46014-88

DR/700
COLORIMETER
PROCEDURES MANUAL

7-30-96-9ed
Rev. 2b, 11/01

TABLE OF CONTENTS

CONTENTS, continued

INTRODUCTION

This manual is divided into three sections:

Section I Chemical Analysis Information
This section relates to all of the procedures. It provides excellent background information or review material for the technician or chemist. Commonly-used procedure steps are explained in detail.

Section II Procedures
Step-by-step illustrated instructions for measuring approximately 100 different parameters or constituents are presented. The instrument is factory calibrated and ready to use (except for procedures requiring user calibration). Clearly written steps are supplemented with helpful notes. Each procedure includes information on sampling and storage, checking accuracy, adjusting for interferences and a listing of all the reagents and apparatus needed to run the test. Additional information on the chemical reactions of many of the procedures is contained in the Hach Water Analysis Handbook, Publication 8353, available free on request.

Section III Technical Support
Technical support is provided to our customers in numerous ways as described in the paragraphs in this section. Hach provides free training workshops and offers publications on various areas of analysis, also free of charge. A staff of trained specialists are on call to give individual assistance via our 800 number throughout each working day

> Before preceding to the analysis procedures in Section II, the analyst should read the instrument manual to learn about the DR/700 features and its operation.

SAMPLE PROCEDURE

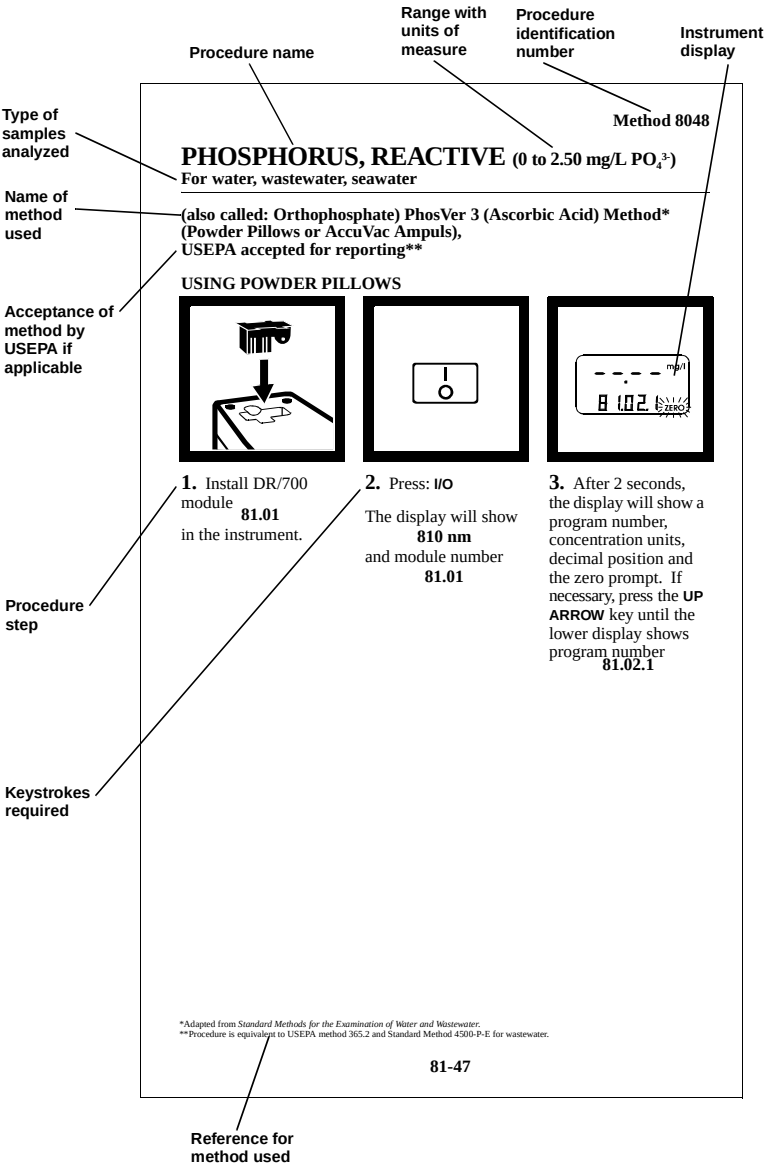
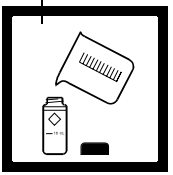


Illustration of procedure steps and instrument keystrokes required

PHOSPHORUS, REACTIVE, continued



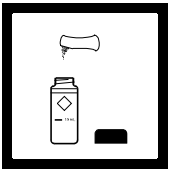
4. Fill a 10-mL cell to the 10-mL line with sample.

Note: *For proof of accuracy, use a 1.0 mg/L Phosphate (0.33 mg/L P) Standard Solution (listed under Optional Reagents) following these steps, in place of the sample.*

Additional information that may be applicable

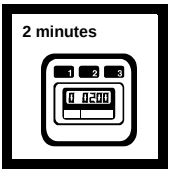
Note: *Run a reagent blank for this test. Use deionized water in place of the sample in Step 4. Subtract this result from all test results run with this lot of PhosVer.*

Note: *Optional 25-mL reagents sample may be used (see Optional Reagents).*



5. Add the contents of one PhosVer 3 Phosphate Powder Pillow to the sample cell (the prepared sample). Cap and invert several times to mix.

Note: *A blue color will form if phosphate is present.*



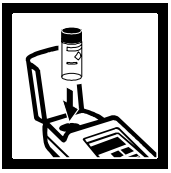
6. Wait two minutes.

Note: *An 8-10 minute reaction period should be used if determining total phosphate following the acid-persulfate digestion.*

Note: *If the sample temperature is less than 15 ^{O}C (59 ^{O}C), allow 9 minutes of reaction time.*

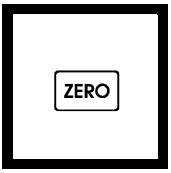
PHOSPHORUS, REACTIVE, continued

7. Fill a 10-mL cell to the 10-mL line with sample (the blank).



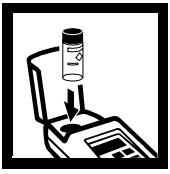
8. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



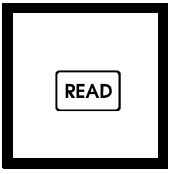
9. Press: **ZERO**

The display will count down to 0 Then the display will show 0.00 mg/l, and the zero prompt will turn off.



10. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



11. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/l phosphate (as PO_4).

Note: *To convert results to other units, see Table 1.*

Conversion Factors Table

Table 1. Conversion Factors

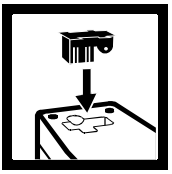
To convert reading from	To	Multiply by
mg/L PO_4	mg/L P_2O_5	0.747
mg/L PO_4	mg/L P	0.326

81-49

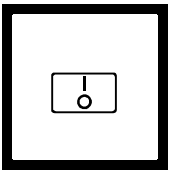
PHOSPHORUS, REACTIVE, continued

Alternate method

USING ACCUVAC AMPULS

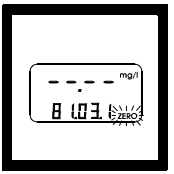


1. Install DR/700 module
81.01
in the instrument.



2. Press: **I/O**

The display will show
810 nm
and module number
81.01

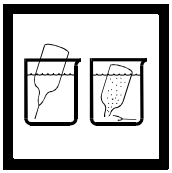


3. After 2 seconds, the display will show a program number, concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
81.03.1

PHOSPHORUS, REACTIVE, continued

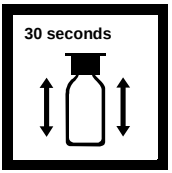
4. Fill a cell with 10 mL of sample (the blank). Cap. Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Run a reagent blank for this test. Use deionized water in place of the sample in Step 4. Subtract this result from all test results run with this lot of PhosVer.*



5. Fill a PhosVer 3 Phosphate AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*



6. Place an ampul cap securely over the tip of the ampul. Shake the ampul for approximately 30 seconds. Wipe off any liquid and finger prints.

Note: *A blue color will form if phosphate is present.*

Note: *Accuracy is unaffected by undissolved powder.*

7. Wait two minutes.

8. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

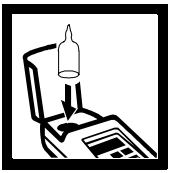
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/l, and the zero prompt will turn off.

81-51

PHOSPHORUS, REACTIVE, continued

10. Insert the AccuVac Vial Adapter into the cell holder.



11. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L phosphate (as PO_4).

Note: *To convert results to other units, see Table 2.*

Table 2. Conversion Factors

To convert reading from	To	Multiply by
mg/L PO_4	mg/L P_2O_5	0.747
mg/L PO_4	mg/L P	0.326

If sample cannot be run immediately, follow these steps

SAMPLING AND STORAGE

Collect sample in plastic or glass bottles that have cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in phosphate analysis.

Most reliable results are obtained when samples are analyzed as soon as possible after collection. If prompt analysis is impossible, preserve samples up to 24 hours by storing at or below 4 C. For longer storage periods, add 4.0 mL of Mercuric Chloride Solution to each liter of sample taken and mix. Use of mercuric chloride is discouraged whenever possible for health and environmental considerations. Sample refrigeration is still required. Samples preserved with mercuric chloride must have a sodium chloride level of 50 mg/L or more to prevent mercury interference. Samples low in chloride should be spiked with 0.1 g sodium chloride per liter of sample.

PHOSPHORUS, REACTIVE, continued

Confirm accuracy with these steps

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Phosphate Voluette Ampule Standard Solution, 50 mg/L PO_4^{-}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 25-mL water sample. Mix each thoroughly. (For AccuVac Ampuls use 50-mL Beakers.)

In addition, may also be used to troubleshoot a test, improve technique, check reagents and to assure cleanliness of glassware

c) Analyze each sample as described above. The phosphate concentration should increase 0.2 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section 1 of the DR/2000 Procedures manual or Water Analysis Handbook) for more information.

INTERFERENCES

Levels of common sample substances or conditions that will cause inaccurate results

Large amounts of turbidity may cause inconsistent results in the phosphate tests because the acid present in the powder pillow may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles. for highly turbid or colored samples, add the contents of one Phosphate Pretreatment Powder Pillow to 25 mL of sample. Mix well. Use this solution to zero the instrument.

The PhosVer 3 Reagent Powder Pillows should be stored in a cool, dry environment.

The following may interfere when present in concentrations exceeding these listed below:

Copper	10 mg/L
Iron	100 mg/L
Silica	50 mg/L
Silicate	10 mg/L

Arsenate and hydrogen sulfide do interfere.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section 1).

PHOSPHORUS, REACTIVE, continued

Expected repeatability of the procedure

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 1.60 mg/L PO_4^{3-} concentration samples, the standard deviation was ±0.007 mg/L PO_4^{3-}.

Testing zero concentration samples, the limit of detection was 0.019 mg/L PO_4^{3-}. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry*, **1980,** *52*, 2242-2249.

Using two representative lots of AccuVacs, the standard deviation was ±0.008 mg/L PO_4^{3-} and the limit of detection was 0.021 mg/L PO_4^{3-}.

Concise explanation of method

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.

The amount of reagents and apparatus needed to perform the procedure as written

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent			
Powder Pillows	1 Pillow	100/pkg	2125-99

REQUIRED REAGENTS (Using AccuVac Ampuls)

PhosVer 3 Phosphate Reagent			
AccuVac Ampuls	1 ampul	25/pkg	25080-25

Items needed to perform the procedure, not included with the instrument

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening			
powder pillows	1	each	968-00
DR/700 Filter Module			
Number 81.01	1	each	46281-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41
Cap, ampul, blue	1	25/pkg	1731-25
DR/700 Filter Module			
Number 81.01	1	each	46281-00

81-54

PHOSPHORUS, REACTIVE, continued

Supplemental reagents and apparatus mentioned in the notes

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Hydrochloric Acid		
Standard Solution, 6.0 N (1:1)	500 mL	884-49
Mercuric Chloride Solution, 10 g/L	100 mL	14994-42
Phosphate Pretreatment Powder Pillows	50/pkg	14501-66
Phosphate Standard Solution,		
1 mg/L as PO_4	500 mL	2569-42
Phosphate Standard Solution, Voluette ampul,		
50 mg/L as PO_4, 10 mL	16/pkg	171-10
PhosVer 3 Phosphate Reagent		
Powder Pillows, 25 mL sample	100/pkg	2125-99
Sodium Chloride, ACS	454 g	182-01
Sodium Hydroxide		
Standard Solution, 5.0 N	100 mL* MDB	2450-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Adapter, AccuVac Vial, DR/700	each	43784-00
Ampule Breaker Kit	each	21968-00
pH Indicator Paper, 1 to 11 pH.	5 rolls/pkg	391-33
pH Meter, Hach One	each	43800-00
Pipet, 2 mL serological	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01	50/pkg	21856-96
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Spoon, measuring, 0.1 g	each	511-00

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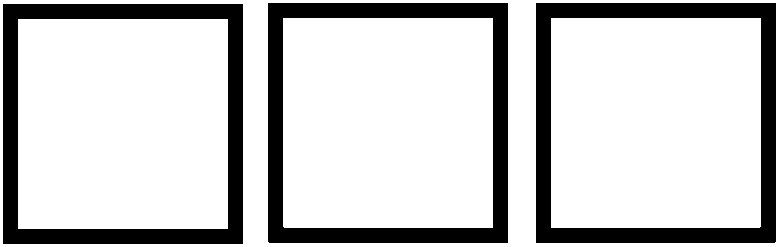
Outside the U.S.A. - Contact the Hach office or distributor serving you.

*Larger sizes available.

SECTION I Chemical Analysis Information



SECTION I CHEMICAL ANALYSIS INFORMATION

ABBREVIATIONS AND CONVERSIONS

Abbreviations

The following abbreviations are used throughout the text of the procedure section:

°C degree(s) Celsius (Centigrade)
°F degree(s) Fahrenheit
ACS American Chemical Society reagent grade purity
A/F Acid/fluoride extraction method for soils
APHA Standard Methods *Standard Methods for the Examination of Water and Wastewater,* published jointly by the American Public Health Association (APHA), the American Water Works Association (AWWA), and the Water Pollution Control Federation (WPCF). Order from Hach requesting Cat. No. 22708-00 or from the Publication Office of the American Public Health Association. This book is the standard reference work for water analysis. Many procedures contained in this manual are based on *Standard Methods.*
AV AccuVac
Bic bicarbonate extraction method for soils
Bicn bicinchoninate
conc concentrated
DB dropping bottle
F&T free and total
FTU Formazin Turbidity Units. Turbidity unit of measure based on a Formazin stock suspension.
FV FerroVer
FZ FerroZine
g grams
gr/gal grains per gallon (1 gr/gal = 17.12 mg/L)
HR high range
kg/ha kilograms per hectare
Lbs/Ac pounds per acre
LR low range
MDB marked dropping bottle
mg/L milligrams per liter (ppm)
µg/L micrograms per liter (ppb)
ml or mL (milliliter)-approximately the same as a cubic centimeter
MR medium range
NPDWR National Primary Drinking Water Regulations
NPDES National Pollutant Discharge Elimination System
P plants
PV PhosVer
S soil
SCDB self-contained dropping bottle
TPTZ (2,4,6-Tri-(2-Pyridyl)-1,3,5-Triazine)
USEPA United States Environmental Protection Agency

Conversions

Conversion factors for many of the commonly used units of measure have been included to make the use of this manual more universal and to simplify calculations. Conversions are categorized by test.

Nitrogen

Nitrite (NO_2) = Nitrogen (N) x 3.28
Nitrate ($N0_3$) = Nitrogen (N) x 4.42
Ammonia (NH_3) = Nitrogen (N) x 1.22
Ammonium (NH_4) = Nitrogen (N) x 1.29

Phosphate

Phosphorus (P) = Phosphate (PO_4) x 0.326
Phosphorus Pentoxide (P_2O_5) = Phosphate (PO_4) x 0.75

Table 1. Hardness Conversion

Units of Measure	mg/l $CaCO_3$	British gr/gal (Imperial) $CaCO_3$	American gr/gal (US) $CaCO_3$	French parts/ 100,000 $CaCO_3$	German parts/ 100,000 CaO	meq/L*	g/L CaO	lb./cu ft $CaCO_3$
mg/L $CaCO_3$	1.0	0.07	0.058	0.1	0.056	0.02	$5.6x10^{-4}$	$6.23x10^{-5}$
English gr/gal $CaCO_3$	14.3	1.0	0.83	1.43	0.8	0.286	$8.0x10^{-3}$	$8.91x10^{-4}$
US gr/gal $CaCO_3$	17.1	1.2	1.0	1.72	0.96	0.343	$9.66x10^{-3}$	$1.07x10^{-3}$
Fr. p/100,000 $CaCO_3$	10.0	0.7	0.58	1.0	0.56	0.2	$5.6x10^{-3}$	$6.23x10^{-4}$
Ger. p/100,000 CaO	17.9	1.25	1.04	1.79	1.0	0.358	$1.0x10^{-2}$	$1.12x10^{-3}$
meq/L	50.0	3.5	2.9	5.0	2.8	1.0	$2.8x10^{-2}$	$3.11x10^{-3}$
g/l CaO	1,790.0	125.0	104.2	179.0	100.0	35.8	1.0	0.112
lb./cu ft $CaCO_3$	16,100.0	1,123.0	935.0	1,610.0	900.0	321.0	9.0	1.0

*or 'epm/L,' or 'mval/L'
N.B. 1 meq/L = N/1000

Oxygen, Dissolved

The following table lists the mg/L dissolved oxygen in water at saturation for various temperatures and atmospheric pressures. The table was formulated in a laboratory using pure water; thus, the values given should be considered as only approximations when estimating the oxygen content of a particular body of surface water.

Table 2. Dissolved Oxygen Saturation In Water

Pressure in Millimeters and Inches Hg										
Temp		775	760	750	725	700	675	650	625	mm
°F	°C	30.51	29.92	29.53	28.45	27.56	26.57	25.59	24.6	inches
32.0	0	14.9	14.6	14.4	13.9	13.5	12.9	12.5	12.0	
33.8	1	14.5	14.2	14.1	13.6	13.1	12.6	12.2	11.7	
35.6	2	14.1	13.9	13.7	13.2	12.9	12.3	11.8	11.4	
37.4	3	13.8	13.5	13.3	12.9	12.4	12.0	11.5	11.1	
39.2	4	13.4	13.2	13.0	12.5	12.1	11.7	11.2	10.8	
41.0	5	13.1	12.8	12.6	12.2	11.8	11.4	10.9	10.5	
42.8	6	12.7	12.5	12.3	11.9	11.5	11.1	10.7	10.3	
44.6	7	12.4	12.2	12.0	11.6	11.2	10.8	10.4	10.0	
46.4	8	12.1	11.9	11.7	11.3	10.9	10.5	10.1	9.8	
48.2	9	11.8	11.6	11.5	11.1	10.7	10.3	9.9	9.5	
50.0	10	11.6	11.3	11.2	10.8	10.4	10.1	9.7	9.3	
51.8	11	11.3	11.1	10.9	10.6	10.2	9.8	9.5	9.1	
53.6	12	11.1	10.8	10.7	10.3	10.0	9.6	9.2	8.9	
55.4	13	10.8	10.6	10.5	10.1	9.8	9.4	9.1	8.7	
57.2	14	10.6	10.4	10.2	9.9	9.5	9.2	8.9	8.5	
59.0	15	10.4	10.2	10.0	9.7	9.3	9.0	8.7	8.3	
60.8	16	10.1	9.9	9.8	9.5	9.1	8.8	8.5	8.1	
62.6	17	9.9	9.7	9.6	9.3	9.0	8.6	8.3	8.0	
64.4	18	9.7	9.5	9.4	9.1	8.8	8.4	8.1	7.8	
66.2	19	9.5	9.3	9.2	8.9	8.6	8.3	8.0	7.6	
68.0	20	9.3	9.2	9.1	8.7	8.4	8.1	7.8	7.5	
69.8	21	9.2	9.0	8.9	8.6	8.3	8.0	7.7	7.4	
71.6	22	9.0	8.8	8.7	8.4	8.1	7.8	7.5	7.2	
73.4	23	8.8	8.7	8.5	8.2	8.0	7.7	7.4	7.1	
75.2	24	8.7	8.5	8.4	8.1	7.8	7.5	7.2	7.0	
77.0	25	8.5	8.4	8.3	8.0	7.7	7.4	7.1	6.8	
78.8	26	8.4	8.2	8.1	7.8	7.6	7.3	7.0	6.7	
80.6	27	8.2	8.1	8.0	7.7	7.4	7.1	6.9	6.6	
82.4	28	8.1	7.9	7.8	7.6	7.3	7.0	6.7	6.5	
84.2	29	7.9	7.8	7.7	7.4	7.2	6.9	6.6	6.4	
86.0	30	7.8	7.7	7.6	7.3	7.0	6.8	6.5	6.2	
87.8	31	7.7	7.5	7.4	7.2	6.9	6.7	6.4	6.1	
89.6	32	7.6	7.4	7.3	7.0	6.8	6.6	6.3	6.0	
91.4	33	7.4	7.3	7.2	6.9	6.7	6.4	6.2	5.9	
93.2	34	7.3	7.2	7.1	6.8	6.6	6.3	6.1	5.8	
95.0	35	7.2	7.1	7.0	6.7	6.5	6.2	6.0	5.7	
96.8	36	7.1	7.0	6.9	6.6	6.4	6.1	5.9	5.6	
98.6	37	7.0	6.8	6.7	6.5	6.3	6.0	5.8	5.6	
100.4	38	6.9	6.7	6.6	6.4	6.2	5.9	5.7	5.5	
102.2	39	6.8	6.6	6.5	6.3	6.1	5.8	5.6	5.4	
104.0	40	6.7	6.5	6.4	6.2	6.0	5.7	5.5	5.3	
105.8	41	6.6	6.4	6.3	6.1	5.9	5.6	5.4	5.2	
107.6	42	6.5	6.3	6.2	6.0	5.8	5.6	5.3	5.1	
109.4	43	6.4	6.2	6.1	5.9	5.7	5.5	5.2	5.0	
111.2	44	6.3	6.1	6.0	5.8	5.6	5.4	5.2	4.9	
113.0	45	6.2	6.0	5.9	5.7	5.5	5.3	5.1	4.8	
114.8	46	6.1	5.9	5.8	5.6	5.4	5.2	5.0	4.8	
116.6	47	6.0	5.9	5.8	5.6	5.3	5.1	4.8	4.7	
118.4	48	5.9	5.8	5.7	5.5	5.3	5.0	4.8	4.6	
120.2	49	5.8	5.7	5.6	5.4	5.2	5.0	4.7	4.5	
122.0	50	5.7	5.6	5.5	5.3	5.1	4.9	4.7	4.4	

ACCURACY AND PRECISION

Accuracy is the nearness of a test result to the true value. Precision refers to the agreement of a set of replicate results or repeatability. Although good precision suggests good accuracy, precise results can be inaccurate. The following paragraphs describe techniques to improve accuracy and precision of analysis.

Standard Additions

Standard additions is a widely accepted technique for checking the validity of test results. Also known as "spiking" and "known additions," the technique also can be used to check the performance of the reagents, the instrument and apparatus, and the procedure.

Standard additions is performed by adding a small amount of a standard solution containing a known amount of the component being measured to an analyzed sample and repeating the analysis—using the same reagent, instrument and technique. The amount of increase in the test result should equal exactly the amount of component added.

For example, if testing shows a 25-mL water sample analyzed for iron contains 1.0 mg/L, the result can be checked by adding 0.10 mL of a 50.0-mg/L iron standard solution to another 25-mL portion of the water sample and repeating the analysis. The result of the analysis on the second sample should be 1.2 mg/L iron because the standard added an equivalent of 0.2 mg/L. For example:

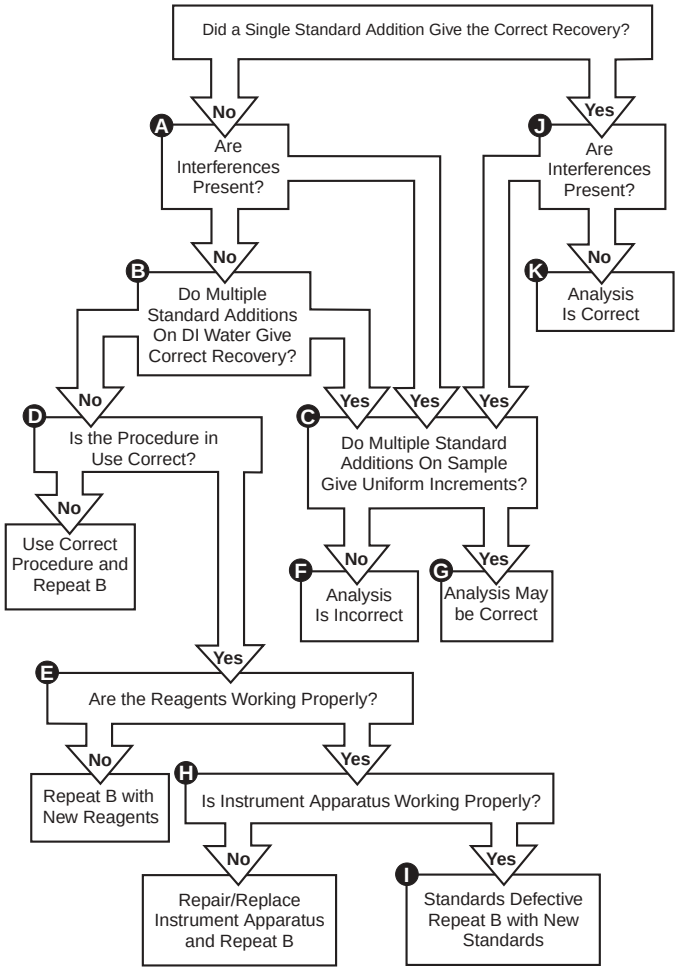
$$\frac{0.10\ \text{mL} \times 50.0\ \text{mg/L}}{25\ \text{mL}} = 0.2\ \text{mg/L}$$

If 0.2 mg/L is recovered from the 0.2 mg/L addition, the analyst can conclude the first answer was correct and the reagents, instrument and method used are all working properly. Because the effect of incremental volume additions is small, the sample volume used in the above equation was 25 mL (not 25 + 0.1). Using 25 mL, instead of 25.1 mL, represents less than 0.4% error. For 0.3 mL standard addition, the error would be less than 1.2% error.

If the second analysis does not give the correct amount of increase in the iron content, it must be concluded the first answer also may be incorrect. The analyst must determine why the technique did not work. The source of the problem can be determined by using a logical troubleshooting

approach whether the fault lies in the reagent, the instrument and apparatus, the test procedure or an interfering substance present in the test sample. A decision tree, such as the one in *Figure 1*, establishes a systematic method for identifying the problem. Request Hach Publication 7004 for additional information on standard additions. Explanations of the various steps follow.

Figure 1
Standard Additions
Decision Tree



Branch A
Suppose a single standard addition to the sample did not give the correct concentration increase. A possible cause could be interferences. Other causes could be defective reagents, an incorrect procedure, a defective instrument and apparatus or a defective standard used for standard additions. If interferences are known or assumed to be absent, proceed to Branch B. If interferences are known to be present, proceed to Branch C.

Branch B
Perform multiple standard additions on a sample of deionized water as in the following example:

1. Conduct an iron analysis on a 25.0-mL sample of deionized water.

2. Add 0.1 mL of a 50-mg/L iron standard solution to a second 25.0-mL sample of deionized water. Analyze this sample for iron.

3. Add 0.2 mL of a 50-mg/L iron standard solution to a third 25.0-mL sample of deionized water. Analyze this sample for iron.

4. Add 0.3 of a 50-mg/L iron standard solution to a fourth 25.0-mL sample of deionized water. Analyze this sample for iron.

5. Tabulate the data as shown below.

mL Std. Added	mg/L Std. Added	mg/L Iron Found
0	0	0
0.1	0.2	0.2
0.2	0.4	0.4
0.3	0.6	0.6

The data shown above indicates several points upon which the following conclusions may be made: First, the chemicals, instrument, procedures and standards are working correctly because iron added to the deionized water sample was recovered entirely in the same uniform steps of addition. Second, because iron added to deionized water was recovered, but was not recovered when an addition was made to an actual water sample (Branch A), the sample contains interferences which prevent the test reagents from operating properly. Third, the first sample analysis gave an incorrect result.

If the results of multiple standard additions gave the correct increments between additions, proceed to Branch C. If the results of multiple standard additions gave other than the correct increments between additions, proceed to Branch D.

Branch C
If interfering ions are present, the analysis may be incorrect. However, it may be possible, with multiple standard additions, to arrive at a close approximation of the correct result. Suppose the result of a sample analyzed for iron was 1.0 mg/L. The analyst, knowing interfering ions could be present, made one standard addition of 0.1 mL of 50-mg/L iron standard to 25.0 mL of sample. Rather than finding an increase of 0.2 mg/L as expected, the analyst found an increase of 0.1 mg/L. The analyst took a third and fourth water sample and added a standard addition of 0.2 and 0.3 mL, respectively. Samples were analyzed and results tabulated. If steps between each addition are roughly uniform (i.e., 0.1 mg/L difference between each addition), proceed to Branch G. If the results are not uniform (i.e., 0.1, 0.08, and 0.05 mg/L), proceed to Branch F.

Branch D
Carefully check the instructions or directions for use of the test, making sure the proper reagents are used in the proper order and time, the colorimeter is adjusted for the correct wavelength and calibration and the glassware in use is that specified. Be sure time for color development and the sample temperature are exactly as specified. If the procedure in use is found to be in error, repeat Branch B using the correct procedure. If the procedure is found to be correct, proceed to Branch E.

Branch E
Check the performance of the reagents. This may be done easily by obtaining a new fresh lot of reagent or by using a known standard solution to run the test. Make sure the color development time given in the procedure is equal to or greater than the time required for the reagent in question. If it is determined reagents are defective, repeat Branch B with new reagents. If the reagents are proven in good condition, proceed with Branch H.

Branch H
Check operation of the instrument and/or apparatus used in the performance of the test. Perform the wavelength and linearity checks

given in your instrument manual. Check glassware used in the procedure, making sure that it is scrupulously clean. Dirty pipets and graduated cylinders are sources of contamination and will not deliver the correct volumes. Hach's TenSette Pipet for dispensing Standards and standards sealed in Voluette Ampules are ideal for standard additions.

If a defect is found in the instrument and/or apparatus, repeat Branch B after repair or replacement of the instrument and/or apparatus. If the instrument and apparatus are found to be in good working order, proceed with Branch I.

Branch I
After determining the procedure, reagents, instrument and/or apparatus are correct and operating properly, an analyst may conclude the only possible cause for standard additions not functioning properly in deionized water is the set of standards used in performing the standard additions. Obtain a new set of standards and repeat Branch B.

Branch F
Examples of non uniform increments between standard additions are shown below.

Example A:

mL Std. Added	mg/L Std. Added	mg/L Found
0	0	1.0
0.1	0.2	1.10
0.2	0.4	1.18
0.3	0.6	1.23

Example B:

mL Std. Added	mg/L Std. Added	mg/L Found
0	0	0
0.1	0.2	0
0.2	0.4	0.2
0.3	0.6	0.4

The two examples illustrate the effect of interferences on the standard addition and on substances in the sample. Data plotted on the *Figure 2*

graph as A and B show that the four data points do not lie on a straight line. Plot A illustrates an interference becoming progressively worse as the concentration of the standard increases. This type of interference is not common and may be caused by an error or malfunction of the procedure, reagents or instrument. It is recommended Branch B be performed to verify the supposed interference.

Plot B illustrates a common chemical interference which becomes less or even zero as the concentration of the standard increases. The graph of the example shows the first standard addition was consumed by the interference and the remaining additions gave the correct incremental increase of 0.2 mg/L.

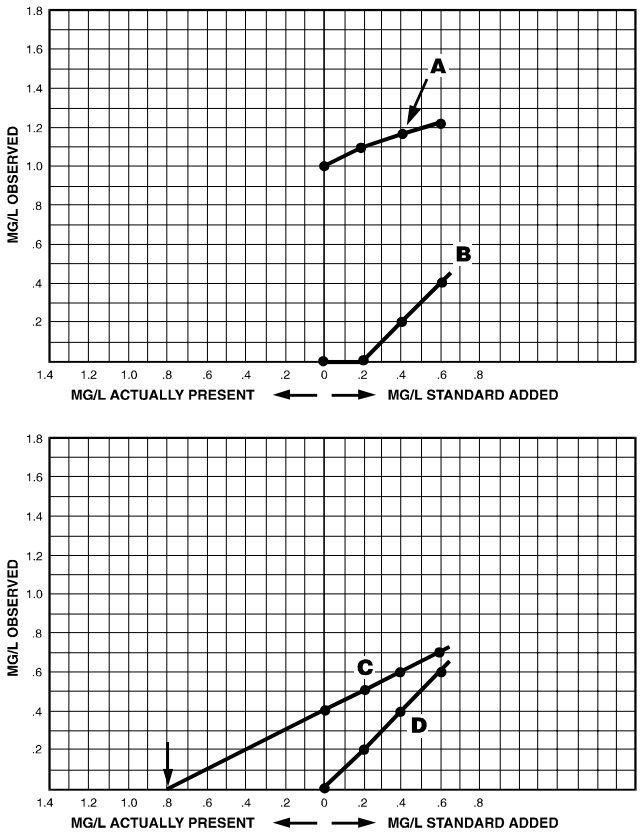


Figure 2 **Multiple Standard Additions Graph**

The apparent interference in Example B could be the result of an error made in the standard addition. The analysis should be repeated.

The two examples illustrate chemical interferences which most certainly mean the result of the first analysis of the water sample was incorrect. When this type of interference occurs, the analyst should attempt to analyze the sample with an alternate method which uses a different type of chemistry.

Branch G
Examples of uniform increments between standard additions are given below.

Example C:

mL Std. Added	mg/L Std. Added	mg/L Found
0	0	0.4
0.1	0.2	0.5
0.2	0.4	0.6
0.3	0.6	0.7

Plot C illustrates a common interference with a uniform effect upon the standard and the substances in the sample. The four data points form a straight line which may be extrapolated back through the horizontal axis. The point intersection with the horizontal axis can be used to determine the concentration of the substance in question. In the example, the first analysis showed 0.4 mg/L. The result located graphically should be much closer to the correct result: 0.8 mg/L.

Apparent interferences also may be caused by a defect in the instrument or the standards. Before assuming the interference is chemical in nature, check Branch B.

Example D:

mL Std. Added	mg/L Std. Added	mg/L Found
0	0	0
0.1	0.2	0.2
0.2	0.4	0.4
0.3	0.6	0.6

Plot D illustrates a problem for the analyst. Increments found are uniform and the recovery of the standard was complete. The result of the first analysis was 0 mg/L and the graph plots back through 0 mg/L. If interferences are known to be present, the interference may be present in an amount equal to the substance in question, thereby preventing the analyst from finding the substance. This would be an uncommon situation.

Branch J
If the standard addition gives the correct result, the analyst must then determine if interfering substances are present. If interfering substances are not present, the result of the analysis prior to the standard addition is correct. If interfering substances are present, proceed to Branch C.

One of the greatest aids to the analyst is knowledge of the sample's composition. An analyst need not know the exact composition of each sample but should be aware of potential interferences in the method of analysis to be used. When performing a particular method, the analyst should know if those interferences are present or not in order to have confidence in the accuracy of the results.

