, the yellow reagent solution turns blue. The blue color is proportional to the dissolved oxygen concentration.

REQUIRED REAGENTS & APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Low Range Dissolved Oxygen AccuVac Ampuls	1 ampul	25/pkg	25010-25
Beaker, 50 mL	1	each	500-41H
Sample Cell, 10-20-25 mL, w/cap	1	6/pkg	24019-06

OPTIONAL REAGENTS AND APPARATUS

AccuVac Snapper Kit	each	24052-00
Sodium Hydrosulfite, technical grade	500 g	294-34

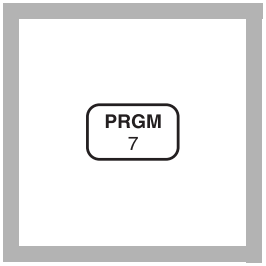
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

OZONE (0 to 0.25 mg/L O_3, 0 to 0.75 mg/L O_3 or 0 to 1.50 mg/L O_3) For water

Indigo Method (Using AccuVac Ampuls)

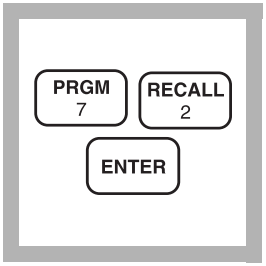


1. Enter the stored program number for Ozone (O_3) AccuVac ampuls.

Press: **PRGM**

The display will show:

PRGM ?

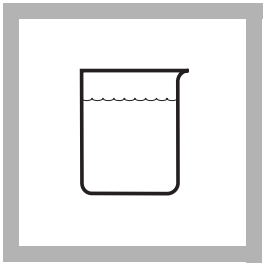


2. Press: **72 ENTER** for low range ozone

Press: **73 ENTER** for mid range ozone

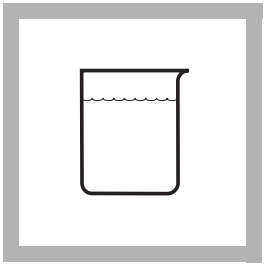
Press: **74 ENTER** for high range ozone.

The display will show **mg/L, O3** and the **ZERO** icon.



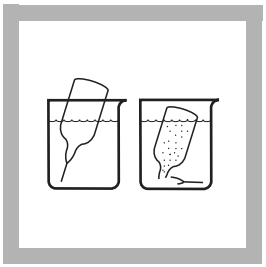
3. Gently collect at least 40 mL of sample in a 50-mL beaker.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis. See* Sampling and Storage *following these steps for proper collection.*



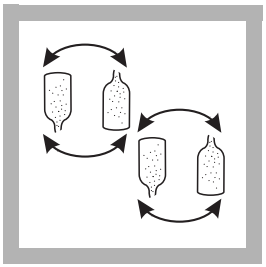
4. Collect at least 40 mL of ozone-free water (blank) in another 50-mL beaker.

Note: *Ozone-free water used for the blank may be deionized water or tap water.*



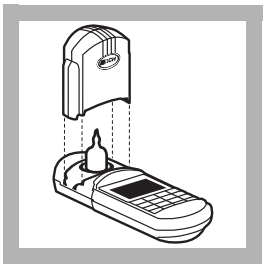
5. Fill one Indigo Ozone Reagent AccuVac Ampul with the sample and one ampul with the blank.

Note: *Keep the tip immersed while the ampul fills.*



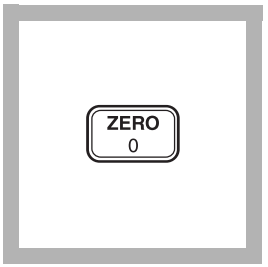
6. Quickly invert both ampuls several times to mix. Wipe off any liquid or fingerprints.

Note: *Part of the blue color will be bleached if ozone is present. (The sample will be lighter than the blank.)*



7. Place the **sample** AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.

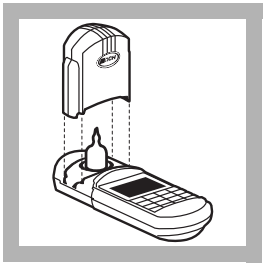
Note: *Standardization for this procedure is intentionally reversed.*



8. Press: **ZERO**

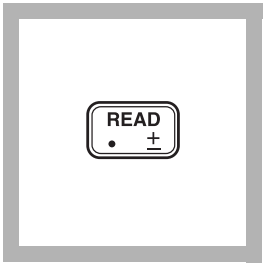
The cursor will move to the right, then the display will show:

0.00 mg/L O3



9. Place the AccuVac ampul containing the **blank** into the cell holder. Tightly cover the ampul with the instrument cap.

Note: *Standardization for this procedure is intentionally reversed.*



10. Press: **READ**

The cursor will move to the right, then the result in mg/L ozone will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling

The chief consideration when collecting a sample is to prevent the escape of ozone from the sample. The sample should be collected gently and analyzed immediately. Warming the sample or disturbing the sample by stirring or shaking will result in ozone loss. After collecting the sample, do not transfer it from one container to another unless absolutely necessary.

Stability of Indigo Reagent

Indigo is light-sensitive. Therefore, the AccuVac Ampuls should be kept in the dark at all times.

However, the indigo solution decomposes slowly under room light after filling with sample. The blank ampul can be used for multiple measurements during the same day.

Method Performance

Precision

In a single laboratory, using standard solutions of 0.15, 0.28 and 0.96 mg/L ozone for the low, mid and high range, respectively, and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.01, ±0.02 and ±0.02 mg/L O_3 for the low, mid and high range tests, respectively. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for the programs #72, #73, and #74 is 0.02 mg/L O_3. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

The reagent formulation adjusts the sample pH to 2.5 after the ampul has filled. The indigo reagent reacts immediately and quantitatively with ozone. The blue color of indigo is bleached in proportion to the amount of ozone present in the sample. Other reagents in the formulation prevent chlorine interference. No transfer of sample is needed in the procedure. Therefore, ozone loss due to sampling is eliminated.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Ozone AccuVac Ampuls			
Select one or more based on range:			
0-0.25 mg/L	2 ampuls	25/pkg	25160-25
0-0.75 mg/L	2 ampuls	25/pkg	25170-25
0-1.50 mg/L	2 ampuls	25/pkg	25180-25
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Beaker, 50 mL	2	each	500-41H

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
AccuVac Ampule sampler	each	24051-00

For Technical Assistance, Price and Ordering

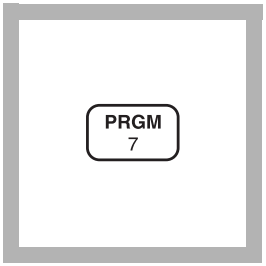
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

pH (6.5 to 8.5 pH units)

Colorimetric pH Determination Using Phenol Red

For water and wastewater

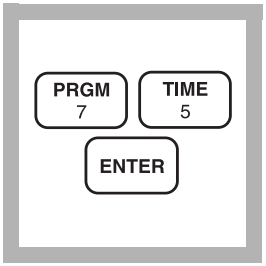


1. Enter the stored program number for the pH method.

Press: **PRGM**

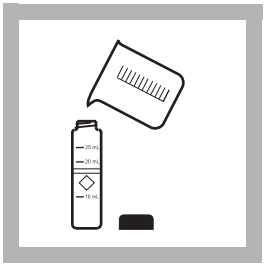
The display will show:

PRGM ?

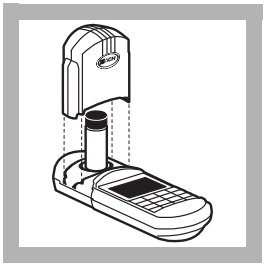


2. Press: **75 ENTER**

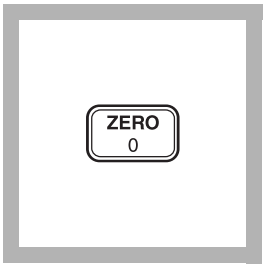
The display will show **PH** and the **ZERO** icon.



3. Fill a sample cell with 10 mL of sample (the blank).



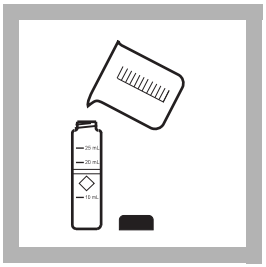
4. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

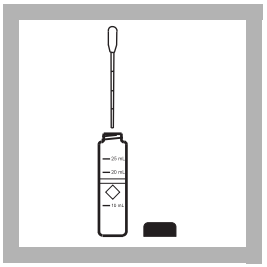
The cursor will move to the right, then the display will show:

6.0 PH

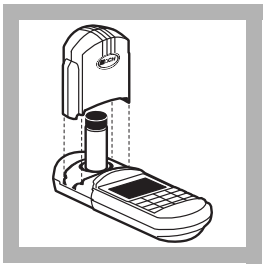


6. Fill another cell with 10 mL of sample.

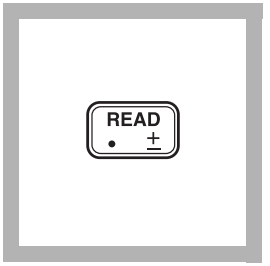
Note: *Sample temperature must be 21-29 °C.*



7. Using a disposable dropper, add 1 mL of Phenol Red Indicator Solution to the cell (the prepared sample). Cap the sample cell and invert twice to mix.



8. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



9. Press: **READ**

The cursor will move to the right, then the result in pH units will be displayed.

Note: *Use of the Standard Adjust feature is highly recommended. See* Accuracy Check.

Note: *Any reading below 6.5 pH units will be erroneous.*

Sampling and Storage

Analyze samples immediately for best results.

Accuracy Check

Standard Solution Method
Using a clear pH 7.0 buffer solution as the sample, perform the pH procedure as described above.

Method Performance

Precision
In a single laboratory using a standard solution of pH 7.0 and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than 0.1 pH units.

Estimated Detection Limit
The estimated detection limit for program 75 is a pH of 6.5.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 7.0 buffer solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **7.0** to edit the standard concentration to match that of the standard used. See *Section 1, Standard Curve Adjustmen*t for more information. Press **ENTER** to complete the curve adjustment.

Interferences

Chlorine does not interfere at levels of 6 mg/L or lower.

Salt water (sea water) will interfere and cannot be analyzed using this method.

Summary of Method

This method uses a sulfonphthalein indicator (Phenol Red) to determine pH colorimetrically. Phenol Red has a working range of pH 6.8 (yellow) to 8.2 (red).

REQUIRED REAGENTS & APPARATUS

Description	Quantity Required Per Test	Units	Cat. No.
Dropper, 0.5 & 1.0 mL marks	1	20/pkg	21247-20
Phenol Red Indicator Solution, spec grade	1.0 mL	50 mL	26575-12
Sample Cells, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

pH 7.0 Buffer Solution		500 mL	12222-49

OPTIONAL APPARATUS

Description	Units	Cat. No.
Thermometer, -20 to 110 °C, Non-Mercury	each	26357-02

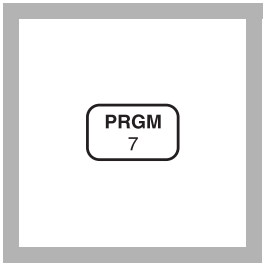
For Technical Assistance, Price and Ordering

In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHONATES (0-2.5 to 0-125 mg/L)

Persulfate UV Oxidation Method*



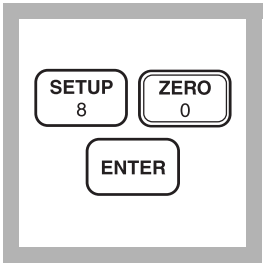
1. Enter the stored program number for phosphonates.

Press: **PRGM**

The display will show:

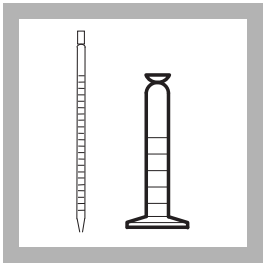
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **80 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**



3. Choose the appropriate sample size from *Table 1* below. Pipet the chosen sample volume into a 50-mL graduated mixing cylinder. Dilute the sample to 50 mL with deionized water. Mix well.

Note: *Clean glassware with 1:1 hydrochloric acid, followed by a deionized water rinse. Do not use commercial detergents containing phosphates to clean glassware.*

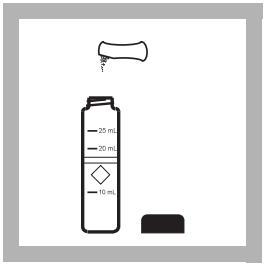
4. Fill a sample cell to the 10-mL mark with diluted sample from Step 3 (label this as the blank).

Fill another sample cell to the 25-mL mark with diluted sample from Step 3 (label this as the sample).

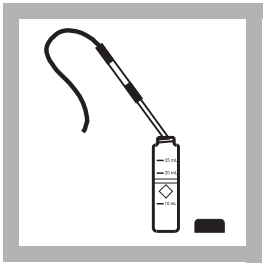
Table 1

Expected Range (mg/L phosphonate)	Sample Volume (mL)
0-2.5	50
0-5	25
0-12.5	10
0-25	5
0-125	1

* Adapted from Blystone, P.; Larson, P., *A Rapid Method for Analysis of Phosphonate Compounds*, International Water Conference, Pittsburgh, Pa. (Oct. 26-28, 1981).



5. Add the contents of one Potassium Persulfate for Phosphonate Powder Pillow to the cell labeled as "sample". Swirl to mix. This cell contains the prepared sample.

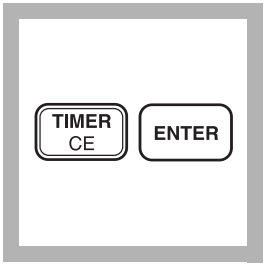


6. Insert the ultraviolet (UV) lamp into the prepared sample.

Note: *Wear UV safety goggles while the lamp is on.*

Note: *Do not handle the lamp surface. Fingerprints will etch the glass. Wipe lamp with a soft, clean tissue between samples. Do not use detergents with phosphates to wash glassware.*

Note: *A specially designed cord adapter is available for performing two digestions with a single power supply. A second UV lamp is required.*



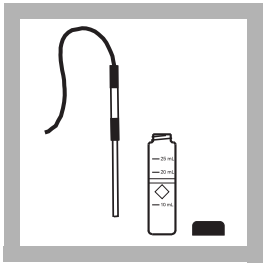
7. Turn on the UV lamp to digest the prepared sample.

Press: **TIMER ENTER**

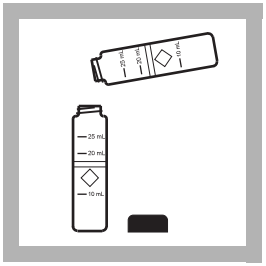
A 10-minute reaction period will begin.

Note: *Phosphonates are converted to ortho-phosphate in this step.*

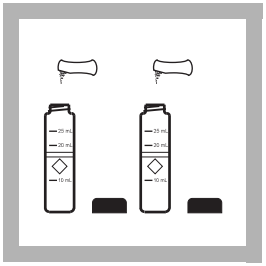
Note: *The digestion step may take less time. Contaminated samples or a weak lamp could result in incomplete digestion. Check efficiency by running a longer digestion to see if readings increase.*



8. When the timer beeps, turn off the UV lamp. Remove it from the sample cell.

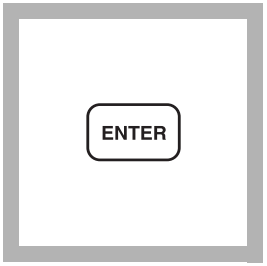


9. Pour 10 mL of sample from the cell labeled as "sample" into a second clean, dry sample cell. This is the prepared sample.



10. Add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow for 10-mL samples to each sample cell. Swirl immediately to mix.

Note: *A blue color will form if phosphate is present. Sample and blank cells may develop color.*

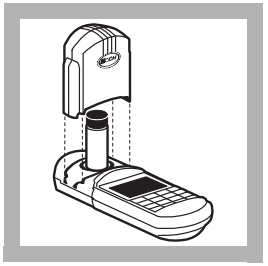


11. The display will show: **2:00 TIMER 2**

Press: **ENTER**

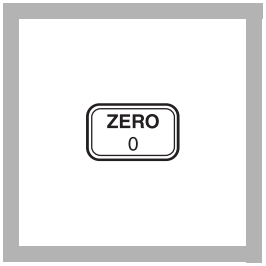
A two-minute reaction period will begin.

Note: *If sample is colder than 15 °C, 4 minutes are required for color development.*



12. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Perform Steps 12-15 within three minutes after the timer beeps.*

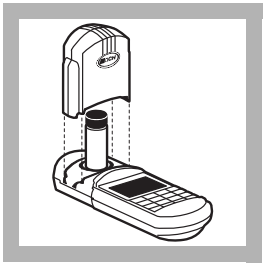


13. Press: **ZERO**

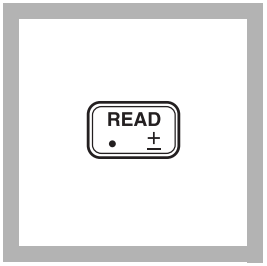
The cursor will move to the right, then the display will show:

0.0 mg/L PO4

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



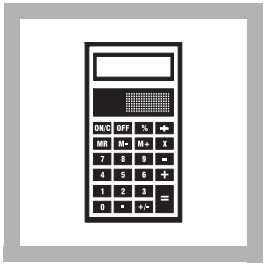
14. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



15. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate will be displayed. Multiply this value by the appropriate multiplier from *Table 2* to obtain the actual concentration of phosphonates as phosphate in the sample.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*



16. Results may be expressed in terms of a specific active phosphonate by using the appropriate conversion factor and the equation found in *Table 3*.

Table 2

Sample Volume (mL) (chosen in Step 3)	Multiplier
50	0.1
25	0.2
10	0.5
5	1.0
1	5.0
Phosphate concentration = Instrument Reading x Multiplier	

Table 3

Phosphonate Type	Conversion Factor
PBTC	2.84
NTP	1.050
HEDPA	1.085
EDTMPA	1.148
HMDTMPA	1.295
DETPMPA	1.207
HPA	1.49
Active Phosphonate (mg/L) = Phosphate concentration from Step 15 x Conversion Factor	

Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water.
Do not use a commercial detergent. If prompt analysis is impossible, adjust the pH to 2 or less with about 2 mL of sulfuric acid, ACS, per liter of sample. Store at 4 °C (39 °F) or below. Preserved samples can be stored at least 24 hours. See *Section 1* for more information on dilution factors, cleaning instructions, etc.

Accuracy Check

Ideally, a solution containing a known amount of the phosphonate product being used should be prepared. This will check the UV conversion of phosphonate to orthophosphate.

Interferences

When testing a 5-mL sample volume, the following may interfere when present in concentrations exceeding those listed below:

The interference levels will decrease as the sample size increases. For example, copper does not interfere at or below 100 mg/L for a 5.00 mL sample. If the sample volume is increased to 10.00 mL, copper will begin to interfere above 50 mg/L.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interferences* in *Section 1*.

Phosphites and organophosphorus compounds other than phosphonates react quantitatively. Meta and polyphosphates do not interfere.

Interfering Substance	Level	Interfering Substance	Level
Aluminum	100 mg/L	EDTA	100 mg/L
Arsenate	all levels	Iron	200 mg/L
Benzotriazole	10 mg/L	Nitrate	200 mg/L
Bicarbonate	1000 mg/L	NTA	250 mg/L
Bromide	100 mg/L	Orthophosphate	15 mg/L
Calcium	5000 mg/L	Silica	500 mg/L
CDTA	100 mg/L	Silicate	100 mg/L
Chloride	5000 mg/L	Sulfate	2000 mg/L
Chromate	100 mg/L	Sulfide	All levels
Copper	100 mg/L	Sulfite	100 mg/L
Cyanide[1]	100 mg/L	Thiourea	10 mg/L
Diethanoldithiocarbamate	50 mg/L		

[1] Increase the UV digestion to 30 minutes.

Summary of Method

This method is directly applicable to boiler and cooling tower samples. The procedure is based on a UV catalyzed oxidation of phosphonate to orthophosphate. Range may be as low as 0 to 2.5 mg/L or as high
as 0 to 125 mg/L.

Phosphonate is converted to orthophosphate during the UV digestion. Both the sample and the blank will develop color if orthophosphate is present in the sample. The increase in color in the sample is proportional to the phosphate produced in the digestion.

REQUIRED REAGENTS

Phosphonates Reagent Set (100 tests) 24297-00
Includes: (2) 21060-69, (1) 20847-69

Description	Quantity Required Per Test	Unit	Cat. No
PhosVer 3 Phosphate Reagent Powder Pillows	2 pillows	100/pkg	21060-69
Potassium Persulfate Pillow for Phosphonate	1 pillow	100/pkg	20847-69
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Cylinder, mixing, graduated, 50 mL	1	each	1896-41
Goggles, UV safety	1	each	21134-00
Pipet, serological, 25 mL	1	each	2066-40
Pipet Filler, safety bulb	1	each	14651-00
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
UV Lamp with power supply, 115 V, with goggles	1	each	20828-00
OR			
UV Lamp with power supply, 230 V	1	each	20828-02

OPTIONAL REAGENTS

Hydrochloric Acid, 6.0 N (1:1)	500 mL		884-49
Sulfuric Acid, ACS	500 mL		979-49

OPTIONAL APPARATUS

pH Paper, 1 to 11 pH units	5 rolls/pkg		391-33
Pipet, serological, 2 mL	each		532-36
Pipet, TenSette, 1-10 mL	each		19700-10
Pipet Tips, for 19700-10 Tensette Pipet	50/pkg		21997-96
Thermometer, -20 to 110 °C, Non-Mercury	each		1877-01

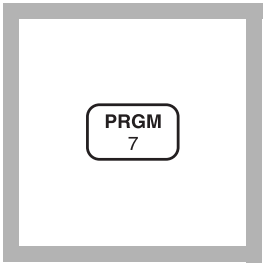
For Technical Assistance, Price and Ordering
In the U.S.A. call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, REACTIVE (0 to 2.50 mg/L PO_4^{3-}) For water, wastewater, seawater

(Also called Orthophosphate) PhosVer 3 (Ascorbic Acid) Method*

(Powder Pillows or AccuVac Ampuls) USEPA Accepted for wastewater analysis reporting**

Using Powder Pillows



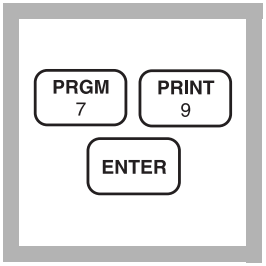
1. Enter the stored program number for reactive phosphorus, ascorbic acid method.

Press: **PRGM**

The display will show:

PRGM ?

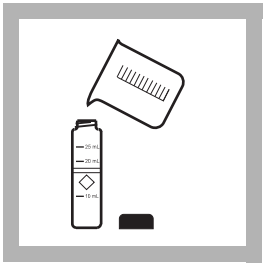
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **79 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

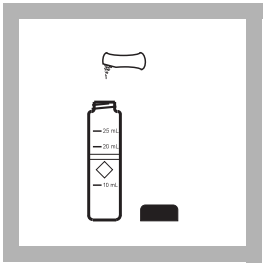
Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*



3. Fill a sample cell with 10 mL of sample.

Note: *For samples with extreme pH, see* Interferences *following these steps.*

Note: *Clean glassware with 1:1 HCl. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

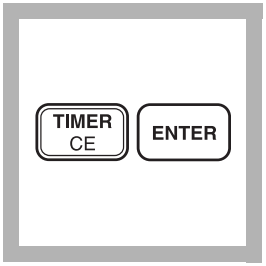


4. Add the contents of one PhosVer 3 Phosphate Powder Pillow for 10-mL sample to the cell (the prepared sample). Shake for 15 seconds.

Note: *A blue color will form if phosphate is present.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

** Procedure is equivalent to USEPA method 365.2 and Standard Method 4500-PE for wastewater.

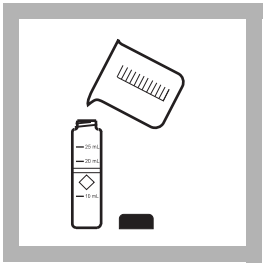


5. Press:

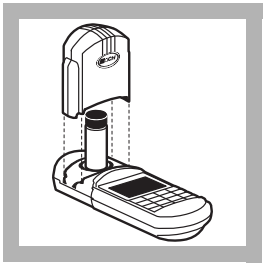
TIMER ENTER

A two-minute reaction period will begin. Perform Steps 6-8 during this period.

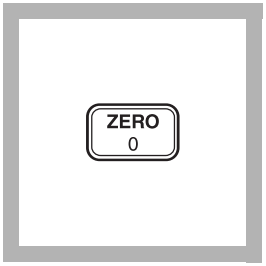
Note: *If the acid-persulfate digestion was used, an 8-10 minute reaction period is required.*



6. Fill another sample cell with 10 mL of sample (the blank).



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

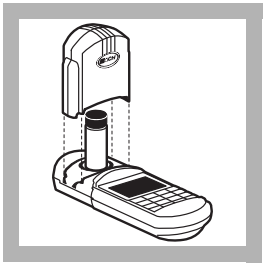


8. Press: **ZERO**

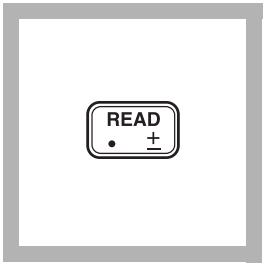
The cursor will move to the right, then the display will show:

0.00 mg/L PO4

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



9. After the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

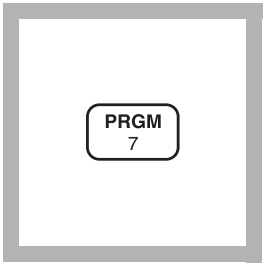


10. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *Standard Adjust may be performed using a 2.0-mg/L PO_4^{3-} standard; see Section 1.*

Using AccuVac Ampuls



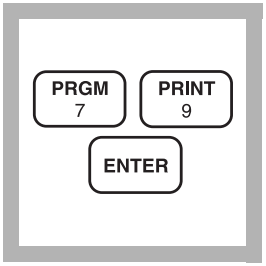
1. Enter the stored program number for reactive phosphorus-ascorbic acid method.

Press: **PRGM**

The display will show:

PRGM ?

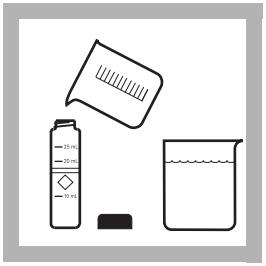
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **79 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

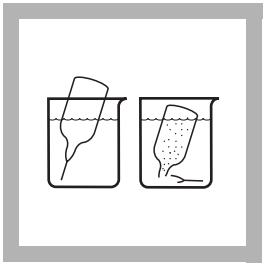
Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*



3. Fill a sample cell (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.

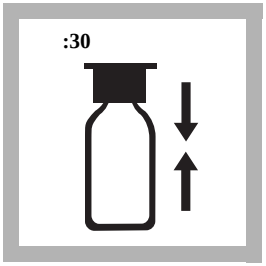
Note: *For samples with extreme pH, see* Interferences.

Note: *Clean glassware with 1:1 HCl. Rinse again with deionized water. Do not use detergent containing phosphates to clean glassware.*



4. Fill a PhosVer 3 Phosphate AccuVac Ampul with sample.

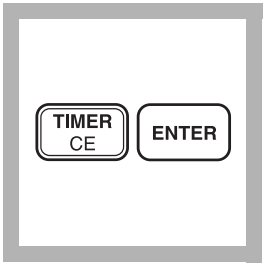
Note: *Keep the tip immersed while the ampul fills completely.*



5. Place an ampul cap securely over the tip of the ampul. Shake the ampul for about 30 seconds. Wipe off any liquid or fingerprints.

Note: *A blue color will form if phosphate is present.*

Note: *Accuracy is not affected by undissolved powder.*

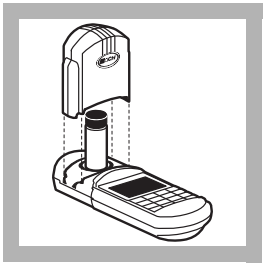


6. Press:

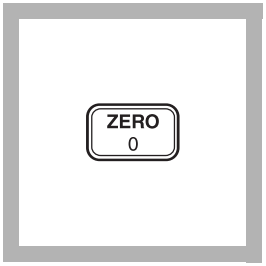
TIMER ENTER

A two-minute reaction period will begin. Perform Steps 7-8 during this period.

Note: *Use an 8-10 minute reaction period if determining total phosphorus following the acid-persulfate digestion.*



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

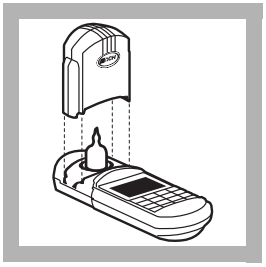


8. Press: **ZERO**

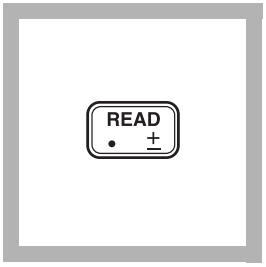
The cursor will move to the right, then the display will show:

0.00 mg/L PO4

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



9. After the timer beeps, place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *Standard Adjust may be performed using a 2.0-mg/L PO_4^{3-} standard; see Section 1.*

Sampling and Storage

Collect sample in plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples for up to 48 hours by filtering immediately and storing samples at 4 °C. Warm to room temperature before testing.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Phosphate PourRite Ampule Standard Solution, 50 mg/L as PO_4^{3-}.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper each and mix thoroughly.

d) For analysis with AccuVacs, transfer solutions to dry, clean 50 mL beakers to fill the AccuVac ampules. For analysis with powder pillows, transfer only 10 mL of solution to the sample cells.

e) Analyze each standard addition sample as described in the procedure. The phosphate concentration should increase 0.2 mg/L PO_4^{3-} for each 0.1 mL of standard added.

f) If these increases do not occur, see *Standard Additions* in *Section 1*.

Standard Solution Method

Prepare a 2.0 mg/L PO_4^{3-} standard solution by pipetting 4.0 mL of Phosphate Standard Solution, 50 mg/L as PO_4^{3-}, into an acid-washed Class A 100-mL volumetric flask. Dilute to volume with deionized water. Stopper and invert to mix. Use this solution in place of the sample in the procedure to insure the accuracy of the test. The mg/L PO_4^{3-} reading should be 2.00 mg/L.

Method Performance

Precision

In a single laboratory using a standard solution of 1.00 mg/L PO_4^{3-} and two lots of reagents with the instrument, a single operator obtained a standard deviation of ±0.05 mg/L PO_4^{3-}.

In a single laboratory using a standard solution of 1.00 mg/L PO_4^{3-} and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of ±0.03 mg/L PO_4^{3-}.

Estimated Detection Limit (EDL)

The EDL for program 79 is 0.05 mg/L PO_4. For more information on the estimated detection limit, see *Section 1*.

Interference

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 200 mg/L
Arsenate	All levels
Chromium	Greater than 100 mg/L
Copper	Greater than 10 mg/L
Hydrogen sulfide	All levels
Iron	Greater than 100 mg/L
Nickel	Greater than 300 mg/L
Silica	Greater than 50 mg/L
Silicate	Greater than 10 mg/L
Turbidity or color	Large amounts may cause inconsistent results in the test because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles. For highly turbid or colored samples, add the contents of one Phosphate Pretreatment Pillow to 25 mL of sample. Mix well. Use this solution to zero the instrument.
Zinc	Greater than 80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment. pH 2 to 10 is recommended.

Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a Phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows 10 mL sample size	1	Pillow 100/pkg	21060-69
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

PhosVer 3 Phosphate Reagent AccuVac Ampuls	1	ampul 25/pkg	25080-25
Beaker, 50 mL	1	each	500-41
Cap, ampul, blue	1	25/pkg	1731-25
Sample Cell, 10-20-25 mL, w/cap	1	6/pkg	24019-06

OPTIONAL REAGENTS

Drinking Water Standard, Inorganic, F^-, NO_3^{-N}, PO_4^{3-}, SO_4^{2-}	500mL	28330-49
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phosphate Standard Solution, 1mg/L	500mL	2569-49
Phosphate Standard Solution, PourRite ampule, 50 mg/L as PO_4^{3-}, 2 mL	20/pkg	171-20
Phosphate Standard Solution, Voluette Ampul, 50 mg/L, 10 mL	16/pkg	171-10
Sodium Hydroxide Standard Solution, 5.0 N	100 mL* MDB	2450-32
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit for 10-ml ampules	each	21968-00
Aspirator, vacuum	each	2131-00
Cylinder, graduated, mixing, 25 mL, tall (3 required)	each	20886-40
Filter Holder, 47 mm, 300 mL, graduated	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	100/pkg	13530-00
Flask, filtering, 500 mL	each	546-49
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *Sens**ion**™**1***, portable with electrode	each	51700-10
Pipet, 2 mL serological	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL TenSette Pipet	each	19700-01
Pipet Tips, for 19700-01	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet Filler, safety bulb	each	14651-00
Pipet, volumetric, Class A, 4.00 mL	each	14515-04
PourRite Ampule Breaker Kit	each	24846-00

Outside the U.S.A.—Contact the Hach office or distributor serving you.

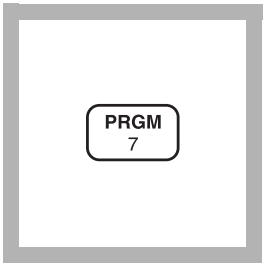
* Larger sizes available.

PHOSPHORUS, REACTIVE (0.00 to 5.00 mg/L PO_4^{3-})

PhosVer 3 Method, Test 'N Tube Procedure

For water, wastewater, and seawater

USEPA accepted for reporting wastewater analysis*



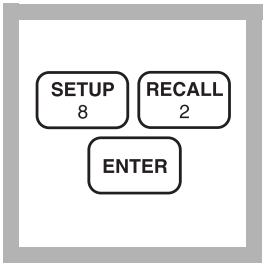
1. Enter the stored program number for reactive phosphorus (PO_4^{3-}), Test 'N Tube.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



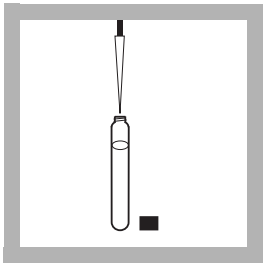
2. Press: **82 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*

3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *A diffuser band covers the light path holes on the adapter to give increased performance.The band should NOT be removed.*



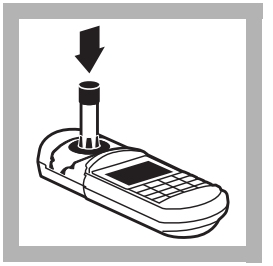
4. Use a TenSette Pipet to add 5.0 mL of sample to a Reactive Phosphorus Test 'N Tube Dilution Vial. Cap and mix.

Note: *For samples with extreme pH, see the* Interference *section.*

* Procedure is equivalent to USEPA Method 365.2 and Standard Method 4500-P E for wastewater.

5. Clean the outside of the vial with a towel.

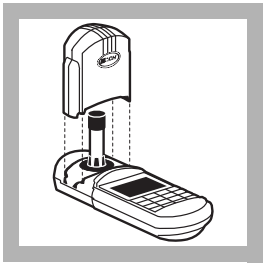
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



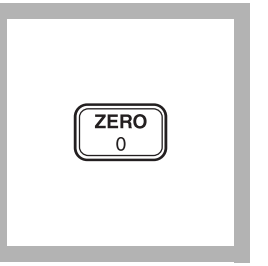
6. Place the sample vial into the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



7. Tightly cover the sample vial with the instrument cap.

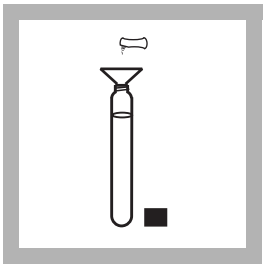


8. Press: **ZERO**

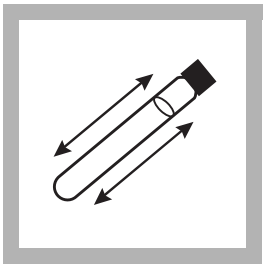
The cursor will move to the right, then the display will show:

0.00 mg/L PO4

Note: *For multiple samples, zero only on the first sample. Read the remaining samples after adding the PhosVer 3 Reagent.*

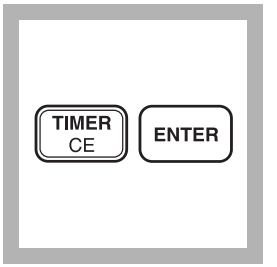


9. Using a funnel, add the contents of one PhosVer 3 Phosphate Powder Pillow to the vial.



10. Cap the vial tightly and shake for 10-15 seconds.

Note: *The powder will not completely dissolve.*



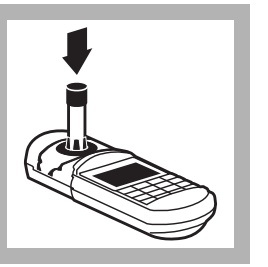
11. Press:

TIMER ENTER

A 2-minute reaction time will begin.

Note: *Read samples between 2 and 8 minutes after the addition of the PhosVer 3 reagent.*

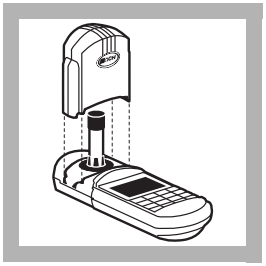
Note: *A blue color will develop if phosphate is present.*



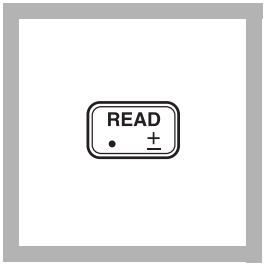
12. Immediately after the timer beeps, place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



13. Tightly cover the vial with the instrument cap.



14. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples for up to 48 hours by filtering immediately and storing at 4 °C. Warm to room temperature before analyzing the sample.

Accuracy Check

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as PO_4^{3-}.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.

d) Analyze each sample as described in the procedure; use 5.0 mL of the prepared standard additions for each test. The concentration should increase as follows: 0.2 mg/L, 0.4 mg/L, 0.6 mg/L PO_4^{3-}, respectively.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To check accuracy, use a 1.0 mg/L Phosphate Standard Solution listed under *Optional Reagents*. Or, prepare a 1.0-mg/L PO_4^{3-} standard by pipetting 2 mL of solution from a Phosphate Voluette Ampule Standard for Phosphate, 50 mg/L as PO_4^{3-}, into an acid-washed, Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

Method Performance

Precision

In a single laboratory, using a standard solution of 5.00 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.08 mg/L PO_4^{3-}.

Estimated Detection Limit (EDL)

The EDL for program 82 is 0.07 mg/L PO_4^{3-}. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding these listed below:

Substance	Interference Level and Treatment
Aluminum	200 mg/L
Arsenate	Interferes at any level
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Sulfide	6 mg/L. Sulfide interference may be removed by oxidation with Bromine Water as follows: 1. Measure 25 mL of sample into a 50-mL beaker. 2. Swirling constantly, add Bromine Water drop-wise until a permanent yellow color develops. 3. Swirling constantly, add Phenol Solution dropwise until the yellow color just disappears. Proceed with *step 1*.
Turbidity (large amounts)	May cause inconsistent results because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see pH Interferences (Section 1).

The PhosVer 3 Phosphate Reagent Powder Pillows should be stored in a cool, dry environment.

Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS

Reactive Phosphorus Test 'N Tube Reagent Set ..... 50 tests 27425-45
Includes: (1) 21060-46, (50) Orthophosphate Dilution Vials*

Description	Quantity Required Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows	1	50/pkg	21060-46
50 Orthophosphate Test 'N Tube Dilution Vials	1	50/pkg	*

REQUIRED APPARATUS

COD/TNT Adapter	1	each	48464-00
Funnel, micro	1	each	25843-35
Pipet, TenSette, 1 to 10 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet	1	50/pkg	21997-96
Test Tube Rack	1-3	each	18641-00

OPTIONAL REAGENTS

Bromine Water, 30 g/L	29 mL	2211-20
Drinking Water Standard, Inorganic, F^-, $NO_3^{-N,}$ PO_4^{3-}, SO_4^{2-}	500mL	28330-49
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phenol Solution, 30 g/L	29 mL	2112-20
Phosphate Standard Solution, 1 mg/L as PO_4^{3-}	500 mL	2569-49
Phosphate Standard Solution, Voluette ampule, 50 mg/L as PO_4^{3-}, 10 mL	16/pkg	171-10
Phosphate Standard Solution, PourRite ampule, 50 mg/L as PO_4^{3-}, 2 mL	20/pkg	171-20H
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49
Water, deionized	4 L	272-56

* These items are not sold separately.

OPTIONAL APPARATUS

Ampule Breaker, Pour Rite (2-mL ampule)	each	24846-00
Ampule Breaker Kit	each	21968-00
Aspirator, vacuum	each	2131-00
Cylinder, graduated, mixing, 25 mL (3 required)	each	20886-40
Dispenser, Repipet Jr., 2 mL	each	22307-01
Filter Holder, 47 mm, 300 mL, graduated	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	100/pkg	13530-00
Flask, filtering, 500 mL	each	546-49
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable with electrode	each	51700-10
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01	50 pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet Filler, Safety Bulb	each	14651-00
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 2.00 mL	each	14515-36

For Technical Assistance, Price and Ordering

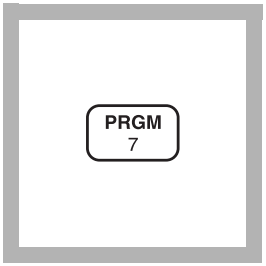
In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, REACTIVE (0 to 30.0 mg/L PO_4^{3-})

Amino Acid Method*

For water, wastewater, seawater



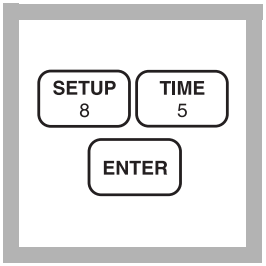
1. Enter the stored program number for reactive phosphate (PO_4^{3-}), amino acid method.

Press: **PRGM**

The display will show:

PRGM ?

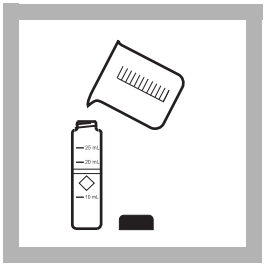
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



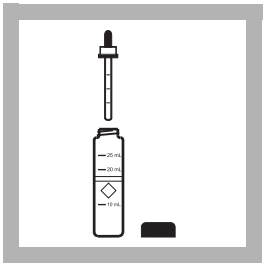
2. Press: **85 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon.

Note: *For alternate forms (P, P_2O_5), press* **CONC**.

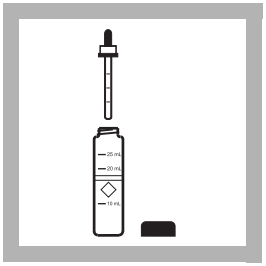


3. Fill a 25-mL sample cell with 25 mL of sample.



4. Add 1 mL of Molybdate Reagent using a 1-mL calibrated dropper.

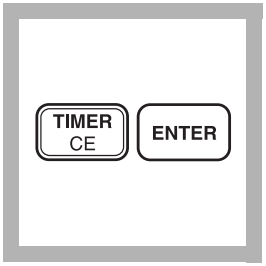
* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



5. Add 1 mL of Amino Acid Reagent Solution. Cap and invert several times to mix (the prepared sample).

Note: *A blue color will form if phosphate is present.*

Note: *You may substitute the contents of one Amino Acid Reagent Powder Pillow for 1 mL of Amino Acid Reagent Solution.*

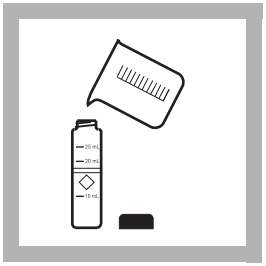


6. Press:

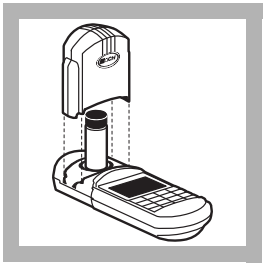
TIMER ENTER

A 10-minute reaction period will begin.

Note: *Perform Step 7 while the timer is running.*



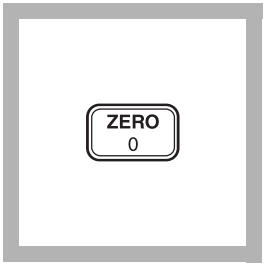
7. Pour 25 mL of sample (the blank) into a sample cell.



8. When the timer beeps, the display will show:

mg/L PO4

Place the blank into the cell holder. Cover the sample cell with the instrument cap.

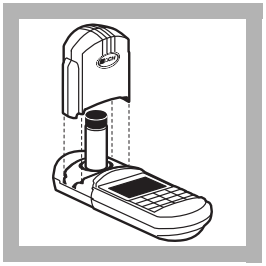


9. Press: **ZERO**

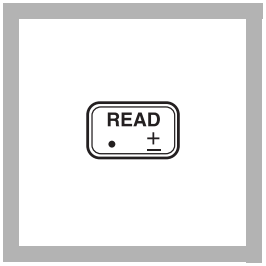
The cursor will move to the right, then the display will show:

0.0 mg/L PO4

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L PO_4 will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use a commercial detergent containing phosphate for cleaning glassware used in this test.

Analyze samples immediately for best results. If prompt analysis is not possible, preserve samples by filtering immediately and storing the sample at 4 °C (39 °F) for up to 48 hours.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Phosphate PourRite Ampule Standard Solution, 500 mg/L as PO_4^{3-}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 25-mL samples. Mix well.

c) Analyze each sample as described in the procedure. Each 0.1-mL addition of standard should cause an increase of 2.0 mg/L orthophosphate (PO_4^{3-}).

d) If these increases do not occur, see *Standard Additions* (*Section 1*) for more information.

Standard Solution Method

Prepare a 10.0-mg/L phosphate standard by pipetting 10.0 mL of a Phosphate Standard Solution, 50 mg/L as PO_4^{3-} into a 50-mL volumetric flask. Dilute to volume with deionized water.

Or, prepare a 10.0-mg/L PO_4^{3-} standard solution by using the TenSette Pipet to add 1.00 mL of Phosphate PourRite Ampule Standard,
500 mg/L as PO_4^{3-}, into a 50-mL volumetric flask. Dilute to volume
with deionized water.

Substitute this standard for the sample and perform the test as described. The mg/L PO_4^{3-} reading should be 10 mg/L.

Method Performance

Precision

In a single laboratory using a standard solution of 15.0 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.12 mg/L PO_4^{3-}.

Estimated Detection Limit

The estimated detection limit for program 85 is 0.14 mg/L PO_4^{3-}. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Calcium (Ca^{2+})	Greater than 10,000 mg/L as $CaCO_3$
Chloride	Greater than 150,000 mg/L as Cl^-
Colored samples	Add 1 mL of 10 N Sulfuric Acid Standard Solution to another 25-mL sample. Use this instead of untreated sample as the blank to zero the instrument. Use a pipet and pipet filler to measure the sulfuric acid standard.
High salt levels (Na^+)	May cause low results. To eliminate this interference, dilute the sample until two successive dilutions yield about the same result.
Magnesium	Greater than 40,000 mg/L as $CaCO_3$
Nitrites (NO_2^-)	Bleach the blue color. Remove nitrite interference by adding 0.05 g of sulfamic acid to the sample. Swirl to mix. Continue with Step 4.
Phosphates, high levels (PO_4^{3-})	As the concentration of phosphate increases, the color changes from blue to green, then to yellow and finally to brown. The brown color may suggest a concentration as high as 100,000 mg/L PO_4^{3-}. If a color other than blue is formed, dilute the sample and retest.
Sulfide (S^{2-})	Sulfide interferes. For samples with sulfide concentration less than 5 mg/L, sulfide interference may be removed by oxidation with Bromine Water as follows: **1.** Measure 50mL of sample into a 125-mL flask. **2.** Add Bromine Water dropwise with constant swirling until permanent yellow color develops. **3.** Add Phenol Solution dropwise until the yellow color just disappears. Use this sample in Steps 3 and 7.
Temperature	For best results, sample temperature should be 21 ±3 °C (70 ±5 °F).
Turbidity	May give inconsistent results for two reasons. Some suspended particles may dissolve because of the acid used in the test. Also, desorption of orthophosphate from particles may occur. For highly turbid samples, add 1 mL of 10 N Sulfuric Acid Standard Solution to another 25-mL sample. Use this instead of untreated sample as the blank to zero the instrument. Use a pipet and pipet filler to measure the sulfuric acid standard.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

Summary of Method

In a highly acidic solution, ammonium molybdate reacts with orthophosphate to form molybdophosphoric acid. This complex is then reduced by the amino acid reagent to yield an intensely colored molybdenum blue compound.

REQUIRED REAGENTS

	Cat. No.
High Range Reactive Phosphorus Reagent Set (100 Test)	22441-00

Includes: (1) 1934-32, (1) 2236-32

Description	Quantity Required Per Test	Units	Cat. No.
Amino Acid Reagent	1 mL	100 mL MDB*	1934-32
Molybdate Reagent	1 mL	100 mL MDB*	2236-32

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Description	Units	Cat. No.
Amino Acid Reagent Powder Pillow	100/pkg	804-99
Bromine Water, 30 g/L	29 mL	2211-20
Hydrochloric Acid Solution, 1:1 (6 N)	500 mL	884-49
Phenol Solution, 30 g/L	29 mL	2112-20
Phosphate Standard Solution, 50 mg/L PO_4^{3-}	500 mL	171-49
Phosphate Standard Solution, PourRite ampule, 500 mg/L PO_4^{3-}, 2 mL	20/pkg	14242-20
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sulfamic Acid	113 g	2344-14
Sulfuric Acid Standard Solution, 10 N	1 L	931-53
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49
Water, deionized	4 L	272-56

* Larger sizes available.

