28372-88

HEALTHY WATER

HEALTHY PEOPLE

Advanced Water Quality Testing Kit

Procedures Manual

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Using the Healthy Water, Healthy People Testing Kit Procedure Manual

The Healthy Water, Healthy People Advanced Testing Kit Procedure Manual provides instructions for conducting the water quality tests in this kit. The procedures are given in narrative and illustrated formats to accommodate diverse learning styles. Refer to the Table of Contents as a guide to help locate the procedures for your chosen parameter.

Healthy Water, Healthy People and Hach Company want to ensure that you have an enjoyable water quality testing experience, so expert technicians and educators are available to help you.

Ordering Replacement Chemicals and Equipment?

Contact Healthy Water, Healthy People toll-free at 1-866-337-5486, or place an order online at www.healthywater.org, or fax (406) 994-1919. Order by part number listed next to the item.

Questions about what Chemical Refills and Equipment to Order?

The Chemical Refills and Replacement Parts are located at the end of the procedures for each parameter.

Need Higher-Level or Additional Equipment?

Check out the Additional Water Quality Testing Materials Sheet in this manual for additional items that will enhance your testing methods.

Contact Us:

Healthy Water, Healthy People

1-866-377-5486

Fax: 406-994-1919

healthywater@montana.edu

www.healthywater.org

P. O. Box 170575

201 Culbertson Hall

Montana State University

Bozeman, MT 59717-0575

Discard of Chemical Waste

In 1976, Congress passed the Resource Conservation and Recovery Act (RCRA) which directed the U.S. Environmental Protection Agency (EPA) to develop and implement a program to protect human health and the environment from improper hazardous waste management practices. The RCRA program is designed to control the management of hazardous waste from its generation to its ultimate disposal – from “cradle-to-grave”.

The provisions of RCRA hold the generator of hazardous waste responsible for proper collection, storage and disposal from the point of generation. Due to the complexity of the regulations, the following resources may be used to ensure that your operations are conducted in compliance with RCRA:

- Check with your Environmental Compliance Officer.
- Check with your Local, State, and/or Federal Hazardous Waste Division.
- Contact a Treatment Storage and Disposal Facility.
- Contact an Environmental Consulting Firm.

What is a Material Safety Data Sheet?

The United States Occupational Safety and Health Administration (OSHA) requires all manufacturers to issue Material Safety Data Sheets (also known as MSDSs) with the first shipment of any hazardous chemical product. Hach Company issues MSDSs for all chemical shipments, from toxic mercuric acid to substances as benign as sodium chloride (salt). The purpose of the MSDS is to relay important information about the nature of the chemical – such as its flammability, toxicity, the need for protective equipment, and spill or clean-up requirements – to ensure the safety of any potential user of that chemical.

At first glance, the MSDS can seem like an intimidating piece of information. The information is not difficult to understand when broken into its component parts. It is a very important document to read and follow.

When you receive a reagent shipment from Hach, we recommend that you:

1. Read the MSDS carefully.
2. Discuss the cautions specified on the MSDS with your students.
3. Utilize necessary protective equipment.
4. Dispose of the reagent properly (see the MSDS for disposal information).
5. Keep file copies of MSDSs in a central location accessible to all.

What to look for in an MSDS (Q and A)

1. *Where do I find an overall product summary and emergency phone numbers?*

   The chemical product and company identification section provides an overall product summary, including manufacturer address, MSDS date, emergency phone numbers, and a brief description of any hazard.

2. *What is this product made of?*

   The composition and ingredients section explains the following information about each component of the product:

   - PCT—Percent by weight of this component
   - CAS—Number Chemical Abstract Services or CAS registry number
   - SARA—Superfund and Reauthorization Act -- better known as the "Community Right to Know Law". If an MSDS indicates that a component is listed in SARA 313 and more than the specified amount is used, it must be reported to the USEPA every year.
   - TLV—Threshold Limit Value is the maximum airborne concentration allowable for an 8-hour exposure, as recommended by the American Conference of Governmental Industrial Hygienists (ACGIH).
   - PEL—Permissible Exposure Limit is the maximum airborne concentration allowable for an 8-hour exposure, as regulated by the Occupational Safety and Health Administration (OSHA).
   - HAZARD—Any physical or health hazards posed by this chemical are explained here.

3. *What are the hazards of using this product?*

   The hazards identification summary starts with an emergency overview, plus ratings of the product from the Hazardous Materials Information System and the National Fire Protection Association (NFPA). This section also outlines the potential harmful effects of this product.

4. *How do I administer first aid in the event of exposure?*

   The First Aid section explains the medical attention required in the event of an exposure. Be sure to read this section carefully!

5. *What do I do in the event of a fire?*

   The fire fighting measures section lists all of the flammability concerns, what media to use to extinguish a fire, and what safety precautions to take, should you be put in a position to fight a fire.

6. *What happens if it spills?*

   The accidental release measures section explains the personnel who are qualified to respond to an emergency involving hazardous substances and outlines containment techniques, clean-up techniques, and any necessary evacuation procedures.

7. *What are the handling and storage requirements?*

   The handling/storage section provides general guidelines for safe handling of the product and lists all storage requirements.

8. *What protective measures do I need to take in the work environment?*

   The exposure controls/protective equipment section outlines safe laboratory practice (e.g. eyewash station nearby) and any necessary protective gear, such as eye, skin, and inhalation protection.

9. *What does it look/act like?*

   The physical/chemical properties section explains information such as the physical state, color, odor, solubility, boiling point, melting point, specific gravity, pH, vapor density, evaporation rate, corrosivity, stability, and storage precautions.

10. *How stable is this product and what storage precautions need to be taken?*

    The stability/reactivity section explains storage conditions, including moisture and temperature requirements and compatibility with substances stored nearby.

11. *How did this product perform in clinical trials?*

    The toxicological information section summarizes data gathered from research with animals, including lethal dosages and concentrations, effects on reproduction, skin toxicity data, and mutation data.

12. *What is the ecological impact of this product?*

    The ecological information section assesses the material's environmental impact on aquatic and terrestrial plants and animals and the potential of the material to persist in the environment.

13. *How do I safely (and legally) dispose of this product?*

    The disposal considerations section outlines any dilution guidelines, container information, and national environmental regulations that may or may not be reinforced by regional or local entities.

14. *How is this product regulated?*

The regulatory information section outlines all known regulatory guidelines spelled out by OSHA, SARA Title III, the Clean Water Act, RCRA, and any known state regulations.

15. *What is the intended use of this product and how was this MSDS compiled?*

The other information section states the product's intended use and lists all reference materials used to write the MSDS.

16. *What are the shipping requirements?*

The transportation information section explains proper shipping name, hazard class, ID number, and packing group for all chemicals regulated by D.O.T., I.C.A.O., and I.M.O.

Comparison of International Drinking Water Guidelines

Table 1 Comparison of International Drinking Water and FDA Bottled Water Guidelines[1]

Parameter	USEPA[2] Maximum Contaminant Level (MCL)	Canada[3] Maximum Acceptable Concentration	EEC[4] Maximum Admissible Concentration	Japan[5] Maximum Admissible Concentration	WHO[6] Guideline	Bottled Water U.S. Federal Drug Administration Level
Aluminum	0.05–0.2 mg/L[7]		0.2 mg/L	0.2 mg/L	0.2 mg/L	
Ammonium			0.5 mg/L	No standard	1.5 mg/L	
Antimony	0.006 mg/L		0.01 mg/L	0.002 mg/L[8]	0.005 mg/L	
Arsenic	0.05 mg/L	0.025 mg/L	0.05 mg/L	0.01 mg/L	0.01 mg/L	0.05 mg/L
Barium	2.0 mg/L	1.0 mg/L	No standard	No standard	0.7 mg/L	2.0 mg/L
Boron		5.0 mg/L	1.0 mg/L	0.2 mg/L[8]	0.3 mg/L	
Cadmium	0.005 mg/L	0.005 mg/L	0.005 mg/L	0.01 mg/L	0.003 mg/L	0.005 mg/L
Chloride	250 mg/L[7]	250 mg/L	250 mg/L	200 mg/L	250 mg/L	
Chromium	0.1 mg/L	0.05 mg/L	0.05 mg/L	0.05 mg/L	0.05 mg/L	0.1 mg/L
Coliforms, total Organisms/100 mL	≤ 5 %positive samples/month	0	0 or MPN ≤ 1	0	0	≤ 1 MF
Coliforms (*E. coli*) Organisms/100 mL	0	0	0	0	0	
Color	15 cu[7]	15 cu	20 mg Pt-Co/L	5 cu	15 cu	< 15 cu
Copper	1.3 mg/L[7]	1.0 mg/L	2.0 mg/L	1.0 mg/L	1–2 mg/L	1.0 mg/L
Cyanides	0.2 mg/L	0.2 mg/L	0.05 mg/L	0.01 mg/L	0.07 mg/L	
Fluoride	2.0-4.0 mg/L[7]	1.5 mg/L	0.7-1.5 mg/L	0.8 mg/L	1.5 mg/L	
Hardness			50 mg/L	300 mg/L		

Table 1 Comparison of International Drinking Water and FDA Bottled Water Guidelines[1] (Continued)

Parameter	USEPA[2] Maximum Contaminant Level (MCL)	Canada[3] Maximum Acceptable Concentration	EEC[4] Maximum Admissible Concentration	Japan[5] Maximum Admissible Concentration	WHO[6] Guideline	Bottled Water U.S. Federal Drug Administration Level
Iron	0.3 mg/L[7]	0.3 mg/L	0.2 mg/L	0.3 mg/L	0.3 mg/L	
Lead	0.015 mg/L	0.01 mg/L	0.01 mg/L	0.05 mg/L	0.01 mg/L	0.005 mg/L
Manganese	0.05 mg/L	0.05 mg/L	0.05 mg/L	0.01–0.05 mg/L	0.1–0.5 mg/L	
Mercury	0.002 mg/L	0.001 mg/L	0.001 mg/L	0.0005 mg/L	0.001 mg/L	0.002 mg/L
Molybdenum				0.07 mg/L	0.07 mg/L	
Nickel	0.1 mg/L		0.02 mg/L	0.01 mg/L[8]	0.02 mg/L	
Nitrate/Nitrite, total	10.0 mg/L as N			10.0 mg/L as N		10 mg/L as N
Nitrates	10.0 mg/L as N	10.0 mg/L as N	50 mg/L	10 mg/L as N	50 mg/L as NO_3^-	
Nitrites	1 mg/L as N	3.2 mg/L	0.1 mg/L	10 mg/L	3 mg/L as NO_2^-	1 mg/L as N
Odor	3 TON[9]		2 dilution no. @ 12 °C; 3 dilution no. @ 25 °C.	3 TON		
pH	6.5–8.5	6.5–8.5	6.5–9.5	5.8–8.6	6.5–8.5	
Phosphorus			5 mg/L	No Standard		
Phenols		0.002 mg/L	0.5 µg/L C_6H_5OH	0.005 mg/L		
Potassium			12 mg/L	No Standard		
Selenium	0.05 mg/L	0.01 mg/L	0.01 mg/L	0.01 mg/L	0.01 mg/L	0.05 mg/L
Silica Dioxide			10 mg/L	No Standard		
Silver	0.1 mg/L[7]	0.05 mg/L	0.01 mg/L	No standard	No standard	