USEPA Approved

The United States Environmental Protection Agency (USEPA) establishes limits for maximum contamination levels for certain constituents in water. They also require that specific methodology be used to analyze for these constituents. These methods originate from several sources. The USEPA has developed some of these methods. In other cases the USEPA has evaluated and approved methods developed by manufacturers, professional groups, and public agencies such as APHA[1], AWWA[2] and WCPF[3] *(Standard Methods for the Examination of Water and Wastewater)*, ASTM[4], USGS[5] and AOAC[6]. All USEPA-approved methods are cited in the *Federal Register* and compiled in the *Code of Federal Regulations.*

USEPA Accepted

Hach has developed analytical methods that are equivalent to USEPA-approved methods. Even though minor modifications may exist, the USEPA has reviewed and accepted certain methods for reporting purposes. These methods are not published in the Federal Register, but are referenced to the equivalent USEPA method.

ADAPTING PROCEDURES

ADAPTING HACH PROCEDURES FOR USE WITH OTHER PHOTOMETERS

Hach test procedures can be used with other instrumentation if calibration curves are established to convert test results from % transmittance or absorbance to the concentration of the constituent being measured. Regardless of the instrument used, the sample and standardizing solutions are prepared the same way and the optimum wavelength specified in these procedures applies to testing with other spectrophotometers. In the example below, a sample calibration for iron concentrations of 0 to 2.4 mg/L is described. A series of iron standard solutions are prepared and measured to establish the calibration curve. The readings are plotted on semilogarithmic paper as % transmittance vs. concentration (or absorbance vs. concentration on linear-linear paper). Points on the graph shown (*Figure 3*) are connected with a smooth curve and the curve is used to make the calibration table if desired. The procedure follows:

1. Prepare several known concentrations with values covering the expected range. At least five standards are recommended. Run tests on 25-mL samples as described in the procedure. Then pour the customary volume of each known solution into separate, clean sample cells of the type specified for your instrument.

2. Select the proper wavelength and standardize the instrument using untreated sample water or a reagent blank as specified by the test procedure.

3. Measure each of the known solutions and plot the % transmittance readings on semilogarithmic graph paper as % transmittance vs. concentration. Plot the % transmittance values on the logarithmic (vertical) scale and the concentration values on the linear (horizontal) scale. In the following example, iron standard solutions of 0.1, 0.2, 0.4, 0.8, 1.2, 1.6 and 2 mg/L were measured on a Spectronic 20 Spectrophotometer at 500 nm. Half-inch test tubes were used. Results were plotted as shown on the graph (*Figure 3*) and the calibration table values (Table 3) were extrapolated from the curve.

To convert transmittance readings to mg/L iron, use Table 3 and select the appropriate line from the "% T Tens" column and the appropriate column from the "% T Units" group of columns. For example, if the

instrument reading was 46%, the 40 line and the 6 column would be selected. The test result would be 0.78 mg/L iron (Fe).

If in Step 3, absorbance values are measured, plot the results on linear-linear paper. Plot the absorbance value on the vertical scale and the concentration values on the horizontal scale. Increasing absorbance values are plotted from bottom to top and increasing concentration values are plotted from left to right. Values of 0.000 absorbance and 0 concentration will both begin in the bottom left corner of the graph. A calibration table can be extrapolated from the curve or concentration values and read directly from the curve in the graph.

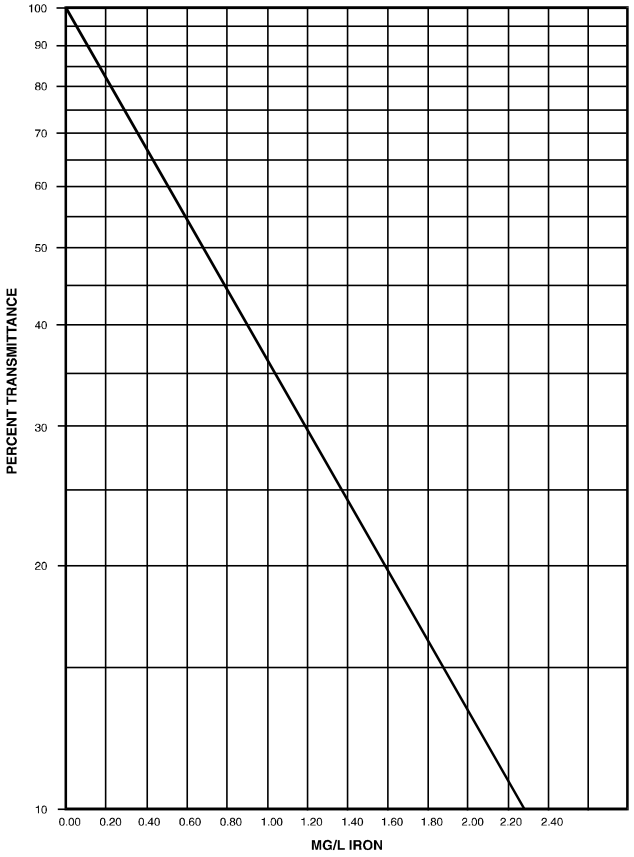


Figure 3 **Calibration Curve Example**

Table 3 Calibration Table

%T	%T UNITS									
Tens	0	1	2	3	4	5	6	7	8	9
0										
10	2.30	2.21	2.12	2.04	1.97	1.90	1.83	1.77	1.72	1.66
20	1.61	1.56	1.51	1.47	1.43	1.39	1.35	1.31	1.27	1.24
30	1.20	1.17	1.14	1.11	1.08	1.04	1.02	.99	.97	.94
40	.92	.89	.87	.84	.82	.80	.78	.77	.73	.71
50	.69	.67	.65	.64	.62	.60	.58	.56	.55	.53
60	.51	.49	.48	.46	.45	.43	.42	.40	.39	.37
70	.36	.34	.33	.32	.30	.29	.28	.26	.25	.24
80	.22	.21	.20	.19	.17	.16	.15	.14	.13	.12
90	.11	.09	.08	.07	.06	.05	.04	.03	.02	.01

Adapting a Buret Titration for Use With a Digital Titrator
Adapt any standard titration procedure using a buret to the Digital Titrator by using the following procedure.

1. Select a titration cartridge from Table 4 with the same active ingredient as the buret solution.

2. Determine the approximate number of digits required. The Digital Titrator dispenses 1 mL per 800 digits on the counter. Using the following equation, determine the digits required for your buret method.

$$\text{Digits Required} = \frac{N_t \times mL_t \times 800}{N_c}$$

Where:
N_t = Normality of buret Titration
mL_t = milliliters of buret titrant required for an average titration
N_c = Normality of Digital Titrator cartridge

3. If the number of digits required is within the range of 70 to 350, you can use the procedure as written, substituting the Digital Titrator directly for the buret. Or, if the number of digits is outside of this range, make the following modifications.

a. If the number of digits required is more than 350, reduce the sample size to save titrant.

b. If the number of digits required is less than 70, increase the sample size to increase precision.
c. If the sample size is altered, adjust the amount of buffering or indicating reagents by the same proportion.

4. When using the Digital Titrator for your buret method, note the number of digits required for a sample titration. To convert the digits required to the equivalent number of milliliters in the buret method was used, calculate:

$$\text{Equivalent Buret Milliliters} = \text{Digits Required} \times \frac{N_c}{800 \times N_t}$$

If the sample size was changed, adjust the equivalent buret milliliters accordingly. If the sample size was increased, reduce the equivalent buret milliliters; if the sample size was reduced, increase the equivalent buret milliliters. Multiply the equivalent milliliters by any normally used factors to calculate concentration in oz/gal, g/L, etc.
Example: Adapt a buret procedure which normally requires about 20 mL of a 0.4 N titrant to the Digital Titrator. Try a 8.0 N titration cartridge. The first equation above gives:

$$\text{Digits Required} = \frac{0.4 \times 20 \times 800}{8.0} = 800 \text{ digits}$$

Because this would use excessive titrant, reduce the sample size to one-fourth its normal size to reduce the digits required to 200, well within the recommended range.

Upon completion of the titration using the smaller sample size, calculate the equivalent buret milliliters by the second equation above. If 205 were the digits required:

$$\text{Equivalent Buret Milliliters} = \frac{205 \times 8.0}{800 \times 0.4} = 5.13 \text{ mL}$$

Multiply the 5.13 mL by four to account for the reduction in sample size to give the true equivalent buret milliliters of 20.5 mL. If the buret method called for multiplying the number of milliliters of titrant by a factor to calculate the concentration of a sample component, then multiply 20.5 by that factor.

Table 4. Titration Cartridges

Description	Cat. No.
CDTA, 0.0800 M, HexaVer	14402-01
CDTA, 0.800 M, HexaVer	14403-01
Ceric Standard Solution, 0.5 N	22707-01
EDTA, 0.0499 M	21253-01
EDTA, 0.0800 M, TitraVer	14364-01
EDTA, 0.142 M	14960-01
EDTA, 0.714 M	14959-01
EDTA, 0.800 M, TitraVer	14399-01
FEAS, ferrous ethylenediammonium sulfate, 0.00564 N	22923-01
Hydrochloric Acid, 8.00 N	14390-01
Iodate-Iodide, potassium, 0.3998 N	14961-01
Iodate-Iodide, potassium, 1.00 N	22944-01
Magnesium Chloride, 0.0800 N	20625-01
Mercuric Nitrate, 0.2256 N	14393-01
Mercuric Nitrate, 2.256 N	921-01
PAO, phenylarsine oxide, 0.00451 N	22599-01
PAO, phenylarsine oxide, 0.0451 N	21420-01
Potassium Dichromate, 1.00 N	21971-01
Potassium Thiocyanate, 1.00 N	22959-01
Silver Nitrate, 0.2256 N	14396-01
Silver Nitrate, 1.128 N	14397-01
Sodium Hydroxide, 0.1600 N	14377-01
Sodium Hydroxide, 0.3636 N	14378-01
Sodium Hydroxide, 0.9274 N	14842-01
Sodium Hydroxide, 1.600 N	14379-01
Sodium Hydroxide, 3.636 N	14380-01
Sodium Hydroxide, 8.00 N	14381-01
Sodium Thiosulfate, Stabilized, 0.02256 N	24091-01
Sodium Thiosulfate, 0.113 N	22673-01
Sodium Thiosulfate, 0.2000 N	22675-01
Sodium Thiosulfate, 0.2068	22676-01
Sodium Thiosulfate, 2.00 N	14401-01
Sodium Vanadate, 0.25 N	22949-01
Sulfuric Acid, 0.1600 N	14388-01
Sulfuric Acid, 1.600 N	14389-01
Sulfuric Acid, 8.00 N	14391-01

INTERFERENCES

Many analytical determinations are subject to interference from substances that may be present in the sample. Most common interferences are mentioned either in the test procedures or in the accompanying notes. Our reagent formulations eliminate many interferences and others are removed by special sample pretreatments described in the procedure.

Interference also may be caused by a high concentration of the constituent under analysis. For example, the presence of a larger excess of chlorine will cause the test to read less than full scale. Dilution of the sample to 5 mg/L will result in a reading higher than full scale. This indicates the need for more dilution until the instrument reading is "on scale."

When an unusual answer is obtained, a color other than that expected is formed, or an unusual odor or turbidity is noticed, the result is suspect. Repeat the test on a sample diluted with deionized water; see *Sample Dilution Techniques*. Compare the result (corrected for the dilution) with the result of the original test. If these two are not identical, the original result probably is in error and an additional dilution should be made to check the second test (first dilution). This process is repeated until the same corrected result is obtained on two successive dilutions.

More complete information about interferences and methods to overcome them is contained in the General Introduction Section of APHA *Standard Methods.* The analyst is urged to obtain this book and refer to it when problems are encountered.

pH Interference

Many of the procedures in this manual are pH dependent. Hach reagents contain built-in buffers to adjust the pH of the typical sample to the correct pH range. However, the reagent buffer capacity may not be sufficient for some unusual samples. This occurs most often with highly buffered samples or samples with extreme sample pH. Check for pH interference in the following manner:

1. From the Sampling and Storage section of your procedure determine the optimum pH range of the test. This is the pH the preserved sample is adjusted to just before running the test. For some procedures this information may not be given. If the pH of your sample is within the optimum pH range, buffering is not needed.

2. Measure the pH of your analyzed sample with a pH meter.

3. Prepare a reagent blank using deionized water as the sample, add all reagents called for in the procedure. Timer sequences, etc., may be ignored. Mix well.

4. Measure the pH of the reagent blank with a pH meter.

5. Compare the pH values of your analyzed sample with the reagent blank.

6. If there is no difference in the pH values of your analyzed sample and the reagent blank, then pH interference is not the problem. Follow the Accuracy Check given in the procedure to more clearly identify the problem.

7. If there is a significant difference between the values of your analyzed sample and the reagent blank, adjust the sample pH to within the optimum pH test range, or if none is given, to the value of the reagent blank before analysis on all future determinations. Use the appropriate acid, usually nitric acid, to lower the pH, and use the appropriate base, usually sodium hydroxide, to raise the pH.

8. Analyze the sample as before.

Interference From Stray Light

Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. If a 25-mL cell is used in the procedure, transfer 10 mL of the solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.

LABORATORY PRACTICE

Boiling Aids

Boiling is included as a necessary step in some procedures. It may be convenient to use a boiling aid such as boiling chips, Cat. No. 14835-31, to reduce bumping. Bumping is caused by the sudden, almost explosive conversion of water to steam as it is heated. Bumping may cause sample loss or a hazardous condition and should be avoided.

All boiling aids used should be checked to verify they will not contaminate the sample. Boiling aids (except glass beads) should not be used again. Loosely covering the sample during boiling will prevent splashing, reduce the possibility of contamination and minimize sample loss.

Filtration of Samples

Filtering is the process of separating particles from the sample by using a medium, generally filter paper, to retain particles but allow the

solution to pass through. This is especially helpful when sample turbidity interferes with calorimetric analysis. Two general methods of filtration are gravity and vacuum. Gravity filtration uses the force of gravity to pull the sample though the filter paper. Vacuum filtration uses the pressure difference created by either an aspirator or vacuum pump plus the force of gravity to move the sample through the filter. Vacuum filtration is faster than gravity filtration. Vacuum filter (see *Figure 4*) as follows:

1. Place a filter paper into the filter holder.

2. Place the filter holder assembly in the filtering flask and wet the filter with deionized water to ensure adhesion to the holder.

3. Position the funnel housing on the filter holder assembly.

4. While applying a vacuum to the filtering flask, transfer the sample to the filtering apparatus.

5. Slowly release the vacuum from the filtering flask and transfer to another container.

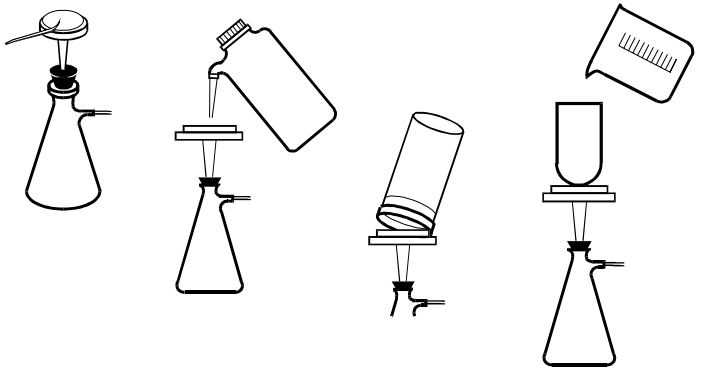


Figure 4 **Vacuum Filtration**

REQUIRED APPARATUS

Description	Unit	Cat. No.
Filter Discs, glass 47 mm	100/pkg	2530-00
Filter Holder, membrane	each	2340-00
Flask, filter, 1000 mL	each	546-53

Many of the procedures in this manual may be filtered with gravity filtration. The only labware required is filter paper, a conical funnel and a receiving flask. This labware is included under the Optional Apparatus listing for each procedure. Gravity filtration provides better retention of fine particles. For optimum filtering speed, add solution until the filter paper cone is three-fourths filled. Never fill the cone completely. Gravity filter (see *Figure 5*) as follows:

1. Place a filter paper into the funnel.

2. Wet the filter with deionized water to ensure adhesion to the funnel.

3. Place the funnel into an erlenmeyer flask or graduated cylinder.

4. Pour the sample into the funnel.

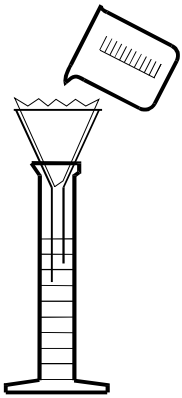


Figure 5 **Gravity Filtration**

REQUIRED APPARATUS

Description	Unit	Cat No.
Cylinder, graduated, 100 mL	each	508-42
Funnel, poly, 65 mm	each	1083-67
Filter Paper, 12.5 cm	each	1894-57
Flask, erlenmeyer, 125 mL	each	505-43

The determination of metals requires acid and heat to pretreat the sample. Because filter paper will disintegrate under these conditions, vacuum filtration with glass fiber filter discs is recommended. Also, glass filter discs do not retain colored species as filter paper would.

Reagent and Standard Stability

Most chemicals and prepared reagents do not deteriorate after manufacture, but storage conditions and packaging have a great influence on their stability. Absorption of moisture, carbon dioxide or other gases from the atmosphere, bacterial action, or light (with photosensitive compounds) may affect the reagent shelf life. In some cases reaction with the storage container or interaction of reagent components may occur.

Hach strives continually to prepare stable formulations and devise ways of packaging them to provide maximum protection. Many unique Hach formulations, methods of analysis and forms of packaging have resulted from these challenges.

Chemicals supplied by Hach have an indefinitely long shelf life when stored under average room conditions, unless designated otherwise. Notations on product labels specify any special storage conditions required. Otherwise, reagents should be stored in a cool, dry, dark place for maximum life. It is always good practice to date chemicals upon receipt and rotate supplies so the older supplies are used first. If in doubt about the reagent shelf life, run a standard to check reagent effectiveness.

Reagent Blank

The term "reagent blank" refers to that portion of the test result contributed solely by the reagent and not the sample. This portion of the test result represents a positive error. In several of the tests, the reagent blank is of such magnitude that compensation must be made each time

the test is performed. This is done by zeroing the instrument on deionized water and reagents.

In most cases, the reagent blank is so small the instrument is zeroed on either an untreated portion of the original water sample or deionized water. This is done routinely without any significant loss of accuracy except where extremely small amounts of a constituent are sought. In such a case, it is best to determine the reagent blank by performing the test on a sample of high-quality, turbidity-free deionized water. The result is expressed in the concentration units of the test and is subtracted from the test results of subsequent samples using that particular lot of reagent. It is necessary to determine the reagent blank only at first use and at intervals of several months unless subsequent contamination is suspected.

Every effort is made to produce reagents with the lowest possible blank. In most cases, it is less than 0.009 absorbance units. In some instances, it is either impossible or not practical to produce reagents with such a low blank. In these cases, it is best to determine the reagent blank as explained above and subtract it from each determination. A note is included in the appropriate procedures describing when this is necessary.

Safety

Safety is the responsibility of each individual when performing analysis procedures, and the analyst must develop and maintain good safety habits. Because many of the procedures in this methods manual require the use of potentially hazardous chemicals and apparatus, it is important for the individual conducting them to minimize chances for accidents by practicing good laboratory techniques. Several rules applying to water analysis in the laboratory and in the field follow. They are not all inclusive, but they emphasize practices that often are key factors in personal injury incidents.

Read Labels Carefully: Each reagent label should be read carefully with particular attention paid to the precautionary information. Never remove the label from a reagent container while it contains reagent. Do not put a different reagent into a labeled container without changing the label. When preparing a reagent or standard solution, be sure to label the container clearly.

Warning labels also appear on some of the apparatus used with the test procedures. The protective shields with the COD Reactor and the Digesdahl Digestion Apparatus point out potential hazards. Be sure these shields are in place during use and observe the precautions they recommend.

Wear Protective Clothing: Protective clothing should be worn when handling chemicals that cause irritation or burns. When caustic materials are being used, eye protection, in particular, is important to guard against spattering and splashes from accidental spills.

Use tongs or finger cots when transferring hot apparatus.

Use Mechanical Pipettors: Never pipet by mouth. Mouth pipetting could result in accidentally ingesting a dangerous chemical. Make a habit of using mechanical pipetting devices for all pipetting. Mistakes that could cause serious injury will be avoided.

Use Special Care With Dangerous Chemicals and Apparatus: Follow the test procedure steps carefully and observe all precautionary measures. It is good practice to read the entire procedure carefully before beginning the procedure. Use the safety equipment—such as pipet fillers, protective clothing and ventilating hoods—appropriate for the test being conducted. Wipe up all spills promptly. Do not smoke or eat in an area where toxic or irritating chemicals are used. Use reagents and apparatus only as they were meant to be used and use them only as directed in the test procedure. Damaged labware and malfunctioning equipment should not be used.

If accidental skin contact with hazardous chemicals occurs, flush the contacted area with water for 15 minutes. Call a physician if necessary. A MSDS (Material Safety Data Sheet) accompanies the first shipment of all products. Refer to the MSDS for safety data essential for day-to-day operations and safety training.

Sample Cell Matching

The sample cells provided with the DR/700 Colorimeter are not optically perfect. Glass imperfections can introduce an error in the true absorbance or percent transmittance measurement. In turn, the true Absorbance or % T error can result in reduced accuracy. For optimum

accuracy and precision, sample cells should be matched prior to use. Refer to Matching Sample Cells in the instrument manual supplied with the colorimeter.

Sample Dilution Techniques

Ten and Twenty-five milliliter (mL) are the specified volumes for most colorimetric tests. However, in some tests, the color developed in the sample may be too intense to be measured. Unexpected colors may develop in other tests. In both cases, it is necessary to dilute the sample or determine if interfering substances are present.

For example, when performing the chromium tests, the colorimeter may detect a concentration above the maximum range limit. This results in a flashing maximum concentration value in the display. A sample solution is necessary. The test can be repeated, for example, with a 25-mL graduated cylinder filled to the 5-mL mark with the sample and then to the 10-mL mark with deionized water. Because the sample was diluted to twice its original volume (5 mL to 10 mL), the test result should be multiplied by 2 to give the correct concentration of chromium.

To accomplish the sample dilution conveniently, pipet the chosen sample portion into a clean graduated cylinder (or clean volumetric flask for more accurate work) and fill the cylinder (or flask) to the desired volume with deionized water. Mix well. Use the diluted sample when running the test.

As an aid, Table 5 shows the amount of sample taken, the amount of deionized water used to bring the volume up to 25 mL and the multiplication factor.

Table 5. Sample Dilutions

Sample Volume (mL)	Deionized Water Used to Bring the Volume to 25 mL (mL)	Multiplication Factor
25.0	0.0	1
12.5	12.5	2
10.0*	15.0	2.5
5.0*	20.0	5
2.5*	22.5	10
1.0*	24.0	25
0.250*	24.75	100

*For sample sizes of 10 mL or less, a pipet should be used to measure the sample into the graduated cylinder or volumetric flask.

The concentration of the sample is equal to the diluted sample reading times the multiplication factor.

An example: A 2.5 mL sample was diluted with 22.5 mL of deionized water. The result was 0.35 mg/L. What is the concentration of the sample?

$$0.35 \times 10 = 3.5 \text{ mg/L}$$

More accurate dilutions can be done with a pipet and a 100-mL volumetric flask. Pipet the sample and dilute to volume with deionized water. Invert several times to mix.

Table 6. Multiplication factors to be used when sample is diluted to 100 mL

Sample Volume (mL)	Multiplication Factor
1	100
2	50
5	20
10	10
25	4
50	2

Sample dilution also influences the level at which a substance may interfere. The effect of the interferences decreases as the sample size decreases. Therefore, the effect of the interference described in the procedure notes will decrease as the sample size decreases. In other words, higher levels of an interfering substance can be present if the sample is diluted.

An example: Copper does not interfere at or below 100 mg/L for a 25.00 mL sample in a procedure. If the sample volume is diluted with an equal volume of water, what is the level at which copper will not interfere?

$$\frac{\text{Total Volume}}{\text{Sample Volume}} = \text{Dilution Factor}$$

$$\frac{25}{12.5} = 2$$

$$\text{Interference level} \times \text{Dilution factor} = \text{Interference level in sample}$$

$$100 \times 2 = 200 \text{ mg/L}$$

The level at which copper will not interfere in the sample is at or below 200 mg/L.

Temperature Considerations
For best results, most tests described in this manual should be performed with sample temperatures between 20 °C (68 °F) and 25 °C (77 °F). If certain tests require closer temperature control, that requirement will be indicated in notes following those procedures.

Use of Pipets and Graduated Cylinders
When small sample quantities are used, the accuracy of measurements is important. *Figure 6* illustrates the proper way of reading the sample level or the meniscus formed when the liquid wets the cylinder or pipet walls.

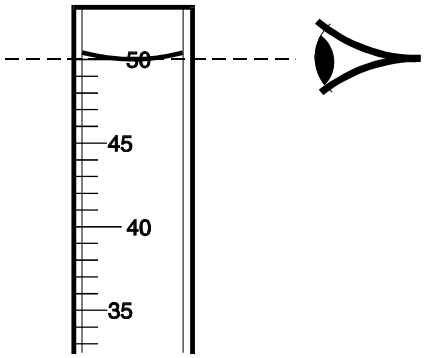


Figure 6 **Reading the Meniscus**

Rinse the pipet or cylinder two or three times with the sample to be tested before filling. Use a pipet filler or pipet bulb to draw the sample into the pipet. Never pipet chemical reagent solutions or samples by mouth. When filling a pipet, keep the tip of the pipet below the surface of the sample as the sample is drawn into the pipet.

Serological pipets are long tubes with a series of calibrated marks to indicate the volume of liquid delivered by the pipet. The calibrated marks may extend to the tip of the pipet or may be only on the straight portion of the tube. Fill serological pipets to the zero mark and discharge the sample by allowing the sample to drain until the meniscus is level with the desired mark. If the serological pipet has calibrated marks extended to the tip of the pipet, the sample must be blown out of the tip for accurate sample measurements.

Volumetric (transfer) pipets are long tubes with a bulb in the middle and a single ring above the bulb to indicate the volume of liquid to be delivered when it is filled to the mark. To discharge the sample from a volumetric pipet, hold the tip of the pipet at a slight angle against the container wall and drain. Do not attempt to discharge sample or reagent remaining in the tip of the pipet after draining. Volumetric pipets are designed to always retain a small reproducible amount of sample in the tip of the pipet.

Use of AccuVac Ampuls

AccuVac ampuls contain pre-measured reagent in optical-quality glass ampuls. The sample is collected in a beaker or other open container. The ampul tip is immersed stem first well below the sample surface and the tip is broken off *(see Figure 7)*. The break must be far enough below the surface to prevent air from being drawn in as the level of the sample lowers. The ampul is inverted several times to dissolve the reagent powder (capping is unnecessary). Test results are not affected by undissolved powder. Wipe the ampul with a towel to remove fingerprints, etc. Insert the ampul into the AccuVac adapter into the colorimeter sample compartment and read the results directly.

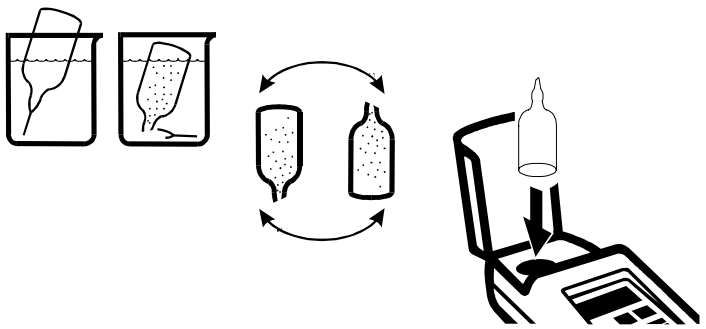


Figure 7 **Using AccuVac Ampuls**

Use of the DR/700 AccuVac Vial Adapter

For safety and ease of use, the DR/700 AccuVac Vial Adapter is provided with the instrument for use with Hach Company's AccuVac Ampul Reagents. Insert the adapter in the cell compartment by aligning

it about 1/4 inch out from the slot in the front side of the cell holder as in *Figure 8*. Push the adapter against the slot sides to seat it. to be sure the adapter is seated, gently slide it upward. The adapter should stop about half way up and stay there.

For measurements, leave the adapter in the up position and insert an AccuVac Ampul in the cell holder (see *Figure 9).* Taking care to avoid sharp edges, gently push the ampule down until it stops. This centers the ampul in the light path. Pushing down on the ampul will not fully seat it. To remove the ampul, pull the adapter up with the side tabs, then pull the ampul out.

The adapter should be removed before testing with round sample cells to allow alignment with the mark on the cells with the tab on the cell holder. To remove the adapter, tilt the top toward the front of the instrument and then pull upwards.

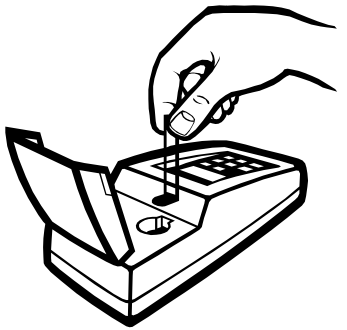


Figure 8 **Inserting AccuVac Vial Adapter**

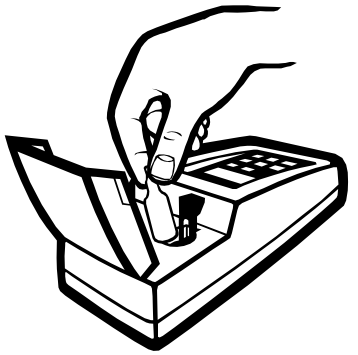


Figure 9 **Inserting AccuVac Ampul**

Use of Reagent Powder Pillows

Dry powdered reagents are used when possible to minimize problems of leakage and deterioration. Powders are packaged in individual, pre-measured, polyethylene "powder pillows." Each pillow contains enough reagent for one test and is opened easily with nail clippers or scissors; *see Figure 10*.

Figure 10 **Opening Powder Pillows**

Using PermaChem Pillows

For best results, slightly tap the pillow on a hard surface to collect the powdered reagent in the bottom. Then:

1. Tear across, from A to B, holding the pillow away from your face.

2. Using two hands, push both sides toward each other to form a spout.

3. Pour the pillow contents into the sample cell and continue the procedure according to the instructions.

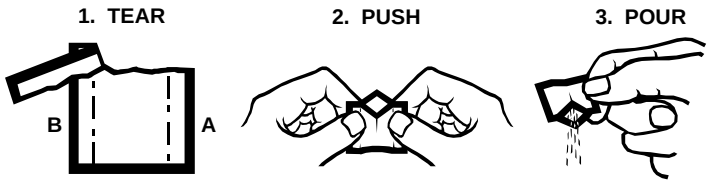


Figure 11 **Using PermaChem Pillows**

Using the TenSette Pipet
For best results, always use a new tip for each pipetting operation. After being used several times, the pipet tip may retain some liquid, causing an error in delivery, Each pipet is supplied with 100 tips. Order Hach replacements, for best results.

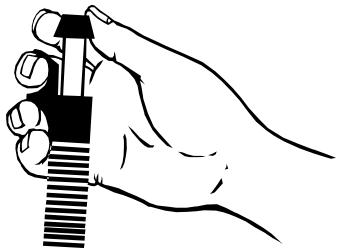
Always use careful and even hand movements for best reproducibility. If the pipet does not operate smoothly, disassemble and coat the piston and retainer with high-quality stopcock grease. The metering turret also may be lightly coated with grease. *Refer to the manual supplied with the TenSette Pipet for more information.*

For best accuracy, both the room temperature and the solution being pipetted should be between 20 and 25°C. Avoid palming the pipet an unnecessarily long time prior to use because the aliquot volume could be affected by elevated temperatures.

Never lay the pipet down with solution in the tip. Solution could leak into the pipet and cause corrosion.

Operating Instructions
1. Attach a clean tip. Holding the TenSette in one hand, gently press the tip onto the tapered nose of the pipet until the tip is held firmly and a good seal is obtained.

2. Turn the turret cap to align the desired volume on the volume-setting ring with the mark on the housing assembly.

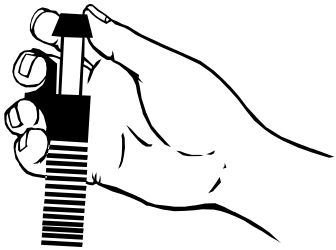


3. Press down on the turret cap with the thumb, using a smooth motion, until the turret reaches the stop. Immerse the tip about 5 mm (¼") below the surface of the solution to avoid drawing air into the tip. Do not insert the tip any deeper, or the delivery volume may be affected.

4. While maintaining a constant pressure, allow the turret to return to the extended position. Do not let the turret snap into place, or the delivery volume may be affected.

5. With the turret up, withdraw the tip from the liquid and move it to the receiving vessel. Avoid placing pressure on the cap while moving the pipet.

6. Use the thumb and forefinger to twist the turret cap to the next higher position on the volume-setting ring to assure full blowout and quantitative transfer of the sample. The "F" position provides full blowout for the 1.0-mL setting.



7. With the tip in contact with the side of the receiving vessel, slowly and smoothly press down on the cap until the turret reaches the stop and the solution is completely discharged.

Mixing Water Samples
The following two methods may be helpful in tests that require mixing sample with chemicals (usually indicated by "swirl to mix" instructions).

1. A swirling motion is recommended when mixing sample in a graduated cylinder or a titration flask. In this case, grip the cylinder (or flask) firmly with the tips of three fingers; *see Figure 12.* Hold the cylinder at a 45-degree angle and twist the wrist. This motion will move the cylinder in an approximately 12-inch circle, giving the liquids an intense rotation to accomplish complete mixing in a few turns.

This swirling procedure is the most gentle and offers the least interference from the atmosphere when testing for carbon dioxide and other gases. Both methods are simple but take a bit of practice in order to obtain the best results.

2. When mixing sample in a square sample cell, the swirling motion is attained by a simple twisting motion; *see Figure 13*. Grasp the neck of the cell with the thumb and index finger of one hand while resting the concave bottom of the cell on the tip of the index finger of other hand. Rotate the cell quickly, first one way and then the other, to mix the sample.

Figure 12 **Swirling Graduated Cylinder**

Figure 13 **Swirling Square Mixing Bottle**

Volume Measurement Accuracy

Sample cells supplied with the colorimeter are marked to indicate approximately 10 mL or 10, 20, and 25 mL. In most tests where volume measurements are critical, the procedure specifies the appropriate method.

If a sample must be diluted, use a pipet for volume measurement. Accuracy is important because a slight mistake in measuring a small sample will cause a substantial error in the result, For instance, a 0.1-mL mistake in the measurement of a 1.0-mL sample produces a 10% error in the test result.

SAMPLE PRETREATMENT

Digestion

Digestion, required in several procedures, refers to the use of acid 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 an absolute process that yields a digest suitable for the determination of metals, total phosphorus and total kjeldahl nitrogen (TKN). It is rapid and convenient. It is the method of choice.

For EPA reporting purposes, EPA-approved digestions are required. EPA presents two digestions (mild and vigorous) for metals analysis. These are much more inconvenient and time consuming compared to the Hach Digesdahl system. Other tedious digestion procedures are required for phosphorus and TKN.

Hach Digesdahl Digestion

In this procedure (pages 38-42) 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 analyses using colorimetric method, see Procedures (Section II).

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 (HCI).

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 below) 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 in appearance with continued refluxing).

6. Again, evaporate to 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 7 below.

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 7 below for the size of flask.