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Ampule Breaker Kit, PourRite	each	24846-00
Aspirator, vacuum	each	2131-00
Cylinder, graduated, 50 mL	each	508-41
Cylinder, graduated, mixing, 25 mL	each	20886-40
Filter Holder, 47 mm, 300 mL, graduated	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	100/pkg	13530-00
Flask, filtering, 500 mL	each	546-49
Flask, erlenmeyer, 125 mL	each	505-43
Flask, volumetric, Class A, 50 mL	each	14574-41
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable with electrode	each	51700-10
Pipet, serological, 2.0 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 10.00 mL	each	14515-38
Pipet Filler, safety bulb	each	12189-00
Spoon, measuring, 0.05 g	each	492-00
Thermometer, -20 to 110 °C, Non-Mercury	each	26357-02

For Technical Assistance, Price and Ordering

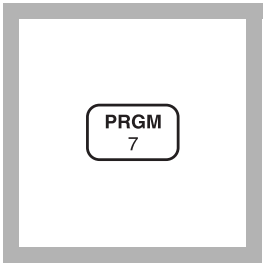
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, REACTIVE (0 to 45.0 mg/L PO_4^{3-}) For water and wastewater

(Also called Orthophosphate) Molybdovanadate Method*
(Reagent Solution or AccuVac Ampuls)

Using Reagent Solution

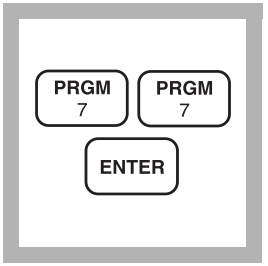


1. Enter the stored program number for high range phosphate (PO_4^{3-}) reagent solution.

Press: **PRGM**

The display will show:

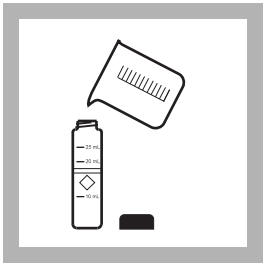
PRGM ?



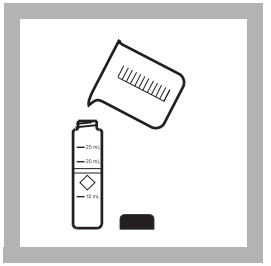
2. Press: **77 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*

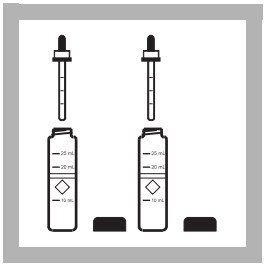


3. Fill a sample cell with 25 mL of deionized water (the blank).



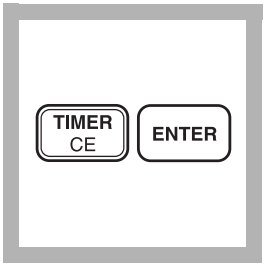
4. Fill another sample cell with 25 mL of sample (the prepared sample).

Note: *For best results, the sample temperature should be 20-25 °C.*



5. Add 1.0 mL of Molybdovanadate Reagent to each sample cell. Cap the cells and invert to mix.

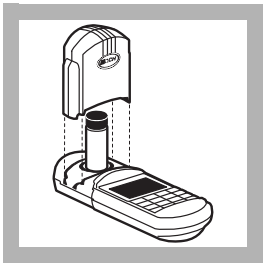
Note: *A yellow color will form if phosphate is present. A small amount of yellow will be present in the blank, because of the reagent.*



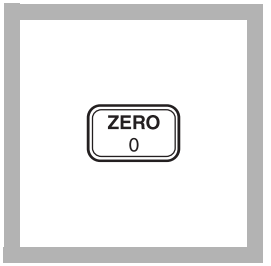
6. Press:

TIMER ENTER

A five-minute reaction period will begin.



7. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

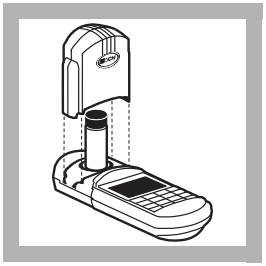


8. Press: **ZERO**

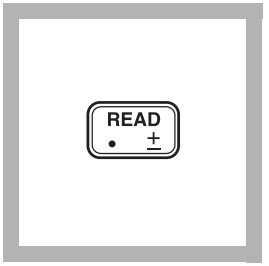
The cursor will move to the right, then the display will show:

0.0 mg/L PO4

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

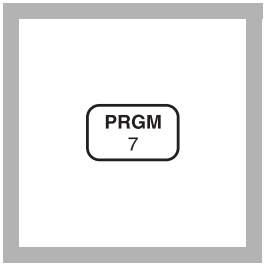


10. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (or alternate form) will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Using AccuVac Ampuls

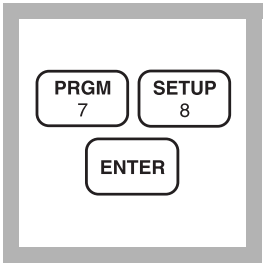


1. Enter the stored program number for high range phosphate (PO_4^{3-})-AccuVac Ampuls.

Press: **PRGM**

The display will show:

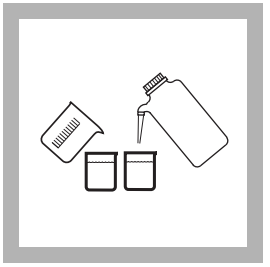
PRGM ?



2. Press: **78 ENTER**

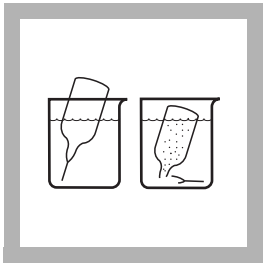
The display will show **mg/L, PO4** and the **ZERO** icon**.**

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*



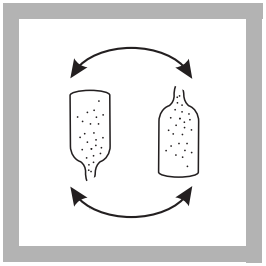
3. Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second beaker.

Note: *For best results, sample temperature should be 20-25 °C.*



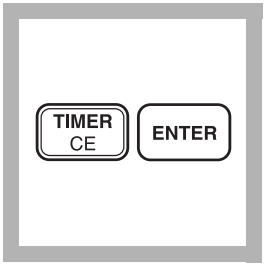
4. Fill a Molybdovanadate Reagent AccuVac Ampul with sample. Fill a second AccuVac Ampul with deionized water (the blank).

Note: *Keep the tip immersed while the ampul fills completely.*



5. Invert the ampul several times to mix, then wipe off any liquid or fingerprints.

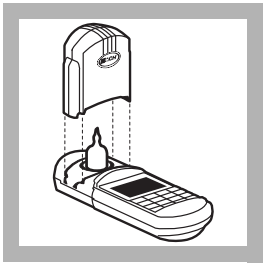
Note: *A yellow color will form if phosphate is present. A small amount of yellow will be present in the blank because of the reagent.*



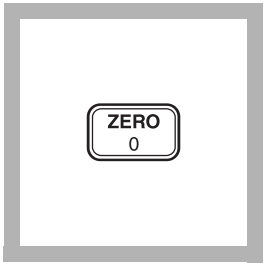
6. Press:

TIMER ENTER

A five-minute reaction period will begin.



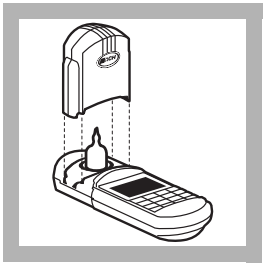
7. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



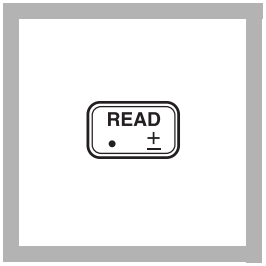
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L PO4



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (or alternate form) will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water.

Do not use a commercial detergent containing phosphate for cleaning glassware used in this test.

Analyze samples immediately for best results. If prompt analysis is impossible, preserve samples by filtering immediately and storing at 4 °C for up to 48 hours.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Phosphate Voluette Ampule Standard Solution, 500 mg/L as PO_4^{3-}.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper and invert to mix well.

d) For analysis with AccuVac Ampuls, transfer the spiked samples to clean, dry 50-mL beakers to facilitate filling of the ampuls. For analysis with reagent solution, transfer the spiked samples to
25-mL sample cells.

e) Analyze each sample as described in the procedure. Each 0.1-mL addition of standard should cause an increase of 2.0 mg/L PO_4^{3-}.

f) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

Standard Solution Method

Obtain a Hach Phosphate Standard Solution, 10.0 mg/L as phosphate. Using this solution as the sample, perform the phosphate procedure as described above.

Standard Adjust

To adjust the calibration curve using the reading obtained with the
10.0 mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustmen*t, *Section 1* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 30.0 mg/L PO_4^{3-}, two lots of reagent, and the instrument, a single operator obtained a standard deviation of ±0.1 mg/L PO_4^{3-} for the reagent solution method and a standard deviation of ±0.2 for the AccuVac Ampul method.

Estimated Detection Limit

The estimated detection limit for program 77 is 0.3 mg/L PO_4^{3-} and
0.4 mg/L PO_4^{3-} for program 78. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatment

Interfering Substance	Interference Level and Treatment
Arsenate	Only interferes if sample is heated.
Iron, ferrous	Blue color caused by ferrous iron does not interfere if iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Only interferes if sample is heated.
Sulfide	Causes a negative interference. Remove interference as follows: **1.** Measure 50 mL of sample into an erlenmeyer flask. **2.** Add Bromine Water drop-wise with constant swirling until a permanent yellow color develops. **3.** Add Phenol Solution drop-wise until the yellow color just disappears. Proceed with step 4 of the procedure (step 3 if using the AccuVac procedure).
Extreme pH or highly buffered samples	May exceed buffering capacity of reagents. See Section 1, *pH Interferences*. Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause negative interference
The following do not interfere in concentrations up to 1000 mg/L: Pyrophosphate, tetraborate, selenate benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, Al^{3+}, Fe^{3+}, Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+}, Li^{+}, Na^{+}, K^{+}, NH_4^{+}, Cd^{2+}, Mn^{2+}, NO_3^{-}, NO_2^{-}, SO_4^{2-}, SO_3^{2-}, Pb^{2+}, Hg^{+}, Hg^{2+}, Sn^{2+}, Cu^{2+}, Ni^{2+}, Ag^{+}, U^{4+}, Zr^{4+}, AsO_3^{-}, Br^{-}, CO_3^{2-}, $Cl0_4^{-}$, CN^{-}, IO_3^{-}, SiO_4^{4-}.	

Summary of Method

In the molybdovanadate method, orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid is formed. The intensity of the yellow color is proportional to the phosphate concentration.

REQUIRED REAGENTS AND APPARATUS (using Reagent Solution)

Description	Quantity Required Per Test		Units	Cat. No.
Molybdovanadate Reagent	2.0 mL	100 mL*	MDB	20760-32
Sample Cell, 10-20-25 mL, w/ cap	2		6/pkg	24019-06
Water, deionized	25 mL		4 L	272-56

REQUIRED REAGENTS AND APPARATUS (using AccuVac Ampuls)

Molybdovanadate Reagent AccuVac Ampuls	2		25/pkg	25250-25
Beaker, 50 mL	2		each	500-41H
Water, deionized	25 mL		4 L	272-56

OPTIONAL REAGENTS

Description	Units	Cat. No.
Bromine Water, 30 g/L	29 mL*	2211-20
Hydrochloric Acid Solution, 1:1 (6.0 N)	500 mL	884-49
Phenol Solution, 30 g/L	29 mL	2112-20
Phosphate Standard Solution, 10.0 mg/L as PO_4^{3-}	946 mL	14204-16
Phosphate Standard Solution, Voluette Ampule, 500 mg/L as PO_4^{3-}, 10 mL	16/pkg	14242-10
Sodium Hydroxide Standard Solution, 5.0 N	100 mL* MDB	2450-32
Sulfuric Acid, ACS	500 mL*	979-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49

OPTIONAL APPARATUS

AccuVac Snapper Kit	each		24052-00
Ampule Breaker Kit	each		21968-00
Cylinder, graduated, 25 mL	each		508-40
Cylinder, graduated, mixing, 25-mL	each		20886-40
Dispenser, fixed volume, 1.0 mL Repipet Jr.	each		21113-02
Flask, erlenmeyer, 50 mL	each		505-41
Flask, volumetric, Class A, 50 mL	each		14574-41
pH Paper, 1 to 11 pH units	5 rolls/pkg		391-33
pH Meter, *Sens**ion**™1*, portable with electrode	each		51700-10

* Contact Hach for larger sizes.

OPTIONAL APPARATUS (continued)

Description	Units	Cat. No.
Pipet, serological, 2.0 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Thermometer, -20 to 110 °C	each	26357-02

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, REACTIVE, HR (0.0 to 100.0 mg/L PO_4^{3-})

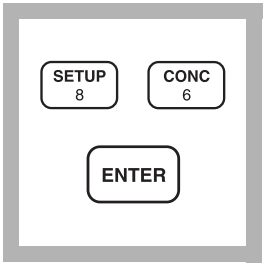
Molybdovanadate Method*, Test 'N Tube™ Procedure **For water and wastewater**

1. Enter the stored program number for phosphorus, reactive, high range, Test 'N Tube.

Press: **PRGM**

The display will show:

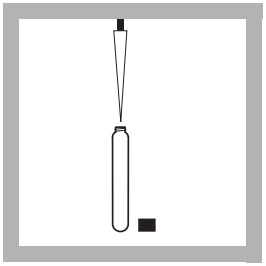
PRGM ?



2. Press: **86 ENTER**

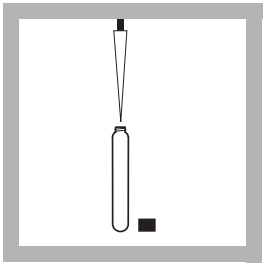
The display will show **mg/L, PO4** and the **ZERO** icon.

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*



3. Use a TenSette® Pipet to add 5.0 mL of deionized water to a Reactive High Range Phosphorus Test 'N Tube Vial (the blank).

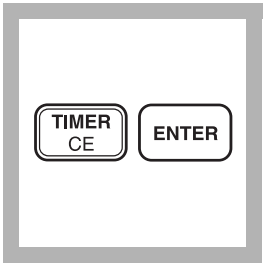
Cap and invert to mix.



4. Use a TenSette Pipet to add 5.0 mL of sample to a Reactive High Range Phosphorus Test 'N Tube Vial (the sample).

Cap and invert to mix.

Note: *For samples with extreme pH, see the Interference section.*



5. Press:

TIMER ENTER

A 7-minute reaction period will begin.

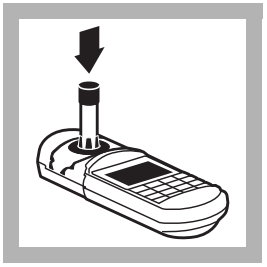
Note: *This reaction time is for samples at 23 °C (73 °F). If the sample temperature is 13 °C (55 °F), wait 15 minutes. If the sample temperature is 33 °C (91 °F), wait two minutes.*

6. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *A diffuser band covers the light path holes on the adapter to give increased performance. The band should NOT be removed.*

7. Clean the outside of the vials with a towel.

Note: *Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.*

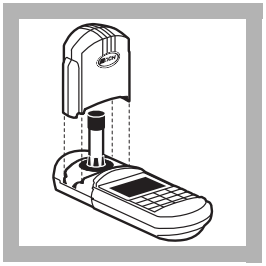


8. When the timer sounds, place the blank vial into the adapter.

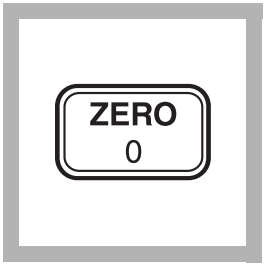
Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



9. Tightly cover the sample cell with the instrument cap.

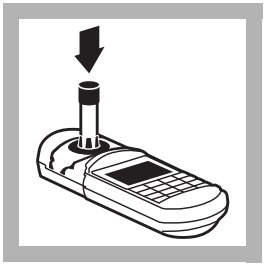


10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L PO4

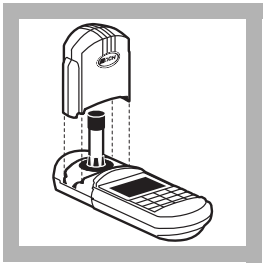
Note: *Reagent blanks for each lot of reagent may be used more than once. At room temperature, the reagent blank is stable for as long as three weeks; then prepare a new one.*



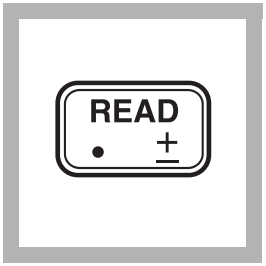
11. Place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Tightly cover the vial with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *For best results, use Standard Adjust with each new lot of reagent. (See* Accuracy Check.*)*

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning the glassware used in this test.

For best results, analyze the samples immediately after collection. If prompt analysis is impossible, preserve the samples for up to 748 hours by filtering immediately and storing at 4 °C. The sample should have a neutral (6–8) pH and be at room temperature before analysis.

Accuracy Check

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

Standard Additions Method

a. Fill three 10-mL graduated mixing cylinders with 10 mL of sample.

b. Snap the neck off a Voluette™ Ampule of Phosphate Standard Solution, 500 mg/L as PO_4^{3-} (Cat. No. 14242-10).

c. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 10-mL aliquots of sample prepared in *step a*. Mix well.

d. Analyze each sample from *step c* as described in the procedure; use 5.0 mL of the prepared sample for each test. The concentration should increase as follows: 5 mg/L, 10 mg/L, and 15 mg/L PO_4^{3-}, respectively.

e. If these increases do not occur, see *Standard Additions* in *Section 1* of the *DR/890 Procedures Manual* for more information.

Standard Solution Method

To check accuracy, prepare an 80 mg/L PO_4^{3-} standard by pipetting 8.0 mL of solution from a 10-mL Voluette Ampule of Phosphate Standard Solution, 500 mg/L as PO_4^{3-}, into an acid-cleaned 50-mL Class A volumetric flask. Fill to the line with deionized water. Substitute this standard for the sample and perform the procedure as described.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 80 mg/mL PO_4^{3-} standard solution, press the **SETUP** key and

scroll, using the arrow keys, to the **STO** option. Press **ENTER** to activate the standard adjust option. Then enter 80.0 to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment, Section 1* of the *Procedures Manual* for more information.

Interferences

Large amounts of sample turbidity may cause inconsistent results in the test because the acid present in the reagents may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.

The following may interfere when present in concentrations exceeding these listed below:

Substance	Interference Level and Treatment
Arsenate	Causes positive interference if the sample is heated.[1]
Iron, ferrous	Blue color caused by ferrous iron does not interfere if the iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Causes positive interference if the sample is heated.*
Sulfide	Causes a negative interference. Remove interference as follows: **1.** Measure 50 mL of sample into an Erlenmeyer flask. **2.** Add Bromine Water drop-wise with constant swirling until a permanent yellow color develops. **3.** Add Phenol Solution drop-wise until the yellow color just disappears. Proceed with *step 1* of the procedure.
Extreme pH or highly buffered samples	May exceed buffering capacity of the reagents. See *pH Interferences* in *Section 1* of the *DR/890 Procedure Manual*. Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause a negative interference.
Temperature, cold (less than 20 °C)	Causes a negative interference.
Temperature, hot (greater than 25 °C)	Causes a positive interference.
The following do not interfere in concentrations up to 1000 mg/L: Pyrophosphate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, Al^{3+}, Fe^{3+}, Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+}, Li^{+}, Na^{+}, K^{+}, NH_4^{+}, Cd^{2+}, Mn^{2+}, NO_3^{-}, NO_2^{-}, SO_4^{2-}, SO_3^{2-}, Pb^{2+}, Hg^{+}, Hg^{2+}, Sn^{2+}, Cu^{2+}, Ni^{2+}, Ag^{+}, U^{4+}, Zr^{4+}, AsO_3^{-}, Br^{-}, CO_3^{2-}, ClO_4^{-}, CN^{-}, IO_3^{-}, SiO_4^{4-}.	

[1] Gentle warming of the sample to reach room temperature will not cause this substance to interfere.

Method Performance

Precision

In a single laboratory, using a standard solution of 80.0 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±3.0 mg/L PO_4^{3-}.

Estimated Detection Limit (EDL)

The EDL for program 86 is 7.0 mg/L PO_4^{3-}. For more information on derivation and use of Hach's estimated detection limit, see *Section 1* of the *DR/890 Procedures Manual*.

Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Consult the Material Safety Data Sheet for information specific to the reagent used.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet for information specific to the reagents used.

Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid forms. The intensity of the yellow color is proportional to the phosphate concentration.

Installing this Program on the DR/800

This procedure will add the current method as a new Hach program to your DR/800.

1. Turn the DR/800 on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key two times so that the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.

6. Enter each of the numbers in the right column, each followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time you may use the arrow keys to scroll back to review or change any number you have already entered.

Line Number	Entry	Line Number	Entry
1	86	29	0
2	4	30	80
3	73	31	50
4	0	32	79
5	0	33	53
6	0	34	0
7	0	35	62
8	65	36	166
9	56	37	246
10	217	38	148
11	21	39	63
12	66	40	63
13	157	41	78
14	197	42	252
15	30	43	4
16	0	44	76
17	0	45	128
18	0	46	0
19	0	47	15
20	80	48	1
21	79	49	164
22	52	50	0
23	0	51	0
24	0	52	0
25	80	53	0
26	0	54	80
27	0	55	0
28	0	56	255

REQUIRED REAGENTS

High Range Reactive Phosphorus Test 'N Tube™ Reagent Set.....50 vials.....27673-45
Includes: (50) Reactive High Range Phosphorus Test 'N Tube™ Vials*, (2) 272-42

Description	Quantity Required Per Test	Unit	Cat. No.
Reactive High Range Phosphorus Test 'N Tube™ Vials	1	50/pkg	*
Water, deionized		100 mL	272-42

REQUIRED APPARATUS

COD/TNT Adapter for DR/800 Series	1	each	48464-00
Pipet, TenSette®, 1 to 10 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette® Pipet	1	50/pkg	21997-96
Test Tube Rack	1–3	each	18641-00

OPTIONAL REAGENTS

Bromine Water, 30 g/L	29 mL**	2211-20
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phenol Solution, 30 g/L	29 mL	2112-20
Phosphate Standard Solution, PourRite ampule, 500 mg/L as PO_4^{3-}, 2 mL	20/pkg	14242-20
Phosphate Standard Solution, Voluette ampule, 500 mg/L as PO_4^{3-}, 10 mL	16/pkg	14242-10
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49

OPTIONAL APPARATUS

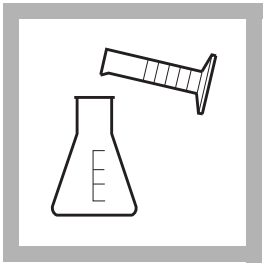
Ampule Breaker Kit	each	21968-00
Aspirator, vacuum	each	2131-00
Cylinder, graduated, mixing, 10 mL, 3 required	each	20886-38
Filter Holder, 47 mm, 300 mL, graduated	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	200/pkg	13530-00
Flask, filtering, 500 mL	each	546-49
Flask, volumetric, Class A, 50-mL	each	14574-41
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion**™1*, portable with electrode	each	51700-10
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette® Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette® Pipet	1000/pkg	21856-28
Pipet Tips, for 19700-10 TenSette® Pipet	250/pkg	21997-25
Pipet, volumetric, Class A, 5.00-mL	each	14515-37
Pipet, volumetric, Class A, 8.00-mL	each	14515-08
PourRite™ Ampule Breaker	each	24846-00

* These items are not sold separately.
** Larger sizes available.

PHOSPHORUS, ACID HYDROLYZABLE

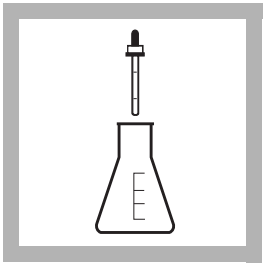
Hydrolysis to Orthophosphate Method*

For water, wastewater, seawater



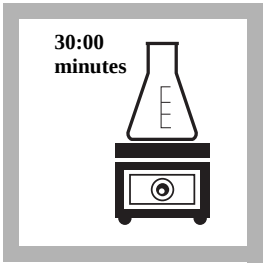
1. Measure 25 mL of sample into a 50-mL erlenmeyer flask using a graduated cylinder.

Note: *Wash all glassware with 6 N hydrochloric acid. Rinse with deionized water. Do not use detergents containing phosphate to clean glassware.*



2. Add 2.0 mL of 5.25 N Sulfuric Acid Solution.

Note: *Use the 1-mL calibrated dropper provided.*

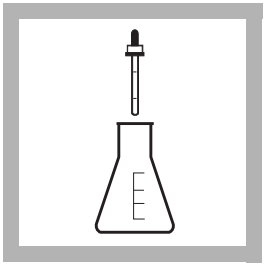


3. Place the flask (the prepared sample) on a hot plate. Boil gently for 30 minutes.

Note: *Samples should be concentrated to less than 20 mL for best recovery. After concentration, maintain the volume near 20 mL by adding small amounts of deionized water. Do not exceed 20 mL.*

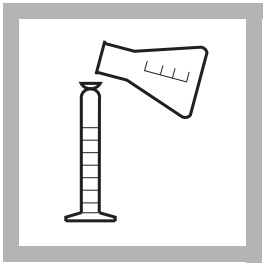
4. Cool the hot prepared sample to room temperature.

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



5. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution to the prepared sample. Swirl to mix.

Note: *Use the 1-mL calibrated dropper provided.*



6. Pour the prepared sample into a graduated cylinder. Add deionized water rinsings from the flask to return the volume to 25 mL. Proceed with the appropriate reactive phosphorus test.

Note: *Results of the reactive phosphorus test at this point will include the orthophosphate plus the acid-hydrolyzable (condensed) phosphate. The condensed phosphate concentration is determined by subtracting the results of a reactive phosphorus test on an untreated sample from this result. Make sure both results are in the same chemical form and units.*

Sampling and Storage

Analyze samples immediately after collection for best results. If prompt analysis is not possible, samples may be preserved up to 48 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

Interferences

If the sample is turbid, use 50 mL of sample and double the reagent volumes. Use the hydrolyzed sample to zero the instrument in the reactive phosphorus procedure. This compensates for any turbidity dissolved by this procedure.

Summary of Method

This procedure lists the necessary steps to convert condensed phosphate forms (meta-, pyro- or other polyphosphates) to reactive orthophosphate before analysis. The procedure uses acid and heat to hydrolyze the sample. Organic phosphates are not converted to orthophosphate by this process, but a very small fraction may be unavoidably included in the result. Thus, the "acid hydrolyzable" phosphate results are primarily a measure of inorganic phosphorus. This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorous content of the sample.

The following reagents and apparatus are required in addition to those required for the reactive phosphorus test.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Drinking Water Standard, Inorganic, F^-, $NO_3^{-N,}$ PO_4^{3-}, SO_4^{2-}	500 mL		28330-49
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL* MDB	2450-32
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL* MDB	2449-32
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL		28332-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL		28331-49

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	2	each	508-40
Flask, erlenmeyer, 50 mL	1	each	505-41

OPTIONAL REAGENTS

Hydrochloric Acid, 6 N	500 mL		884-49
Water, deionized	4 L		272-56

OPTIONAL APPARATUS

Cylinder, graduated, 50 mL		each	508-41
Flask, erlenmeyer, 125 mL		each	505-43
Hot Plate, 4" diameter, 120 Vac		each	12067-01
Hot Plate, 4" diameter, 240 Vac		each	12067-02
Pad, cooling, 4" x 4"		each	18376-00
pH indicator Paper, 1 to 11 pH	5	rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable with electrode		each	51700-10
Thermometer, -20 to 110 °C, Non-Mercury		each	26357-02

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

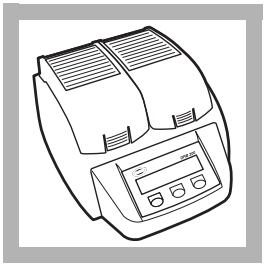
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes.

PHOSPHORUS, ACID HYDROLYZABLE (0.00 to 5.00 mg/L PO_4^{3-})

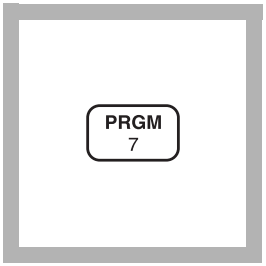
PhosVer 3 with Acid Hydrolysis Test 'N Tube™ Procedure

For water, wastewater, and seawater



1. Turn on the COD Reactor. Heat to 150 °C.

Note: *See DRB200 instrument manual for selecting preprogrammed temperature applications.*



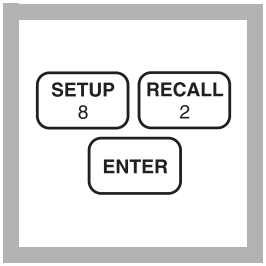
2. Enter the stored program number for acid hydrolyzable phosphorus (PO_4^{3-}), Test 'N Tube.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



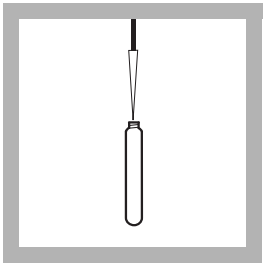
3. Press: **82 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

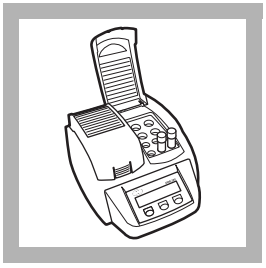
Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*

4. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

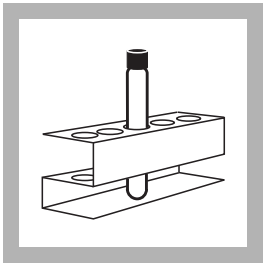
Note: *A diffuser band covers the light path holes on the adapter to give increased performance. The band should NOT be removed.*



5. Use a TenSette Pipet to add 5.0 mL of sample to a Total and Acid Hydrolyzable Test Vial. Cap and mix.

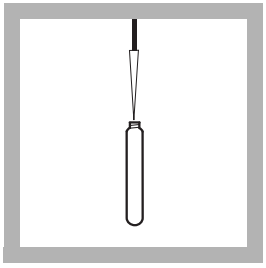


6. Heat the vial in the DRB200 Reactor for 30 minutes.

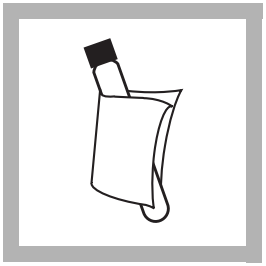


7. Carefully remove the vial from the reactor. Place it in a test tube rack and allow to cool to room temperature.

Note: *Vials will be hot.*

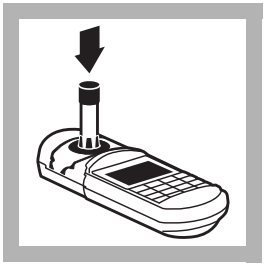


8. Remove the cap from the vial. Use a TenSette Pipet to add 2.0 mL of 1.00 N sodium hydroxide to the vial. Cap and mix.



9. Clean the outside of the vial with a towel.

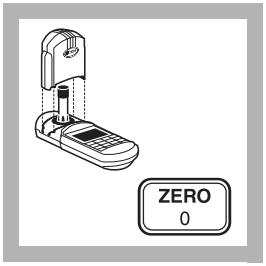
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



10. Place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



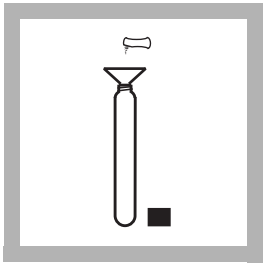
11. Tightly cover the vial with the instrument cap.

Press: **ZERO**

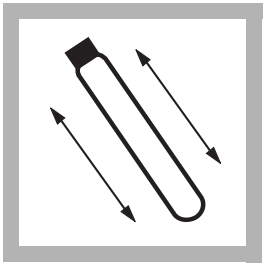
The cursor will move to the right, then the display will show:

0.00 mg/L PO4

Note: *For multiple samples, zero on the first sample. Read the remaining samples after adding the PhosVer 3 reagent. Subtract the reagent blank value from each reading.*

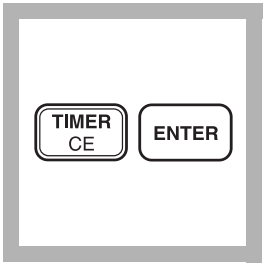


12. Remove the cap from the vial. Using a funnel, add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to the vial.



13. Cap tightly and shake for 10-15 seconds.

Note: *The powder will not completely dissolve.*



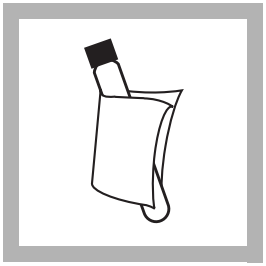
14. Press:

TIMER ENTER

A 2-minute reaction period will begin.

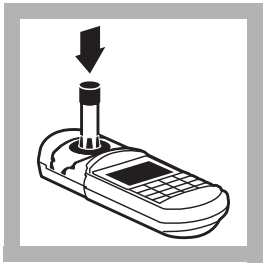
Note: *Read samples between 2 and 8 minutes after adding the PhosVer 3 reagent.*

Note: *A blue color will form if phosphate is present.*



15. After the timer beeps, clean the outside of the sample vial with a towel.

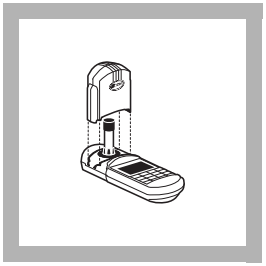
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



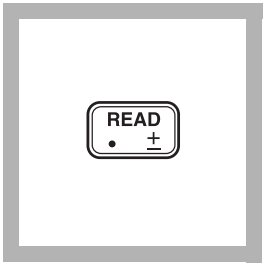
16. Place the prepared sample in the adapter

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



17. Tightly cover the vial with the instrument cap.



18. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water.
Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, the sample may be preserved up to 48 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

Accuracy Check

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse with deionized water. Do not use detergents containing phosphate to clean glassware.*

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as PO_4^{3-}.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.

d) Analyze each sample as described in the procedure. Use 5.0 mL of the prepared standard additions for each test; the concentration should increase as follows: 0.2 mg/L, 0.4 mg/L, and 0.6 mg/L PO_4^{3-}, respectively.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Obtain a 1.0 mg/L Phosphate Standard Solution listed under *Optional Reagents*. Or, this can be prepared by pipetting 2 mL of a Voluette Ampule Standard for Phosphate, 50 mg/L as PO_4^{3-}, into an acid washed Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Substance	Interference Level and Treatment
Aluminum	200 mg/L
Arsenate	Interferes at any level.
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Sulfide	9 mg/L. Sulfide interference may be removed by oxidation with Bromine Water as follows: 1. Measure 25 mL of sample into a 50-mL beaker. 2. Swirling constantly, add Bromine Water drop-wise until a permanent yellow color develops. 3. Swirling constantly, add Phenol Solution dropwise until the yellow color just disappears. Proceed with *step 1*.

Turbidity (large amounts)	May cause inconsistent results because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see pH Interferences (Section 1).

The PhosVer 3 Phosphate Reagent Powder Pillows should be stored in a cool, dry environment.

Method Performance

Precision

In a single laboratory, using a standard solution of 3.00 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.06 mg/L PO_4^{3-}.

Estimated Detection Limit

The estimated detection limit for program 82 is 0.07 mg/L PO_4^{3-}. For more information on the estimated detection limit, see *Section 1*.

Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS

Total and Acid Hydrolyzable Test 'N Tube Reagent Set ..... 50 tests 27427-45
Includes: (1) 272-42, (1) 1045-42, (1) 20847-66, (1) 21060-46, (50) Total and Acid Hydrolyzable Test Vials* (1) 27430-42

Description	Quantity Required Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows	1	50/pkg	21060-46
Potassium Persulfate powder Pillows	1	50/pkg	20847-66
Sodium Hydroxide Solution, 1.0 N	2 mL	100 mL	1045-42
Total and Acid Hydrolyzable Test Vials	1	50/pkg	*
Water, deionized for reagent blanks	5 mL	100 mL	272-42

REQUIRED APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
COD/TNT Adapter	1	each	48464-00
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Funnel, micro	1	each	25843-35
Pipet, TenSette, 1 to 10 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet		50/pkg	21997-96
Test Tube Rack	1-3	each	18641-00

OPTIONAL REAGENTS

Bromine Water, 30 g/L ..... 29 mL 2211-20
Drinking Water Standard, Inorganic, F^-, $NO_3^{-N,}$ PO_4^{3-}, SO_4^{2-} 500mL 28330-49
Hydrochloric Acid Standard Solution, 6.0 N (1:1) ..... 500 mL 884-49
Phenol Solution, 30 g/L ..... 29 mL 2112-20
Phosphate Standard Solution, 1 mg/L as PO_4^{3-} ..... 500 mL 2569-49
Phosphate Standard Solution, PourRite ampule,
50 mg/L as PO_4^{3-}, 2 mL ..... 20/pkg 171-20H
Phosphate Standard Solution, Voluette ampule,
50 mg/L as PO_4^{3-}, 10 mL 16/pkg 171-10
Sodium Hydroxide Standard Solution, 5.000 N ..... 1000 mL 2450-53
Sulfuric Acid Standard Solution, 1.000 N ..... 1 L 1270-53
Wastewater Effluent Standard, Inorganic
(NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC) ..... 500 mL 28332-49
Water, deionized ..... 4 L 272-56

* These items are not sold separately.

OPTIONAL APPARATUS

Description	Units	Cat. No.
Ampule Breaker Kit, Voluette	each	21968-00
Ampule Breaker, PourRite	each	24846-00
Cylinder, graduated, mixing, 25 mL (3 required)	each	20886-40
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm		LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm		LTV082.52.30001
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable with electrode	each	51700-10
Pipet, TenSette, 0.1-1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet Filler, safety bulb	each	14651-00

For Technical Assistance, Price and Ordering

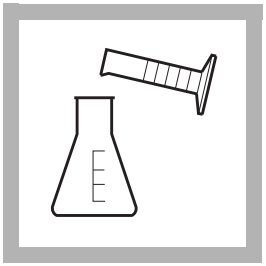
In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, TOTAL

(Also called Organic and Acid Hydrolyzable) Acid Persulfate Digestion Method*

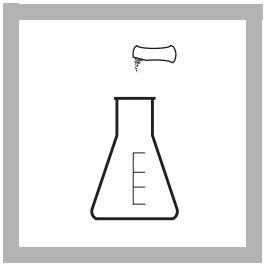
USEPA Accepted for reporting wastewater analysis**



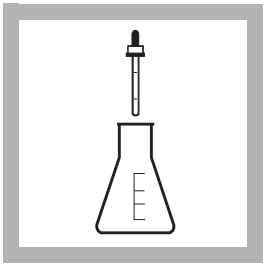
1. Measure 25 mL of sample into a 50-mL erlenmeyer flask using a graduated cylinder.

Note: *Rinse all glassware with 1:1 Hydrochloric Acid Solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

Note: *Adjust the pH of stored samples before digestion.*



2. Add the contents of one Potassium Persulfate Powder Pillow. Swirl to mix.



3. Add 2.0 mL of 5.25 N Sulfuric Acid Solution.

Note: *Use the 1-mL calibrated dropper provided.*

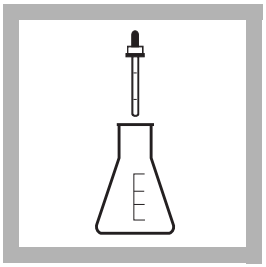
4. Place the flask on a hot plate. Boil gently for 30 minutes.

Note: *Samples should be concentrated to less than 20 mL for best recovery. After concentration, maintain the volume near 20 mL by adding small amounts of deionized water. Do not exceed 20 mL.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

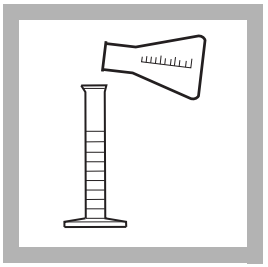
** Procedure is equivalent to USEPA Method 365.2 and Standard Method 4500-P B,5 & P E.

5. Cool the sample to room temperature.



6. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution. Swirl to mix.

Note: *Use the 1-mL calibrated dropper provided.*



7. Pour the sample into a 25-mL graduated cylinder. Return the volume to 25 mL. Proceed with a reactive phosphorus test of the expected total phosphorus concentration range.

Note: *Use deionized water rinsings from the flask to adjust the volume.*

Note: *Results of the reactive phosphorus test at this point will include the organic phosphate plus the orthophosphate and the acid-hydrolyzable (condensed) phosphate. The organic phosphate concentration is determined by subtracting results of an acid hydrolyzable phosphorus test from this result. Make sure that both results are in the same units before taking the difference.*

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid-washed with 1:1 HCl and rinsed with deionized water. Do not use detergents containing phosphates for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples up to 28 days by adjusting the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter) and storing at 4 °C. Warm to room temperature before testing. Correct results for volume additions; see *Volume Additions* (Section 1) for more information.

Interferences

For turbid samples, use 50 mL of sample and double the reagent quantities. Use digested sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed by this procedure. For alkaline or highly buffered samples it may be necessary to add additional acid in Step 3 to drop the pH of the solution below 1.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate. Organically bound phosphates are thus determined indirectly by subtracting the result of an acid hydrolyzable phosphorus test from the total phosphorus result.

This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorus content of the sample. If the ascorbic acid (PhosVer 3) method is used to measure the reactive phosphorus, this method is EPA approved for NPDES reporting.

The following reagents and apparatus are required in addition to those required for the reactive phosphorus test.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Potassium Persulfate Powder Pillows	1 pillow	100/pkg	2451-99
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL* MDB	2450-32
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL* MDB	2449-32

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	2	each	508-40
Flask, erlenmeyer, 50 mL	1	each	505-41
Sample Cell, 10-20-25 mL, w/caps	2	6/pkg	24019-06

OPTIONAL REAGENTS

Drinking Water Standard, Inorganic, F^-, $NO_3^{-N,}$ PO_4^{3-}, SO_4^{2-}		500mL	28330-49
Hydrochloric Acid, 6 N		500 mL	884-49
Sodium Hydroxide Solution, 5.0 N		1 L	2450-53
Sulfuric Acid		500 mL	979-49
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28332-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28331-49
Water, deionized		4 L	272-56

* Marked Dropper Bottle - Contact Hach for larger sizes.

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Cylinder, graduated, 50 mL	each	508-41
Flask, erlenmeyer, 125 mL	each	505-43
Hot Plate, 4" diameter, 120 Vac	each	12067-01
Hot Plate, 4" diameter, 240 Vac	each	12067-02
Pads, cooling, 4 x 4"	each	18376-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *Sens**ion***™*1*, portable with electrode	each	51700-10

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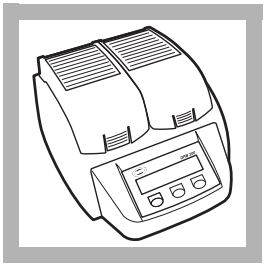
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

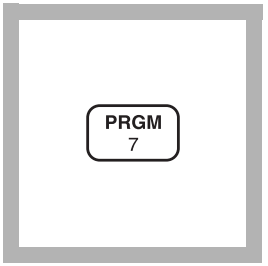
PHOSPHORUS, TOTAL (0.00 to 3.50 mg/L PO_4^{3-}) For water, wastewater and seawater

PhosVer 3 with Acid Persulfate Digestion* USEPA Accepted for reporting wastewater analysis** Test 'N Tube Procedure



1. Turn on the DRB200 Reactor. Heat the reactor to 150 °C.

Note: *See DRB200 instrument manual for selecting preprogrammed temperature applications.*



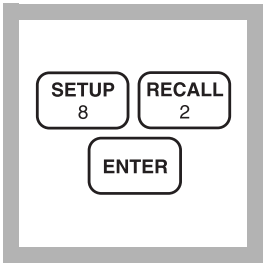
2. Enter the stored program number for total phosphorus, (PO_4^{3-}), Test 'N Tube.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



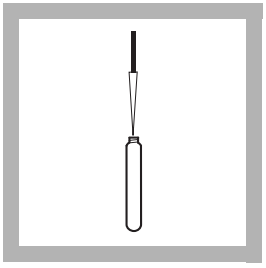
3. Press: **82 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*

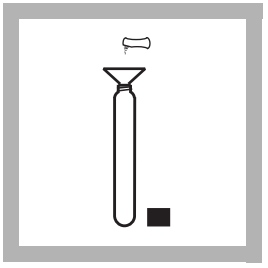
4. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *A diffuser band covers the light path holes on the adapter to give increased performance. The band should NOT be removed.*

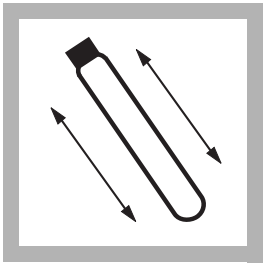


5. Use a TenSette Pipet to add 5.0 mL of sample to a Total and Acid Hydrolyzable Test Vial.

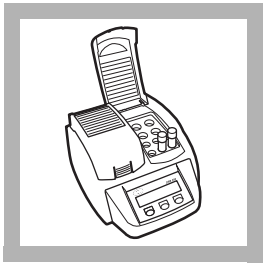
Note: *Adjust the pH of stored samples to 6-8 before analysis.*



6. Using a funnel, add the contents of one Potassium Persulfate Powder Pillow for Phosphonate to the vial.



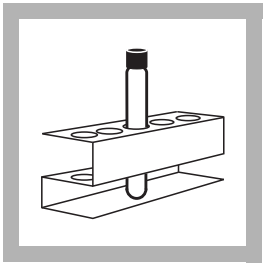
7. Cap tightly and shake to dissolve.



8. Place the vial in the DRB200 Reactor. Heat the vial for 30 minutes.

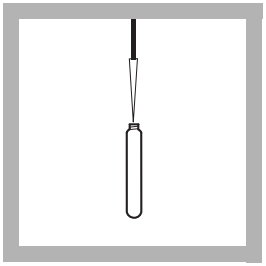
* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

** Procedure is equivalent to USEPA Method 365.2 and Standard Method 4500-P B, 5 and P.E.

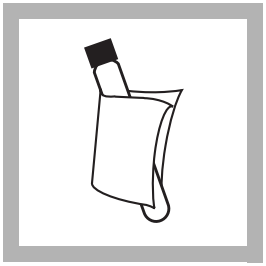


9. Carefully remove the vial from the reactor. Place it in a test tube rack and allow to cool to room temperature.

Note: *Vials will be hot.*

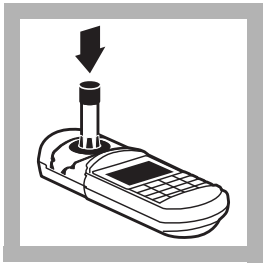


10. Use a TenSette Pipet to add 2.0 mL of 1.54 N sodium hydroxide to the vial. Cap and mix.



11. Clean the outside of the vial with a towel.

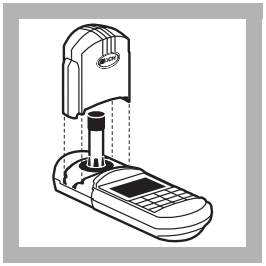
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



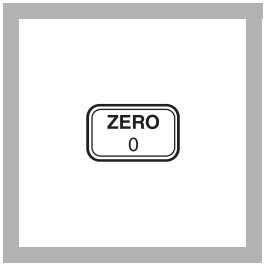
12. Place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



13. Tightly cover the vial with the instrument cap.

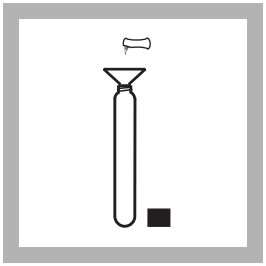


14. Press: **ZERO**

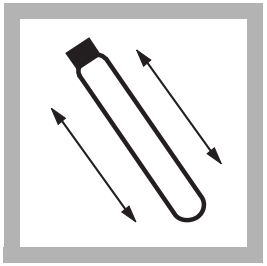
The cursor will move to the right, then the display will show:

0.00 mg/L PO4

Note: *For multiple samples, zero only on the first sample. Read the remaining samples after adding the PhosVer 3 reagent.*

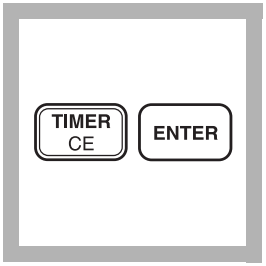


15. Remove the cap from the vial. Using a funnel, add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to the vial.



16. Cap tightly and shake for 10-15 seconds.

Note: *The powder will not completely dissolve.*



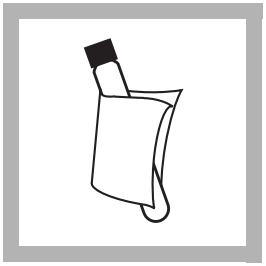
17. Press:

TIMER ENTER

A 2-minute waiting period will begin.

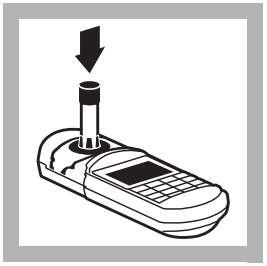
Note: *Read samples between 2 and 8 minutes after the addition of the PhosVer 3 reagent.*

Note: *A blue color will form if phosphate is present.*



18. After the timer beeps, clean the outside of the sample vial with a towel.

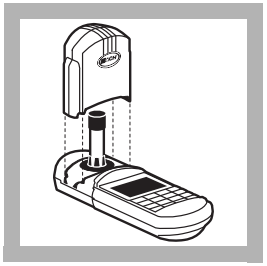
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



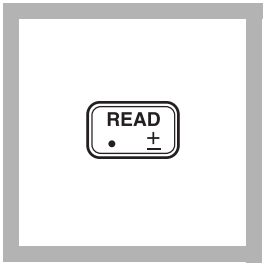
19. Place the prepared sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



20. Tightly cover the vial with the instrument cap.



21. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

IMPORTANT NOTE:

The test range for total phosphate is limited to 0 to 3.5 mg/L PO_4^{3-}. Values above 3.5 mg/L may be used to estimate dilution ratios, but should NOT be used for reporting purposes. If a value above 3.5 mg/L PO_4^{3-} is obtained, dilute the sample and repeat the digestion and the colorimetric test.

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphates for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve the sample for up to 28 days by adjusting the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter) and storing at 4 °C. Neutralize and warm the sample to room temperature before analysis. Correct test results for volume additions; see *Volume Additions* in *Section 1*.

Accuracy Check

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

Standard Additions Method

a) Fill three 25 mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as PO_4^{3-}.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.

d) Analyze each sample as described in the procedure using 5.0 mL of the prepared standard additions for each test. The concentration should increase 0.2 mg/L, 0.4 mg/L, and 0.6 mg/L PO_4^{3-}, respectively.

e) If these increases do not occur, see *Standard Additions* (Section 1).

Standard Solution Method

To check accuracy, use a 1.0 mg/L Phosphate Standard Solution (see Optional Reagents). Or, prepare a standard by pipetting 2 mL of solution a Voluette Ampule Standard for Phosphate Standard, 50 mg/L as PO_4^{3-}, into an acid-cleaned Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described. The mg/L PO_4^{3-} reading should be 1.0 mg/L.