Table 1 Comparison of International Drinking Water and FDA Bottled Water Guidelines[1] (Continued)

Parameter	USEPA[2] Maximum Contaminant Level (MCL)	Canada[3] Maximum Acceptable Concentration	EEC[4] Maximum Admissible Concentration	Japan[5] Maximum Admissible Concentration	WHO[6] Guideline	Bottled Water U.S. Federal Drug Administration Level
Solids, total dissolved	500 mg/L[7]	500 mg/L	No standard	500 mg/L	1000 mg/L	
Sodium			75-150 mg/L	200 mg/L	200 mg/L	
Sulfate	250 mg/L[7]	500 mg/L	250 mg/L	No Standard	250 mg/L	
Turbidity	0.3–1 NTU	1 NTU	4 JTU	1–2 units	5 NTU	< 5 NTU
Zinc	5 mg/L[7]	5.0 mg/L	No Standard	1.0 mg/L	3.0 mg/L	

1 To our knowledge, data in this table were accurate and current at the publication date. Contact the regulatory agency in your area for the most current information.

2 United States Environmental Protection Agency.

3 These limits are established by Health Canada.

4 In the EEC (European Economic Community), these limits are set by the European Committee for Environmental Legislation.

5 In Japan, these limits are established by the Ministry of Health and Welfare.

6 World Health Organization.

7 U.S. Secondary MCL.

8 Identified as a parameter to be regulated in the future.

9 Threshold Odor Number.

Replacement Standards and Buffers

Standards	Units	Catalog Number	Advanced Kit	Rivers, Streams, Ponds, and Lakes Kit	Classroom Drinking Water Kit	Drinking Water (Urban) Kit	Watershed Kit
pH Buffers (Singlets)							
pH 7.00 and pH 10.01	10 ea/pkg	27698-00	x	x	x	x	x
pH 4.01 and pH 7.00	10 ea/pkg	27699-20	x	x	x	x	x
pH 4.01	20/pkg	27700-20	x	x	x	x	x
pH 7.00	20/pkg	27701-20	x	x	x	x	x
pH 10.01	20/pkg	27702-20	x	x	x	x	x
Conductivity Standards							
180 ±10 us/cm, 90 ±5 mg/L TDS	100 mL	23075-20	x	x			
1000 ±10 us/cm, 500 ±5 mg/L TDS	100 mL	14400-42	x	x			
1991 ±10 us/cm, 995 ±5 mg/L TDS	100 mL	2105-42	x	x			
Single Parameter Standards							
Alkalinity, 0.500 N	10-mL Voluette® ampules	14278-10	x	x	x		x
BOD, 300 mg/L, Glucose plus Glutamic Acid	10-mL Voluette® ampules	14865-10	x	x			
Chlorine, Free and Total, 23–30 mg/L*	2-mL ampules	26300-20	x		x	x	

Standards	Units	Catalog Number	Advanced Kit	Rivers, Streams, Ponds, and Lakes Kit	Classroom Drinking Water Kit	Drinking Water (Urban) Kit	Watershed Kit
Single Parameter Standards							
Copper, 4 mg/L[1]	2-mL ampules	26057-20				x	
Hardness, 1000 mg/L	500 mL	22403-49	x		x		
Iron, 1 mg/L	500 mL	139-49	x		x		
Manganese, 10 mg/L	2-mL ampules	26058-20	x				
Nitrate -N, 10 mg/L	500 mL	307-49	x		x	x	x
Nitrate-N, 5 mg/L	10-mL Voluette® ampules	25578-10		x			
Nitrate-N, 1 mg/L	500 mL	2046-49	x	x			
Phosphate, 1 mg/L	500 mL	2569-49	x	x			
Phosphate, 15 mg/L	100 mL	14243-42		x			x
Sulfate, 50 mg/L	500 mL	2578-49	x	x			

* Some standards are not available in levels within the test range and will require dilution.

Additional Water Quality Testing Materials Available Through *Healthy Water, Healthy People*

Item	Part #	Cost $*
Digital Titrator	16900-01	105.00
Turbidity Tube	63010-00	25.00
Pocket Turbidimeter	46500-00	837.00
Chlorine Pocket Colorimeter™†	46700-00	335.30
LeadTrak®‡ Pocket Colorimeter™	46700-21	443.45
Nitrate Pocket Colorimeter™	46700-02	335.30
Phosphate Pocket Colorimeter™	46700-06	335.30
sens*ion*™** 156 pH, DO, Conductivity probes	54650-15	1,350.00
sens*ion* 378 ph, Conductivity, TDS, Salinity, Temperature, DO probes	54750-21	1,575.00
Arsenic Test Kit	28000-00	95.00

Microbiology

Paddle Testers	26109-10	29.15
BART™†† testers, combo pack: IRB (3), SRB (3), SLYM (3)	24348-09	80.00
MEL/850 Potable Water Lab	26888-00	2,453.90
MEL/MPN Total Coliform and E. Coli Lab	25698-00	1,537.00
Portable Incubator, with rechargeable 12 V dc battery	25699-00	850.00
Battery Charger for Portable Incubator, 115 V ac	25803-00	163.00

* Prices subject to change without notice.

† Pocket Colorimeter is a trademark of Hach Company.

‡ LeadTrak is a registered trademark of Hach Company.

** sens*ion* is a trademark of Hach Company.

†† BART is a trademark of Droycon Bioconcepts Inc.

General Apparatus

Glasses, Safety Standard (Clear Plastic)	18421-00	4.65
Glove, Latex, Powder-Free/100 pk (size Small)	25904-01	24.75
Glove, Latex, Powder-Free/100 pk (size Medium)	25904-02	24.75
Notebook, Data, Pocket (64 waterproof pages)	20917-00	16.50
Stopwatch, Water Resistant	26302-00	61.80

General Apparatus, continued

Tech Board (clipboard including clock, stopwatch, calculator)	27473-00	46.35
Tech Board Plus (same as above with an advanced scientific calculator)	27991-00	61.80
Timer, Mini (up to 99 minutes w/1 second intervals)	27664-00	37.10
Thermometer, Pocket/non mercury in protective case. Dual Range (-10 to 110°C/ 0 to 220°F)	26764-00	22.70
TenSette®* Pipet, 0.1 to 1.0 mL in 0.1 mL increments	19700-01	165.00
Pipet Tips for19700-01, pk/50	21856-96	7.50
TenSette Pipet, 1.0 to 10.0 mL in 1.0 mL increments	19700-10	165.00
Pipet Tips for 19700-10, pk/50	21997-96	6.70
Voluette®† Ampule breaker (for Standards), 10-mL	21968-00	10.75
Voluette Ampule breaker (for Standards), 2-mL	25640-00	10.60

pH, Conductivity, and TDS Testers

pH Testr™ 2‡, Oakton, Waterproof, –0.1 to 15 pH, double junction	27993-00	81.40
pH Testr 2, Oakton, Waterproof, –0.1 to 15 pH	27235-00	81.40
ECTestr, Low, Oakton Conductivity Tester, 0-1990 microS, auto-temperature compensation, push-button calibration, waterproof replaceable sensor	28455-00	59.95

* TenSette is a registered trademark of Hach Company.
† Voluette is a registered trademark of Hach Company.
‡ pHTestr 2 is a trademark of Oakton Instruments.

pH, Conductivity, and TDS Testers, continued

ECTestr, High, Oakton Conductivity Tester, 0-19.90 mS, auto-temperature compensation, push-button calibration, waterproof replaceable sensor ..... 28456-00 ..... 59.95

TDSTestr, Low, Oakton, 0 to 1990 ppm, auto-temperature compensation, push-button calibration, waterproof replaceable sensor ..... 28457-00 ..... 59.95

TDSTestr, High, Oakton, 1 to 10 ppt, auto-temperature compensation, push-button calibration, waterproof replaceable sensor ..... 28458-00 ..... 59.95

Replacement Sensor for EC and TDS Testers ..... 28459-00 ..... 37.00

Standards and Buffers (see Replacement Standards and Buffers on page 12)

Refer to the Water Analysis Handbook for specific instructions on how to use standards.

Water Analysis Handbook ..... 23196-00 ..... no charge

DR/890 Procedures Manual ..... 48470-22 ..... 40.00

To Order:

Contact Healthy Water, Healthy People:
toll free 1-866-337-5486
email healthywater@montana.edu
website www.healthywater.org
fax (406) 994-1919

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201 Culbertson Hall
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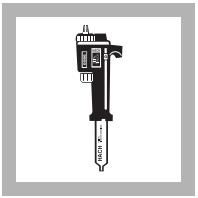
Hints for Performing Chemical Tests

- Review the Material Safety Data Sheet (MSDS).
- Read all of the instructions for each test before you perform the procedures.
- Practice! The more familiar you are with the tests, the easier they will be to perform, and the more accurate your results will be.
- Wear protective equipment indicated on the MSDS (i.e. protective gloves and safety goggles).
- When you are finished, wash your hands.
- Obtain your water sample from the stream mainstream flow (usually the middle). Take the sample 3-5 inches under the surface.
- Rinse collection tubes or bottles with sample water before collecting the sample.
- Rinse testing tubes and bottles with distilled water after completing each test.
- Clean lab ware with non-abrasive detergents or a solvent such as isopropyl alcohol. Use a soft cloth for wiping or drying. Do not use paper towels or tissue on plastic tubes as they may scratch.

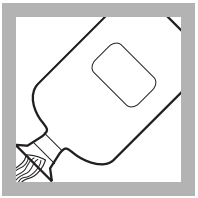
Biological Oxygen Demand (BOD)

WARNING: Handling chemical samples, standards, and reagents can be dangerous. Review the Material Safety Data Sheets before handling any chemicals.

1. Collect one sample in a 300-mL BOD bottle, filling it completely and preventing air from being trapped in the bottle. Allow the sample to overflow the bottle for 2-3 minutes to ensure air bubbles are not trapped or stopper the bottle while both are underwater.