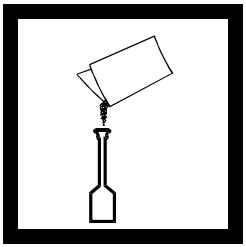
8. Adjust the sample to pH 4 by drop-wise addition of 5.0 N Sodium Hydroxide Standard Solution; mix thoroughly and check the pH after each addition. Dilute to volume with deionized water. Multiply the

result by the correction factor in column 5 of the table below. A reagent blank also should be carried through the digestion and measurement procedures.

Table 7. Vigorous Digestion

Expected Metal Concentration	Suggested Sample for Digestion	Suggested Volume 1:1 HC1	Suggested Final Volume After Digestion	Correction Factor
1 mg/L	50 mL	10 mL	200 mL	4
10	5	10	200	40
100	1	25	500	500

GENERAL DIGESDAHL DIGESTION



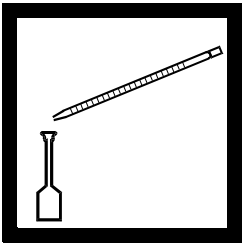
1. Transfer a preweighed or a premeasured amount of sample into a 100-mL volumetric flask; see Table 8. The amount transferred should not contain more than 0.5 g of solids or organic liquids. The maximum volume for water samples is 50 mL. In samples with more than 1% solids present, use the formula below:

$$\text{Water Sample Volume (mL)} = \frac{50}{\%\ \text{solids}}$$

Example: *If solids are 10% of total volume of sample, the maximum volume of liquid sample would be 5 mL.*

Note: *Several 50-mL sample aliquots of the sample may be digested in succession to concentrate a sample.*

Note: *If liquid is too viscous to measure, preweigh the sample into the digestion flask.*



2. Add concentrated sulfuric acid according to Table 8 to the volumetric flask and two or more silicon carbide (Carborundum) boiling chips for liquid samples.

Note: *Boiling chips can be pretreated by soaking in 1:1 nitric acid and rinsing thoroughly with deionized water. Treatment may be particularly important in low level work. Silicon carbide boiling chips are recommended.*

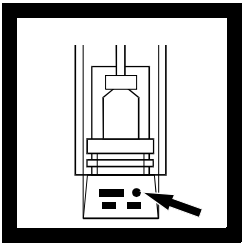
Note: *Use only Hach digestion flasks. Volumetric flasks with concave bottom should* ***not*** *be used.*

Warning

A safety shield placed between the operator and the Digesdahl is required. Safety glassed are mandatory.

Caution

Experimentation with the Digesdahl Apparatus is not recommended. See Safety Considerations following these steps.

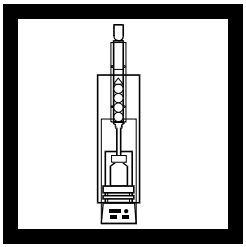


3. Turn on the water to the aspirator and make sure there is suction to the fractionating column. Turn the temperature dial to a heat setting of 440 °C (825 °F). For meat digestion, set to 468 °C (875 °F).

Note: *Wait for the proper temperature to be reached before sample is placed on the heater.*

Note

Specific method manuals for a variety of sample types are available, free of charge from Hach Company. See Application - Specific Manuals following these steps for a complete listing. New methods are continually being developed. Please contact Hach Company World Headquarters, (303) 669-3050, for a current listing.

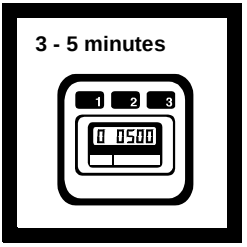


4. Place the flask weight followed by the fractionating column with funnel on the flask. Place the flask on the heater and heat until the boiling point of sulfuric acid is reached (refluxing sulfuric acid will be visible).

Note: *White acid vapors usually will be present but their presence alone does not indicate that the boiling point of sulfuric acid has been reached.*

Note: *Liquid samples require total evaporation of water before vapors are visible.*

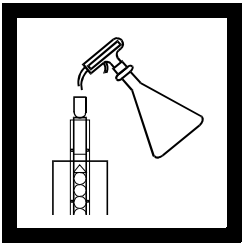
Note: *If sample starts to foam up into the neck of the flask, lower temperature to 335 °C (600 °F). Continue heating at lower temperature until all water is evaporated off. Then return to original digestion temperature.*



5. Heat 3-5 minutes. Do not boil sample to dryness.

Note: *Discard sample if it evaporates to dryness and use larger amount of concentrated sulfuric acid for digestion in Step 2.*

Note: Some organic samples may need more than five minutes for complex digestion. See Table 8.



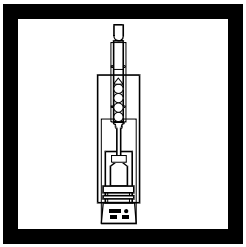
6. Be sure you have added the correct amount of sulfuric acid. Add 10 mL of 50% hydrogen peroxide to the charred sample via the funnel on the fractionating head.

Note: If the digest does not turn colorless, add 5 mL increments of peroxide until the digest becomes clear.

Note: Visually confirm the presence of sulfuric acid in the flask before adding hydrogen peroxide.

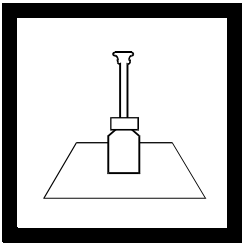
Note

If foaming or bumping is not stopped by lowering temperature or volume, then liquid samples that will not clog the capillary funnel may be added to the flask via the capillary funnel, 10 mL at a time. Decrease amount added if foaming still persists.



7. Boil off excess hydrogen peroxide by heating for one more minute after addition of hydrogen peroxide is complete. Do not heat to dryness.

Note: *If the sample goes to dryness, turn off the Digesdahl and cool completely. Add water to flask before handling. Repeat digestion from the beginning.*



8. Take the flask off the heater and allow the flask to cool. Remove the fractionating column from the digestion flask.

Note: *Use finger cots to remove the digestion flask. Place it on a cooling pad for at least one minute. Then remove the column.*

REQUIRED REAGENTS

Description	Quantity Per Digestion	Unit	Cat. No.
Hydrogen Peroxide, 50%	10 mL	500 mL	21196-49
Potassium Hydroxide Standard Solution, 1 N	varies	59 SCDB*	23144-26
Potassium Hydroxide Standard Solution, 8 N	varies	500 mL	282-49
Sulfuric Acid, ACS (concentrated, specific gravity 1.84)	≥3 mL	4 kg	979-09
Water, deionized	varies	3.78 L	272-17

REQUIRED APPARATUS

Dispenser, pour-out, 10 mL	1	each	22200-38
Pipet, serological, 10 mL	1	each	532-38
Pipet Filler, safety bulb	1	each	14651-00
Boiling Chips, silicon carbide	varies	500 g	20557-34

REQUIRED APPARATUS (continued)

Description	Quantity Per Digestion	Unit	Cat. No.
Safety Glasses	1	each	18421-00
Safety Shield, for Digesdahl	1	each	20974-00
Select one based on available voltage:			
Digesdahl Apparatus, 115 Vac	1	each	23130-20
Digesdahl Apparatus, 230 Vac	1	each	23130-21

OPTIONAL REAGENTS

Kjeldahl Reduction Reagent (for fluid fertilizers)	40 g	23653-04
2,4-Dinitrophenol Indicator Solution	100 mL MDB	1348-32
Nitric Acid Solution, 1:1	500 mL	2540-49
Potassium Hydroxide, 1 N	59 mL MDB	23144-26
Sodium Hydroxide, 5 N	59 mL* SCDB	2450-26
Sodium Hydroxide, 1 N	100 mL* MDB	1045-32
Hydrogen Peroxide, 30%	500 mL	144-49

OPTIONAL APPARATUS

Balance, Sartorius, B310S, 110 V	each	24030-00
Balance, Sartorius, B310S, 220 V	each	24030-02
Beaker, 400 mL	each	500-48
Beaker, Berzelius, 200 mL	12/pkg	22761-75
Bottle, Wash, 1 L	each	620-16
Bulb, dropper, 2 mL	12/pkg	21189-00
Cylinder, graduated, 50 mL	each	508-41
Dispenser, 1-5 mL, (for H_2SO_4, meat)	each	23121-37
Dispenser, 10-50 mL, (for H_2SO_4, meat)	each	23121-41
Filter Discs, glass, 47 mm	100/pkg	2530-00
Filter Holder, membrane	each	2340-00
Flask, filter, 500 mL	each	546-49
Flask, flat-bottom volumetric, 100 mL	each	23125-42
Fume Scrubber Apparatus, 115 V	each	23266-00
Fume Scrubber Apparatus, 230 V	each	23266-02
Oven, laboratory, 120 V	each	14289-00
Paper, weighing, 76 x 76 mm	500/pkg	14738-00
pH Paper, pH 1-11	5 roll/pkg	391-33

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
pH Meter, EC10, Portable	each	50050-00
Pipet, Pasteur, disposable, 229 mm	250/pkg	21234-01
Safety Glasses	each	18421-00
Spatula, stainless, 127 mm	each	561-64
Spoon, measuring, 0.05 g	each	492-00
Stir Bar, PTFE, 1"	each	20953-51
Stir Plate, magnetic, Thermolyne, 120V, 50/60 Hz	each	23444-00
Stir Plate, magnetic, Thermolyne, 240V, 50/60 Hz	each	23444-02
Stopper, hollow size #5	6/pkg	14480-05
Syringe, 5 mL, plastic	100/pkg	23433-33
Watch Glass, 65 mm	12/pkg	578-97

For additional ordering information, see final section.
In the U.S.A. call 800-227-4224 to place an order.

*Contact Hach for larger sizes.

Table 8. Digestion Guidelines for Specific Sample Types

Sample Type	Sample Weight	Amount of Acid	Preheat Time	Amount of Peroxide	Special Instructions
Plant Tissue	0.25 to 5 g	4 mL	4 min.	10 mL	Use N-free paper to weigh sample
Meat & Poultry	0.5 g or predigestion	4 mL or as in predigest	4 min.	10 mL	May use predigestion procedure; see *Systems for Food, Feed and Beverage Analysis* (Lit. Code 3120.)
Fluid Fertilizers	0.1 to 0.25 g	4 mL	4 min.	10 mL	Add 0.4 g Kjeldahl Reduction Powder to flask before adding sulfuric acid. Place the flask in an 80 °C oven 15 minutes before digestion. Use N-free paper to weigh sample
Feed & Forage	0.25 g	4 mL	4 min.	10 mL	Use N-free paper to weigh sample
Dairy	0.25 to 2.0 g	4 mL	4 min.	10 mL	Use N-free paper to weigh dry samples (cheese)
Cereal	0.25 to 0.5 g	4 mL	4 min.	10 mL	Use N-free paper to weigh sample
Beverage	approx. 5 g (pipet into funnel)	4 mL	1 min.	10 mL	Preheat acid for 1 minute then add sample through funnel. Heat flask 30 seconds after sample is in flask.
Sludge	< 2.5 g wet sludge < 0.5 g dried sludge	4 mL	3 to 5 min.	10 mL or increase in 5 mL increments	Heat the diluted digest for 15 minutes and filter.
Water & Waste-water	not more than 0.5 g solid (mL = 50/C; C = % solids)	3 mL	until acid is refluxing	10 mL or increase in 5 mL increments	Water must evaporate before acid will reflux. **Boiling chips required.**
Bath Solutions	0.3 to 10 mL	4 mL	4 minutes	10 mL	Water must evaporate before acid will reflux. **Boiling chips** required.

Table 8. Digestion Guidelines for Specific Sample Types (continued)

Sample Type	Sample Weight	Amount of Acid	Preheat Time	Amount of Peroxide	Special Instructions
Edible Oils	0.25 to 0.5 g	4-6 mL	4 min.	5 mL immediately and 5 mL later	Weigh sample into flask and record exact weight.
Ion Exchange Resin	equivalent of 0.25 g dry resin	10 -15 mL	12 min.	20 mL	Digest will be clear with particles on bottom if metal oxides are not soluble in H_2SO_4. Add aqua regia or suitable solvent to dissolve particles. If particles are floating, start again using 15 mL H_2SO_4 and longer char time.
Soil	0.25 to 1.0 g	6 mL	4 min.	10 to 20 mL	
Fuels/ Lubricants	0.25 to 0.5 g	6 mL	4 min.	20 mL	Heat the diluted digest for 15 minutes and filter. Temperature of heater may need to be lowered slightly if foaming or burning occur.

SAFETY CONSIDERATIONS

Digesdahl Digestion Apparatus

For safe Digesdahl operation:

•Sample size-Never exceed 0.5 grams of sample (dry weight)

•Oils and organic liquids should be considered as solids when determining sample size.

•Acid type- Only use acid specified in Hach step-by-step procedures.

•Acid volume- Never use less than 3 mL.

•Always follow the order of steps indicated.

•**Always** wear safety glasses.

•Always perform digestion behind a safety shield or in a closed fume hood.

•If the sample goes to dryness, remove immediately and cool. Repeat procedure with smaller sample volume or more acid.

The following additional specific safety precautions are appropriate when using hydrogen peroxide in the Digesdahl digestion applications:

Do not mix hydrogen peroxide with any chemical reagents except as specified in the instructions.

Do not add hydrogen peroxide directly to the column on the digestion flask. Always add hydrogen peroxide in a slow and controlled manner; use the capillary funnel.

Hydrogen peroxide should be added to the organic materials in the flask ***only*** when sulfuric acid is present.

Do not add alcohol, acetone or other organic solvents to the digestion flask before or after digestion.

During digestion, use the heat setting and digestion time specified in the instructions.

When digesting a new substance for the first time, begin with a smaller size and work up to the optimum quantity for digestion.

The digestion flask and attached fractionating column must be vented at all times.

During operation, the Digesdahl heating element and associated glassware become very hot. Handle this glassware with the provided finger cots protecting the thumb and index finger. A hot digestion flask can scorch an unprotected surface. Use the cooling pad. If a flask should break during a digestion, perform the following procedures to avoid injury to personnel or damage to equipment:

1. Unplug the heater assembly and wait for the unit to cool.

2. Do not breathe any fumes that may be produced.

3. Hold the parts exposed to the digestion mixture under running water, avoiding getting water or the digestion mixture into the heater base.

If the aspirator fails during a digestion, immediately turn the heater off. Do not breathe any fumes that may be expelled from the manifold. After the flask has cooled and the fumes subsided, clean or replace the aspirator.

If the flask boils dry during the digestion, unplug the heater assembly and allow the flask to cool. Remove the flask and discard the contents. Repeat the digestion using less sample or more acid. If hydrogen peroxide was added to the flask before it went dry, wait until the flask cools completely. Add water to the flask before handling.

Chemicals

Concentrated sulfuric acid and hydrogen peroxide used in the digestion process should be handled correctly and with caution. Sulfuric acid is a strong acid and can cause burns if splashed on the skin and permanent damage if eye contact occurs. Hydrogen peroxide (30% or 50%) is a powerful oxidant and should never be stored near flammable materials. Like sulfuric acid, it can cause burns and eye damage. In case of eye or skin contact with either chemical, flush eyes and/or skin with water for 15 minutes. Remove contaminated clothing. Call a physician.

Both sulfuric acid and hydrogen peroxide are highly corrosive and should be cleaned up with water if spilled on instruments or a counter top. Read and observe all warnings on the reagent labels and Material Safety Data Sheets (MSDS).

Proper handling and storage procedures involving hydrogen peroxide should always address two major characteristics of the product: first, it is a strong oxidizing agent (corrosive), and second, it can decompose, releasing heat and oxygen. The chemical nature of hydrogen peroxide makes it an irritant to the skin, to mucus membranes and particularly to the eyes. It will cause chemical burns at industrial concentrations and may cause spontaneous combustion upon immediate or prolonged contact with combustibles.

Hydrogen peroxide decomposes to form water and oxygen. The natural decomposition rate of the normal industrial grade product is very low, but it will accelerate when contaminated by materials such as dust,

metallic ions, or alkali. Accelerated decomposition from contamination will result in the significant production of oxygen and liberation of heat. These products will support combustion and will cause pressure bursts in confined spaces. Commercial grades of hydrogen peroxide contain small quantities of additives (termed "stabilizers") to prevent accelerated decomposition from occurring during normal product usage.

Please observe the following precautions for handling and storage of hydrogen peroxide:

Do store in a cool place away from direct sunlight.

Do store in the original containers with closures as supplied and keep closed when not in use. (Be sure the containers are vented. Hach hydrogen peroxide bottles are shipped with a special permeable cap liner.)

Do wear gloves and safety glasses when handling the material.

Do use silicon carbide boiling chips when digesting liquid samples.

Do wash contaminated skin and body quickly with plenty of water. Remove contaminated clothing and wash well before using again. Wash regularly.

Do wash eyes with plenty of water if contaminated and do get medical advice quickly.

Do get medical advice without delay if the material is ingested.

Do flush all spillage with large amounts of water.

Do not store near heat sources or in contact with combustible or organic materials.

Do not allow materials to be stored or trapped in confined spaces.

Do not inhale vapors or ingest the materials.

Do not allow contact with eyes or body.

Do not allow contact with decomposition catalysts (metals, dust, alkali, etc.).

Do not use unapproved materials (brass, copper, carbon steel, rubber, etc.) for transfer or storage systems.

Caps on the reagent bottles are made with a special porous liner that allows venting of gas. The venting cap always must be used on the bottle of hydrogen peroxide. As a precaution, the reagent bottles are shipped in a plastic bag. If there is evidence of leakage during shipment, wear gloves when removing the bottle from the bag and rinse the bottle with water when removed from the bag. Rinse the bag before disposal.

APPLICATION-SPECIFIC MANUALS

Operating procedures for the Digesdahl Digestion Apparatus vary according to the type and form of the sample and have been published in a series of procedure manuals dedicated to specific applications. They are available on request and provide the analyst with step-by step instructions for sample digestion and subsequent analysis of specific parameters. Specific setup and operating information is given in the Digesdahl Digestion Apparatus instruction manual, included with each Digesdahl. Application-specific manuals available include:

Literature Code	Title
3120	Systems for Food, Feed and Beverage Analysis
3201	Fluid Fertilizer Analysis Manual
8353	Water Analysis Handbook

To receive a free copy of these manuals, contact Hach Customer Services and request by literature code number.

DISTILLATION

Distillation is an effective way of separating chemical components for analysis. The Hach Distillation Apparatus (see Figure 14) is adapted easily for a variety of test needs. Sample distillations are easy and safe to perform. It is suitable for water and wastewater requiring sample distillation pretreatment. Applications for the General Purpose Apparatus include: fluoride, albuminoid nitrogen, ammonia nitrogen, phenols, selenium and volatile acids. Arsenic and cyanide require specialty glassware sets in addition to the General Purpose Set. These sets are 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 and Support Apparatus provide efficient heating and anchoring of the glassware.

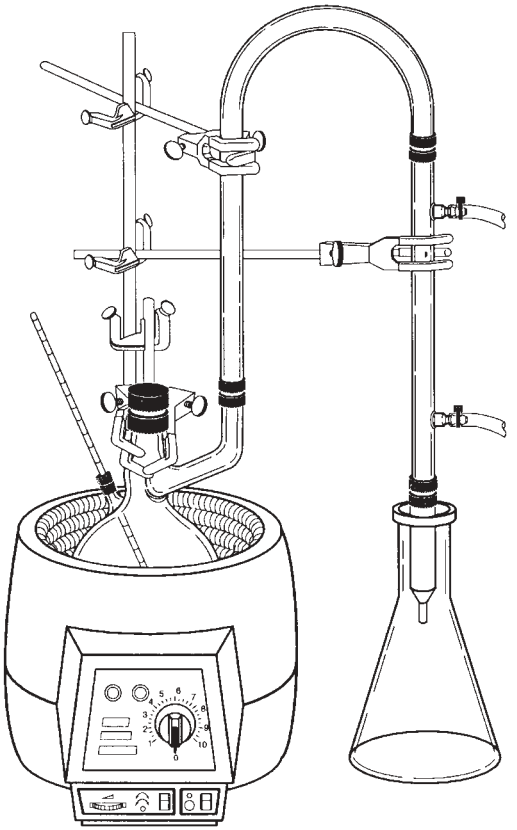


Figure 14 **General Purpose Distillation Apparatus with Heater and Support Apparatus**

SAMPLING AND STORAGE

Correct sampling and storage are critical to the accuracy of each test. For greatest accuracy minimize contamination from the sampling device, remove residues of previous samples from sample container and preserve the sample properly, if necessary.

Taking Water Samples

Collect samples for analysis carefully to make sure the most representative sample possible is obtained. In general, they should be

taken near the center of the vessel or duct and below the surface. Use only clean containers (bottles, beakers) for collecting samples. Rinse the container several times first with the water to be sampled.

Take samples as closely as possible to the source of the supply to minimize the effects of a distribution system. Allow the water to run for sufficient time to flush the system, and the sample container should be filled slowly with a gentle stream to avoid turbulence and air bubbles. Collect water samples from wells after the pump has run long enough to deliver water representative of the ground water feeding the well.

It is difficult to obtain a truly representative sample when collecting surface water samples. Best results are obtained by running a series of tests with samples taken from several locations and depths at different times. Results then can be used to establish patterns applicable to that particular body of water.

Generally, as little time as possible should elapse between collecting the sample and making the analysis.

Depending on the nature of the test, special precautions in handling the sample also may be necessary to prevent natural interferences such as organic growth or loss or gain of dissolved gases. Sample preservatives and storage techniques are described in each procedure for sample held for later testing.

Acid Washing Bottles

A procedure may suggest acid-washing the sample bottles to minimize the effect of interferences. This is accomplished by using a detergent to clean the glassware or plastic-ware, rinsing with tap water, rinsing with a 1:1 Hydrochloric Acid Solution or 1:1 Nitric Acid Solution, rinsing with deionized water. This may require successive rinses, up to 12-15 may be necessary if chromium is being determined. Air dry. The nitric acid rinse also is important if lead is being determined.

Chromic acid or chromium-free substitutes may be used to remove organic deposits from glass containers, but rinse containers thoroughly with water to remove traces of chromium.

Glassware for phosphate determinations should be washed with phosphate-free detergents and acid-washed with 1:1 HC1. This

glassware must be rinsed thoroughly with distilled water. For ammonia and Kjeldahl nitrogen, the glassware must be rinsed with ammonia-free water.

Storage and Preservation

The most cost-effective sample containers are made of polypropylene or polyethylene. The best and most expensive containers are made of quartz or TFE (tetrafluoroethylene, Teflon). Avoid soft glass containers for metals in the microgram-per-liter range. Store samples for silver determination in light-absorbing containers.

Avoid introducing contaminating metals from containers, distilled water or membrane filters. Thoroughly clean sample containers as described under Acid Washing Bottles.

Preservation techniques retard the chemical and biological changes continuing after sample is taken. These changes may change the amount of a chemical species available for analysis. As a general rule, it is best to analyze the samples as soon as possible after collection. This is especially true when the concentration is expected to be low. Analyzing immediately reduces the potential for error and minimizes labor.

Preservation methods are limited generally to pH control, chemical addition, refrigeration and freezing. The recommended preservation for various constituents is given in Table 9. Other information provided in the table is the suggested type of container and the maximum recommended holding times for properly preserved samples.

Aluminum, cadmium, chromium, cobalt, copper, iron, lead, nickel, phosphorus, potassium, silver and zinc samples can be preserved for at least 24 hours by the addition of one Nitric Acid Solution Powder Pillow 1:1 (Cat. No. 2540-98) per liter of sample. Check the pH with pH indicator paper or a pH meter to assure the pH is 2 or less. Add additional pillows if necessary. Adjust the sample pH prior to analysis by adding an equal number of Sodium Carbonate Anhydrous Powder Pillows (Cat. No. 179-98). Or, raise the pH to 4-5 with Sodium Hydroxide Standard Solution, 1 N or 5 N.

Table 9. Required Containers, Preservation Techniques and Holding Times[1]

Parameter No./Name	Container[2]	Preservation[3,4]	Maximum Holding Time[5]
Table 1A-Bacterial Tests: **1-4.** Coliform, fecal and total	P G	Cool, 4°C, 0.008% $Na_2S_2O_3$[6]	6 hours.
5. Fecal streptococci	P, G	do	do.
Table 1B-Inorganic Tests: **1.** Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, G	do	do.
4. Ammonia	P, G	Cool, 4 °C, H_2SO_4 to pH< 2	28 days.
9. Biochemical oxygen demand	P, G	Cool, 4 °C	48 hours.
11. Bromide	P, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4 °C	48 hours.
15. Chemical oxygen demand	P, G	Cool, 4 °C, H_2SO_4 to pH< 2	28 days.
16. Chloride	P, G	None required	do.
17. Chlorine, total residual	P, G	do	Analyze immediately.
21. Color	P, G	Cool, 4 °C	48 hours.
24-24. Cyanide, total and amenable to chlorination	P, G	Cool, 4 °C, NaOH to pH >12, 0.6g ascorbic acid[6]	14 days[7].
25. Fluoride	P	None required	28 days.
27. Hardness	P, G	HNO_3 to pH< 2, H_2SO_4 to pH< 2	6 months.
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately
31,43. Kjeldahl and organic nitrogen	P, G	Cool, 4 °C, H_2SO_4 to pH < 2	28 days.
Metals:[8] **18.** Chromium VI	P, G	Cool, 4 °C	24 hours.

Table 9 Required Containers, Preservation Techniques and Holding Times[1] (continued)

Parameter No./Name	Container[2]	Preservation[3,4]	Maximum Holding Time[5]
Metals (continued): **35.** Mercury	P, G	HNO_3 to pH < 2	28 days.
3, 5-8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals, except chromium VI and mercury.	P, G	do	6 months.
38. Nitrate	P, G	Cool, 4 °C	48 hours.
39. Nitrate-nitrite[5]	P, G	Cool, 4 °C, H_2SO_4 to pH < 2	28 days.
40. Nitrite	P, G	Cool, 4 °C	48 hours.
41. Oil and grease	G	Cool, 4 °C, H_2SO_4 to pH < 2	28 days.
42. Organic carbon	P, G	Cool, 4 °C, HCl or H_2SO_4 to pH < 2	do.
44. Orthophosphate	P, G	Filter immediately, Cool, 4 °C	48 hours.
46. Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately.
47. Winkler	do	Fix on site and store in dark	8 hours.
48. Phenols	G only	Cool, 4 °C, H_2SO_4 to pH < 2	28 days.
49. Phosphorus (elemental)	G	Cool, 4 °C	48 hours.
50. Phosphorus, total	P, G	Cool, 4 °C, H_2SO_4 to pH < 2	28 days.
53. Residue, total	P, G	Cool, 4 °C	7 days.
54. Residue, Filterable	P, G	do	7 days.
55. Residue, Nonfilterable (TSS)	P, G	do	7 days.
56. Residue, Settleable	P, G	do	48 hours.
57. Residue, volatile	P, G	do	7 days.

Table 9 Required Containers, Preservation Techniques and Holding Times[1] (continued)

Parameter No./Name	Container[2]	Preservation[3,4]	Maximum Holding Time[5]
Metals (continued):			
61. Silica	P	do	28 days.
64. Specific conductance	P, G	do	do.
65. Sulfate	P, G	do	do.
66. Sulfide	P, G	Cool, 4 °C add zinc acetate plus sodium hydroxide to pH >9.	7 days.
67. Sulfite	P, G	None required	Analyze immediately
68. Surfactants	P, G	Cool, 4 °C	48 hours.
69. Temperature	P, G	None required	Analyze immediately
73. Turbidity	P, G	Cool, 4 °C	48 hours.

[1] This table was taken from Table II published in the *Federal Register*, October 1, 1991, 40 CFR, Part 136.3, pages 308-309.
[2] Polyethylene (P) or glass (G).
[3] Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved by maintaining at 4 °C until compositing and sample splitting is completed.
[4] When any sample is to be shipped by common carrier or sent through United States Mails, it must comply with the Department of Transportation Hazardous Material Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (about pH 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
[5] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administer under §136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obliged to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See §136.3(e) for details.
[6] Should only be used in the presence of residual chlorine.
[7] Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to a pH of 12.
[8] Samples should be filtered immediately on-site before adding preservative for dissolved metals.

Volume Additions, Correction For
When significant amounts of preservative are used, a volume correction should be made. This will account for the acid added to preserve the sample and the base used to adjust the pH to the range of the procedure. This correction is made as follows:

1. Determine the total volume of initial sample, acid added and base added.

2. Divide the total volume by the initial volume.

3. Multiply the test result by this factor.

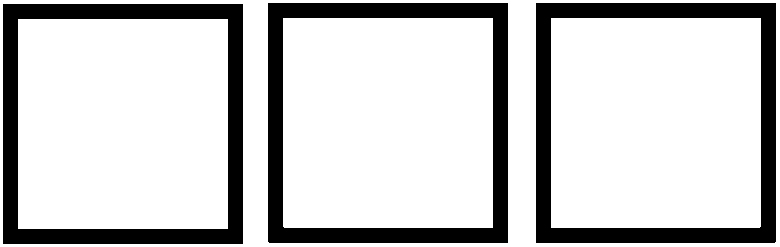
An example:
A one-liter sample was taken and preserved with 2 mL of nitric acid. It was neutralized with 5 mL of sodium hydroxide, 6 N. The result of the analysis procedure was 10.00 mg/L. What is the volume correction factor and correct result?

1. Total volume = 1000 mL + 2 mL + 5 mL = 1007.

2. $\frac{1007}{1000}$ = 1.007 = volume correction factor

3. 10.00 mg/L x 1.007 = 10.07 mg/L = correct result

The addition of a Sodium Carbonate Anhydrous Power Pillow does not need to be corrected for.



DR/700 MODULE SELECTION GUIDE

Parameters and Ranges (alphabetical)

Description	Range	Module Number (Cat. No.)		Reagent Package
Aluminum, Aluminon Method	0-1.0 mg/L	52.01	(46252-00)	22420-00
Aluminum, Eriochrome Cyanine R Method	0-0.25 mg/L	52.01	(46252-00)	
Arsenic*	0-0.2 mg/L	52.01	(46252-00)	
Barium	0-300 mg/L	45.01	(46245-00)	12064-66
Benzotriazole	0-15 mg/L	42.01	(46242-00)	21412-66
Boron	0-15 mg/L	61.01	(46261-00)	
Bromine	0-4 mg/L	52.01	(46252-00)	21056-69
Cadmium, Anion Exchange	0-100 µg/L	45.01		
Cadmium, Dithizone	0-70 µg/L	52.01	(46252-00)	22422-00
Chloride	0-20 mg/L	45.01	(46245-00)	23198-00
Chlorine, Free*	0-3.5 mg/L	52.01	(46252-00)	21055-69
Chlorine, Total*	0-3.5 mg/L	52.01	(46252-00)	21056-69
Chromium, Hexavalent*	0-1.000 mg/L	55.01	(46255-00)	25050-25
Chromium, Total	0-0.700 mg/L	55.01	(46255-00)	22425-00
Cobalt	0-1.2 mg/L	61.01	(46261-00)	22426-00
Color, Apparent and True	0-500 Units	45.01	(46245-00)	
Copper, Autocatalytic	0-3 g/L	81.01	(46281-00)	1042-66
Copper, Bicinchoninate*	0-3 mg/L	55.01	(46255-00)	21058-69
Copper, Porphyrin	0-200 µg/L	42.01	(46242-00)	22427-00
Cyanide*	0-0.2 mg/L	61.01	(46261-00)	
Cyanuric Acid	0-65 mg/L	50.01	(46250-00)	
DEHA	0-300 µg/L	55.01	(46255-00)	
Fluoride, SPADNS*	0-2 mg/L	57.01	(46257-00)	25060-25
Formaldehyde	0-625 µg/L	61.01	(46261-00)	22577-00
Hardness, Calcium as $CaCO_3$	0-2.7 mg/L	52.01	(46252-00)	23199-00
Hardness, Magnesium as $CaCO_3$	0-2 mg/L	52.01	(46252-00)	23199-00
Hydrazine	0-400 µg/L	45.01	(46245-00)	1790-37
Iodine	0-6 mg/L	52.01	(46252-00)	21056-69
Iron, Ferrous	0-5.00 mg/L	50.01	(46250-00)	1037-69
Iron, FerroZine Method	0-0.9 mg/L	55.01	(46255-00)	2301-66
Iron, Total, FerroVer Method	0-5.00 mg/L	50.01	(46250-00)	21057-69
Iron, Total, TPTZ Method	0-1 mg/L	57.01	(46257-00)	22756-68
Lead, Anion Exchange	0-120 µg/L	48.01		
Lead, Dithizone*	0-140 µg/L	52.01	(46252-00)	22431-00
Lead, LeadTrak	0-150 µg/L	48.01	(46248-00)	23750-00
Manganese, High Range*	0-20.0 mg/L	52.01	(46252-00)	
Manganese, Low Range	0-0.800 mg/L	55.01	(46255-00)	22433-00
Molybdenum, Molybdate, High Range	0-50 mg/L	42.01	(46242-00)	22434-00
Molybdenum, Molybdate, Low Range	0-3.00 mg/L	61.01		
Nickel, Autocatalytic	0-9 g/L	57.01	(46257-00)	14321-98
Nickel, Heptoxime*	0-1.5 mg/L	42.01	(46242-00)	22435-00
Nickel, PAN	0-0.6 mg/L	55.01	(46255-00)	22426-00
Nitrate, High Range	0-30 mg/L	50.01	(46250-00)	21061-69
Nitrate, High Range AccuVac	0-30 mg/L	50.01	(46250-00)	25110-25
Nitrate, Low Range	0-0.5 mg/L	50.01	(46250-00)	
Nitrite, High Range	0-150 mg/L	57.01	(46257-00)	21075-69
Nitrite, Low range*	0-0.2 mg/L	50.01	(46250-00)	21071-69
Nitrogen, Ammonia, Nessler Method*	0-3.0 mg/L	42.01	(46242-00)	
Nitrogen, Ammonia, Monochloramine and Free Ammonia	0-1.0 mg/L	61.01	46261-00	26184-00

Description	Range	Module Number (Cat. No.)		Reagent Package
Nitrogen, Ammonia, Salicylate Method	0-0-1.00 mg/L	61.01	(46261-00)	22437-00
Oxygen, Dissolved, High Range	0-10 mg/L	52.01	(46252-00)	25150-25
Oxygen, Dissolved, Low Range	0-800 µg/L	61.01	(46261-00)	25010-25
Oxygen, Dissolved, Super High Range	0-40 mg/L	69.01	(46269-00)	25150-25
Oxygen Demand, Chemical, Reactor Digestion Method, H.R. & S.H.R.*	0-1500 & 0-15,000 mg/L	61.01	(46261-00)	
Oxygen Demand, Chemical, Reactor Digestion Method, Low Range*	0-150 mg/L	42.01	(46242-00)	
Ozone, High Range AccuVac	0-1.50 mg/L	61.01	(46261-00)	25180-25
Ozone, Low Range AccuVac	0-0.25 mg/L	61.01	(46261-00)	25160-25
Ozone, Medium Range AccuVac	0-0.75 mg/L	61.01	(46261-00)	25170-25
Palladium	0-250 mg/L	42.01	(46242-00)	23123-00
Phenols*	0-0.2 mg/L	45.01	(46245-00)	22439-00
Phosphonates	0-125 mg/L	81.01	(46281-00)	22440-00
Phosphorus, Acid Hydrolyzable, Test 'N Tube™	0-5.00 mg/L	81.01	(46281-00)	
Phosphorus, Reactive, Amino Acid	0-20 mg/L	52.01	(46252-00)	22441-00
Phosphorus, Reactive, Molybdovanadate	0-45.0 mg/L	42.01	(46242-00)	20760-37
Phosphorus, Reactive, PhosVer 3*	0-2.50 mg/L	81.01	(46281-00)	21060-69
Phosphorus, Reactive, Test 'N Tube™	0-5.00 mg/L	81.01	(46281-00)	
Phosphorus, Total, Test 'N Tube™	0-5.00 mg/L	81.01	(46281-00)	
Platinum	0-10 mg/L	50.01	(46250-00)	
Polyacrylic Acid	0-20mg/L	50.01	(46250-00)	22252-00
Potassium, Tetraphenylborate Method	0-8 mg/L	45.01	(46245-00)	
Quaternary Ammonium Compounds	0-5.0 mg/L	57.01	(46257-00)	
Residue, Nonfilterable	0-750 mg/L	81.01	(46281-00)	
Rhodium	0-15 g/gal	42.01	(46242-00)	
Silica, High Range	0-40 mg/L	42.01	(46242-00)	
Silica, Low Range	0-3 mg/L	81.01	(46281-00)	
Silver, Colorimetric Method	0-0.6 mg/L	57.01	(46257-00)	22966-00
Sodium Chromate	0-1000 mg/L	45.01	(46245-00)	2127-99
Sulfate	0-100 mg/L	45.01	(46245-00)	21067-69
Sulfide*	0-1 mg/L	61.01	(46261-00)	22445-00
Surfactants, Anionic (as LAS)	0-0.2 mg/L	61.01	(46261-00)	
Tannin and Lignin	0-9.00 mg/L	69.01	(46269-00)	22446-00
Tolyltriazole	0-15 mg/L	42.01	(46242-00)	21412-66
Volatile Acids (as HOAC)	0-2500 mg/L	50.01	(46255-00)	22447-00
Zinc, Zincon Method*	0-3.00 mg/L	61.01	(46261-00)	