OR

Prepare a 2.5 mg/L standard solution by pipetting 5 mL of a 50-mg/L Phosphate Voluette Ampule Standard into an

acid-washed 100-mL Class A volumetric flask. Dilute to the mark with deionized water.

Method Performance

Precision

In a single laboratory, using a standard solution of 3.00 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.06 mg/L PO_4^{3-}.

Estimated Detection Limit

The estimated detection limit for program 82 is 0.07 mg/L PO_4^{3-}. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Substance	Interference Level and Treatment
Aluminum	200 mg/L
Arsenate	Interferes at any level.
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Sulfide	90 mg/L
Turbidity (large amounts)	May cause inconsistent results because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see pH Interferences (Section 1).

Store PhosVer 3 Reagent Powder Pillows in a cool, dry environment.

Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS

Total Phosphorus Test 'N Tube Reagent Set ..... 50 tests 27426-45
Includes: (1) 272-42, (1) 20847-66, (1) 21060-46, (1) 27430-42, (50) Acid Dilution Vials*

Description	Quantity Required Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows	1	50/pkg	21060-46
Potassium Persulfate powder Pillows	1	50/pkg	20847-66
Sodium Hydroxide Solution, 1.54 N	2 mL	100 mL	27430-42
Test 'N Tube Acid Dilution Vials	1	50/pkg	*
Water, deionized for reagent blank	5 mL	100 mL	272-42

REQUIRED APPARATUS

COD/TNT Adapter	1	each	48464-00
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Funnel, micro	1	each	25843-35
Test Tube Rack	1-3	each	18641-00
Pipet, TenSette, 1 to 10 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet	varies	50/pkg	21997-96

* These items are not sold separately.

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Drinking Water Standard, Inorganic, F^-, NO_3^{-N}, PO_4^{3-}, SO_4^{2-}	500mL	28330-49
Total and Acid Hydrolyzable Test 'N Tube Reagent Set	each	27427-45
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phosphate Standard Solution, 1 mg/L as PO_4^{3-}	500 mL	2569-49
Phosphate Standard Solution, PourRite ampule, 50 mg/L as PO_4^{3-}, 2 mL	20/pkg	171-20H
Phosphate Standard Solution, Voluette ampule, 50 mg/L as PO_4^{3-}, 10 mL	16/pkg	171-10
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53
Total and Acid Hydrolyzable Test 'N Tube Reagent Set	each	27427-45
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Ampule Breaker, PourRite ampules	each	24846-00
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm		LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm		LTV082.52.30001
Cylinder, graduated, mixing, 25 mL (3 required)	each	20886-40
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *Sens**ion**™1*, portable with electrodes	each	51700-10
Pipet Filler, safety bulb	each	14651-00
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet, TenSette, 0.1-1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28

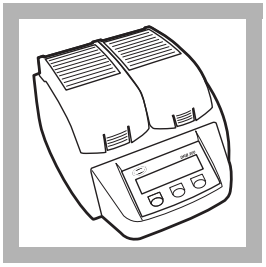
For Technical Assistance, Price and Ordering

In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, TOTAL, HR (0.0 to 100.0 mg/L PO_4^{3-})

Molybdovanadate Method with Acid Persulfate Digestion*
Test 'N Tube™ Procedure **For water and wastewater**



1. Turn on the DRB200 Reactor. Heat to 150 °C.

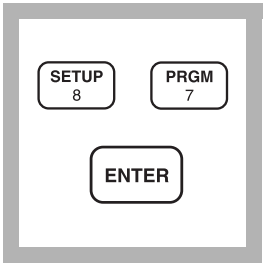
Note: *See DRB200 instrument manual for selecting preprogrammed temperature applications*

2. Enter the stored program number for phosphorus total high range, Test 'N `Tube.

Press: **PRGM**

The display will show:

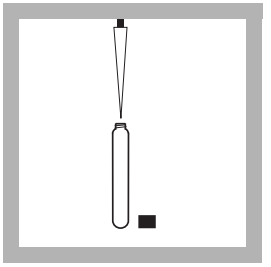
PRGM ?



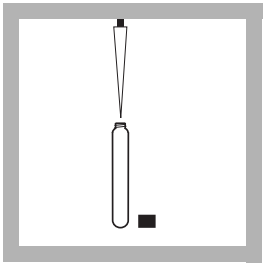
3. Press: **87 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon.

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*

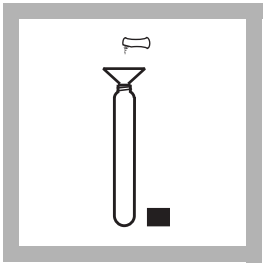


4. Use a TenSette® Pipet to add 5.0 mL of deionized water to a Total Phosphorus Test 'N Tube Vial (the blank).

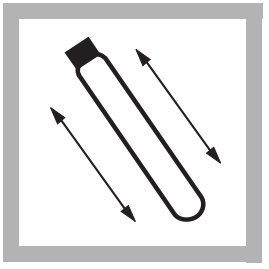


5. Use a TenSette Pipet to add 5.0 mL of sample to a Total Phosphorus Test 'N Tube Vial (the sample).

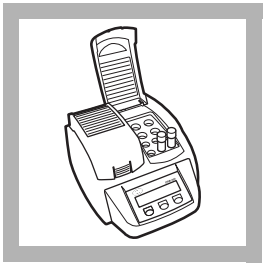
Note: *Adjust the pH of stored samples to 6–8 before analysis.*



6. Use a funnel to add the contents of one Potassium Persulfate Powder Pillow for Phosphonate to each vial.



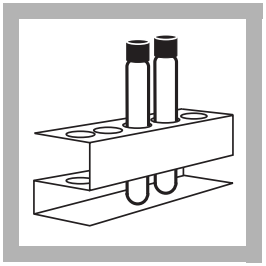
7. Cap tightly and shake to dissolve.



8. Place the vials in the DRB200 Reactor. Heat for 30 minutes.

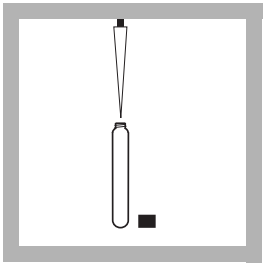
Press: **TIMER ENTER** to time the heating period.

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



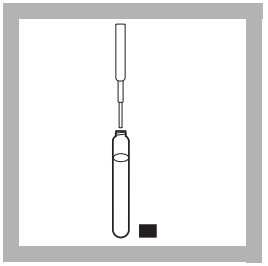
9. Carefully remove the vials from the reactor. Place them in a test tube rack and allow to cool to room temperature (18–25 °C).

Note: *Vials will be hot.*



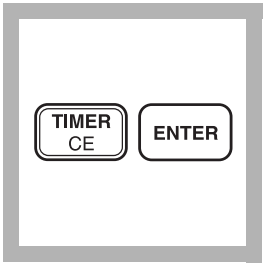
10. Use a TenSette Pipet to add 2.0 mL of 1.54 N sodium hydroxide to each vial.

Cap and invert to mix.



11. Use a polyethylene dropper to add 0.5 mL of Molybdovanadate Reagent to each vial.

Cap and invert to mix.



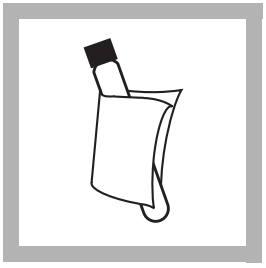
12. Press:

TIMER ENTER

A 7-minute reaction period will begin.

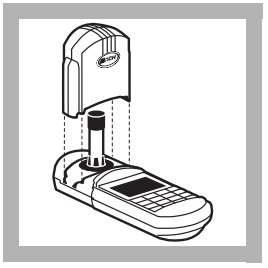
Note: *Read the samples between 7 and 9 minutes.*

13. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.



14. Clean the outside of the vials with a towel.

Note: *Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.*

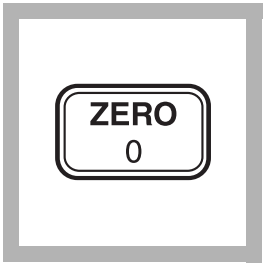


15. When the timer sounds, place the blank vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Tightly cover the vial with the instrument cap.

Note: *Do not move the vial from side to side as this can cause errors.*

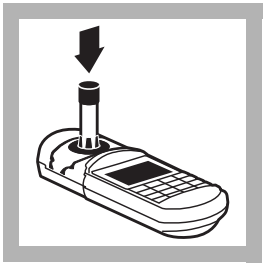


16. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L PO4

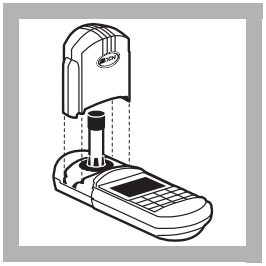
Note: *Reagent blanks for each lot of reagents may be used more than once, but should not be used for longer than one day.*



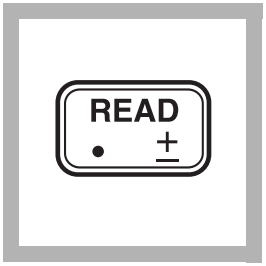
17. Place the prepared sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



18. Tightly cover the vial with the instrument cap.



19. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *For best results, use Standard Adjust with each new lot of reagent. (See* Accuracy Check.*)*

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphates for cleaning the glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve the sample for up to 28 days by adjusting the pH to 2 or less with concentrated H_2SO_4 (about 2 mL per liter) and storing at 4 °C. Warm the sample to room temperature and neutralize with 5.0 N NaOH before analysis.

Correct test results for volume additions; see *Volume Additions* in *Section 1*of the *DR/890 Procedures Manual*.

Accuracy Check

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

Standard Additions Method

a. Fill each of three 10-mL graduated mixing cylinders with 10 mL of sample.

b. Snap the neck off a 10-mL Voluette® Ampule of Phosphate Standard Solution, 500 mg/L as PO_4^{3-} (Cat. No. 14242-10).

c. Use a TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 10-mL aliquots of the water sample prepared in *step a*. Mix well.

d. Analyze samples from *step c* as described in the procedure. Use 5.0 mL of the prepared sample for each test. The concentration should increase: 5 mg/L, 10 mg/L, and 15 mg/L PO_4^{3-}, respectively.

e. If these increases do not occur, see *Standard Additions (Section 1 of the DR/890 Procedures Manual*) for more information.

Standard Solution Method

To check accuracy, prepare an 80 mg/L standard by pipetting 8.0 mL of solution from a 10-mL Voluette® Ampule of Phosphate Standard Solution, 500 mg/L as PO_4^{3-} into an acid-cleaned, Class A, 50-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 80 mg/L PO_4^{3-} standard solution, press the **SETUP** key and scroll, using the arrow keys, to the **STO** option. Press **ENTER** to activate the standard adjust option. Then enter 80.0 to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment, Section 1* of the *Procedures Manual* for more information.

Interferences

Large amounts of sample turbidity may cause inconsistent results in the test because the acid present in the reagents may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.

The following may interfere when present in concentrations exceeding those listed below:

Interfering Substance	Interference Level and Treatment
Arsenate	Causes positive interference if the sample is heated.[1]
Iron, ferrous	Blue color caused by ferrous iron does not interfere if iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Causes positive interference if the sample is heated.*
Extreme pH or highly buffered samples	May exceed buffering capacity of the reagents. See *pH Interferences* in *Section 1* of the *DR/890 Procedures Manual*. Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause a negative interference.
Temperature, Cold (less than 18 °C)	Causes a negative interference.
Temperature, Hot (greater than 25 °C)	Causes a positive interference. Post-digestion samples should be brought to room temperature (18–25 °C) before the addition of the Molybdovanadate Reagent or sodium hydroxide.
The following do not interfere in concentrations up to 1000 mg/L: Pyrophosphate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, Al^{3+}, Fe^{3+}, Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+}, Li^{+}, Na^{+}, K^{+}, NH_4^{+}, Cd^{2+}, Mn^{2+}, NO_3^{-}, NO_2^{-}, SO_4^{2-}, SO_3^{2-}, Pb^{2+}, Hg^{+}, Hg^{2+}, Sn^{2+}, Cu^{2+}, Ni^{2+}, Ag^{+}, U^{4+}, Zr^{4+}, AsO_3^{-}, Br^{-}, CO_3^{2-}, ClO_4^{-}, CN^{-}, IO_3^{-}, SiO_4^{4-}.	

[1] Gentle warming of the sample to reach room temperature will not cause this substance to interfere.

Method Performance

Precision

In a single laboratory, using a standard solution of 80.0 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±3.0 mg/L PO_4^{3-}.

Estimated Detection Limit

The estimated detection limit for program 87 is 7.0 mg/L PO_4^{3-}. For more information on the estimated detection limit, see *Section 1* of the *DR/890 Procedures Manual*.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet for information specific to the reagents used.

Sample Disposal Information

The final samples will contain molybdenum. In addition, the final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Consult the Material Safety Data Sheet for information specific to the reagent used.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid forms. The intensity of the yellow color is proportional to the phosphate concentration.

Installing this Program on the DR/800

This procedure will add the current method as a new Hach program to your DR/800.

1. Turn the DR/800 on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key two times so that the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.
6. Enter each of the numbers in the right column, each followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time you may use the arrow keys to scroll back to review or change any number you have already entered.

Line Number	Entry	Line Number	Entry
1	87	18	0
2	4	19	0
3	73	20	80
4	0	21	79
5	0	22	52
6	0	23	0
7	0	24	0
8	0	25	80
9	0	26	0
10	0	27	0
11	0	28	0
12	66	29	0
13	175	30	80
14	48	31	50
15	32	32	79
16	0	33	53
17	0	34	0
35	62	46	0

Line Number	Entry	Line Number	Entry
36	166	47	15
37	246	48	7
38	148	49	8
39	63	50	1
40	63	51	164
41	78	52	0
42	252	53	0
43	4	54	40
44	76	55	0
45	128	56	255

REQUIRED REAGENTS

Total High Range Phosphorus Test 'N Tube™ Reagent Set ..... 50 vials 27672-45
Includes: (50) Total Phosphorus Test 'N Tube™ Vials*, (2) 272-42, (1) 20847-66
(1) 20760-26, (1) 27430-42

Description	Quantity Required Per Test	Unit	Cat. No.
Molybdovanadate Reagent	0.5 mL	25 mL	20760-26
Potassium Persulfate Powder Pillows	1	50/pkg	20847-66
Sodium Hydroxide Solution, 1.54 N	2 mL	100 mL	27430-42
Total Phosphorus Test 'N Tube™ Vials	1	50/pkg	*
Water, deionized		100 mL	272-42

REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes LTV082.52.40001
COD/TNT Adapter, DR/800 series ..... 1 ..... each 48464-00
Dropper, LDPE, 0.5 to 1.0 mL ..... 1 ..... 20/pkg 21247-20
Pipet, TenSette®, 1 to 10 mL ..... 1 ..... each 19700-10
Pipet Tips, for 19700-10 TenSette® Pipet ..... varies .. 50/pkg 21997-96
Test Tube Rack ..... 1–3 ..... each 18641-00

* These items are not sold separately.

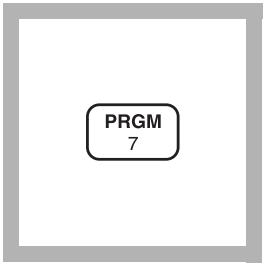
OPTIONAL REAGENTS

Description	Unit	Cat. No.
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phosphate Standard Solution, PourRite™ ampule, 500 mg/L as PO_4^{3-}, 2-mL	20/pkg	14242-20
Phosphate Standard Solution, Voluette™ ampule, 500 mg/L as PO_4^{3-}, 10-mL	16/pkg	14242-10
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53
Sulfuric Acid, ACS, concentrated	500 mL	979-491
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Aspirator, vacuum	each	2131-00
Cylinder, graduated, mixing, 10 mL (3 required)	each	20886-38
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm		LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm		LTV082.52.30001
Filter Holder, 47 mm, 300 mL, graduated	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	200/pkg	13530-01
Flask, filtering, 500-mL	each	546-49
Flask, volumetric, Class A, 50-mL	each	14574-41
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion**™1*, portable with electrode	each	51700-10
Pipet Filler, Safety Bulb	each	14651-00
Pipet, TenSette®, 0.- to 1.0-mL	each	19700-01
Pipet Tips, for 19700-01	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 8.00-mL	each	14515-08
Stopper, No. 7 one hole	6/pkg	2119-07
Tubing, rubber	12 feet	560-19

Heteropoly Blue Method*

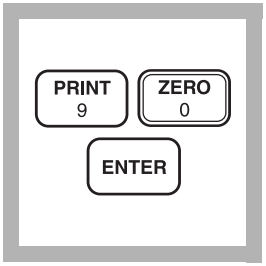


1. Enter the stored program number for low range silica (SiO_2).

Press: **PRGM**

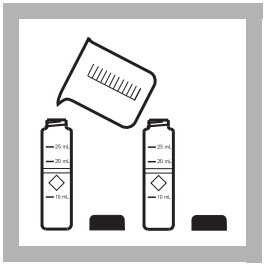
The display will show:

PRGM ?

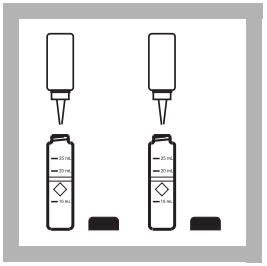


2. Press: **90 ENTER**

The display will show **mg/L, SiO2** and the **ZERO** icon**.**

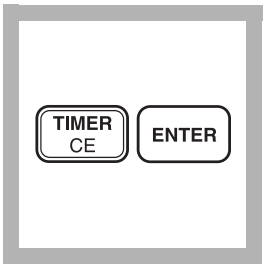


3. Fill two sample cells to the 10-mL line with sample.



4. Add 15 drops of Molybdate 3 Reagent to each sample cell. Swirl to mix.

Note: *For greatest accuracy, hold dropping bottle vertical.*

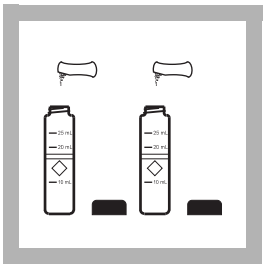


5. Press:

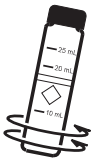
TIMER ENTER

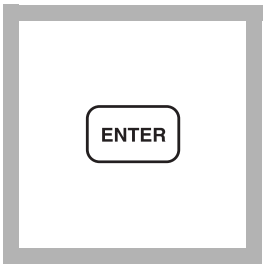
A 4-minute reaction period will begin.

Note: *Reaction time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait 8 minutes. If the sample temperature is 30 °C (86 °F), wait 2 minutes.*



6. After the timer beeps, add the contents of one Citric Acid Reagent Powder Pillow to each sample cell. Swirl to mix.





7. The display will show:

1:00 TIMER 2

Press: **ENTER**

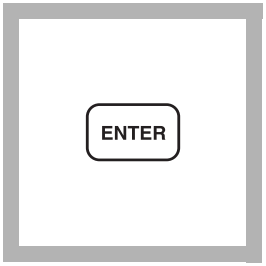
A 1-minute reaction period will begin. Phosphate interference is eliminated during this period.

Note: *The time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait two minutes. If the sample is 30 °C (86 °F), wait 30 seconds.*

8. After the timer beeps, add the contents of one Amino Acid F Reagent Powder Pillow to one of the sample cells (the prepared sample). Invert to mix.

Note: *The sample cell without the Amino Acid F Reagent is the blank.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



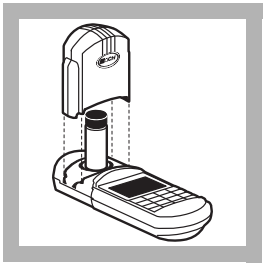
9. The display will show:

2:00 TIMER 3

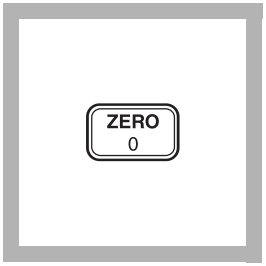
Press: **ENTER**

A 2-minute reaction period will begin.

Note: *A blue color will develop if silica is present.*



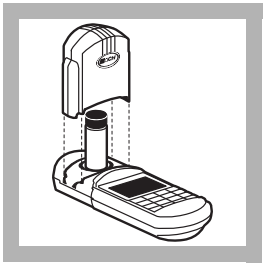
10. After the timer beeps, place the blank (solution without Amino Acid F Reagent) into the cell holder. Tightly cover the sample cell with the instrument cap.



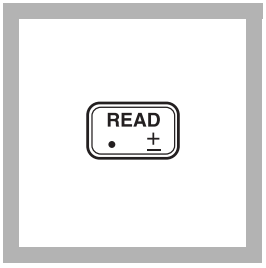
11. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L SiO2



12. Place the sample into the cell holder. Tightly cover the sample cell with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L SiO2 will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic bottles. Analyze samples as soon as possible after collection. If prompt analysis is not possible, store samples for up to 28 days by cooling to 4 °C (39 °F) or below. Warm samples to room temperature before analysis.

Accuracy Check

Standard Additions Method

a) Open a Silica Standard Solution Bottle, 25 mg/L SiO_2.

b) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of standard to three 10-mL samples. Mix thoroughly.

c) Analyze each sample as described above. The silica concentration should increase 0.25 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Adjust

To adjust the calibration curve using the reading obtained with the
1.00-mg/L Standard Solution (see *Optional Reagents*), press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **1.00** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1*, *Standard Curve Adjustment* for more information.

Method Performance

Precision

In a single laboratory, using standard solutions of 1.00 mg/L silica and two representative lots of reagent and a instrument, a single operator obtained a standard deviation of ±0.025 mg/L silica.

Estimated Detection Limit (EDL)

The estimated detection limit for program 90 is 0.020 mg/L SiO2. For more information on the estimated detection limit, see *Section 1*. If testing for very low levels of silica, use the ultra-low range silica method on the Hach DR/2010 or DR/4000 Spectrophotometers.

Interferences

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample.
Phosphate	Phosphate does not interfere at levels less than 50 mg/L PO_4. At 60 mg/L PO_4, an interference of -2% occurs. At 75 mg/L PO_4 the interference is -11%.
Iron	Large amounts of iron interfere.
Slow reacting forms of silica	Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these "molybdate-unreactive" forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the Examination of Water and Wastewater* under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help in lieu of the bicarbonate pretreatment.
Sulfides	Interfere at all levels
Turbidity	Eliminated by zeroing the instrument with the original sample.

Reagent Preparation

To prepare Amino Acid F Reagent Solution, dissolve 11.4 grams of Amino Acid F Reagent Powder in 100 mL of 1.0 N Sodium Hydroxide Solution. The solution is stable for at least one month if stored in a plastic bottle.

Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Acid reduces the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration.

REQUIRED REAGENTS

	Cat. No.
Low Range Silica Reagent Set, 10 mL sample (100 tests)	24593-00
Includes: (1) 22540-69, (1) 21062-69 (2) 1995-26	

Description	Quantity Required Per Test	Units	Cat. No.
Amino Acid F Reagent Powder Pillows	1 pillow	100/pkg	22540-69
Citric Acid Powder Pillows	2 pillows	100/pkg	21062-69
Molybdate 3 Reagent	28 drops	50 mL SCDB	1995-26

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Silica Standard Solution, 1.00 mg/L SiO_2	500 mL	1106-49
Silica Standard Solution, 25 mg/L SiO_2	236 mL	21225-31
Sodium Bicarbonate, ACS	454 g	776-01
Sodium Hydroxide Standard Solution, 1.000 N	900 mL	1045-53
Sulfuric Acid Standard Solution, 1.0 N	1000 mL	1270-53

OPTIONAL APPARATUS

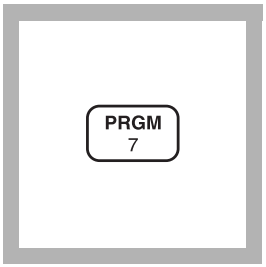
Bottle, 118 mL, polyethylene, oblong	6/pkg	23184-06
Dropper, 0.5- & 1.0-mL marks	6/pkg	23185-06
Pipet, serological, 2 mL, poly	each	2106-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 Pipet	1000/pkg	21856-28
Standard Methods for the Examination of Water and Wastewater	each	22708-00
Thermometer, - 20 to 110 °C, Non-Mercury	each	26357-02

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

Silicomolybdate Method



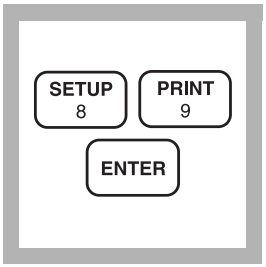
1. Enter the stored program number for high range silica (SiO_2).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **89 ENTER**

The display will show **mg/L, SiO2** and the **ZERO** icon**.**

Note: *For alternate form (Si), press the* **CONC** *key.*

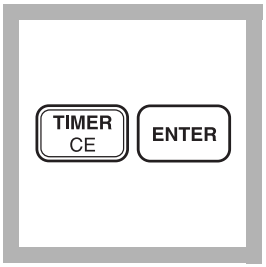
3. Fill two sample cells with 10 mL of sample. Set one aside as the blank.

Note: *Sample temperature should be 15 to 25 °C (59 to 77 °F).*

4. To the other cell, add the contents of one Molybdate Reagent Powder Pillow for High Range Silica (the prepared sample). Cap and invert to mix.

5. Add the contents of one Acid Reagent Powder Pillow for High Range Silica. Cap and invert to mix.

Note: *Silica or phosphate will cause a yellow color to develop.*



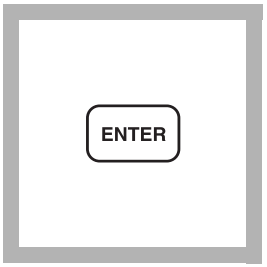
6. Press:

TIMER ENTER

A 10-minute reaction period will begin.

7. When the timer beeps, add the contents of one Citric Acid Powder Pillow to the prepared sample. Cap and invert to mix.

Note: *The yellow color due to phosphate will disappear.*

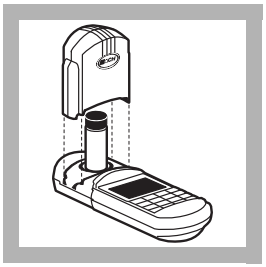


8. The display will show: **2:00 Timer 2**

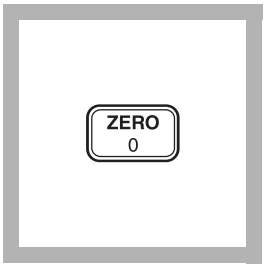
Press: **ENTER**

A two-minute reaction period will begin.

Note: *Perform Steps 9-12 within three minutes after the timer beeps.*



9. When the timer beeps, place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

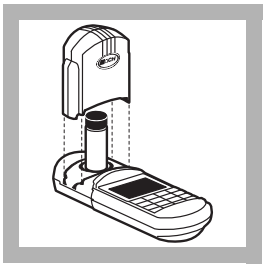


10. Press: **ZERO**

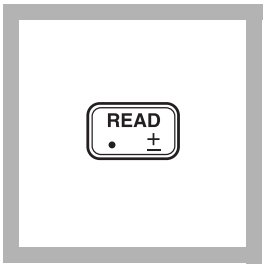
The cursor will move to the right, then the display will show:

0.0 mg/L SiO2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L silica (SiO_2) will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. Store samples up to 28 days at 4 ˚C (39 ˚F) or below. Warm samples to room temperature before analyzing.

Accuracy Check

Standard Additions Method

a) Open a High Range Silica Standard Solution, 1000 mg/L SiO_2.

b) Use the TenSette Pipet to add 0.1 mL, 0.3 mL, and 0.5 mL of the standard to three 10-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The silica concentration should increase 10.0 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To check the accuracy of the method, use the Silica Standard Solutions, 25 and 50 mg/L as SiO_2, listed under Optional Reagents. Analyze according to the above procedure using deionized water as the blank.

Standard Adjust

To adjust the calibration curve using the reading obtained with the
50.0 mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **50.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1*, *Standard Curve Adjustmen*t for more information.

Method Performance

Precision

In a single laboratory, using a standard solution of 50.0 mg/L SiO_2 and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±1.0 mg/L silica.

Estimated Detection Limit

The estimated detection limit for program 89 is 1.00 mg/L SiO_2. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample.
Iron	High levels of Fe^{2+} and Fe^{3+} interfere.
Phosphate	Does not interfere below 50 mg/L PO_4^{3-}. At 60 mg/L PO_4^{3-}, a negative 2% interference occurs. At 75 mg/L PO_4^{3-} the interference is negative 11%.
Sulfides (S^{2-})	High levels interfere.
Turbidity	Eliminated by zeroing the instrument with the original sample.

Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these “molybdate-unreactive” forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the*

Examination of Water and Wastewater under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help in lieu of the bicarbonate treatment.

Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Silica is then determined by measuring the remaining yellow color.

REQUIRED REAGENTS

	Cat. No.
High Range Silica Reagent Set, 10-mL sample (100 tests)	24296-00
Includes: (1) 21074-69, (1) 21062-69, (1) 21073-69	

Description	Quantity Required Per Test	Units	Cat. No.
Acid Reagent Powder Pillows for High Range Silica	1	100/pkg	21074-69
Citric Acid Powder Pillows	1	100/pkg	21062-69
Molybdate Reagent Powder Pillows for HR Silica	1	100/pkg	21073-69

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

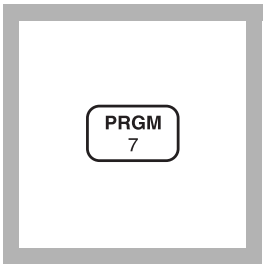
Silica Standard Solution, 10 mg/L	500 mL	1403-49
Silica Standard Solution, 25 mg/L	236 mL	21225-31
Silica Standard Solution, 50 mg/L	200 mL	1117-29
Silica Standard Solution, 1000 mg/L	500 mL	194-49
Sodium Bicarbonate, ACS	454 g	776-01
Sulfuric Acid Standard Solution, 1.000 N	100 mL MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 Pipet	1000/pkg	21856-28
Standard Methods for the Examination of Water and Wastewater	each	22708-00
Thermometer, -20 to 110 ˚C, Non-Mercury	each	26357-02

For Technical Assistance, Price and Ordering
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

Silicomolybdate Method



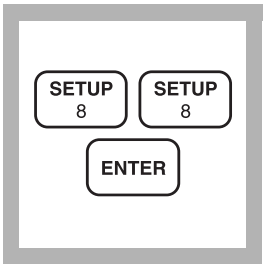
1. Enter the stored program number for ultra high range silica (SiO_2).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



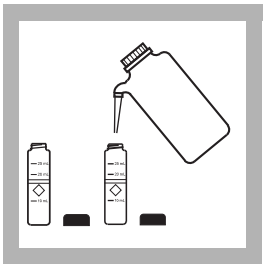
2. Press: **88 ENTER**

The display will show **mg/L, SiO2** and the **ZERO** icon**.**

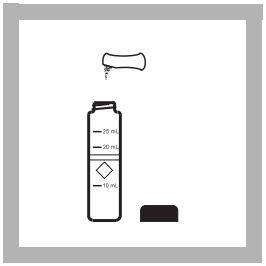
Note: *For alternate form (Si), press the* **CONC** *key.*

3. Fill 2 sample cells with 10 mL of sample.

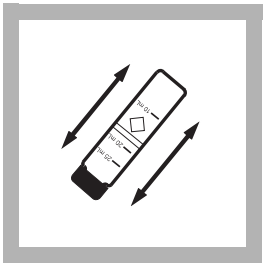
Note: *Sample temperature should be 15 to 25 °C (59 to 77 °F).*



4. Fill both sample cells to the 25-mL line with deionized water. Set one sample cell aside as the blank.

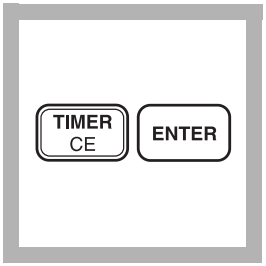


5. To the other cell, add the contents of one Molybdate Reagent Powder Pillow for High Range Silica (the prepared sample). Cap and invert to mix.



6. Add the contents of one Acid Reagent Powder Pillow for High Range Silica to the prepared sample. Cap and invert to mix.

Note: *Silica or phosphate will cause a yellow color to develop.*



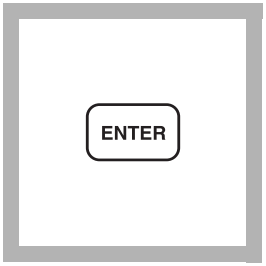
7. Press:

TIMER ENTER

A 10-minute reaction period will begin.

8. When the timer beeps, add the contents of one Citric Acid Powder Pillow to the prepared sample. Cap and invert to mix.

Note: *The yellow color due to phosphate will disappear.*

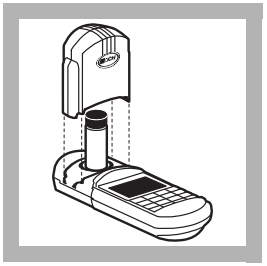


9. The display will show: **2:00 Timer 2**

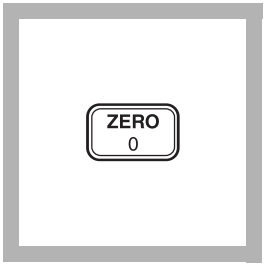
Press: **ENTER**

A two-minute reaction period will begin.

Note: *Perform Steps 10-13 within three minutes after the timer beeps.*



10. When the timer beeps, place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

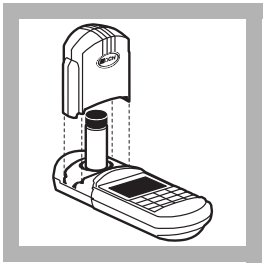


11. Press: **ZERO**

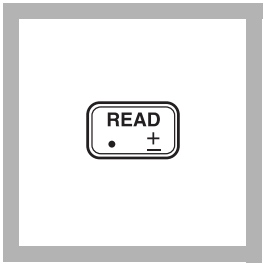
The cursor will move to the right, then the display will show:

0 mg/L SiO2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



12. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L silica (SiO_2) will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. Store samples up to 28 days at 4 °C (39 °F) or below. Warm samples to room temperature before analyzing.

Accuracy Check

Standard Additions Method

a) Open a High Range Silica Standard Solution, 1000 mg/L SiO_2.

b) Use the TenSette Pipet to add 0.1 mL, 0.3 mL, and 0.5 mL of the standard to three 10-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The silica concentration should increase 4 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To prepare a 160-mg/L silica standard, pipet 40.0 mL of a 1000-mg/L Silica Standard Solution into a 250-mL volumetric flask. Dilute to the line with deionized water. Analyze according to the above procedure using deionized water as the blank.

Standard Adjust

To adjust the calibration curve using the reading obtained with the
160-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **160.** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1*, *Standard Curve Adjustmen*t for more information.

Method Performance

Precision

In a single laboratory, using a standard solution of 100.0 mg/L SiO_2 and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±2.0 mg/L silica.

Estimated Detection Limit

The estimated detection limit for program 88 is 3.0 mg/L SiO_2. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample.
Iron	High levels of Fe^{2+} and Fe^{3+} interfere.
Phosphate	Does not interfere below 50 mg/L PO_4^{3-}. At 60 mg/L PO_4^{3-}, a negative 2% interference occurs. At 75 mg/L PO_4^{3-} the interference is negative 11%.
Sulfides (S^{2-})	High levels interfere.
Turbidity	Eliminated by zeroing the instrument with the original sample.

Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these "molybdate-unreactive" forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the Examination of Water and Wastewater* under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help in lieu of the bicarbonate treatment.

Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Silica is then determined by measuring the remaining yellow color.

REQUIRED REAGENTS

	Cat. No.
High Range Silica Reagent Set, 25-mL sample (100 tests)	22443-00
Includes: (1) 1042-99, (1) 14548-99, (1) 1041-99	

Description	Quantity Required Per Test	Units	Cat. No.
Acid Reagent Powder Pillows for High Range Silica	1	100/pkg	1042-99
Citric Acid Powder Pillows	1	100/pkg	14548-99
Molybdate Reagent Powder Pillows for HR Silica	1	100/pkg	1041-99
Water, deionized	30 mL	4 L	272-56

REQUIRED APPARATUS

Sample 10-20-15 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Silica Standard Solution, 10 mg/L	500 mL	1403-49
Silica Standard Solution, 25 mg/L	236 mL	21225-31
Silica Standard Solution, 50 mg/L	200 mL	1117-29
Silica Standard Solution, 1000 mg/L	500 mL	194-49
Sodium Bicarbonate, ACS	454 g	776-01
Sulfuric Acid Standard Solution, 1.000 N	100 mL MDB	1270-32

OPTIONAL APPARATUS

Flask, volumetric, 250 mL, Class A	each	14574-46
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 100 mL	each	14515-42
Pipet Filler, safety bulb	each	14651-00
Standard Methods for the Examination of Water and Wastewater	each	22708-00
Thermometer, -20 to 110 °C, Non-Mercury	each	26357-02

For Technical Assistance, Price and Ordering

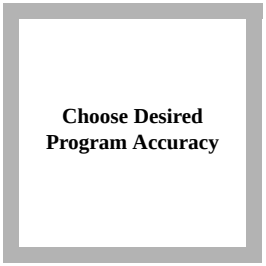
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

SULFATE (0 to 70 mg/L)

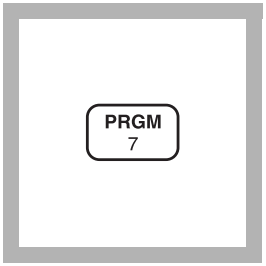
SulfaVer 4 Method* (Powder Pillows or AccuVac Ampuls); USEPA accepted for reporting wastewater analysis**

Using Powder Pillows



1. A User-Entered Calibration is necessary to obtain the most accurate results. See the *User Calibration* section at the back of this procedure. Program 91 can be used for process control or applications where a high degree of accuracy is not needed.

Note: *The nature of turbidimetric tests and reagent lot variation requires user calibration for best results.*

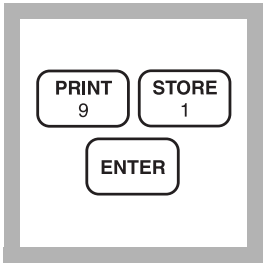


2. Enter the stored program number for sulfate (SO_4^{-}).

Press: **PRGM**

The display will show:

PRGM ?



3. Press: **91 ENTER** or the program number selected for a user-entered calibration.

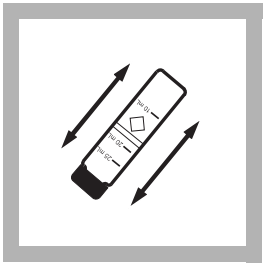
The display will show **mg/L, SO4** and the **ZERO** icon.

4. Fill a clean sample cell with 10 mL of sample.

Note: *Filter highly turbid or colored samples. Use filtered sample in this step and as the blank.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

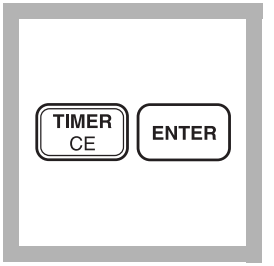
** Procedure is equivalent to USEPA method 375.4 for wastewater.



5. Add the contents of one SulfaVer 4 Sulfate Reagent Powder Pillow to the sample cell (the prepared sample). Cap the cell and invert several times to mix.

Note: *A white turbidity will develop if sulfate is present in the sample.*

Note: *Accuracy is not affected by undissolved powder.*

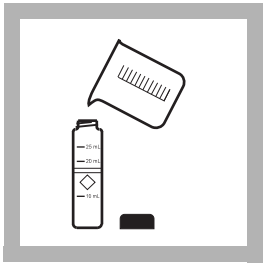


6. Press:

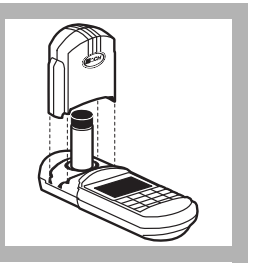
TIMER ENTER

A 5-minute reaction period will begin.

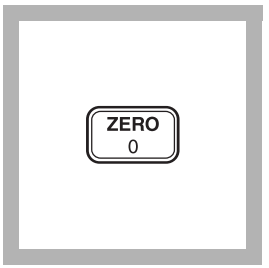
Allow the cell to stand undisturbed.



7. After the timer beeps, fill a second sample cell with 10 mL of sample (the blank).



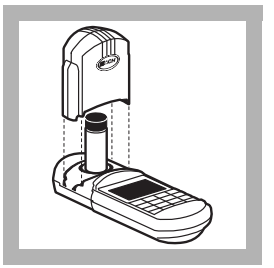
8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



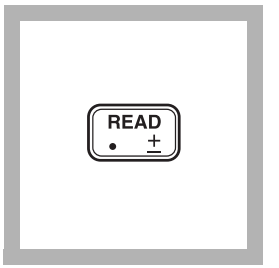
9. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L SO4



10. Within five minutes after the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



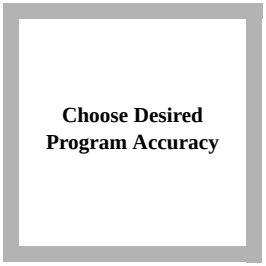
11. Press: **READ**

The cursor will move to the right, then the result in mg/L sulfate will be displayed.

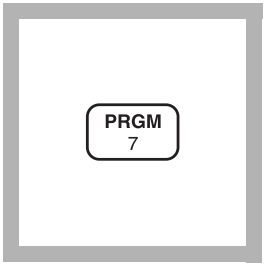
Note: *If Program 91 is used, use of the Standard Adjust is highly recommended. See Accuracy Check.*

Note: *Clean the sample cells with soap and a brush.*

Using AccuVac Ampuls



1. A User-Entered Calibration is necessary to obtain the most accurate results. See User Calibration Section at the back of this procedure. Program 92 can be used for process control or applications where a high degree of accuracy is not needed.

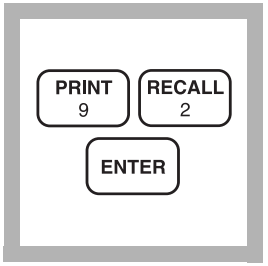


2. Enter the stored program number for sulfate (SO_4^{-})-AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

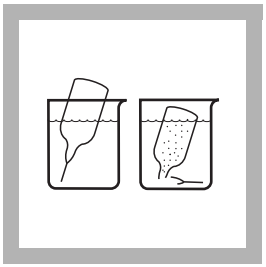


3. Press: **92 ENTER**

The display will show **mg/L, SO4** and the **ZERO** icon**.**

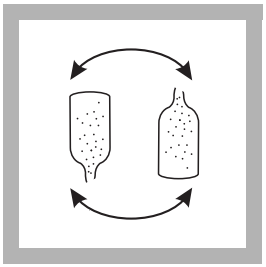
4. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Filter highly turbid or colored samples. Use filtered sample in this step and as the blank.*



5. Fill a SulfaVer 4 Sulfate AccuVac Ampul with sample.

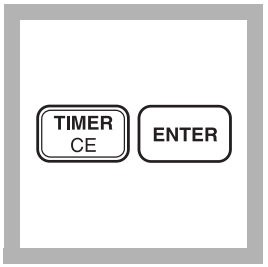
Note: *Keep tip immersed until the ampul fills completely.*



6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A white turbidity will develop if sulfate is present.*

Note: *Accuracy is not affected by undissolved powder.*

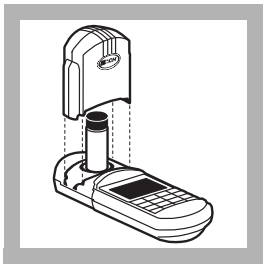


7. Press:

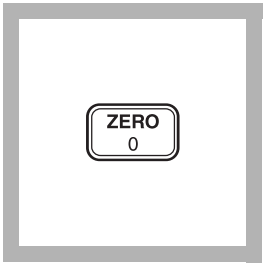
TIMER ENTER

A 5-minute reaction period will begin.

Note: *Allow the ampul to stand undisturbed.*



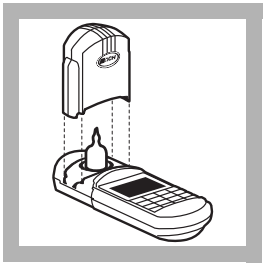
8. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



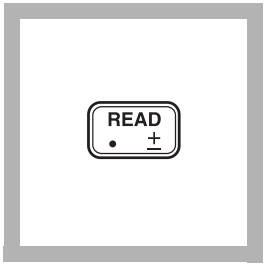
9. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L SO4



10. Within five minutes after the timer beeps, place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L sulfate will be displayed.

Note: *If Program 92 is used, use of the Standard Adjust is highly recommended. See Accuracy Check.*

User- Entered Calibration

There are various programs to determine sulfate, each with a different level of accuracy. Best results are obtained by performing a user-entered calibration with each new lot of reagent. Programs 91 and 92 can be run when a high degree of accuracy is not needed. Use of the Standard Adjust feature will improve performance when using programs 91 and 92. It should NOT be used with a user calibration, as it will hinder performance.

Using Class A glassware, prepare standards of 10, 20, 30, 40, 50, 60, and 70 mg/L sulfate by pipetting 1, 2, 3, 4, 5, 6, and 7 mL of a 1000-mg/L sulfate standard into 100-mL volumetric flasks. Dilute to the mark with deionized water and mix well.

Zero the instrument with water. The user-entered settings for sulfate are:

Program number: #101 to 105
Wavelength: 520 nm
Resolution: 0 mg/L

See *Creating User-Entered Program* in the instrument manual for specific instructions on entering a user-entered program.

Sampling and Storage

Collect samples in clean plastic or glass bottles. Samples may be stored up to 28 days by cooling to 4 °C (39 °F) or lower. Warm to room temperature before analysis.

Accuracy Check

Standard Additions Method- Powder Pillows

a) Snap the neck off a Sulfate Standard PourRite Ampule, 1000 mg/L SO_4^{2-} .

b) Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to the three 10-mL samples. Mix thoroughly.

c) Analyze each sample as described above. The sulfate concentration should increase 10 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method- AccuVac Ampuls

a) Snap the neck off a Sulfate Standard PourRite Ampule, 2500 mg/L SO_4^{2-} .

b) Fill three 25- mL graduated cylinders with 25 mL of sample. Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to the three cylinders. Mix thoroughly. For AccuVac Ampuls, transfer to a 50-mL beaker.

c) Analyze each sample as described above. The sulfate concentration should increase 10 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Check the accuracy of the test by using the Sulfate Standard Solution, 50 mg/L, listed under Optional Reagents. Or, prepare this solution by pipetting 1.0 mL of a PourRite Ampule Standard for Sulfate (2500 mg/L) into a 50-mL volumetric flask. Dilute to volume with deionized water. The final concentration is 50 mg/L sulfate. Substitute this standard for the sample and proceed with the test as described in the procedure.

Standard Adjust
Standard adjust is recommended when using stored programs 91 or 92. It **should not** be used with a user-entered calibration.

To adjust the calibration curve using the reading obtained with the
50-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **50** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustmen*t for more information.

Method Performance

Precision
In a single laboratory, using a standard solution of 50 mg/L sulfate and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of ±0.5 mg/L sulfate.

In a single laboratory, using a standard solution of 50 mg/L sulfate and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±3 mg/L sulfate.

Estimated Detection Limit (EDL)
The EDL for program 91 is 4.9 mg/L SO_4 and the EDL for program 92 is 3 mg/L SO_4. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

The following interfere at levels above those concentrations listed:

Calcium: 20,000 mg/L as $CaCO_3$	Magnesium: 10,000 mg/L as $CaCO_3$
Chloride: 40,000 mg/L as Cl^-	Silica: 500 mg/L as $CaCO_3$

Summary of Method

Sulfate ions in the sample react with barium in the SulfaVer 4 Sulfate Reagent to form insoluble barium sulfate. The amount of turbidity formed is proportional to the sulfate concentration. The SulfaVer 4 also contains a stabilizing agent to hold the precipitate in suspension.

REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Units	Cat. No.
SulfaVer 4 Sulfate Reagent Powder Pillows	1 pillow	100/pkg	21067-69
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

REQUIRED REAGENTS AND APPARATUS (Using AccuVac Ampuls)

SulfaVer 4 Sulfate AccuVac Ampuls	1 ampul	25/pkg	25090-25
Beaker, 50-mL	1	each	500-41H

OPTIONAL REAGENTS

Standard, Drinking Water Inorganics, F^-, NO_3^{-N}, PO_4^{-3}, SO_4^{-2}		500 mL	28330-49
Standard, Wastewater Effluent Inorganics, NH_3^{-N}, NO_3^{-N}, PO_4^{-3}, COD, SO_4^{-2}, TOC		500 mL	28332-49
Sulfate Standard Solution, 50 mg/L		500 mL	2578-49
Sulfate Standard Solution, 1000 mg/L		500 mL	21757-49
Sulfate Standard Solution, PourRite Ampule, 2500 mg/L, 10 mL		16/pkg	14252-10
Sulfate Standard Solution, PourRite Ampule, 1000 mg/L, 2 mL		20/pkg	21757-20
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit		each	24052-00
Cylinder, graduated mixing, 25 mL		each	20886-40
Filter Paper, folded, 12.5 cm		100/pkg	1894-57
Flask, volumetric, 50 mL, Class A		each	14574-41
Funnel, poly, 65 mm		each	1083-67
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 Pipet		50/pkg	21856-96
Pipet, volumetric, 1.00 mL, Class A		each	14515-35
Pipet, volumetric, 2.00 mL, Class A		each	14515-36
Pipet, volumetric, 3.00 mL, Class A		each	14515-03
Pipet, volumetric, 4.00 mL, Class A		each	14515-04
Pipet, volumetric, 5.00 mL, Class A		each	14515-37
Pipet, volumetric, 6.00 mL, Class A		each	14515-06
Pipet, volumetric, 7.00 mL, Class A		each	14515-07
Pipet Filler, safety bulb		each	14651-00
PourRite Ampule Breaker		each	24846-00

For Technical Assistance, Price and Ordering

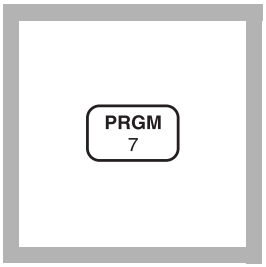
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

SULFIDE (0 to 0.70 mg/L S^{2-})

For water, wastewater and seawater

Methylene Blue Method* USEPA accepted for reporting wastewater analysis**

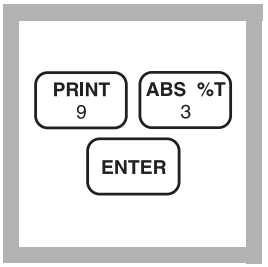


1. Enter the stored program number for sulfide (S).

Press: **PRGM**

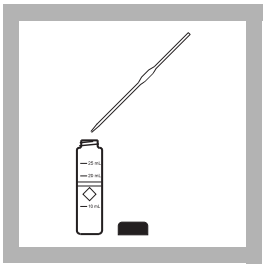
The display will show:

PRGM ?