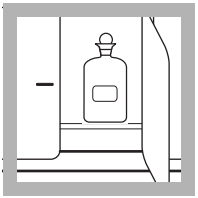


2. Determine the dissolved oxygen concentration with the digital titrator method on page 20 for a 300-mL bottle. Record this value as $DO_{initial}$.

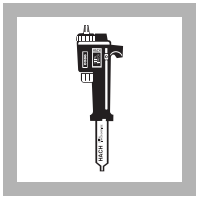


3. Discard the titrated sample into a waste container. Clean the sample bottle thoroughly before proceeding to step 4.

4. Collect another sample in the 300-mL BOD bottle, filling it completely and preventing air from being trapped in the bottle. Allow the sample to overflow the bottle for 2-3 minutes to ensure air bubbles are not trapped or stopper the bottle while both are underwater.



5. Store the second sample in a warm, dark location for 5 days. If possible, wrap the bottle in aluminum foil and place it on a hot water heater or in a similar warm location.



6. After 5 days, determine the dissolved oxygen concentration of the second sample with the digital titrator method on page 20 for a 300-mL bottle. Record this value as DO_{final}.

7. Calculate the 5-Day BOD of the sample with the following equation:

$$BOD = DO_{initial} - DO_{final}$$

Note: *This procedure is a modification of the Standard Methods 5-Day BOD Test (Standard Methods for the Examination of Water and Wastewater, 5210). The results of this modified procedure may not be equivalent to results from the Standard Methods 5-Day BOD Test and may not be compared directly. If BOD results from this test are recorded, note that the results were obtained using the modified procedure.*

Note: *If dissolved oxygen is not detected after the 5-day incubation period, the BOD of the sample cannot be determined. In this case, report BOD as $DO_{initial}$ and note that no DO was detectable after incubation. Incubation in the Standard Methods BOD test is performed at 20 ±1°C*

Oxygen, Dissolved (1 to >10 mg/L as DO), using a 300-mL BOD Bottle

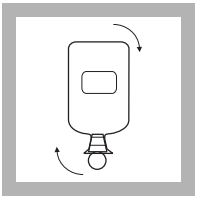
Note: *Refer to the Digital Titrator manual included with this kit for detailed information about the Digital Titrator.*

1. Collect one sample in a 300-mL BOD bottle, filling it completely and preventing air from being trapped in the bottle. Allow the sample to overflow the bottle for 2-3 minutes to ensure air bubbles are not trapped or stopper the bottle while both are underwater.

2. Add the contents of one Manganous Sulfate Powder Pillow and one Alkaline Iodide-Azide Reagent Powder Pillow to the 300-mL BOD bottle.

3. Immediately insert the stopper so air is not trapped in the bottle. Invert several times to mix.

Note: *A flocculent precipitate will form. It will be orange-brown if oxygen is present or white if oxygen is absent.*

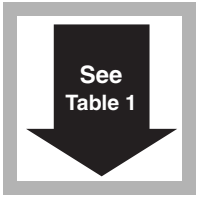


4. Wait until the floc in the solution has settled (at least 5 minutes, in salt water, the floc will settle more slowly.) Again invert the bottle several times and wait until the floc has settled.

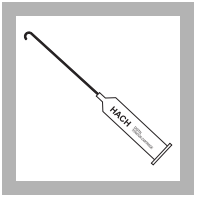
Note: *Waiting until floc has settled twice assures complete reaction of the sample and reagents.*

5. Remove the stopper and add the contents of one Sulfamic Acid Powder Pillow. Replace the stopper without trapping air in the bottle and invert several times.

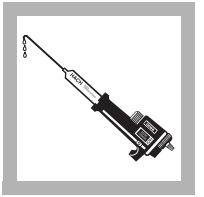
Note: *The floc will dissolve and leave a yellow color if oxygen is present.*



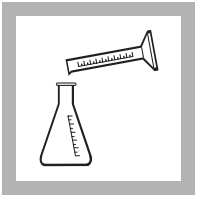
6. Select a sample volume and Sodium Thiosulfate Titration Cartridge corresponding to the expected dissolved oxygen (D.O.) concentration from Table 1.



7. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body.

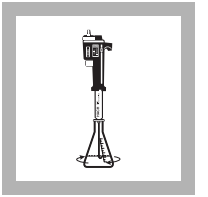


8. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

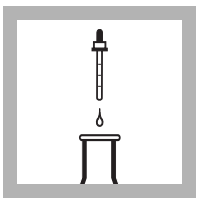


9. Use a graduated cylinder to measure the sample volume from Table 1. Transfer the sample into a 250-mL Erlenmeyer flask.

Note: *Rinse the cylinder and flask with prepared sample before use.*



10. Place the delivery tube tip into the solution and swirl the flask while titrating with sodium thiosulfate to a pale yellow color. Do not reset the titrator digits.



11. Add two 1-mL droppers of Starch Indicator Solution and swirl to mix.

A dark blue color will develop.

12. Continue the titration to a colorless end point. Record the number of digits required.

13. Calculate:

Digits Required x Digit Multiplier = mg/L Dissolved Oxygen

Table 1 Sample Volumes

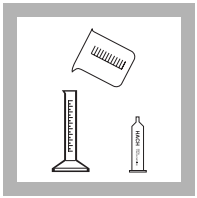
Range (mg/L D.O.)	Sample Volume (mL)	Titration Cartridge (N $Na_2S_2O_3$)	Catalog Number	Digit Multiplier
1-5	200	0.200	22675-01	0.01
2-10	100	0.200	22675-01	0.02
>10	200	2.000	14401-01	0.1

Replacement Parts

Alkaline Iodide-Azide Reagent Powder Pillows.....50/pkg..... 1072-66
BOD bottle, with stopper, 300-mL.....each..... 621-00
Cylinder, graduated measuring, 100-mL.....each..... 1081-42
Delivery Tube for Digital Titrator .....each..... 17205-00
Digital Titrator Assembly.....each.....16900-01
Erlenmeyer Flask, 250-mL .....each.....505-46
Manganous Sulfate Reagent Powder Pillows .....50/pkg..... 1071-66
Sodium Thiosulfate Titration Cartridge, 0.200 N.....each.....22675-01
Sulfamic Acid Reagent Powder Pillows .....50/pkg.....20762-66

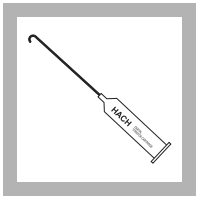
Alkalinity (10 to 4000 mg/L as $CaCO_3$)

- Refer to the Digital Titrator manual included with this kit for detailed information about the Digital Titrator.
- Four drops of Phenolphthalein Indicator Solution may be substituted for the Phenolphthalein Indicator Powder Pillow.
- Rinse glassware with sample before use.

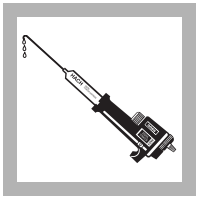


1. Select the sample volume and Sulfuric Acid (H_2SO_4) Titration Cartridge corresponding to the expected alkalinity concentration as mg/L calcium carbonate ($CaCO_3$) from Table 1.

Note: *Alkalinity test strips (Cat. No. 27448-50) can be used to estimate the alkalinity range.*

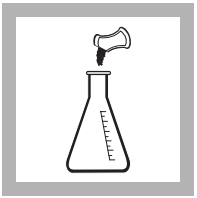


2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip with a clean cloth.

4. Use a graduated cylinder or pipet to measure the sample volume from Table 1. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

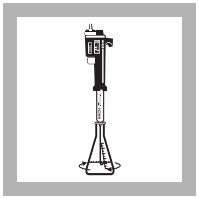


5. Add the contents of one Phenolphthalein Indicator Powder Pillow and swirl to mix.

Note: *A solution of one pH 8.3 Buffer Powder Pillow and one Phenolphthalein Powder Pillow in 50 mL of deionized water is recommended as a comparison for determining the proper end point color.*

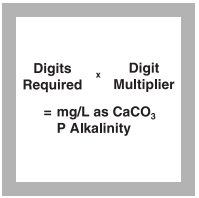
Table 1 Sample Volumes

Range (mg/L as $CaCO_3$)	Sample Volume (mL)	Titration Cartridge (N Sulfuric Acid)	Catalog Number	Digit Multiplier
10-40	100	0.1600	14388-01	0.1
40-160	25	0.1600	14388-01	0.4
100-400	100	1.600	14389-01	1.0
200-800	50	1.600	14389-01	2.0
500-2000	20	1.600	14389-01	5.0
1000-4000	10	1.600	14389-01	10.0



6. If the solution turns pink, titrate to a colorless end point. Place the delivery tube tip into the solution and swirl the flask while titrating with sulfuric acid. Record the number of digits required.

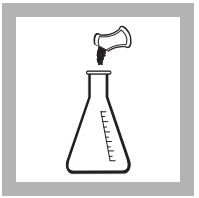
Note: *If the solution is colorless before titrating with sulfuric acid, the Phenolphthalein (P) Alkalinity is zero; proceed with step 8.*



7. Calculate:

Digits Required x Digit Multiplier = mg/L $CaCO_3$ Phenolpthalein Alkalinity

Note: *The digit multiplier is found in Table 1 on page 23.*



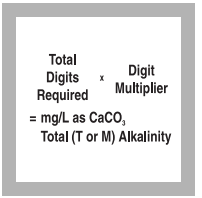
8. Add the contents of one Bromcresol Green-Methyl Red Indicator Powder Pillow to the flask and swirl to mix.

Note: *Four drops of Methyl Purple Indicator Solution may be substituted for the Bromcresol Green-Methyl Red Indicator Powder Pillow. Titrate from green to a gray end point (pH 5.1).*

Note: *Four drops of Bromcresol Green-Methyl Red Indicator Solution may be substituted for the Bromcresol Green-Methyl Red Indicator Powder Pillow.*

9. Continue the titration with sulfuric acid to a light pink color (pH 4.5). If using a pH meter, titrate to the pH endpoint appropriate to sample composition (Table 2). Record the number of digits required.