*USEPA Approved procedure

420-nm Filter Module (42.01) Cat. No. 46000-10

Parameter	Range
Benzotriazole, Tolytriazole	15 mg/L
Copper, Porphyrin	200 ug/L
Molybdenum, High Range	50 mg/L
Nickel, Heptoxime*	1.5 mg/L
Nitrogen, Ammonia Nessler*	3.0 mg/L
Oxygen Demand, Chemical, L.R*	150 mg/L
Palladium	250 mg/L
Phosphorus, Acid Hydrolyzable	NA
Phosphorus, Molybdovanadate	45.0 mg/L
Phosphorus, Total*	NA
Rhodium	15 g/gal
Silica, High Range	40 mg/L

450-nm Filter Module (45.01) Cat. No. 46000-11

Parameter	Range
Barium	300 mg/L
Cadmium, Anion Exchange	100 ug/L
Chloride	20 mg/L
Color	500 Units
Hydrazine	0-400 µg/L
Oil in Water	700 mg/L
Phenols*	0.2 mg/L
Potassium (user calibration)	8 mg/L
Sodium Chromate	1000 mg/L
Sulfate	100 mg/L

480-nm Filter Module (48.01) Cat. No. 46000-12

Parameter	Range
Lead, Anion Exchange	120 ug/L
Lead, LeadTrak	150 ug/L

500-nm Filter Module (50.01) Cat. No. 46000-13

Parameter	Range
Cyanuric Acid	65 mg/L
Iron, Ferrous	5.00 mg/L
Iron, Total, FerroVer	5.00 mg/L
Nitrogen, Nitrate, High Range	30 mg/L
Nitrogen, Nitrate, H.R. AccuVac	40 mg/L
Nitrogen, Nitrite, Low Range*	0.2 mg/L
Platinum (user calibration)	10 mg/L
Polyacrylic Acid	20 mg/L
Volitile Acids	2500 mg/L

525-nm Filter Module (52.01) Cat. No. 46000-14

Parameter	Range
Aluminum, Aluminon	1.0 mg/L
Aluminum, Erichrome Cyanine R	0.25 mg/L
Arsenic (user calibration)*	0.2 mg/L
Bromine	4 mg/L
Cadmium, Dithizone*	70 ug/L
Chlorine, Free	*3.5 mg/L
Chlorine, Total*	3.5 mg/L
Hardness, Calcium	2.7 mg/L
Hardness, Magnesium	2.0 mg/L
Iodine	6 mg/L
Lead, Dithizone*	140 µg/L
Manganese, High Range*	20.0 mg/L
Oxygen, Dissolved, HRDO	10 mg/L
Phosphorus, acid hydrolyzable	NA
Phosphorus, Amino Acid	20 mg/L
Phosphorus, total*	NA

550-nm Filter Module (55.01) Cat. No. 46000-15

Parameter	Range
Chromium, Hexavalent*	1.000 mg/L
Chromium, Total	0.700 mg/L
Copper, Bicinchoninate*	3 mg/L
DEHA	300 ug/L
Iron, FerroZine	0.9 mg/L
Manganese, Low Range	0.800 mg/L
Nickel, PAN	0.7 mg/L

575-nm Filter Module (57.01) Cat. No. 46000-16

Parameter	Range
Fluoride*	2 mg/L
Iron, Total, TPTZ	1 mg/L
Nickel, Autocatalytic	9 g/L
Nitrogen, Nitrite, High Range	150 mg/L
Quaternary Ammonium Compounds	5.0 mg/L
Silver, Colorimetric	0.6 mg/L

610-nm Filter Module (61.01) Cat. No. 46000-17

Parameter	Range
Boron	15 mg/L
Cobalt	1.2 mg/L
Cyanide*	0.2 mg/L
Formaldehyde	625 ug/L
Molybdenum, Low Range	3.00 mg/L
Nitrogen, Ammonia, Salicylate	1 mg/L
Nitrogen, Monochloramine and Free Ammonia	0-1.00 mg/L

610-nm Filter Module (61.01) Cat. No. 46000-17 (continued)

Parameter	Range
Oxygen Dissolved, LRDO AccuVac	800 ug/L
Oxygen Demand, Chemical, High Range and High Range Plus	1500 & 15,000 mg/L
Ozone, Low Range	0.25 mg/L
Ozone, Medium Range	0.75 mg/L
Ozone, High Range	1.50 mg/L
Surfactants, Anionic	0.2 mg/L
Sulfide*	1 mg/L
Zinc, Zincon*	3.00 mg/L

690-nm FIlter Module (69.01) Cat. No. 46000-18

Parameter	Range
Oxygen, Dissolved, SHRDO	40 mg/L
Tannin and Lignin	9.00 mg/L

810-nm Filter Module (81.01) Cat. No. 46000-19

Parameter	Range
Copper, Autocatalytic	3 g/L
Phosphonates	125 mg/L
Phosphorus, Acid Hydrolyzable	0-5.00 mg/L
Phosphorus, PhosoVer 3*	2.50 mg/L
Phosphorus, Reactive, Test 'N Tube	0-5.00 mg/L
Phosphorus, Total*, Test 'N Tube	0-5.00 mg/L
Residue, Nonfilterable	750 mg/L
Silica, Low range	3 mg/L

Key to Abbreviations

LR = Low Range	SHR = Super High Range	g/L = grams per liter
MR = Medium Range	HR+ = High Range Plus	g/gal = grams per gallon
HR = High Range	mg/l (milligrams per liter) = ppm (parts per million)	

*USEPA approved procedure

Module 42.01
420 nm

Hach warrants equipment it manufactures against defective materials or workmanship for at least one year from the shipping date. Warranties do not apply to limited-life electrical components such as batteries. Full warranty information is located on the reverse side of Hach invoices.

IMPORTANT NOTICE

A lamp intensity adjustment must be performed:
- Before first or initial use
- When new filter modules are installed
- On each filter module when the lamp is replaced

Refer to Lamp Intensity Adjustment in the instrument manual.

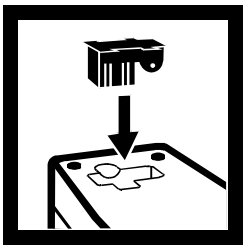
Table of Contents for 420-nm parameters

BENZOTRIAZOLE OR TOLYLTRIAZOLE

(0 to 15.0 mg/L Benzotriazole; 0 to 20.0 mg/L Tolylriazole)

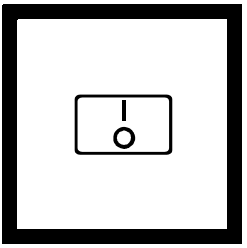
For cooling and boiler water

UV Photolysis Method*



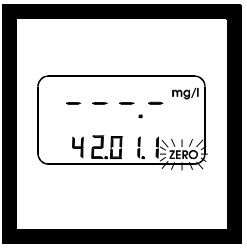
1. Install module
42.01
in a DR/700.

Note: *The most reliable results are obtained when samples are analyzed as soon as possible after collection.*



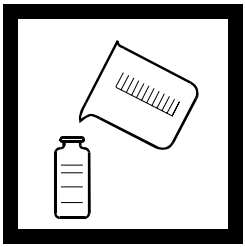
2. Press: **I/O**

The display will show
420 nm
and module
42.01



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
42.01.1
for benzotriazole or
42.10.1
for tolyltriazole.

*Adapted from Harp, D., *Proceedings 45th International Water Conference*, October 1984, 299



4. Fill a square mixing bottle with 25 mL of sample.

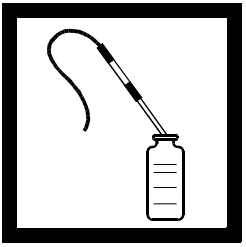
Note: *For proof of accuracy, use a 5.0 mg/L benzotriazole standard solution (preparation given in Accuracy Check) in place of the sample.*

Note: *Sample temperature should be between 20 to 25 °C (68 to 70 °F).*

Note: *If sample contains nitrite or borax (sodium borate), adjust the pH to between 4 to 6 with 1 N Sulfuric Acid.*

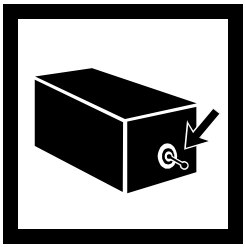
5. Add the contents of one Triazole Reagent Powder Pillow (the prepared sample). Swirl to dissolve completely.

Note: *If sample contains more than 500 mg/L hardness (as $CaCO_3$), add 10 drops of Rochelle Salt Solution.*



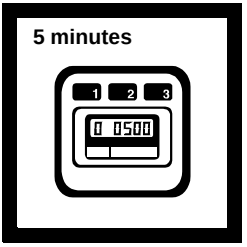
6. Insert the ultraviolet lamp into the mixing bottle.

Note: UV safety goggles should be worn while the lamp is on.



7. Turn the UV lamp on.

Note: *A yellow color will develop if triazole is present.*

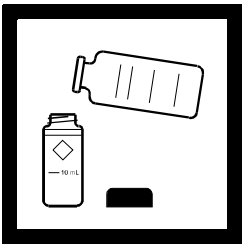


8. Wait 5 minutes.

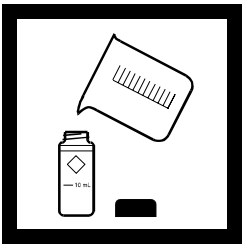
9. Turn the lamp off. Remove lamp from the bottle Swirl to mix thoroughly.

Note: *Low results will occur if photolysis (lamp on) takes place for more or less than five minutes.*

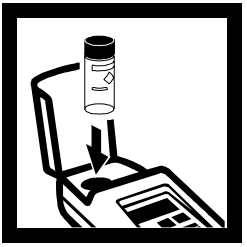
Note: *Avoid fingerprints on the quartz surface of the lamp. Rinse the lamp and wipe with a soft, clean tissue between tests.*



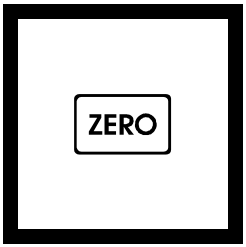
10. Fill a 10-mL cell to the 10-mL line with the prepared sample.



11. Fill another 10-mL cell to the 10-mL line with sample (the blank).

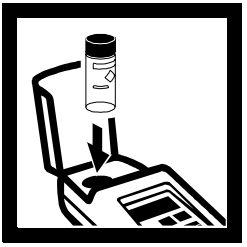


12. Place the blank in the cell holder.



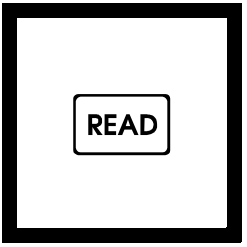
13. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



14. Place the prepared sample in the cell holder.

Note: In bright sunlight it may be necessary to close the cell compartment cover.



15. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L benzotriazole or tolyltriazole.

SAMPLING AND STORAGE
The most reliable results are obtained when samples are analyzed as soon as possible after collection.

ACCURACY CHECK
Standard Additions Method
a) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard solution, 500 mg/L benzotriazole, to three 25-mL samples. Perform the test according to the above procedure.

Note: *The test will not distinguish between benzotriazole and tolyltriazole.*

b) Each addition of 0.1 mL of standard solution should increase the benzotriazole reading by 2 mg/L over the reading of an unspiked sample.

c) If these increases are not obtained see Standard Additions (Section I) for more information.

UV Lamp Check

To verify the ultraviolet lamp (normal life equals 5000 hours) is working properly, perform the following test:

a) Prepare a 5.0 mg/L benzotriazole standard solution by pipetting 10.0 mL of benzotriazole standard solution, 500 mg/L benzotriazole, into a 1-L volumetric flask. Dilute to volume.

b) Analyze according to the above procedure. If the result is significantly below 5.0 mg/L, replace the lamp.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 10.0 mg/L tolyltriazole concentration solutions, the standard deviation was ±0.22 mg/L tolyltriazole. Testing zero concentration samples, the limit of detection was 0.17 mg/L tolyltriazole.

Testing 10.0 mg/L benzotriazole concentration solutions, the standard deviation was ±0.11 mg/L benzotriazole. Testing zero concentration samples, the limit of detection was 0.16 mg/L benzotriazole.

The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

The following may interfere when present in concentrations exceeding those listed below:

Acrylates (as methyl acrylate)	50 mg/L
Alum	400 mg/L
Borate (as sodium tetraborate)	4000 mg/L
Chlorine (as Cl_2)	20 mg/L
Chromium (as chromate)	12 mg/L
Copper	10 mg/L
Hardness	500 mg/L as $CaCO_3$
Iron	20 mg/L
Lignosulfonates	40 mg/L
Magnesium	300 mg/L as $CaCO_3$
Molybdenum (as molybdate)	200 mg/L
Nitrite	4000 mg/L
Phosphonates (AMP or HEDP)	100 mg/L
Sulfate	200 mg/L
Zinc	80 mg/L

Strong oxidizing or reducing agents present in the sample will interfere directly.

SUMMARY OF METHOD

Benzotriazole or tolyltriazole, used in many applications as corrosion inhibitors for copper and copper alloys, are determined by a proprietary catalytic ultraviolet (UV) photolysis procedure requiring less than 10 minutes to perform.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Triazole Reagent Powder Pillows	1 pillow	50/pkg	21412-66

REQUIRED APPARATUS

Bottles, square, mixing, 25-mL mark	1	each	17042-00
Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 42.01	1	each	46242-00
Stopwatch	1	each	14645-00
UV Safety Goggles	1	each	21134-00

Select one based on available voltage:

Lamp, UV, with power supply, 115 Vac, 60 Hz		each	20828-00
Lamp, UV, with power supply, 230 Vac, 50 Hz		each	20828-02

OPTIONAL REAGENTS

Benzotriazole Standard Solution, 500 mg/L	100 mL		21413-42
Rochelle Salt Solution	29 mL*	DB	1725-33
Sulfuric Acid Standard Solution, 1.0 N	100 mL	MDB	1270-32

OPTIONAL APPARATUS

Cap for 10- and 25-mL Sample Cells	12/pkg		24018-12
Flask, volumetric, 1000 mL	each		14574-53
Lamp, UV, (lamp only)	each		20823-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg		391-33
Pipet Filler, safety bulb	each		14651-00

*Contact Hach for larger sizes.

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Tensette Pipet	50/pkg	21856-96
Pipet, volumetric, Class B, 10 mL	each	515-38
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Single to dual UV lamp cord adapter	each	19485-00
Timer, interval, 1 second to ten hours	each	23480-00

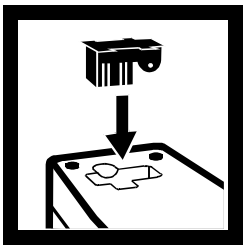
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

COPPER (0 to 250 µg/L)

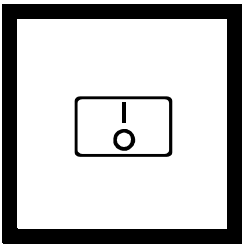
For water, wastewater and seawater

Porphyrin Method*



1. Install module
42.01
in a DR/700.

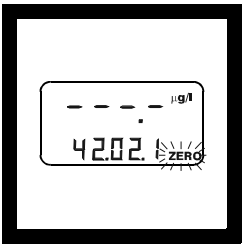
Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*



2. Press: **I/O**

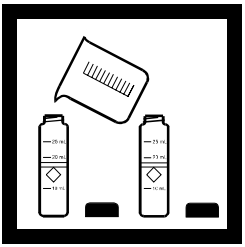
The display will show
420 nm
and module number
42.01

Note: *Total copper determination needs a prior digestion; use either the Digesdahl or vigorous digestion (Section 1).*



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
42.02.1

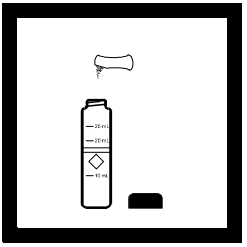
*Adapted from Ishii and Koh, *Buseki Kagaku, 28,* 473 (1979)



4. Fill two 25-mL cells to the 25-mL line with sample.

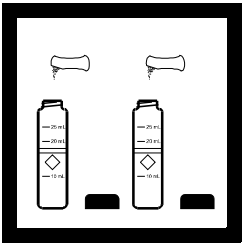
Note: *Wash all glassware with detergent. Rinse with tap water. Rinse again with Nitric Acid Solution, 1:1. Rinse a third time with copper-free, demineralized water.*

Note: *For proof of accuracy, use a 100 µg/L copper standard solution (preparation given in Accuracy Check) in place of the sample.*

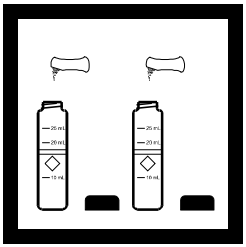


5. Add the contents of one Copper Masking Powder Pillow to one of the sample cells (the blank). Cap and invert several times to mix.

Note: *The other sample cell is the prepared sample.*

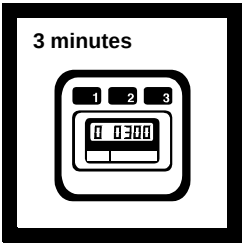


6. Add the contents of one Porphyrin 1 Reagent Powder Pillow to each sample cell. Cap and invert several times to mix.

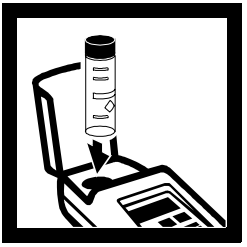


7. Add the contents of one Porphyrin 2 Reagent Powder Pillow to each sample cell. Cap and invert several times to mix.

Note: *The yellow color will turn blue momentarily. If any copper is present, the sample will return to yellow.*

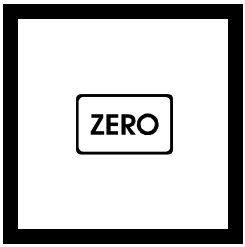


8. Wait 3 minutes.



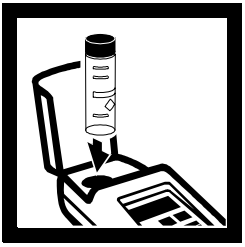
9. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



10. Press: **ZERO**

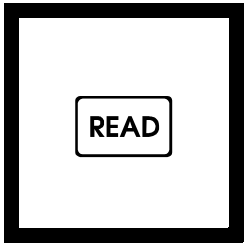
The display will count down to 0. Then the display will show 0 µg/L and the zero prompt will turn off.



11. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to 10-mL sample cell and proceed.*

Note: *If standards or samples with high levels of metal are analyzed, a slight metallic deposit or yellow buildup may appear on the sample cell wall. Remove by rinsing with nitric acid.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in µg/L copper (Cu Porphyrin).

Note: *For most accurate results, run the test using copper-free demineralized water. Subtract the value obtained in Step 12 from all following tests. Repeat for each new lot of reagents.*

SAMPLING AND STORAGE

Collect samples in acid-washed plastic bottles. To preserve, adjust the pH to 2 or less with nitric acid (about 5 mL per liter). Store preserved samples up to six months at room temperature.

Before testing, adjust the pH of the sample to between 2 and 6. If the sample is too acidic, adjust the pH with 5.0 N Sodium Hydroxide Standard Solution. Correct test results for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Additions Method

a) Using a TenSette Pipet, add 0.1 mL of Copper Standard Solution, 10.0 mg/L Cu, to two sample cells containing 25 mL of sample.

b) Repeat, using 0.2 mL and 0.3 mL additions of standard.

c) Analyze the samples as described above. The copper concentration reading should increase by 40 µg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

To assure the accuracy of the test, prepare a 100-µg/L copper standard:

a) Pipet 1.00 mL of copper standard solution, 10.0 mg/L Cu, into a 100-mL volumetric flask.

b) Dilute to volume with copper-free, reagent-grade water.

c) Use this standard in place of the sample in the procedure.

d) Prepare this solution daily.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 150 µg/L Cu concentration solutions, the standard deviation was ±1.2 µg/L Cu.

Testing zero concentration samples, the limit of detection was 1.7 µg/L Cu. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

The following may interfere when present in concentrations exceeding those listed below:

Aluminum	60 mg/L
Cadmium	10 mg/L
Calcium	1,5000 mg/L
Chloride	90,000 mg/L
Chromium (Cr^{6+})	110 mg/L
Cobalt	100 mg/L
Fluoride	30,000 mg/L
Iron	6 mg/L
Lead	3 mg/L
Magnesium	10,000 mg/L
Manganese	140 mg/L
Mercury	3 mg/L
Molybdenum	11 mg/L
Nickel	60 mg/L
Potassium	60,000 mg/L
Sodium	90,000 mg/L
Zinc	9 mg/L

Chelating agents, such as EDTA, interfere at all levels unless either the Digesdahl or vigorous digestion (Section I) is performed.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment: see Interferences, pH (Section I)

SUMMARY OF METHOD

The porphyrin method is very sensitive to trace amounts of free copper. The method is free from most interferences and does not require any sample extraction or preconcentration. Interferences from other metals are eliminated by the copper masking reagent. The porphyrin indicator forms an intense, yellow-colored complex with any free copper present in sample.

REQUIRED REAGENTS

	Cat. No.
Copper Reagent Set (100 Tests)	22427-00
Includes: (2) 21873-66, (4) 21874-66, (2) 21875-69	

Description	Quantity Per Test	Unit	Cat. No.
Copper Masking Reagent			
Powder Pillows	1 pillow	100/pkg	21873-99
Porphyrin 1 Reagent			
Powder Pillows	2 pillows	100/pkg	21874-69
Porphyrin 2 Reagent			
Powder Pillows	2 pillows	100/pkg	21875-69

REQUIRED APPARATUS

Clippers, for opening			
powder pillows	1	each	968-00
DR/700 Filter Module			
Number 42.01	1	each	46242-00

OPTIONAL REAGENTS

Copper Standard Solution, 10 mg/L Cu	100 mL MDB	129-32
Hydrochloric Acid Solution, 1:1 (6 N)	500 mL	884-49
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 5 N	1 L	2450-53
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Beaker, 100 mL	each	500-42
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Flask, volumetric, Class A, 50 mL	each	14574-41
Flask, volumetric, Class A, 100 mL	each	14574-42
Hot Plate, 7" x 7", 120 Vac	each	23441-00
Hot Plate, 7" x 7", 240 Vac	each	23441-02
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, Mohr, 5 mL	each	20934-37
Pipet, TenSette, 0.1 to 1.0 mL	each	14515-35
Pipet, volumetric, Class A, 1 mL	each	14515-35
Pipet, volumetric, Class A, 50 mL	each	14515-41
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Watch Glass	each	578-70

For Technical Assistance, Prices and Ordering

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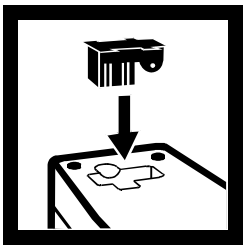
Outside the U.S.A. - Contact the Hach office or distributor serving you.

MOLYBDENUM, MOLYBDATE, HR

(0 to 40 mg/L)

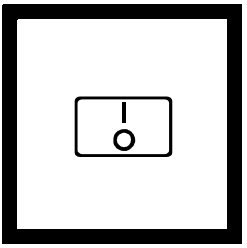
For water and wastewater

Mercaptoacetic Acid Method*



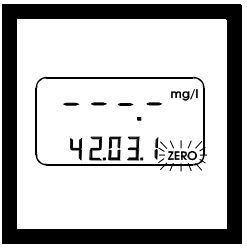
1. Install module
42.01
in a DR/700.

Note: *Collect samples in glass or plastic bottles.*



2. Press: **I/O**

The display will show
420 nm
and module number
42.01



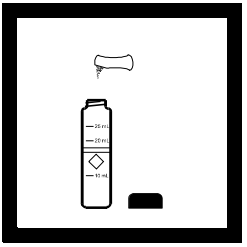
3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
42.03.1

*Adapted from *Analytical Chemistry,* 25, (9) 1363 (1953).

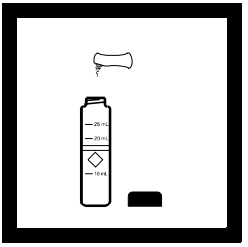
4. Fill a 25-mL cell to the 25-mL line with sample.

Note: *For proof of accuracy, use a 10.0 mg/L Molybdenum Standard Solution (listed under Optional Reagents) in place of the sample.*

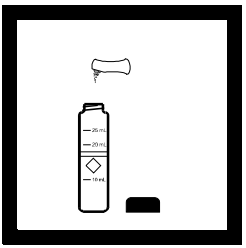
Note: *Filter turbid samples using the labware listed under Optional Apparatus.*



5. Add the contents of one MolyVer 1 Reagent Powder Pillow. Cap and invert several times to mix.

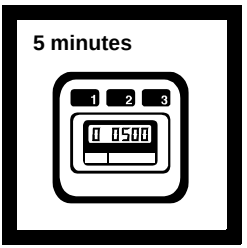


6. Add the contents of one MolyVer 2 Reagent Powder Pillow. Cap and invert several times to mix.



7. Add the contents of one MolyVer 3 Reagent Powder Pillow (the prepared sample). Cap and invert several times to mix.

Note: *If molybdenum is present a yellow color will develop.*

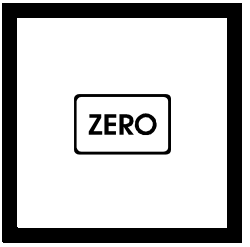


8. Wait 5 minutes.

9. Fill a 25-mL cell to the 25-mL line with sample (the blank). Cap.

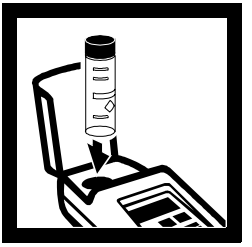
10. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



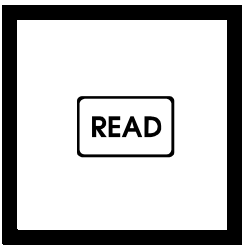
11. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



12. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



13. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L molybdenum (Mo^{6+}).

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Molybdenum Voluette Ampule Standard Solution, 500 mg/L Mo^{6+}.

b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to three 25-mL samples, Mix thoroughly.

c) Analyze the spiked sample according to the above procedure. The molybdenum concentration reading should increase by 2.0 mg/L for each 0.1 mL addition of standard.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

To assure the accuracy of the test, use a Molybdenum Standard Solution, 10.0 mg/L Mo^{6+}, listed under Optional Reagents.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched cells and two representative lots of testing reagents. Testing 20.0 mg/L Mo concentration samples, the standard deviation was ±0.11 mg/L Mo.

Testing zero concentration samples, the limit of detection was 0.18 mg/L Mo. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry* **1980,** 52, 2242-2249).

INTERFERENCES

Samples containing 10 mg/L copper or more will exhibit an increasing positive interference upon standing. Read these samples as soon as possible after the 5-minute reaction period of Step 8.

Aluminum, iron and nickel do not interfere in concentrations up to 50 mg/L. Chromium does not interfere in concentrations up to 1000 mg/L.

Interference from nitrite up to 2000 mg/L as NO_2^- can be eliminated by adding one Sulfamic Acid Powder Pillow in Step 4.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

SUMMARY OF METHOD

MolyVer 1 and 2 Reagents are added to buffer and condition the sample. MolyVer 3 provides the mercaptoacetic acid which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration.

REQUIRED REAGENTS

	Cat. No.
Molybdenum Reagent Set (100 Tests)	22434-00
Includes (1) 14146-69, (1) 14148-69, (1) 14178-69	

Description	Quantity Per Test	Unit	Cat. No.
MolyVer 1 Molybdenum Reagent Powder Pillows	1 pillow	100/pkg	14146-69
MolyVer 2 Molybdenum Reagent Powder Pillows	1 pillow	100/pkg	14148-69
MolyVer 3 Molybdenum Reagent Powder Pillows	1 pillow	100/pkg	14178-69

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 42.01	1	each	46242-00

OPTIONAL REAGENTS

Molybdenum Standard Solution, 10 mg/L Mo^{6+}		105 mL	14187-42
Molybdenum Standard Solution, Voluette ampule, 500 mg/L Mo^{6+}	10 mL	16/pkg	14265-10
Sulfamic Acid Powder Pillows		100/pkg	1055-99
Water, demineralized		4 L	272-56

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, erlenmeyer, 250 mL	each	505-46
Funnel, poly, 65 mm	each	1083-67
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

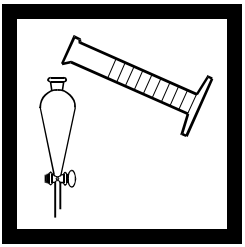
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

NICKEL (0 to 1.80 mg/L Ni)

For water, wastewater and seawater

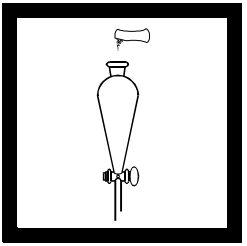
Heptoxime Method*; EPA accepted for reporting (Digestion is required; see Section 1)**



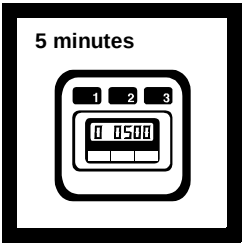
1. Measure 300 mL of sample in a 500-mL graduate cylinder. Pour into a 500-mL separatory funnel.

Note: *If sample cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*

Note: *For proof of accuracy, use a 1.0 mg/L nickel standard solution (preparation given in Accuracy Check) in place of the sample.*



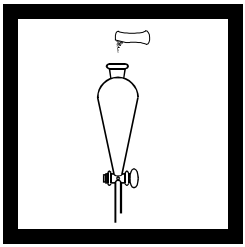
2. Add the contents of one Nickel 1 Reagent Powder Pillow to the funnel. Stopper and shake to mix.



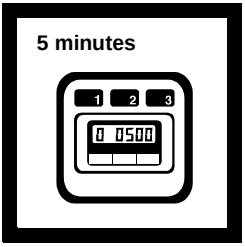
3. Wait 5 minutes.

*Adapted from *Chemie Analytique*, 36, 43 (1954).

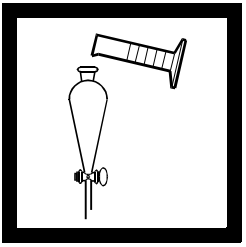
**Procedure is equivalent to Standard Method 3500-Ni D for wastewater.



4. Add the contents of one Nickel 2 Reagent Powder Pillow to the funnel. Stopper and shake to mix.

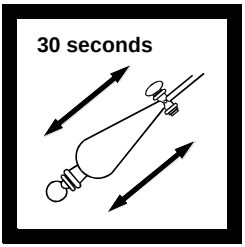


5. Wait five minutes.

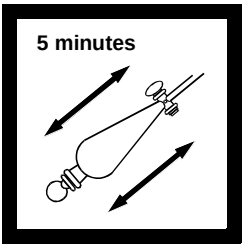


6. Add 10 mL of chloroform. Stopper and shake gently. Holding the stopper in, invert the funnel and slowly open the stopcock to vent the pressure buildup.

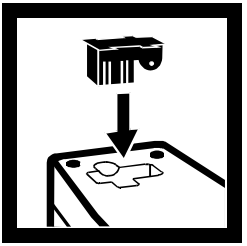
Note: *Point the funnel in a safe direction when venting.*



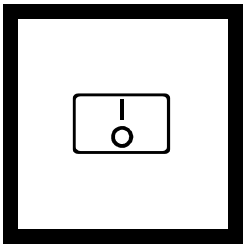
7. Close the stopcock. Shake for 30 seconds.



8. Shake the funnel several times over the next five minutes. Vent after shaking.

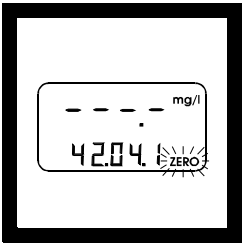


9. Install module **42.01** in a DR/700.

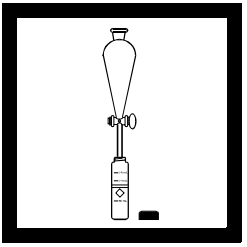


10. Press: **I/O**

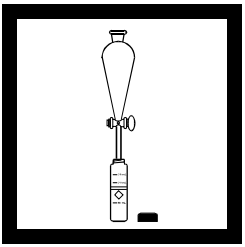
The display will show
420 nm
and module number
42.01



11. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
42.04.1



12. Wait for the layers to separate (about 5 minutes). Insert a pea-size cotton plug into the delivery tube of the separatory funnel. Drain the chloroform layer into a sample cell (the prepared sample). Stopper.

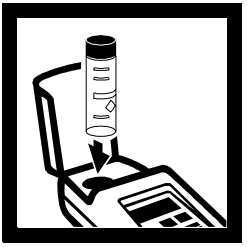


13. Repeat Steps 6 to 8 and Step 12 two additional times with 10-mL portions of chloroform. Drain the chloroform layer into the same sample cell.

Note: *The five-minute reaction period is not necessary for the second and third extractions. Shake with chloroform; wait for layers to separate, then continue.*

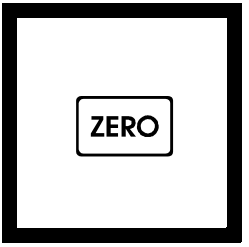
Note: *The final volume of extract will be about 25 mL due to the slight solubility of chloroform in water. Use all the chloroform extracted, exact final volume is not important.*

Note: *Swirl sample cell to mix extracts.*



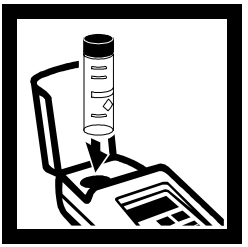
14. Fill a second sample cell with chloroform (the blank). Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open, In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If a 10-mL cell is used for the blank a 10-mL cell must also be used for the sample.*



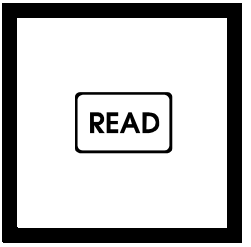
15. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



16. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open, In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If a 10-mL cell is used for the blank a 10-mL cell must also be used for the sample.*



17. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L nickel (Ni).