2. Press: **93 ENTER**

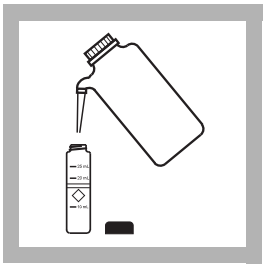
The display will show **mg/L, S** and the **ZERO** icon**.**



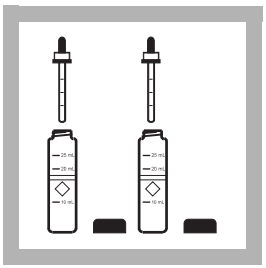
3. Pipet 25 mL of sample into a clean sample cell. This will be the prepared sample.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis. Use a pipet to avoid agitation.*

Note: *For field testing, a 25-mL graduated cylinder may be used.*

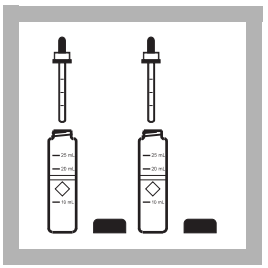


4. Fill a second sample cell with 25 mL of deionized water (the blank).



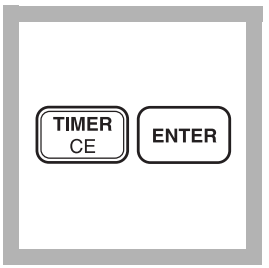
5. Add 1.0 mL of Sulfide 1 Reagent to each cell. Swirl to mix.

Note: *Use the calibrated 1-mL dropper.*



6. Add 1.0 mL of Sulfide 2 Reagent to each cell. Immediately swirl to mix.

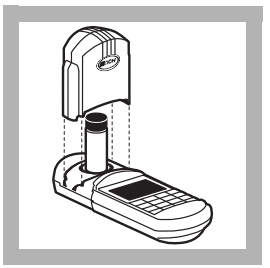
Note: *A pink color will develop, then the solution will turn blue if sulfide is present.*



7. Press:

TIMER ENTER

A 5-minute reaction period will begin.

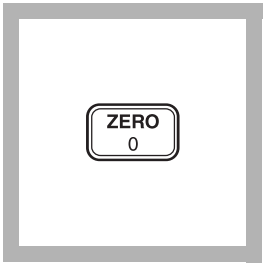


8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

---

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

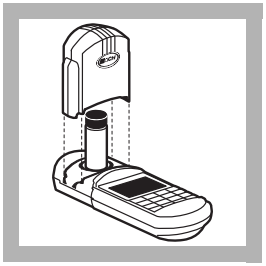
** Procedure is equivalent to USEPA method 376.2 or Standard Method 4500-S^{2-} D for wastewater.



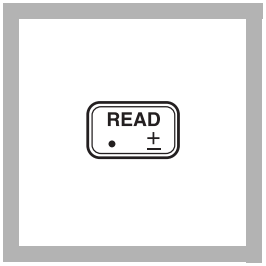
9. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L S



10. After the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L sulfide will be displayed.

Note: *Some sulfide loss may occur if dilution is necessary.*

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Avoid excessive agitation or prolonged exposure to air. Analyze samples immediately.

Accuracy Check

Sulfide standards are unstable and must be user prepared. See Sandard Methods, 4500S^{-} for preparation and standardization directions.

Method Performance

Precision

In a single laboratory, using standard solutions of 0.73 mg/L sulfide and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ± 0.02 mg/L sulfide.

Estimated Detection Limit (EDL)

The EDL for program 93 is 0.01 mg/L S^{2-}. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Strong reducing substances (sulfite, thiosulfate and hydrosulfite)	Interfere by reducing the blue color or preventing its development.
Sulfide, high levels	High concentrations of sulfide may inhibit full color development and require sample dilution. Some sulfide loss may occur when the sample is diluted.
Turbidity	For turbid samples, prepare a sulfide-free blank as follows. Use it in place of the deionized water blank in the procedure. **1.** Measure 25 mL of sample into a 50-mL erlenmeyer flask. **2.** Add Bromine Water dropwise with constant swirling until a permanent yellow color just appears. **3.** Add Phenol Solution dropwise until the yellow color just disappears. Use this solution in Step 4 in place of deionized water.

Soluble Sulfides

Determine soluble sulfides by centrifuging the sample in completely filled, capped tubes and analyzing the supernatant. Insoluble sulfides are then estimated by subtracting the soluble sulfide concentration from the total sulfide result.

Summary of Method

Hydrogen sulfide and acid-soluble metal sulfides react with N, N-dimethyl-p-phenylenediamine oxalate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration. High sulfide levels in oil field waters may be determined after dilution.

Pollution Prevention and Waste Management

Sulfide 2 Reagent contains potassium dichromate. The final solution will contain hexavalent chromium (D007) at a concentration regulated as a hazardous waste by Federal RCRA. See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS

	Cat. No.
Sulfide Reagent Set (100 tests)	22445-00
Includes: (2) 1816-42, (2) 1817-42	

Description	Quantity Required Per Test	Units	Cat. No.
Sulfide 1 Reagent	2 mL	100 mL MDB	1816-32
Sulfide 2 Reagent	2 mL	100 mL MDB	1817-32
Water, deionized	25 mL	4 L	272-56

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508-40
Pipet, volumetric, Class A, 25.00 mL	1	each	14515-40
Pipet Filler, safety bulb	1	each	14651-00
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Description	Units	Cat. No.
Bromine Water, 30 g/L	29 mL	2211-20
Phenol Solution, 30 g/L	29 mL	2112-20
Sodium Sulfide, hydrate	114 g	785-14

OPTIONAL APPARATUS

Bottle, Wash, 250 mL	each	620-31
Dropper, for 1 oz. bottle	each	2258-00
Flask, erlenmeyer, 50 mL	each	505-41
Standard Methods for the Examination of Water and Wastewater	each	22708-00

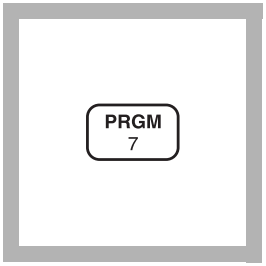
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

SURFACTANTS, ANIONIC (0 to 0.300 mg/L)

(Also called: Detergents) Crystal Violet Method*

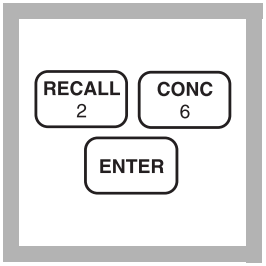


1. Enter the stored program number for Surfactants, anionic (LAS).

Press: **PRGM**

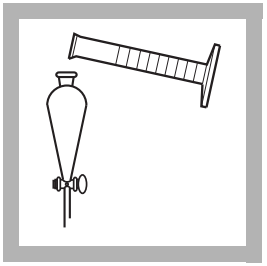
The display will show:

PRGM ?

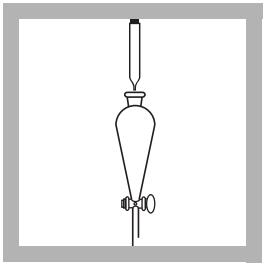


2. Press: **26 ENTER**

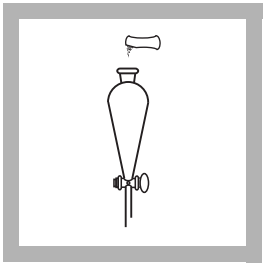
The display will show **mg/L, LAS** and the **ZERO** icon**.**



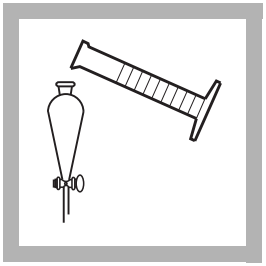
3. Fill a clean 500-mL graduated cylinder to the 300-mL mark with sample. Pour the sample into a clean 500-mL separatory funnel.



4. Add 10 mL of Sulfate Buffer Solution. Stopper the funnel. Shake the funnel for five seconds.



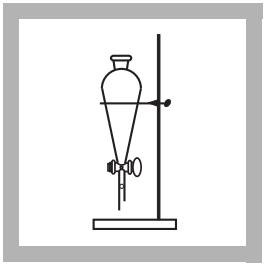
5. Add the contents of one Detergents Reagent Powder Pillow to the funnel. Stopper the funnel and shake to dissolve the powder.



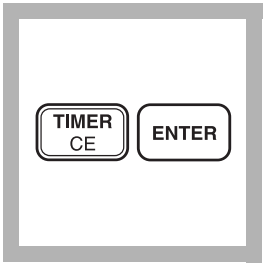
6. Add 30 mL of benzene to the funnel. Stopper the funnel and shake gently for one minute.

Note: *Spilled reagent will affect test accuracy and is hazardous to the skin and other materials.*

Note: *Use benzene only in a well-ventilated area.*



7. Place the separatory funnel in a support stand.



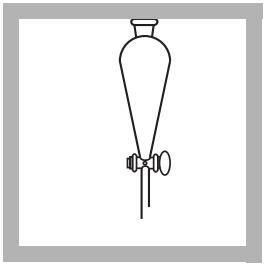
8. Press:

TIMER ENTER

A 30-minute reaction period will begin.

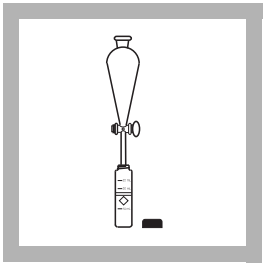
Note: *Excessive agitation may cause an emulsion, requiring a longer time for phase separation. If this occurs, remove most of the water layer, then gently agitate the funnel with a clean inert object in the funnel such as a Teflon-coated magnetic stirring bar.*

* *Analytical Chemistry*, 38, 791(1966).



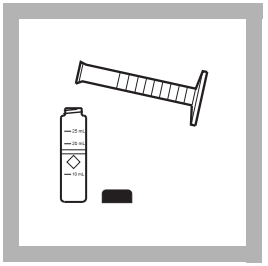
9. After the timer beeps, remove the stopper and drain the bottom water layer. Discard this layer.

Note: *Benzene solutions are a regulated waste and cannot be poured down the drain. See Section 3 for proper disposal of these materials.*

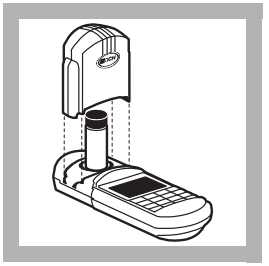


10. Drain the top benzene layer into a clean sample cell (the prepared sample).

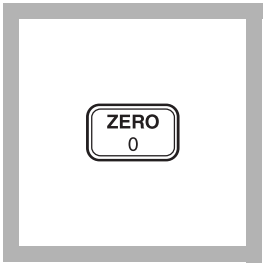
Note: *The benzene layer cannot be filtered before color measurement. Filtration removes the blue color.*



11. Fill another sample cell to the 25-mL mark with pure benzene (the blank).



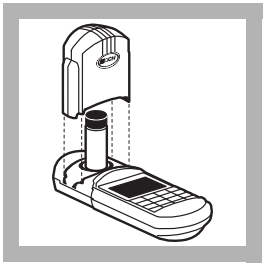
12. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



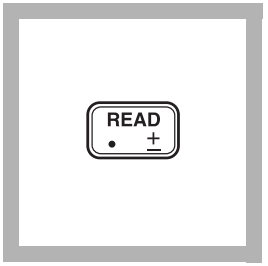
13. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.000 mg/L LAS



14. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



15. Press: **READ**

The cursor will move to the right, then the result in mg/L anionic surfactants (LAS) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Note: *Acetone may be used to clean benzene from glassware.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible, but they may be stored at least 24 hours by cooling to 4 °C
(39 °F). Warm to room temperature before testing.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Detergent Voluette Ampule Standard Solution, 60 mg/L as LAS (The molecular weight of linear alkylate sulfonate used to make the standard is 342).

b) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of standard to three 300-mL samples. Mix thoroughly.

c) Analyze each as described above. The anionic surfactants reading should increase 0.02 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.150 mg/L LAS, two lots of reagent, and the instrument, a single operator obtained a standard deviation of ±0.010 mg/L LAS as anionic surfactant.

Estimated Detection Limit

The estimated detection limit for program 26 is 0.020 mg/L LAS. For more information on the estimated detection limit, see *Section 1*.

Interferences

Perchlorate and periodate ions will interfere. High amounts of chloride, such as those levels found in brines and seawater, will cause low results.

Summary of Method

Detergents, ABS (alkyl benzene sulfonate) or LAS (linear alkylate sulfonate) are determined by association with crystal violet dye and extraction of the ion-pair complex into benzene.

Pollution Prevention and Waste Management

Benzene (D018) solutions are regulated as hazardous waste by Federal RCRA. Do not pour these materials down the drain. Collect water saturated with benzene solutions for disposal with laboratory solvent wastes. See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Benzene, ACS	55 mL	500 mL	14440-49
Buffer Solution, sulfate type	10 mL	500 mL	452-49
Detergent Reagent Powder Pillow	1 pillow	25/pkg	1008-68

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, 25 mL	1	each	508-40
Cylinder, graduated, 50 mL	1	each	508-41
Cylinder, graduated, 500 mL	1	each	508-49
Funnel, separatory, 500 mL	1	each	520-49
Ring, support, 4 inch	1	each	580-01
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06
Stand, support, 127 x 203 mm (5 x 8")	1	each	563-00

OPTIONAL REAGENTS

Acetone, ACS	500 mL	14429-49
Detergent Standard Solution, Voluette ampule, 60 mg/L as LAS, 10 mL	16/pkg	14271-10

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 Pipet	1000/pkg	21856-28
Thermometer, -20 to 110 °C, Non-Mercury	each	26357-02

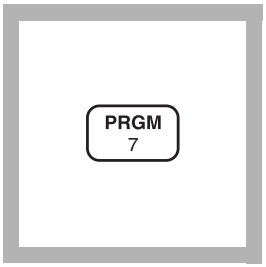
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

SUSPENDED SOLIDS (0 to 750 mg/L)

Photometric Method* (Also called Nonfilterable Residue)

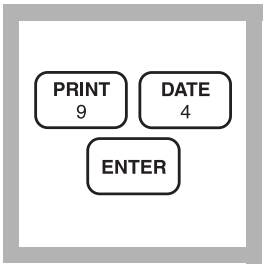


1. Enter the stored program number for suspended solids.

Press: **PRGM**

The display will show:

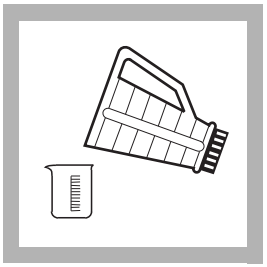
PRGM ?



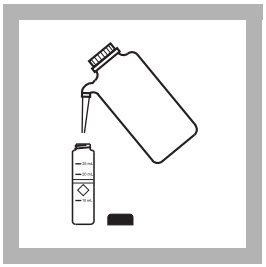
2. Press: **94 ENTER**

The display will show **mg/L, SuSld** and the **ZERO** icon**.**

3. Blend 500 mL of sample in a blender at high speed for exactly 2 minutes.

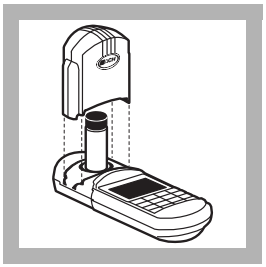


4. Pour the blended sample into a 600-mL beaker.

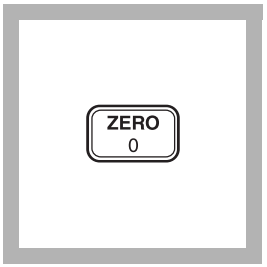


5. Fill a sample cell with 25 mL of tap water or deionized water (the blank).

Note: *Remove gas bubbles in the water by swirling or tapping the bottom of the cell on a table.*



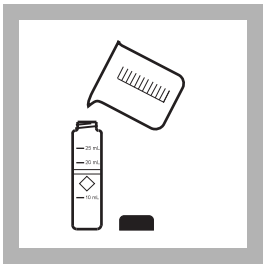
6. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



7. Press: **ZERO**

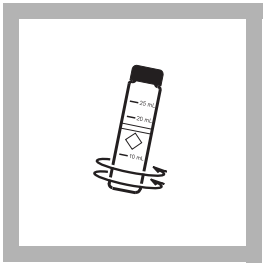
The cursor will move to the right, then the display will show:

0 mg/L SuSld

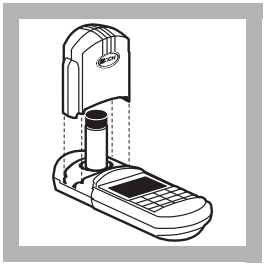


8. Stir the sample thoroughly and immediately pour 25 mL of the blended sample into a sample cell (the prepared sample).

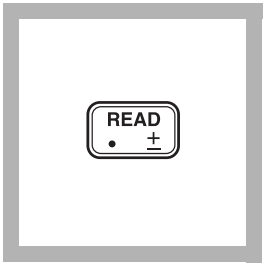
* Adapted from *Sewage and Industrial Wastes*, 31, 1159 (1959).



9. Swirl the prepared sample cell to remove any gas bubbles and uniformly suspend any residue.



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L suspended solids will be displayed.

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. The sample may be stored seven days by cooling to 4 °C (39 °F).

Method Performance

Precision

In a single laboratory, using a standard solution of 847.4 mg/L Suspended Solids with the instrument, a single operator obtained a standard deviation of ±18.2 mg/L Suspended Solids.

For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program 94 is 22.1 mg/L Suspended Solids. For more information on the estimated detection limit, see *Section 1*.

Interferences

Calibration for this test is based on parallel samples using the gravimetric technique on sewage samples from a municipal sewage plant. For most samples, this calibration will provide satisfactory results. When higher accuracy is required, run parallel photometric and gravimetric determinations with portions of the same sample. The new calibration should be made on your particular sample using a gravimetric technique as a basis.

Summary of Method

This method of determining suspended solids is a simple, direct measurement which does not require the filtration or ignition and weighing steps that gravimetric procedures do. The USEPA specifies the gravimetric method for solids determinations, while this method is often used for checking in-plant processes.

REQUIRED APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Beaker, 600 mL, low form	1	each	1080-52
Blender, 1.2 L, 120 V	each	each	26161-00
Blender, 1.2 L, 240 V	each	each	26161-02
Cylinder, graduated, 500 mL, poly	1	each	1081-49
Pipet, serologic, 25 mL	1	each	2066-40
Pipet, Filler, safety bulb	1	each	14651-00

OPTIONAL APPARATUS

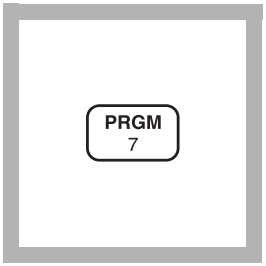
Stirring Rod, glass		3/pkg	1770-01
Wash Bottle, 250 mL		each	620-31

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

Tyrosine Method*

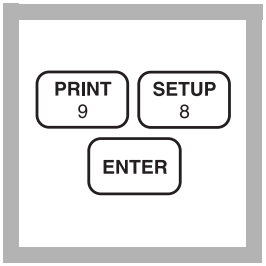


1. Enter the stored program number for tannin and lignin.

Press: **PRGM**

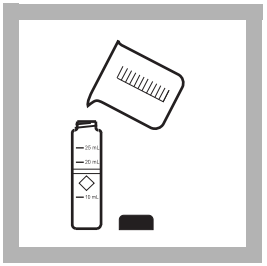
The display will show:

PRGM ?

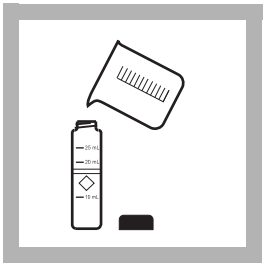


2. Press: **98 ENTER**

The display will show **mg/L, tanic** and the **ZERO** icon**.**

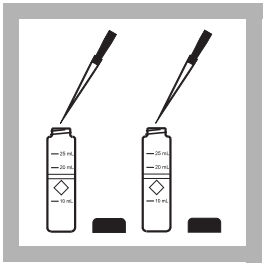


3. Fill a clean sample cell to the 25-mL mark with deionized water (the blank).

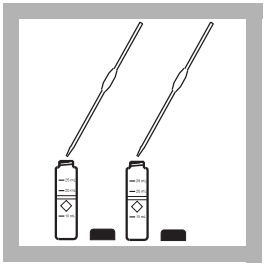


4. Fill a clean sample cell to the 25-mL mark with sample (the prepared sample).

Note: *Filter turbid samples and report results as mg/L soluble tannic acid.*

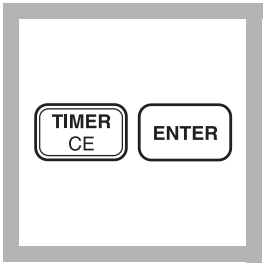


5. Pipet 0.5 mL of TanniVer 3 Tannin-Lignin Reagent into each cell. Swirl to mix.



6. Pipet 5.0 mL of Sodium Carbonate Solution into each cell. Swirl to mix.

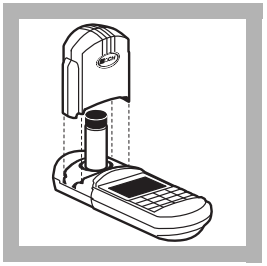
Note: *A blue color will develop if tannins and/or lignins are present.*



7. Press:

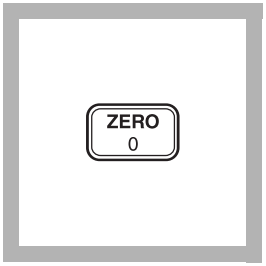
TIMER ENTER

A 25-minute reaction period will begin.



8. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

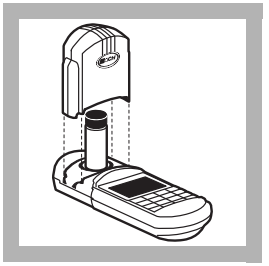
* Adapted from Kloster, M.B., *Journal American Water Works Association,* Vol. 66, No. 1, p. 44 (1974).



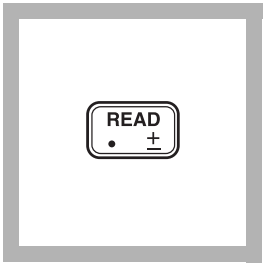
9. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L tanic



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L tannic acid will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles.

Accuracy Check

Standard Solution Method

Prepare a 200-mg/L tannic acid standard solution by dissolving 0.200 grams of tannic acid in deionized water and diluting to 1000 mL. Prepare this solution monthly. A 2.0 mg/L tannic acid standard is prepared by diluting 10.00 mL of the stock solution to 1000 mL with deionized water. Prepare this standard daily.

Method Performance

Precision

In a single laboratory, using standard solutions of 4.0 mg/L tannic acid and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.1 mg/L tannic acid.

Estimated Detection Limit

The estimated detection limit for program 98 is 0.1 mg/L tannin and lignin. For more information on the estimated detection limit, see *Section 1*.

Interferences

Substance	Interference Level and Treatment
Ferrous iron	Causes a positive interference. Two mg/L of ferrous iron produces a color equivalent to about 1 mg/L of tannic acid. To eliminate interference of ferrous iron up to 20 mg/L, add one 0.2-g scoop of sodium pyrophosphate to the sample before testing.
Sulfide	Eliminated by adding 1 mL of formaldehyde to the sample before testing the sample.

Summary of Method

This test measures all hydroxylated aromatic compounds, including tannin, lignin, phenol and cresol. This method produces a blue color proportional to the amount of these compounds present in the sample. Report results as total tannin and lignin expressed as mg/L tannic acid.

REQUIRED REAGENTS

Cat. No.

Tannin and Lignin Reagent Set (up to 100 tests) 22446-00
Includes: (2) 675-49, (1) 2560-42

Description	Quantity Required Per Test	Unit	Cat. No
Sodium Carbonate Solution	10 mL	500 mL	675-49
TanniVer 3 Tannin-Lignin Reagent	1 mL	100 mL	2560-42
Water, deionized	25 mL	4 L	272-56

REQUIRED APPARATUS

Pipet Filler, safety bulb	1	each	14651-00
Pipet, volumetric, Class A, 5.0 mL	1	each	14515-37
Pipet, volumetric, Class A, 0.5 mL	1	each	14515-34
Sample Cell, 10-20-25-mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Formaldehyde	100 mL	2059-32
Sodium Pyrophosphate, ACS	50 g	14295-25
Tannic Acid	113 g	791-14

OPTIONAL APPARATUS

Description	Unit	Cat. No
Balance, analytical, 115 V	each	28014-01
Balance, analytical, 230 V	each	28014-02
Cylinder, graduated, 25 mL	each	508-40
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, volumetric, 1000 mL	each	14547-53
Funnel, poly, 65 mm	each	1083-67
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 10.00 mL	each	14515-38
Pipet, Filler, safety bulb	each	14651-00
Spoon, measuring, 0.2 g	each	638-00
Weighing Boat, 67/47 mm	500/pkg	21790-00

For Technical Assistance, Price and Ordering

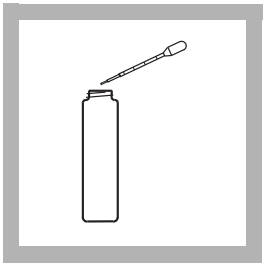
In the U.S.A.—Call 800-227-4224

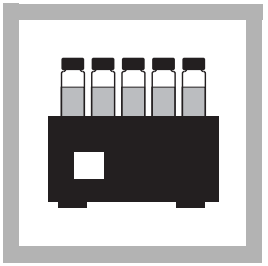
Outside the U.S.A.—Contact the Hach office or distributor serving you.

TOXTRAK™ TOXICITY TEST*

Colorimetric Method**

Inoculum Development



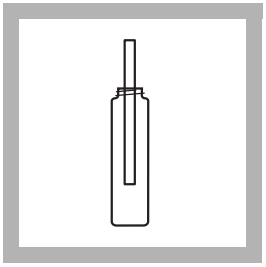


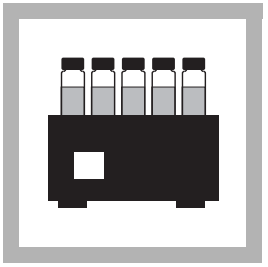
Using Indigenous Biomass

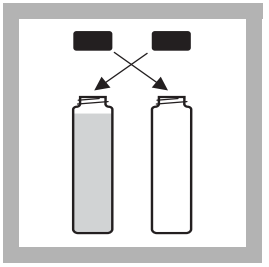
1. Using one of the dropper pipets provided, add 1.0 mL of source culture to a Tryptic Soy Broth Tube.

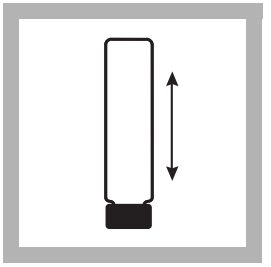
2. Incubate at 37 °C until the vial contents are visibly turbid (turbidity indicates bacterial growth).

Inoculum Development Using Aqua QC-Stiks









1. Inoculate a Lauryl Tryptose broth tube with an *E. coli* Aqua QC-Stik™ according to the instructions that come with the stick.

2. Incubate the Lauryl Tryptose Broth Tube at 35°C (95°F) until the medium is visibly turbid (approxamately 12 hours). Turbidity develops much faster in an incubator than at room temperature.

3. Inoculate a new Lauryl Tryptose Broth Tube by first inverting the tube and switching the caps of the two tubes.

In this way, several medium vials can be inoculated from one Aqua–QC Stick™.

4. Invert the new tube. After incubation, this new vial may be used in subsequent tests.

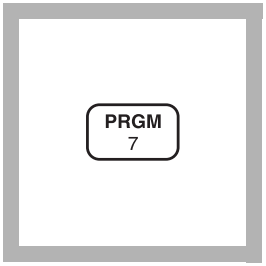
If toxicity tests will be run on consecutive days, inoculum may be kept several days at room temperature.

Cultures 10 to 72 hours old give best results.

* U.S. Patent number 5,413,916.

** Liu, D., *Bull. Environ. Contam. Toxicol.* 26, 145-149 (1981).

Colorimetric Reaction

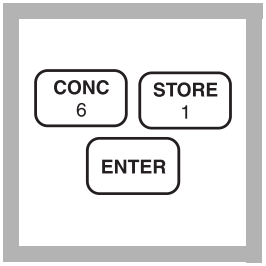


1. Enter the stored program number for toxicity.

Press: **PRGM**

The display will show:

PRGM ?

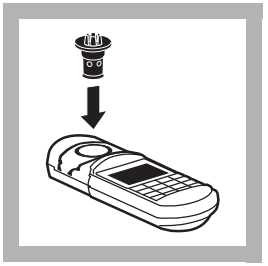


2. Press: **61 ENTER**

The display will show:

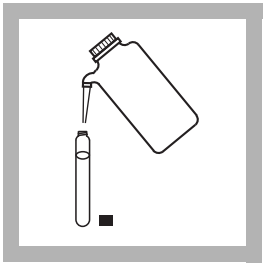
ABS 610 nm

and the zero icon.

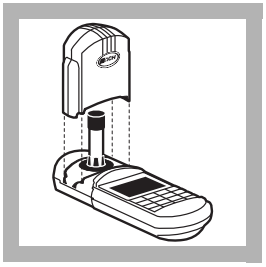


3. Insert the TNT/COD Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

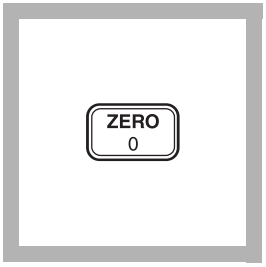
Note: *A diffuser band covers the light path holes on the adapter to give increased performance. The band should NOT be removed.*



4. Fill a Test 'N Tube vial with deionized water. Label this vial as the "blank". Wipe the outside of all the vials with a tissue to remove fingerprints and smudges.



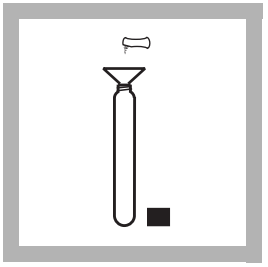
5. Place the blank in the adapter. Tightly cover the vial with the instrument cap.



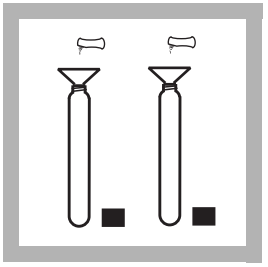
6. Press: **ZERO**

The cursor will move to the right, then the display will show:

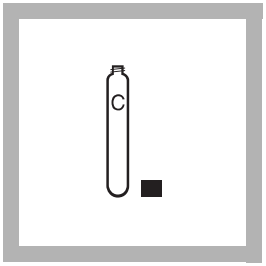
0.000 ABS



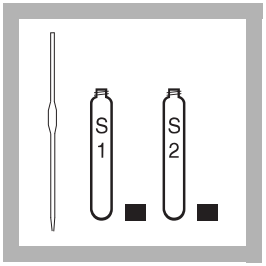
7. Label a vial "control." Open one ToxTrak Reagent Powder Pillow and add the contents to the empty reaction vial.



8. Label each sample or dilution vial clearly. Add the contents of one ToxTrak Reagent Powder Pillow to each labeled vial.

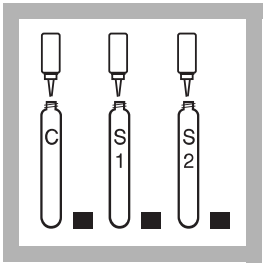


9. Add 5.0 mL of deionized water to the control tube.

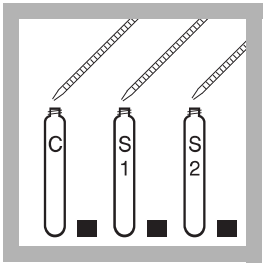


10. Add 5.0 mL of the sample (or dilution) to each sample vial.

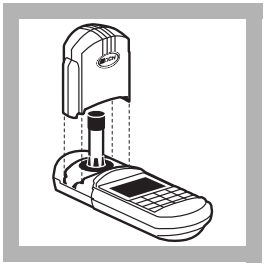
Note: *To determine the approximate threshold level of toxicity for a sample, dilute 1 mL of sample to 10 mL of deionized water and run the test. Continue to make serial 1:10 dilutions until a level is reached which gives a 0% Inhibition in Step 18.*



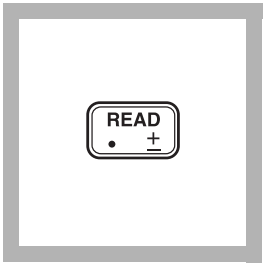
11. Add 2 drops of Accelerator Solution to each vial. Cap and invert to mix.



12. Add 0.5 mL of inoculum (previously prepared) to each vial. Cap and invert to mix.



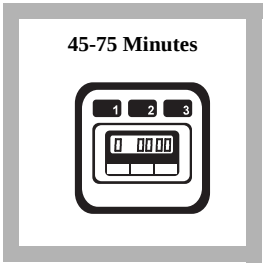
13. Place the control vial in the cell holder. Tightly cover the vial with the instrument cap.



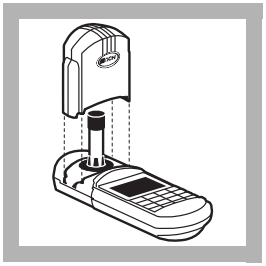
14. Press: **READ**

The cursor will move to the right, then the result in ABS will be displayed. Record the absorbance of the "control" vial.

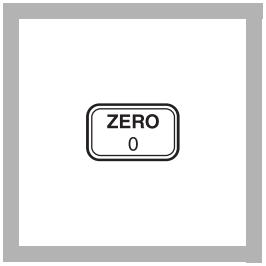
15. Repeat Steps 13-14 for all samples and dilutions. Be sure to record each absorbance.



16. Allow the solutions in the tubes to react until the absorbance of the **control tube** decreases 0.60 ±0.10.
This should take about 45-75 minutes.



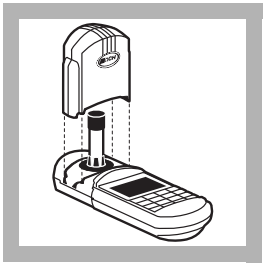
17. After the absorbance of the "control" vial has decreased 0.60 ±0.10 absorbance units, place the blank in the adapter. Tightly cover the vial with the instrument cap.



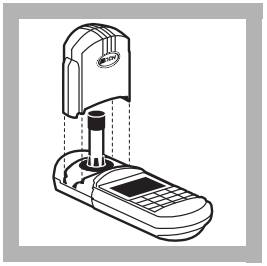
18. Press: **ZERO**

The cursor will move to the right, then the display will show:

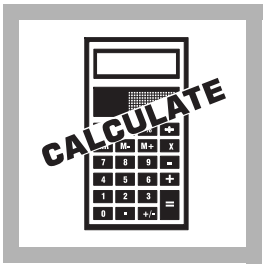
0.000 ABS



19. Place the "control" vial in the cell holder. Tightly cover the vial with the instrument cap. Record the absorbance value of the control.



20. Place each sample or dilution vial in the cell holder. Tightly cover the vial with the instrument cap. Record each absorbance value.



21. Calculate the % Inhibition as follows:

$$\%I = \left[1 - \left(\frac{\Delta \text{Abs sample}}{\Delta \text{Abs control}}\right)\right] \times 100$$

ΔA is the initial absorbance value minus the final absorbance value.

See the example following this step.

Note: *Some toxins increase respiration and will give a negative % inhibition on all respiration-based toxicity tests. After repeated testing, samples which always give a % inhibition in Step 21 that is more negative than –10% should be considered toxic.*

Example

The control tube (C) has an initial absorbance of 1.6 and decreases to 1.0 Abs. The sample tube has an initial absorbance of 1.7 and decreases to 1.3 Abs.

ΔAbs. Sample = 1.7 – 1.3 = 0.4 ΔAbs. Control = 1.6 – 1.0 = 0.6

$$\%I = \left(1 - \left(\frac{0.4}{0.6}\right)\right) \times 100 \qquad \%I = 33.3$$

Interpreting Results

The Percent Inhibition results obtained are only a relative measurement. They do not represent a true quantitative measurement of toxic concentration. The Percent Inhibition does not necessarily increase in direct proportion to the concentration of toxins. To determine the minimum inhibition concentration of a toxin, it is possible to make tenfold dilutions of the sample and determine the Percent Inhibition for the dilutions. When the sample is diluted so that no inhibition is observed, this is the No Observed Effect Concentration (NOEC).

Due to the many variables involved in the test, the limits of detection are on the order of 10% Inhibition. This would correlate to the Lowest Observable Effect Concentration (LOEC). If a sample shows less that 10% Inhibition, repeat the test. After several repetitions, look at the series of data to determine the likelihood of toxicity. Results below 10% are not reliable, but can be used to surmise some presence of toxicity if they are consistent. See the table below.

Toxicity Results

Data Points: Percent Inhibition	Conclusion
7%, 9%, 5%, 8%, 5%	May be slightly toxic
7%, –4%, 5%, 5%, 1%	Most likely not toxic
–7%, –9%, 5%, –8%, –5%	May be slightly toxic

Some toxins will increase respiration and will give a negative Percent Inhibition on this and all other respiration-based toxicity tests. After repeated testing, samples that always give a Percent Inhibition that is more negative than –10% should be considered toxic.

Disposal of Test Cultures

Dispose of active bacterial cultures by using one of these methods:

1. Autoclave used test containers at 121 °C for 15 minutes at 15 pounds of pressure. Once the containers are sterile, pour the contents down the drain with running water. The reaction tubes may be washed
and re-used.

2. Sterilize test containers by using a 1:10 dilution of commercial

laundry bleach. Pour the test container contents and test containers into the bleach solution. Allow 10-15 minutes of contact time with the bleach solution. Then pour the liquid down the drain and wash the reaction tubes for re-use.

Summary of Method

Resazurin is a redox-active dye, which changes from pink to blue when it is reduced. Bacterial respiration occurring in the sample reduces resazurin. If toxic substances are present, they inhibit the rate of resazurin reduction. The sample color is compared to a toxin-free control tube to determine how toxic the sample is to an indigenous culture or a culture of *E. coli*. A chemical accelerant reduces the reaction time of the procedure.

REQUIRED REAGENTS

Description	Cat. No.
ToxTrak Reagent Set (25 tests)	25972-00
Includes: (1) 25607-66, (1) 25608-36, (1) 22777-00, (1) 24092-32	

Description	Quantity Required Per Test	Unit	Cat. No.
Aqua QC–Stiks, Escherichia Coli	1	3 cultures	27063-03
Sodium Thiosulfate Standard Solution	varies	100 mL	24092-32
ToxTrak Reagent Powder Pillows	1 pillow	50/pkg	25607-66
ToxTrak Accelerator Solution	2 drops	15 mL SCDB	25608-36
Tryptic Soy Broth Tubes	1	15/pkg	22777-00
Tube, culture, with cap	1	10/pkg	20962-08
Water, deionized	varies	200 mL	272-29

REQUIRED APPARATUS

Cap, White	1	6/pkg	22411-06
Clippers, to open powder pillows	1	each	936-00
COD/TNT Adapter	1	each	48464-00
Dropper Pipet, 1 mL	varies	10/pkg	21247-20
Forceps, flat square tip	1	each	14537-00
Pipet, volumetric, 5.00 mL, Class A	1	each	14515-37
Pipet Filler, Safety Bulb	1	each	14651-00
Vial, Test ‘N Tube	1	6/pkg	25831-25

OPTIONAL APPARATUS

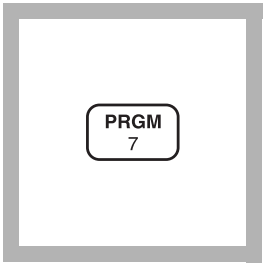
Description	Unit	Cat. No.
Burner, Alcohol, 60 mL	each	20877-42
Burner, Bunsen	each	21627-00
Germicidal Cloth	50/pkg	24632-00
Incubator, Dri Bath, 25 well, 120/230 V	each	45900-00
Incubator, Dri Bath, 25 well, 120/230 V, with European power cord	each	45900-02
Pipet, Sterile Transfer	15/pkg	22325-12
Timer	each	26305-00

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

TURBIDITY (0 to 1000 FAU)

Absorptometric Method*



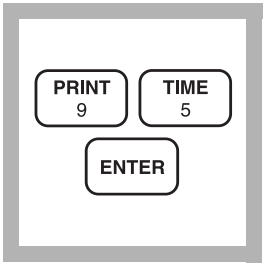
1. Enter the stored program number for turbidity.

Press: **PRGM**

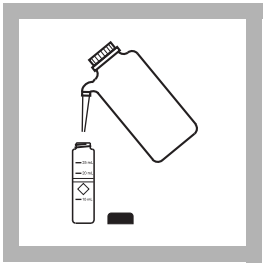
The display will show:

PRGM ?

Note:
1 FAU=1 NTU=1 FTU when measuring formazin. These are not equivalent when measuring other types of standards or samples.



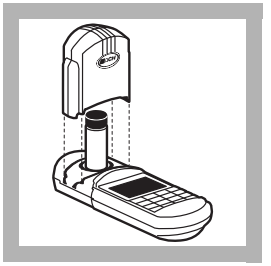
2. Press: **95 ENTER**
The display will show **FAU** and the **ZERO** icon.



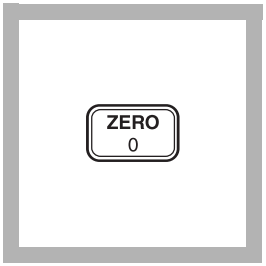
3. Fill a sample cell with 10 mL of deionized water (the blank).

Note: *Wipe the surface of the cell with a soft cloth.*

Note: *For highly colored samples, use a filtered portion of sample in place of the deionized water.*



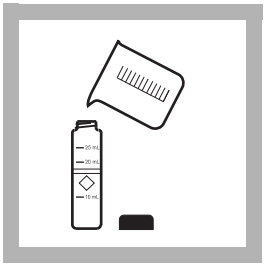
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

The cursor will move to the right, then the display will show:

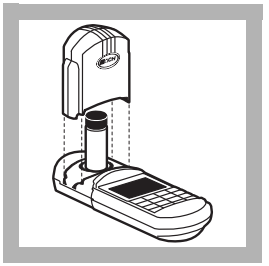
0 FAU



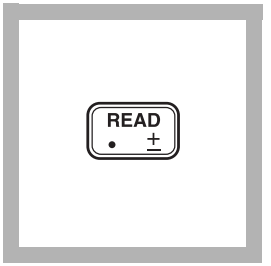
6. Fill another sample cell with 10 mL of sample.

Note: *Mix the sample well before transferring it to the sample cell.*

Note: *Wipe the surface of the cell with a soft cloth.*



7. Place the sample cell into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **READ**

The cursor will move to the right, then the result in Formazin Attenuation Units (FAU) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section I).*

* Adapted from FWPCA *Methods for Chemical Analysis of Water and Wastes*, 275 (1969)

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible. Store samples up to 48 hours by cooling to 4 °C (39 °F). Analyze the sample at the same temperature as it was collected.

Accuracy Check

Standard Solution Method

The stored program has been calibrated using formazin, the primary standard for turbidity. A 200 FAU formazin solution for checking the accuracy of the test can be prepared using the following procedure.

1. Pipet 5.00 mL of a 4000 NTU Formazin stock solution into a 100-mL volumetric flask.

2. Dilute to the mark with deionized water. Prepare this daily.

Convenient stabilized turbidity stock solution (200 NTU StablCal™ Standard) is available from Hach.

Standard Adjust

To adjust the calibration curve using the reading obtained with the
200 FAU formazin standard, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **200** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

Method Precision

Precision

In a single laboratory, using a turbidity standard solution of 200 FAU with the instrument, a single operator obtained a standard deviation of ±2 FAU.

Estimated Detection Limit

The estimated detection limit for program 95 is 21 FAU. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Air Bubbles	Interfere at all levels. Degass samples using the Degassing Kit or an ultrasonic bath.
Color	Interferes if the color absorbs light at 520 nm.
Temperature extremes	May interfere by changing the turbidity of the sample. Analyze samples as soon as possible after collection. Analyze at the same temperature as the original sample.

Summary of Method

This turbidity test measures an optical property of the sample which results from scattering and absorption of light by particles in the sample. The amount of turbidity measured depends on variables such as the size, shape, color, and refractive properties of the particles.

This procedure is calibrated using formazin turbidity standards and the readings are in terms of Formazin Attenuation Units (FAU). This test cannot be used for USEPA reporting purposes, but it may be used for daily in-plant monitoring. One FAU is equivalent to one Nephelometric Turbidity Unit (NTU) of Formazin. However, the optical method of measurement for FAU is very different than the NTU method (1 NTU = 1 FTU = 1 FAU when traced to formazin primary standards.)

REQUIRED APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

REQUIRED REAGENTS

Description	Units	Cat. No.
Formazin Stock Solution, 4000 NTU	500 mL	2461-49
Silicone Oil	15 mL DB	1269-36
StablCal Stabilized Turbidity Standard, 200 NTU	500 mL	26604-49
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Description	Units	Cat. No.
Bath, ultrasonic	each	24895-00
Bottle, wash, 250 mL	each	620-31
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, filter, 500 mL	each	546-49
Filter Holder	each	13529-00
Filter Pump, aspirator	each	2131-00
Oiling cloth, for applying silicone oil	each	26873-00
Pipet Filler, safety bulb	each	14651-00
Pipet, volumetric, Class A, 5.0 mL	each	14515-37
Sample Degassing Kit	each	43975-00
Stopper, rubber, one-hole, No. 7	6/pkg	2119-07
Tubing, rubber, 5/16” I.D.	12 feet	560-19
Tweezers, plastic	each	14282-00

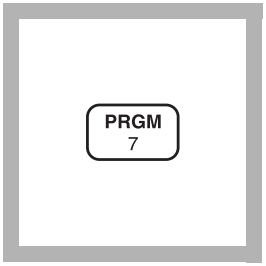
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

VOLATILE ACIDS (0 to 2800 as mg/L HOAc)

Esterification Method*



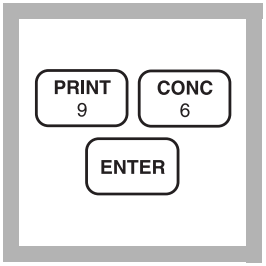
1. Enter the stored program number for Volatile Acids as acetic acid (HOAc).

Press: **PRGM**

The display will show:

PRGM ?

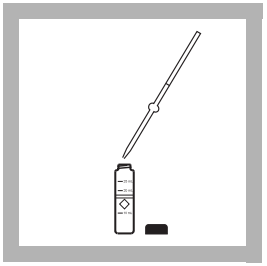
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **96 ENTER**

The display will show **mg/L, HOAc** and the **ZERO** icon**.**

Note: *If high levels of dissolved solids or mineral acids are present, distill as described in the Hach Distillation Apparatus manual.*



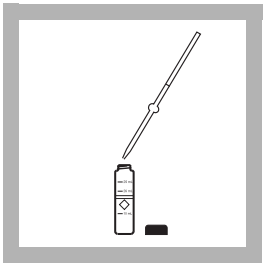
3. Pipet 0.5 mL of deionized water into a dry 25-mL sample cell (the blank).

Note: *Use a Class A or TenSette Pipet.*

Note: *Adjust the pH of stored samples before analysis.*

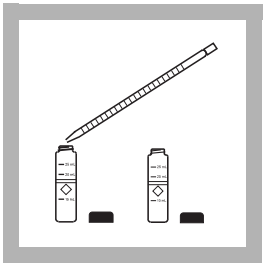
4. Filter or centrifuge 25 mL of the sample.

Note: *Centrifugation is faster than filtration.*

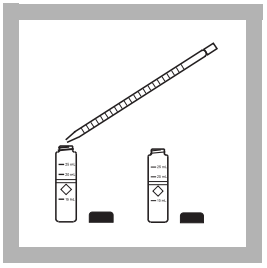


5. Pipet 0.5 mL of the filtrate or supernatant into another dry 25-mL sample cell (the prepared sample).

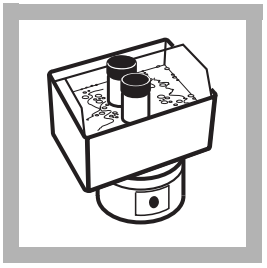
Note: *Use a Class A or TenSette Pipet.*



6. Pipet 1.5 mL of ethylene glycol into each sample cell. Swirl to mix.



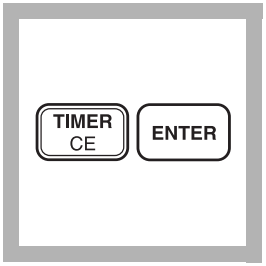
7. Pipet 0.2 mL of 19.2 N Sulfuric Acid Standard Solution into each cell. Swirl to mix.



8. Place both cells into a boiling water bath.

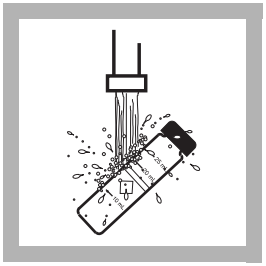
Note: *Samples may be boiled in a 600-mL beaker.*

* Adapted from *The Analyst*, 87, 949 (1962)

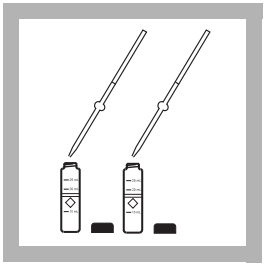


9. Press: **TIMER ENTER**

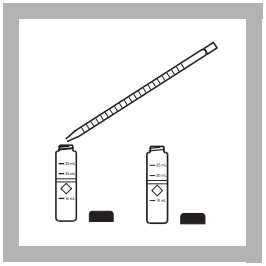
A 3-minute reaction period will begin.



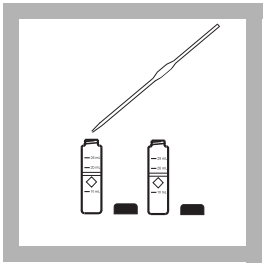
10. When the timer beeps, cool solutions to 25 °C (until cells feel cool) with running tap water. Then dry the cells with a soft cloth.



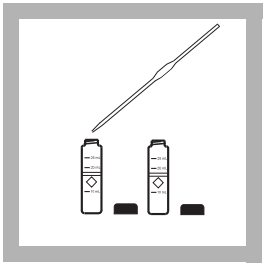
11. Pipet 0.5 mL of Hydroxylamine Hydrochloride Solution into each cell. Swirl to mix.