Note: *A solution of one Bromcresol Green-Methyl Red Powder Pillow and one pillow of the appropriate pH buffer in 50 mL of deionized water is recommended as a comparison for judging the proper end point color. If the pH 3.7 end point is used, use a Bromphenol Blue Powder Pillow instead of a Bromcresol Green-Methyl Red and titrate to a green end point.*



10. Calculate:

Total Digits Required x Digit Multiplier = mg/L as $CaCO_3$ Total (T or M) Alkalinity

Note: *Carbonate, bicarbonate and hydroxide concentrations may be expressed individually using the relationships shown in Table 3.*

Note: *meq/L Alkalinity = mg/L as $CaCO_3$ ÷ 50.*

Table 2 pH Endpoint for Sample Concentration

Sample Composition	End Point
Alkalinity about 30 mg/L	pH 4.9
Alkalinity about 150 mg/L	pH 4.6
Alkalinity about 500 mg/L	pH 4.3
Silicates or Phosphates present	pH 4.5
Industrial waste or complex system	pH 4.5

Sampling and Storage

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Avoid excessive agitation or prolonged exposure to air. Samples should be analyzed as soon as possible after collection but can be stored at least 24 hours by cooling to 4 °C (39 ° F) or below. Warm to room temperature before analyzing.

Alkalinity Relationship Table

Total alkalinity primarily includes hydroxide, carbonate and bicarbonate alkalinities. The concentration of these alkalinities in a sample may be determined when the phenolphthalein and total alkalinities are known (see Table 3).

Table 3

Row	Result of Titration Carbonate	Hydroxide Alkalinity is equal to:	Carbonate Alkalinity is equal to:	Bicarbonate Alkalinity is equal to:
1	Phenolphthalein Alkalinity = 0	0	0	Total Alkalinity
2	Phenolphthalein Alkalinity equal to Total Alkalinity	Total Alkalinity	0	0
3	Phenolphthalein Alkalinity less than one half of Total Alkalinity	0	Two times the Phenolphthalein Alkalinity	Total Alkalinity minus two times Phenolphthalein Alkalinity
4	Phenolphthalein Alkalinity equal to one half of Total Alkalinity	0	Total Alkalinity	0
5	Phenolphthalein Alkalinity greater than one half of Total Alkalinity	Two times the Phenolphthalein minus Total Alkalinity	Two times the difference between Total and Phenolphthalein Alkalinity	0

To use Table 3, follow these steps:

1. Does the phenolphthalein alkalinity equal zero? If yes, use Row 1.
2. Does the phenolphthalein alkalinity equal total alkalinity? If yes, use Row 2.
3. Multiply the phenolphthalein alkalinity by 2.
4. Select Row 3, 4, or 5 based on comparing the result of step c with the total alkalinity.
5. Perform the required calculations in the appropriate row, if any.
6. Check your results. The sum of the three alkalinity types will equal the total alkalinity.

For example:

A sample has 170 mg/L as $CaCO_3$ phenolphthalein alkalinity and 250 mg/L as $CaCO_3$ total alkalinity. What is the concentration of hydroxide, carbonate and bicarbonate alkalinities?

The phenolphthalein alkalinity does not equal 0 (it is 170 mg/L), see step 1.

The phenolphthalein alkalinity does not equal total alkalinity (170 mg/L vs. 250 mg/L), see step 2.

The phenolphthalein alkalinity multiplied by 2 = 340 mg/L, see step 3.

Because 340 mg/L is greater than 250 mg/L, select Row 5, see step 4.

The hydroxide alkalinity is equal to: (see step 5).

340 – 250 = 90 mg/L hydroxide alkalinity

The carbonate alkalinity is equal to:

250 – 170 = 80

80 x 2 = 160 mg/L carbonate alkalinity

The bicarbonate alkalinity equals 0 mg/L. Check: (see step 6).

90 mg/L hydroxide alkalinity + 160 mg/L carbonate alkalinity +
0 mg/L bicarbonate alkalinity = 250 mg/L

The above answer is correct; the sum of each type equals the total alkalinity.

Accuracy Check

Standard Additions Method

Perform this accuracy check when interferences are suspected or to verify analytical technique.

1. Snap the neck off an Alkalinity Standard Solution Voluette® Ampule, 0.500 N.
2. Use a TenSette® Pipet to add 0.1 mL of standard to the sample titrated in step 6 or 9.
3. Resume titration back to the same end point. Record the number of digits needed.
4. Repeat, using two more additions of 0.1 mL. Titrate to the end point after each addition.
5. Each 0.1 mL addition of standard should require 25 additional digits of 1.600 N titrant or 250 digits of 0.1600 N titrant.

Interferences

- Highly colored or turbid samples may mask the color change at the end point. Use a pH meter for these samples to indicate when an endpoint has been reached.
- Chlorine may interfere with the indicators. Add one drop of 0.1 N Sodium Thiosulfate, prior to analysis, to eliminate this interference.

Summary of Method

The sample is titrated with sulfuric acid to a colorimetric endpoint corresponding to a specific pH. Phenolphthalein alkalinity is determined by titration to a pH of 8.3, as evidenced by the color change of phenolphthalein indicator, and indicates the total hydroxide and one half the carbonate present. M (methyl orange) or T (total) alkalinity is determined by titration to a pH between 3.7 and 5.1, and includes all carbonate, bicarbonate and hydroxide.

Replacement Parts

Bromcresol Green-Methyl Red Indicator Powder Pillows	100/pkg	943-99
Delivery Tube, 180 degree hook	5/pkg	17205-00
Digital titrator Assembly	each	16900-01
Erlenmeyer Flask, 250-mL	each	505-46
Phenolphthalein Indicator Powder Pillows	10/pkg	942-99
Sulfuric Acid Titration Cartridge, 0.1600 N	each	14388-01
Sulfuric Acid Titration Cartridge, 1.600 N	each	14389-01

Chlorine, Free (0 to 2.00 mg/L)

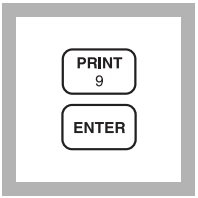
- For most accurate results, perform a Reagent Blank Correction using deionized water.
- Samples must be analyzed immediately and cannot be preserved for later analysis.
- Standard Adjust may be performed using a prepared standard.
- If the sample temporarily turns yellow after reagent addition, or the 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. Or, use the High Range Free Chlorine test, program #8.
- Wipe sample cells with a lint-free cloth before placing into the instrument.

1. Enter the stored program number for free and total chlorine (Cl_2) using powder pillows.

Press: **PRGM**

The display will show:

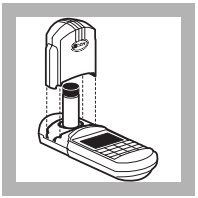
PRGM?



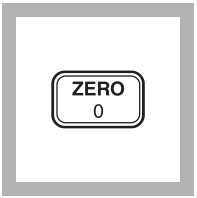
2. Press: **9 ENTER**

The display will show mg/L, Cl_2 and the **ZERO** icon.

3. Fill a sample cell with 10 mL of sample (the blank).



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

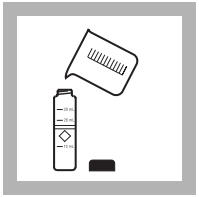


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Cl_2

Note: *If Reagent Blank Correction is on, the display may flash “limit”.*

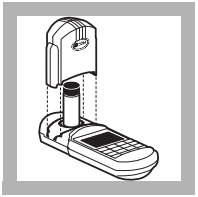


6. Fill another cell with 10 mL of sample.

Note: *Rinse the sample cell with sample before use.*

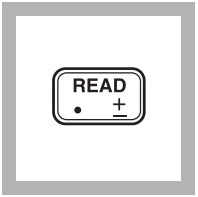
7. Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and swirl vigorously to dissolve the powder.

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.

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.

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.

Estimated Detection Limit

The estimated detection limit for program 9 is 0.02 mg/L Cl_2.

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).

Interferences

Interfering Substance	Interfering 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.
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.
Bromine	Interferes at all levels.
Chlorine Dioxide	Interferes at all levels.
Chloramines, organic	May interfere.

Interfering Substance	Interfering Levels and Treatments
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.

Summary of Method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,Ndiethyl-p-phenylenediamine) indicator to form a magenta color which is proportional to the chlorine concentration.

Replacement Parts

DPD Free Chlorine Reagent Powder Pillows 100/pkg 21055-69
Sample Cell, 10-20-25 mL 6/pkg 24019-06

Chlorine, Total (0 to 2.00 mg/L)

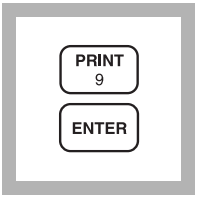
- For most accurate results, perform a Reagent Blank Correction using deionized water.
- Samples must be analyzed immediately and cannot be preserved for later analysis.
- Standard Adjust may be performed using a prepared standard.
- If the sample temporarily turns yellow after reagent addition, or the 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. Or, use the High Range Total Chlorine test, program #8.

1. Enter the stored program number for total chlorine (Cl_2) using powder pillows.

Press: **PRGM**

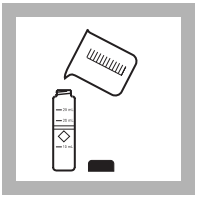
The display will show:

PRGM?

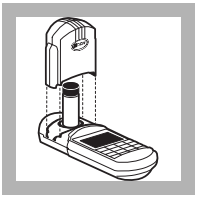


2. Press: **9 ENTER**

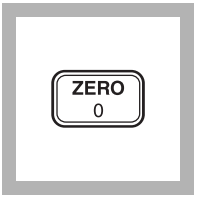
The display will show mg/L, Cl_2 and the ZERO icon.



3. Fill a sample cell with 10 mL of sample (the blank).



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

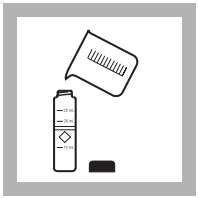


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Cl_2

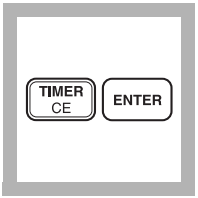
Note: *If Reagent Blank Correction is on, the display may flash “limit”.*



6. Fill another cell with 10 mL of sample.

7. 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.

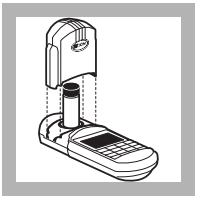
It is not necessary that all powder dissolves.



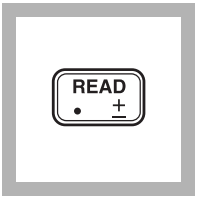
8. Press: **TIMER**, then **ENTER**

A three-minute reaction period will begin.

A pink color will develop if free chlorine is present.



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.

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.

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.

Estimated Detection Limit

The estimated detection limit for program 9 is 0.02 mg/L Cl_2.

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).

Interferences

Interfering Substance	Interfering 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, 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.
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 $CaCO_3$.

Interfering Substance	Interfering Levels and Treatments
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.

Oakton ECTestr

Follow the instructions included with the Oakton ECTestr.