SAMPLING AND STORAGE

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 5 mL per liter). Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the sample pH to between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 8 as this may cause some loss of nickel as a precipitate. Correct the test results for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Nickel Voluette Ampule Standard Solution, 300 mg/L Ni.

b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to three 300-mL samples.

c) Analyze each sample as described above. The nickel concentration should increase 0.10 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 1.0 mg/L nickel standard solution by diluting 50.0 mL of a 10-mg/L working standard solution to 500 mL in a volumetric flask. The working stock solution should be prepared daily by diluting 10.00 mL of Nickel Standard Solution, 1000 mg/L as Ni, to 1000 mL with demineralized water.

Or, use the TenSette Pipet to add 1.0 mL of a Nickel Voluette Ampule Standard Solution, 300 mg/L Ni, into a 500-mL volumetric flask and dilute to volume with demineralized water. This solution is 0.6 mg/L nickel.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 1.00 mg/L Ni concentration samples, the standard deviation was ±0.026 mg/L Ni.

Testing zero concentration samples, the limit of detection was 0.013 mg/L Ni. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** 52, 2242-2249).

INTERFERENCES

Cobalt, copper and iron interferences can be overcome by adding one or more additional Nickel 1 Reagent Powder Pillows in Step 2. The tolerance limits of these interferences are shown in the following table:

Table 1. Tolerance Limits

Number of Nickel 1 Pillows needed	Concentration of interfering substance (mg/L)		
	Cobalt	Copper	Iron
1	1	10	20
2	7	16	65
3	13	22	110
4	18	28	155
5	25	35	200

A preliminary acid digestion is required to determine any suspended or precipitated nickel and to eliminate interference by organic matter. To eliminate this interference or to determine total recoverable nickel perform the EPA approved digestion in Digestion (Section I).

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section 1).

SUMMARY OF METHOD

Nickel ion reacts with heptoxime to form a yellow-colored complex which is then extracted into chloroform to concentrate the color and enable a more sensitive determination. Chelating agents are added to the sample to overcome the interferences caused by cobalt, copper and iron.

REQUIRED REAGENTS

	Cat. No.
Nickel Reagent Set (50 Tests) . . .	22435-00
Includes: (3) 14458-49, (2) 2123-68, (2) 2124-68	

Description	Quantity Per Test	Unit	Cat. No.
Chloroform, ACS	55 mL	500 mL	14458-49
Nickel 1 Reagent Powder Pillows	1 pillow	25/pkg	2123-68
Nickel 2 Reagent Powder Pillows	1 pillow	25/pkg	2124-68

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cotton balls, absorbent	1	100/pkg	2572-01
Cylinder, graduated, 10 mL	1	each	508-38
Cylinder, graduated, 500 mL	1	each	508-49
DR/700 Filter Module Number 42.01	1	each	46242-00
Funnel, separatory, 500 mL	1	each	520-49
Ring, support, 4"	1	each	580-01
Stand, support, 127x203 mm	1	each	563-00
Stopper, hollow, poly, Size 0	2	6/pkg	14480-00

OPTIONAL REAGENTS

Nickel Standard Solution, 1000 mg/L Ni	100 mL	14176-42
Nickel Standard Solution, Voluette ampule, 300 mg/L Ni, 10 mL	16/pkg	14266-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Caps for 10- and 25-mL sample cells	12/pkg	24018-12
Flask, erlenmeyer, 500 mL	each	505-49
Flask, volumetric, Class A, 500 mL	each	14574-49
Flask, volumetric, Class A, 1000 mL	each	14574-53
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
Pipet, serological, 1 mL	each	532-35
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 10.00 mL	each	14515-38
Pipet Filler, safety bulb	each	14651-00
Pipet, volumetric, Class A, 50.00 mL	each	14515-41
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

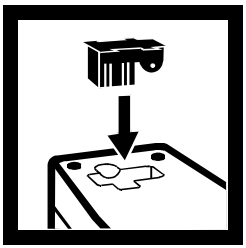
For Technical Assistance Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or the distributor serving you.

NITROGEN, AMMONIA (0 to 3.00 mg/L NH_3-N)

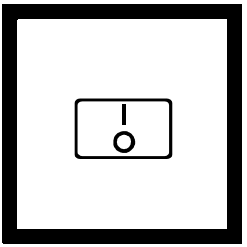
For water, wastewater*, seawater*

Nessler Method**, EPA accepted for reporting (distillation required)†



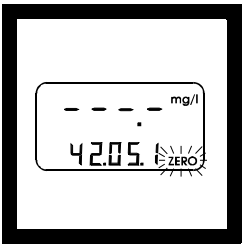
1. Install module
42.01
in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*



2. Press: **I/O**

The display will show
420 nm
and module number
42.01

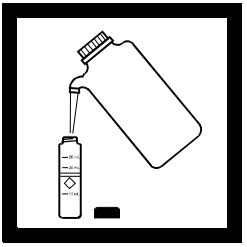


3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
42.05.1

*Requires distillation.
** Adapted from *Standard Methods for the Examination of Water and Wastewater.*
†Procedure is equivalent to USEPA Method 350.2 and Standard Method 4500-NH_3 B and C for wastewater.

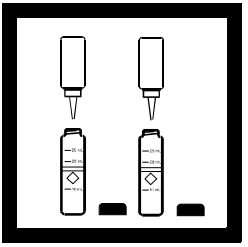
4. Fill a 25-mL sample cell to the 25-mL mark with sample (the prepared sample).

Note: *For proof of accuracy, use a 1.0 mg/L Ammonia Nitrogen Standard Solution (listed under Optional Reagents) in place of the sample.*

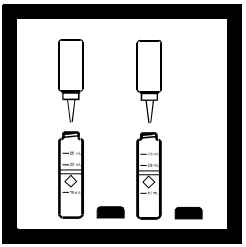


5. Fill another 25-mL sample cell with demineralized water (the blank).

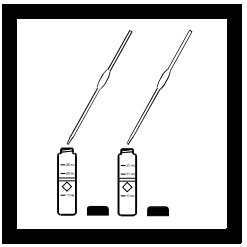
Note: *The demineralized water and sample should be at 20 ± 1°C (68 ±2°F) for best results. Higher temperatures cause high results; lower temperatures cause low results.*



6. Add three drops of Mineral Stabilizer to each sample cell. Cap and invert several times to mix.



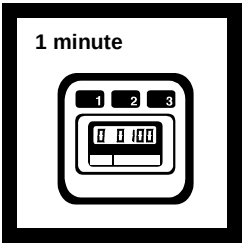
7. Add three drops of Polyvinyl Alcohol Dispersing Agent to each sample cell by holding the dropping bottle straight. Invert several times to mix.



8. Pipet 1.0 mL of Nessler Reagent to each sample cell. Cap and invert several times to mix.

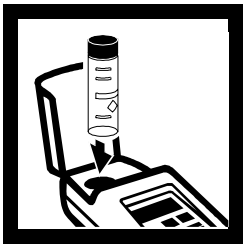
Note: *Nessler Reagent is toxic and corrosive. Pipet carefully.*

Note: *A yellow color will develop if ammonia is present. The reagent will cause a faint yellow color in the blank.*



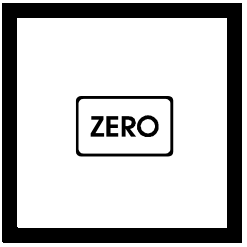
9. Wait 1 minute.

Note: *Do not wait more than five minutes after Step 8 before performing Step 13.*



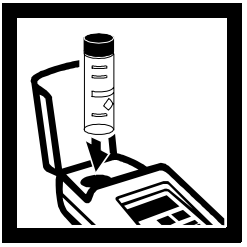
10. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10-mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



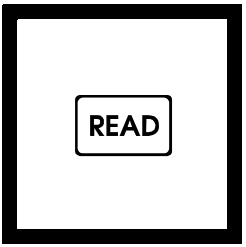
11. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



12. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10-mL of the prepared sample to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



13. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L ammonia nitrogen (NH_3-N).

Note: To convert results to other units, see Table 1.

Table 1. Conversion Factors

To convert reading from	To	Multiply by
mg/L NH_3-N	mg/L NH_3	1.22
mg/L NH_3-N	mg/L NH_4^+	1.29

SAMPLING AND STORAGE

Collect samples in clean glass or plastic bottles. If chlorine is present, add one drop of sodium thiosulfate, 0.1 N, for each 0.3 mg/L Cl_2 in a 1-liter sample. Preserve the sample by reducing the pH to 2 or less with sulfuric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize with sodium hydroxide, 5 N, before analysis. Correct the test result for volume additions: see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Nitrogen Ammonia Voluette Ampule Standard Solution, 50 mg/L NH_3-N.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The nitrogen concentration should increase 0.20 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

To check accuracy, use a 1.0 mg/L Nitrogen Ammonia Standard Solution listed under Optional Reagents. Or, this can be prepared by diluting 1.00 mL of solution from a Voluette Ampule Standard for Ammonium Nitrogen to 50.0 mL with demineralized water.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched cells and two representative lots of testing reagents. Testing 1.50 mg/L NH_3- concentration samples, the standard deviation was ±0.013 mg/L NH_3-N.

Testing zero concentration samples, the limit of detection was 0.015 mg/L NH_3-N. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** 52, 2242-2249)

INTERFERENCES

A solution containing a mixture of 500 mg/L $CaCO_3$ and 500 mg/L Mg as $CaCO_3$ does not interfere. If the hardness concentration exceeds these concentration, extra Mineral Stabilizer should be added.

Iron and sulfide interfere by causing a turbidity with Nessler Reagent.

Residual chlorine must be removed by addition of sodium arsenite solution. Use two drops to remove each mg/L Cl from a 250 mL sample. Sodium thiosulfate can be used in place of sodium arsenite. See Sampling and Storage Section.

Less common interferences, such as glycine, various aliphatic and aromatic amines, organic chloramines, acetone, aldehydes and alcohols may cause greenish or other off colors or turbidity. It may be necessary to distill the sample if these compounds are present.

Seawater samples may be analyzed by addition of 1.0 mL (27 drops) of Mineral Stabilizer to the sample before analysis. This will complex the high magnesium concentrations found in sea water, but the sensitivity of the test will be reduced by 30 percent due to the high chloride concentration. For best results, perform a calibration, using standards spiked to the equivalent chloride concentration, or distill the sample as described below.

DISTILLATION

a) Measure 250 mL of sample into a 250-mL graduated cylinder and pour into a 400-mL beaker. Destroy chlorine, if necessary, by adding 2 drops of Sodium Arsenite Solution per mg/L Cl_2.

b) Add 25 mL of Borate Buffer Solution and mix. Adjust the pH to about 9.5 with 1 N sodium hydroxide solution. Use a pH meter.

c) Set up the general purpose distillation apparatus as shown in the Hach Distillation Apparatus Manual. Pour the solution into the distillation flask. Add a stir bar.

d) Use a graduated cylinder to measure 25 mL of demineralized water into a 250-mL erlenmeyer flask. Add the contents of one Boric Acid Powder Pillow. Mix thoroughly. Place the flask under the still drip tube. Elevate so the end of the tube is immersed in the solution.

e) Turn on the heater power switch. Set the stir control to 5 and the heat control to 10. Turn on the water and adjust to maintain a constant flow through the condenser.

f) Turn off the heater after collecting 150 mL of distillate. Immediately remove the collection flask to avoid sucking solution into the still. Measure the distillate to assure 150 mL was collected (total volume 175 mL).

g) Adjust the pH of the distillate to about 7 with 1 N sodium hydroxide. Use a pH meter.

h) Pour the distillate into a 250-mL volumetric flask. Rinse the erlenmeyer with several small volumes of demineralized water and add the rinsings to the volumetric flask. Dilute to the mark. Stopper. Mix thoroughly. Analyze as described above.

SUMMARY OF METHOD

The Mineral Stabilizer complexes hardness in the sample. The Polyvinyl Alcohol Dispersing Agent aids the color formation in the reaction of Nessler Reagent with ammonium ions. A yellow color is formed proportional to the ammonia concentration.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Nessler Reagent	2 mL	500 mL	21194-49
Mineral Stabilizer	6 drops	59 mL* SCDB	23766-26
Polyvinyl Alcohol Dispersing Agent	6 drops	59 mL* SCDB	23765-26
Water, demineralized	25 mL	4 L	272-56

REQUIRED APPARATUS

DR/700 Filter Module Number 42.01	1	each	46242-00
Pipet, 1 mL	2	each	515-35
Pipet Filler, safety bulb	1	each	14651-00

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Borate Buffer Solution	946 mL	14709-16
Boric Acid Powder Pillows	50/pkg	14817-66
Nitrogen, Ammonia Standard Solution, 1 mg/L NH_3-N	500 mL	1891-49
Nitrogen, Ammonia Standard Solution, Voluette ampule, 50 mg/L NH_3-N	16/pkg	14791-10
Sodium Arsenite Solution, 5 g/L	100 mL MDB	1047-32
Sodium Hydroxide Standard Solution, 5.0 N	100 mL* MDB	2450-32
Sodium Hydroxide Standard Solution, 1.0 N	100 mL* MDB	1045-32
Sodium Thiosulfate Solution, 0.1 N	100 mL* MDB	323-32
Sulfuric Acid, ACS	500 mL*	979-49

OPTIONAL APPARATUS

Beaker, 400 mL	each	500-48
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, 25 mL	each	508-40
Cylinder, graduated, 250 mL	each	508-46
Distillation apparatus general purpose accessories	each	22653-00
Distillation heater and support apparatus set, 115 V	each	22744-00
Distillation heater and support apparatus set, 230 V	each	22744-02
Dropper, plastic, 0.5 and 1.0-mL marks	10/pkg	21247-10
Flask, erlenmeyer, 250-mL	each	505-46
Flask, volumetric, 50 mL	each	547-41
Flask, volumetric, 250 mL	each	547-46
pH Meter, EC10, portable	each	50050-00
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 1 mL	each	515-35
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Thermometer, -20 to 105 °C	each	1877-01

For Technical Assistance, Prices and Ordering

In the U.S.A.--Call 800-227-4224 toll-free for more information.
Outside the U.S.A.--Contact the Hach office or distributor serving you.

*Contact Hach for larger sizes

OXYGEN DEMAND, CHEMICAL (COD)

(0 to 150 mg/L)

For water, wastewater and seawater

Reactor Digestion Method*; USEPA approved for reporting†

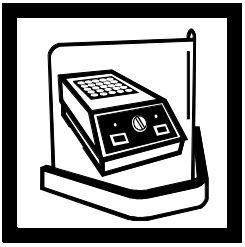
DIGESTION

1. Homogenize 100 mL sample for 2 minutes in a blender.

Note: *For samples with high solid content, blending ensures distribution of solids and improves accuracy and reproducibility.*

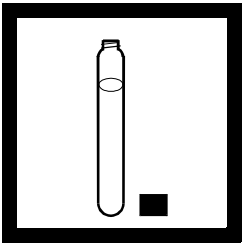
Note: *Pour homogenized sample into a 250-mL beaker and stir with magnetic stirrer.*

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*



2. Turn on the COD Reactor. Preheat to 150 °C. Place the plastic shield in front of the reactor.

Caution: Ensure safety devices are in place to protect analyst from splattering should reagent leaking occur.



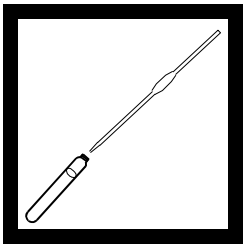
3. Remove the cap from a 0 to 150 mg/L COD Digestion Reagent Vial.

Note: *The reagent mixture is light sensitive. Keep unused vials in the opaque shipping container, in a refrigerator if possible. The amount of light striking the vials during testing will not affect the results.*

Caution

Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if improperly handled or accidentally misused. Please read all warnings and the safety section of this manual. Appropriate eye protection and clothing should be used for adequate user protection. If contact occurs, flush the affected area with running water. Follow instructions carefully.

*Jirka, A.M.; Carter, M.J. *Analytical Chemistry,* **1975,** 47(8), 1397.
†*Federal Register,* **April 21, 1980,** 45(78), 26811-26812.



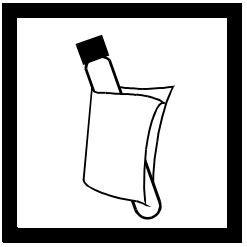
4. Hold the vial at a 45-degree angle. Pipet 2.00 mL of sample into the vial.

Note: *To ensure a uniform sample aliquot, stir sample with magnetic stirrer while drawing sample aliquot into the pipet.*

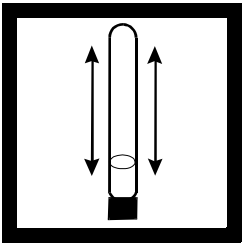
Note: *For greater accuracy, three replicates should be analyzed and the results averaged.*

Note: *Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Do not run tests with vials which have been spilled. If some spills, wash with running water.*

Note: *For proof of accuracy, use COD standard solutions (preparation given in the Accuracy Check) in place of the sample.*

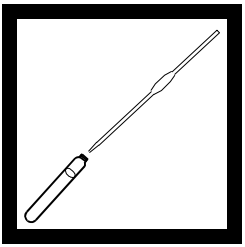


5. Replace the vial cap tightly. Rinse the COD vial with demineralized water and wipe the vial clean with a paper towel.



6. Hold the vial by the cap and over a sink. Invert several times to mix the contents. Place the vial in the preheated COD Reactor.

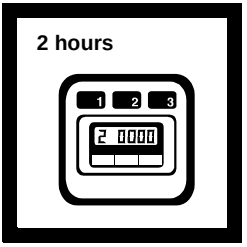
Note: *The vial will become hot during mixing.*



7. Prepare a blank by repeating Steps 3 to 6, substituting 2.00 mL demineralized water for the sample.

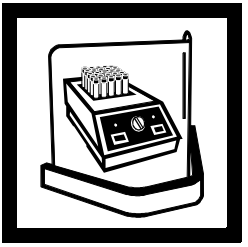
Note: *Be sure the pipet is well rinsed or use a clean pipet.*

Note: *One blank must be run with each set of samples. All tests (samples and blanks) should be run with the same lot of vials. The lot number appears on the container label.*

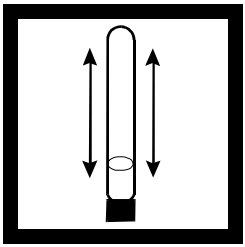


8. Heat the vials for 2 hours.

Note: *Many wastewater samples contain easily digested materials that are digested in less than 2 hours. If desired, measure the concentration (while still hot) at 15-minute intervals until it remains unchanged. At this point, the sample is completely digested. Cool vials to room temperature for final measurement.*



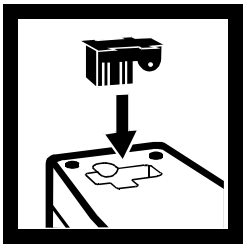
9. Turn the reactor off. Wait 20 minutes for the vials to cool to 120 °C or less.



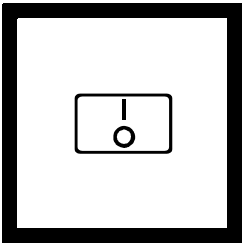
10. Invert each vial several times while still warm. Place the vials into a rack. Wait until the vials have cooled to room temperature.

Note: *If a pure green color appears in the reacted sample, the reagent capacity may have been exceeded. For most accurate results with samples near 150 mg/L COD, repeat the test with a diluted sample.*

COLORIMETRIC DETERMINATION, 0 to 150 mg/L COD

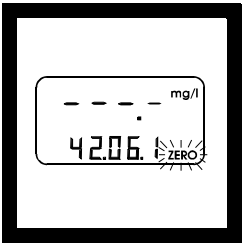


1. Install module
42.01
in a DR/700.

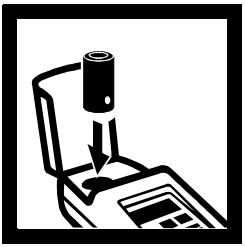


2. Press: **I/O**

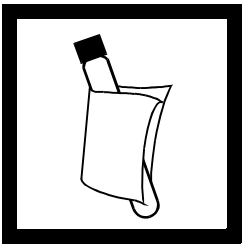
The display will show
420 nm
and module number
42.01



3. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display number shows
42.06.1

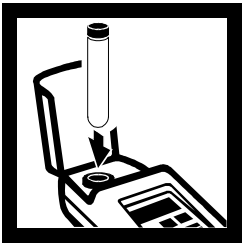


4. Fully insert a COD Vial Adaptor into the cell holder with the tabs in the square slot.



5. Clean the outside of the blank with a towel.

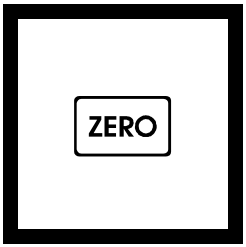
Note: *Wiping with a damp towel, followed by a dry one will remove fingerprints or other marks.*



6. Place the blank into the adapter with the Hach logo facing the front of the instrument.

Note: *The blank is stable when stored in the dark; see Blanks for Colorimetric Determination following these steps.*

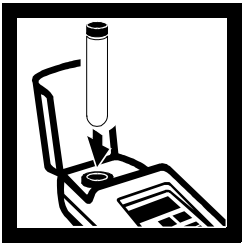
Note: *Avoid bright light when making measurements.*



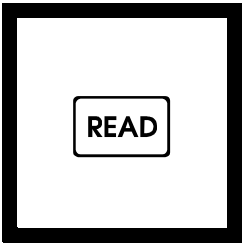
7. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L, and the zero prompt will turn off.

8. Clean the outside of the sample vial with a towel.



9. Place the sample vial into the adapter with the Hach logo facing the front of the instrument.



10. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L COD.

Note: *For most accurate results with samples near 150 mg/L COD, repeat the analysis with a diluted sample.*

SAMPLING AND STORAGE

Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days. Correct results for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Solution Method

Check the accuracy of the 0 to 150 mg/L range with a 100 mg/L standard. Prepare by dissolving 85 mg of dried (120 °C, overnight) potassium acid phthalate (KHP) in 1 liter of demineralized water. Use 2 mL as the sample volume. The expected result will be 100 mg/L COD. Or, dilute 10 mL of 1000-mg/L COD Standard Solution to 100 mL to produce a 100-mg/L standard.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagents.

Testing 150 mg/L COD concentration samples, the standard deviation was ±5 mg/L COD.

Testing zero concentration samples, the limit of detection was 10 mg/L COD. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

Chloride is the primary interference when determining COD concentration. Each COD vial contains mercuric sulfate that will eliminate chloride interference up to 2000 mg/L. Dilute samples with higher chloride concentrations. Dilute the sample enough to reduce the chloride concentration to 1000 mg/L.

If sample dilution will cause the COD concentration to be too low for accurate determination, add 0.50 g of mercuric sulfate ($HgSO_{4)}$ to each COD vial before the sample is added. The additional mercuric sulfate will raise the maximum chloride concentration allowable to 8000 mg/L.

BLANKS FOR COLORIMETRIC DETERMINATION

The blank may be used repeatedly for measurements using the same lot of vials. Store it in the dark. Monitor decomposition by measuring the absorbance. Zero the instrument in the absorbance mode, using a vial containing 5 mL of demineralized water and measure the absorbance of the blank. Record the value. Prepare a new blank when the absorbance has changed by about 0.01 absorbance units.

SUMMARY OF METHOD

The mg/L COD results are defined as the mg of O_2 consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion ($Cr_2O_7^{2-}$) to green chromic ion (Cr^{3+}). When the 0-150 mg/L colorimetric or titrimetric method is used, the amount of Cr^{6+} remaining is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex chloride interferences.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Select the appropriate COD Digestion Reagent Vial:			
Low Range, 0 to 150 mg/L COD	1 to 2 vials	25/pkg	21258-25
Water, demineralized	varies	4 L	272-56

REQUIRED APPARATUS

COD Reactor, 120/240 Vac	1	each	45600-00
COD Vial Adapter, DR/700	1	each	46008-00
DR/700 Filter Module Number 42.01	1	each	46242-00
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet, volumetric, Class A, 2.00 mL	1	each	14515-36
Pipet Filler, safety bulb	1	each	14651-00
Test Tube Rack	1 to 2 racks	each	18641-00

OPTIONAL REAGENTS

Description	Unit	Cat. No.
COD Digestion Reagent Vials, 0 to 150 mg/L COD	150/pkg	21258-15
COD Standard Solution, 1000 mg/L	236 mL	22539-31
Potassium Acid Phthalate, ACS	500 g	315-34
Sulfuric Acid, ACS	500 mL*	979-49
Mercuric Sulfate, ACS	28 g*	1915-20

OPTIONAL APPARATUS

Beaker, 250 mL	each	500-46
Blender		obtain locally
Cylinder, graduated, 5 mL	each	508-37
Electromagnetic Stirrer, 120 V, with electrode stand	each	45300-01
Electromagnetic Stirrer, 230 V, with electrode stand	each	45300-02
Flask, volumetric, Class A, 1000 mL	each	14574-53
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, serological, 5 mL	each	532-37
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg*	21856-96
Pipet, volumetric, Class A, 10 mL	each	14515-38
Safety shield, for COD reactor	each	23810-00
Spoon, measuring, 0.5 g	each	907-00
Stir Bar, 22.2 x 4.76 mm ($^{7}/_{8}$" x $^{3}/_{16}$")	each	45315-00
Stir Bar Retriever	each	15232-00

RELATED LITERATURE

Ask for your copy by literature code number.

Title	Literature Code No.
COD Disposal Information Brochure	4088

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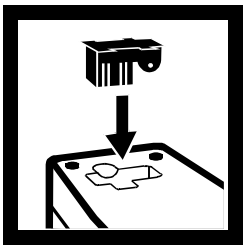
*Contact Hach for larger sizes.

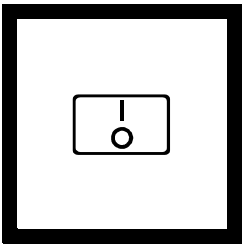
PALLADIUM (0 to 250 mg/L)

For palladium-tin activator baths

N,N'-Dimethyldithiooxamide Method

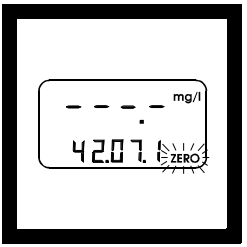


1. Install module
42.01
in a DR/700.

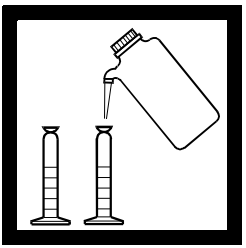


2. Press: **I/O**

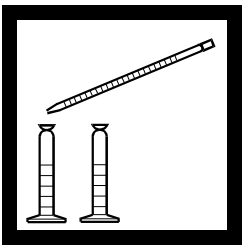
The display will show
420 nm
and module number
42.01



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
42.07.1

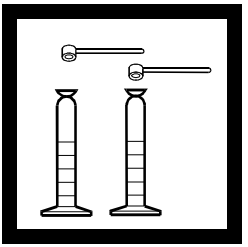


4. Fill two 25-mL mixing cylinders to the 20-mL mark with demineralized water.



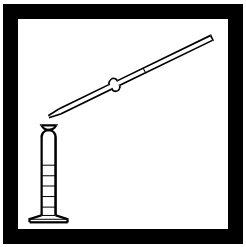
5. Add 5.0 mL of concentrated hydrochloric acid to each cylinder. Swirl to mix.

Note: *Use a Mohr pipet and pipet filler.*



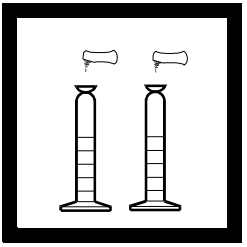
6. Add on 0.2-gram scoop of 2,2'bipyridine to each cylinder. Cap and invert to mix.

Note: *Because of 2,2'bipyridine's density, approximately 0.1 gram fills a 0.2-gram scoop.*

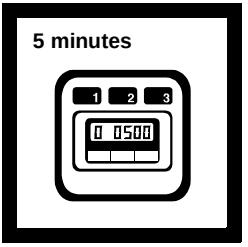


7. Add 0.5 mL of the palladium activator sample to one of the mixing cylinders (the prepared sample). The other cylinder will be the blank.

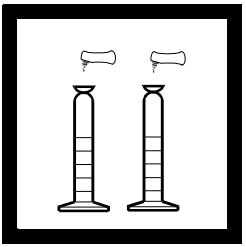
Note: *Use a TenSette pipet or 0.5 volumetric pipet.*



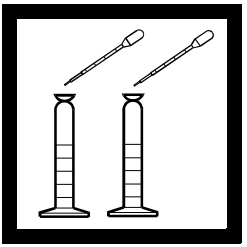
8. Add the contents of one Chromium 1 Reagent Powder Pillow to each cylinder. Stopper and invert several times to mix.



9. Wait 5 minutes.

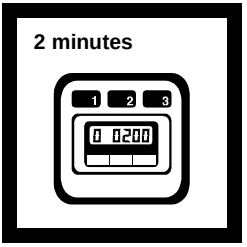


10. Add the contents of one Sodium Metabisulfite Reagent Powder Pillow to each cylinder. Stopper and invert several times to mix.



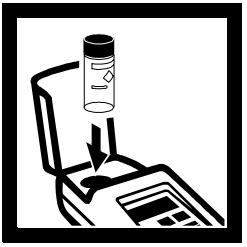
11. Add 1.0 mL of N,N'-Dimethyl-dithiooxamide Indicator Solution to each cylinder. Stopper and invert several times to mix.

Note: *Pressure may build up when the indicator is added. Use a paper towel to remove the stopper if this occurs.*



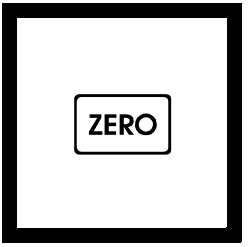
12. Wait 2 minutes.

13. Fill a 10-mL cell to the 10-mL line with the blank. Cap.



14. Place the blank in the cell holder.

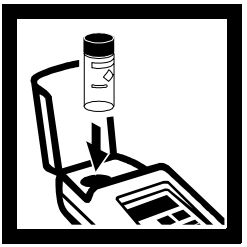
Note: *In bright light, it may be necessary to close the cell compartment cover.*



15. Press: **ZERO**

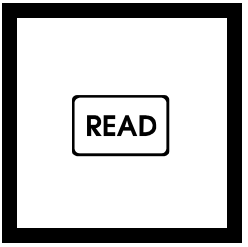
The display will count down to 0. Then the display will show 0 mg/L and the zero prompt will turn off.

16. Fill a 10-mL sample cell with 10 mL of the prepared sample.



17. Place the prepared sample in the cell holder.

Note: *In bright light, it may be necessary to close the cell compartment cover.*



18. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L palladium (Pd).

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Analyze activator bath samples as soon as possible after collection. Mix well before pipetting the sample.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 1.54 mg/L Pd concentration solutions, the standard deviation was ±1.79 mg/L Pd.

Testing zero concentration samples, the limit of detection was 6.19 mg/L Pd. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry,* **1980**, *52*, 2242-2249).

INTERFERENCES

Copper and nickel do not interfere under the reaction conditions of the test. Gold does not interfere in concentrations up to 50 mg/L.

SUMMARY OF METHOD

The test for palladium uses a hypobromite oxidation step which destroys the reducing agent present in palladium-tin activator solutions. The excess hypobromite is destroyed with sodium bisulfite. The palladium then reacts under acid conditions with N,N'-dimethyldithiooxamide to form a yellow color proportional to the palladium present. 2,2'Bipyridine is used to mask copper. This method is designed primarily for analyzing the palladium content of activator solutions used in the autocatalytic plating of printed circuit boards. Electrolytic and electroless palladium plating baths may also by analyzed by this method if the bath sample is first diluted to less than 250 mg/L palladium.

REQUIRED REAGENTS

	Cat. No.
Palladium Reagent Set (50 Tests)	23123-00
Includes: (2) 116-22, (1) 2043-99, (2) 134-49, (1) 23087-37, (2) 7095-99	

REQUIRED REAGENTS (continued)

Description	Quantity Per Test	Unit	Cat. No.
2,2'Bipyridine	0.2 g	5 g	116-22
Chromium 1 Reagent Powder Pillows	2 pillows	100/pkg	2043-99
Hydrochloric Acid, ACS	10 mL	500 mL	134-49
N,N'-Dimethyldithiooxamide Indicator Solution	2 mL	105 mL MDB	23087-32
Sodium Metabisulfite Reagent Powder Pillows	2 pillows	100/pkg	7095-99
Water, demineralized	40 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, mixing, tall-form, 25 mL	2	each	21190-40
DR/700 Filter Module Number 42.01	1	each	46242-00
Pipet, Mohr, 10 mL	1	each	20934-38
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	1	50/pkg	21856-96
Pipet Filler, safety bulb	1	each	14651-00
Spoon, measuring, 0.2 g	1	each	638-00

OPTIONAL APPARATUS

Cap for 10-and 25-mL sample cells		12/pkg	24018-12
Pipet, volumetric, Class A, 0.5 mL		each	14515-34
Sample Cell, 10-mL with screw cap		6/pkg	24276-06
Sample Cell, 25-mL with screw cap		6/pkg	24019-06

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PHOSPHORUS, ACID HYDROLYZABLE

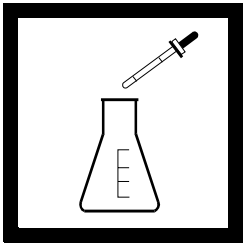
For water, wastewater, seawater

Hydrolysis to Orthophosphate Method*

1. Measure 25 mL of sample into a 50-mL erlenmeyer flask using a graduated cylinder.

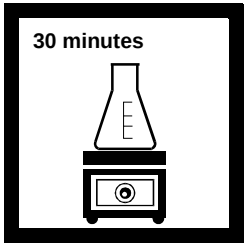
Note: *Wash all glassware with hydrochloric acid, 6 N. Rinse with demineralized water.*

Note: *If prompt analysis is impossible, see Sampling and Storage following these steps.*



2. Add 2.0 mL of Sulfuric Acid Solution, 5.25 N.

Note: *Use the 1-mL calibrated dropper provided.*

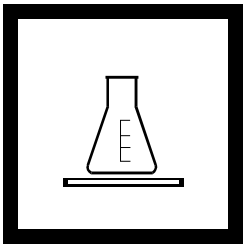


3. Place the flask (the prepared sample) on a hot plate. Boil gently for 30 minutes.

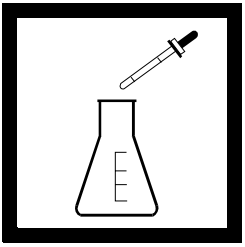
Note: *Maintain the volume near 20 mL by adding small amounts of demineralized water. Do not exceed 20 mL.*

Note: *This is more easily accomplished with a "double-boiler" technique. Place the flask in a pan or beaker of boiling water which is just deeper than the solution level in the flask. Continue boiling for 30 minutes.*

*Adapted from *Standard Methods for the Examination of Water and Wastewater*

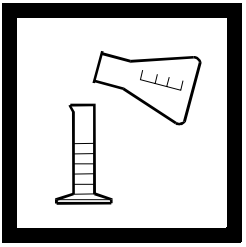


4. Cool the prepared sample to room temperature.



5. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution. Swirl to mix.

Note: *Use the 1-mL calibrated dropper provided.*



6. Pour the prepared sample into a graduated cylinder. Add demineralized water rinsings from the flask to return the volume to 25 mL. Proceed with the appropriate reactive phosphorus test.

Note: *Results of the reactive phosphorus test at this point will include the orthophosphate plus the acid-hydrolyzable (condensed) phosphate. The condensed phosphate concentration is determined by subtracting the results of a reactive phosphorus test on an untreated sample from this result.*

SAMPLING AND STORAGE

Most reliable results are obtained when samples are analyzed immediately. If prompt analysis is not possible, samples may be preserved up to 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

INTERFERENCES

If the sample is turbid, use 50 mL of sample and double the reagent volumes. Use 25 mL of the hydrolyzed sample to zero the instrument in the reactive phosphorus procedure. This compensates for any turbidity dissolved by this procedure.