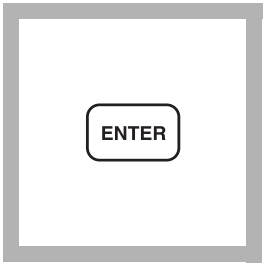
12. Pipet 2.0 mL of 4.5 N Sodium Hydroxide Standard Solution into each cell. Cap and invert to mix.



13. Add 10 mL of Ferric Chloride Sulfuric Acid Solution to each cell. Cap and invert to mix.



14. Add 10 mL of deionized water to each cell. Cap and invert to mix.

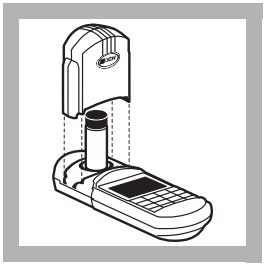


15. The display will show: **3:00 TIMER 2**

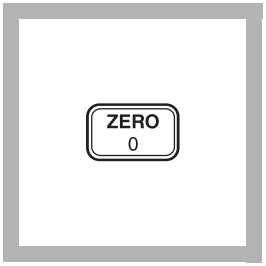
Press: **ENTER**

A 3-minute reaction period will begin.

Note: *After this three-minute reaction period, proceed immediately through steps 16-19.*



16. When the timer beeps, immediately place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

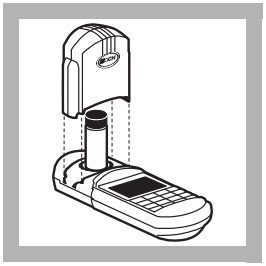


17. Press: **ZERO**

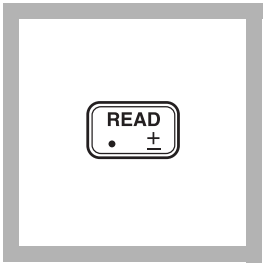
The cursor will move to the right, then the display will show:

0 mg/L HOAc

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



18. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



19. Press: **READ**

The cursor will move to the right, then the result in mg/L Volatile Acids as acetic acid will be displayed.

Sampling and Storage

Collect samples in plastic or glass bottles. Analyze samples as soon as possible after collection. Samples can be stored up to 24 hours by cooling to 4 °C (39 °F) or below. Warm to room temperature before testing.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Volatile Acids PourRite Ampule Standard Solution, 62,500 mg/L as acetic acid.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to three 25-mL graduated mixing cylinders, each containing 25 mL of filtered sample. Stopper. Shake well to mix.

c) Remove a 0.5 mL aliquot of sample from each cylinder; add to three dry sample cells. Analyze all three samples along with the original test sample beginning with Step 5 of the procedure. The volatile acid concentration should increase 250 mg/L volatile acids as acetic acid for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1.*

Standard Solution Method

Prepare a 500 mg/L volatile acid standard by using the TenSette Pipet to add 0.8 mL of a Volatile Acids PourRite Ampule Standard Solution (62,500 mg/L as acetic acid) to a 100-mL volumetric flask. Dilute to volume with deionized water. Stopper and invert to mix.

Method Performance

Precision

In a single laboratory, using a standard solution of 500 mg/L volatile acids as acetic acid and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±8 mg/L.

Estimated Detection Limit

The estimated detection limit for program 96 is 17 mg/L HOAc. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

The volatile acids test is designed specifically for the determination of volatile acids in digestor sludges. The method is based on esterification of the carboxylic acids present and determination of the esters by the ferric hydroxamate reaction. All volatile organic acids present are reported as their equivalent mg/L acetic acid.

REQUIRED REAGENTS

	Cat. No.
Volatile Acids Reagent Set (90 tests)	22447-00
Includes: (1) 2039-53, (2) 2042-53, (1) 818-42, (1) 2040-53, (1) 2038-32	

Description	Quantity Required Per Test	Units	Cat. No.
Ethylene Glycol	3 mL	1000 mL	2039-53
Ferric Chloride-Sulfuric Acid Solution	20 mL	1000 mL	2042-53
Hydroxylamine Hydrochloride Solution, 100 g/L	1 mL	100 mL	818-42
Sodium Hydroxide Standard Solution, 4.5 N	4 mL	1000 mL	2040-53
Sulfuric Acid Standard Solution, 19.2 N	0.4 mL	100 mL	2038-32
Water, deionized	20.5 mL	4 L	272-56

REQUIRED APPARATUS

Description	Quantity Required Per Test	Units	Cat. No.
Cots, finger	2	2/pkg	14647-02
Cylinder, graduated, 10 mL	1	each	508-38
Filter Paper, folded, 12.5 cm	1	100/pkg	1894-57
Flask, erlenmeyer, 50 mL	1	each	505-41
Funnel, poly, 65 mm	1	each	1083-67
Hot Plate, circular, 3.5-inch diameter	1	each	12067-01
Pipet Filler, safety bulb	1	each	14651-00
Pipet, serological, 2 mL	2	each	532-36
Pipet, volumetric, Class A, 0.5 mL	3	each	14515-34
Pipet, volumetric, Class A, 10.00 mL	3	each	14515-38
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
Water Bath and Rack	1	each	1955-55

OPTIONAL REAGENTS

Volatile Acids Standard Solution, PourRite ampule, 62,500 mg/L as acetic acid, 10 mL	16/pkg	14270-10

OPTIONAL APPARATUS

Ampule Breaker, PourRite	each	24846-00
Beaker, 600 mL	each	500-52
Bottle, wash, 500 mL	each	620-11
Centrifuge, laboratory, 115 Vac	each	26765-00
Centrifuge, laboratory, 230 Vac	each	26765-02
Centrifuge Tubes, 15 mL	10/pkg	22787-39
Centrifuge Tube Caps	20/pkg	25852-20
Cylinder, graduated, mixing, 25 mL	each	1896-40
Cylinder, graduated, plastic, 250 mL	each	1081-46
Distillation Apparatus	each	22653-00
Distillation Heater and Support Apparatus	each	22744-00
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet Tips, for 19700-10	50/pkg	21997-96

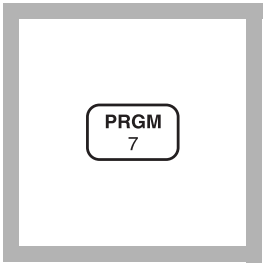
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

ZINC (0 to 3.00 mg/L Zn)

Zincon Method* USEPA approved for wastewater analysis** (digestion needed; see Section 2)



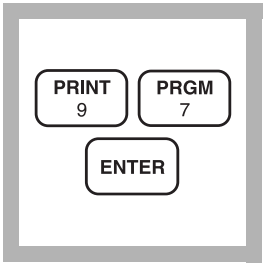
1. Enter the stored program number for zinc (Zn).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

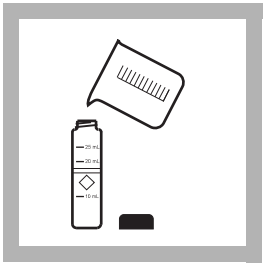


2. Press: **97 ENTER**

The display will show **mg/L, Zn** and the **ZERO** icon**.**

Note: *Total zinc requires a prior digestion; use either the Digesdahl or mild digestion (Section 2).*

Note: *Adjust the sample to pH 4-5; see Sampling and Storage following these steps.*



3. Fill a 25-mL sample cell with 20 mL of sample.

Note: *Rinse glassware with 1:1 hydrochloric acid and deionized water before use.*

Note: *If samples cannot be analyzed immediately, see Sampling and Storage.*

4. Add the contents of one ZincoVer 5 Reagent Powder Pillow. Cap. Invert several times to completely dissolve the powder. If the sample does not turn orange, see the note below.

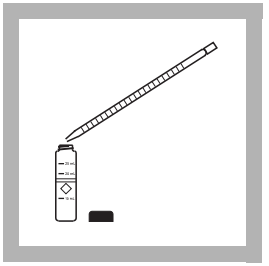
Note: *Powder must be completely dissolved or inconsistent results may occur.*

Note: *The sample should be orange. If it is brown or blue, dilute the sample and repeat the test. Either the zinc concentration is too high or an interference is present.*

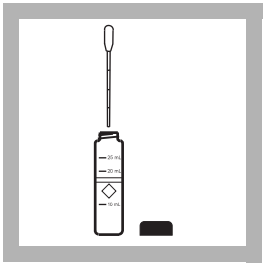
Caution: **ZincoVer 5 contains cyanide and is very poisonous if taken internally or inhaled. Do not add to an acidic sample. Store away from water and acids.**

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

** *Federal Register,* 45 (105) 36166 (May 29, 1980).

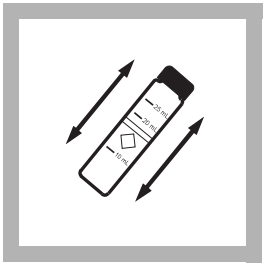


5. Measure 10 mL of the orange solution into another sample cell (the blank).



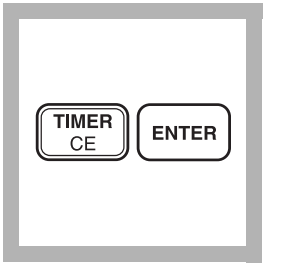
6. Add 0.5 mL of cyclohexanone to the remaining orange solution in the first sample cell (the prepared sample).

Note: *Use a plastic squeezer. Rubber bulbs may contaminate the cyclohexanone.*



7. Tightly cap the cell. Shake vigorously for 30 seconds (the prepared sample).

Note: *The sample will be red-orange, brown or blue, depending on the zinc concentration.*

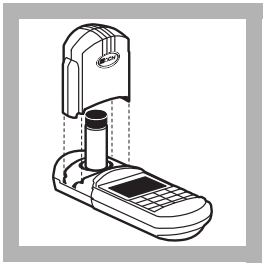


8. Press:

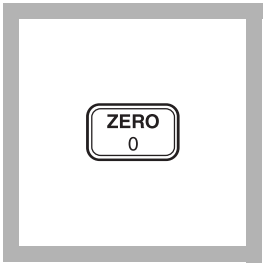
TIMER ENTER

A 3-minute reaction period will begin.

Note: *Steps 9-11 must be completed within 10 minutes after the timer beeps.*



9. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

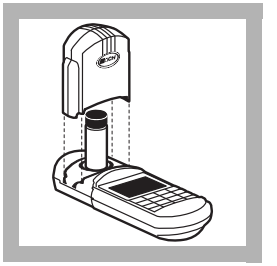


10. Press: **ZERO**

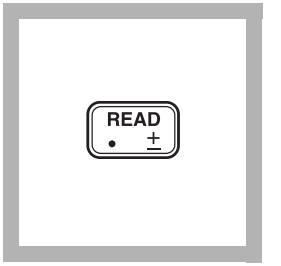
The cursor will move to the right, then the display will show:

0.00 mg/L Zn

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



11. Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L Zn will be displayed.

Note: *Standard Adjust may be performed using a prepared 0.50 mg/L standard. See Section 1.*

Sampling and Storage

Collect samples in acid-washed plastic bottles. For storage, adjust the pH to 2 or less with nitric acid (about 2 mL per liter). The preserved samples can be stored up to six months at room temperature.

Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as zinc may be lost as a precipitate. Correct the test result for volume additions; see *Sampling and Storage, Volume Additions,* in *Section 1* for more information.

If only dissolved zinc is to be determined, filter the sample before the acid addition.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Zinc PourRite Ampule Standard, 25 mg/L Zn.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The zinc concentration should increase 0.1 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 0.50 mg/L zinc standard solution by diluting 5.00 mL of Zinc Standard Solution, 100 mg/L as Zn, to 1000 mL with deionized water in a Class A 1000-mL volumetric flask. Prepare this solution daily. Use this solution as the sample and perform the zinc procedure as described above.

Method Performance

Precision

In a single laboratory, using a standard solution of 1.50 mg/L Zn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.02 mg/L Zn.

Estimated Detection Limit (EDL)

The EDL for program 97 is 0.02 mg/L Zn. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below.

Interfering Substance	Interference Level and Treatments
Aluminum	6 mg/L
Cadmium	0.5 mg/L
Copper	5 mg/L
Iron (ferric)	7 mg/L
Manganese	5 mg/L
Nickel	5 mg/L
Organic material	Large amounts may interfere. Perform the mild digestion (Section 2) to eliminate this interference.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment (see pH Interference in Section 1). Adjust pH to 4-5.

Pollution Prevention and Waste Management

ZincoVer 5 reagent contains potassium cyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Cyanide should be collected for disposal as reactive (D003) waste. Be sure that cyanide solutions are stored in a caustic solution with pH >11 to prevent the release of hydrogen cyanide gas.

In the event of a spill or release, clean up the area by following these steps:

a) Use a fume hood or supplied-air or self-contained breathing apparatus.

b) While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochlorite (household bleach).

c) Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.

d) Neutralize and flush the solution down the drain with a large excess of water.

Summary of Method

Zinc and other metals in the sample complex with cyanide. Adding cyclohexanone selectively releases zinc. The zinc then reacts with the 2-carboxy-2'-hydroxy-5'-sulfoforamazyl benzene (zincon) indicator and forms a blue color that is proportional to the zinc concentration.

REQUIRED REAGENTS

Zinc Reagent Set, 20 mL size (100 tests) 24293-00
Includes: (1) 14033-32, (1) 21066-69

Description	Quantity Required Per Test	Units	Cat. No.
Cyclohexanone	0.5 mL	100 mL MDB	14033-32
ZincoVer 5 Reagent Powder Pillows	1 pillow	100/pkg	21066-69

REQUIRED APPARATUS

Pipet, serological, 10 mL	1	each	532-38
Pipet Filler, safety bulb	1	each	14651-00
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
Squeezers, plastic dropper	1	20/pkg	21247-20

OPTIONAL REAGENTS

Bleach, household	1 gal	buy locally
Cylinder, graduated, mixing, 25mL	each	20886-40
Hydrochloric Acid Standard Solution, 6 N	500 mL	884-49
Nitric Acid, ACS	500 mL	152-49
Nitric Acid 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	50 mL SCDB	2450-26
Water, deionized	4 L	272-56
Zinc Standard Solution, 100 mg/L Zn	100 mL	2378-42
Zinc Standard Solution, PourRite ampule, 25 mg/L as Zn, 2mL	20/pkg	14246-20

OPTIONAL APPARATUS

Ampule Breaker, PourRite ampules	each	24846-00
Aspirator, vacuum	each	2131-00
Beaker, glass, 1000 mL	each	500-53
Cylinder, graduated, 100 mL	each	508-42
Cylinder, graduated, mixing, 250 mL	each	26362-46
Filter discs, glass, 47 mm	100/pkg	2530-00
Filter holder, 47 mm	each	2340-00
Flask, erlenmeyer, 250 mL	each	505-46
Flask, filtering, 500 mL	each	546-19

OPTIONAL APPARATUS (continued)

Description	Units	Cat. No.
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 1000 mL	each	14574-53
Hot plate, micro 115 V	each	12067-01
Hot plate, micro 230 V	each	12067-02
pH paper, 1 to 11 pH	5 rolls/pkg	391-33
pH meter, *Sens**ion**™1*, portable with electrode	each	51700-10
Pipet filler, safety bulb	each	14651-00
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet, TenSette, tips for 19700-01	50/pkg	21856-96
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet, TenSette, tips for 19700-01	1000/pkg	21856-28
Pipet, TenSette, tips for 19700-10	50/pkg	21997-96
Pipet, TenSette, tips for 19700-10	250/pkg	21997-25
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 0.5 mL	each	14515-34

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

By Phone:
6:30 a.m. to 5:00 p.m. MST
Monday through Friday
800-227-HACH (800-227-4224)

By Mail:
Hach Company
P. O. Box 389
Loveland, Colorado 80539-0389 U.S.A.

By FAX:
970-669-2932 (Hach Loveland)

Information Required:

- Hach account number (if available)
- Billing address
- Shipping address
- Your name and phone number
- Purchase order number
- Catalog number
- Brief description or model number
- Quantity

Technical and Customer Service

Hach Technical and Customer Service Department personnel are eager to answer questions about our products and their use and to take your orders. Specialists in analytical methods, they are happy to put their talents to work for you. Call **1-800-227-4224**.

International Customers

Hach maintains a network of dealers and distributors throughout the world.

In Canada
Hach Sales and Service Canada Ltd.
1313 Border Street, Unit 34
Winnipeg, Manitoba R3H 0X4
Telephone: (204) 632-5598
FAX: (204) 694-5134

In other countries, contact:
Hach Company World Headquarters
P. O. Box 389
Loveland, Colorado, U.S.A. 80539-0389
Telephone: (1) (970) 669-3050
FAX: (1) (970) 669-2932

Information presented on these pages applies only to Hach products manufactured for use within the United States. Exportation of these products renders these terms void.

Prices and Terms

Prices are subject to change without notice. All prices are FOB from the shipping point (usually Ames, Iowa). Hach offers instant credit up to $200 on Net 30 Day terms. Larger orders are subject to credit review. Customers may send remittance with orders or we can ship C.O.D. if you prefer.

Warranty

Hach warrants its products to be of high quality, to be free of material defects on the date of shipment and to be as specified.

Limits of Usage

Our chemicals and reagents are offered for laboratory and manufacturing use ONLY. They may not be used as drugs, cosmetics or food additives.

MSDS

Hach Material Safety Data Sheets, among the most complete and informative in the industry, provide comprehensive safety data essential for day-to-day operations and safety training.

An MSDS accompanies all Hach chemical products including test kits. For an additional cost, we will print MSDSs on your own forms.

ADDITIONAL INFORMATION

Label Information

Labels on Hach chemicals and reagents supply the following:

- Product Name -- In French, German, Italian and Spanish as well as English is printed on all but the smallest-size labels.
- Hach Catalog Number -- Makes reordering easy and helps match the appropriate MSDS.
- Storage Information and Lot Numbers -- Lot numbers made up of letters and numbers indicate an extended shelf life; a four-digit number indicates items should be rotated and checked with a standard to confirm performance. The lot number is essential if you call for technical assistance or with questions about reagent performance.

Shipping

Our experienced warehouse staff packages your orders for safe arrival. Unless we are instructed otherwise, the best and most efficient mode of transportation is selected. Motor freight shipments will be sent freight collect unless you specify otherwise at the time you order.

If you have questions about methods for shipment and availability of special packaging, please ask when you place your order.

Claims and Returns

We take extreme care to fill, check, re-check and pack orders properly. If errors or damages should occur, please report details to our Loveland Customer Service Department and to the carrier immediately. Be sure to keep all containers and packing materials.

AUTHORIZATION MUST BE OBTAINED from Hach when returning items for any reason. Call 1-800-227-4224 toll free. **ALL "FREIGHT COLLECT" SHIPMENTS OR MERCHANDISE RETURNED WITHOUT PROPER AUTHORIZATION FROM HACH WILL BE REFUSED.**

USER MANUAL
DOCUMENT #626973-01REF

ProDSS

USER MANUAL

YSI
a xylem brand

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Contents

Introduction

Thank you for purchasing the YSI Professional Digital Sampling System (ProDSS).

ProDSS features include:

- Digital smart probes that are automatically recognized by the instrument when connected
- Waterproof (IP-67) case
- Long-life rechargeable lithium-ion battery pack
- Color display and backlit keypad
- User-selectable cable options
- USB connectivity
- Global Positioning System (GPS) (optional)
- Depth sensor (optional)
- Large memory with extensive site list capabilities
- Rugged enclosure with rubber over-molded case and miltary-spec (MS) connectors
- KorDSS data management software included with each instrument (Please see installation instructions on page 67)

Safety information

Please read this entire manual before unpacking, setting up or operating this equipment. Pay attention to all precautionary statements. Failure to do so could result in serious injury to the operator or damage to the equipment. Make sure that the protection provided by this equipment is not impaired. Do not use or install this equipment in any manner other than that specified in this manual.

NOTICE: The manufacturer is not responsible for any damages due to misapplication or misuse of this product including, without limitation, direct, incidental and consequential damages, and disclaims such damages to the full extent permitted under applicable law. The user is solely responsible to identify critical application risks and install appropriate mechanisms to protect processes during a possible equipment malfunction.

Precautionary symbols

NOTE: *Information that requires special emphasis*

NOTICE: Indicates a situation which, if not avoided, may cause damage to the instrument

⚠ **CAUTION:** Indicates a potentially hazardous situation that may result in minor or moderate injury

⚠ **WARNING:** Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury

Product components

Carefully unpack the instrument and accessories and inspect for damage. If any parts or materials are damaged, contact YSI Customer Service at 800-897-4151 (+1 937 767-7241) or the authorized YSI distributor from whom the instrument was purchased.

Battery use and battery life

The ProDSS uses a rechargeable lithium-ion (Li-Ion) battery pack as a power source. The battery comes pre-installed in the ProDSS and does not need to be replaced until the battery charge capacity is deemed unacceptable by the user. The battery is shipped at less than 50% full capacity and charging the battery is not required before first use.

Battery life depends on use, enabled parameters, LCD brightness, and GPS use. As with all lithium-ion batteries, battery life will decline over time and use. This decay is typical and should be expected.

A new ProDSS battery is expected to last for the following durations (25 °C (77 °F), auto sampling, GPS on, keypad backlight off):

- ProDSS instrument only - 48 hours
- ProDSS with fully loaded 4 port cable assembly and 25% LCD brightness - 20 hours
- ProDSS with fully loaded 4 port cable assembly and 100% (Default) LCD brightness - 14 hours

To increase battery life, enable manual sampling mode (Sampling on page 20). Manual sampling mode powers the sensor/s on to take a measurement and then powers down to conserve battery life. Battery life may also depend on the battery charging practices used. For maximum battery life, keep the battery 40% to 80% charged. Also, a larger discharge (e.g. to 50%) is better than a small discharge (e.g. to 90%) between recharges.

Charging the battery pack

A USB cable is included with the ProDSS to charge the instrument battery pack and connect the instrument to a PC. The instrument battery pack can be charged from the AC power adapter, directly from a computer USB connection or from an external, portable USB battery pack (sold separately, see ProDSS accessories on Page 79).

Plug the USB connector into the AC power adapter, computer USB connector or external USB battery pack, then plug the micro USB connector into the ProDSS instrument (Figure 1).

NOTE: *The ProDSS internal charge controller only allows the battery pack to be charged if the temperature is between 0 and 45 °C (32 and 113 °F).*

⚠ **WARNING:** Charge the battery pack in an open area away from flammable materials, liquids, and surfaces. Read Rechargeable Lithium-Ion battery pack safety warnings and precautions on page 80.

The ProDSS will charge faster when plugged into an AC outlet for charging rather than a PC's USB port. For the instrument to recognize that it is using AC power, you must start charging the ProDSS while on. After the instrument recognizes it is being charged, it can be turned off to finish charging.

When using the AC adapter, it takes approximately 14 hours to charge the ProDSS battery when the instrument is turned ***off*** during the charge. The amount of time required to completely charge the battery pack when the ProDSS is initially turned ***on*** during the charge is approximately 9 hours.

Figure 1 Connecting the ProDSS to AC power supply

Battery replacement

NOTE: *The battery pack is pre-installed in the ProDSS instrument.*

⚠ **WARNING:** Do not charge or handle a battery pack that is hot to the touch. Failure to follow the safety warnings and precautions can result in personal injury and/or instrument damage not covered under warranty. Read Rechargeable Lithium-Ion battery pack safety warnings and precautions on page 80.

1. Remove the battery pack cover by unscrewing (counter-clockwise) the four screws with a flat or Phillips head screwdriver (Figure 2 on page 7).

   NOTE: *The retaining screws are captured into the battery pack cover and are not removable.*

2. If replacing an existing battery pack, remove the Li-Ion battery pack and battery pack gasket/cradle. With two fingers, grasp the battery pack connector and pull the connector straight up to disconnect and remove.

   NOTE: *Properly dispose of the old battery pack (See Battery Disposal on page 81).*

   NOTE: *A new gasket/cradle is included with a new battery pack to prevent water leaking into the instrument case. When replacing the battery pack, use the new battery pack gasket/cradle supplied with the replacement battery pack.*

3. Inspect the replacement battery pack and battery pack gasket/cradle for damage. Contact YSI customer service if the new battery pack and/or replacement gasket/cradle is damaged.
4. Correctly align and seat the battery pack gasket/cradle and battery pack into the instrument.
5. Align the battery pack connector wire terminals with the three instrument pins, then connect the battery pack to the instrument.

   NOTICE: Make sure that the three wire terminal connectors and three instrument pins are correctly aligned before connecting the battery pack connector. Incorrect installation can damage the battery pack connectors or instrument pins.

6. Install the battery pack cover, then hand tighten the cover screws with a screwdriver. DO NOT use any power tools. Make sure that the cover sealing surface is correctly aligned and free of any contamination or damage.

   NOTICE: Overtightening the cover screws can damage the battery cover.

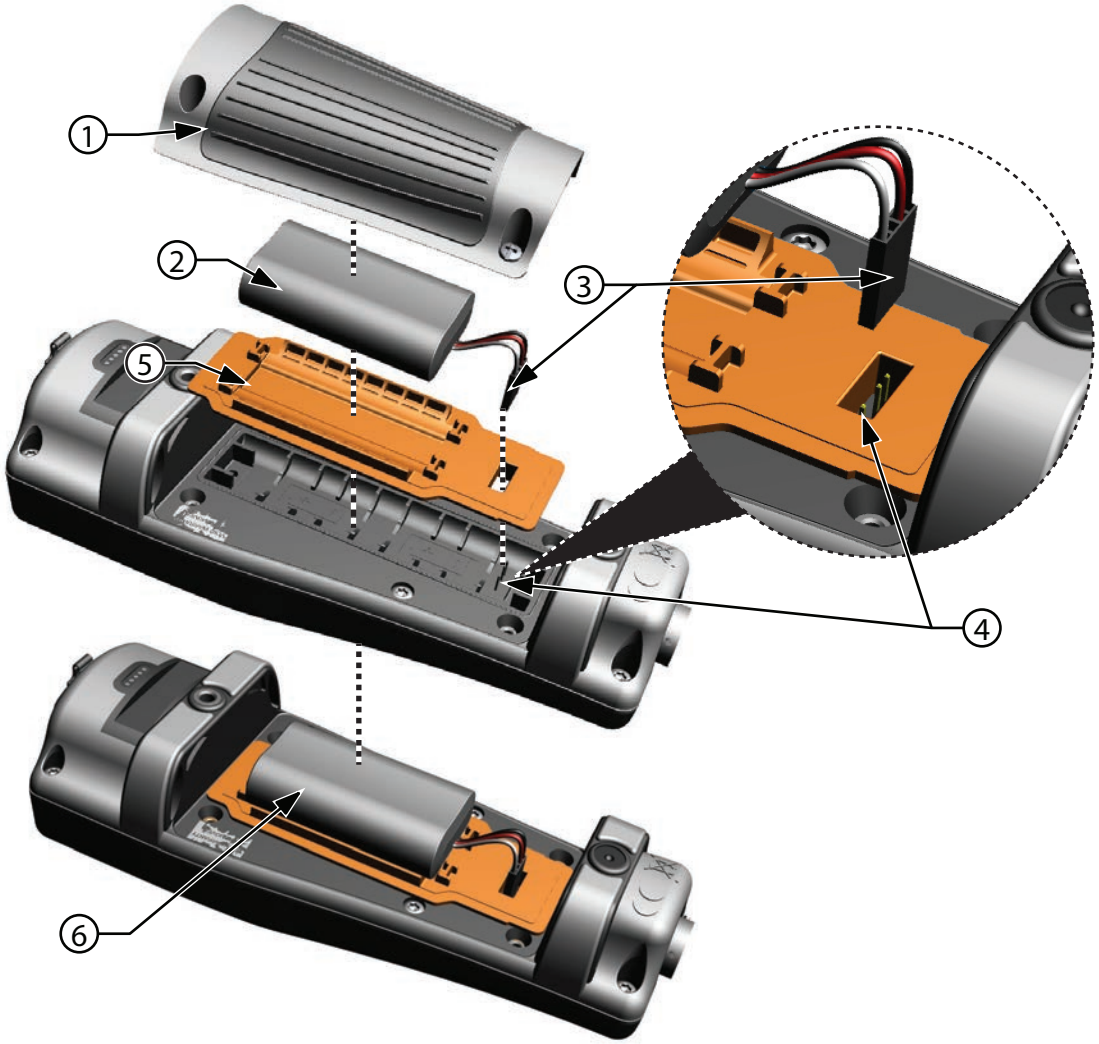


Figure 2 Battery replacement

1 Battery pack cover	**4** Instrument pin connectors
2 Battery pack	**5** Battery pack gasket/cradle*
3 Battery pack connector	**6** Battery pack gasket/cradle installed

*Color shown for reference

Connect the handheld to the cable assembly

The ProDSS cable connectors are keyed for positive mating and to prevent connector damage (Figure 3). The ProDSS instrument retains its IP-67 rating when the cable is disconnected. However, the connectors are not wet-mateable and should be clean and dry before connecting.

Align the keys on the cable assembly connector with the slots on the instrument connector. Push together firmly, then twist the outer ring clockwise until it locks into place.

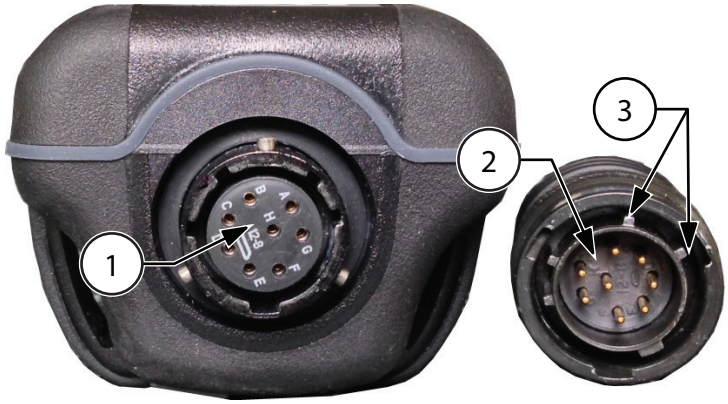


Figure 3 Keyed connectors

1	Handheld female connector	**3**	Keyed area of connectors
2	Cable male connector		

ProDSS sensor installation/removal

ProDSS cable assemblies that feature user-replaceable sensors include 4 port cables with (626910 and 626911) and without (626909) a built-in depth sensor. ***This section pertains to these cable assemblies.***

Other ProDSS cable assemblies, like the ProDSS ODO/CT (627150), ProODO (626250) and ProOBOD (626400 and 626401), feature integral (i.e. built-in) sensors. Therefore, sensors on these cable assemblies cannot be replaced by the user. A complete list of cable options can be found in the Accessories section.

NOTICE: The ProDSS bulkhead and sensor connectors are not wet-mateable. Make sure that the sensor and bulkhead connectors are clean and dry before sensor installation.

NOTE: *Sensor ports on the bulkhead (4 port cables only) are numbered (Figure 5). If multiple sensors of the same type are installed, the sensor port number will be added to the Run screen display to clarify the measurement value of each sensor.*

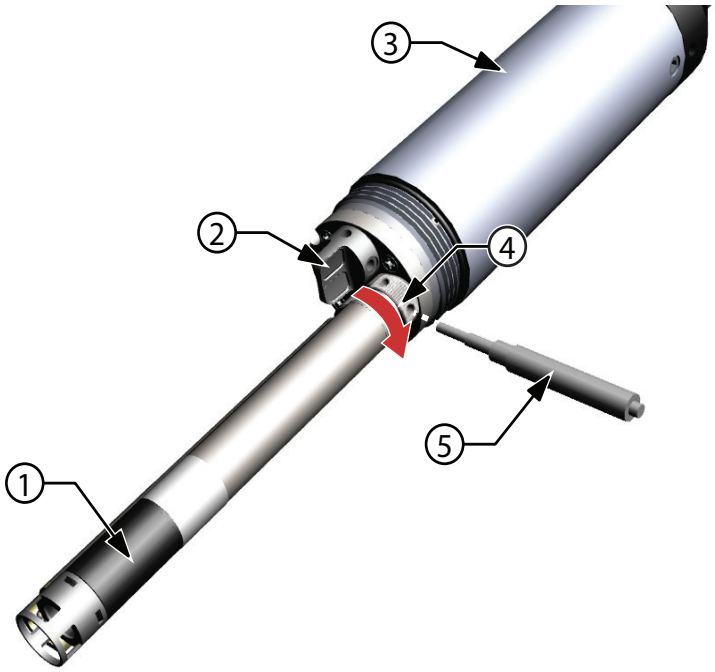


Figure 4 Sensor installation

1	Sensor	**4**	Sensor retaining nut
2	Port plug	**5**	Sensor installation/removal tool
3	Bulkhead (4 port pictured)		

Sensor installation

The ports on the ProDSS bulkhead are universal; therefore, you can install any sensor into any port.

NOTE: *A conductivity/temperature sensor (626902) must be installed in a 4 port ProDSS cable for accurate measurement of all parameters except turbidity and TSS. All sensors, including conductivity/temperature, must be ordered separately.*

1. Remove the port cover shipped with ProDSS cables. This cover fits over the bulkhead to protect the sensor connectors from contamination and damage during shipment. This cover can be kept for long-term cable storage.
2. Inspect the bulkhead port for contamination. If the port is dirty or wet, clean it with compressed air.
3. Apply a thin coat of o-ring lubricant to the sensor o-rings. Wipe off excess o-ring grease with a lint-free cloth.

4. Carefully align the sensor and bulkhead connectors by inserting the sensor into the port then gently rotating the sensor until the connectors align. Once aligned, push the sensor toward the bulkhead until the sensor seats in the port.
5. Carefully finger-tighten the retaining nut clockwise.

   NOTICE: If any resistance is felt, loosen the retaining nut completely to prevent cross-threading. Incorrect installation may cause damage to the sensor or bulkhead that is not covered by the warranty.

6. Use the sensor installation/removal tool to tighten the retaining nut clockwise until snug, about a 1/4 to 1/2 additional turn of the retaining nut.

   NOTICE: Do not over-tighten the retaining nut. Over-tightening can cause damage to the sensor or bulkhead not covered by the warranty.

Sensor removal

To remove a sensor, insert the sensor installation/removal tool into the retaining nut, then rotate the retaining nut counterclockwise to loosen. After the retaining nut has been completely unscrewed from the bulkhead, pull the sensor straight out of the port and place it on a clean surface.

NOTICE: Install a port plug if not reinstalling a sensor in the exposed port. Exposure to water can cause damage or corrosion to the bulkhead connectors not covered by the warranty.

Port plugs

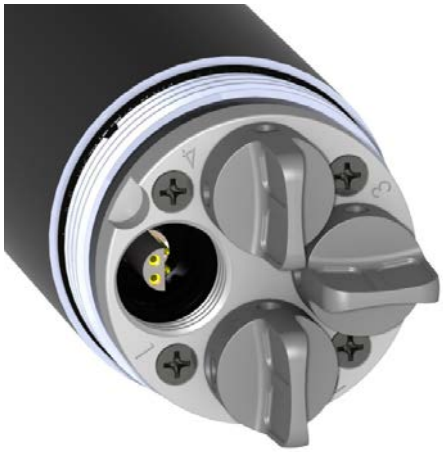


Figure 5 Sensor port plugs and port numbering (4 port cables)

To protect the bulkhead connectors from damage, install a port plug into any port without an installed sensor. Port plugs and a tube of o-ring lubricant are included in the maintenance kit that ships with all 4 port ProDSS cables. Refer to the Accessories section if an additional maintenance kit is needed.

NOTICE: Do not submerge the bulkhead without a sensor or port plug installed in all ports.

Installation

1. Apply a thin coat of o-ring lubricant to the o-rings on the plug port.
2. Remove any excess lubricant from the o-rings and port plug with a lint-free cloth.
3. Insert the port plug into the empty port and press until firmly seated.
4. Finger-tighten the port plug clockwise to install. If necessary, use the sensor installation tool to make sure that the plug is fully seated into the port.

   NOTICE: The o-rings will not be visible if a port plug is correctly installed. Do not over-tighten the port plug. Over-tightening can cause damage to the port plug or bulkhead not covered by the warranty.

Sensor guard and weight installation

1. Carefully slide the sensor guard over the bulkhead and attached sensors/port plugs. Push the sensor guard toward the bulkhead until the sensor guard threads align with the bulkhead threads.
2. Carefully finger-tighten the sensor guard clockwise.

NOTICE: If any resistance is felt, loosen the sensor guard completely to prevent cross-threading. Incorrect installation may cause damage to the sensor guard or bulkhead that is not covered by the warranty.

NOTICE: Do not submerge the bulkhead without a sensor or port plug installed in all ports.

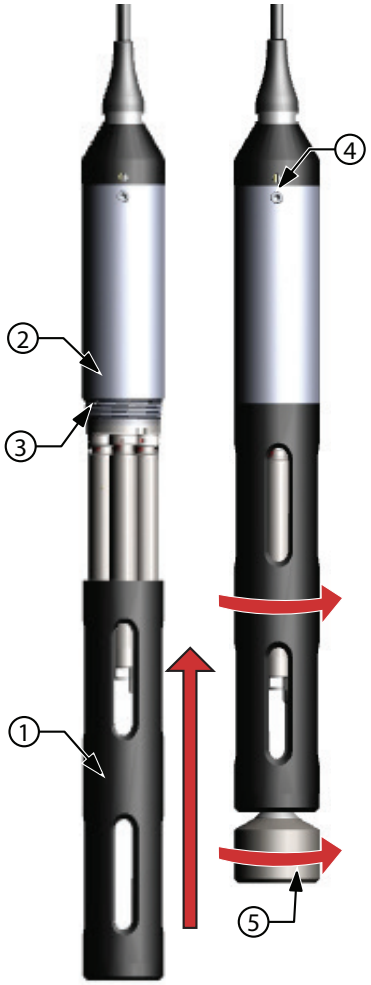


Figure 6 Sensor guard and weight installation on a 4 port cable assembly

1 Sensor guard	**4** Depth sensor (if equipped)
2 Bulkhead	**5** Weight
3 Bulkhead threads	

Sensor guard weights

To help stabilize the sensors when profiling at deeper depths, a 1 lb. sensor guard weight is supplied with 4 port assemblies 10 meters and longer. To attach the weight, carefully hand-tighten it clockwise on to the bottom of the sensor guard (Figure 6 on page 11).

NOTICE: If any resistance is felt, loosen the sensor guard weight completely to prevent cross-threading. Incorrect installation may cause damage to the sensor guard.

The bottom of the weight is threaded so that additional weights can be added if needed. See ProDSS accessories on page 79.

NOTE: *Do not have any weights installed on the sensor guard when calibrating using the calibration cup.*

Operation

Keypad and navigation

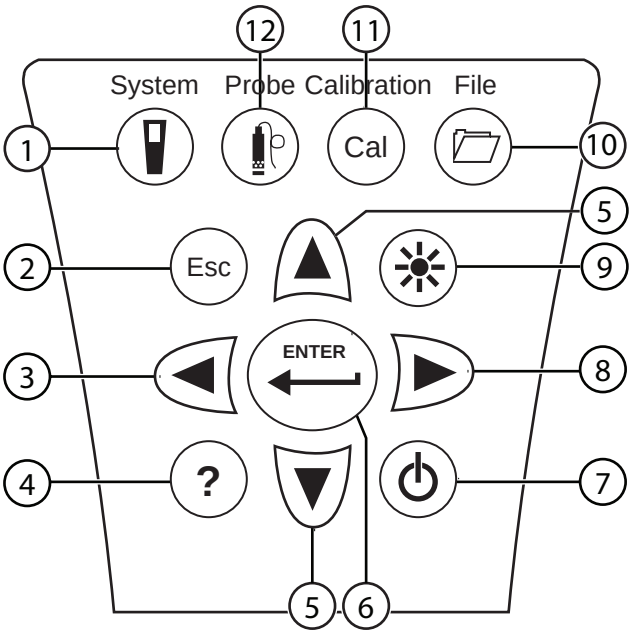


Figure 7 Keypad description

1 **System:** Opens the system menu. Use to adjust system settings	**7** **ON/OFF:** Turn on or turn off the instrument
2 **Exit/Escape key:** Exits to the Run screen. When in an alpha/numeric entry screen, returns to previous menu	**8** **Right arrow key:** Navigate right in an alpha/numeric entry screen. On the Run screen, push to show graphical representations of the displayed measurements. Push the right or left arrow to return to the Run screen. In the View Data screen, push to view additional parameters in the data set
3 **Left arrow key:** Navigate left in an alpha/numeric entry screen. Push to return to previous menu in all screens except alpha/numeric entry. On the Run screen, push to show graphical representations of the displayed measurements. Push the right or left arrow to return to the Run screen	**9** **Backlight:** Turns the keypad backlight on or off for use in low light conditions
4 **Help:** Shows context sensitive help	**10** **File:** Opens the file menu. Use to view logged data and GLP files, backup data to a USB stick, and delete data
5 **Up/down arrow keys:** Scroll through menus or enter numbers and letters	**11** **Calibrate:** Opens the calibration menu. Use to calibrate all parameters except temperature
6 **Enter key:** Push to confirm selections. On the Run screen, push to log a single data point or start continuous data logging	**12** **Probe:** Opens the sensor menu. Use to setup sensors, change the measurements shown on the run screen, select the sensor averaging mode, and turn on/off Auto Stable and GPS

Operation

Startup

Push the ⏻ key to turn on the handheld. If the handheld does not turn on, make sure that the battery pack is correctly installed and charged. Push and hold the ⏻ key for 1.5 seconds to turn the handheld off.

Navigation

The ProDSS contains menus to change user-defined options, functions, and parameters. Use the arrow keys (▲ and ▼) to highlight different options within menus and sub-menus, then push the ENTER key to select the option. Push the ◄ key to return to the previous menu.

> ***NOTE:*** *When in an alpha/numeric screen, the ◄ key is for alpha/numeric navigation only. Push the Esc key to return to the previous menu.*

Push the Esc key to return to the Run screen. To enable or disable an option, highlight the option, then push the ENTER key. Enabled functions appear as a circle with a dot ◉ or a box with a check mark ☑ . Disabled functions appear as a circle only ○ or an empty box ☐.

Alpha/numeric entry

When required, an alpha or alpha/numeric entry screen will be shown. When finished entering information, highlight **ENTER**, then push the ENTER key to save the entry (Figure 8).

> ***NOTE:*** *When in an alpha/numeric screen, the ◄ key is for alpha/numeric navigation only. Push the Esc key to return to the previous menu.*

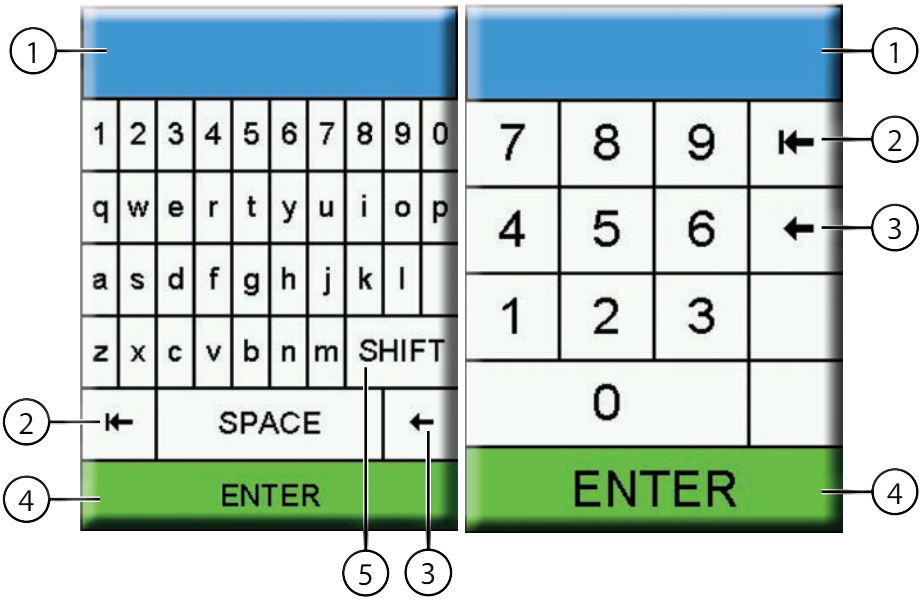


Figure 8 Alpha/numeric and numeric entry screens

1	User entry field	**4**	Enter selection
2	Delete entire entry	**5**	Upper/lowercase
3	Backspace		

Main display description

The main display (Run screen) shows the current measurements as defined in the Sensor Display menu (Sensor Display on page 25). If more measurements are selected than can be displayed on the Run screen, a scroll bar will be shown. Use the ▲ and ▼ arrow keys to view the additional measurements (Figure 9).

The message area shows status messages, error messages, and information about selected functions.

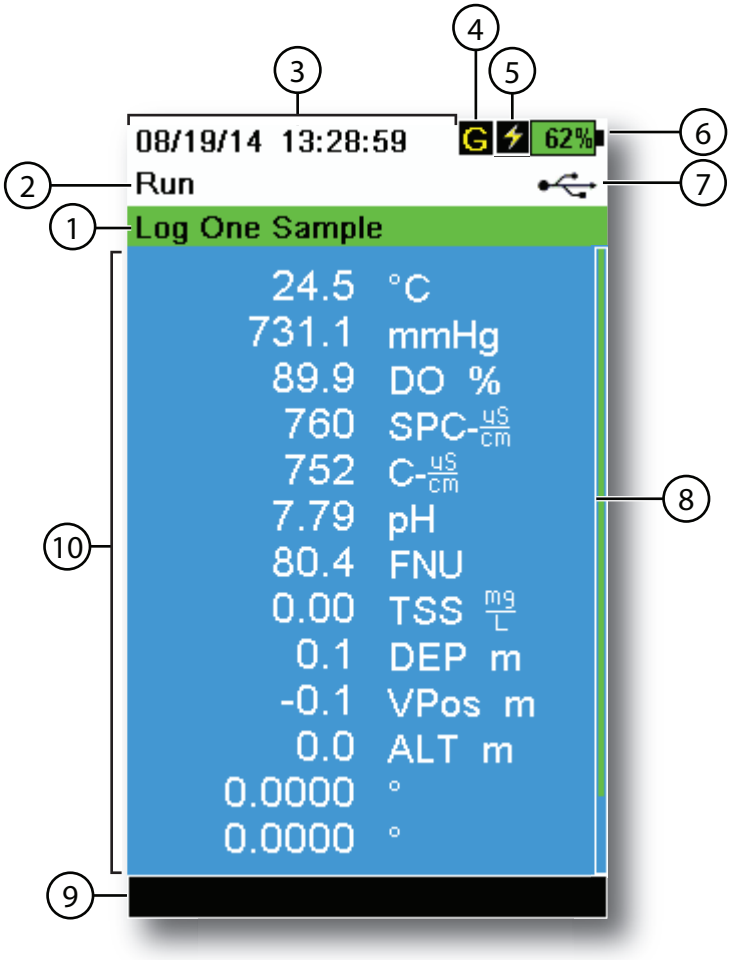


Figure 9 Main display example

1	Log or sampling (update measurements) prompt on Run screen (single or continuous)	**6**	Battery charge %
2	Current screen/menu	**7**	USB/PC connection indicator
3	Date/Time	**8**	Scroll bar
4	GPS signal indicator	**9**	Message area
5	Battery charging indicator	**10**	Displayed measurements

System menu

Push the System key to view and adjust instrument settings. Highlight a sub-menu then push the ENTER key to view the sub-menu options (Figure 10).

Pre-defined or user-selected options are noted within brackets (**[]**). See Alpha/numeric entry on page 14.

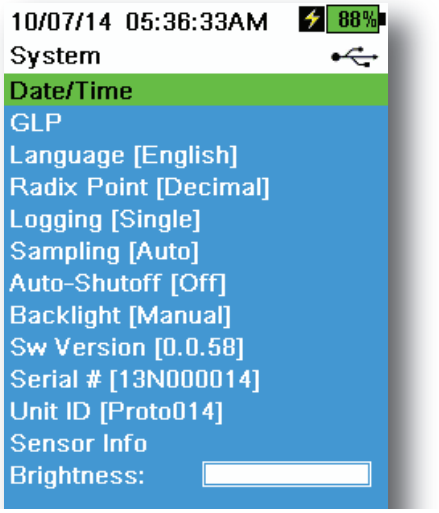


Figure 10 System menu

Use the System menu to:

- Set the date and time (Date/Time on page 17)
- Change the user-defined Good Laboratory Practices (GLP) options (GLP menu on page 17)
- Change the instrument language setting (Language on page 19)
- Change logging options (Logging on page 19)
- Change sampling options (Sampling on page 20)
- Set the handheld auto-shutoff time (Auto-Shutoff on page 20)
- Change the radix point (Radix Point on page 21)
- Set the backlight mode (Backlight on page 21)
- View the software version (Software version on page 21)
- View the handheld serial number (Serial # on page 21)
- View and adjust the Unit ID (Unit ID on page 22)
- View the sensor specific information (Sensor info on page 22)
- Adjust the display brightness (Brightness on page 22)

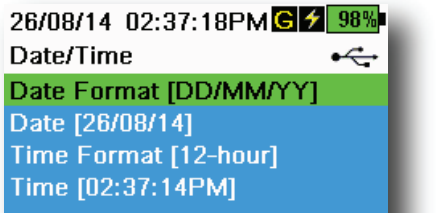


Figure 11 Date/Time

Date/Time

→ **Date/Time**

For accurate logging and GLP data, correctly set the date and time options (Figure 11). Select any of the following options to set the Date/Time in the ProDSS.

Date/Time options:

- Set YY/MM/DD, MM/DD/YY, DD/MM/YY or YY/DD/MM date format
- Set the correct date
- Select 12 or 24 hour time format
- Set the correct time

GLP menu

Detailed sensor calibration information is stored in the Good Laboratory Practice (GLP) file for later review.

One GLP file is used to store all calibration records. The instrument's internal memory can save up to 400 individual calibration records. After 400 records, the instrument will overwrite previously stored calibration records, starting with the oldest.

To prevent the permanent loss of GLP records, periodically download the GLP file to a computer using the KorDSS software.

NOTE: *Information included in each GLP record can be seen on page 30.*

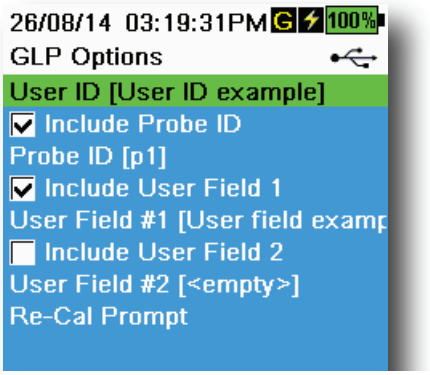


Figure 12 GLP Options

GLP Options

→ **GLP** → **Options**

User ID, Probe ID, or User Field #1 or 2 can be user-defined for positive GLP file identification of:

- The person calibrating the instrument.
- The sensor/cable serial number used during calibration (or other, user-defined Probe ID).
- Other user-specific identification (User Field #1 and #2) (Figure 12).