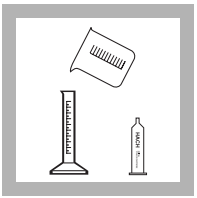
Replacement Parts

ECTestr, Low, Oakton Conductivity Tester, 0-1990 microS..... each 28455-00
ECTestr, High, Oakton Conductivity Tester, 0-19.90 mS ..... each 28456-00

Hardness, Total (10 to 4000 mg/L as $CaCO_3$)

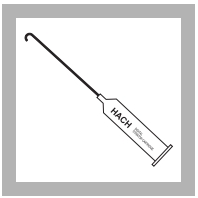
WARNING: Handling chemical samples, standards, and reagents can be dangerous. Review the Material Safety Data Sheets before handling any chemicals.

- mg/L Total Hardness as Ca = mg/L Total Hardness as ($CaCO_3$) × 0.400
- Total Hardness (as $CaCO_3$) = mg/L Ca (as $CaCO_3$) + mg/L Mg (as $CaCO_3$)
- Collect at least 100 mL of sample in a glass or polyethylene container. Samples may be kept up to seven days before analysis if stored at 4 °C and acidified to pH 2 with concentrated nitric acid. Neutralize acidified sample to pH 7 with ammonium hydroxide before testing.
- Four drops of ManVer® Hardness Indicator Solution or a 0.1 g scoop of ManVer® 2 Hardness Indicator Powder (Cat. No. 280-14) may be substituted for the powder pillow.

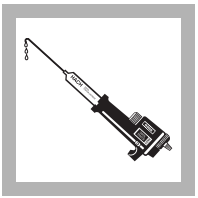


1. Select a sample size and an EDTA Titration Cartridge corresponding to the expected total hardness as calcium carbonate ($CaCO_3$) concentration. Use Table 1 for concentrations in mg/L.

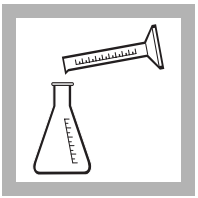
Note: *Hardness Test Strips (Cat. No. 27452-50) can be used to estimate hardness concentration.*



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

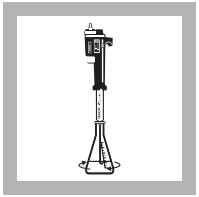


4. Use a graduated cylinder or pipet to measure the sample volume from Table 1 or Table 2. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

Note: *Rinse all glassware with sample before use.*

5. Add 2 mL of Hardness 1 Buffer Solution and swirl to mix.

6. Add the contents of one ManVer® 2 Hardness Indicator Powder Pillow and swirl to mix.



7. Place the delivery tube tip into the solution and swirl the flask while titrating with EDTA from red to pure blue. Record the number of digits required.

Note: *Titrate slowly near the end point because the reaction is slow, especially in cold samples.*

8. Use one of the following formulas to calculate the final concentration:

Digits Required x Digit Multiplier (Table 1) = mg/L Total Hardness as $CaCO_3$.

Note: *The magnesium concentration may be determined by subtracting the results of the calcium determination from total hardness.*

Table 1 Sample Volumes

Range (mg/L as $CaCO_3$)	Sample Volume (mL)	Titration Cartridge (M EDTA)	Catalog Number	Digit Multiplier
10-40	100	0.0800	14364-01	0.1
40-160	25	0.0800	14364-01	0.4
100-400	100	0.800	14399-01	1.0
200-800	50	0.800	14399-01	2.0
500-2000	20	0.800	14399-01	5.0
1000-4000	10	0.800	14399-01	10.0

Accuracy Check

Standard Additions Method

To verify analytical technique, use 20 mL of the Calcium Standard Solution, 1000 mg/L as $CaCO_3$. Perform the procedure as described above (Steps 1–8). This solution will read 1000 mg/L. Perform this accuracy check when interferences are suspected.

1. Snap the neck off a Hardness Standard Solution Voluette® Ampule, 10,000 mg/L as $CaCO_3$.
2. Use a TenSette® Pipet to add 0.1 mL of standard to the sample titrated in step 7. Resume titration back to the same end point. Record the number of digits required.
3. Repeat, using two more additions of 0.1 mL. Titrate to the end point after each addition.
4. Each 0.1 mL addition of standard should require 10 additional digits of 0.800 M titrant, 100 digits of 0.0800 M titrant, 11 digits of 0.714 M, or 56 digits of 0.1428 M titrant. If these uniform increases do not occur, refer to *Accuracy Check and Standard Additions* in the *DR/850 Procedures Manual.*

Interferences

- Although less common than calcium and magnesium, other polyvalent metal ions cause the same hardness effects and will be included in the results.
- Some transition and heavy metals complex the indicator and prevent the color change at the end point.
- Iron does not interfere in concentrations up to 15 mg/L. Above this level it causes a red-orange to green end point which is sharp and usable up to 30 mg/L iron. Substitute a 0.0800 M CDTA or 0.800 M CDTA titration cartridge for the 0.0800 M EDTA or 0.800 M EDTA titration cartridges, respectively, if iron interference is probable.
- Manganese titrates directly up to 20 mg/L but masks the end point above this level. Adding a 0.1-gram scoop of hydroxylamine hydrochloride monohydrate raises this level to 200 mg/L manganese.
- Copper and aluminum interfere at levels above 0.10 and 0.20 mg/L, respectively. Cobalt and nickel interfere at all levels and must be absent or masked. A 0.5-gram scoop of potassium cyanide removes interference from up to 100 mg/L copper, 100 mg/L zinc, 100 mg/L cobalt, and 100 mg/L nickel. It raises the permissible aluminum level to 1 mg/L. Metals masked with cyanide will not be included in the hardness result.

WARNING: Potassium cyanide is toxic. Always add it after the potassium hydroxide. Excess potassium cyanide does not affect results. All cyanide wastes should be disposed of by adding an excess of strongly alkaline sodium hypochlorite solution (bleach) with stirring. Use good ventilation. Allow to stand for 24 hours before disposal.

- Orthophosphate causes a slow end point and polyphosphate must be absent for accurate results.
- Acidity and alkalinity at 10,000 mg/L (as $CaCO_3$) do not interfere.
- Saturated sodium chloride solutions do not give a distinct end point, but the titration can be run directly on sea water.
- Adding the contents of one CDTA Magnesium Salt Powder Pillow removes metal interferences at or below the levels shown in Table 2.

Table 2

Metal	CDTA Removes Interference Below This Level
Aluminum	50 mg/L
Cobalt	200 mg/L
Copper	100 mg/L
Iron	100 mg/L
Manganese	200 mg/L
Nickel	400 mg/L
Zinc	300 mg/L

- If more than one metal is present at or above the concentrations shown above, an additional CDTA Magnesium Salt Powder Pillow may be required. Results obtained by this procedure include the hardness contributed by the metals. If the concentration of each metal is known, a correction can be applied to obtain the calcium and magnesium hardness concentration. The hardness (in mg/L as $CaCO_3$) contributed by each mg/L of metal is listed below, and can be subtracted from the total hardness value obtained above to determine the calcium and magnesium hardness. See Table 3.

Table 3

Metal	Metal Hardness as $CaCO_3$ Contributed by Each mg/L of Metal
Aluminum	3.710
Barium	0.729
Cobalt	1.698
Copper	1.575
Iron	1.792
Manganese	1.822
Nickel	1.705
Strontium	1.142
Zinc	1.531

- Barium, strontium and zinc titrate directly.
- Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.

Summary of Method

After the sample is buffered to pH 10.1, ManVer® 2 Hardness Indicator is added, and forms a red complex with a portion of the calcium and magnesium in the sample. EDTA titrant reacts first with the free calcium and magnesium ions, then with those bound to the indicator, causing it to change to a blue color at the end point.

Replacement Parts

Cylinder, graduated, 25-mL.....each.....1081-40
Cylinder, graduated, 100-mL.....each.....1081-42
Delivery Tube for Digital Titrator.....each..... 17205-00
Digital Titrator Assembly .....each..... 16900-01
Hardness 1 Buffer Solution .....100 mL..... 424-32
ManVer® 2 Hardness Indicator Powder Pillows, 50-mL..... 100/pkg.....851-99
TitraVer EDTA Titration Cartridge, 0.0800 M .....each..... 14364-01
TitraVer EDTA Titration Cartridge, 0.800 M .....each..... 14399-01

Iron, Total (0 to 3.00 mg/L)

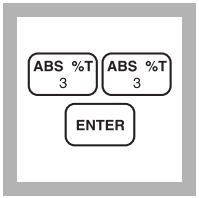
- *Sample Pretreatment For Metals Analysis* on page 45 may be performed to determine total iron content.
- Adjust pH of stored samples before analysis.
- Standard Adjust may be performed using a prepared standard.
- Wipe sample cells with a clean, dry cloth before placing in the instrument.

1. Enter the stored program number for iron (Fe) powder pillows.

Press: **PRGM**

The display will show:

PRGM?



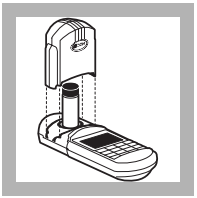
2. Press: **33 Enter**

The display will show **mg/L, Fe** and the **ZERO** icon.

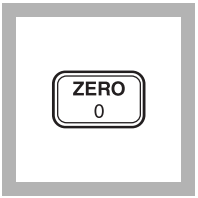
3. Fill a sample cell with 10 mL of sample (the blank).

Wipe the cell with a soft cloth.

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**

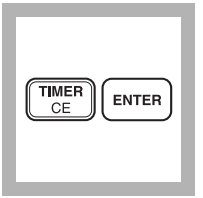
The cursor will move to the right, then the display will show:

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.*

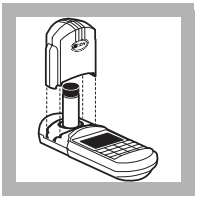


8. Press: **TIMER ENTER**

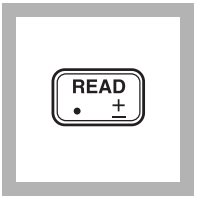
A three-minute reaction period will begin.

Note: *An orange color will form if iron is present.*

Note: *Samples containing visible rust should be allowed to react at least five minutes.*



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.

Sampling and Storage

Collect samples in acid-cleaned glass or plastic containers. Clean glassware with 1:1 nitric acid solution. If unavailable, other dilute acid solutions may be used. Rinse three times with deionized water.

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. If only dissolved iron is to be determined, filter the sample before adding the acid.

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.

Estimated Detection Limit

The EDL for program 33 is 0.03 mg/L Fe.