SUMMARY OF METHOD

This procedure lists the necessary steps to convert condensed phosphate forms (meta-, pyro- or other polyphosphates) to orthophosphate before analysis. The procedure uses acid and heat to hydrolyze the sample. Organic phosphates are not converted to orthophosphate by this process, but a very small fraction may be unavoidably included in the result. Thus, the "acid hydrolyzable" phosphate results are primarily a measure of inorganic phosphorus. This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorous content of the sample.

The following reagents and apparatus are required in addition to those required for the reactive phosphorus test.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL* MDB	2450-32
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL* MDB	2449-32

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508-40
Flask, erlenmeyer, 50 mL	1	each	505-41

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Hydrochloric Acid, 6 N	500 mL	884-49
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Cylinder, graduated, 50 mL	each	508-41
Flask, erlenmeyer, 125 mL	each	505-43
Hot Plate, 3 $^{1}/_{2}$" diameter, 120 Vac	each	12067-01
Hot Plate, 3 $^{1}/_{2}$" diameter, 240 Vac	each	12067-02
Pad, cooling, 4" x 4"	each	18376-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00

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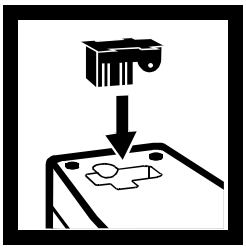
*Contact Hach for larger sizes.

PHOSPHORUS, REACTIVE (0 to 20 mg/L PO_4^{3-})

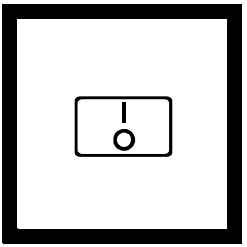
For water and wastewater

(also called Orthophosphate) Molybdovanadate Method*



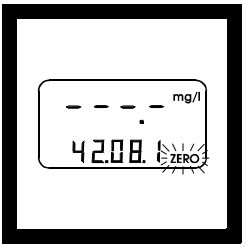
1. Install module **42.01** in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage, below.*

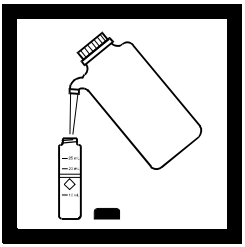


2. Press: **I/O**

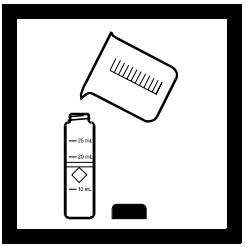
The display will show
420 nm
and module number
42.01



3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
42.08.1

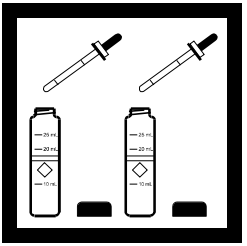


4. Fill a 25-mL cell to the 25-mL line with demineralized water (the blank). Cap.



5. Fill a second 25-mL cell to the 25-mL line with sample (the prepared sample). Cap.

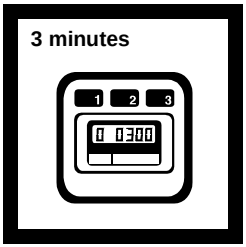
Note: *For proof of accuracy, use a 10.0 mg/L phosphate (3.3 mg/L phosphorus) standard solution (preparation given in Accuracy Check) in place of the sample.*



6. Add 1.0 mL of Molybdovanadate Reagent to each sample cell. Cap and invert several times to mix.

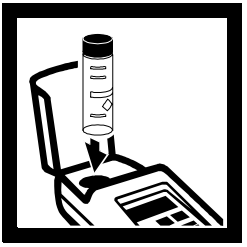
Note: *A yellow color will develop if phosphate is present. A small amount of yellow color will be present in the blank due to the reagent.*

Adapted from *Standard Methods for the Examination of Water and Wastewater, 12th ed.*



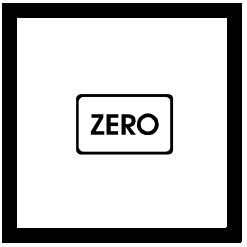
7. Wait 3 minutes.

Note: *If the sample concentration is greater than 24 mg/L, read at exactly 3 minutes or make a 1:1 dilution.*



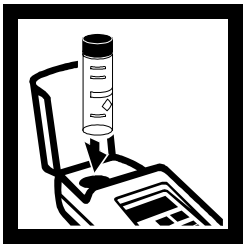
8. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



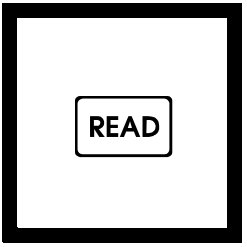
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



10. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample*



11. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L phosphate (PO_4). See Table 1 to convert results to other units.

Table 1. Conversion Factors

To convert reading from	To	Multiply by
mg/L PO_4^{3-}	mg/L P_2O_5	0.747
mg/L PO_4^{3-}	mg/L P	0.326

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with demineralized water. Do not use a commercial detergent because the phosphate content will contaminate the sample.

If samples cannot be analyzed the same day, adjust the pH to 2 or less by adding about 2 mL of sulfuric acid, ACS, per liter of sample. Store the sample at 4 °C (39 °F) or below. Samples can be stored up to 24 hours. For longer storage periods, add 4.0 mL of Mercuric Chloride Solution for each liter of sample taken and mix. Use of mercuric chloride is discouraged to minimize the amount of mercury released to the environment. Sample refrigeration is still required. Sample preserved with mercuric chloride must be spiked with 0.1 g sodium chloride per liter of sample, to bring the sodium chloride level to 50 mg/L or more if the sample is low in chloride. The addition of chloride prevents mercury interference in the test.

Before analysis, adjust the acidified sample to about pH 7 by adding 5 N Sodium Hydroxide Standard Solution. Mix thoroughly. Warm to room temperature before analyzing.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Phosphate Voluette Ampule Standard Solution, 500 mg/L as PO_4^{3-}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard respectively to three 25-mL water samples. Mix well.

c) Analyze each sample as described in the procedure and compare the results with that of the original test sample. Each 0.1-mL addition of standard should cause an increase of 2.0 mg/L PO_4^{3-} or 0.67 mg/L P.

d) If these increases do not occur, see Standard Additions (Section 1) for more information.

Standard Solution Method

A 10.0 mg/L phosphate standard can be prepared by pipetting 10.0 mL of a Phosphate Standard Solution, 50 mg/L PO_4^{3-}, into a 50-mL volumetric flask. Dilute to volume with demineralized water.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and representative lots of testing reagents. Testing 20.00 mg/L PO_4^{3-} concentration samples, the standard deviation was ±0.11 mg/L PO_4^{3-}.

Testing zero concentration samples, the limit of detection was 0.27 mg/L PO_4^{3-}. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical (Chemistry,* **1980,** *52,* 2242-2249).

INTERFERENCES.

Sulfide interference may be removed by oxidation with Bromine Water as follows:

a) Measure 25 mL of sample into a sample cell.

b) Add Bromine Water drop-wise with constant swirling until permanent yellow color develops.

c) Add Phenol Solution drop-wise until the yellow color just disappears. Proceed with Step 5.

Positive interferences are caused by silica and arsenate only if the sample is heated. Negative interferences are caused by arsenate, fluoride, thorium, bismuth, sulfide, thiosulfate, thiocyanate or excess molybdate. Blue color is caused by ferrous iron but this does not affect results if ferrous iron concentration is less than 100 mg/L.

Ions that do not interfere in concentrations up to 1000 mg/L are pyrophosphate, molybdate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, Al^{3+}, Fe^{3+}, Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{3+}, Li^{+}, Na^{+}, K^{+}, NH_4^{+}, Cd^{3+}, Mn^{2+}, NO_3^{-}, NO_2^{-}, SO_4^{2-}, SO_3^{2-}, Pb^{2+}, Hg^{+}, Hg^{2+}, Sn^{2+}, Cu^{2+}, Ni^{2+}, Ag^{+}, U^{4+}, Zr^{4+}, AsO_3^{-}, Br^{-}, CO_3^{2-}, $C10_4^{-}$, CN^{-}, IO_3^{-}, SiO_4^{4-}.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section 1).

SUMMARY OF METHOD

In the molybdovanadate method, orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid is formed. The intensity of the yellow color is proportional to the phosphate concentration.

REQUIRED REAGENTS

Description	Quantity Per Test	Units	Cat. No.
Molybdovanadate Reagent	2.0 mL	100 mL* MDB	20760-32
Water, demineralized	25 mL	4 L	272-56

REQUIRED APPARATUS

DR/700 Filter Module			
Number 42.01	1	each	46242-00

OPTIONAL REAGENTS

Bromine Water	29 mL*	2211-20
Hydrochloric Acid Solution, 1:1	500 mL	884-49
Mercuric Chloride Solution, 10 g/L	100 mL	14994-42
Phenol Solution, 30 g/L	29 mL	2112-20
Phosphate Standard Solution, 50 mg/L as PO_4^{3-}	500 mL	171-49
Phosphate Standard Solution, Voluette ampule, 500 mg/L as PO_4^{3-}, 10 mL	16/pkg	14242-10
Sodium Chloride, ACS	454 g	182-01
Sodium Hydroxide Standard Solution, 5.0 N	100 mL**MDB	2450-32
Sulfuric Acid, ACS	500 mL*	979-49
Molybdovanadate Reagent	1000 mL	20760-53

OPTIONAL APPARATUS

Caps for 10- and 25-mL sample cells	12/pkg	24018-12
Dispenser, fixed volume, 1.0 mL Repipet Jr.	each	21113-02
Flask, erlenmeyer, 50 mL	each	505-41
Flask, volumetric, Class A, 50 mL	each	14574-41
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00

OPTIONAL APPARATUS (continued)

Description	Per Test	Units	Cat. No.
Pipet, serological, 2.0 mL		each	532-36
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet, volumetric, Class A, 10.00 mL		each	14515-38
Pipet Filler		each	12189-00
Sample Cell, 10-mL with screw cap		each	24276-06
Sample Cell, 25-mL with screw cap		each	24019-06
Spoon, measuring, 0.1 g		each	511-00

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*Contact Hach for larger sizes

PHOSPHORUS, TOTAL

For water, wastewater and seawater

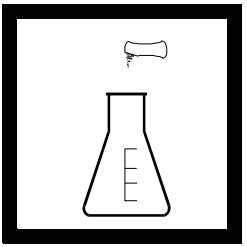
(also called Organic and Acid Hydrolyzable) Acid Persulfate Digestion Method*; USEPA accepted for reporting

1. Measure 25 mL of sample into a 50-mL erlenmeyer flask.

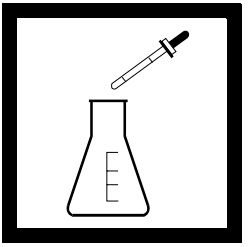
Note: *Use a graduated cylinder to measure the sample.*

Note: *Rinse all glassware with 1:1 Hydrochloric Acid Solution. Rinse again with demineralized water.*

Note: *If prompt analysis is impossible, see Sampling and Storage following these steps.*



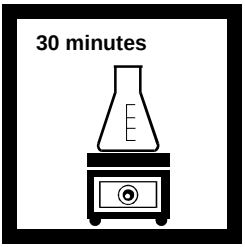
2. Add the contents of one Potassium Persulfate Powder Pillow. Swirl to mix.



3. Add 2.0 mL of 5.25 N Sulfuric Acid Solution.

Note: *Use the 1-mL calibrated dropper provided.*

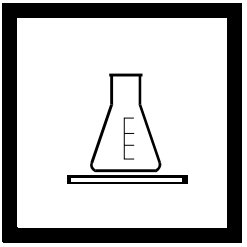
* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



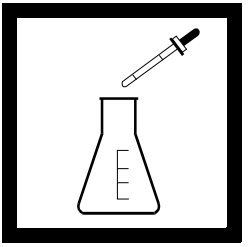
4. Place the flask on a hot plate. Boil gently for 30 minutes.

Note: *Maintain the volume near 20 mL by adding small amounts of demineralized water. Do not exceed 20 mL.*

Note: *This is more easily accomplished with a "double-boiler" technique. Place the flask in a pan or beaker of boiling water which is deeper than the solution level in the flask, Continue boiling for 30 minutes.*

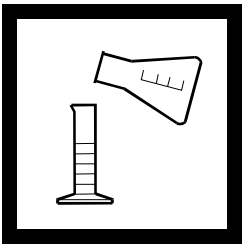


5. Cool the sample to room temperature.



6. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution. Swirl to mix.

Note: *Use the 1-mL calibrated dropper provided.*



7. Pour the sample into a 25-mL graduated cylinder. Using demineralized water rinsings from the flask, return the volume in the cylinder to 25 mL. Proceed with a reactive phosphorus test of the expected total phosphorus concentration range.

Note: *Results of the reactive phosphorus test at this point will include the organic phosphate plus the orthophosphate and the acid hydrolyzable (condensed) phosphate. The organic phosphate concentration is determined by subtracting the results of an acid hydrolyzable phosphorus test from this result. Make sure that both results are in the same units, either mg/L* PO_4^{3-} *or mg/L P before taking the difference.*

SAMPLING AND STORAGE

Most reliable results are obtained when samples are analyzed immediately. If prompt analysis is not possible, samples may be preserved up to 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

INTERFERENCES

For turbid samples, use 50 mL of sample and double the reagent quantities. Use 25 mL of the digested sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed by this procedure. For alkaline or highly buffered samples it may be necessary to use additional acid in Step 3 to drop the pH of the solution below 1.

SUMMARY OF METHOD

Phosphates present in organic and condensed inorganic forms (meta-, pyro-, or other polyphosphates) must be converted to orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate. Organically bound phosphates are thus determined indirectly by subtracting the result of an acid hydrolyzable phosphorus test from the total phosphorus result.

This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorus content of the sample. If the ascorbic acid (PhosVer 3) method is used to measure the reactive phosphorus, this method is EPA accepted for NPDES reporting.

The following reagents and apparatus are required beside those required for the reactive phosphorus test.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Potassium Persulfate Powder Pillows	1 pillow	50/pkg	2451-66
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL*MDB	2450-32

REQUIRED REAGENTS (continued)

Description	Quantity Per Test	Unit	Cat. No.
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL*MDB	2449-32
Water, demineralized	25 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, 25 mL	1	each	508-40
Flask, erlenmeyer, 50 mL	1	each	505-41

OPTIONAL REAGENTS

Hydrochloric Acid, 6 N (1:1)	500 mL	884-49
Sodium Hydroxide Solution, 5.0 N	1 L	2450-53

OPTIONAL APPARATUS

Cylinder, graduated, 50 mL	each	508-41
Flask, erlenmeyer, 125 mL	each	505-43
Hot Plate, 3 $^{1}/_{2}$-inch diameter, 120 Vac	each	12067-01
Hot Plate, 3 $^{1}/_{2}$-inch diameter, 240 Vac	each	12067-02
Pads, cooling, 4" x 4"	each	18376-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00

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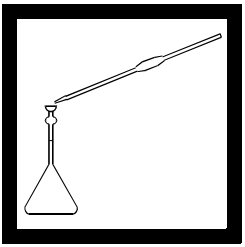
RHODIUM (0 to 14 g/gal)

For surface finishing solutions

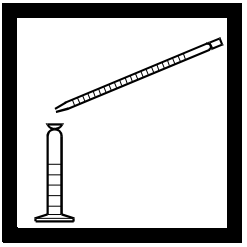
N,N'-Dimethyldithiooxamide Method*



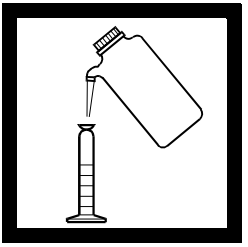
1. Dilute the rhodium bath solution by pipetting 1.0 mL of bath solution into a 250-mL volumetric flask. Fill the flask to the mark with demineralized water. Cap and invert at least 10 times to mix.

Note: *If the bath solution has more than 15 g/gal of rhodium a larger dilution is necessary.*

Note: *The DR/700 must be calibrated before samples can be analyzed. See Calibration section.*

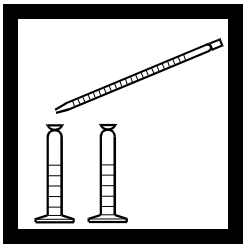


2. Fill a 25-mL graduated mixing cylinder to the 10-mL mark with the diluted bath solution (the prepared sample).



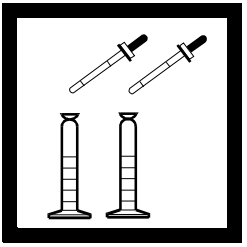
3. Fill a second 25-mL graduated mixing cylinder to the 10-mL mark with demineralized water (the blank).

*User calibration required; range is approximate

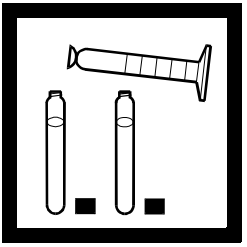


4. Carefully add 15.0 mL of concentrated Hydrochloric Acid to each cylinder. Stopper and invert to mix.

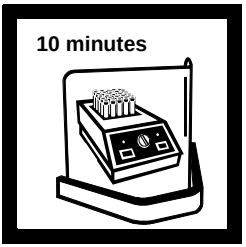
Note: *Use a pipet bulb and Mohr pipet.*



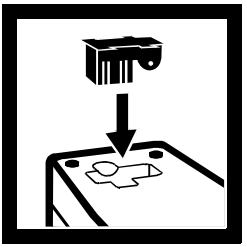
5. Using the 1.0 mL calibrated dropper, add 1.0 mL of N,N'-Dimethyldithiooxamide Indicator Solution to each cylinder. Stopper and invert to mix.



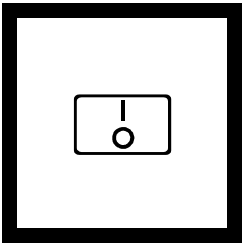
6. Pour 5 mL of each prepared solution into a heavy walled 16 x 100 mm culture tube. Cap tightly and wipe the tube walls to remove any liquid or fingerprints.



7. Place the tubes in a COD reactor at 150 ºC for 10 minutes.

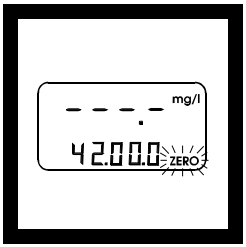


8. Install module **42.01** in a DR/700.



9. Press: **I/O**

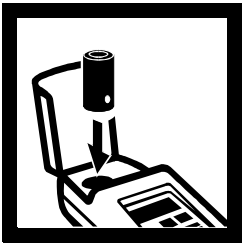
The display will show **420 nm** and module number **42.01**



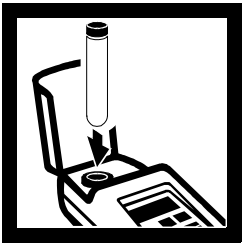
10. After 2 seconds, the display will show a program number, concentration units and the zero prompt. Press **PROGRAM** until the display shows program number

42.000

The upper display will show the S1 concentration.

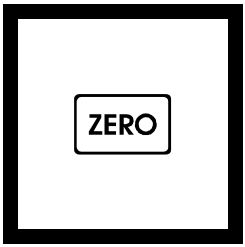


11. Fully insert a COD Vial Adapter into the cell holder with the tabs in the square slot.

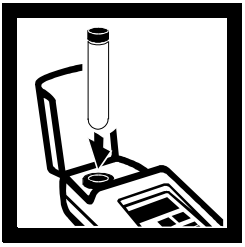


12. After the 10 minute period, grasp the cap of the hot tube and place the blank in the adapter with the Hach logo facing to the front of the instrument.

Note: *Measurements in bright sunlight should be avoided.*

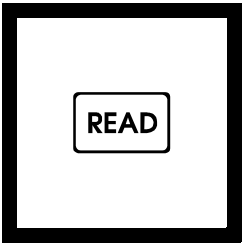


13. Press: **ZERO**
The display will count down to 0. Then the display will show 0.00 and the zero and S1 prompts will turn off.



14. Grasp the prepared sample by the cap and place it in the adapter with the Hach logo facing to the front of the instrument

Note: *Measurements in bright sunlight should be avoided.*



15. Press: **READ**
The display will count down to 0. Then the display will show the results in mg/L Rh in the diluted bath sample.

Note: *To convert results to other units, see Table 1.*

Note: *If necessary, use the following formula to correct diluted bath concentrations to actual bath concentration:*

$$\frac{\text{Diluted Bath (mg/L) Rh X Flask Volume}}{\text{Pipet Volume}} = \text{Bath mg/L Rh}$$

Table 1. Conversion Factors

To convert results from	To	Multiply by
mg/L Rh	g/L Rh	0.001
mg/L Rh	g/gal Rh	0.00378

CALIBRATION

If a 1.0 to 250 mL dilution is used in Step 1, calibrating with a 15.0 mg/L rhodium standard is proportional to having 3750 mg/L, 3.765 g/L or 14.2 g/gal of rhodium in the original bath. When analyzing samples, the DR/700 can display the diluted bath concentration or one of the proportional values. It will display in the same way as it was calibrated.

To make a 15.0 mg/L rhodium standard, pipet 15.0 mL of 1000 mg/L Rhodium Standard Solution into a 1000-mL volumetric flask. Dilute to volume with demineralized water. Cap the flask and invert at least 10 times to mix. This is Standard 2. Demineralized water is Standard 1.

Perform Steps 2 to 11 of the rhodium procedure using the 15 mg/L rhodium standard as the diluted bath solution in Step 2.

Start to perform the Operator Program Calibrating Using Two Standards procedure. For Standard 1's concentration, make the display show 0.000 mg/L or zero of another concentration unit of your choice.

After the tubes have been heated for 10 minutes, grasp the cap of the hot "blank" tube (prepared Standard 1) and put it in the adapter.

For Standard 2's concentration make the display show 15.0 mg/L or a proportional concentration in other units. The hot "sample" is prepared Standard 2. Complete the calibration as described.

SAMPLING AND STORAGE

Several locations within the bath should be sampled and combined to obtain a representative sample of the bath solution. Store samples in clean plastic or glass bottles. Analyze bath sample as soon as possible after collection.

INTERFERENCES

Metals normally present in rhodium baths (nickel, iron, copper, tin, lead and zinc) do not interfere under the reaction conditions of the test.

SUMMARY OF METHOD

Rhodium present in sulfate- or phosphate-type electrolytic plating baths reacts when heated with N,N'- Dimethyldithiooxamide to give a yellow color proportional to the amount of rhodium present.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat No.
Hydrochloric Acid, ACS	30.0 mL	2.8 kg	134-06
N-N'-Dimethyldithiooxamide Indicator Solution	2.0 mL	100 mL MDB	23807-32
Rhodium Standard Solution 1000 mg/L	15.0 mL	100 mL	23209-42
Water, demineralized	1.25 L	4 L	272-56

REQUIRED APPARATUS

Cap, for 22758-00	1	each	22411-00
COD Reactor	1	each	45600-00
COD Vial Adapter	1	each	46008-00
Cylinder, mixing, 25 mL, tall form	1	each	21190-40
Flask, volumetric Class A, 250 mL	1	each	14574-46
Flask, volumetric Class A, 1000 mL	1	each	14574-53
Laboratory Bench Safety Shield	1	each	23810-00
DR/700 Filter Module Number 42.01	1	each	46242-00
Pipet Filler, safety bulb	1	each	14651-00
Pipet, Mohr, 25 mL	1	each	20934-40
Pipet, volumetric Class A, 1.00 mL	1	each	14515-35
Pipet, volumetric Class A, 15.00 mL	1	each	14515-39
Tube, culture, 16 x 100 mm	1	each	22758-00

OPTIONAL APPARATUS

Pipet Filler, 3-valve	each	12189-00
Test Tube Rack, stainless steel	each	18641-00

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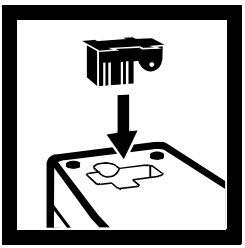
In the U.S.A.--Call 800-227-4224 toll-free for more information.
Outside the U.S.A.--Contact the Hach Office or distributor serving you.

SILICA, HR (0 to 40 mg/L)

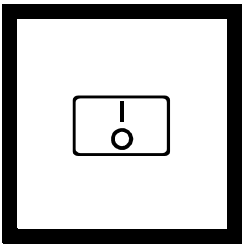
For water and wastewater

Silicomolybdate Method*



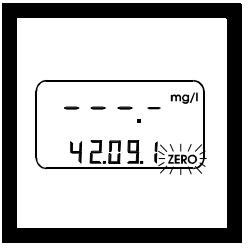
1. Install module
42.01
in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*



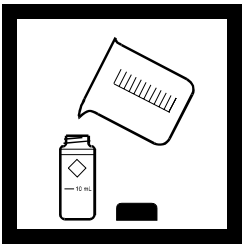
2. Press: **I/O**

The display will show
420 nm
and module number
42.01



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
42.09.1

*Adapted from *Standard Methods for the Examination of Water and Wastewater.*



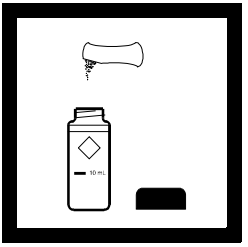
4. Fill a 10-mL cell to the 10-mL line with sample.

Note: *A 25-mL sample can be tested by using 25-mL sample cells and optional reagents.*

Note: *For proof of accuracy, use a 50 mg/L Silica Standard Solution (listed under Optional Reagents) in place of the sample.*

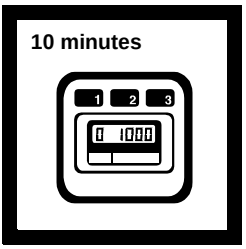
Note: *Sample temperature should be 15 to 25°C (59 to 77°F).*

5. Add the contents of one Molybdate Reagent Powder Pillow For High Range Silica. Cap and invert to mix. Remove cap.



6. Add the contents of one Acid Reagent Powder Pillow For High Range Silica. Cap and invert to mix.

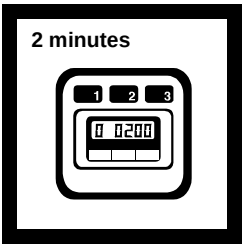
Note: *Silica or phosphate will cause a yellow color to develop.*



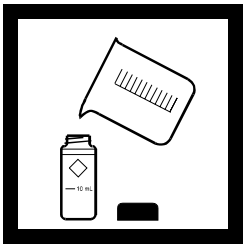
7. Wait 10 minutes.

8. Add the contents of one Citric Acid Powder Pillow to the sample cell (the prepared sample). Cap and invert to mix.

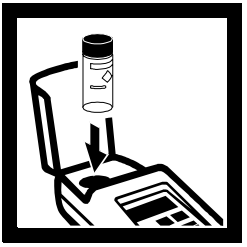
Note: *The yellow color due to any phosphate present will be removed.*



9. Wait 2 minutes.

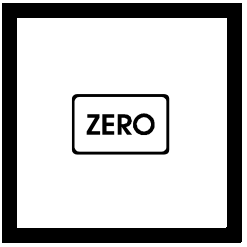


10. Fill a second 10-mL cell to the 10-mL line with sample (the blank).



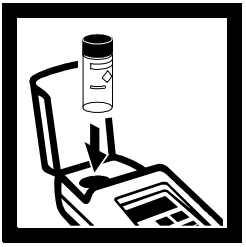
11. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



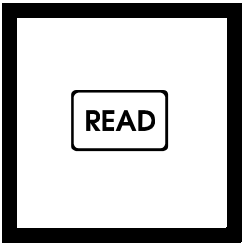
12. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



13. Within 3 minutes after the 2 minute period, place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



14. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L silica.

SAMPLING AND STORAGE
Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. Store samples up to seven days at 4 °C (39 °F) or below. Warm samples to room temperature before analyzing.

ACCURACY CHECK
Standard Additions Method
a) Open a High Range Silica Standard Solution Pillow, 250 mg/L SiO_2.

b) Use the TenSette Pipet to add 0.10 mL, 0.20 mL, and 0.30 mL of standard, respectively, to three 25-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The silica concentration should increase 1.0 mg/L for each 0.10 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method
To check the accuracy of the method, use the Silica Standard Solutions, 10 and 25 mg/L as SiO_2, listed under Optional Reagents. Analyze according to the above procedure using demineralized water as the blank.

STATISTICAL EVALUATION
A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 20.0 mg/L SiO_2 concentration samples, the standard deviation was ±0.42 mg/L SiO_2.

Testing zero concentration samples, the limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry*, **1980**, 52, 2242-2249)

INTERFERENCES
Color and turbidity interferences are eliminated by zeroing the instrument with the original water sample.

Sulfides and large amounts of iron interfere.

There is no interference from phosphate below 50 mg/L PO_4^{3-}. At 60 mg/L PO_4^{3-}, an interference of minus 2% is observed. At 75 mg/L, the interference is minus 11%.

Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these "molybdate-unreactive" forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the Examination of Water and Wastewater*, Silica--Digestion with Sodium Bicarbonate. A longer reaction time of sample with the molybdate and acid reagents, before the addition of citric acid, is often helpful in lieu of the bicarbonate pretreatment.

SUMMARY OF METHOD

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid preferentially destroys the phosphate complexes. Silica is then determined by measuring the remaining yellow color.

REQUIRED REAGENTS

	Cat. No.
High Range Silica Reagent Set (100 tests)	22443-00
Includes: (2) 1042-66, (1)14548-99, (2) 1041-66	

Description	Quantity Per Test	Unit	Cat. No
Acid Reagent Powder Pillows for High Range Silica	1	100/pkg	21074-69
Citric Acid Powder Pillows	1	100/pkg	21062-69
Molybdate Reagent Powder Pillows for High Range Silica	1	100/pkg	21073-69

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 42.01	1	each	46242-00

OPTIONAL REAGENTS

High Range Silica Reagent Set (100 Tests) for 25-mL samples			22443-00
Includes: (2) 1042-66, (1) 14548-99, (2) 1041-66			
Silica Standard Solution, 10 mg/L		500 mL	1403-49

OPTIONAL REAGENTS

Description	Quantity Per Test	Unit	Cat. No
Silica Standard Solution, 50 mg/L		237 mL	1117-31
Acid Reagent Powder Pillows for High Range Silica (25 mL sample)	1 pillow	100/pkg	21074-69
Citric Acid Powder Pillows (25 mL sample)	1 pillow	100/pkg	21062-69
Molybdate Reagent Powder Pillows for High Range Silica (25 mL)	1 pillow	100/pkg	21073-69
Silica Standard Solution, 10 mg/L	1	500 mL	1403-49
Silica Standard Solution, 50 mg/L		237 mL	1117-31
Silica Standard Solution Pillows, 250 mg/L as SiO_2		16/pkg	14244-10
Sodium Bicarbonate, ACS		454 g	776-01
Sulfuric Acid Standard Solution, 1.000 N		100 mL MDB	1270-32

OPTIONAL APPARATUS

Cap for 10 and 25-mL sample cells		12/pkg	24018-12
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Sample Cell, 10-mL with screw cap		6/pkg	24276-06
Sample Cell, 25-mL with screw cap		6/pkg	24019-06
Standard Methods for the Examination of Water and Wastewater, 19th edition		each	22708-00
Thermometer, - 20 to 105 °C		each	1877-01

For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

Module 45.01
450 nm

Hach warrants equipment it manufactures against defective materials or workmanship for at least one year from the shipping date. Warranties do not apply to limited-life electrical components such as batteries. Full warranty information is located on the reverse side of Hach invoices.

IMPORTANT NOTICE

A lamp intensity adjustment must be performed:
- Before first or initial use
- When new filter modules are installed
- On each filter module when the lamp is replaced

Refer to Lamp Intensity Adjustment in the instrument manual.

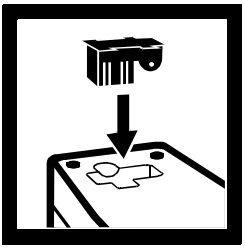
Table of Contents for 450-nm parameters

BARIUM (0 to 300 mg/L)

For water, wastewater, oil-field water and seawater

Turbidimetric Method* (Powder Pillows or AccuVac Ampuls)

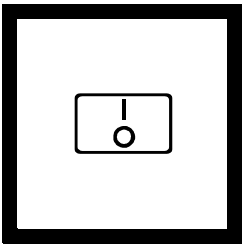
USING POWDER PILLOWS



1. Install module
45.01
in a DR/700.

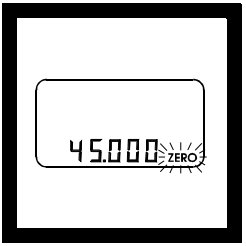
Note: *If sample cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of samples before analysis.*

Note: *The DR/700 must be calibrated before sample analysis. See Calibration following these steps.*



2. Press: **I/O**

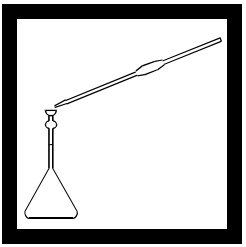
The display will show
450 nm
and module number
45.01



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. Press **PROGRAM** until the display shows program number
45.000

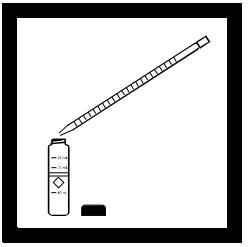
The upper display will show the concentration of a previous calibration standard. If desired, press the **UP ARROW** key to display the other standard.

*Adapted from Snell and Snell, *Colorimetric Methods of Analysis*, Vol. II, 769 (1959)

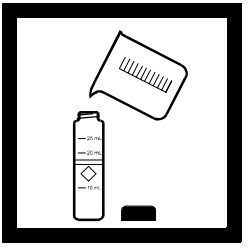


4. Prepare the displayed standard. For each 10 mg/L displayed, pipet 1.00 mL from a 5000-mg/L Barium Voluette Ampule into a 500-mL volumetric flask. Add demineralized water to the mark. Cap and invert 10 times to mix.

Example: *To make a 50 mg/L standard, pipet 5.00 mL into the flask. For a zero standard, use only demineralized water.*

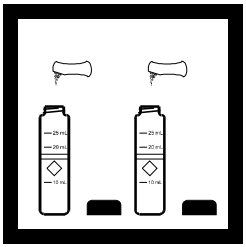


5. Fill a 25-mL cell to the 25-mL line with the displayed standard. Cap.



6. Fill a 25-mL cell to the 25-mL line with sample.

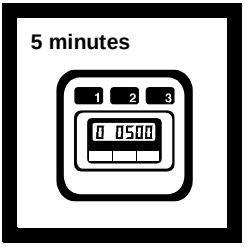
Note: *Filter highly colored or turbid water samples using labware listed under Optional Apparatus. Large amounts of color or turbidity will interfere and cause high readings.*



7. Add the contents of one BariVer 4 Barium Reagent Powder Pillow to each sample cell. Cap and invert several times to mix.

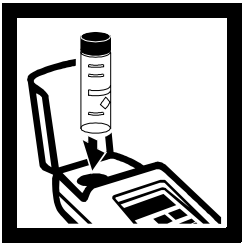
Note: *A white turbidity will develop if barium is present.*

Note: *If the BariVer 4 Reagent does not dissolve readily, use a 25-mL graduated mixing cylinder. Mix the reagent with the sample in the cylinder, then pour it into the sample cell.*



8. Wait 5 minutes.

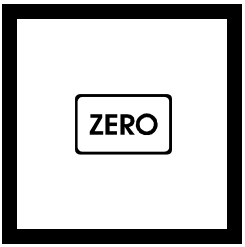
Note: The sample should not be disturbed during the five minute turbidity development period.