NOTE: *User Field can be used to describe the condition of the probe. For example, new sensor or new ODO cap.*

Figure 13 Re-Cal Prompts

Re-Cal Prompts

→ **GLP** → **Options** → **Re-Cal Prompts**

Re-Cal Prompts provide a reminder to recalibrate a probe in the user-defined number of days (Figure 13).

The Re-Cal prompt will be displayed in the message area of the main display when the set time has elapsed (Figure 9 on page 15).

Select the desired sensor Re-Cal prompt, then enter the desired number of days before the Re-Cal prompt occurs.

Set the sensor value to zero (0) days (default) to turn off Re-Cal prompts.

NOTE: *When enabled and the set amount of time since the last calibration has passed, the Re-Cal prompt will be shown when the instrument is turned on.*

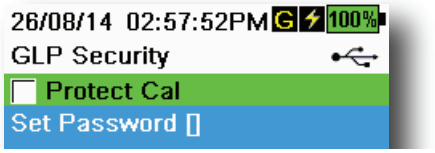


Figure 14 GLP Security

GLP Security

→ **GLP** → **Security**

The Calibration menu can be password protected to prevent accidental or unauthorized sensor calibration (Figure 14).

1. From the GLP menu, select **Security**, then enter the default password "ysi123".
2. Select **Set Password []** and change the default password.
3. Select the **Protect Cal** check box to password protect the Calibration menu.

NOTE: *Write down and keep the password in a safe place. Contact YSI Technical Support if you lose the password* (Technical support on page 82).

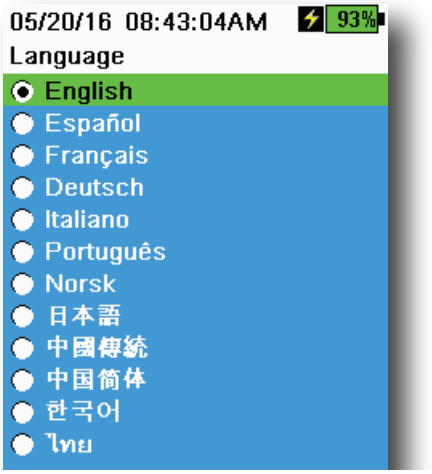


Figure 15 Language

Language

→ **Language**

The ProDSS is shipped with English enabled. If a different language is desired and selected, the ProDSS will take approximately 10 to 20 seconds to enable the new language (during the first installation only).

Optional languages:

- Spanish
- French
- German
- Italian
- Portuguese
- Norwegian
- Japanese
- Simplified Chinese
- Traditional Chinese
- Korean
- Thai

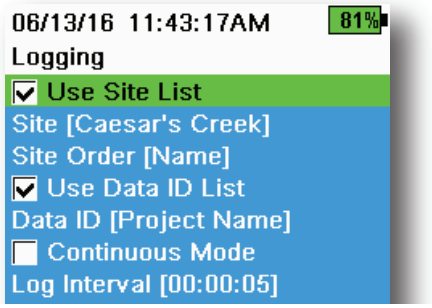


Figure 16 Logging

Logging

→ **Logging**

The ProDSS can add a user-defined Site and/or Data ID to a data record if these functions are enabled under the Logging menu. A check mark in the box next to these features indicates they are enabled (Figure 16).

After selecting **Site [ ]** or **Data ID [ ]**, the Site List or Data ID List will be shown (Figure 17). New entries can be created by choosing **Add new...** When creating a new site, GPS coordinates and altitude can be entered.

NOTE: *If the instrument has GPS signal, the current GPS coordinates will be used when creating a new site.*

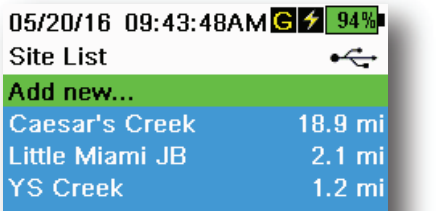


Figure 17 Site List

Choose an entry from the Site List or Data ID List to **Select** (i.e. will be added to data record), **Edit**, or **Delete** the entry (Figure 18).

NOTE: *KorDSS can be used to send a picture of the Site to the instrument.*

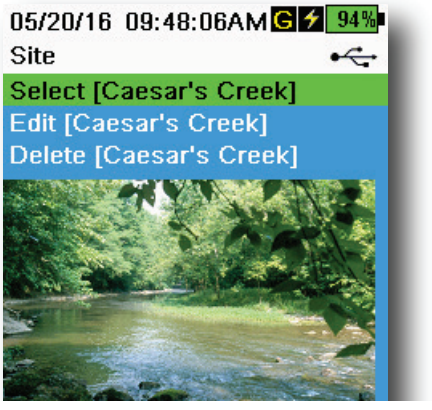


Figure 18 Site

NOTE: *Sites can be listed in order of **Name** (i.e. alphanumeric order) or **Distance** from the current position (Figure 16).*

Continuous Mode (Interval logging): Select the Continuous Mode check box and enter the user-defined Log Interval (in HH:MM:SS - hours:minutes:seconds) to log samples continuously at the specified time interval. The Run screen will display **Start Logging...** when in Continuous Mode. Press ENTER to begin logging.

One sample logging: Clear the Continuous Mode check box. The Run screen will display **Log One Sample**. A sample will be logged each time the ENTER key is pushed when in the Run screen.

NOTE: *An option to change Site and/or Data ID (if enabled) appears once ENTER is pressed to begin logging.*

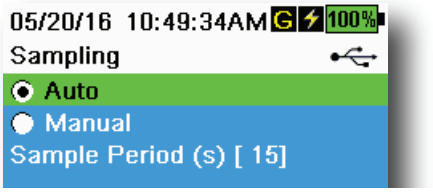


Figure 19 Sampling

Sampling

→ Sampling

Auto sampling mode continuously updates measurements on the display (Figure 19).

Manual mode helps conserve battery power. The user-defined Sample Period determines the measurement time limit.

When in Manual mode, the instrument will take measurements for the duration of the Sample Period then "lock" or hold the readings on the display (sample period default 50 seconds, user-defined between 15 to 60 seconds).

Once the measurements are locked, push the ENTER key to log the held data, or the Esc key and then the ENTER key to take a new measurement.

Enter the desired Sample Period time.

NOTE: *When both Continuous Logging Mode and Manual Sampling mode are enabled, the ProDSS will power the sensors on and take measurements for 15 seconds before logging a data set.*

Auto-Shutoff

→ Auto-Shutoff

To conserve battery power, auto-shutoff powers off the instrument after a user-defined time period (in minutes). Set to 0 (zero) to disable Auto-Shutoff.

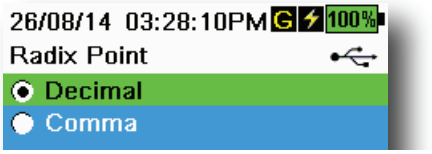


Figure 20 Radix Point

Radix Point

→ **Radix Point**

The radix point can be changed to display a comma or a decimal in numeric displays (e.g. 1.00 becomes 1,00 when Comma is selected) (Figure 20).

Backlight

→ **Backlight**

In Automatic mode, the instrument display will dim 60 seconds after the last key was pushed. Once any key is pushed, the instrument display will return to the user-defined brightness setting and the keypad backlight will turn on. The screen will dim and the keypad backlight will turn off after another 60 seconds of inactivity.

In manual mode, the instrument display remains at the user-defined brightness until manually changed and the keypad backlight is turned on and off by the Backlight key.

NOTE: *In bright conditions, set the backlight to Manual mode.*

Software (Sw) Version

→ **Sw Version**

Sw Version shows the ProDSS software version number. The latest instrument software and update instructions are available at ysi.com. Instrument software can be updated through the KorDSS PC software program when connected to the internet or if the update file has been transferred to the PC. See the KorDSS help section for more information.

Serial

→ **Serial #**

Serial # shows the serial number of the ProDSS handheld instrument. Note the serial number when contacting YSI support.

Unit ID

→ **Unit ID**

Unit ID identifies the instrument in the KorDSS PC software program that was included with the instrument.

Select **Unit ID** to change the default ID.

Sensor info

→ **Sensor info**

Sensor info shows measurement data, and hardware/software information for each component of the system: instrument, sensor, and bulkhead. Use the ▲ and ▼ arrow keys to scroll through the components.

Brightness

→ **Brightness**

The screen brightness can be adjusted to accommodate lighting conditions and to conserve battery power (Figure 21).

Select **Brightness** then use the ◀ and ▶ arrow keys to adjust the screen brightness.

NOTE: *In bright conditions, set the screen brightness to 75% or greater.*

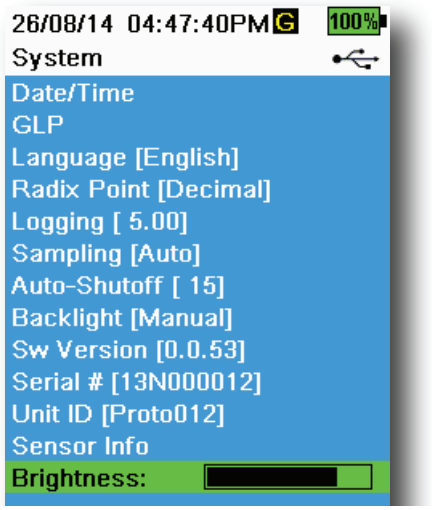


Figure 21 Display Brightness

Sensor menu

Use the Probe key to access the Sensor menu and change sensor settings (if applicable), enable the measurement units displayed on the Run screen, set Auto Stable parameters, change the sensor averaging mode, and if equipped, turn on/off GPS.

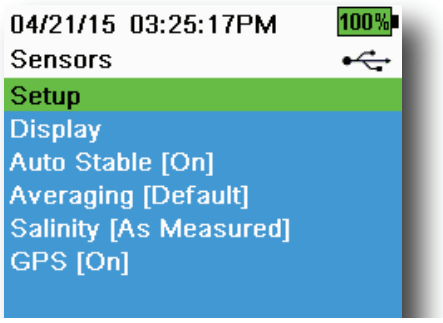


Figure 22 Probe (Sensor) menu

Push the key to access the sensor menu (Figure 22). Highlight a sub-menu then push the ENTER key to view sub-menu options.

Pre-defined or user-selected sensor settings are noted within brackets ([]).

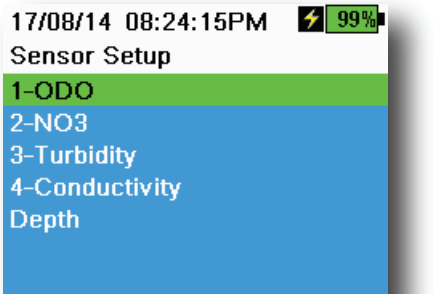


Figure 23 Sensor Setup

Sensor Setup

→ **Setup**

The Sensor Setup menu will show all sensors connected to the instrument (Figure 23). If a sensor is connected but is not listed on the Sensor Setup menu (**<None>** displayed), check the sensor and cable connections (ProDSS sensor installation/removal on page 9).

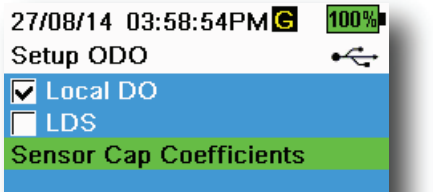


Figure 24 Setup ODO

Setup ODO

→ **Setup** → **ODO**

Local DO: Enable or disable localized DO% measurements. When enabled, the calibration value is set to 100% regardless of altitude or barometric pressure. When enabled, an L will be shown next to DO% on the run screen. DO mg/L measurements are unaffected when Local DO is enabled (Figure 24).

LDS: Last Digit Supression (LDS) rounds the DO value to the nearest tenth, e.g. 8.27 mg/L becomes 8.3 mg/L.

Sensor Cap Coefficients: The sensor cap coefficients must be updated after sensor cap replacement. Update the sensor cap coefficients using the KorDSS software and the coefficient sheet provided with the new sensor cap.

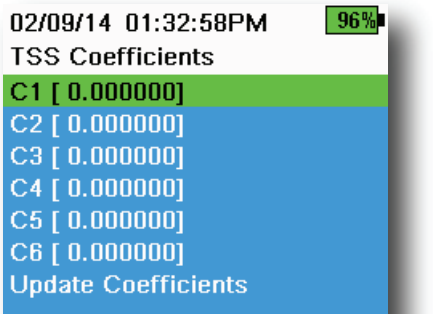


Figure 25 TSS coefficients

Setup Turbidity

→ **Setup** → **Turbidity**

TSS Coefficients: Total Suspended Solids (TSS) can be measured if correlation coefficients are calculated in KorDSS.

To obtain these coefficients, collect turbidity data at the sampling site with corresponding grab samples. Analyze the samples in a lab to determine a true TSS measurement (mg/L). At least 2 and up to 6 value pairs of turbidity and TSS measurements can be used.

NOTE: *For highest accuracy, obtain 6 value pairs.*

NOTE: *Correlation data must be collected for each unique sampling site, as this correlation is site-specific.*

In KorDSS, enter the field-obtained turbidity measurements and the corresponding lab-obtained TSS measurements. Coefficients can then calculated with KorDSS and sent to the sensor.

NOTE: *Although correlation coefficients can be entered directly into the ProDSS (Figure 25), only KorDSS can calculate the coefficients.*

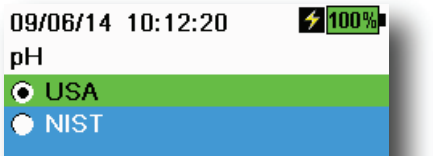


Figure 26 Setup pH

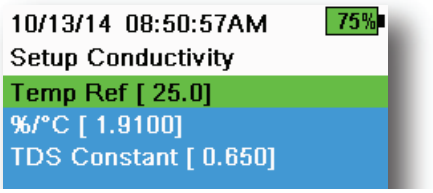


Figure 27 Setup Conductivity

Setup pH

→ **Setup** → **pH**

Select USA auto-buffer recognition (4.00, 7.00, and 10.00) or NIST auto-buffer recognition (4.01, 6.86, and 9.18) (Figure 26). Calibration values are automatically compensated for temperature for both buffer sets.

Setup Conductivity

→ **Setup** → **Conductivity**

Temp Ref (Temperature reference): Reference temperature used to calculate temperature compensated specific conductance. All specific conductance values are compensated to the Temp Ref temperature. The default value is 25 °C (77 °C) (Figure 27). Enter a new value between 15.00 °C (59 °F) and 25.00 °C (77 °F).

%/°C (Percent per degree Celsius): Temperature coefficient used to calculate temperature compensated specific conductance. The default is 1.91% based on KCl standards. Enter a new value between 0 and 4%.

TDS Constant: Multiplier used to calculate an estimated Total Dissolved Solids (TDS) value from conductivity. The multiplier is used to convert specific conductance in mS/cm to TDS in g/L. The default value is 0.65. Enter a new value between 0 and 0.99.

This multiplier is highly dependent on the nature of the ionic species present in the water sample. To be assured of moderate accuracy for the conversion, you must determine a multiplier for the water at your sampling site. Use the following procedure to determine the multiplier for a specific sample:

1. Determine the specific conductance of a water sample from the site.
2. Filter a portion of water from the site.
3. Carefully measure a volume of the filtered water. Completely evaporate to yield a dry solid.
4. Accurately weight the remaining solid.
5. Divide the weight of the solid (in grams) by the volume of water used (in liters) to yield the TDS value in g/L for the site.
6. Divide the TDS value in g/L by the specific conductance of the water in mS/cm to yield the conversion multiplier.

NOTE: *Make sure to use the correct units.*

NOTE: *If the nature of the ionic species at the site changes between sampling studies, the TDS values will be in error. TDS cannot be calculated accurately from specific conductance unless the make-up of the chemical species in the water remains constant.*

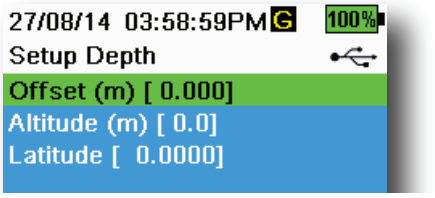


Figure 28 Setup Depth

Setup Depth

→ **Setup** → **Depth**

For ProDSS bulkheads with the depth sensor:

The ProDSS cable assemblies with a depth sensor in the bulkhead can measure virtual vented depth. The virtual vented depth measurement allows for real time compensation for atmospheric pressure using the instrument's barometer.

Depth offset: Depth offset can be used if referencing water elevation against a known datum. If a depth offset is entered (in meters), the output value will shift by the value of the offset (Figure 28).

Altitude/Latitude: To compensate for atmospheric pressure based on elevation and gravitational pull, enter the local altitude in meters relative to sea level and latitude in degrees where the ProDSS is sampling.

Latitude effect: Varying latitudes cause a 200 mm change in depth from equator to pole.

Altitude effect: Varying altitudes cause approximately 90 mm change from sea level to 8000 m. A 100 m change causes 1.08 mm of change to the readings.

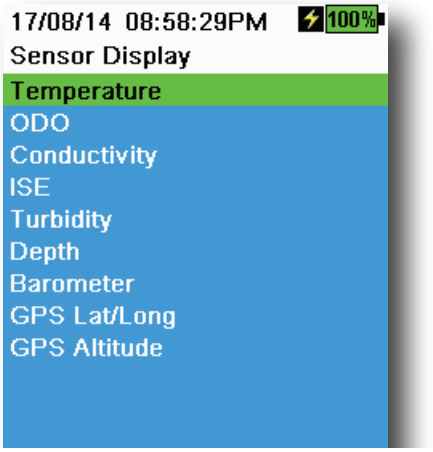


Figure 29 Sensor Display

Sensor Display

→ **Display** (Figure 29)

The Sensor display menu determines the measurements that are shown on the Run screen (Figure 9). The Run screen will only show measurements for sensors that are attached to the cable bulkhead.

If more measurements are selected than can be displayed on one screen, a scroll bar will be shown. Use the ▲ and ▼ keys to scroll through the measurements.

NOTE: *For depth profiling, enable Vertical Position under Depth Display to view the real-time position of the depth sensor in the water column. This is helpful in profiling applications to ensure the depth sensor is lowered to the desired depth without waiting for the depth data to stabilize.*

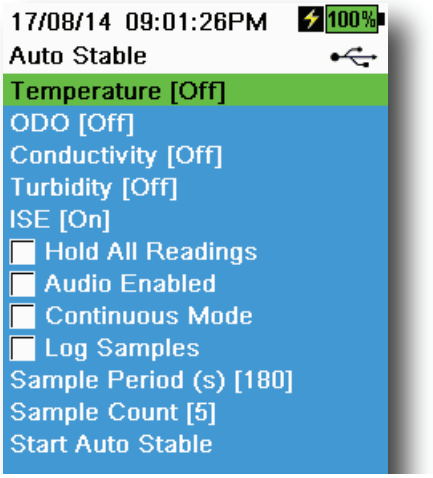


Figure 30 Auto Stable

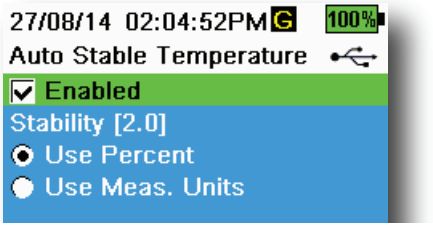


Figure 31 Auto Stable stability threshold

Auto Stable

→ **Auto Stable**

Auto Stable indicates when a measurement is stable. Sensors with Auto Stable enabled will have A S flash beside the measurement on the Run screen.
A S will flash green when the measurement is stable.

Select a sensor to enable or disable Auto Stable. Set the stability threshold parameters (Figure 30).

The Auto Stable stability threshold can be set by percent of measurement or in the units of measurement selected in the Sensor Display menu.

Enter the stability value, then select **Use Percent** or **Use Meas. Units** (Figure 31).

This threshold is used to compare the last reading with the previous. The smaller the number entered in % or units, the longer it will take for the instrument to reach the auto stable criteria.

Example: For temperature in °C, if unit threshold is set to 0.2 and the temperature reading changes by more than 0.2 degrees, A S will continue to be red until the reading does not change by more than 0.2 °C over the defined sample period and sample count.

Hold All Readings: After all sensors have reached their stability criteria, the measurements will be held or 'locked' on the display. If disabled, the sensor measurements will continue to change in real time.

Audio Enabled: An audio alert will sound when stability is reached.

Continuous Mode: The ProDSS will continuously check sensor values against the stability criteria even after the sample period and sample count have been met.

Log Samples: Logs the sample/s defined by the Sample Period to memory.

Sample Period: Time interval between the sensor measurements (sample) that are used to determine stability. Set the interval in seconds (1 to 900).

Sample Count: Number of consecutive samples required for stability (1 to 10).

Select **Start Auto Stable** to enable.

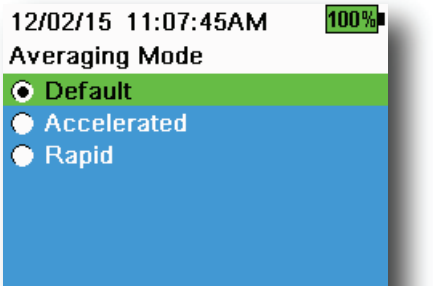


Figure 32 Averaging

Averaging

→ **Averaging** (Figure 32)

The averaging mode determines how the ProDSS will filter data. A smaller time frame for the rolling average window allows changes in the sensor's measurements to be more quickly observed, while a larger rolling window provides more stable measurement readings and a smooth result. Each averaging mode will decrease the time span of the rolling window if a large change in the sensor measurement is detected, allowing the ProDSS to adapt when an event occurs.

The **Default** mode provides optimum averaging for all sensors. This mode has up to 40 seconds of averaging on the sensors.

In **Accelerated** mode, changes in sensor measurements are more quickly observed than default (5-10 seconds of averaging). This mode is recommended when the sensors are moving through the water, such as profiling studies and most spot sampling applications.

NOTE: *For profiling applications, enable Vertical Position under Depth Display to view unfiltered depth measurements. This helps to ensure the depth sensor is lowered to the desired depth without waiting for the averaged measurement.*

In **Rapid** mode, sensor response is very fast (2-3 seconds of averaging), but the instrument will never settle on a single steady number. This mode is recommended when the sensors are moving quickly through the water, such as rapid profiling and towed applications.

Salinity

→ **Salinity**

Salinity is determined by calculations derived from the conductivity and temperature sensors.

When a conductivity sensor is installed, the instrument will automatically use the salinity measurement for DO and "As Measured" will be displayed. If no conductivity sensor is installed (e.g. ProODO cable assembly used), the salinity value will be user-selectable.

Because salinity is an important factor in determining dissolved oxygen, YSI does not recommend calibrating or taking dissolved oxygen measurements without conductivity and temperature sensors.

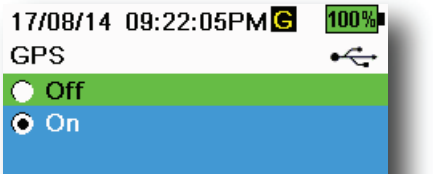


Figure 33 GPS

GPS (optional)

→ **GPS**

GPS turns the ProDSS Global Positioning System On or Off. The G symbol is shown when a GPS signal is received (Figure 33).

When enabled, the GPS coordinates will be saved with the GLP file and logged data.

NOTE: *GPS data will be most accurate when there is a clear line of sight to satellites. GPS will not typically receive a signal while inside a building.*

Calibration menu

Push the Cal key to access the Calibration menu (Figure 34). Highlight a sub-menu then push the ENTER key to view sub-menu options.

Pre-defined or user-selected parameters are noted within brackets (**[]**). See Alpha/numeric entry on page 14.

Refer to the Calibration section for sensor specific calibration procedures (Calibration on page 33).

NOTE: *Attached sensors are listed according to the bulkhead port in which they are installed.*

NOTE: *User ID, Probe ID, and User Field #1 and #2 must be enabled in the GLP menu to appear in the Calibration menu* (GLP Options on page 17).

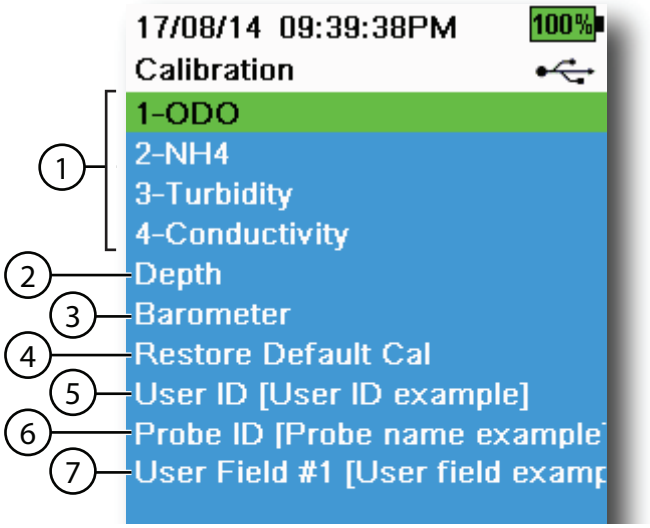


Figure 34 Calibration menu

1	Sensors connected to bulkhead	**5**	User ID
2	Optional Depth sensor calibration	**6**	Probe ID
3	Barometer calibration	**7**	User Field #1
4	Restore Default Calibration - restores all calibrations to factory default		

Files menu

Push the key to access the Files menu (Figure 35). Highlight a sub-menu then push the ENTER key to view sub-menu options.

Use the Files menu to view, delete or backup logged data or the GLP file. Data can be filtered by a specific date and time range and by user-created site and Data ID lists.

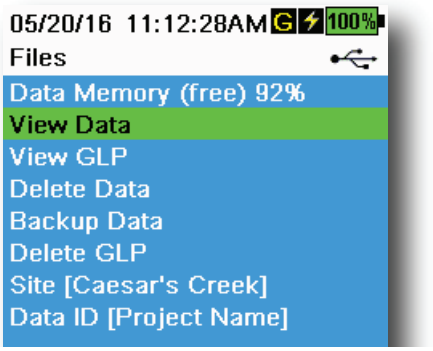


Figure 35 Files menu

Data Memory (free) % shows the remaining memory available. Download or delete data to free available internal memory.

The Site List and/or Data ID List can be seen by selecting **Site [ ]** or **Data ID [ ]**. To enable the use of Site and/or Data ID when logging data, view the Logging menu on page 19.

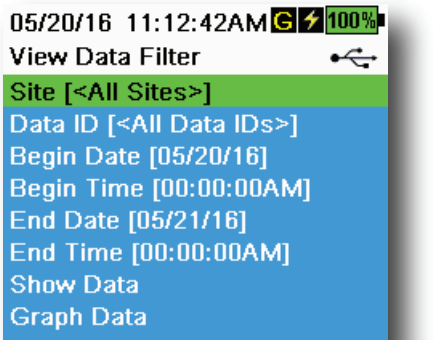


Figure 36 View Data Filter

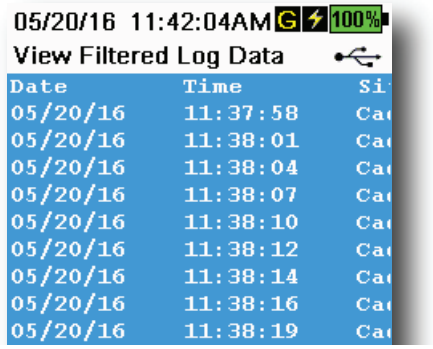


Figure 37 View Filtered Log Data

View Data Filter

→ **View Data**

Enter the desired filter criteria, then select **Show Data** or **Graph Data** to view the tabular or graphical data. If necessary, use the ▲ and ▼ arrow keys to scroll through the data (Figure 36 and Figure 37).

Site: View data from one site or all sites.

Data ID: View data from one ID or all IDs.

Begin/End: View data within specific date and time ranges.

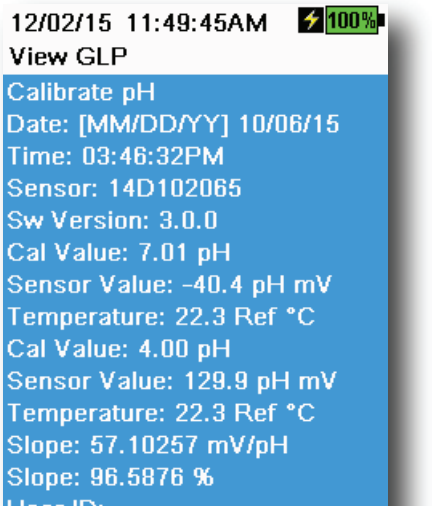


Figure 38 View GLP

View GLP

→ **View GLP**

Select **View GLP** to show the stored sensor calibrations (Figure 38).

Use the arrow keys to scroll through the GLP file data.

GLP saved information

Information in each GLP record

- Sensor calibrated
- Date/time stamp
- Sensor ID
- Sensor software version
- User ID (optional)
- Probe ID (optional)
- User fields #1 and #2 (optional)
- Calibration status
- Calibration value
- Temperature

Parameter-specific GLP information

- Calibration method - ODO, Depth, Conductivity
- Sensor value - pH, ODO, Ammonium, Nitrate, Chloride, Depth, Turbidity
- Pre cal value - Depth, Barometer, Turbidity, Conductivity, ORP
- Cell constant - Conductivity
- Cal offset - ORP
- Slope - pH
- Gain - ODO

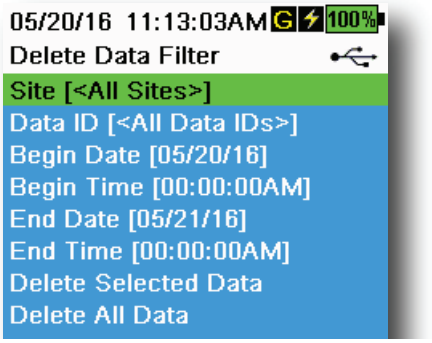


Figure 39 Delete Data Filter

Delete Data

→ **Delete Data**

Enter the desired filter criteria, then select **Delete Selected Data** to *permanently* delete the data (Figure 39).

Select **Delete All Data** to *permanently* delete *all* logged data from the ProDSS.

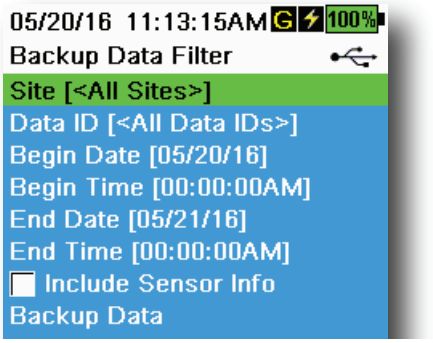


Figure 40 Backup Data

Figure 41 Micro USB female connector

Backup Data

→ **Backup Data**

This function allows you to backup logged data to a flash drive based on Site, Data ID, and log date (Figure 40). A USB female to micro USB male adapter is included with new instruments for this data backup.

NOTE: *The USB storage device must be formatted as FAT32, not NTFS or exFAT. The handheld will only support FAT32.*

If the box next to "**Include Sensor Info**" is checked, each data set will be sent to a flash drive as a separate file with sensor serial number and sensor software information included. If the box is not checked (default), all data sets will be sent in a single backup file with no sensor serial number or sensor software information.

NOTE: *It is suggested to send data to the USB flash drive as a single file (i.e. box is not checked) unless this sensor information is needed. This makes importing the data much faster and easier.*

Once the filter settings are configured, select **Backup Data** to send the data to a flash drive.

NOTE: *The data is exported in a CSV file.*

NOTE: *If the data backup is not successful, ensure the correct filter criteria are selected and the USB connection indicator can be seen at the top of the screen (Figure 9).*

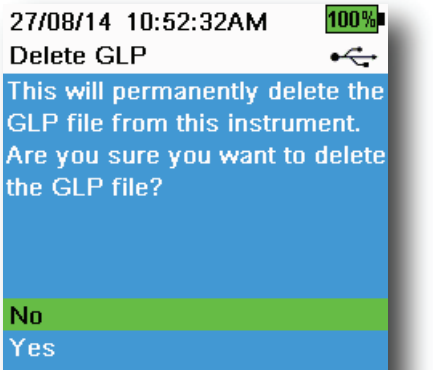


Figure 42 Delete GLP

Delete GLP

→ **Delete GLP**

To permanently delete the GLP file from the instrument, select **Yes**, then push the ENTER key (Figure 42).

Taking measurements

For the highest accuracy, calibrate the instrument before taking measurements (Calibration on page 33).

1. Create site and Data ID lists for logged data (if applicable) (Logging on page 19).
2. Set the logging method (single or interval) (Logging on page 19).
3. Set the Auto Stable parameters (if applicable) (Auto Stable on page 26).
4. Verify that the sensors and/or port plugs are correctly installed in all bulkhead ports (page 9).
5. Install the sensor guard (page 11).
6. Insert the sensors into the sample.

   NOTE: *Make sure to submerge the sensors completely. If using a depth sensor, submerge to where the cable assembly attaches to the bulkhead.*

7. Move the bulkhead in the sample to release any air bubbles and to provide a fresh sample to the sensors.
8. Wait for the sensor/s to stabilize in the sample.
9. On the main run screen, press 

to begin logging (single or interval) (Logging on page 19).

   NOTE: *An option to change Site and/or Data ID (if enabled) appears once* 

is pressed to begin logging.

Calibration

ProDSS sensors (except temperature) require periodic calibration to maintain accurate measurements. Calibration procedures follow the same basic steps with variations for specific parameters.

Before calibration

- Enter GLP user-defined data if applicable to user requirements (User ID, Probe ID, User Field #1/2) (GLP Options on page 17).
- Set up sensor options, settings, and coefficients as applicable (Sensor menu beginning on page 22).

NOTICE: Install a gray port plug in all exposed ports. Exposure to water can cause damage or corrosion to the bulkhead connectors not covered by the warranty.

Calibration setup (pH, ORP, ISE, conductivity, turbidity)

NOTE: *Make sure the calibration cup, sensor guard, and all sensors are clean.*

NOTE: *If using the calibration cup, make sure to install the sensor guard before placing the sensors into the calibration cup.*

NOTE: *The sensor guard and calibration cup should be used for the turbidity and DO calibration. All other calibrations can be performed in other laboratory glassware.*

1. Install a clean, dry sensor (Figure 4 on page 9) and sensor guard (Figure 6 on page 11) onto the bulkhead.

   NOTICE: Install a gray port plug in any exposed port on 4 port ProDSS cable assemblies. All sensor ports must have either a sensor or port plug installed.

2. Fill the calibration cup with a moderate amount of water and tighten the calibration cup onto the bulkhead. Use the water to rinse the cup and the sensor to be calibrated. Discard the rinse.
3. Thoroughly rinse the calibration cup with a small amount of the calibration standard for the sensor to be calibrated. Discard the standard.
4. For ***4 port cable assemblies***, refill the calibration cup with fresh calibration standard to approximately the first line for pH, ORP, and turbidity calibration. Fill to the second line for conductivity calibration (Figure 43 on page 34). If using the ***ODO/CT cable assembly*** and calibrating conductivity, ensure the vent holes at the top of the sensor are completely immersed and the solution level is at least 1/2 inch higher than these top vent holes (Figure 46 on page 36). A cylinder is included with ODO/CT cable assemblies for the purpose of calibrating conductivity.

   NOTE: *Volumes will vary. Make sure the temperature sensor and the sensor to be calibrated are submerged in calibration solution, except when performing a DO% saturation calibration.*

   NOTE: *Be careful to avoid cross-contamination with other standards.*

   NOTE: *These rinsing recommendations are only suggested guidelines for highest data accuracy. Make sure to follow your organization Standard Operating Procedures (SOPs) for instrument calibration and operation.*

5. Immerse the sensor(s) in the standard and tighten the calibration cup onto the bulkhead.
6. Calibrate the sensor(s).

Alternately, pH, ORP, and conductivity calibrations can be completed in a beaker or other container using the same basic procedure described above. Make sure that the temperature sensor and the sensor to be calibrated are completely submerged. When submerging the conductivity sensor, make sure that the calibration solution covers the vent holes on the conductivity sensor.

Calibration cup installation

1. Ensure the calibration cup gasket is correctly seated (Figure 43). Loosely install the retaining nut on the cup.
2. Slide the calibration cup over the sensors and sensor guard and tighten the retaining nut.

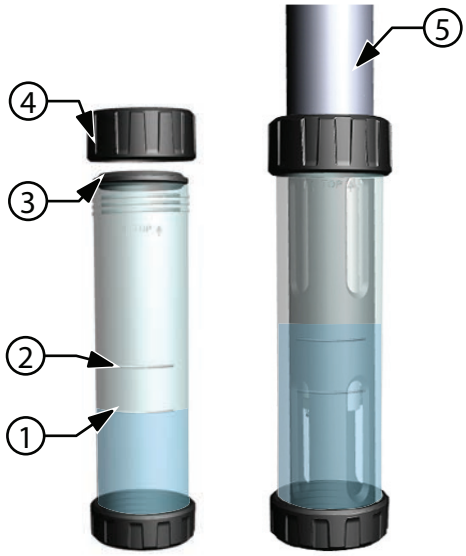
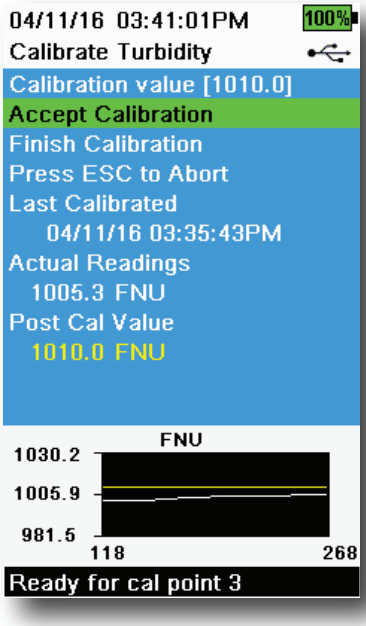


Figure 43 Calibration cup standard volume **(4 port cable)**

1	Fill line one (used for Turbidity, pH, and ORP calibration solution)	**4**	Retaining nut
2	Fill line two (used for conductivity calibration solution)	**5**	Calibration cup installed
3	Gasket		

NOTE: *When the 4 port calibration cup is empty (i.e. no sensor guard or sensors), it takes ~170 mL of solution to fill the calibration cup to line 1 while it takes ~225 mL to fill the cup to line 2.*

Calibration screen layout



The calibration screen has the same basic layout for each parameter (Figure 44).

Calibration value: The value the sensor will be calibrated to. *The Yellow Line on the graph corresponds to this value.*

Accept Calibration: Calibrates the sensor to the calibration value.

Finish Calibration: Only available with multi-point calibrations (i.e. pH, ISE, turbidity). Finishes the calibration by applying previously accepted points.

Press ESC to Abort: Press the ESC key to leave the calibration. The sensor will not be calibrated to any points. The last successful calibration will be used.

Last Calibrated: Date and time of the last successful sensor calibration.

Actual Readings: The current measurement value on the Run screen. *The White Line on the graph corresponds to this value. Observe the White Line to ensure the measurement is stable before choosing* ***Accept Calibration****.*

Post Cal Value: The same as the calibration value. This will be the measurement value in the current solution after the calibration is finished.

Figure 44 Layout of calibration screen

Conductivity

A conductivity/temperature sensor must be installed on the bulkhead (Figure 4 on page 9) for accurate temperature compensation and measurements of all parameters except turbidity and TSS. Temperature calibration is not available or required for accurate temperature measurements.

The conductivity/temperature sensor can measure and calculate conductivity, specific conductance (temperature compensated conductivity), salinity, non-linear function (nLF) conductivity, TDS, resistivity, and density. Calibration is only available for specific conductance, conductivity, and salinity. Calibrating one of these options automatically calibrates the other conductivity/temperature parameters listed above. For both ease of use and accuracy, YSI recommends calibrating specific conductance.

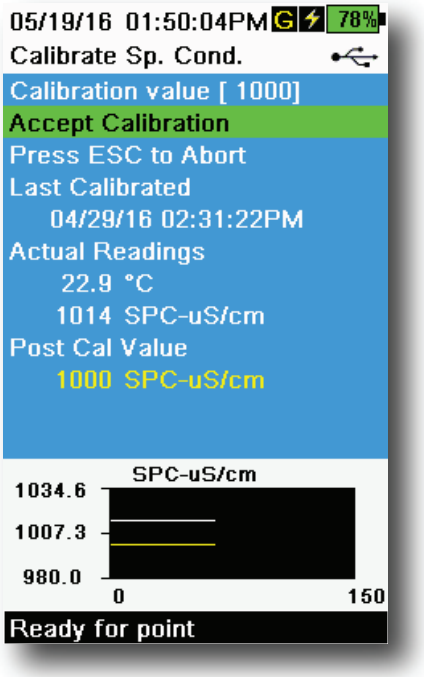


Figure 45 Calibrate specific conductance

Conductivity calibration

1. If necessary, clean the conductivity cell with the supplied soft brush. See Conductivity/temperature sensor maintenance on page 56.
2. Perform the Calibration setup (pH, ORP, ISE, conductivity, turbidity) on page 33.
3. Place the correct amount of conductivity standard into a clean and dry or pre-rinsed calibration cup.

   NOTE: *Select the appropriate calibration standard for the conductivity of the sampling environment. Standards greater than 1 mS/cm (1000 µs/cm) are recommended for the greatest stability. For fresh water applications, calibrate to 1,000 or 10,000 µS. For salt water applications, calibrate to 50,000 µS.*

4. Carefully immerse the sensors into the solution. Make sure the solution is above the vent holes on the side of the conductivity sensor. If using the ODO/CT assembly, ensure the vent holes at the top of the sensor are completely immersed and the solution level is at least 1/2 inch higher than these top vent holes (Figure 46).
5. Gently rotate and/or move the sensor up and down to remove any bubbles from the conductivity cell. Allow at least one minute for temperature equilibration before proceeding.
6. Push the Cal key, select **Conductivity**, then select **Specific Conductance**.

   NOTE: *Calibrating any conductivity calibration option will automatically calibrate the other options. Specific conductance is recommended for both ease of use and accuracy.*

7. Select **Calibration value** then enter the calibration value of the standard used. Note the measurement units the instrument is reporting and calibrating and be sure to enter in the correct calibration value for the units being used. For example, 10,000 µS = 10 mS. Make sure that the units are correct and match the units displayed on the handheld.
8. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration** (Figure 45). "Calibration successful!" will be displayed in the message area.

NOTE: *If the data is not stabilized after 40 seconds, gently rotate the sensor or remove/reinstall the calibration cup to make sure that no air bubbles are in the conductivity cell.*

NOTE: *If the actual measurement data is about 1/2 if the expected calibration value, the conductivity sensor is not completely submerged. Add more calibration standard to the calibration cup.*

NOTE: *If you get calibration error messages, check for proper sensor immersion, verify the calibration solutions is fresh, the correct value has been entered into the ProDSS, and/or try cleaning the sensor.*

9. Rinse the bulkhead and sensors in clean water then dry.

Figure 46 ProDSS ODO/CT Cable Assembly

Barometer

The barometer is factory calibrated and should rarely need to be recalibrated. The barometer is used for DO calibration, %Local measurements, and for virtual depth measurements. Verify that the barometer is accurately reading "true" barometric pressure and recalibrate as necessary.

Laboratory barometer readings are usually "true" (uncorrected) values of air pressure and can be used "as is" for barometer calibration. Weather service readings are usually not "true", i.e. they are corrected to sea level and cannot be used until they are "uncorrected". Use this approximate formula:

True BP in mmHg=[Corrected BP in mmHg] - [2.5* (Local altitude in ft. above sea level/100)]

Example:

Corrected BP = 759 mmHg

Local altitude above sea level = 978 ft

True BP = 759 mmHg - [2.5*(978ft/100)] = 734.55 mmHg

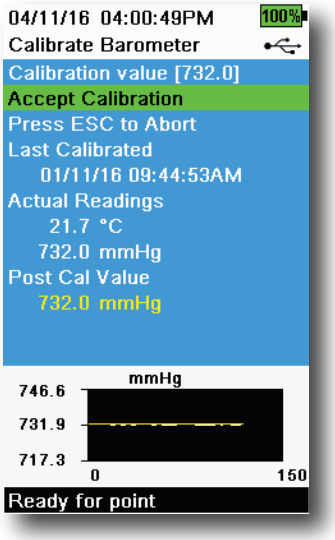


Figure 47 Calibrate Barometer

Barometer calibration

1. Push the Cal key, then select **Barometer**.

2. Select **Calibration value** then enter the correct "true" barometric pressure.

NOTE: *The measurement units during calibration are dictated by what is enabled in the sensor setup menu. Be sure to enter in the correct units.*

- BP in mmHg=25.4 x BP inHg
- BP in mmHg=0.750062 x BP mb
- BP in mmHg=51.7149 x BP psi
- BP in mmHg=7.50062 x BP kPa
- BP in mmHg=760 x BP atm

3. Select **Accept Calibration** (Figure 47). "Calibration successful!" will be displayed in the message area.

Dissolved oxygen

ODO calibration requires the current "true" barometric pressure. Make sure that the barometer is reading accurately and recalibrate the barometer as necessary.

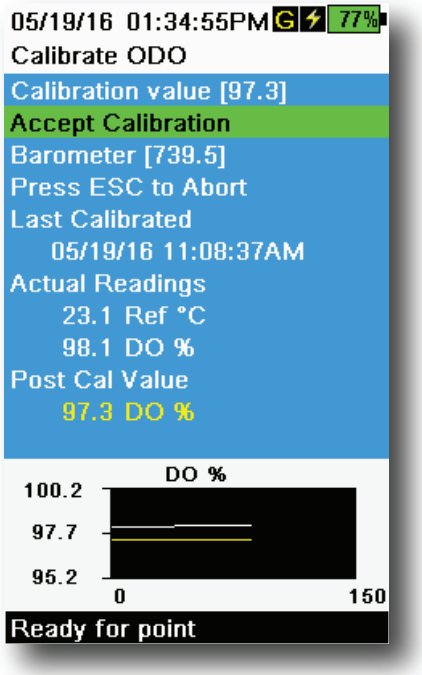


Figure 48 Calibrate ODO %

ODO% and ODO% local - water saturated air calibration

NOTE: *This method calibrates the instrument's DO% measurement or DO% Local measurement if DO% local is enabled in the sensor setup menu.*

NOTE: *Calibrating in DO% or DO% local automatically calibrates the mg/L and ppm measurement. There is no reason to calibrate both parameters. For both ease of use and accuracy, we recommend that you calibrate DO% or DO% Local and not mg/L.*

1. Place a small amount of clean water (1/8 inch) into the calibration cup.
2. Make sure there are no water droplets on the ODO sensor cap or temperature sensor.
3. Attach the sensor guard to the bulkhead and carefully place the guard/sensor into the calibration cup. Partially tighten the calibration cup to the bulkhead.

   NOTE: *Do not fully tighten the calibration cup to the bulkhead. Atmospheric venting is required for accurate calibration.*

   NOTE: *Make sure the ODO and temperature sensors are not immersed in water.*

4. Turn the instrument on and wait approximately 5 to 15 minutes for the air in the storage container to be completely saturated with water.
5. Push the (Cal) key, then select **ODO**. Select **DO%**. This will calibrate the instrument's DO% measurement or DO% Local measurement if DO% Local is enabled in the sensor setup menu.
6. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration** (Figure 48). "Calibration successful!" will be displayed in the message area.

   NOTE: *If you see a calibration error message, verify the barometer reading and inspect the sensor cap. Clean and/or replace the sensor cap as needed.*

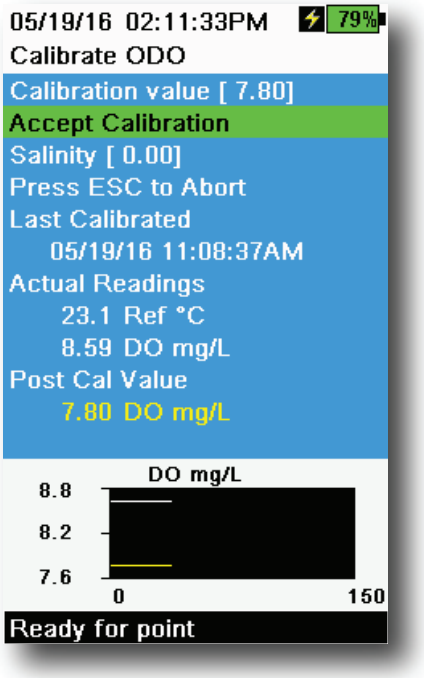


Figure 49 Calibrate ODO mg/L

ODO mg/L calibration

1. Place the ODO and conductivity/temperature sensor into a water sample that has been titrated by the Winkler method to determine the dissolved oxygen concentration in mg/L.
2. Push the Cal key, then select **ODO**. Select **DO mg/L**.
3. Select **Calibration value**.
4. Enter the dissolved oxygen concentration of the sample in mg/L.
5. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration** (Figure 49). "Calibration successful!" will be displayed in the message area.
6. Rinse the bulkhead and sensors in clean water then dry.