Interferences

Interfering Substance	Interfering Levels and Treatments
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 (*Sample Pretreatment For Metals Analysis*). 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.
Turbidity	1. Add 0.1 g scoop of RoVer® Rust Remover to the blank in on page 42. 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 step 3 and step 6.
Sample pH (extreme)	Adjust pH to 3–5.
Highly Buffered Samples	Adjust pH to 3–5.

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.

Replacement Parts

FerroVer® Iron Reagent Powder Pillows 100/pkg 21057-69
Sample Cell, 10-20-25 mL 6/pkg 24019-06

Sample Pretreatment For Metals Analysis

Digestion is only required for total recoverable metal determination. It is not required for dissolved metals testing.

Digestion

Some metals procedures may 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 1 on page 46) 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 1 Vigorous Digestion Volumes .
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 1 . A reagent blank also should be carried through the digestion and measurement procedures.

Table 1 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

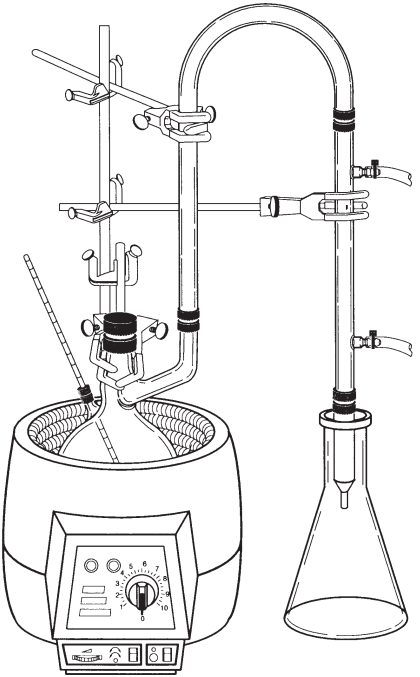
Distillation is an effective way of separating chemical components for analysis. The Hach Distillation Apparatus (see Figure 1) is adapted easily for many test needs and is suitable for water and wastewater samples. Sample distillations are easy and safe to perform.

Applications for the General Purpose Distillation Apparatus include:

- fluoride
- phenols
- albuminoid nitrogen
- selenium
- ammonia nitrogen
- 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 1 General Purpose Distillation Apparatus with Heater and Support Apparatus



Manganese, High Range (0 to 20.0 mg/L)

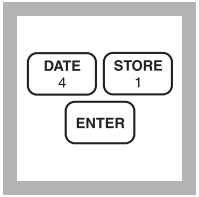
- *Sample Pretreatment For Metals Analysis* on page 45 may be performed to determine total manganese content.
- Adjust pH of stored samples before analysis.
- Standard Adjust may be performed using a prepared standard.

1. Enter the stored program number for manganese, periodate oxidation method.

Press: **PRGM**

The display will show:

PRGM?

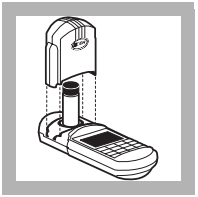


2. Press: **41 Enter**

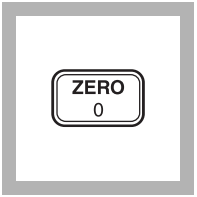
The display will show **mg/L**, **Mn** and the **ZERO** icon.

3. Fill a sample cell with 10 mL of sample (the blank).

Wipe the cell with a soft cloth.



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



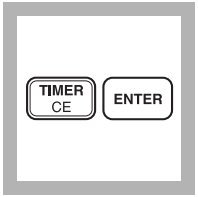
5. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L 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.

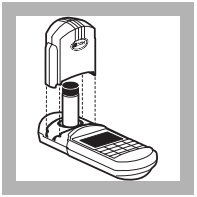
7. Add the contents of one Sodium Periodate Powder Pillow to the sample cell (the prepared sample). Cap the sample cell. Invert for 10 seconds to mix.



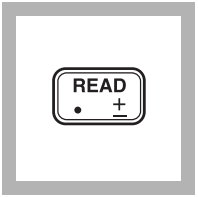
8. Press: **TIMER ENTER**

A two-minute reaction period will begin.

Note: *A violet color will form if manganese is present.*



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.

Sampling and Storage

Collect samples in acid-washed plastic bottles. Manganese may be lost by adsorption to glass container walls. To acid wash bottles, rinse with dilute nitric acid then rinse thoroughly with deionized water.

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. If only dissolved Mn is to be determined, filter before acid addition.

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.

Interferences

Interfering Substance	Interfering Levels and Treatments
Calcium	700 mg/L
Chloride	70,000 mg/L
Iron	5 mg/L
Magnesium	100,000 mg/L

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.

Replacement Parts

Citrate Buffer Powder Pillows ..... 100/pkg..... 21076-69
Sodium Periodate Reagent Powder Pillows, 10-mL ..... 100/pkg..... 21077-69
Sample Cell, 10-20-25 mL .....6/pkg..... 24019-06

Nitrate, Low Range (0 to 0.50 mg/L NO_3^-–N)

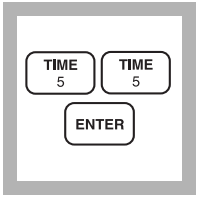
- For most accurate results, perform a Reagent Blank Correction using deionized water.
- Adjust the pH of stored samples before analysis.
- Remove all the powder from the NitraVer® 6 Powder Pillow by tapping repeatedly until no more powder comes out.
- Shaking time and technique influence color development. 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.
- Use of the standard adjust feature with each new lot of reagent is highly recommended.
- 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.

1. Enter the stored program number for low range nitrate nitrogen using powder pillows.

Press: **PRGM**

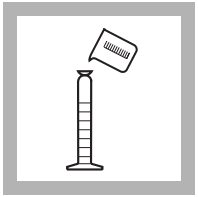
The display will show:

PRGM?

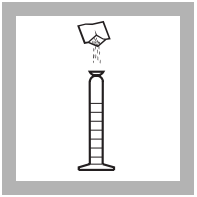


2. Press: **55 Enter**

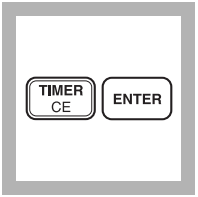
The display will show mg/L, NO_3^-–N and the **ZERO** icon.



3. Fill a 25-mL graduated mixing cylinder to the 15-mL mark with sample.

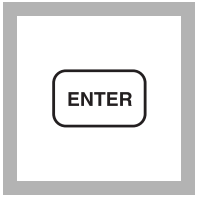


4. Add the contents of one NitraVer 6 Nitrate Reagent Powder Pillow to the cylinder. Stopper.



5. Press: **TIMER ENTER**

A 3-minute reaction period will begin. Shake the cylinder vigorously until the timer beeps.



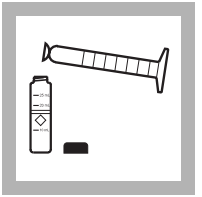
6. After the timer beeps, the display will show:

2:00 TIMER 2

Press: **ENTER**

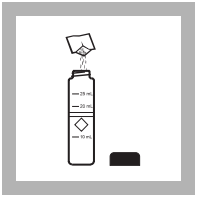
A 2-minute reaction period will begin.

A cadmium deposit will remain after the NitraVer 6 Nitrate Reagent Powder dissolves and will not affect test results.



7. After 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.*

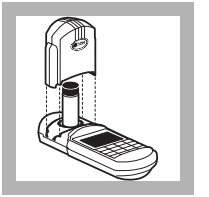
9. The display will show:

15:00 TIMER 3

Press: **ENTER**

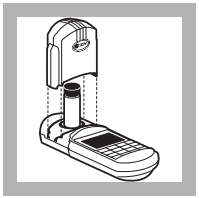
A 15-minute reaction period will begin.

Note: *If Reagent Blank Correction is on, the display may flash "limit".*

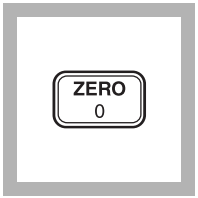


10. Fill another sample cell (the blank) with 10 mL of sample.

11. After the timer beeps, wipe off any liquid or fingerprints from the sample cell.



12. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

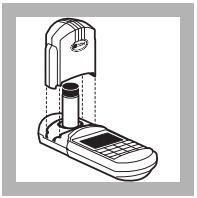


13. Press: **ZERO**

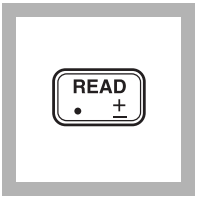
The cursor will move to the right, then the display will show:

0.0 mg/L NO_3^-–N

Note: *If Reagent Blank Correction is on, the display may flash "limit".*



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 NO_3^-–N (or NO_3) will be displayed.

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.

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 powder pillows with the instrument, a single operator obtained a standard deviation of ±0.03 mg/L nitrate nitrogen.

Estimated Detection Limit

The estimated detection limit for program 55 is 0.01 mg/L NO_3^-–N.

Pollution Prevention and Waste Management

NitraVer 6 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.

Interferences

Interfering Substance	Interfering 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 interfere.
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 used for 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	All levels interfere.

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 product.

Replacement Parts

Cylinder, graduated, 25-mL.....each.....1081-40
NitraVer® 6 Reagent Powder Pillows, 10-mL..... 100/pkg..... 21072-49
NitriVer® 3 Reagent powder Pillows, 10-mL..... 100/pkg.....21071-69

Nitrate, High Range (0 to 30.0 mg/L NO_3^-–N)

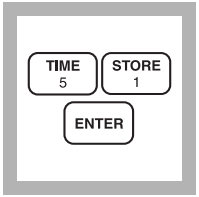
- For most accurate results, perform a Reagent Blank Correction using deionized water.
- Adjust the pH of stored samples before analysis.
- Remove all the powder from the NitraVer® 5 Powder Pillow by tapping repeatedly until no more powder comes out.
- Shaking time and technique influence color development. 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.
- Use of the standard adjust feature with each new lot of reagent is highly recommended.
- 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.

1. Enter the stored program number for high range nitrate nitrogen using powder pillows.

Press: **PRGM**

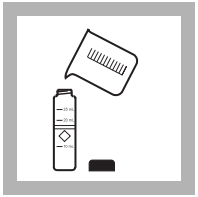
The display will show:

PRGM?

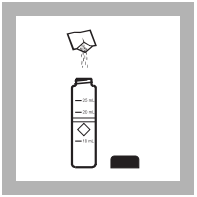


2. Press: **51 Enter**

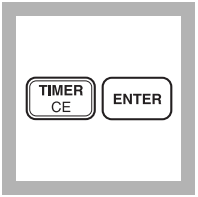
The display will show mg/L, NO_3^-–N and the **ZERO** icon.