9. Within 5 minutes after the 5 minute period, place the treated standard in the cell holder.

Note: *If the display is blank, repeat Steps 2 and 3.*

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



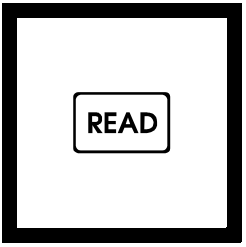
10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 g/L and the zero prompt will turn off.

Note: *This step adjusts the DR/700 to the calibration curve previously entered.*

11. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*

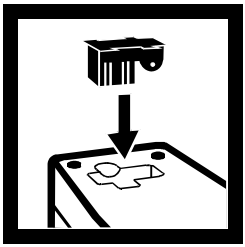


12. Press: **READ**

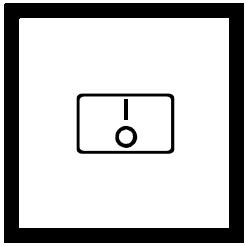
The display will count down to 0. Then the display will show the results in mg/L barium.

Note: *After each test, clean the sample cell with soap, water and a brush. A white film of barium sulfate will form on the sample cell walls and cause errors in future determinations if cleaning is not done soon after each test.*

USING ACCUVAC AMPULS

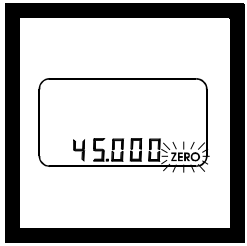


1. Install module
45.01
in a DR/700.



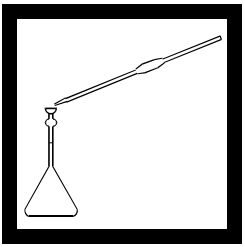
2. Press: **I/O**

The display will show
450 nm
and module number
45.01



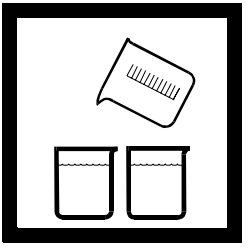
3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. Press **PROGRAM** until the display shows program number
45.000

The upper display will show the concentration of a previous calibration standard. If desired, press the **UP ARROW** key to display the other standard.



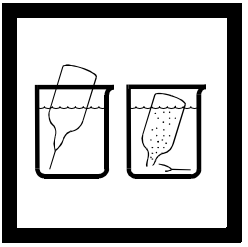
4. Prepare the displayed standard. For each 10 mg/L displayed, pipet 1.00 mL from a 5000-mg/L Barium Voluette Ampule into a 500-mL volumetric flask. Add demineralized water to the mark. Cap and invert 10 times to mix.

Example: *To make a 50 mg/L standard, pipet 5.00 mL into the flask. For a zero standard, use only demineralized water.*



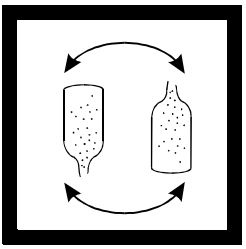
5. Collect at least 40 mL of sample in a 50-mL beaker. Collect at least 40 mL of the displayed standard in another 50-mL beaker.

Note: *Filter highly colored or turbid samples using labware listed under Optional Apparatus.*



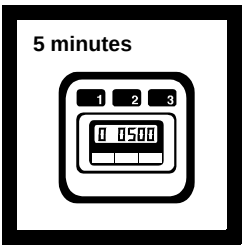
6. Fill a Barium AccuVac Ampul with the displayed standard. Fill a second AccuVac Ampul with sample.

Note: *Keep the tip immersed while ampul fills completely.*



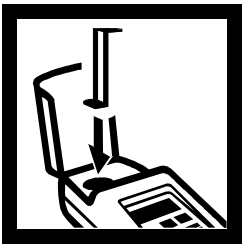
7. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A white turbidity will develop if barium is present.*

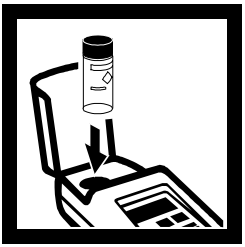


8. Wait 5 minutes.

Note: *The sample should not be disturbed during the five minute turbidity-development period.*

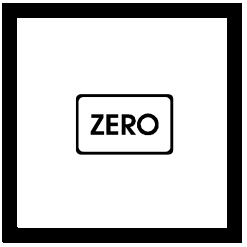


9. Insert the AccuVac Vial Adapter into the cell holder.



10. Within five minutes after the five minute development time, place the blank in the cell holder.

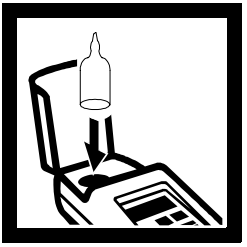
Note: *In bright sunlight, it may be necessary to close the cell compartment cover.*



11. Press: **ZERO**

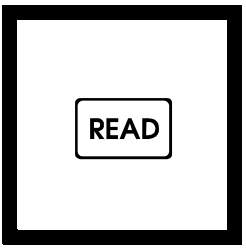
The display will count down to 0. Then the display will show the concentration of the standard and the zero prompt will turn off.

Note: *This step adjusts the DR/700 to the calibration curve previously entered.*



12. Place the prepared sample in the cell holder.

Note: *In bright light close the cell compartment cover.*



13. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L barium.

SAMPLING AND STORAGE

Collect samples in an acid cleaned glass or plastic container. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the pH to 5 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Barium Voluette Ampule Standard, 5000 mg/L Ba.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 25-mL samples and mix each thoroughly (for AccuVac ampuls, use 50-mL beakers).

c) Analyze each sample as described above. The barium concentration should increase 20 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

CALIBRATION

A new calibration is necessary for each new manufacturing lot of BariVer 4 Reagent Powder Pillows or AccuVac Ampuls. A new calibration is also needed when the water sample to be tested has a barium concentration which is not between that of the standards used to calibrate the DR/700.

To perform a calibration, make 2 barium standards using the technique described in Step 4 of the method. Make them so that one has a slightly lower and the other a slightly higher barium concentration than that anticipated to be in the water sample. Process each in the same way as the "displayed standard" described in this method.

Next, perform paragraph 3.2.4.1, Calibration Using Two Prepared Standards, in the DR/700 instrument manual. Either standard can be used as Standard 1 or Standard 2.

INTERFERENCES

The following may interfere when present in concentrations exceeding those listed below:

Silica	500 mg/L
Sodium Chloride	130,000 mg/L as NaCl
Magnesium	100,000 mg/L as $CaCO_3$
Calcium	10,000 mg/L as $CaCO_3$
Strontium	Interferes at any level

If strontium is known to be present, the total concentration between barium and strontium may be expressed as a PS (Precipitated by Sulfate). While this does not distinguish between barium and strontium, it gives an accurate indication of scaling tendency.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

SUMMARY OF METHOD

The BariVer 4 Barium Reagent Powder combines with barium to form a barium sulfate precipitate, which is held in suspension by a protective colloid. The amount of turbidity present caused by the fine white dispersion of particles is directly proportional to the amount of barium present.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
BariVer 4 Barium Reagent Powder Pillows	2 pillows	50/pkg	12064-66
Barium Standard Solution, 5000 mg/L, Voluette ampule, 10 mL	varies	16/pkg	14251-10
Water, demineralized	500 mL	4 L	272-56

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 45.01	1	each	46245-00

REQUIRED APPARATUS (Using Powder Pillows) (cont.)

Description	Quantity Per Test	Unit	Cat. No.
Flask, volumetric, 500 mL	1	each	14574-49
Pipet, Mohr, 25 mL	1	each	20934-40
Pipet, volumetric, 1.00 mL, Class A	1	each	14515-35
Pipet, volumetric, 2.00 mL, Class A	1	each	14515-36
Pipet, volumetric, 3.00 mL, Class A	1	each	14515-03
Pipet, volumetric, 4.00 mL, Class A	1	each	14515-04
Pipet, volumetric, 5.00 mL, Class A	1	each	14515-37
Pipet, volumetric, 10.00 mL, Class A	1	each	14515-38

REQUIRED REAGENTS (Using AccuVac Ampuls)

BariVer 4 Barium Reagent AccuVac Ampuls	1 ampul	25/pkg	25130-25
Barium Standard Solution, 5000 mg/L, Voluette ampule, 10 mL	varies	16/pkg	14251-10
Water, demineralized	500 mL	4 L	272-56

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac Vial	1	each	46025-00
Beaker, 50 mL	1	each	500-41
Flask, volumetric, 500 mL	1	each	14574-49
Pipet, Mohr, 25 mL	1	each	20934-40
Pipet, volumetric, 1.00 mL, Class A	1	each	14515-35
Pipet, volumetric, 2.00 mL, Class A	1	each	14515-36
Pipet, volumetric, 3.00 mL, Class A	1	each	14515-03
Pipet, volumetric, 4.00 mL, Class A	1	each	14515-04
Pipet, volumetric, 5.00 mL, Class A	1	each	14515-37

REQUIRED APPARATUS (Using AccuVac Ampuls) (cont.)

Description	Quantity Per Test	Unit	Cat. No.
Pipet, volumetric, 10.00 mL, Class A	1	each	14515-38

OPTIONAL REAGENTS

Barium Standard Solution, 50 mg/L Ba		500 mL	1951-49
Nitric Acid, ACS		500 mL	152-49
Nitric Acid Solution, 1:1		500 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N		1 L	2450-53

OPTIONAL APPARATUS

Ampule Breaker Kit		each	21968-00
AccuVac Snapper Kit		each	24052-00
Adapter, AccuVac Vial for DR/700		each	46025-00
Brush		each	690-00
Filter Paper, folded, 12.5 cm		100/pkg	1894-57
Funnel, poly, 65 mm		each	1083-67
pH Indicator Paper, 1 to 11 pH		5 rolls/pkg	391-33
pH Meter, EC10, portable		each	50050-00
Pipet, serological, 2 mL		each	532-36
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Filler, safety bulb		each	14651-00
Sample cell, 10-mL with screw cap		6/pkg	24276-06
Sample cell, 25-mL with screw cap		6/pkg	24019-06

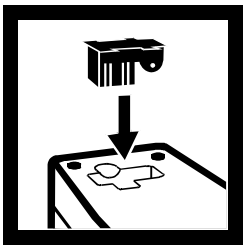
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

CHLORIDE (0 to 20.0 mg/L Cl^-)

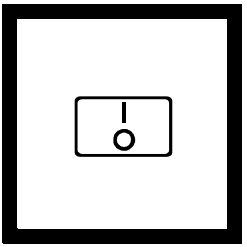
For water and wastewater

Mercuric Thiocyanate Method*



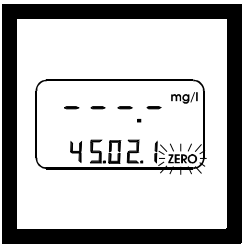
1. Install module number
45.01
in a DR/700.

Note: *Samples can be stored for at least 28 days at room temperature in glass or plastic bottles.*



2. Press: **I/O**

The display will show
455 nm
and module number
45.01

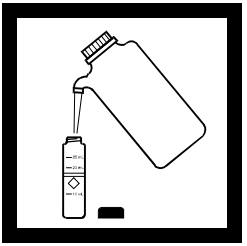


3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
45.02.1

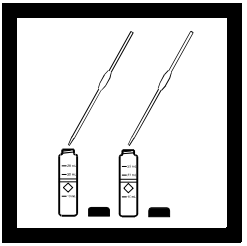
4. Fill a 25-mL sample cell to the 25-mL line with sample.

Note: *Filter turbid samples through a moderately rapid filter paper before analysis.*

Note: *For proof of accuracy, use a 10.0 mg/L chloride standard (preparation given in Accuracy Check) in place of the sample.*

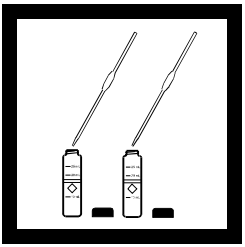


5. Fill a 25-mL sample cell to the 25-mL line with demineralized water (the blank).



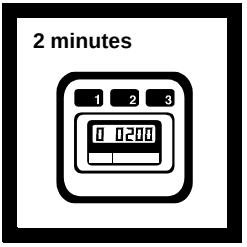
6. Pipet 2.0 mL of Mercuric Thiocyanate Solution into each sample cell. Swirl to mix.

*Adapted from Zall, et. al., *Analytical Chemistry*, **1956**, 28 (11), 1665

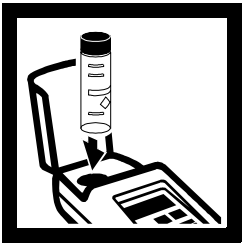


7. Pipet 1.0 mL of Ferric Ion Solution into each cell. Cap each cell and invert several times to mix.

Note: *An orange color will develop if chloride is present.*

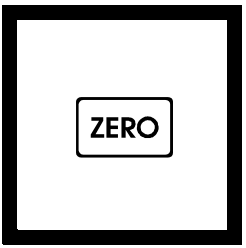


8. Wait 2 minutes.



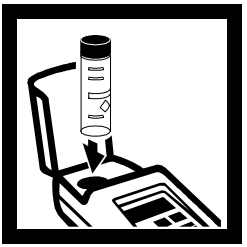
9. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



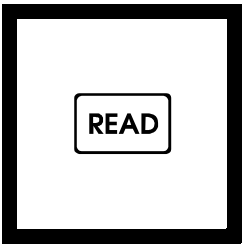
10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L and the zero prompt will turn off.



11. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L chloride (Cl^-).

ACCURACY CHECK

Standard Additions Method

a) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of Chloride Standard Solution, 1000 mg/L as Cl^-, to each of three 25-mL water samples. Mix each thoroughly.

b) Analyze each sample as described above.

c) The chloride concentration should increase 4.0 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 10.0 mg/L chloride standard solution by diluting 5.00 mL of Chloride Standard Solution, 1000 mg/L, to 500 mL with demineralized water.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 12.0 mg/L Cl^- concentration samples, the standard deviation was ±0.34 mg/L Cl^-.

Testing zero concentration samples, the limit of detection was 0.14 mg/L Cl^-. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

The pH of the sample after addition of reagents should be about 2. If the sample is strongly acid or alkaline, adjust a portion of sample before testing to a pH of about 7. Use either 5.0 N Sodium Hydroxide Standard Solution or a 1:5 dilution of perchloric acid. Use pH paper, as most pH electrodes will contaminate the sample with chloride.

SUMMARY OF METHOD

Chloride in the sample reacts with mercuric thiocyanate to form mercuric chloride and liberate thiocyanate ion. Thiocyanate ions react with the ferric ions to form an orange ferric thiocyanate complex.

The amount of this complex is proportional to the chloride concentration. Chloride at these levels also can be determined directly using the Chloride Ion Selective Electrode (Cat. No. 50255-00)

REQUIRED REAGENTS

	Cat. No.
Chloride Reagent Set (50 Tests*)	23198-00
Includes: (1) 22122-42, (1) 22121-31	

Description	Quantity Per Test	Unit	Cat. No.
Ferric Ion Solution	2 mL	100 mL	22122-42
Mercuric Thiocyanate Solution	4 mL	236 mL	22121-31
Water, demineralized	25 mL	4 L	272-56

REQUIRED APPARATUS

DR/700 Filter Module Number 45.01	1	each	46245-00
Pipet, volumetric, 1.0 mL	1	each	515-35
Pipet, volumetric, 2.0 mL	1	each	515-36
Pipet Filler, safety bulb	1	each	14651-00
OR			
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette pipet	1	50/pkg	21856-96

OPTIONAL REAGENTS

Chloride Standard Solution, 1000 mg/L as Cl	500 mL	183-49
Perchloric Acid, ACS, 70%	680 g	757-65
Sodium Hydroxide Standard Solution, 5.0 N	59 mL SCDB	2450-26

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Filter Paper, folded, mod. rapid, 12.5 cm	100/box	692-57
Flask, erlenmeyer, 125 mL	each	505-43
Flask, volumetric, 500 mL	each	547-49
Funnel, filtering, polypropylene, 75 mm	each	1083-68

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
pH Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

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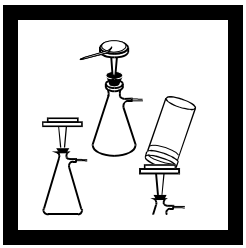
Outside the U.S.A. - Contact the Hach office or distributor serving you.

*50 tests equals 25 samples and 25 blanks.

COLOR, TRUE AND APPARENT (0 to 500 units)

For water, wastewater and seawater

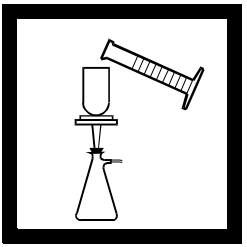
APHA Platinum-Cobalt Method*



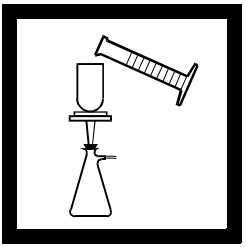
1. Assemble the filtering apparatus (membrane filter, filter holder, filter flask and aspirator).

Note: *To test for apparent color, do not filter; omit Steps 1 to 3.*

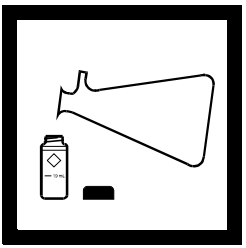
Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*



2. Rinse the filter by pouring about 50 mL of demineralized water through the filter.

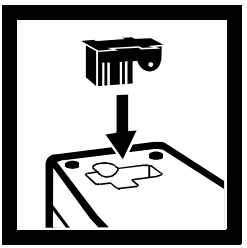


3. Pour another 50 mL of demineralized water through the filter.

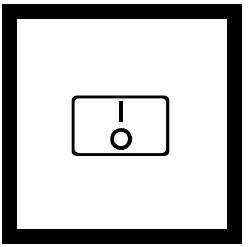


4. Fill a 10-mL cell to the 10-mL line with filtered demineralized water (the blank). Discard the excess.

Note: *For apparent color, use unfiltered demineralized water.*



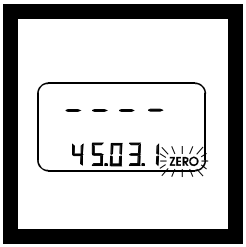
5. Install module
45.01
in a DR/700.



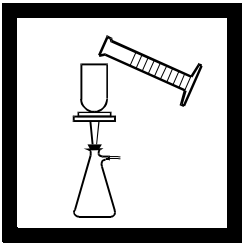
6. Press: **I/O**

The display will show
450 nm
and module number
45.01

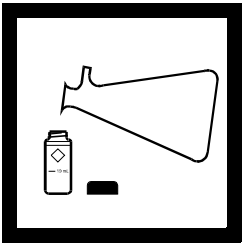
*Adapted from *Standard Methods for the Examination of Water and Wastewater* and *Wat. Res.* Vol. 30, No. 11, pp. 2771-2775. 1996.



7. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **45.03.1**

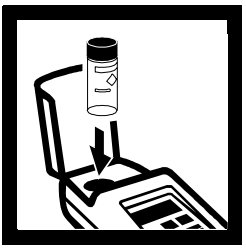


8. Pour about 50 mL of sample through the filter.



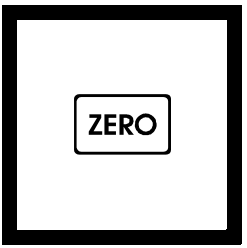
9. Fill a second 10-mL cell to the 10-mL line with the filtered sample (the prepared sample).

Note: *For proof of accuracy, use a 250 unit platinum-cobalt color standard solution (preparation given in Accuracy Check) in place of the filtered sample.*



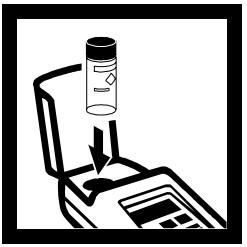
10. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



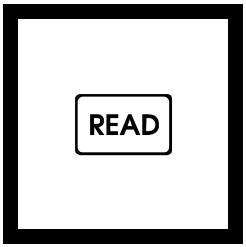
11. Press: **ZERO**

The display will count down to 0. Then the display will show 0 Units PtCo color and the zero prompt will turn off.



12. Place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



13. Press: **READ**

The display will count down to 0. Then the display will show the results in PtCo color units.

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after the collection. If prompt analysis is impossible, full bottles completely and cap tightly. Avoid excessive agitation or prolonged contact with air. Sample can be stored for 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

ACCURACY CHECK

Standard Solution Method

A 500 platinum-cobalt units color standard solution is available under Optional Reagents for checking test accuracy. A 250 platinum-cobalt units standard can be made by pipetting 50.0 mL of the 500 platinum-cobalt units standard into a 100-mL volumetric flask and diluting to volume with demineralized water.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 500 color units concentration solutions, the standard deviation was ±7.0 color units.

Testing zero concentration samples, the limit of detection was 4.2 mg/L color units. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

SUMMARY OF METHOD

Color may be expressed as "apparent" or "true" color. The apparent color includes that from dissolved materials plus that from suspended matter. By filtering or centrifuging out the suspended materials, the true color can be determined. The procedure describes true color analysis. If apparent color is desired, it can be determined by measuring an unfiltered water sample. The stored program is used for both forms of color.

The stored program is calibrated in color units based on the APHA-recommended standard of 1 color unit being equal to 1 mg/L platinum as chloroplatinate ion.

REQUIRED REAGENTS

Description	Quantity Per Test	Units	Cat. No.
Water, demineralized	50 mL	4 L	272-56

REQUIRED APPARATUS

Aspirator, vacuum	1	each	2131-00
DR/700 Filter Module Number 45.01	1	each	46245-00
Filter Holder, 47 mm, 300 mL graduated	1	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	1	100/pkg	13530-00
Flask, filtering, 500 mL	1	each	546-49
Stopper, No 7, one hole	1	6/pkg	2119-07
Tubing, rubber	1	12 ft	560-19

OPTIONAL REAGENTS

Color Standard Solution, 500 platinum-cobalt units	1 L	1414-53

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, 50 mL	each	1081-41
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, volumetric, Class A, 50 mL	each	14515-41
Sample Cell, 10-mL with screw cap	6/pkg	24276-06

OPTIONAL APPARATUS (continued)

Description	Units	Cat. No.
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

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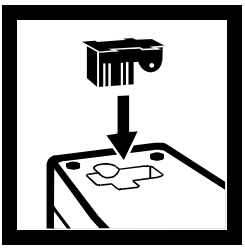
Outside the U.S.A. - Contact the Hach office or distributor serving you.

HYDRAZINE (0 to 400 µg/L)

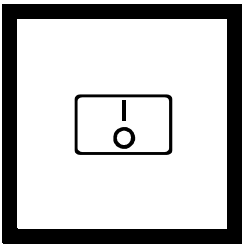
For boiler water/feedwater, water and seawater

p-Dimethylaminobenzaldehyde Method*



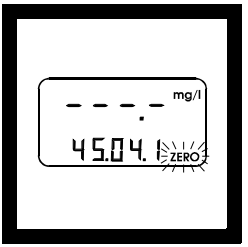
1. Install module
45.01
in a DR/700.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



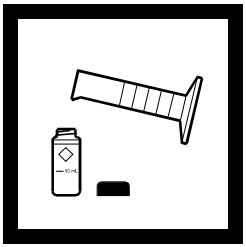
2. Press: **I/O**

The display will show
450 nm
and module number
45.01

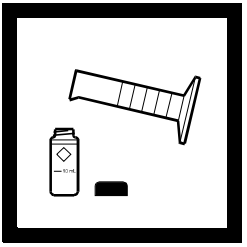


3. After 2 seconds, the display will show the program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
45.04.1

*Adapted from *ASTM Manual of Industrial Water*, D1385-78, 376 (1979)



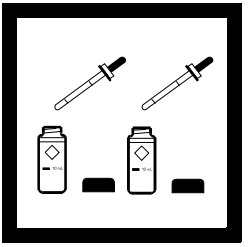
4. Pour 10.0 mL of demineralized water into a 10-mL sample cell (the blank) using a graduated cylinder.



5. Pour 10.0 mL of sample into a second 10-mL sample cell (the prepared sample) using a graduated cylinder.

Note: *For proof of accuracy, use a 100 µg/L hydrazine standard solution (preparation given in Accuracy Check) in place of the sample.*

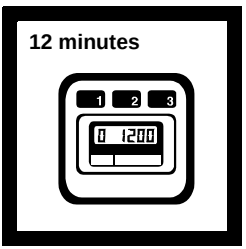
Note: *Sample temperature should be 21°C ± 4°C (70°C ± 7°F).*



6. Add 0.5 mL of HydraVer 2 Hydrazine Reagent to each sample cell. Cap and invert several times to mix.

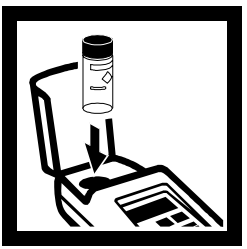
Note: *A yellow color will develop if hydrazine is present.*

Note: *HydraVer 2 Reagent will cause a faint yellow color to appear in the blank.*



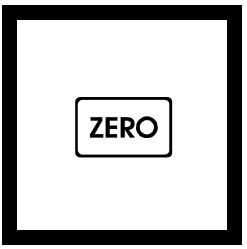
7. Wait 12 minutes.

Note: *Complete Steps 8 through 10 during this time.*



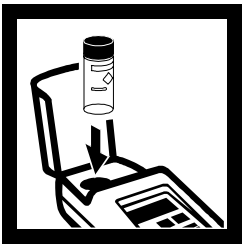
8. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



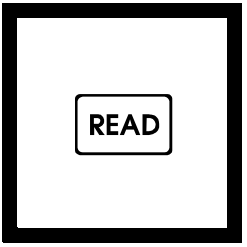
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 µg/L and the zero prompt will turn off.



10. Place the prepared sample in the cell holder. Cap.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



11. Immediately after the 12-minute period press: **READ**

The display will count down to 0. Then the display will show the results in µg/L hydrazine.

SAMPLING AND STORAGE

Samples collected in glass or plastic bottles should be filled completely and capped tightly. Avoid excessive agitation or exposure to air. Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

ACCURACY CHECK

Standard Solution Method

To assure the accuracy of the test, prepare the following solutions:

a) Prepare a 25 mg/L stock solution by dissolving 0.1016 g of hydrazine sulfate in demineralized water then diluting to 1000 mL. Prepare stock solution daily.

b) Prepare a 0.1 mg/L (1000 µg/L) hydrazine working solution by diluting 0.4 mL of the 25 mg/L stock solution to 100 mL with demineralized water. Prepare just before analysis.

c) Use the working solution in place of the sample in Step 5. The result should be 100 µg/L hydrazine.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two

representative lots of testing reagents. Testing 300 µg/L N_2H_4 concentration samples, the standard deviation was ±3.4 µg/L N_2H_4.

Testing zero concentration samples, the limit of detection was 2.4 µg/L N_2H_4. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980**, 52, 2242-2249).

INTERFERENCES

For highly colored or turbid samples, prepare a blank by oxidizing the hydrazine in a portion of the sample. This can be accomplished with a 1:1 mixture of demineralized water and a household bleach such as Clorox. Add one drop of the mixture to 25 mL of sample in a graduated mixing cylinder and invert to mix. Use this solution in Step 4, in place of demineralized water, to prepare the blank. There are no other common interferences.

Ammonia does not interfere up to 10 mg/L. At 20 mg/L, a positive error of up to 20% may occur.

Morpholine does not interfere up to 10 mg/L.

SUMMARY OF METHOD

Hydrazine in the sample reacts with the p-dimethyl-aminobenzaldehyde from the HydraVer 2 Reagent to form a yellow color which is proportional to the hydrazine concentration.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
HydraVer 2			
Hydrazine Reagent	1 mL	100 mL* MDB	1790-32
Water, demineralized	25 mL	4 L	272-56

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508-40
DR/700 Filter Module			
Number 45.01	1	each	46245-00

OPTIONAL REAGENTS

Hydrazine Sulfate, ACS	100 g	742-26

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, mixing, 25 mL	each	1896-40
Flask, volumetric, 100 mL	each	547-42
Flask, volumetric, 1000 mL	each	547-53
Pipet, serological, 1 mL	each	532-35
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Thermometer, -20 to 105 °C	each	1877-01

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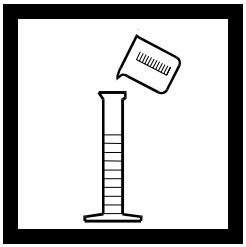
*Contact Hach for larger sizes.

PHENOLS (0 to 0.200 mg/L)

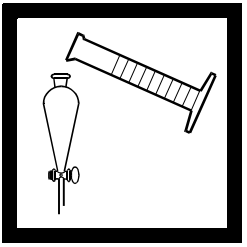
For water, wastewater and seawater

4-Aminoantipyrine Method*, USEPA accepted for reporting (distillation is required; see Section 1)**

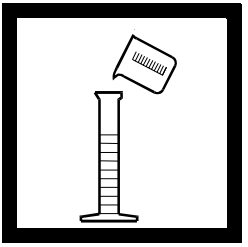


1. Measure 300 mL of demineralized water in a 500-mL graduated cylinder.

Note: *Analyze samples within four hours to avoid oxidation; see Sampling and Storage following these steps.*

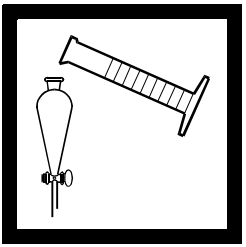


2. Pour the measured demineralized water into a 500-mL separatory funnel (the blank).

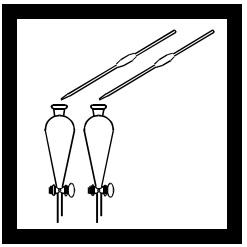


3. Measure 300 mL of sample in a 500-mL graduated cylinder.

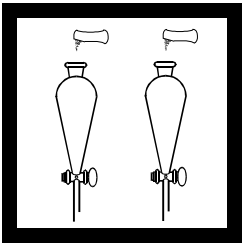
Note: *For proof of accuracy, a phenol standard solution (preparation given in Accuracy Check) can be used in place of the sample.*



4. Pour the measured sample into another 500-mL separatory funnel (the prepared sample).



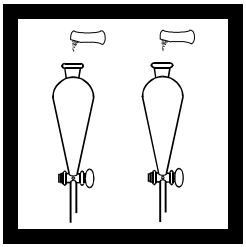
5. Add 5 mL of Hardness 1 Buffer to each separatory funnel. Stopper. Shake to mix.



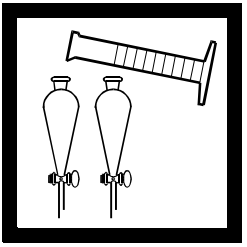
6. Add the contents of one Phenol Reagent Powder Pillow to each separatory funnel. Stopper. Shake to dissolve.

Note: *Spilled reagent will affect test accuracy and is hazardous to skin and other materials.*

*Adapted from *Standard Methods for the Examination of Water and Wastewater*
**Procedure is equivalent to USEPA Method 420.1 for wastewater

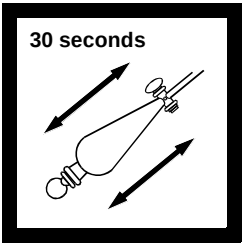


7. Add the contents of one Phenol 2 Reagent Powder Pillow to each separatory funnel. Stopper. Shake to dissolve.

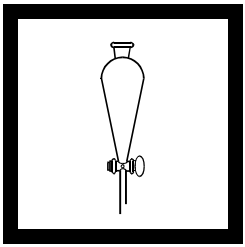


8. Add 30 mL of chloroform to each separatory funnel. Stopper each funnel.

Caution: Use chloroform only with proper ventilation.

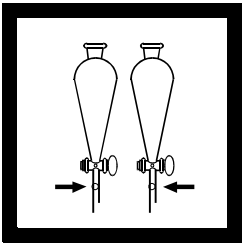


9. Holding the stopper in, invert each funnel and temporarily vent. Shake each funnel briefly and vent. Then vigorously shake each funnel for a total of 30 seconds.

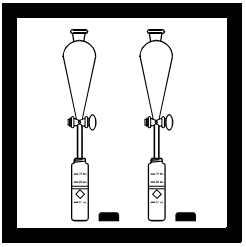


10. Remove the stoppers. Allow both funnels to stand until the chloroform settles to the bottom of the funnel.

Note: *The chloroform will be yellow to amber if phenol is present.*



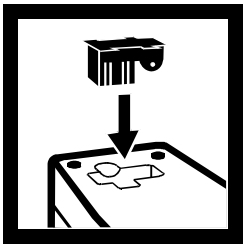
11. Insert a pea-size cotton plug into the delivery tube of each funnel.



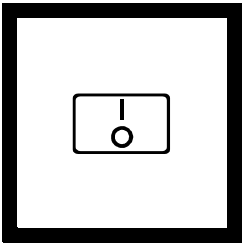
12. Drain the chloroform layers into separate sample cells - one for the blank and one for the prepared sample.

Note: *Filtering the chloroform layer through dry cotton will remove any suspended water or particles. The volume of the chloroform extract will be about 25 mL due to slight solubility of chloroform in water.*

Note: *Proceed promptly through the rest of the procedure since the chloroform will evaporate, causing high readings.*

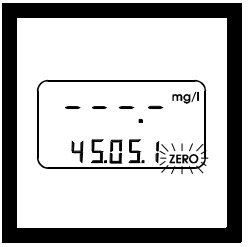


13. Install module **45.01** in a DR/700.

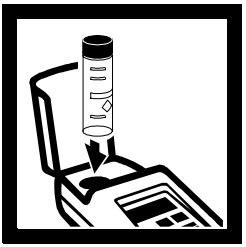


14. Press: **I/O**

The display will show **450 nm** and module number **45.01**

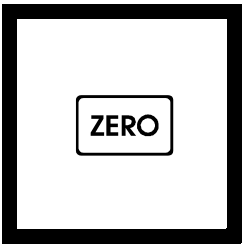


15. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **45.05.1**



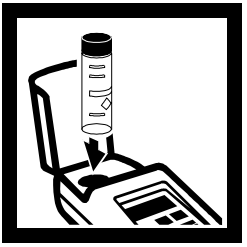
16. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright light, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



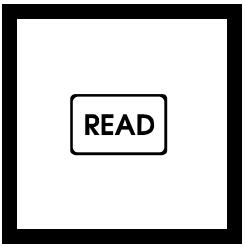
17. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L.



18. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright light, it may be necessary to close the cell compartment cover. Transfer 10 mL of the sample solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



19. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L phenols.

SAMPLING AND STORAGE

Most reliable results are obtained when samples are analyzed within four hours after collection. The following storage instructions are necessary **only** when prompt analysis is impossible. Collect 500 mL of sample in clean glass containers and add the contents of two Copper Sulfate Powder Pillows. Adjust the sample pH to 4 or below with 10% Phosphoric Acid Solution. Store at 4 °C (39 °F) or lower and analyze within 24 hours.

ACCURACY CHECK

Standard Solution Method

Verify accuracy of the test by performing the analysis procedure using known phenol standard solutions in place of the test sample. For greatest accuracy, standard solutions should be analyzed periodically to verify test accuracy and when new reagent lots are first used. Prepare standards as follows:

a) Weigh out 1.00 g of phenol, ACS. Transfer to a 1-liter volumetric flask. Dilute to the mark with freshly boiled and cooled demineralized water. This is a 1-g/L stock solution.

b) Transfer 1 mL of the 1-g/L stock solution to a 100-mL volumetric flask. Dilute to the mark with demineralized water. This is a 10-mg/L working solution.

c) Prepare 0.06, 0.12 and 0.18 mg/L standard solutions by using a pipet to add 3, 6 and 9 mL of the 10-mg/L working solution, respectively, to

three separate 500-mL volumetric flasks. Dilute each to the mark with demineralized water.

d) Perform the procedure with each of the three standard solutions and verify that the test results are correct.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 0.100 mg/L phenols concentration solutions, the standard deviation was ±0.0047 mg/L phenols.