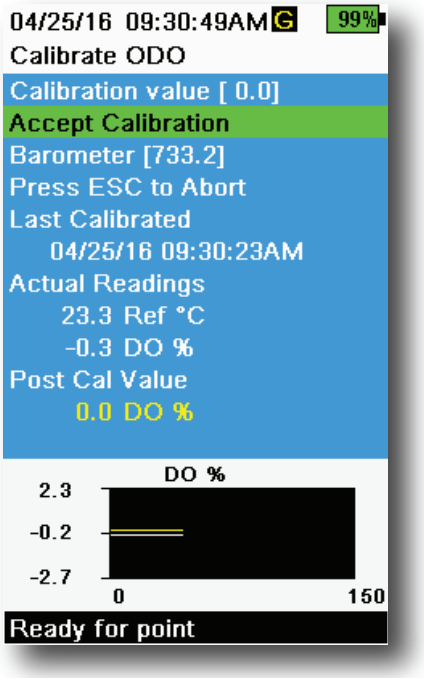


Figure 50 Calibrate ODO zero point

ODO zero point calibration

1. Place the ODO and Conductivity/Temperature sensors in a solution of zero DO.

   NOTE: *A zero DO solution can be made by dissolving approximately 8-10 grams of sodium sulfite into 500 mL of tap water. Mix the solution thoroughly. It may take the solution 60 minutes to be oxygen-free.*

2. Push the Cal key, then select **ODO**. Select **Zero**.
3. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration** (Figure 50). "Calibration successful!" will be displayed in the message area.
4. Thoroughly rinse the bulkhead and sensors in clean water then dry.
5. Perform a ODO % water-saturated air calibration after performing a zero point calibration.

pH/ORP

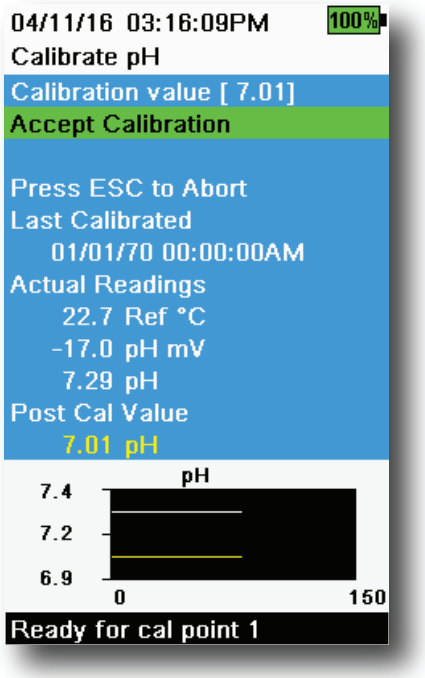


Figure 51 Calibrate pH 1-point

pH calibration 1-point

NOTE: *If performing a 1-point calibration, use buffer 7 (6.86) as your calibration point for highest accuracy.*

NOTE: *Observe the pH mV readings during calibration to understand the condition and response of the pH sensor. In buffer 7, pH mVs should be between -50 and +50. In buffer 4, the mVs should be a +165 to 185 away from the pH 7 mV value. In buffer 10, the mVs should be a -165 to -185 away from the pH 7 mV value. Ideal slope is -59 mV per pH unit.*

1. Perform the Calibration setup (pH, ORP, ISE, conductivity, turbidity) on page 33.
2. Fill the calibration cup to the appropriate level with pH 7 buffer solution (or 6.86 if using NIST buffers).
3. Carefully immerse the probe end of the sensors into the buffer solution.
4. Push the Cal key, then select **pH** or **pH/ORP**.

   NOTE: *If using a pH/ORP sensor, select* **pH/ORP**, *then* **pH**.
5. Allow at least one minute for temperature stabilization. The **Calibration value** will automatically be adjusted based on the selected buffer set and temperature. Alternatively, the Calibration value can be manually entered.
6. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration** (Figure 51). "Ready for cal point 2" will be displayed in the message area.
7. After calibrating to the first point, select **Finish Calibration** for a 1-point calibration or continue on to the 2-3 point calibration procedure (Calibration cup installation on page 34).

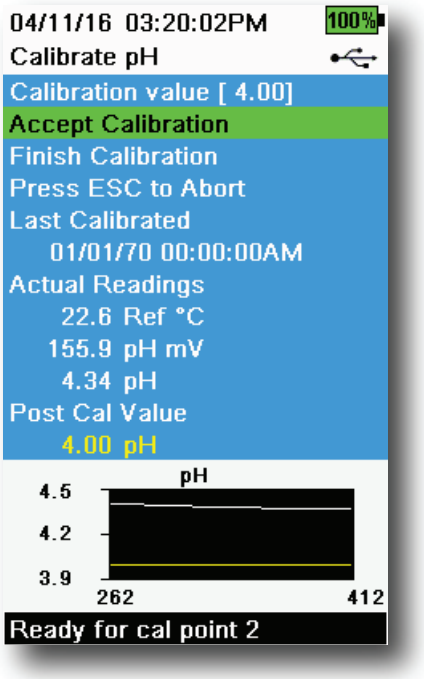


Figure 52 Calibrate pH 2- or 3-point

pH calibration 2- or 3-point

NOTE: *If performing a 2- or 3-point calibration, one point should be in buffer 7; however, the calibration points can be in any order.*

1. Perform steps 1-7 of the pH calibration 1-point procedure (pH calibration 1-point on page 39).
2. Rinse the sensor 2-3 times with a small amount of pH 4 or pH 10 buffer solution.
3. Rinse, then fill the calibration cup to the appropriate level with the buffer solution that is the same value (pH 4 or pH 10) used to rinse the sensor.
4. Carefully immerse the sensors into the solution.
5. Allow at least one minute for temperature stabilization. The **Calibration value** will automatically be adjusted based on the selected buffer set and temperature. Alternatively, the Calibration value can be manually entered.
6. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration** (Figure 52). "Ready for cal point 3" will be displayed in the message area.
7. After calibrating to the second point, select **Finish Calibration** for a 2-point calibration or continue with an additional buffer to complete a 3-point calibration. The procedure will automatically finish after calibrating using a third buffer.

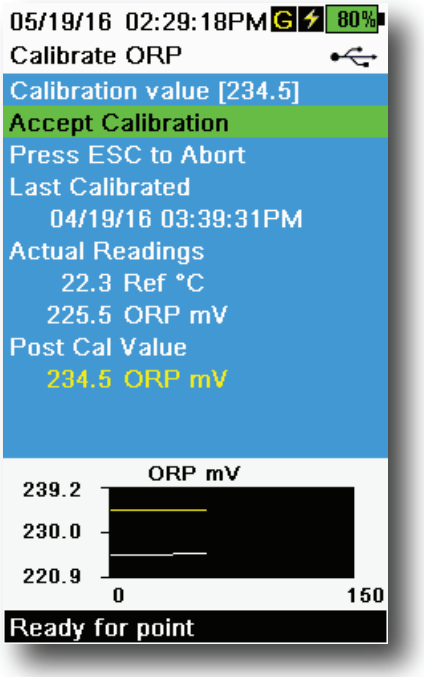


Figure 53 Calibrate ORP

ORP calibration

1. Obtain/prepare a standard with a known oxidation reduction potential (ORP) value.

   NOTE: *YSI recommends Zobell solution.*

2. Perform the Calibration setup (pH, ORP, ISE, conductivity, turbidity) on page 33.
3. Fill the calibration cup to the appropriate level with standard solution.
4. Carefully immerse the sensors into the solution.
5. Push the Cal key, then select **pH/ORP**, then **ORP**.
6. Allow the temperature of the standard to stabilize. If using YSI Zobell solution, the **Calibration value** will automatically be adjusted based on the temperature. Alternatively, the Calibration value can be manually entered.
7. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration** (Figure 53). "Calibration successful!" will be displayed in the message area.

Depth

NOTE: *This calibration option is available only if your bulkhead is equipped with a depth sensor. The depth sensor is located where the cable connects to the bulkhead* (Figure 60 on page 54).

For the calibration, make sure that the depth sensor is clean and in air, not immersed in any solution. For highest accuracy, keep the bulkhead still and in one position while calibrating.

NOTE: *Cables 10 m and longer are supplied with a weight that can be attached to the sensor guard for sampling at water depths 10 m and greater.*

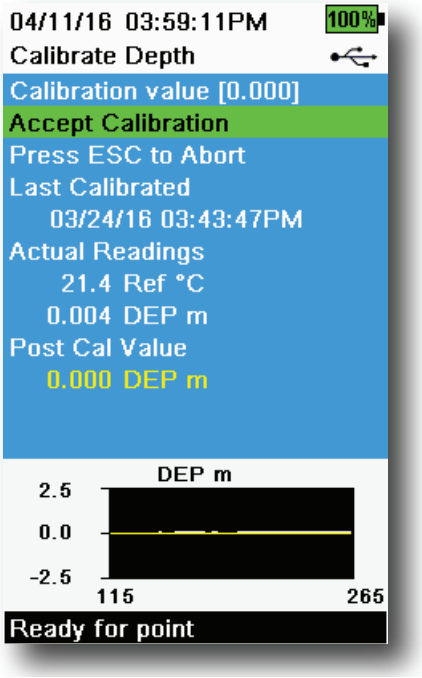


Figure 54 Calibrate Depth

Depth calibration

1. If applicable, enter the depth offset, altitude, and latitude (Figure 28 Setup Depth on page 25).

   NOTE: *Depth offset allows you to set the depth measurement to something other than zero. If the depth offset is used, the depth measurement will be adjusted by the offset after calibration. Enter the altitude and latitude of your sampling location to increase the accuracy of your depth measurement.*

2. Push the (Cal) key, then select **Depth**.
3. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration** (Figure 54). "Calibration successful!" will be displayed in the message area.

Turbidity

Before performing the calibration, review "Calibration setup (pH, ORP, ISE, conductivity, turbidity)" on page 33.

For proper calibration, you must use standards that have been prepared according to details in Standard Methods for the Treatment of Water and Wastewater (Section 2130 B).

Acceptable standards include:

- AMCO-AEPA standards prepared specifically for the ProDSS turbidity sensor manufactured by YSI (YSI turbidity standards)
- Formazin prepared according to Standard Methods, especially for calibration points greater than 1010
- Dilutions of 4000 FNU (NTU) formazin concentrate purchased from Hach
- Hach StablCal™ standards in various FNU (NTU) denominations

The use of standards other than those mentioned above will result in calibration errors and inaccurate field readings. It is important to use the same type of standard for all calibration points. (i.e. do not mix formazine and AMCO-AEPA standard for different points in a multi-point calibration).

Calibration limits

Because of the non-linear response of the turbidity sensor, calibration ranges may be limited. A 1-, 2- or 3-point calibration can be completed using the following limits:

1st calibration point	2nd calibration point	3rd calibration point
0-1 FNU (NTU)	5-200 FNU (NTU)	400-4200 FNU (NTU)

Calibration standards

The following standards are available for the ProDSS turbidity sensor:

608000	0 (all turbidity sensors); 1 gallon
607200	12.4 FNU (NTU) (ProDSS); 1 gallon
607300	124 FNU (NTU) (ProDSS); 1 gallon
607400	1010 FNU (NTU) (ProDSS); 1 gallon

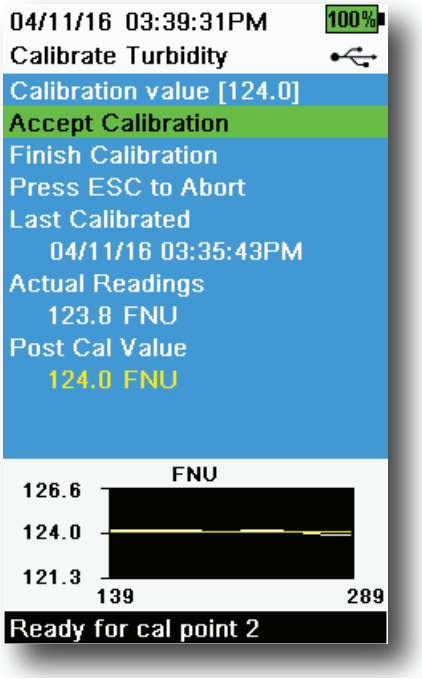


Figure 55 Calibrate Turbidity

Turbidity calibration 1-, 2- or 3-point

NOTE: *The sensor guard must be installed for the turbidity sensor calibration.*

NOTE: *When performing a turbidity calibration, the first point must be zero.*Select **Calibration Value** and enter 0.00.

1. Perform the Calibration setup (pH, ORP, ISE, conductivity, turbidity) on page 33. Rinse the sensor 2-3 times with a small amount of 0 FNU (NTU) standard.
2. Fill the calibration cup to the appropriate level with 0 FNU (NTU) standard (clear deionized or distilled water is suitable). Immerse the sensors into the water.

   NOTE: *With the calibration cup empty (i.e. no sensor guard or sensors), filling the calibration cup to line 1 will provide a sufficient amount of solution for calibration.*

3. Push the Cal key, then select **Turbidity**.
4. Select **Calibration Value** and enter 0.00.
5. Observe the data points readings for stability with the 0 FNU (NTU) standard (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration**. "Ready for cal point 2" will be displayed in the message area.
6. Select **Finish Calibration** to complete a 1-point calibration or continue for the 2- or 3-point calibration.
7. Rinse the sensors, calibration cup, and sensor guard 2-3 times with a small amount of standard #2. Discard the standard after each rinse.
8. Fill the calibration cup to the appropriate level with standard #2. Immerse the sensors in the second calibration standard.
9. Select **Calibration Value** and enter the value of the second calibration standard.
10. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration** (Figure 55). "Ready for cal point 3" will be displayed in the message area.
11. Select **Finish Calibration** to complete a 2-point calibration or continue for the 3-point calibration.
12. Rinse the sensors, calibration cup, and sensor guard 2-3 times with a small amount of standard #3. Discard the standard after each rinse.
13. Fill the calibration cup to the appropriate level with standard #3. Immerse the sensors in the third calibration standard.
14. Select **Calibration Value** and enter the value of the third calibration standard.
15. Observe the data points readings for stability, then select **Finish Calibration**. "Calibration successful!" will be displayed in the message area.
16. Rinse the sensors in clean water then dry.

ISEs: Ammonium, Nitrate, & Chloride

Before performing the calibration, review Calibration setup (pH, ORP, ISE, conductivity, turbidity) on page 33.

The ISE sensors can be calibrated to one, two or three points. A 2-point calibration without chilling a third calibration solution is extremely accurate and is the preferred method. However, if there is a large temperature variation during sampling, a chilled third calibration point is recommended.

Higher calibration accuracy can be obtained if the standards used have a least one order of magnitude difference between them. For example, 1 mg/L and 10 mg/L or 10 mg/L and 100 mg/L.

mV information for the ISE calibration

Ammonium mV values

- NH_4 1 mg/L = 0 mV +/- 20 mV (new sensor only)
- NH_4 100 mg/L = 90 to 130 mV > 1 mg/L mV value
- The mV span between 1 mg/L and 100 mg/L values should be ≈ 90 to 130 mV. The slope should be 45 to 65 mV per decade.

Nitrate mV values

- NO_3 1 mg/L = 200 mV +/- 20 mV (new sensor only)
- NO_3 100 mg/L = 90 to 130 mV < 1 mg/L mV value
- The mV span between 1 mg/L and 100 mg/L values should be ≈ 90 to 130 mV. The slope should be -45 to -65 mV per decade.

Chlroide mV values

- Cl 10 mg/L = 225 mV +/- 20 mV (new sensor only)
- Cl 1,000 mg/L = 80 to 130 mV < 10 mg/L mV value
- The mV span between 10 mg/L and 1000 mg/L values should be ≈ 80 to 130 mV. The slope should be -40 to -65 mV per decade.

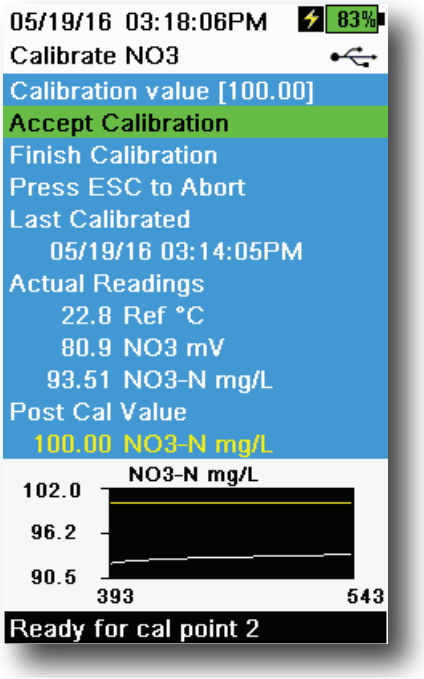


Figure 56 Calibrate ISE

ISE calibration 3-point

1. Perform the Calibration setup (pH, ORP, ISE, conductivity, turbidity) on page 33. Rinse the sensor 2-3 times with a small amount of standard #1.

   NOTE: *It is best to calibrate in order of increasing concentration (e.g. if using 1 mg/L and 100 mg/L standards, calibrate with 1 mg/L first).*

2. Push the Cal key, then select the applicable ISE sensor.
3. Carefully immerse the sensors into a solution of standard #1.
4. Allow the temperature of the standard to stabilize, then select **Calibration value**. Enter the calibration value that corresponds to standard #1.
5. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration** (Figure 56). "Ready for cal point 2" will be displayed in the message area.
6. Select **Finish Calibration** to complete a 1-point calibration. Otherwise, continue the calibration procedure to complete at least a 2-point calibration.

   NOTE: *A 2-point calibration is extremely accurate and is the preferred method.*

7. Rinse the sensor 2-3 times with a small amount of standard #2. Discard the standard after rinsing.
8. Carefully immerse the sensors into a fresh solution of standard #2.
9. Allow the temperature of the solution to stabilize then select **Calibration value**. Enter the calibration value that corresponds to standard #2.
10. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Accept Calibration** (Figure 56). "Ready for cal point 3" will be displayed in the message area.
11. Select **Finish Calibration** to complete a 2-point calibration. Otherwise, continue the calibration procedure to complete a 3-point calibration.

    NOTE: *To calibrate with a chilled third standard, see* Chilled third calibration point on page 46.

12. Rinse the sensor 2-3 times with a small amount of standard #3. Discard the standard after rinsing.
13. Carefully immerse the sensors into a fresh solution of standard #3.
14. Allow the temperature of the solution to stabilize then select **Calibration value**. Enter the calibration value that corresponds to standard #3.
15. Observe the actual measurement readings for stability (white line on graph shows no significant change for 40 seconds), then select **Finish Calibration**. "Calibration successful!" will be displayed in the message area.

Chilled third calibration point

The 3-point calibration method assures maximum accuracy when the temperature of the media to be monitored cannot be anticipated. If you must perform a chilled 3-point calibration, the following procedure requires one portion of the high concentration calibration solution and two portions of the low concentration calibration solution.

The high concentration solution and one of the low concentration solutions should be at ambient temperature. The other low concentration solution should be chilled to less than 10 °C (50 °F) to prior calibration point.

See ISE calibration 3-point on page 45.

1. When "Ready for cal point 3" is displayed in the message area during ISE calibration, place the proper amount of chilled 1 mg/L standard (10 mg/L for the chloride) into a clean, dry or pre-rinsed calibration cup.
2. Carefully immerse the sensor into the solution. Allow for temperature equilibration. If necessary, select **Calibration value** to manually enter the standard #3 value.
3. Once the readings are stable, select **Accept Calibration**. "Calibration successful!" will be displayed in the message area.

Preparing chloride standards

The following recipes are provided for preparation of 10 and 1000 mg/L chloride reagents. Nitrate and Ammonium standards can be purchased from YSI or other laboratory supply companies.

⚠ **WARNING:** Some of the chemicals required for these solutions could be hazardous under some conditions. It is the responsibility of the user to obtain and study the MSDS for each chemical and to follow the required instructions with regard to handling and disposal of these chemicals.

You will need:

- Solid sodium chloride or a certified 1000 mg/L chloride solution from a supplier
- Magnesium sulfate
- High-purity water
- A good quality analytical balance
- 1000 mL volumetric flask
- An accurate 10 mL measuring devices
- And 1000 mL glass or plastic storage vessels.

1000 mg/L Standard

1. Accurately weigh 1.655 grams of anhydrous sodium chloride and transfer into a 1000 mL volumetric flask.
2. Add 0.5 grams of anhydrous magnesium sulfate to the flask.
3. Add 500 mL of water to the flask, swirl to dissolve all of the reagents, then dilute to the volumetric mark with water.
4. Mix well by repeated inversion, then transfer the 1000 mg/L standard to a storage bottle.
5. Rinse the flask extensively with water prior to its use in the preparation of the 10 mg/L standard. Alternatively, simply add 0.5 grams of magnesium sulfate to a liter of a 1000 mg/L chloride standard from a certified supplier.

10 mg/L Standard

1. Accurately measure 10 mL of the above 1000 mg/L standard solution into a 1000 mL volumetric flask.
2. Add 0.5 grams of anhydrous magnesium sulfate to the flask.
3. Add 500 mL of water, swirl to dissolve the solid reagents, then dilute to the volumetric mark with water.
4. Mix well by repeated inversion, then transfer the 10 mg/L standard to a storage bottle.

Calibration

Preparing nitrate standards

We recommend using YSI calibration solutions whenever possible. However, qualified users can save cost by following these recipes for 1 and 100 mg/L nitrate standards. Other concentrations can be made by altering the amount of potassium nitrate. All other concentrations should remain unchanged.

⚠ **CAUTION:** Some of these chemicals are hazardous and therefore, the standards should only be prepared by qualified chemists in laboratories where proper safety precautions are possible. It is the responsibility of the user to obtain and study the MSDS for each chemical and to follow the required instructions with regard to handling and disposal of these materials.

You will need:

- Solid potassium nitrate or a certified 1000 mg/l NO_3-N from a supplier
- Magnesium sulfate, high purity water
- A good quality analytical balance
- 1000 mL volumetric flask
- Accurate volumetric measuring devices for 100 mL, 10 mL and 1 mL of solution
- And 1000 mL glass or plastic storage vessels.
-

100 mg/L standard

1. Accurately weigh 0.7222 g of anhydrous potassium nitrate and transfer quantitatively into a 1000 mL volumetric flask. Add 1.0 g of anhydrous magnesium sulfate to the flask.
2. Add approximately 500 mL of water to the flask. Swirl to dissolve all of the reagents, and then dilute to the volumetric mark with distilled or deionized water.
3. Mix well by repeated inversion and then transfer the 100 mg/L standard to a storage bottle.
4. Rinse the flask extensively with water prior to its use in the preparation of the 1 mg/l standard. Alternatively, 100 mL of certified 1000 mg/L NO_3-N standard can be used in place of the solid potassium nitrate.

1 mg/L standard

1. Accurately measure 10.0 mL of the above 100 mg/L standard solution into a 1000 mL volumetric flask. Add 1.0 g of anhydrous magnesium sulfate to the flask.
2. Add approximately 500 mL of distilled or deionized water. Swirl to dissolve the solid reagents, and then dilute to the volumetric mark with water.
3. Mix well by repeated inversion and then transfer the 1 mg/L standard to a storage bottle.

NOTE: *Recipes are given for 1 and 100 mg/L. Other concentrations can be made by altering the amount of potassium nitrate. All other concentrations should remain unchanged.*

Preparing ammonium standards

We recommend using YSI calibration solutions whenever possible. However, qualified users can save cost by following these recipes for 1 and 100 mg/L standards. Other concentrations can be made by altering the amount of ammonium chloride. All other ingredient concentrations should remain unchanged.

⚠ **CAUTION:** Some of these chemicals are hazardous and therefore, the standards should only be prepared by qualified chemists in laboratories where proper safety precautions are possible. It is the responsibility of the user to obtain and study the MSDS for each chemical and to follow the required instructions with regard to handling and disposal of these materials.

You will need:

- Solid ammonium chloride or a certified 100 mg/L NH_4^+-N from a supplier
- Lithium acetate dihydrate
- Concentrated hydrochloric acid
- High purity water
- A good quality analytical balance
- A 1000 mL volumetric flask
- Accurate volumetric measuring devices for 100 mL and 10 mL of solution
- And a 1000 mL glass or plastic storage vessels.

⚠ **CAUTION:** Hydrochloric acid is highly corrosive and toxic and should therefore be handled with extreme care in a well-ventilated fume hood. The user could also add the equivalent amount of a less-hazardous, more dilute sample of the acid if preferred.)

100 mg/L Standard

1. Accurately weigh 0.3817 g of ammonium chloride and transfer quantitatively into a 1000 mL volumetric flask. Add 2.6 g of lithium acetate dihydrate to the flask.
2. Add approximately 500 mL of distilled or deionized water to the flask. Swirl to dissolve all of the reagents and then dilute to the volumetric mark with distilled or deionized water.
3. Mix well by repeated inversion and then transfer the 100 mg/L standard to a storage bottle.
4. Add 3 drops of concentrated hydrochloric acid to the bottle, then seal and agitate to assure homogeneity. Alternatively, 100 mL of certified 100 mg/L NH4+-N standard can be used in place of the solid ammonium chloride.

1 mg/L Standard

1. Accurately measure 10.0 mL of the above 100 mg/L standard solution into a 1000 mL volumetric flask. Add 2.6 g of lithium acetate dihydrate to the flask.
2. Add approximately 500 mL of distilled or deionized water. Swirl to dissolve the solid reagents and then dilute to the volumetric mark with water.
3. Mix well by repeated inversion and then transfer the 1 mg/L standard to a storage bottle.
4. Add 3 drops of concentrated hydrochloric acid to the bottle, then seal and agitate to assure homogeneity.

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Maintenance and storage

Follow all maintenance and storage procedures in this section.

NOTICE: Incorrect or unapproved maintenance and/or storage can cause handheld, sensor or cable damage not covered by the warranty.

Unless otherwise specified, storage terms are defined as follows:

Short-term storage (less than 4 weeks): Storage when the ProDSS will be used at regular intervals (daily, weekly, biweekly, etc.)

Long-term storage: Storage when the ProDSS will have long periods of inactivity (over winter, end of monitoring season, etc.)

NOTICE: Perform sensor maintenance before long-term storage.

NOTICE: To prevent damage, do not store sensors in corrosive solutions.

ProDSS handheld instrument

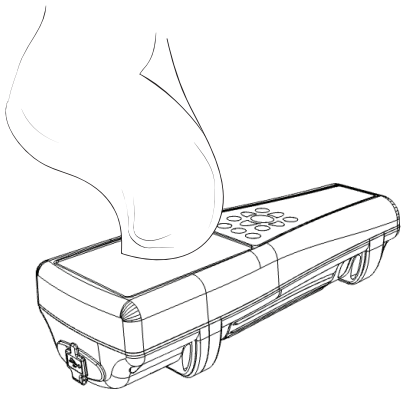

Figure 57 Handheld cleaning

Handheld instrument maintenance

Wipe the keypad, screen, and case with a cloth dampened with a mild solution of clean water and dish soap (Figure 57).

Handheld storage temperature

Optimal storage temperature of the handheld instrument:

- With battery pack installed: 0-45 °C (32-113 °F)
- Without battery pack installed: 0-60 °C (32-140 °F)

NOTICE: The battery pack permanently loses capacity at a faster rate when above 45 °C (113 °F).

Handheld short-term storage (less than 4 weeks)

Power off the handheld and store in a secure location (Startup on page 14).

Handheld long-term storage

1. Clean the handheld instrument.
2. Remove the battery pack to prevent possible battery leaks (Figure 2). Reinstall the battery cover.
3. Ensure the USB port cover is installed.
4. Store the handheld and removed battery pack in a secure location. See Rechargeable Lithium-Ion battery pack safety warnings and precautions on page 80.

Cable, bulkhead, and connectors

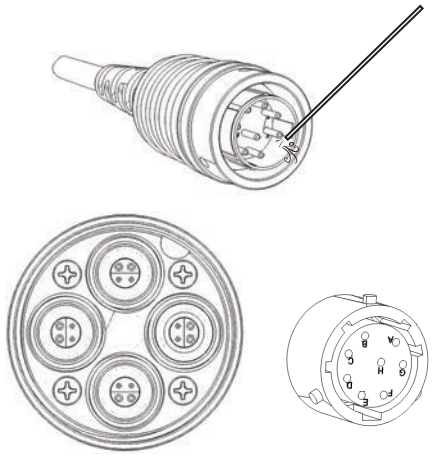

Figure 58 Cable, bulkhead, connector maintenance

Cable, bulkhead, and connector maintenance

Wipe the bulkhead cable with a cloth dampened with a mild solution of clean water and dish soap.

NOTICE: Install sensors or port plugs in ProDSS 4 port cable assemblies so the bulkhead ports do not get wet when cleaning. Exposure to water can cause damage or corrosion to the bulkhead connectors not covered by the warranty.

Inspect the bulkhead ports and cable connectors for contamination. If dirty or wet, clean it with compressed air (Figure 58).

Cable, bulkhead, and connector storage

Clean the connectors and bulkhead cable. For ProDSS 4 port cables, install the cap that protected the bulkhead during initial shipment. Alternatively, install bulkhead port plugs when not in use (Port plugs on page 10).

Sensor guard

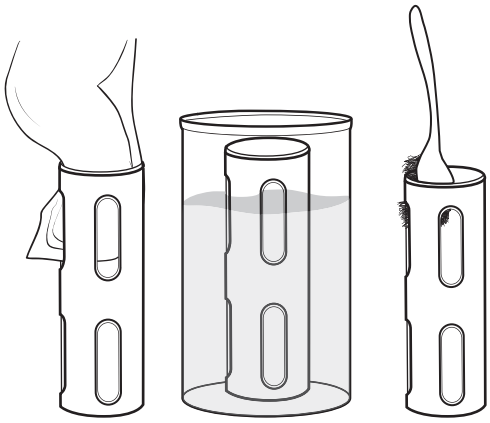

Figure 59 Sensor guard maintenance

Sensor guard maintenance

Remove minimal bio-fouling with a cloth soaked in a mild solution of clean water and dish soap (Figure 59).

Remove heavy bio-fouling by soaking the guard in a with a solution of clean water and dish soap. Soak in vinegar to remove hard growth and deposits.

Use a plastic scrub brush to remove any remaining bio-fouling. Rinse the sensor guard with clean water.

NOTICE: Do not sand or polish the guard. Removal of the guard coating can affect turbidity readings.

Depth sensor maintenance and storage

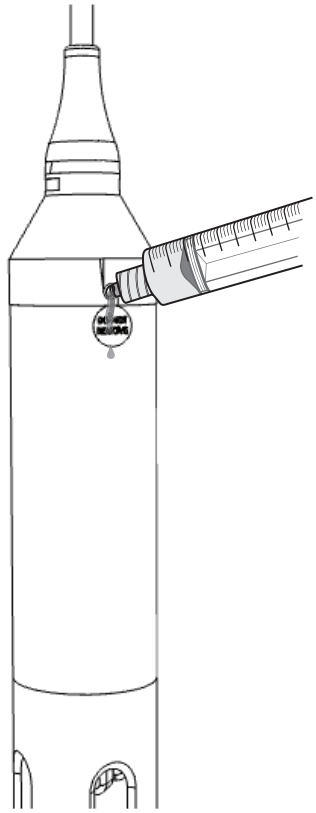

Figure 60 Depth sensor flush

Depth sensor storage

The ProDSS optional depth sensor on 4 port ProDSS cables accesses water through ports located in the bulkhead (Figure 60). Although not directly accessible, correct maintenance and storage is necessary for reliable operation.

The depth sensor can be stored dry, in water-saturated air or submerged in water.

NOTICE: To prevent damage to the sensor's strain gauge, do not store the sensor in corrosive solutions.

Depth sensor maintenance

Periodically clean the depth ports with the syringe included in the ProDSS maintenance kit (626990). Fill the syringe with clean water and gently force water into one of the ports. Flush until clean water flows from the opposite depth port.

NOTICE: Do not insert objects into the depth ports. Damage to the depth transducer from incorrect cleaning is not covered by the warranty.

Turbidity sensor

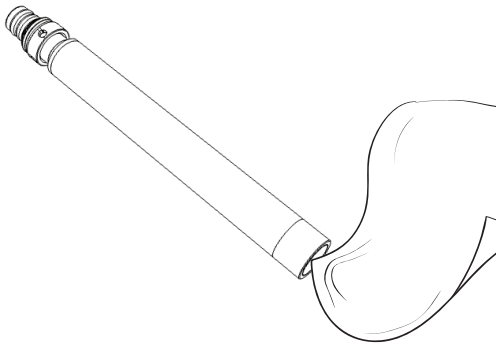

Figure 61 Turbidity sensor window

Turbidity sensor maintenance

Clean the sensing window with a non-abrasive, lint-free cloth (Figure 61).

NOTICE: Clean the window carefully to prevent scratches. If necessary, use mild soapy water.

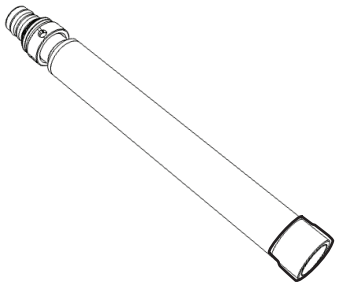

Figure 62 Turbidity sensor storage

Turbidity sensor short-term storage (less than 4 weeks)

When in regular field use, the turbidity sensor can remain installed on the bulkhead in an environment of water-saturated air (Figure 62).

NOTE: *The turbidity sensor can be stored dry if stored separate from other sensors.*

Place approximately 0.5 in (1 cm) of any water (tap or environmental) in calibration cup.

Install the calibration cup on the bulkhead and firmly tighten to prevent evaporation.

Turbidity sensor long-term storage

Store the turbidity sensor in dry air. The turbidity sensor can be left on the bulkhead or removed for storage.

If removed from the bulkhead, install the shipping cap on the sensor to prevent scratches or damage to the optical sensing window.

NOTICE: Install a port plug into the empty port on the bulkhead.

Conductivity/temperature sensor

NOTICE: Use care when handling the conductivity/temperature sensor to prevent any impact on the exposed thermistor.

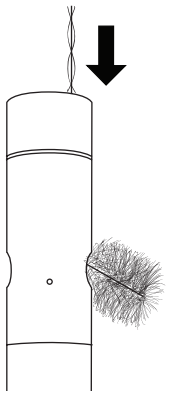

Figure 63 Channel brush

Conductivity/temperature sensor maintenance

1. Dip the sensor's cleaning brush (included with the maintenance kit) in clean water.
2. Insert the brush at the top of the channels, and sweep the channels 15 to 20 times (Figure 63).

   NOTICE: If deposits have formed on the electrodes, use a mild solution of dish soap and water to brush the channels. For heavy deposits, soak the sensor in white vinegar to assist cleaning, then scrub with the cleaning brush after soaking.
3. Rinse the channels with clean water following the sweepings or soak.

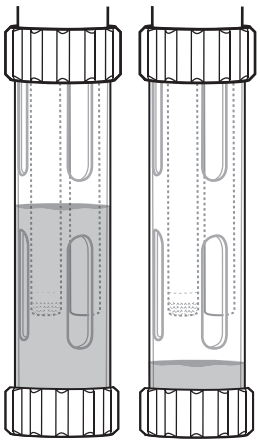

Figure 64 Conductivity/Temperature Short-term storage

Conductivity/temperature sensor short-term storage (less than 4 weeks)

When in regular field use, the conductivity/temperature sensor should remain installed on the bulkhead in a dry or water-saturated air environment.

Place approximately 0.5 in (1 cm) of any water (deionized, distilled or environmental) in calibration cup.

Install the calibration cup on the bulkhead and firmly tighten to prevent evaporation (Figure 64).

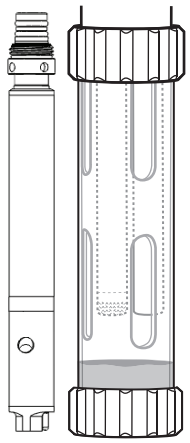

Figure 65 Conductivity/Temperature Long-term storage

Conductivity/temperature sensor long-term storage

The Conductivity/Temperature sensor can be stored dry or wet. For ProDSS 4 port cable assemblies, the sensor can be installed on the bulkhead or detached (Figure 65).

Dissolved oxygen sensor

ODO sensor caps are warranted for 1 year but have a typical working life of 18 to 24 months. The ODO Extended Warranty Sensor Cap (627180) that comes pre-installed on ODO/CT cable assemblies features a 2 year warranty. As the ODO sensor caps ages, large scratches in the paint/dye layer and changes in the dye layer can reduce measurement stability and response time.

Periodically inspect the sensor cap for damage and large scratches in the paint/dye layer. Replace the cap when readings become unstable and cleaning the cap and DO recalibration do not remedy the symptoms.

Cleaning the sensor cap

The sensor cap should be kept clean since some types of fouling may consume oxygen which could affect the dissolved oxygen measurements. To clean the sensor cap, gently wipe away any fouling with a lens cleaning tissue that has been moistened with water.

NOTICE: Do not use organic solvents to clean the sensor cap. Using an organic solvent to clean the sensor cap may cause permanent damage to the cap. For example, alcohol will dissolve the outer paint layer and other organic solvents will likely dissolve the dye in the cap.

ODO sensor cap replacement

The sensor cap should be replaced about once per year for those with a 1 year warranty, but the cap may last longer. It should also be replaced if it is cracked or damaged. The instruction sheet shipped with the replacement ODO sensor cap includes the calibration coefficients specific to your sensor cap.

The instructions for replacing the sensor cap on ProDSS ODO sensors (626900) are different than the instructions for integral (i.e. built-in) ODO sensors on ODO/CT (627150) and ProODO (626250) cable assemblies, so ensure the correct directions are being followed when replacing the sensor cap.

NOTE: *Make sure to save the ODO sensor cap instruction sheet in case you need to reload the calibration coefficients.*

NOTE: *The replacement ODO sensor cap is shipped in a humidified container and the package should not be opened until immediately before sensor cap replacement.*

Once the sensor cap has been installed on the ODO sensor, it is important to keep the sensor in a 100% humid environment. If the sensor dries out, refer to the rehydration procedure (ODO sensor rehydration on page 60).

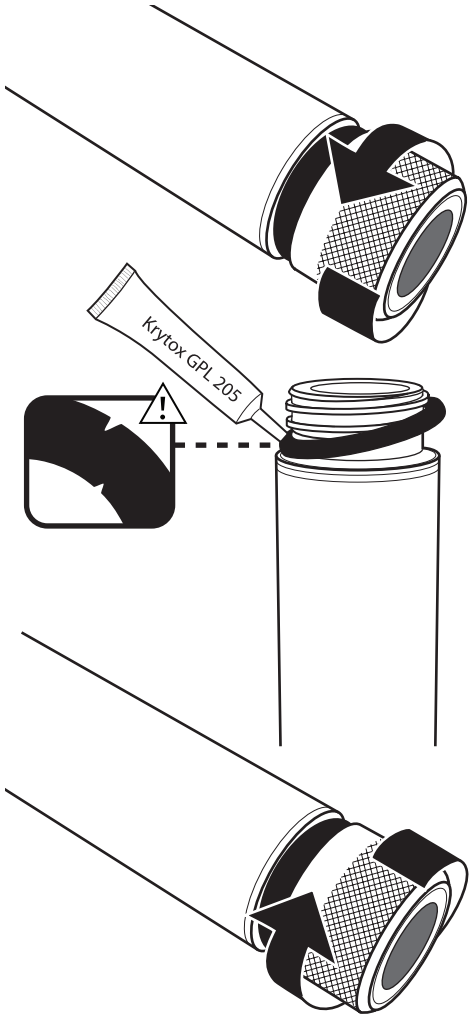

Figure 66 ProDSS ODO cap replacement

Sensor cap replacement - ProDSS ODO Sensors

1. Turn the used sensor cap counterclockwise to remove it from the sensor.

   NOTE: If possible, do not use a tool to remove the cap from the sensor. If necessary, carefully turn the cap counterclockwise with pliers until it breaks loose. Do not use the pliers on the sensor body. Make sure to not damage the sensor cap threads.

2. Without using tools, remove the used o-ring from the sensor body (pinch the o-ring out, then roll it upward over the threads), then discard it.
3. Clean the sensor threads with a clean, lint-free cloth.
4. Visually inspect the new o-ring for nicks, tears, contaminants or particles. Discard damaged o-rings.
5. Without twisting it, carefully install the new o-ring over the threads and into the o-ring groove.
6. Apply a thin coat of o-ring lubricant to the o-ring only. Wipe any excess from the threads and sensor body.
7. Clean the sensor window with a clean, lint-free cloth.
8. Make sure the new sensor cap cavity is completely dry, then carefully finger-tighten the cap clockwise onto the sensor. The o-ring should be compressed between the sensor cap and body, not pinched.

   NOTICE: Do not over-tighten the sensor cap. Do not use tools.

9. Store the ODO sensor in a moist environment.

   NOTE: If the o-ring is pinched, remove and discard it. Repeat steps 3 to 8.

Sensor cap replacement - Built-in ODO sensors on ODO/CT and ProODO cables

1. Remove the old sensor cap assembly from the probe by grasping the probe body with one hand and rotating the sensor cap counterclockwise until it is completely free. Do not use any tools for this procedure.
2. Inspect the o-ring on the probe for damage. If there is any indication of damage, carefully remove the o-ring and replace it with the new o-ring included with the replacement sensor cap. Do not use any tools to remove the o-ring.
3. Ensure the o-ring installed on the probe is clean. If necessary, wipe clean with a lint free cloth or replace the o-ring as described in the previous step.
4. Locate the o-ring lubricant included with the new sensor cap. Apply a thin coat of o-ring lubricant to the installed o-ring. After application, there should be a thin coat of o-ring lubricant on the o-ring only. Remove any excess o-ring lubricant from the o-ring and/or probe with a lens cleaning tissue.
5. Remove the new sensor cap from its hydrated container and dry the inside cavity of the sensor cap with lens cleaning tissue. Make sure the cavity is completely dry before proceeding with the installation. Next, clean the clear surface of the sensor on the end of the probe with lens cleaning tissue.
6. Using clockwise motion, thread the new sensor cap onto the probe assembly until it is finger-tight. The o-ring should be compressed between the sensor cap and probe. Do not over-tighten the sensor cap and do not use any tools for the installation process.
7. After installing the new sensor cap, store the sensor in either water or in humidified air in the calibration sleeve.

Updating the ODO sensor cap coefficients

After installing a new sensor cap, connect the bulkhead cable assembly to the ProDSS instrument and turn the instrument on. Locate the Calibration Code Label on the ODO sensor cap instruction sheet and note the eight sequences of letters and/or numbers listed as K1 through K7 and KC. These contain the calibration code for this particular sensor cap.

Follow the procedures below to enter the new calibration coefficients into the instrument.

1. Push the Probe key to access the Sensor menu, then select **Setup**, then **ODO**.
2. Select **Sensor Cap Coefficients**.
3. Highlight each coefficient in turn (K1 through KC) and use the numeric entry screen to enter the corresponding new coefficient from the Calibration Code Label. Push the ENTER key after each entry and then proceed to the next K selection.
4. After all the new coefficients have been entered, select **Update Sensor Cap Coefficients**.
5. A message will appear warning that you will be overwriting the current sensor cap coefficients and you should confirm that you wish to carry out this action. Select **Yes** to confirm the new coefficients.

After updating the Coefficients, the Serial # in the Sensor Cap menu will be updated automatically based on your entries. If errors are made in entering the Sensor Cap Coefficients, the instrument will block the update and an error message will appear on the display.

If you see this error message, re-enter the coefficients and check them carefully for correct transcription from the Calibration Code Label prior to selecting Update Sensor Cap Coefficients. If you receive an error message after several entry attempts, contact YSI Technical Support for assistance.

After entering the new Sensor Cap coefficients, perform a 1-point DO calibration (ODO% and ODO% local - water saturated air calibration on page 37).

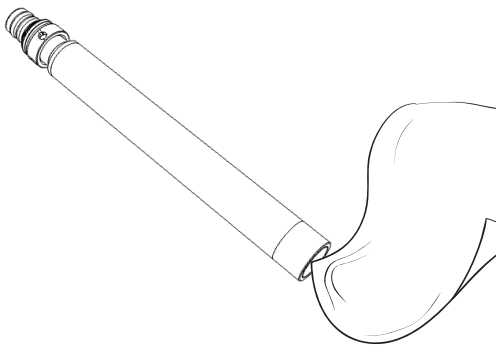

Figure 67 ODO sensor window

ODO sensor maintenance

Clean the sensing window with a non-abrasive, lint-free cloth (Figure 67).

NOTICE: Clean the window carefully to prevent scratches. Do not use organic solvents to clean the ODO sensor or sensor cap.

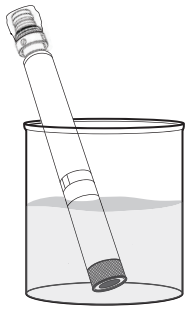

Figure 68 ODO rehydration

ODO sensor rehydration

To prevent sensor drift, always store the ODO sensor in a wet or water-saturated air environment. If the ODO sensor has accidentally been left dry for longer than 8 hours, it must be rehydrated.

If rehydration is necessary, soak the ODO sensor cap in warm (room temperature) tap water for approximately 24 hours. After the soak, calibrate the sensor (Figure 68).

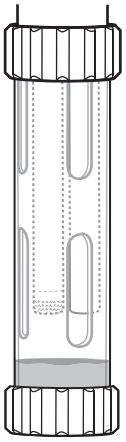

Figure 69 ODO short-term storage

ODO sensor short-term storage (less than 4 weeks)

When in regular field use, the ODO sensor should remain installed on the bulkhead. Place approximately 0.5 in (1 cm) of any water (tap or environmental) in the calibration cup (Figure 69).

Install the calibration cup onto the bulkhead and firmly tighten to prevent evaporation.

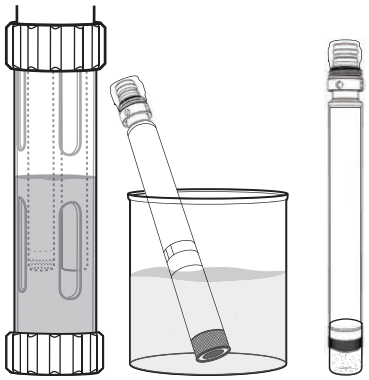

Figure 70 ODO long-term storage

ODO sensor long-term storage

For ProDSS 4 port cable assemblies, the ODO sensor can be left on the bulkhead or removed for long-term storage (Figure 70).

Installed on bulkhead

Fill the calibration cup with clean water (use distilled or deionized water if a pH sensor is not installed). Submerge the sensor in the calibration cup then firmly tighten to prevent evaporation.

Removed from bulkhead

Remove the sensor from the bulkhead (Sensor removal on page 10).

Method 1: Cover the sensor connector end with the plastic storage cap. Submerge the sensing end of the sensor in a container of clean water (use distilled or deionized water if a pH sensor is not installed). Periodically check the level of the water to make sure that it does not evaporate.

Method 2: Wet the sponge located in the cap originally included with the ODO sensor, then install on sensing end of the ODO sensor. Replace the sponge if it becomes dirty.

pH - pH/ORP sensors

NOTE: *pH and pH/ORP sensors require periodic maintenance to clear contamination from the sensing elements. These contaminants can slow sensor response time. Clean the sensors when deposits, bio-fouling or other contamination appears on the glass or when the sensor response time is noticeably slow.*

NOTICE: Do not physically scrub or swab the glass bulb. The bulbs are fragile and will break if pressed with sufficient force.

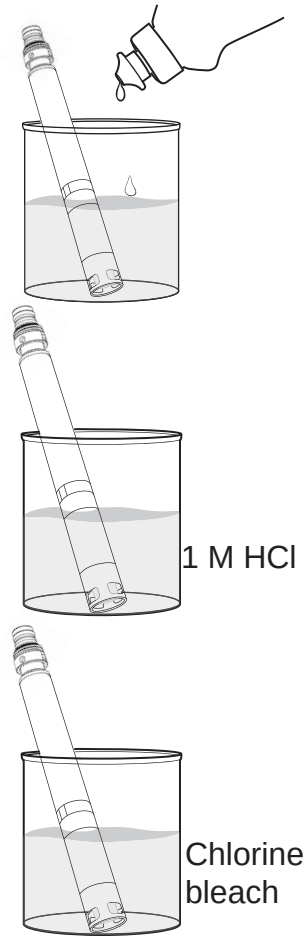

Figure 71 pH and pH/ORP sensor maintenance

pH - pH/ORP sensor maintenance

1. Remove the sensor from the bulkhead and soak for 10 to 15 minutes in a mild solution of clean water and dish soap (Figure 71).
2. Rinse the sensor with clean tap water and inspect.
3. If contaminants are removed, attach the sensor to the bulkhead and test the response time (ProDSS sensor installation/removal on page 9).

   OR

   If contaminants remain or response time does not improve, continue to the hydrochloric acid (HCl) soak in step 4.
4. Soak the sensor for 30 to 60 minutes in one molar (1 M) HCl.

   NOTE: *HCl reagent can be purchased from most chemical or laboratory distributors. If HCl is not available, soak in white vinegar.*

⚠ **CAUTION:** To prevent injury, carefully follow the HCl manufacturer's instructions.

5. Rinse the sensor in clean tap water.
6. Soak the sensor in clean tap water for 60 minutes, stirring occasionally. Repeat the clean tap water rinse.
7. Attach the sensor to the bulkhead and test the response time. If response time does not improve or biological contamination of the reference junction is suspected, continue to the chlorine bleach soak in step 8.
8. Soak the sensor for approximately one hour in a 1:1 dilution of chlorine bleach and tap water.
9. Rinse the sensor with clean tap water.
10. Soak the sensor in clean tap water for one hour or longer. Repeat the clean tap water rinse.

pH - pH/ORP sensor storage

The pH - pH/ORP sensors are shipped with their tips in a storage bottle containing KCl. Store the pH - pH/ORP sensors in the shipping container when not in use.

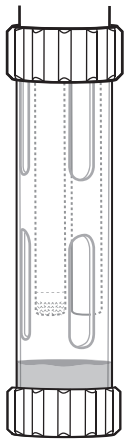

Figure 72 pH - pH/ORP short-term storage

pH - pH/ORP sensor short-term storage (less than 4 weeks)

When in regular field use, the pH-pH/ORP sensors should remain installed on the bulkhead. Place approximately 0.5 in (1 cm) of any water (tap or environmental) in the calibration cup (Figure 72).

Install the calibration cup onto the bulkhead and firmly tighten to prevent evaporation.

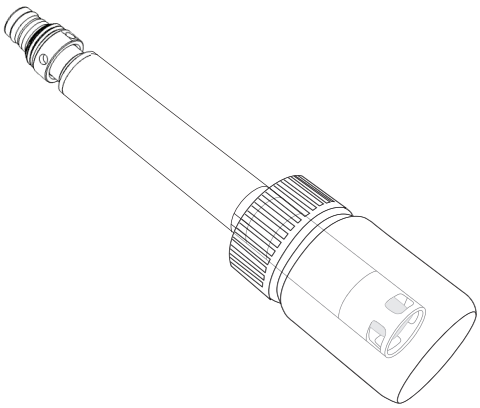

Figure 73 pH - pH/ORP long-term storage

pH - pH/ORP sensor long-term storage

Remove the sensor from the bulkhead and insert the sensing end into the shipping bottle. Install the bottle o-ring and tighten (Figure 73).

The shipping bottle contains a 2 molar solution of pH 4 buffer. If this solution is not available, store the sensor in tap water.

NOTICE: To prevent damage, do not store the pH - pH/ORP sensors in Zobell solution or DI water.

ISE sensors

Do not let the ISE sensor reference electrode junctions dry out. Clean the sensors when deposits, bio-fouling or other contamination appears on the membrane.

Ammonium and nitrate sensor maintenance

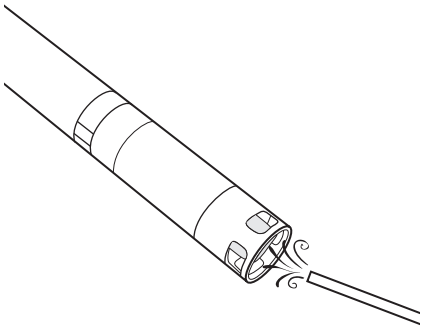

Figure 74 Ammonium and nitrate maintenance

1. Carefully clean the ammonium or nitrate sensor by rinsing with DI water followed by soaking in the high standard calibration solution (Figure 74).
2. Carefully dab the sensor dry with a clean, lint-free cloth.