3. Fill a sample cell with 10 mL of sample.

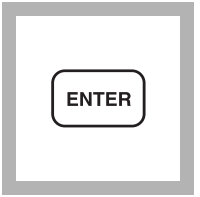


4. Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow to the cell (the prepared sample). Cap the cell.



5. Press: **TIMER ENTER**

A one-minute reaction period will begin. Shake the sample vigorously until the timer beeps.



6. After the timer beeps, the display will show:

5:00 TIMER 2

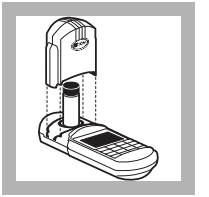
Press: **ENTER**

A five-minute reaction period will begin.

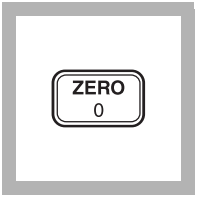
A cadmium deposit will remain after the NitraVer 5 Nitrate Reagent Powder dissolves and will not affect test results.

An amber color will develop if nitrate nitrogen is present.

7. Fill another cell with 10 mL of sample (the blank). Wipe off any liquid or fingerprints from the sample cell.



8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

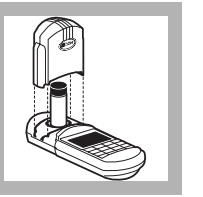


9. When the timer beeps press: **ZERO**

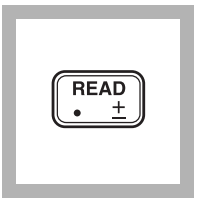
The cursor will move to the right, then the display will show:

0.0 mg/L NO_3–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.



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.

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.

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 +0.3 mg/L nitrate nitrogen for program 51.

Estimated Detection Limit

The estimated detection limit for program 51 is 0.8 mg/L NO_3^-–N.

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.

Interferences

Interfering Substance	Interfering 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 interfere.
Nitrite	All levels interfere. Compensate for nitrite interference as follows: 1. Add 30-g/L Bromine Water a drop at a time to the sample in step 3 on page 56 until a yellow color remains. 2. Add one drop of 30-g/L Phenol Solution to destroy the color. 3. Proceed with step 3 on page 56. 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	All levels interfere.

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.

Replacement Parts

NitraVer® 5 Nitrate Reagent Powder Pillows, 10-mL ..... 100/pkg.....21061-69
Sample Cell, 10-20-25 mL .....6/pkg..... 24019-06

Nitrite, Low Range (0 to 0.350 mg/L NO_2^-–N)

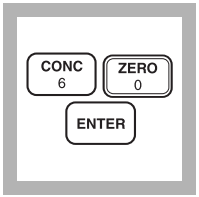
- For most accurate results, perform a Reagent Blank Correction using deionized water .
- If Reagent Blank Correction is on, the display may flash “limit”.

1. Enter the stored program number for nitrite nitrogen (NO_2^-–N) powder pillows.

Press: **PRGM**

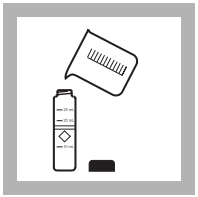
The display will show:

PRGM?



2. Press: **60 Enter**

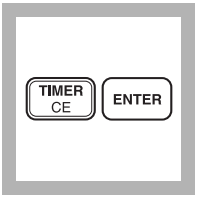
The display will show mg/L, NO_2^--N and the ZERO icon.



3. Fill a sample cell with 10 mL of sample.

4. Add the contents of one NitriVer® 3 Nitrite Reagent Powder to the sample cell. Cap and shake to dissolve.

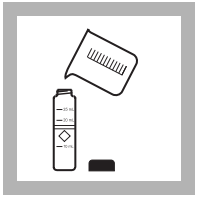
Accuracy is not affected by undissolved powder.



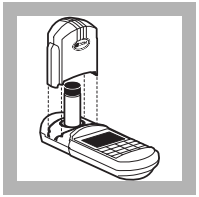
5. Press: **TIMER ENTER**

A 15-minute reaction period will begin.

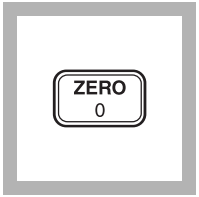
A pink color will develop if nitrite is present.



6. When the timer beeps, fill an empty sample cell with 10 mL of sample (the blank).



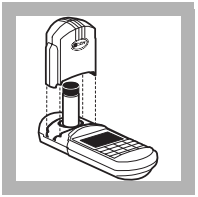
7. Wipe the outside of the blank with a towel. Place into the cell holder. Tightly cover the sample cell with the instrument cap.



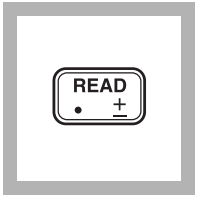
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.000 mg/L NO_2^--N



9. Wipe the outside of the prepared sample cell with a towel. 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.

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.

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.

Estimated Detection Limit

The estimated detection limit for program 60 is 0.005 mg/L NO_2^-–N.

Interferences

Interfering Substance	Interfering Levels and Treatments
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 ions	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.

Replacement Parts

NitriVer® 3 Nitrite Reagent Powder Pillows, 10-mL.....100/pkg.....21071-69
Sample Cell, 10-20-25 mL.....6/pkg..... 24019-06

Oakton pH Test™ 2

Follow the instructions included with the Oakton pH Testr 2.

Replacement Parts

pH Testr™ 2*, Oakton, Waterproof, –0.1 to 15 pH, double junction .....each..... 27993-00
pH Testr™ 2, Oakton, Waterproof, –0.1 to 15 pH.....each..... 27235-00

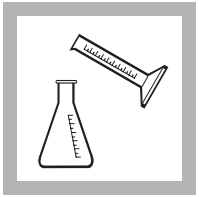
* pHTestr 2 is a trademark of Oakton Instruments.

Total Phosphorus Modified Digestion Notes

While the modified total phosphorus procedure does not include boiling, a traditional phosphorus digestion includes boiling the sample on a hot plate after acid is added. Because boiling is omitted in this procedure, the modified digestion will give lower results than a traditional hot plate digestion. Results from the original and modified methods cannot be directly compared. Results from a modified total phosphorus digestion may not reflect a true total phosphorus measurement and should not be reported as such.

If facilities and resources are available, a hot plate digestion can be performed on the sample to obtain the true total phosphorus results. After *step 4* of the modified procedure, boil the sample for 30 minutes. Deionized water may be added to maintain the volume at 25 mL. Do not boil to dryness. Allow the sample to cool and proceed with *step 6*. If the sample is digested by boiling, **do not** multiply the DR/850 reading in *step 16* by 1.2.

1. Rinse all glassware with a 1:1 Hydrochloric Acid Solution. Rinse 3 times with deionized water. Do not use detergents containing phosphates to clean glassware.



2. Using a graduated cylinder, measure 25 mL of sample into a 250 mL Erlenmeyer flask or a DR/850 sample cell.

Note: *Adjust the pH of stored samples before digestion.*

3. Using the 1-mL dropper provided, add 2.0 mL of 5.25 N Sulfuric Acid Solution. Mix thoroughly.

4. Add one Potassium Persulfate Powder Pillow. Mix thoroughly.

5. Cover the sample and let sit for at least 12 hours, but no longer than 24 hours, before proceeding to the next step.

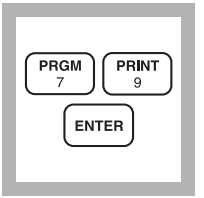
6. Using the 1-mL dropper provided, add 2.0 mL of 5.0 N Sodium Hydroxide Solution. Mix thoroughly.

7. Enter the stored program number for reactive phosphorus, ascorbic acid method.

Press: **PRGM**

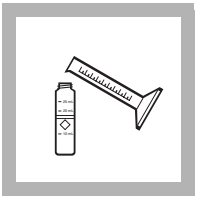
The display will show:

PRGM?



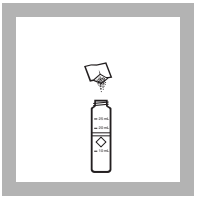
8. Press: **79 ENTER**

The display will show **mg/L, PO_4** and the **ZERO** icon.



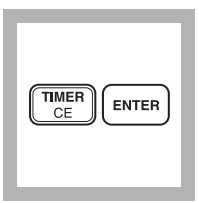
9. Fill a sample cell with 10 mL of digested sample.

Note: *For samples with extreme pH, see Interferences following these steps.*



10. 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.*

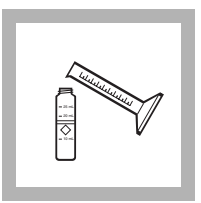


11. Press:

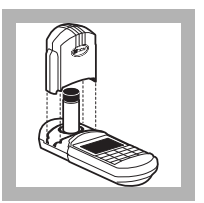
TIMER ENTER

A two-minute reaction period will begin. Perform steps 12-14 during this period.

Note: *If the acid-persulfate digestion was used, an 8-10 minute reaction period is required.*

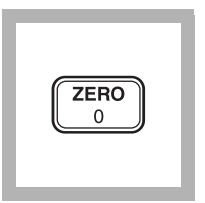


12. Fill another sample cell with 10 mL of sample (the blank).



13. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

Press: **EXIT**

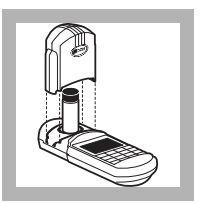


14. Press: **ZERO**

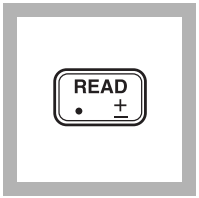
The cursor will move to the right, then the display will show:

0.00 mg/L PO_4

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



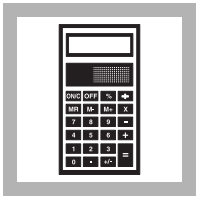
15. After the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



16. Press: **READ**

The cursor will move to the right, a value 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.*



17. The dilution performed in the modified digestion procedure requires you to multiply the result in step 16 by a factor of 1.2. This is the final result.

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 28 days by adjusting the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter) and storing samples at 4 °C. Warm to room temperature before testing. Correct results for volume additions.

Interferences

For turbid samples, use 50 mL of sample and double all the reagent quantities. Use digested sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed in the 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.

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-}.

Estimated Detection Limit (EDL)

The EDL for program 79 is 0.05 mg/L PO_4.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro-, or 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 orthophosphates 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 method for determination of the phosphorus content of the sample. If the ascorbic acid (PhosVer3) method is used to measure the reactive phosphorus, this method is EPA approved for NPDES reporting.

Replacement Parts

Cylinder, graduated, 25-mL	each	1081-40
Erlenmeyer Flask, 250-mL	each	505-46
PhosVer® 3 Reagent Powder Pillows, 10-mL	100/pkg	21060-69
Potassium Persulfate Reagent Powder Pillow	100/pkg	2451-99
Sample Cell, 10-20-25 mL	6/pkg	24019-06
Sodium Hydroxide Solution, 5.0 N	100 mL	2450-32
Sulfuric Acid Solution, 5.25 N	100 mL	2449-32

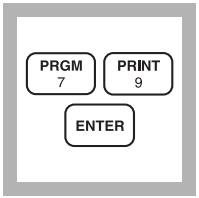
Orthophosphate (Reactive Phosphorus), (0–2.50 mg/L PO_4^{3-})

1. Enter the stored program number for reactive phosphorus, ascorbic acid method.

Press: **PRGM**

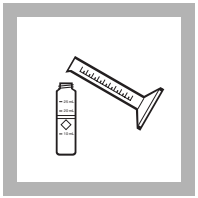
The display will show:

PRGM?



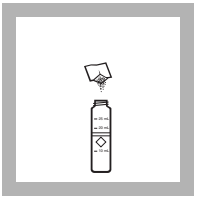
2. Press: **79 ENTER**

The display will show **mg/L, PO_4** and the **ZERO** icon.



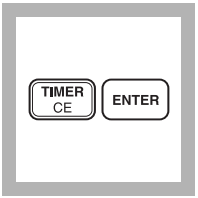
3. Fill a sample cell with 10 mL of sample.

Note: *For samples with extreme pH, see Interferences following these steps.*



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.*

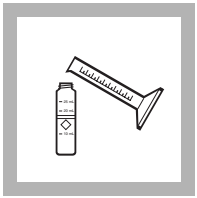


5. Press:

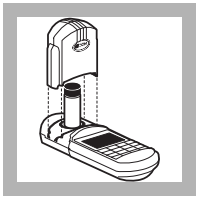
TIMER ENTER

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.*

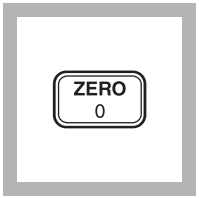


6. Fill another sample cell with 10 mL of sample (the blank).



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

Press: **EXIT**

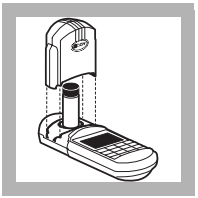


8. Press: **ZERO**

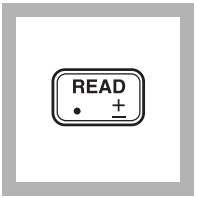
The cursor will move to the right, then the display will show:

0.00 mg/L PO_4

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, a value in mg/L orthophosphate (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 28 days by adjusting the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter) and storing samples at 4 °C. Warm to room temperature before testing. Correct results for volume additions.

Interferences

For turbid samples, use 50 mL of sample and double all the reagent quantities. Use digested sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed in the 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.

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 ±0.03 mg/L PO_4^{3-}.

Estimated Detection Limit (EDL)

The EDL for program 79 is 0.05 mg/L PO_4.

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.

Replacement Parts

PhosVer® 3 Reagent Powder Pillows, 10-mL 100/pkg 21060-69
Sample Cell, 10-20-25 mL 6/pkg 24019-06

Sulfate (0 to 70 mg/L)

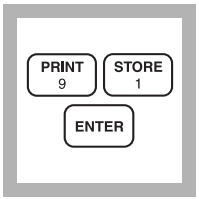
- A User-Entered Calibration is necessary to obtain the most accurate results. See the User Calibration section at the back of this procedure. Program 91 can be used for process control or applications where a high degree of accuracy is not needed. The nature of turbidimetric tests and reagent lot variation requires user calibration for best results.

1. Enter the stored program number for sulfate (SO_4-).

Press: **PRGM**

The display will show:

PRGM?

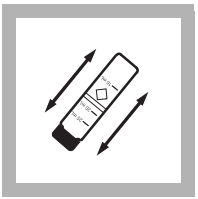


2. Press: **91 ENTER** or the program number selected for a user entered calibration.

The display will show **mg/L, SO_4** and the **ZERO** icon.

3. Fill a clean sample cell with 10 mL of sample.

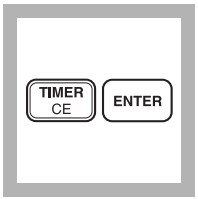
Note: *Filter highly turbid or colored samples. Use filtered sample in this step and as the blank.*



4. 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.

A white turbidity will develop if sulfate is present in the sample.

Accuracy is not affected by undissolved powder.

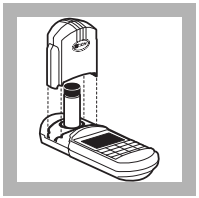


5. Press:

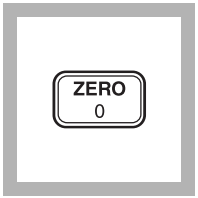
TIMER ENTER

A 5-minute reaction period will begin. Allow the cell to stand undisturbed.

6. After the timer beeps, fill a second sample cell with 10 mL of sample (the blank).



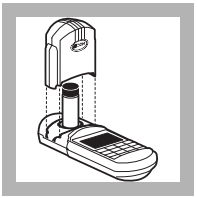
7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



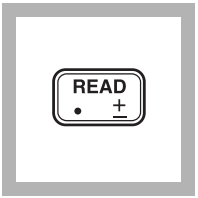
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L SO_4



9. Within five 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 sulfate will be displayed.

Note: *If Program 91 is used, use of the Standard Adjust is highly recommended.*

Note: *Clean the sample cells with soap and a brush, then acid wash before next use.*

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. Program 91 can be run when a high degree of accuracy is not needed. Use of the Standard Adjust feature will improve performance when using program 91. 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 *DR/890 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.

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.

Estimated Detection Limit

The EDL for program 91 is 4.9 mg/L SO_4.

Interferences

Interfering Substance
Calcium: 20,000 mg/L as $CaCO_3$
Chloride: 40,000 mg/L as Cl^-
Magnesium: 10,000 mg/L as $CaCO_3$
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.

Replacement Parts

Sample Cell, 10-20-25 mL 6/pkg 24019-06
SulfaVer® 4 Sulfate Reagent Powder Pillow 100/pkg 21067-69

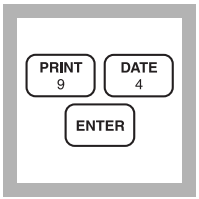
Suspended Solids (0–750 mg/L)

1. Enter the stored program number for suspended solids.

Press: **PRGM**

The display will show:

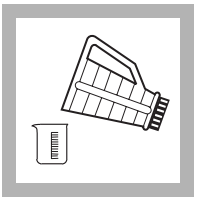
PRGM?



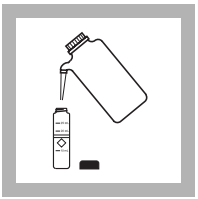
2. Press: **94 Enter**

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. If a blender is not available, proceed to step 4, but make sure to collect a well-mixed sample.

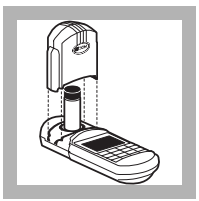


4. Pour the blended sample into a 600-mL beaker. If a blender was not used, pour 500 mL of the sample directly into the beaker.

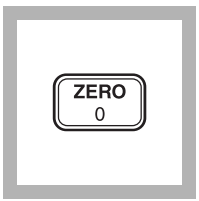


5. Fill a sample cell with 25 mL of tap water or deionized water (the blank).

Remove gas bubbles in the water by swirling the cell or tapping it on a table.



6. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

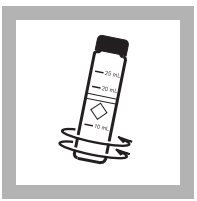


7. Press: **ZERO**

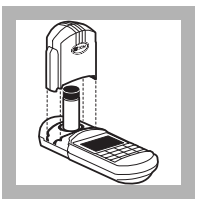
The cursor will move to the right, then the display will show:

0 mg/L SuSld

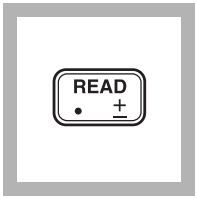
8. Stir the sample thoroughly and immediately pour 25 mL of the blended sample into a sample cell (the prepared sample).



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 sample 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.

Estimated Detection Limit

The estimated detection limit for program 94 is 22.1 mg/L Suspended Solids.

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 determination 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 photometric method is often used for checking process efficiency and trending environmental samples.

Replacement Parts

Beaker, 600-mL ..... each 1080-52
Sample Cell, 10-20-25 mL 6/pkg 24019-06

Turbidity (0 to 1000 FAU)

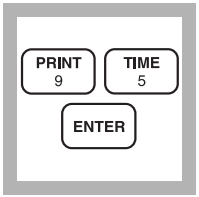
Note: 1 FAU=1 NTU=1 FTU when measuring formazin only. These are not equivalent when measuring other types of standards or samples. A correlation must be established by the user to relate FAU to NTU.

1. Enter the stored program number for turbidity.

Press: **PRGM**

The display will show:

PRGM?

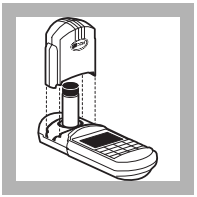


2. Press: **95 Enter**

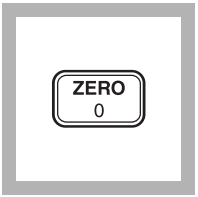
The display will show FAU and the ZERO icon.

3. Fill a sample cell with 10 mL of deionized water (the blank).

Wipe the cell with a soft cloth.



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

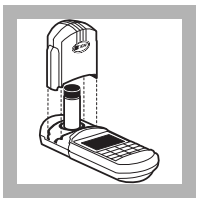
The cursor will move to the right, then the display will show:

0 FAU

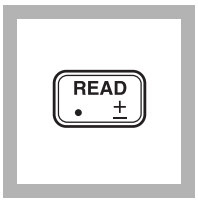
6. Mix the sample well before transferring it to the sample cell.

Fill another sample cell with 10 mL of sample.

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.*

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.

Method Performance

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.

Interferences

Interfering Substance	Interfering Levels and Treatments
Air Bubbles	Interfere at all levels. Degas samples using the Degassing Kit or an ultrasonic bath.
Color	Interfere at all levels. Degas samples using the Degassing Kit or an ultrasonic bath.
Temperature	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 environmental trending. 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.)

Replacement Parts

Sample Cell, 10-20-25 mL .....6/pkg..... 24019-06