NOTICE: The ion-selective membranes are very fragile. Do not use coarse material (e.g. paper towels) to clean the membranes or permanent damage to the sensor can occur. The only exception is fine emery cloth on the chloride sensor.

Chloride sensor maintenance

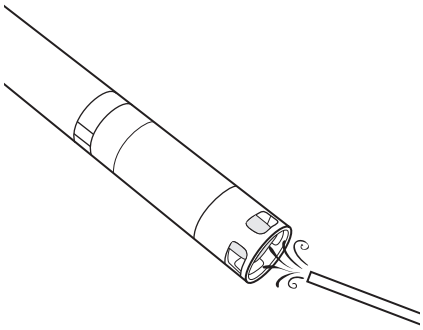

Figure 75 Chloride maintenance

Carefully clean the chloride sensor by carefully polishing with fine emery paper in a circular motion to remove deposits or discoloration (Figure 75).

Carefully rinse with DI water to remove any debris.

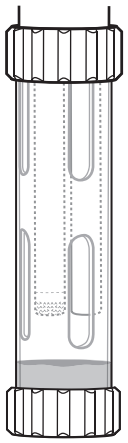

Figure 76 ISE short-term storage

ISE sensor short-term storage (less than 4 weeks)

When in regular field use, the ISE sensors should remain installed on the bulkhead in an environment of water-saturated air. Place approximately 0.5 in (1 cm) of any water (deionized, distilled or environmental) in the calibration cup (Figure 76).

Install the calibration cup onto the bulkhead and firmly tighten to prevent evaporation.

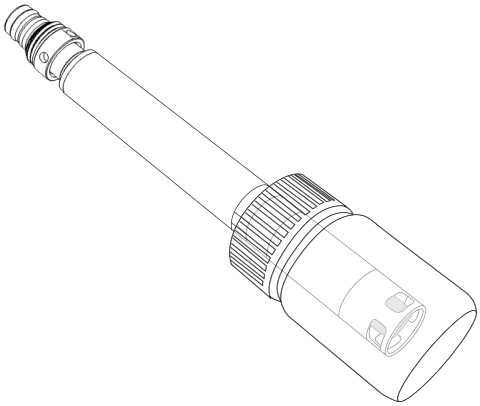

Figure 77 ISE long-term storage

ISE sensor long-term storage

NOTICE: Do not let the ISE junctions dry out. Junctions that have been allowed to dry out by improper storage may be irreparably damaged by dehydration and will require replacement.

1. Place a small amount of high-calibration solution or tap water in the storage bottle originally included with the sensor.
2. Remove the sensor from the bulkhead and insert the sensing end into the shipping bottle.
3. Install the bottle o-ring and tighten (Figure 77).

NOTICE: The sensors should not be immersed in water.

NOTICE: Do not store the ISE sensors in conductivity standard, pH buffer or salt water.

Rehydrating the reference junction

If an ISE sensor has been allowed to dry, soak the sensor for several hours (preferably overnight) in the sensor's high-calibration solution. If the sensor is irreparably damaged, the sensor module must be replaced.

ProDSS sensor module replacement

ProDSS pH, pH/ORP, ammonium, chloride and nitrate sensors feature replaceable sensor modules. These modules can be replaced by the user as needed. Typical working life of a pH or pH/ORP sensor module is 18 to 24 months. Typical working life of ammonium, chloride and nitrate sensor modules is 4 to 8 months.

Perform the pH - pH/ORP and ISE sensor module replacement in a clean, dry laboratory environment.

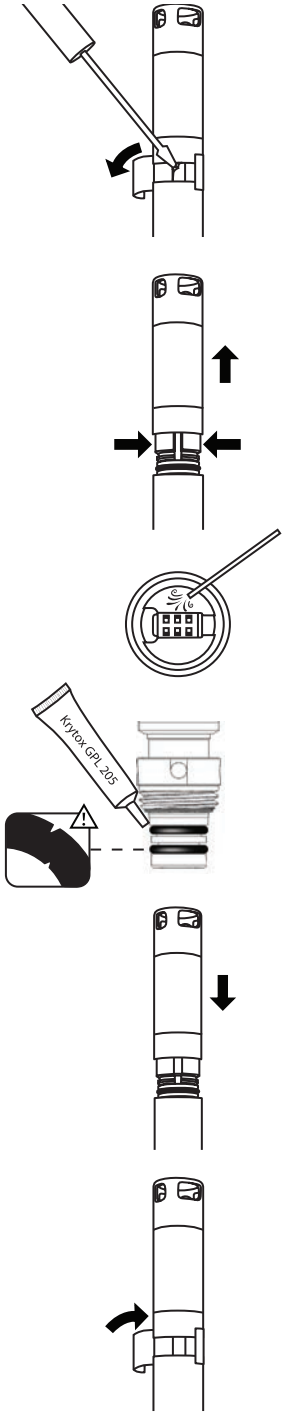

Figure 78 pH - pH/ORP sensor module replacement

Module replacement

1. Peel off and discard the sticker that covers the junction of the sensor body and the module (Figure 78).
2. With a small, flat-blade screwdriver, carefully remove the small rubber plug from the gap in the hard plastic ring at the base of the sensor module.
3. Using two fingers, squeeze the sensor module's hard plastic ring so that it compresses the gap left by the rubber plug.
4. Steadily pull the sensor module straight from the sensor body, rocking slightly if necessary.

   NOTICE: The o-ring is unusable after removal from the sensor body. Do not reinstall the removed sensor module or o-ring after removal. Dispose of the module according to you organization's guidelines or return it to YSI for recycling (Service information on page 82).
5. Inspect the sensor connector port for debris or moisture. If detected, remove it with lint-free cloth or a light blast of compressed air.
6. Visually inspect the two new o-rings for nicks, tears, contaminants or particles. Discard damaged o-rings.
7. Without twisting, carefully install the new o-rings over the threads and into the o-ring grooves.
8. Apply a thin coat of o-ring lubricant to the o-rings only. Wipe any excess from the threads and sensor module.

   NOTICE: If a sensor module is removed for any reason, the o-rings must be replaced.
9. Align the prongs on the base of the sensor module with the slots in the sensor body. The sensor module is keyed to insert in only one orientation.
10. Push the sensor module firmly into position until it clicks. Wipe any excess o-ring lubricant from the assembled components.
11. Wrap the junction of the sensor module and sensor body with the new sticker included in the sensor module kit. The sticker helps keep the sensor module junction clean and retain the rubber plug throughout deployment.
12. Write the replacement date on the sticker.
13. Calibrate the sensor (pH/ORP on page 39 or ISE calibration 3-point on page 45).

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KorDSS software installation

NOTE: *YSI recommends that you have administrative privileges on the PC in which KorDSS will be installed. This level of permission will allow the user to install software onto the PC. A warning will commonly indicate that software installation cannot be completed due to the lack of administrative privileges.*

On personal PC systems, the owner will typically have administrative privileges. Users of business and networked systems may not have administrative privileges. Information technology (IT) departments can customize these settings, so different levels of administrative privileges can exist. To obtain administrative privileges on your business or networked PC, please contact the IT department of your employer.

Follow these steps to complete the KorDSS installation process:

1. Install the KorDSS software from the USB flash drive included with the instrument.
2. Install the ProDSS instrument driver.
3. Start KorDSS for the first time and complete the KorDSS Startup Wizard.

System requirements

Supported 32 bit (x86) Microsoft Operating Systems:

- Microsoft Windows XP Home SP3
- Microsoft Windows XP Professional SP3
- Microsoft Windows 7 Home Basic SP1
- Microsoft Windows 7 Home Premium SP1
- Microsoft Windows 7 Professional SP1
- Microsoft Windows 7 Enterprise SP1
- Microsoft Windows 7 Ultimate SP1
- Microsoft Windows 8/8.1
- Microsoft Windows 8/8.1 Professional
- Microsoft Windows 8/8.1 Enterprise

Supported 64 bit (x64) Microsoft Operating Systems:

- Microsoft Windows 7 Home Basic SP1
- Microsoft Windows 7 Home Premium SP1
- Microsoft Windows 7 Professional SP1
- Microsoft Windows 7 Enterprise SP1
- Microsoft Windows 7 Ultimate SP1
- Microsoft Windows 8/8.1
- Microsoft Windows 8/8.1 Professional
- Microsoft Windows 8/8.1 Enterprise

Ram Memory Requirement:

- Minimum of 2 GB of RAM installed

Hard Disk Free Space:

- Minimum of 500 MB of free hard drive space

Internet Access Required to Support:

- Software and device updates, software licensing, and maps

Install the KorDSS software

1. Insert the supplied USB flash drive into a USB port on your computer.
2. Depending on the PC operating system and system settings, the KorDSS Installer Guide may appear. If it does not appear, double-click **Start.exe** to start the installer guide (Figure 79).

NOTE: *If desired, view the ProDSS User Manual or the end-user license agreement.*

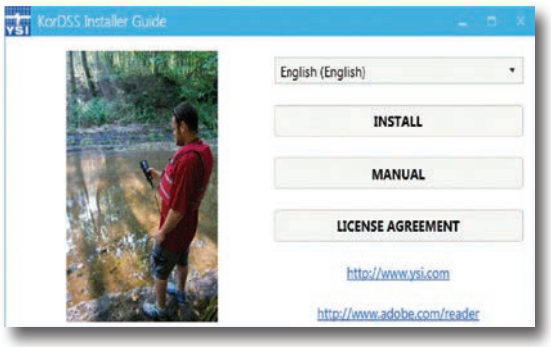

Figure 79 KorDSS Installer Guide

3. Click **Install** on the KorDSS Installer Guide.
4. Check the license agreement box. Click **Install** (Figure 80).

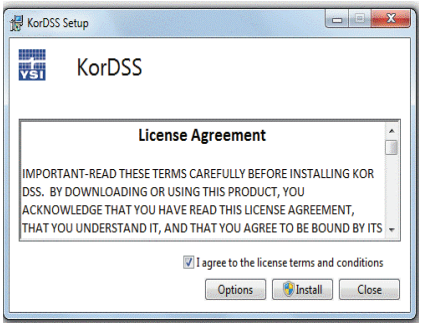

Figure 80 KorDSS license agreement

5. You may be asked if you want to allow a program from an unknown publisher to make changes on the computer. If so, select **Yes**.

KorDSS is now installed. Before using KorDSS to manage data, you must install the driver for the ProDSS instrument on your PC.

NOTE: *KorDSS has a built-in help file under the "File" tab or the program. This document provides an overview of all program features.*

ProDSS driver installation

NOTE: *KorDSS must be installed on your PC before the ProDSS driver can be installed. The driver is partially installed on the computer during the KorDSS install process. Connect the ProDSS instrument to your PC and follow the instructions for your operating system to finish the driver installation.*

NOTE: *The ProDSS driver installation procedure allows the KorDSS software to recognize the instrument. A COM port will be assigned to each ProDSS instrument that is connected to your PC, but the driver installation procedure is required only one time.*

The driver installation procedure is different for each operating system. Follow the applicable installation procedure carefully.

Windows XP ProDSS driver on page 70
Windows 8 and 8.1 ProDSS driver on page 71
Windows 10 ProDSS driver on page 72

Windows 7 ProDSS driver

1. Turn the instrument on and connect it to the PC with the included USB cable.

If a message appears indicating successful download of the driver, proceed to the KorDSS Startup Wizard (page 73).

If you do not see a message indicating the successful download of the driver or if you see a message indicating unsuccessful download of the driver, continue this driver installation procedure.

2. Open the Device Manager. To access: Click the **Start** button, click **Control Panel**, click **System and Security**, and then, under **System**, click **Device Manager**.
3. Under **Other devices**, right click on **smxUSBD Serial Emulator** and select **Update Driver Software** (Figure 81).

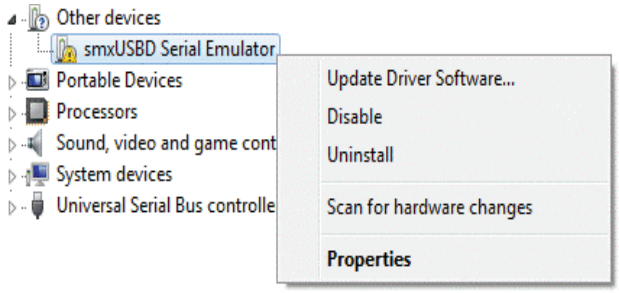

Figure 81 Device Manager Windows 7

4. Click **Browse my computer for driver software**.
5. Click **Browse**, then navigate to the file location: **C:\Program Files (x86)\YSI\KorDSS** for 64 bit systems or **C:\Program Files\YSI\KorDSS** for 32 bit systems. Click **Next**.
6. A warning will appear indicating that Windows can't verify the publisher of the driver software. Select **Install this driver software anyway**.
7. After driver installation, proceed to the KorDSS Startup Wizard (page 73).

Windows XP ProDSS driver

1. Turn the instrument on and connect it to the PC with the included USB cable.
2. On the **Found New Hardware Wizard** window, select **No, not at this time** when asked if Windows can connect to Windows Update. Click **Next**.
3. Select **Install from a list or specific location**, then click **Next**.
4. Select **Search for the best driver in these locations**, then **Include this location in the search:**. Click **Browse**, then navigate to the file location: **C:\Program Files (x86)\YSI\KorDSS** for 64 bit systems or **C:\Program Files\YSI\KorDSS** for 32 bit systems (Figure 82). Click **Next**.

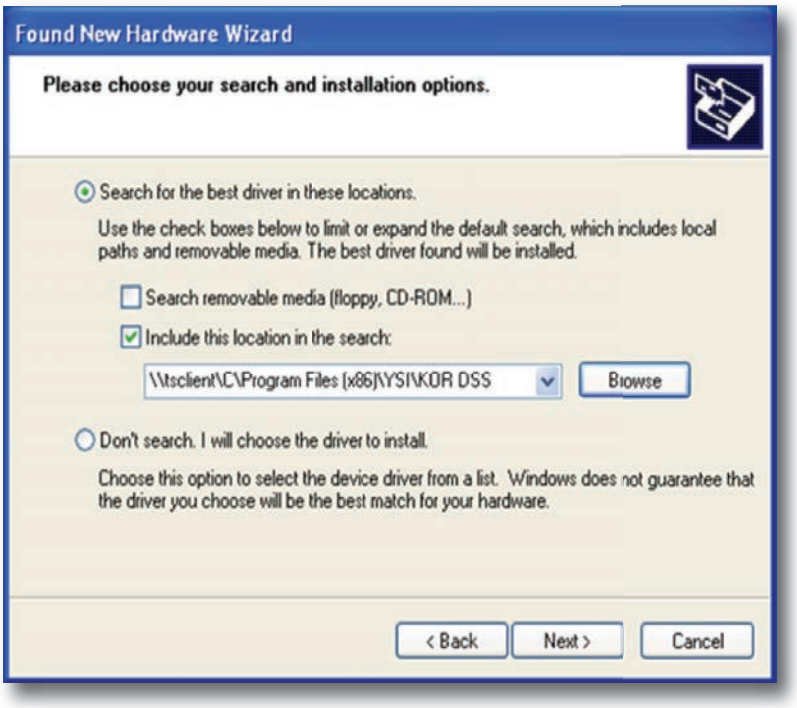

Figure 82 Found New Hardware Wizard file location

5. Select **Continue Anyway** when warned that the software has not passed Windows Logo testing.
6. Click **Finish** to close the New Hardware Wizard.
7. After driver installation, proceed to the KorDSS Startup Wizard (page 73).

Windows 8 and 8.1 ProDSS driver

1. Save any open files and close all programs. Your computer will restart during this process.
2. Open **Settings** by moving the computer mouse to the bottom right corner of the computer screen. If using a touch screen, swipe the screen from the right to reveal the Settings charm. Alternately, settings can be opened by pressing the Windows key + I.
3. Complete the following navigation steps under Settings:

- For Windows 8.1: **Change PC Settings** → **Update and Recovery** → **Advanced Setup** → **Restart now**
- For Windows 8: **Change PC Settings** → **General** → **Advanced Setup** → **Restart now**

4. When the **Choose an option** appears, select **Troubleshoot**, then **Advanced Options**.
5. Select **Startup Settings**, then **Restart**.
6. After the computer reboots, the **Startup Settings** screen will be shown. Use the **F7** or **7** key to select **Disable driver signature enforcement**.
7. Connect the ProDSS to the PC with the included USB cable. After connection, turn the instrument on.
8. Open Device Manager by pressing the Windows Key + X to open the Start Menu, then selecting **Device Manager**. Alternately, search for **devmgmt**, then select **Device Manager**.
9. Under **Other devices**, right click on **smxUSBD Serial Emulator** and select **Update Driver Software** (Figure 83).

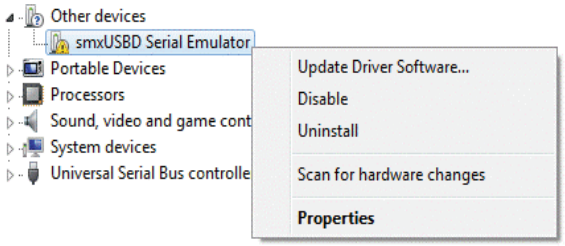

Figure 83 Device Manager Windows 8/8.1

10. Click **Browse my computer for driver software**.
11. Click **Browse**, then navigate to the file location: **C:\Program Files (x86)\YSI\KorDSS** for 64 bit systems or **C:\Program Files\YSI\KorDSS** for 32 bit systems (Figure 84). Click **Next**.

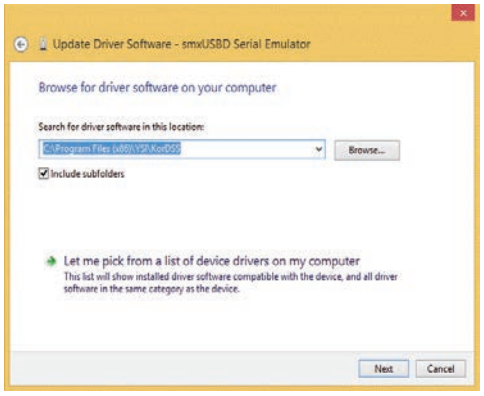

Figure 84 Driver location Windows 8/8.1

12. A warning will appear indicating that Windows can't verify the publisher of the driver software. Select **Install this driver software anyway**.
13. After driver installation, reboot the computer, then proceed to the KorDSS Startup Wizard (page 73).

Windows 10 ProDSS driver

NOTE: *KorDSS compatibility with Windows 10 has not been fully tested, but will likely work. The following driver install instructions can be used if you install KorDSS on a computer with Windows 10.*

1. Turn the instrument on and connect it to the PC with the included USB cable.
2. Open **Device Manager** by typing "Device Manager" in the search box on the Windows 10 taskbar (Figure 85).

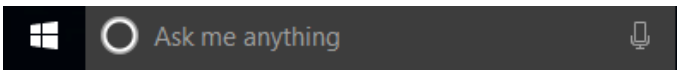

Figure 85 Windows 10 search box

3. Under **Ports**, right click on **USB Serial Device** and select **Update Driver Software** (Figure 86).

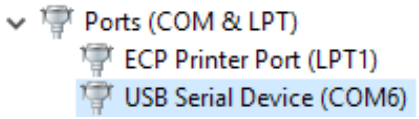

Figure 86 Device Manager Windows 10

4. Click **Browse my computer for driver software**.
5. Click Browse, then navigate to the file location: **C:\Program Files (x86)\YSI\KorDSS** for 64 bit systems (Figure 87) or **C:\Program Files\YSI\KorDSS** for 32 bit systems. Click **Next**.

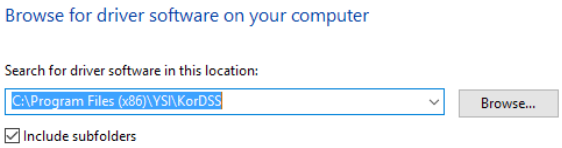

Figure 87 Driver location Windows 10

6. After successful driver installation, proceed to the KorDSS Startup Wizard (page 73).

KorDSS startup wizard

1. After Windows has successfully updated the driver software, start KorDSS and set the language preference (Figure 88). Click **Next**.

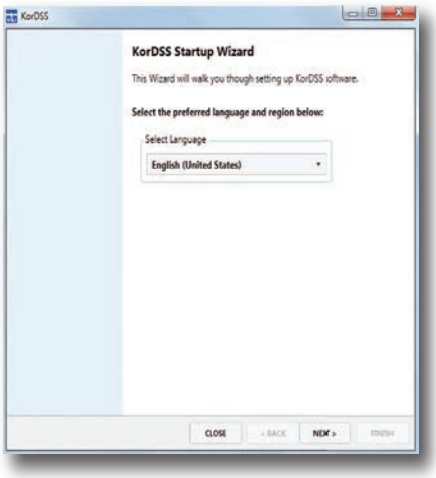

Figure 88 KorDSS language preference

2. On the Software Licensing Mode screen, select **Premium Mode** if you would like to view sampling locations on a map (internet connection required) (Figure 89). To upgrade to Premium Mode for free, follow the link, register your ProDSS, then use the code sent to you via email to upgrade to the Premium Mode. You can upgrade to Premium Mode at any time by going to the File tab in KorDSS.

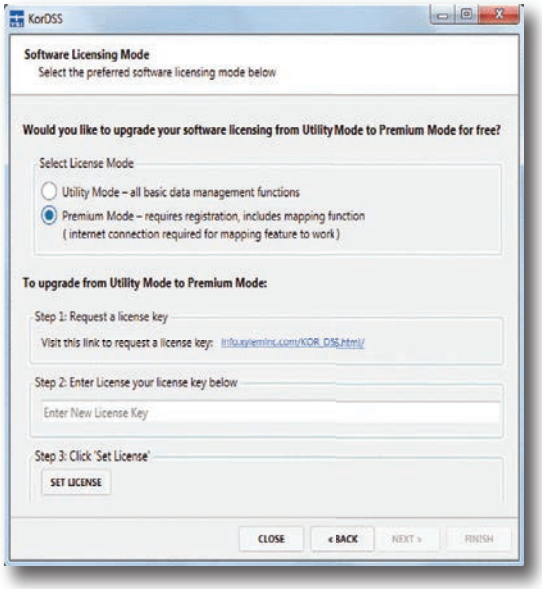

Figure 89 Software licensing mode screen

3. Select your ProDSS and KorDSS update preference to finish the installation process. Consult the HTML help file, found under the File tab of the KorDSS software, for a complete description of all KorDSS features.

Accessories

Ordering

Telephone: 800 897 4151 (USA)
+1 937 767 7241 (Globally) Monday through Friday, 8:00 AM to 5:00 ET
Fax: +1 937 767 9353 (orders)
Email: info@ysi.com
Mail: YSI Incorporated 1725 Brannum Lane Yellow Springs, OH 45387 USA
Internet: ysi.com

When placing an order please have the following available:

1. YSI account number (if available)
2. Name and phone number
3. Purchase Order or Credit Card number
4. Model Number or brief description
5. Billing and shipping addresses
6. Quantity

ProDSS handhelds

YSI Item #	Description
626870-1	ProDSS handheld, no GPS
626870-2	ProDSS handheld with GPS

ProDSS 4 port cable assemblies (No sensors included)

YSI Item #	Description
626909-1	ProDSS-1 meter 4 port cable assembly, no depth
626909-4	ProDSS-4 meter 4 port cable assembly, no depth
626909-10	ProDSS-10 meter 4 port cable assembly, no depth
626909-20	ProDSS-20 meter 4 port cable assembly, no depth
626909-30	ProDSS-30 meter 4 port cable assembly, no depth
626909-40	ProDSS-40 meter 4 port cable assembly, no depth
626909-50	ProDSS-50 meter 4 port cable assembly, no depth
626909-60	ProDSS-60 meter 4 port cable assembly, no depth
626909-70	ProDSS-70 meter 4 port cable assembly, no depth
626909-80	ProDSS-80 meter 4 port cable assembly, no depth
626909-90	ProDSS-90 meter 4 port cable assembly, no depth
626909-100	ProDSS-100 meter 4 port cable assembly, no depth
626910-1	ProDSS-1 meter 4 port cable assembly, with depth
626910-4	ProDSS-4 meter 4 port cable assembly, with depth
626910-10	ProDSS-10 meter 4 port cable assembly, with depth
626911-20	ProDSS-20 meter 4 port cable assembly, with depth
626911-30	ProDSS-30 meter 4 port cable assembly, with depth
626911-40	ProDSS-40 meter 4 port cable assembly, with depth
626911-50	ProDSS-50 meter 4 port cable assembly, with depth
626911-60	ProDSS-60 meter 4 port cable assembly, with depth
626911-70	ProDSS-70 meter 4 port cable assembly, with depth
626911-80	ProDSS-80 meter 4 port cable assembly, with depth
626911-90	ProDSS-90 meter 4 port cable assembly, with depth
626911-100	ProDSS-100 meter 4 port cable assembly, with depth

ProDSS smart sensors

YSI Item #	Description
626900	ProDSS Optical Dissolved Oxygen sensor
626902	ProDSS conductivity and temperature sensor
626901	ProDSS turbidity sensor
626903	ProDSS pH sensor with module
626904	ProDSS pH/ORP sensor with module
626906	ProDSS ammonium sensor with module
626905	ProDSS nitrate sensor with module
626907	ProDSS chloride sensor with module

Replacement sensor modules and ODO sensor caps

YSI Item #	Description
626890	Replacement ProDSS Optical Dissolved Oxygen sensor cap (for 626900 smart sensor)
626320	Replacement ProODO Optical Dissolved Oxygen sensor cap (for 626250 probe/cable assemblies)
626482	Replacement ProOBOD Optical Dissolved Oxygen sensor cap (for 626400 or 626401 lab probes)
627160	Replacement ProDSS ODO/CT ***ODO Sensor Cap*** (for 627150 probe/cable assemblies)
627180	Replacement ProDSS ODO/CT ***ODO Extended Warranty Sensor Cap*** (for 627150 probe/cable assemblies; comes pre-installed on new ProDSS ODO/CT assemblies)
626963	Replacement ProDSS pH sensor module
626964	Replacement ProDSS pH/ORP sensor module
626966	Replacement ProDSS Ammonium sensor module
626965	Replacement ProDSS Nitrate sensor module
626967	Replacement ProDSS Chloride sensor module

ProDSS ODO/CT sensor and cable assemblies - DO/Conductivity/Temp only

NOTE: *ProDSS ODO/CT cable assemblies feature non-replaceable temperature, conductivity, and optical DO sensors. There is no depth option with ODO/CT cables.*

NOTE: *There are two replacement sensor cap options for the ODO sensor on ODO/CT cable assemblies. The* ***ODO Sensor Cap*** *(item # 627160) features a 1 year warranty, while the* ***ODO Extended Warranty Sensor Cap*** *(item # 627180) is more rugged and features a 2 year warranty. The ODO Extended Warranty Sensor Cap comes pre-installed on new ODO/CT cable assemblies, with calibration coefficients of the sensor cap pre-loaded into the probe at the factory.*

YSI Item #	Description
627150-1	ProDSS ODO/CT-1 meter cable assembly with non-replaceable sensors, no depth
627150-4	ProDSS ODO/CT-4 meter cable assembly with non-replaceable sensors, no depth
627150-10	ProDSS ODO/CT-10 meter cable assembly with non-replaceable sensors, no depth
627150-20	ProDSS ODO/CT-20 meter cable assembly with non-replaceable sensors, no depth
627150-30	ProDSS ODO/CT-30 meter cable assembly with non-replaceable sensors, no depth
627150-40	ProDSS ODO/CT-40 meter cable assembly with non-replaceable sensors, no depth
627150-50	ProDSS ODO/CT-50 meter cable assembly with non-replaceable sensors, no depth
627150-60	ProDSS ODO/CT-60 meter cable assembly with non-replaceable sensors, no depth
627150-70	ProDSS ODO/CT-70 meter cable assembly with non-replaceable sensors, no depth
627150-80	ProDSS ODO/CT-80 meter cable assembly with non-replaceable sensors, no depth
627150-90	ProDSS ODO/CT-90 meter cable assembly with non-replaceable sensors, no depth
627150-100	ProDSS ODO/CT-100 meter cable assembly with non-replaceable sensors, no depth

ProODO/ProOBOD sensor and cable assemblies - DO/Temp only

NOTE: *ProODO/ProOBOD cable assemblies feature non-replaceable temperature and optical DO sensors. Sensor caps for optical DO sensors are replaceable (626320 for ProODO cable assemblies; 626482 for ProOBOD cable assemblies). There is no depth option with ProODO/ProOBOD cable assemblies.*

YSI Item #	Description
626250-1	ProODO-1 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626250-4	ProODO-4 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626250-10	ProODO-10 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626250-20	ProODO-20 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626250-30	ProODO-30 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626250-40	ProODO-40 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626250-50	ProODO-50 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626250-60	ProODO-60 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626250-70	ProODO-70 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626250-80	ProODO-80 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626250-90	ProODO-90 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626250-100	ProODO-100 meter cable assembly with non-replaceable ODO/temperature sensors, no depth
626400	ProOBOD BOD probe/cable assembly, lab probe; U.S./Japanese version with power supply
626401	ProOBOD BOD probe/cable assembly, lab probe; International version with power supply

Calibration standards

YSI Item #	Description
065270	Conductivity standard, 1000 µmhos/cm (quart, glass); ideal for fresh water
065272	Conductivity standard, 10000 µmhos/cm (quart, glass); ideal for brackish water
065274	Conductivity standard, 100000 µmhos/cm (quart, glass); ideal for supersaturated sea water
060907	Conductivity standard, 1000 µmhos/cm (box of 8 individual pints, plastic); ideal for fresh water
060906	Conductivity standard, 1413 µmhos/cm, ±1%, 0.01 M KCl (box of 8 individual pints, plastic)
060911	Conductivity standard, 10000 µmhos/cm (box of 8 individual pints, plastic); ideal for brackish water
060660	Conductivity standard, 50000 µmhos/cm (box of 8 individual pints, plastic); ideal for sea water
061320	ORP (mV) standard, Zobell solution, powder - needs hydrated (125 mL bottle, plastic)
061321	ORP (mV) standard, Zobell solution, powder - needs hydrated (250 mL bottle, plastic)
061322	ORP (mV) standard, Zobell solution, powder - needs hydrated (500 mL bottle, plastic)
003821	pH 4 buffer (box of 6 individual pints, plastic); ideal for storage solution for pH sensor
003822	pH 7 buffer (box of 6 individual pints, plastic)
003823	pH 10 buffer (box of 6 individual pints, plastic)
603824	Assorted case of pH 4, 7, and 10 buffers (2 individual pints of each buffer, plastic)
005580	Confidence solution to verify conductivity, pH and ORP system (box of 6 individual 475 mL bottles, plastic). ***Note:*** *Not for calibration*
003841	Ammonium standard, 1 mg/L (500 mL, plastic)
003842	Ammonium standard, 10 mg/L (500 mL, plastic)
003843	Ammonium standard, 100 mg/L (500 mL, plastic)
003885	Nitrate standard, 1 mg/L (500 mL, plastic)
003886	Nitrate standard, 10 mg/L (500 mL, plastic)
003887	Nitrate standard, 100 mg/L (500 mL, plastic)
608000	Turbidity standard, 0 FNU (1 gallon, plastic)
607200	Turbidity standard, 12.4 FNU (1 gallon, plastic)
607300	Turbidity standard, 124 FNU (1 gallon, plastic)
607400	Turbidity standard, 1010 FNU (1 gallon, plastic)

ProDSS accessories

YSI Item #	Description
626946	Large, hard-sided carrying case (Fits ProDSS 4 port cables 10, 20, and 30 meters in length, cable management kit, handheld, and accessories)
603075	Large, soft-sided carrying case
626945	Small, hard-sided carrying case (Fits ProDSS 4 port cables 1 and 4 meters in length, handheld, flow cell, and accessories)
599080	Flow cell for ProDSS 4 port cables
603076	Flow cell for ProDSS ODO/CT cables (requires single port adapter; 603078)
603078	Adapter required for ProDSS ODO/CT flow cell (603076)
603056	Flow cell mounting spike
063507	Tripod (screws into back of meter)
063517	Ultra clamp (screws into back of meter)
603070	Shoulder strap
603069	Belt clip (screws into back of meter)
626942	USB car charger
626943	Small external Li-Ion rechargeable battery pack (Typical performance: will charge a completely discharged ProDSS battery to about 50%)
626944	Large external Li-Ion rechargeable battery pack (Typical performance: will charge a completely discharged ProDSS battery to full charge, plus have power to charge a second battery to 20%)
626940	AC charger (USA). Includes power supply and USB cable (included with ProDSS handheld)
626941	AC charger (international). Includes power supply, USB cable and outlet adapters (included with ProDSS handheld)
626846	Replacement Lithium-ion battery pack
626969	ProDSS USB flash drive (included with ProDSS handheld)
626991	Cable for charging and PC connection (included as part of 626940 and 626941)
626992	Cable for connection to USB drive (included with ProDSS handheld)
626990	ProDSS maintenance kit (included with all ProDSS 4 port cables): • 3 port plugs • 1 Krytox tube • 1 brush • 1 syringe • 1 sensor installation/removal tool • O-rings (6)
626919	Sensor guard for 4 port ProDSS cable assembly (included with all ProDSS cables)
599786	Calibration/storage cup for 4 port ProDSS cable assembly (included with all 4 port ProDSS cables)
627195	Calibration cup for ProDSS ODO/CT cable assembly (included with all ProDSS ODO/CT cables)
603062	Cable management kit (included with **ProDSS 4 port cables** 10, 20, and 30-meters long; **ProDSS ODO/CT cables** 4, 10, 20, 30, 40, and 50-meters long; and **ProODO cables** 4, 10, 20, and 30-meters long)
626918	1 lb weight (included with ProDSS 4 port cables 10-meters and longer)
605978	4.9 oz weight

Safety and support

Rechargeable Lithium-Ion battery pack safety warnings and precautions

⚠ **CAUTION:** Failure to follow the safety warnings and precautions can result in fire, personal injury and/or equipment damage not covered under warranty.

⚠ **CAUTION:** If the internal battery fluid comes into contact with skin, wash the affected area(s) with soap and water immediately. If it comes into contact with your eye(s), flush them with generous amounts of water for 15 minutes and seek immediate medical attention.

⚠ **CAUTION:** Always keep batteries away from children.

⚠ **WARNING:** In the unlikely event a lithium-ion battery catches fire, **DO NOT** attempt to put the fire out with water, use a Class A, B or C fire extinguisher.

Do:

- Store the battery pack in a cool, dry, ventilated area.
- Store the battery pack in a non-conductive and fireproof container.
- Store the battery pack at approximately 50% of the capacity.
- Disconnect the battery pack when not in use and for long-term storage.
- Follow applicable laws and regulations for transporting and shipping of batteries.
- *Immediately discontinue* use of the battery pack if, while using, charging or storing the battery pack:
  - Emits an unusual smell
  - Feel hot
  - Changes color
  - Changes shape
  - Appears abnormal in any other way.

Battery pack general precautions:

- **DO NOT** put the battery in fire or heat the battery.
- **DO NOT** connect the positive and the negative terminal of the battery to each other with any metal object (e.g. wire).
- **DO NOT** carry or store the battery pack with neckaces, hairpins or other metal objects.
- **DO NOT** carry or store the battery pack with hazardous or combustible materials.
- **DO NOT** pierce the battery pack with nails, strike with a hammer, step on or otherwise subject the battery pack to strong impacts or shocks.
- **DO NOT** solder directly onto the battery pack.
- **DO NOT** expose the battery pack to water or salt water or allow it to get wet.
- **DO NOT** disassemble or modify the battery pack. The battery contains safety and protection devices that, if damaged, can cause the battery to generate heat, rupture or ignite.
- **DO NOT** place the battery pack on or near fires, stoves or other high-temperature locations.
- **DO NOT** place the battery pack in direct sunlight or extreme temperatures for extended periods of time or store the battery pack inside cars in hot weather. Doing so may cause the battery pack to generate heat, rupture or ignite. Using the battery pack in this manner may also result in a loss of performance and a shortened life expectancy.
- **DO NOT** place the battery pack in microwave ovens, high-pressure containers or on induction cookware.
- **DO NOT** ship damaged or potentially defective batteries to YSI or any of our authorized service centers unless instructed otherwise. All federal and international shipping laws should be consulted prior to shipping lithium-ion batteries.

Charging/discharging/handling the battery pack

⚠ **WARNING:** Failure to follow the battery pack charging/discharging instructions can cause the battery to become hot, rupture or ignite and cause serious injury and/or equipment damage.

⚠ **WARNING:** Only charge the battery using charging devices designed specifically for the ProDSS by YSI. Use of unapproved chargers can result in battery failure and potentially serious injury to the user.

If at any time the battery pack becomes damaged, hot or begins to balloon or swell, discontinue charging (or discharging) immediately. Quickly and safely disconnect the charger. Then place the battery pack and/or charger in a safe, open area way from flammable materials. After one hour of observation, remove the battery pack from service. **DO NOT** continue to handle, attempt to use or ship the battery.

Damaged or swollen batteries can be unstable and very hot. **DO NOT** touch batteries until they have cooled. In the event of a fire use a Class A, B, or C fire extinguisher. **DO NOT** use water.

- **DO NOT** attach the battery pack to a power supply plug or directly to a car's cigarette lighter.
- **DO NOT** place the battery pack in or near fire or into direct extended exposure to sunlight. When the battery pack becomes hot, the built-in safety equipment is activated, preventing the battery pack from charging further. Heating the battery pack can destroy the safety equipment and cause additional heating, breaking or ignition.
- **DO NOT** leave the battery pack unattended while charging.

  NOTICE: The ambient temperature range over which the battery pack can be discharged is -20°C to 60°C (-4°F to 140°F). Use of the battery pack outside of this temperature range may damage the performance of the battery pack or may reduce its life expectancy.

- **DO NOT** discharge the battery pack using any device except for the ProDSS handheld. When the battery pack is used in other devices it may damage the performance of the battery or reduce its life expectancy. Use of a non-approved device to discharge the battery pack can cause an abnormal current to flow, resulting in the battery pack to become hot, rupture or ignite and cause serious injury.
- **DO NOT** leave the battery pack unattended while discharging.

Battery Disposal

When the battery pack is worn out, insulate the terminals with adhesive tape or similar materials before disposal. Dispose of the battery pack in the manner required by your city, county, state or country. For details on recycling lithium-ion batteries, please contact a government recycling agency, your waste-disposal service or visit reputable online recycling sources such as www.batteryrecycling.com.

This product must not be disposed of with other waste. Instead, it is the user's responsibility to dispose of their waste equipment by handing it over to a designated collection point for the recycling of waste electrical and electronic equipment. The separate collection and recycling of your waste equipment at the time of disposal will help to conserve natural resources and ensure that it is recycled in a manner that protects human health and the environment.

For more information about where you can drop off your waste equipment for recycling, please contact your local city office, or your local waste disposal service. **DO NOT ship batteries to YSI or a YSI authorized service center unless instructed to do otherwise.**

Contact YSI Technical Support at (937) 767-7241 if you have additional questions.

Service information

YSI has authorized service centers throughout the United States and Internationally. For the nearest service center information, please visit ysi.com and click 'Support' or contact YSI Technical Support directly at 800-897-4151 (+1 937-767-7241).
When returning a product for service, include the Product Return form with cleaning certification. The form must be completely filled out for a YSI Service Center to accept the instrument for service. The form may be downloaded from ysi.com.

Technical support

Telephone: 800 897 4151 (USA)

+1 937 767 7241 (Globally) Monday through Friday, 8:00 AM to 5:00 ET

Fax: +1 937 767 9353 (orders)

Email: info@ysi.com

Mail: YSI Incorporated 1725 Brannum Lane Yellow Springs, OH 45387 USA

Internet: ysi.com

Declaration of Conformity

The undersigned hereby declares on behalf of the named manufacturer under our sole responsibility that the listed product conforms to the requirements for the listed European Council Directive(s) and carries the CE mark accordingly.

Manufacturer:	YSI Incorporated 1725 Brannum Lane Yellow Springs, OH 45387 USA
Product Name:	ProDSS
Conforms to the following:	
Directives:	EMC 2004/108/EC RoHS 2011/65/EU WEEE 2012/19/EU
Harmonized Standards:	EN61326-1:2013 (IEC 61326-1:2012) IEC 61000-3-2:2005 +A1:2008+A2:2009 IEC 61000-3-3:2008
Supplementary Information:	All performance met the operation criteria as follows: 1. ESD, IEC 61000-4-2:2008 2. Radiated Immunity, IEC 61000-4-3:2006 +A1:2007+A2:2010 3. Electrical Fast Transient (EFT), IEC 61000-4-4:2004 +A1:2010 4. Immunity to Surge, IEC 61000-4-5:2005 5. Radio Frequency, Continuous Conducted Immunity, IEC61000-4-6:2008 6. IEC 61000-4-8:2009 7. IEC 61000-4-11:2004
Authorized EU Representative	Xylem Analytics UK Ltd Unit 2 Focal Point, Lacerta Court, Works Road Letchworth, Hertfordshire, SG6 1FJ UK

Signed: Lisa M. Abel
Title: Director of Quality

Date: June 3, 2016

The undersigned hereby declares on behalf of the named manufacturer under our sole responsibility that the listed product conforms to the requirements for electrical equipment under US FCC Part 15 and ICES-003 for unintentional radiators.

Manufacturer:	YSI Incorporated 1725 Brannum Lane Yellow Springs, OH 45387 USA
Product Name:	Professional Digital Sampling System Instrument
Model Numbers	
Instrument/Accessory:	ProDSS non-GPS (626870-1) / ProDSS GPS (626870-2)
Probe/Cable Assemblies:	626909-1, 626909-4, 626909-10, 626909-20, 626909-30, 626909-40, 626909-50, 626909-60, 626909-70, 626909-80, 626909-90, 626909-100, 626910-1, 626910-4, 626910-10, 626911-20, 626911-30, 626911-40, 626911-50, 626911-60, 626911-70, 626911-80, 626911-90, 626911-100 627150-1, 627150-4, 627150-10, 627150-20, 627150-30, 627150-40, 627150-50, 627150-60, 627150-70, 627150-80, 627150-90, 627150-100 626250-1, 626250-4, 626250-10, 626250-20, 626250-30, 626250-40, 626250-50, 626250-60, 626250-70, 626250-80, 626250-90, 626250-100 626400, 626401
Sensors:	626900, 626902, 626901, 626903, 626904, 626906, 626905, 626907
Conforms to the following:	
Standards:	• FCC 47 CFR Part 15-2008, Subpart B, Class B, Radio Frequency Devices • ICES-003:2004, Digital Apparatus
Supplementary Information:	Tested using ANSI C63.4-2003 (excluding sections 4.1, 5.2, 5.7, 9, and 14)

Signed: Lisa M. Abel Date: June 3, 2016
Title: Director of Quality

Warranty

The YSI Professional Digital Sampling System (ProDSS) is warranted for three (3) years from date of purchase by the end user against defects in materials and workmanship. The ProDSS bulkhead, sensors and cable (ProDSS 4 port, ProDSS ODO/CT, ProODO, and ProOBOD) assemblies are warranted for two (2) years from date of purchase by the end user against defects in material and workmanship. The ODO Extended Warranty Sensor Cap (627180) for ProDSS ODO/CT cable assemblies is warranted for two (2) years from date of purchase by the end user against defects in material and workmanship. ProDSS pH and pH/ORP sensor modules, optical ODO sensor caps (all but the 627180 cap previously mentioned), and Li-Ion battery pack are warranted for one (1) year from date of purchase by the end user against defects in material and workmanship (6 months for ammonium, nitrate, chloride sensor modules). ProDSS systems (instrument, cables & sensors) are warranted for 1 year (excluding sensor modules) from date of purchase by the end user against defects in material and workmanship when purchased by rental agencies for rental purposes. Within the warranty period, YSI will repair or replace, at its sole discretion, free of charge, any product that YSI determines to be covered by this warranty.

To exercise this warranty, call your local YSI representative, or contact YSI Customer Service in Yellow Springs, Ohio at +1 937 767-7241, 800-897-4151 or visit www.YSI.com (Support tab) for a Product Return Form. Send the product and proof of purchase, transportation prepaid, to the Authorized Service Center selected by YSI. Repair or replacement will be made and the product returned, transportation prepaid. Repaired or replaced products are warranted for the balance of the original warranty period, or at least 90 days from date of repair or replacement.

LIMITATION OF WARRANTY

This Warranty does not apply to any YSI product damage or failure caused by:

1. Failure to install, operate or use the product in accordance with YSI's written instructions;
2. Abuse or misuse of the product;
3. Failure to maintain the product in accordance with YSI's written instructions or standard industry procedure;
4. Any improper repairs to the product;
5. Use by you of defective or improper components or parts in servicing or repairing the product;
6. Modification of the product in any way not expressly authorized by YSI.

THIS WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. YSI's LIABILITY UNDER THIS WARRANTY IS LIMITED TO REPAIR OR REPLACEMENT OF THE PRODUCT, AND THIS SHALL BE YOUR SOLE AND EXCLUSIVE REMEDY FOR ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY. IN NO EVENT SHALL YSI BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY.

Appendix A - DO% calibration values

Calibration Value	Pressure			
D.O. %	**in Hg**	**mmHg**	**kPa**	**mbar**
101%	30.22	767.6	102.34	1023.38
100%	29.92	760.0	101.33	1013.25
99%	29.62	752.4	100.31	1003.12
98%	29.32	744.8	99.30	992.99
97%	29.02	737.2	98.29	982.85
96%	28.72	729.6	97.27	972.72
95%	28.43	722.0	96.26	962.59
94%	28.13	714.4	95.25	952.46
93%	27.83	706.8	94.23	942.32
92%	27.53	699.2	93.22	932.19
91%	27.23	691.6	92.21	922.06
90%	26.93	684.0	91.19	911.93
89%	26.63	676.4	90.18	901.79
88%	26.33	668.8	89.17	891.66
87%	26.03	661.2	88.15	881.53
86%	25.73	653.6	87.14	871.40
85%	25.43	646.0	86.13	861.26
84%	25.13	638.4	85.11	851.13
83%	24.83	630.8	84.10	841.00
82%	24.54	623.2	83.09	830.87
81%	24.24	615.6	82.07	820.73
80%	23.94	608.0	81.06	810.60
79%	23.64	600.4	80.05	800.47
78%	23.34	592.8	79.03	790.34
77%	23.04	585.2	78.02	780.20
76%	22.74	577.6	77.01	770.07
75%	22.44	570.0	75.99	759.94
74%	22.14	562.4	74.98	749.81
73%	21.84	554.8	73.97	739.67
72%	21.54	547.2	72.95	729.54

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Appendix B - oxygen solubility table

Solubility of oxygen in mg/L in water exposed to water-xaturated air at 760 mm Hg pressure.

Salinity = Measure of quantity of dissolved salts in water.

Chlorinity = Measure of chloride content, by mass, of water.

S(0/00) = 1.80655 x Chlorinity (0/00)

Temp °C	Chlorinity : 0 Salinity: 0	5.0 ppt 9.0 ppt	10.0 ppt 18.1 ppt	15.0 ppt 27.1 ppt	20.0 ppt 36.1 ppt	25.0 ppt 45.2 ppt
0.0	14.62	13.73	12.89	12.10	11.36	10.66
1.0	14.22	13.36	12.55	11.78	11.07	10.39
2.0	13.83	13.00	12.22	11.48	10.79	10.14
3.0	13.46	12.66	11.91	11.20	10.53	9.90
4.0	13.11	12.34	11.61	10.92	10.27	9.66
5.0	12.77	12.02	11.32	10.66	10.03	9.44
6.0	12.45	11.73	11.05	10.40	9.80	9.23
7.0	12.14	11.44	10.78	10.16	9.58	9.02
8.0	11.84	11.17	10.53	9.93	9.36	8.83
9.0	11.56	10.91	10.29	9.71	9.16	8.64
10.0	11.29	10.66	10.06	9.49	8.96	8.45
11.0	11.03	10.42	9.84	9.29	8.77	8.28
12.0	10.78	10.18	9.62	9.09	8.59	8.11
13.0	10.54	9.96	9.42	8.90	8.41	7.95
14.0	10.31	9.75	9.22	8.72	8.24	7.79
15.0	10.08	9.54	9.03	8.54	8.08	7.64
16.0	9.87	9.34	8.84	8.37	7.92	7.50
17.0	9.67	9.15	8.67	8.21	7.77	7.36
18.0	9.47	8.97	8.50	8.05	7.62	7.22
19.0	9.28	8.79	8.33	7.90	7.48	7.09
20.0	9.09	8.62	8.17	7.75	7.35	6.96
21.0	8.92	8.46	8.02	7.61	7.21	6.84
22.0	8.74	8.30	7.87	7.47	7.09	6.72
23.0	8.58	8.14	7.73	7.34	6.96	6.61
24.0	8.42	7.99	7.59	7.21	6.84	6.50
25.0	8.26	7.85	7.46	7.08	6.72	6.39
26.0	8.11	7.71	7.33	6.96	6.62	6.28
27.0	7.97	7.58	7.20	6.85	6.51	6.18
28.0	7.83	7.44	7.08	6.73	6.40	6.09
29.0	7.69	7.32	6.93	6.62	6.30	5.99
30.0	7.56	7.19	6.85	6.51	6.20	5.90
31.0	7.43	7.07	6.73	6.41	6.10	5.81
32.0	7.31	6.96	6.62	6.31	6.01	5.72

Appendix B - oxygen solubility table

Temp °C	Chlorinity : 0 Salinity: 0	5.0 ppt 9.0 ppt	10.0 ppt 18.1 ppt	15.0 ppt 27.1 ppt	20.0 ppt 36.1 ppt	25.0 ppt 45.2 ppt
33.0	7.18	6.84	6.52	6.21	5.91	5.63
34.0	7.07	6.73	6.42	6.11	5.82	5.55
35.0	6.95	6.62	6.31	6.02	5.73	5.46
36.0	6.84	6.52	6.22	5.93	5.65	5.38
37.0	6.73	6.42	6.12	5.84	5.56	5.31
38.0	6.62	6.32	6.03	5.75	5.48	5.23
39.0	6.52	6.22	5.98	5.66	5.40	5.15
40.0	6.41	6.12	5.84	5.58	5.32	5.08
41.0	6.31	6.03	5.75	5.49	5.24	5.01
42.0	6.21	5.93	5.67	5.41	5.17	4.93
43.0	6.12	5.84	5.58	5.33	5.09	4.86
44.0	6.02	5.75	5.50	5.25	5.02	4.79
45.0	5.93	5.67	5.41	5.17	4.94	4.72

Item #626973-01REF
Rev D
September, 2016