Testing zero concentration samples, the limit of detection was 0.006 mg/L phenols. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

The sample pH must be between 3 and 11.5 for the best results. In the presence of sulfides or suspended matter, the following pretreatment will be necessary:

a) Take a water sample by filling a clean 500-mL graduated cylinder to the 350-mL mark. Pour the sample into a clean 500-mL erlenmeyer flask.

b) Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix.

c) Filter 300 mL of the sample through a folded filter paper. Use this solution in Step 4.

Interference can be caused by reducing agents and oxidizing agents such as chlorine.

Sample distillation as described in the following steps will eliminate interferences.

a) Set up the distillation apparatus for the test by assembling the general purpose apparatus as shown in the Hach Distillation Apparatus Manual. Use the 500-mL erlenmeyer flask to collect the distillate. It may be

necessary to elevate the flask with a laboratory jack. Place a stirring bar into the distillation flask.

b) Measure 300 mL of sample in a 500-mL graduated cylinder. Pour it into the distillation flask.

c) Using a serological pipet, add 1 mL of Methyl Orange Indicator Solution to the distillation flask. Turn on the heater power switch. Set the stir control to 5.

d) Add Phosphoric Acid Solution, 10%, drop-wise, until the indicator changes from yellow to orange.

e) Add the contents of one Copper Sulfate Powder Pillow and allow to dissolve. (Omit this step if copper sulfate was used to preserve the sample.)

f) Turn on the water and adjust so a constant flow of water is maintained through the condenser. Set the heat control setting to 10.

g) Turn off the still after collecting 275 mL of distillate.

h) Fill a 25-mL graduated cylinder to the 25-mL mark with demineralized water. Turn the still back on. Add the water to the flask. Resume heating until another 25 mL of distillate is collected.

i) Using a graduated cylinder, remeasure the distillate to be certain 300 mL has been collected. The distillate may now be analyzed for phenol by the 4-aminoantipyrine method.

SUMMARY OF METHOD

The 4-aminoantipyrine method determines all ortho- and meta-substituted phenols or napthols but not para-substituted phenols. These phenols react with 4-aminoantipyrine in the presence of potassium ferricyanide to form a colored antipyrine dye. This dye is then extracted from aqueous solution with chloroform and the color is measured at 460 nm. Sensitivity of the method varies with the type of phenolic compound. Because a water sample may contain various types of phenolic compounds, the results of the test are expressed as the equivalent concentration of phenol. Wastewater and seawater samples may require pretreatment.

REQUIRED REAGENTS

	Cat. No.
Phenols Reagent Set (100 Tests)	22439-00
Includes: (3) 424-49, (2) 14458-17, (4) 1836-66, (8) 872-68	

Description	Quantity Per Test	Unit	Cat. No.
Buffer Solution, Hardness 1	10 mL	500 mL	424-49
Chloroform, ACS	60 mL	4 L	14458-17
Phenol 2 Reagent Powder Pillow	2 pillows	50/pkg	1836-66
Phenol Reagent Powder Pillow	2 pillows	25/pkg	872-68
Water, demineralized	300 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cotton Balls	2	100/pkg	2572-01
Cylinder, 50 mL graduated	1	each	508-41
Cylinder, 500 mL graduated	1	each	508-49
DR/700 Filter Module Number 45.01	1	each	46245-00
Funnel, 500 mL separatory	2	each	520-49
Pipet, volumetric, Class B, 5 mL	1	each	515-37
Ring, support, 4"	2	each	580-01
Stand, support, 5" x 8" base	1	each	563-00

OPTIONAL REAGENTS

Copper Sulfate Powder Pillows	50/pkg	14818-66
Methyl Orange Indicator Solution	500 mL	148-49
Phenol, ACS	113 g	758-14
Phosphoric Acid Solution, 10%	105 mL	14769-32
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	2418-99

OPTIONAL APPARATUS

Balance, analytical	each	22310-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, 25 mL, graduated	each	508-40
Distillation Apparatus General Purpose Accessories	each	22653-00

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
Distillation Apparatus Heater, 115 V	each	22744-00
Distillation Apparatus Heater, 230 V	each	22744-02
Filter Paper, 12.5 cm folded	100/pkg	1894-57
Flask, 500 mL erlenmeyer	each	505-49
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 500 mL	each	14574-49
Flask, volumetric, Class A, 1000 mL	each	14574-53
Funnel, 65 mm poly	each	1083-67
Jack, laboratory	each	22743-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, serological, 1.0 mL	each	532-35
Pipet, volumetric, Class A, 1 mL	each	14515-35
Pipet, volumetric, Class A, 3 mL	each	14515-03
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

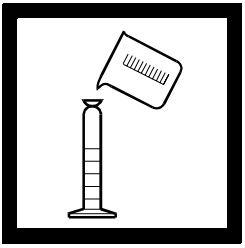
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

POTASSIUM (0 to 8.0 mg/L)

For water, wastewater and seawater

Tetraphenylborate Method*

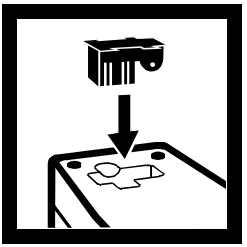


1. Fill a graduated mixing cylinder with 25 mL of sample.

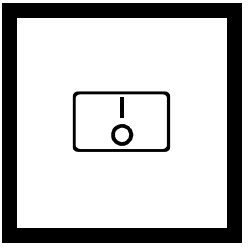
Note: *Filter highly turbid or colored samples.*

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

Note: *The DR/700 must be calibrated before sample analysis. See Calibration following these steps.*



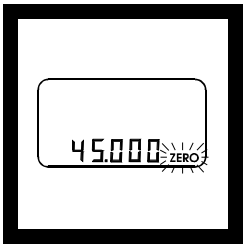
2. Install module **45.01** in a DR/700.



3. Press: **I/O**

The display will show **450 nm** and module number **45.01**

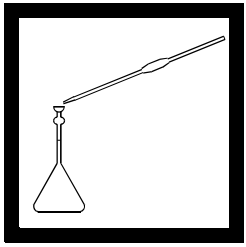
*User calibration required; range is approximate.



4. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. Press **PROGRAM** once or twice until the lower display shows program number

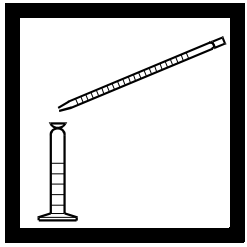
45.000

The upper display will show the concentration of a previous calibration standard. If desired, press the **UP ARROW** key to display the other standard.

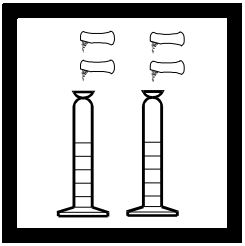


5. Make the displayed standard. For each mg/L displayed, pipet 1 mL from a 250-mg/L Potassium Voluette Ampule into a 250-mL volumetric flask. Add demineralized water to the mark. Cap and invert 10 times to mix.

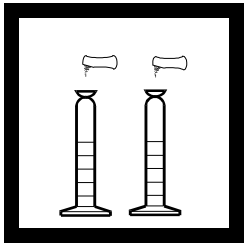
Note: *For example, to make a 4 mg/L standard, pipet 4.00 mL of the 250-mg/L standard solution into the flask. To make a 0-mg/L standard, use only demineralized water.*



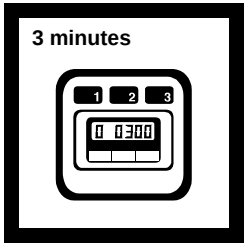
6. Fill a 25-mL graduated mixing cylinder with 25 mL of the displayed standard.



7. Add the contents of one Potassium 1 Reagent Powder Pillow and the contents of one Potassium 2 Reagent Solution Pillow to each cylinder. Stopper and invert several times until the powder dissolves.

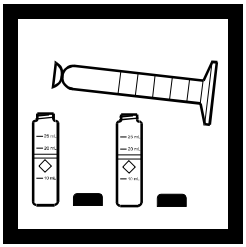


8. After the solution clears, add the contents of one Potassium 3 Reagent Powder Pillow to each cylinder. Stopper and shake for 30 seconds.

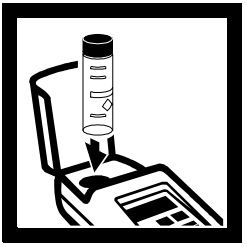


9. Wait 3 minutes.

Note: *Complete Steps 10-14 within 7 minutes after the 3-minute waiting period.*

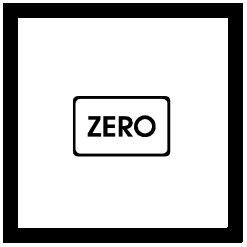


10. Pour the solutions from each cylinder into separate 25-mL sample cells. Cap each cell.



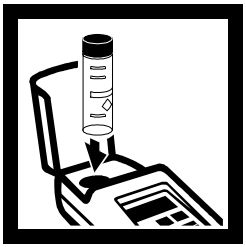
11. Place the treated standard in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright light, it may be necessary to close the cell compartment cover. Transfer 10 mL of the treated standard solution to a 10-mL cell. If the 10-mL cell is used for the treated standard, another 10-mL cell must be used for the prepared sample.*



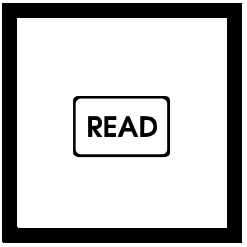
12. Press: **ZERO**

The display will count down to 0. Then the display will show the concentration of the standard and the zero prompt will turn off.



13. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright light, it may be necessary to close the cell compartment cover. Transfer 10 mL of the prepared sample to a 10-mL cell. If the 10-mL cell is used for the treated standard, another 10-mL cell must be used for the prepared sample.*



14. Within 7 minutes after the 3-minute period, press: **READ**

The display will count down to 0. Then the display will show the results in mg/L potassium (K).

Note: *Clean the cells with soap and a brush after use.*

CALIBRATION

A new calibration is needed for each new lot of Potassium 3 Reagent Powder Pillows. A new calibration is also needed when the expected potassium concentration of the water sample to be tested is not between the standards last used to calibrate the DR/700.

To perform a calibration, make 2 potassium standards. Make them so that one has somewhat less potassium and the other somewhat more potassium than the expected potassium concentration of the water sample. Make each as described in step 5 of the potassium procedure. Continue by doing steps 2 and 6 through 10, processing both standards like the displayed standard.

Next, perform paragraph 3.2.4.1, Calibration Using Two Prepared Standards, in the DR/700 Instrument Manual. Either standard can be

used as Standard 1 or Standard 2. Complete the calibration within 7 minutes after the 3-minute period.

SAMPLING AND STORAGE

Collect samples in acid-washed plastic bottles. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored at least six months at room temperature. Before analysis, adjust the pH to 4 to 5 with 5.0 N sodium hydroxide. Do not measure pH in the sample container with a pH electrode, as this will introduce potassium from the filling solution. Use pH paper or pour off sample and test pH in a separate beaker. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section 1) for more information.

ACCURACY CHECK

Standard Addition Method

a) Snap the neck off a Potassium Voluette Ampule Standard Solution, 250 mg/L.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The potassium concentration should increase 1.0 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section 1) for more information.

INTERFERENCES

The following ions do not interfere below the concentration shown:

Ammonium Nitrogen	15 mg/L as N
Calcium	7000 mg/L as $CaCO_3$
Chloride	15,000 mg/L
Magnesium	6000 mg/L as $CaCO_3$

SUMMARY OF METHOD

Potassium in the sample combines with sodium tetraphenylborate, an insoluble white solid. The amount of turbidity produced is proportional to the potassium concentration.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Potassium 1 Reagent Pillows	1 pillow	25/pkg	14321-98
Potassium 2 Reagent Pillows	1 pillow	25/pkg	14322-98
Potassium 3 Reagent Pillows	1 pillow	50/pkg	14323-96
Potassium Standard Solution, Voluette Ampule, 250 mg/L, 10 mL	10 mL	16/pkg	14790-10
Water, demineralized	250 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, mixing, graduated, 25 mL	1	each	1896-40
DR/700 Filter Module Number 45.01	1	each	46245-00

OPTIONAL REAGENTS

Nitric Acid, ACS	500 mL	152-49
Nitric Acid, 1:1	500 mL	2540-49
Potassium Standard Solution, 5 mg/L	500 mL	20583-49
Potassium Standard Solution, 1000 mg/L	100 mL	22404-42
Sodium Hydroxide Solution, 5.0 N	59 mL SCDB	2450-26

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, volumetric, 1000 mL	each	547-53
Funnel, poly, 65 mm	each	1083-67
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
Pipet Filler, 3-valve	each	12189-00
Pipet Filler, safety bulb	each	14651-00
Pipet, Mohr, 25 mL	each	20934-40
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
Sample Cell, 10-mL with screw cap	6/pkg	24278-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

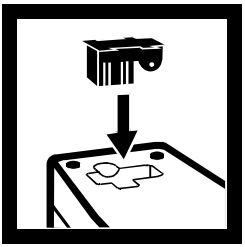
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

SODIUM CHROMATE (0 to 1,000 mg/L)

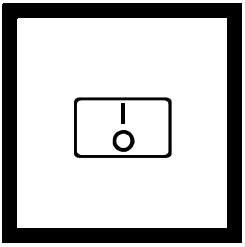
For water, wastewater and seawater

Direct Colorimetric Method



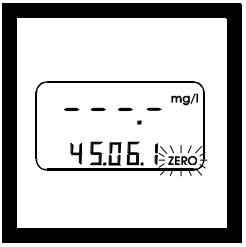
1. Install module **45.01** in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage, below.*

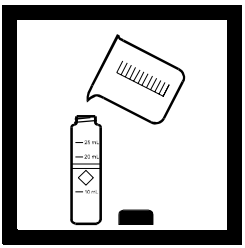


2. Press: **I/O**

The display will show **450 nm** and module **45.01**



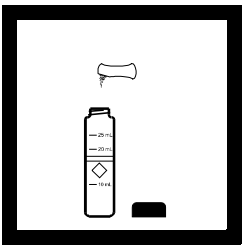
3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **45.06.1**



4. Fill a 25-mL cell to the 25-mL line with sample.

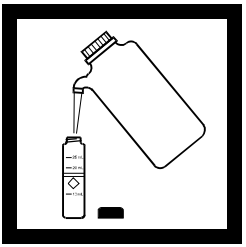
Note: *For proof of accuracy, use a 1000 mg/L Sodium Chromate Standard Solution (listed under Optional Reagents) in place of the sample.*

Note: *Filter turbid samples using the labware listed under Optional Apparatus.*

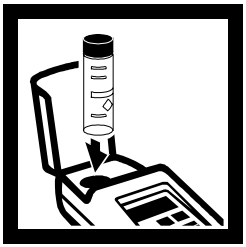


5. Add the contents of one Neutralizing Reagent Powder Pillow. Cap and invert to mix (the prepared sample).

Note: *The Neutralizing Reagent Powder Pillow is necessary only if the sample is orange or yellow-orange. If the color is in doubt, add the powder pillow contents.*

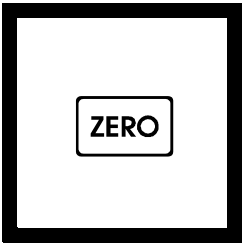


6. Fill a 25-mL cell to the 25-mL demineralized water (the blank). Cap.



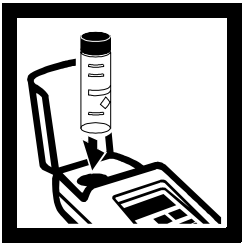
7. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10-mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



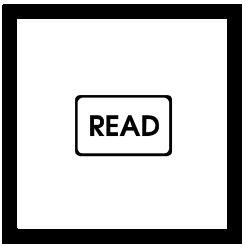
8. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



9. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10-mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



10. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L sodium chromate (Na_2CrO_4).

Note: *See table below to convert sodium chromate to other units.*

Table 1. Conversion Factors

To convert reading from	To	Multiply by
mg/L Na_2CrO_4	CrO_4^{2-}	0.72
mg/L Na_2CrO_4	Cr^{6+}	0.321

SAMPLING AND STORAGE
Collect samples in clean glass or plastic bottles.

ACCURACY CHECK
Standard Additions Method
a) Snap the neck off a Sodium Chromate Voluette Ampule Standard Solution, 25,000 mg/L Na_2CrO_4.

b) Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to three 25-mL samples. Mix well.

c) Analyze the sample according to the above procedure. The sodium chromate concentration should increase by 100 mg/L for each 0.1 mL addition of standard.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method
Use a 1000 mg/L Sodium Chromate Standard Solution listed under Optional Reagents to check accuracy.

INTERFERENCES
Large amounts of turbidity will result in high readings.

STATISTICAL EVALUATION
A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 934 mg/L Na_2CrO_4 concentration samples, the standard deviation was ±5.4 mg/L Na_2CrO_4.

Testing zero concentration samples, the limit of detection was 8.1 mg/L Na_2CrO_4. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52,* 2242-2249).

SUMMARY OF METHOD
The test directly measures the intensity of the alkaline yellow color of the sodium chromate solution. In acid media, the solution is orange and must be treated. A neutralizing agent is added to raise the pH, giving the yellow color necessary for the determination.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No
Neutralizing Reagent			
Powder Pillows	1 pillow	100/pkg	2127-99
Water, demineralized	25 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening			
powder pillows	1	each	968-00
DR/700 Filter Module			
Number 45.01	1	each	46245-00

OPTIONAL REAGENTS

Sodium Chromate Standard Solution,			
1000 mg/L Na_2CrO_4		500 mL	2503-49
Sodium Chromate Standard Solution,			
Voluette Ampule, 25,000 mg/L Na_2CrO_4		16/pkg	14255-10

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells		12/pkg	24018-12
Funnel, poly, 65 mm		each	1083-67
Filter Paper, folded, 12.5 cm		100/pkg	1894-57
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 Pipet		50/pkg	21856-96
Sample Cell, 10-mL with screw cap		6/pkg	24276-06
Sample Cell, 10-mL with screw cap		6/pkg	24019-06

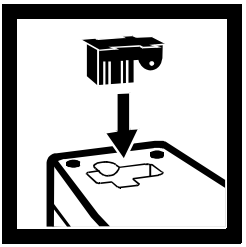
For Technical Assistance, Prices and Ordering
In the U.S.A.-Call 800-227-4224 toll-free for more information.
Outside the U.S.A.-Contact the Hach office or the distributor serving you.

SULFATE (0 to 100 mg/L SO_4^{2-})

For water, wastewater and seawater

SulfaVer 4 Method*‡ (Powder Pillows or AccuVac Ampuls), USEPA accepted for reporting**

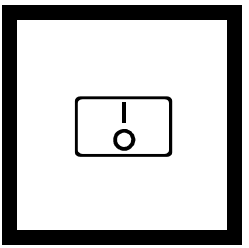
USING POWDER PILLOWS



1. Install module
45.01
in a DR/700.

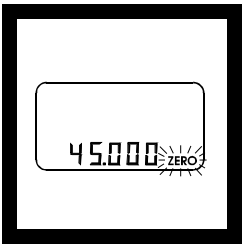
Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

Note: *The DR/700 must be calibrated before sample analysis. See Calibration following these steps.*



2. Press: **I/O**

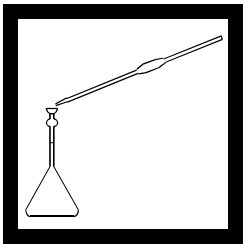
The display will show
450 nm
and module number
45.01



3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. Press the **PROGRAM** key once or twice until the lower display shows program number
45.000

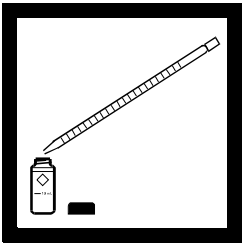
The upper display will show the concentration of a previous calibration standard. If desired press the **UP ARROW** key to display the other standard.

‡ User calibration is required, range is approximate.
*Adapted from *Standard Methods for the Examination of Water and Wastewater*
** Procedure is equivalent to USEPA method 375.4 for wastewater.

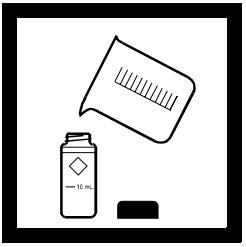


4. Prepare the displayed standard. For each mg/L displayed, pipet 1.00 mL of 1000 mg/L of Sulfate Standard Solution into a 1000-mL volumetric flask. Add demineralized water to the mark. Cap and invert 10 times to mix.

Note: *For example: to make a 50 mg/L standard, pipet 50.0 mL into the flask, For a zero concentration standard, use only demineralized water.*



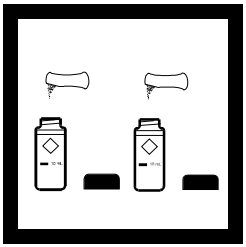
5. Fill a 10-mL cell to the 10-mL line with the displayed sample. Cap.



6. Fill a 10-mL cell to the 10-mL line with sample.

Note: *Filter highly colored or turbid samples. Use labware listed under Optional Apparatus to filter.*

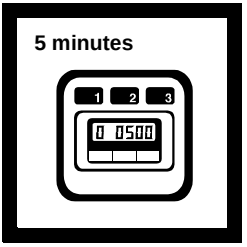
Note: *A 25-mL sample can be tested by using 25-mL sample cells and optional reagents.*



7. Add the contents of one SulfaVer 4 Sulfate Reagent Powder Pillow to each sample cell. Cap and invert several times to mix.

Note: *A white turbidity will develop if sulfate is present.*

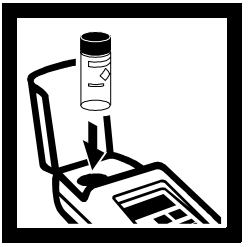
Note: *Accuracy is not affected by undissolved powder.*



8. Wait 5 minutes.

Note: *Allow cells to stand undisturbed.*

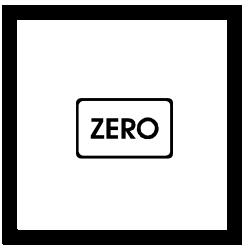
Note: *Perform Steps 9-12 within 5 minutes after the 5-minute waiting period.*



9. Place the prepared standard into the cell holder.

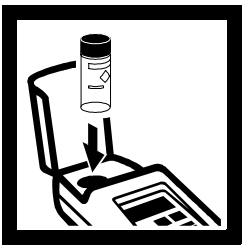
Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

Note: *If the display is blank, repeat Steps 2 and 3.*



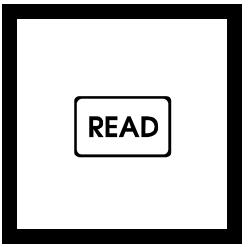
10. Press: **ZERO**

The display will count down to 0. Then the display will show the standards concentration and the zero prompt will turn off.



11. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

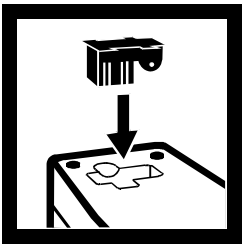


12. Within 5 minutes after the 5-minute period, press: **READ**

The display will count down to 0. Then the display will show the results in mg/L sulfate (SO_4^{2-}).

Note: *Clean the sample cells with soap and a brush.*

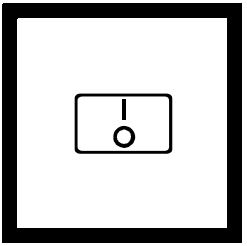
USING ACCUVAC AMPULS



1. Install module
45.01
in a DR/700.

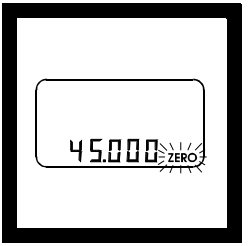
Note: *If sample cannot be analyzed immediately, see Sampling and Storage, below.*

Note: *The DR/700 must be calibrated before sample analysis. See Calibration following these steps.*



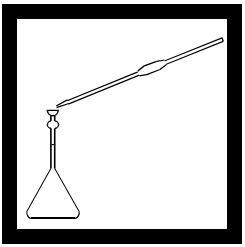
2. Press: **I/O**

The display will show
450 nm
and module number
45.01



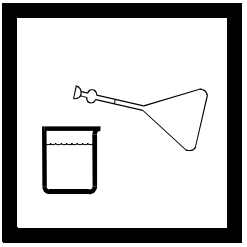
3. After 2 seconds, the display will show a program number, concentration units, decimal position and the zero prompt. Press the **PROGRAM** key once or twice until the lower display shows program number
45.000

The upper display will show the concentration of a previous calibration standard. If desired, press the **UP ARROW** key to display the other standard.

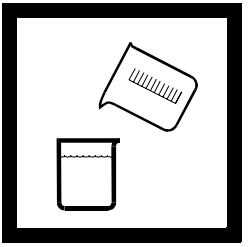


4. Prepare the displayed standard. For each mg/L displayed, pipet 1.00 mL of 1000 mg/L of Sulfate Standard Solution into a 1000-mL volumetric flask. Add demineralized water to the mark. Cap and invert 10 times to mix.

Note: *For example: to make a 50 mg/L standard, pipet 50.0 mL into the flask, For a zero concentration standard, use only demineralized water.*

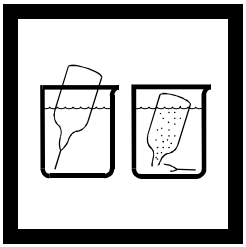


5. Pour at least 40 mL of the displayed standard into a 50-mL beaker.



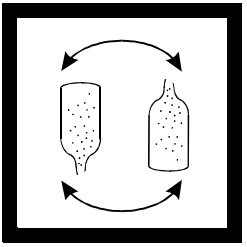
6. Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Filter highly colored or turbid samples. Use labware listed under Optional Apparatus.*



7. Fill a SulfaVer 4 Sulfate AccuVac Ampul with displayed standard. Fill a second AccuVac Ampul with sample.

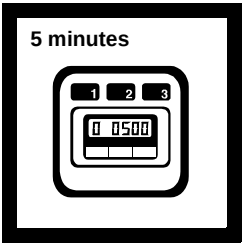
Note: *Keep tip immersed while the ampul fills completely.*



8. Quickly invert the sample several time to mix. Wipe off any liquid or fingerprints.

Note: *A white turbidity will form if sulfate is present.*

Note: *Accuracy is not affected by undissolved powder.*

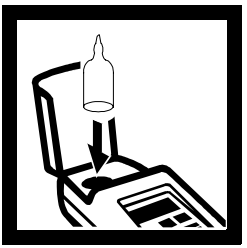


9. Wait 5 minutes.

Note: *Allow the ampul to stand undisturbed.*

Note: *Complete Steps 10-14 within 5 minutes after the 5-minute waiting period.*

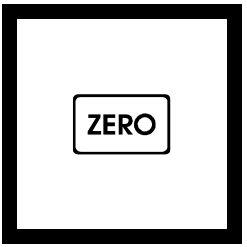
10. Insert the AccuVac Vial Adapter into the cell holder.



11. Place the AccuVac containing the prepared standard into the cell holder.

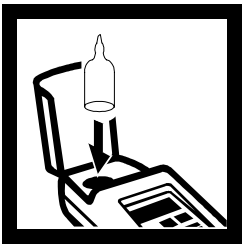
Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

Note: *If the display is blank, repeat Steps 2 and 3.*



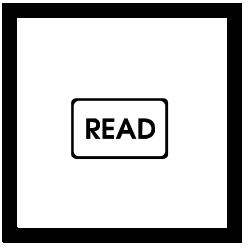
12. Press: **ZERO**

The display will count down to 0. Then the display will show the standard's concentration and the zero prompt will turn off.



13. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



14. Within five minutes after the five-minute waiting period, press: **READ**

The display will count down to 0. Then the display will show the results in mg/L sulfate (SO_4^{2-}).

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Samples may be stored up to 7 days by cooling to 4 °C (39 °F) or lower. Warm to room temperature before analysis.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Sulfate Voluette Ampule Standard Solution, 2500 mg/L.

b) Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to three 25-mL samples. Mix thoroughly. For AccuVac ampuls, use 50-mL beakers.

c) Analyze each sample as described above. The sulfate concentration should increase 10 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

CALIBRATION

A new calibration is necessary when different manufacturing lots of SulfaVer 4 powder Pillows or AccuVacs are used. A new calibration is

also needed when the water sample to be tested has an expected sulfate concentration which is not between that of the standards last used to calibrate the DR/700 module. Perform the calibration as follows.

Make 2 sulfate standards using the technique described in Step 4 of the test procedure, Make them so one has a slightly lower and the other a slightly higher concentration than that anticipated to be in the water sample. Process each on the same way as the "displayed standard" described in the test procedure.

Next perform paragraph 3.2.4.1, Calibration Using Two Prepared Standards, in the DR/700 instrument manual. Either standard can be used as Standard 1 (S1) or Standard 2 (S2).

INTERFERENCES

Silica and Calcium may interfere at levels above 500 and 20,000 mg/L as $CaCO_3$, respectively.

Chloride and magnesium do not interfere at levels up to at least 40,000 mg/L as Cl and 10,000 mg/L as $CaCO_3$, respectively.

SUMMARY OF METHOD

Sulfate ions on the sample react with barium in the SulfaVer 4 Sulfate Reagent and form insoluble barium sulfate turbidity. The amount of turbidity formed is proportional to the sulfate concentration.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No
SulfaVer 4 Reagent Powder Pillows, 10 mL	2 pillows	100/pkg	21067-69
Sulfate Standard Solution, 1000 mg/L	varies	500 mL	21757-49
Water, demineralized	1 L	4 L	272-56

REQUIRED REAGENTS (Using AccuVac Ampuls)

SulfaVer 4 AccuVac Ampuls	1 ampul	25/pkg	25090-25
Sulfate Standard Solution, 1000 mg/L	varies	500 mL	21757-49
Water, demineralized	1 L	4 L	272-56

REQUIRED APPARATUS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No
Brush	1	each	690-00
Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 45.01	1	each	46245-00
Flask, volumetric, 1000 mL	1	each	14574-53
Pipet Filler, safety bulb	1	each	14651-00
Choose one or more based on Calibration Volume (all Class A)			
Pipet, volumetric, 1.00 mL	1	each	14515-35
Pipet, volumetric, 2.00 mL	1	each	14515-36
Pipet, volumetric, 3.00 mL	1	each	14515-03
Pipet, volumetric, 4.00 mL	1	each	14515-04
Pipet, volumetric, 5.00 mL	1	each	14515-37
Pipet, volumetric, 10.00 mL	1	each	14515-38
Pipet, volumetric, 15.00 mL	1	each	14515-39
Pipet, volumetric, 20.00 mL	1	each	14515-20
Pipet, volumetric, 25.00 mL	1	each	14515-40
Pipet, volumetric, 50.00 mL	1	each	14515-41
Pipet, volumetric, 100.00 mL	1	each	14515-42

REQUIRED APPARATUS (For AccuVac Ampuls)

Description	Quantity Per Test	Unit	Cat. No
Beaker, 50 mL	2	each	500-41
DR/700 Filter Module Number 45.01	1	each	46245-00
Flask, volumetric, 1000 mL	1	each	14574-53
Pipet Filler, safety bulb	1	each	14651-00
Choose one or more based on Calibration Volume (all Class A)			
Pipet, volumetric, 1.00 mL	1	each	14515-35
Pipet, volumetric, 2.00 mL	1	each	14515-36
Pipet, volumetric, 3.00 mL	1	each	14515-03
Pipet, volumetric, 4.00 mL	1	each	14515-04
Pipet, volumetric, 5.00 mL	1	each	14515-37
Pipet, volumetric, 10.00 mL	1	each	14515-38
Pipet, volumetric, 15.00 mL	1	each	14515-39
Pipet, volumetric, 20.00 mL	1	each	14515-20
Pipet, volumetric, 25.00 mL	1	each	14515-40

REQUIRED APPARATUS (For AccuVac Ampuls) (continued)

Description	Quantity Per Test	Unit	Cat. No
Pipet, volumetric, 50.00 mL	1	each	14515-41
Pipet, volumetric, 100.00 mL	1	each	14515-42

OPTIONAL REAGENTS

Sulfate Standard Solution, 50 mg/L	500 mL	2578-49
Sulfate Standard Solution, Voluette Ampule, 2500 mg/L, 10 mL	16/pkg	14252-10
SulfaVer 4 Reagent Powder Pillows, 25 mL	50/pkg	12065-66

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac Vial, DR/700	each	43784-00
Ampule Breaker Kit	each	21968-00
Beaker, 50 mL	each	500-41
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Funnel, poly, 65 mm	each	1083-67
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Sample cell, 10-mL with screw cap	6/pkg	24276-06
Sample cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

Module 48.01
480 nm

Hach warrants equipment it manufactures against defective materials or workmanship for at least one year from the shipping date. Warranties do not apply to limited-life electrical components such as batteries. Full warranty information is located on the reverse side of Hach invoices.

IMPORTANT NOTICE

A lamp intensity adjustment must be performed:
- Before first or initial use
- When new filter modules are installed
- On each filter module when the lamp is replaced

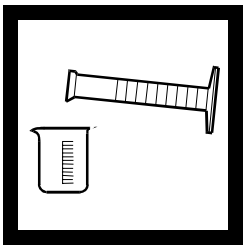
Refer to Lamp Intensity Adjustment in the instrument manual.

Table of Contents for 480-nm parameters

LEAD (0 to 150 µg/L)

For drinking water

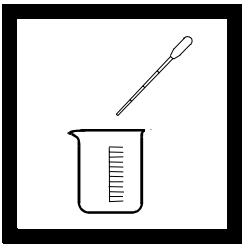
LeadTrak® Fast Column Extraction Method



1. Fill a 100-mL plastic graduated cylinder with 100 mL of the water to be tested. Pour the measured sample into a 250-mL plastic beaker.

Note: *The sampling requirements for "first-draw" analysis are detailed in the Sampling and Storage section.*

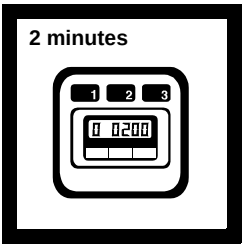
Note: *For proof of accuracy, use a 50-µg/L Lead Standard Solution (preparation given in Accuracy Check) in place of the sample.*



2. Using a plastic 1-mL dropper, add 1.0 mL of pPb-1 Acid Preservative Solution to the sample and swirl to mix.

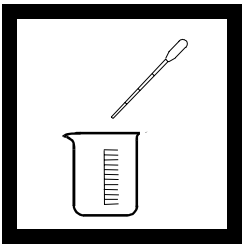
Note: *If the sample has been preserved previously with pPb-1 Acid Preservative at a rate of 1.0 mL per 100 mL sample, omit steps 2 and 3.*

Note: *Samples preserved with Nitric Acid require Steps 2 and 3.*



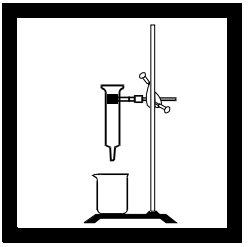
3. Wait 2 minutes.

*U.S. Patent No. 5,019,516



4. Use a second 1-mL plastic dropper to add 2.0 mL of pPb-2 Fixer Solution to the sample. Swirl to mix and proceed quickly to the next step.

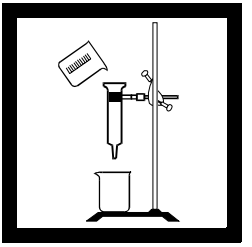
Note: *Field samples that have been acid-preserved with nitric acid or samples that have been digested may exceed the buffer capacity of the Fixer Solution. After step 4 check the pH of these samples and adjust with 5 N sodium hydroxide to a pH of 6.7-7.1 before proceeding with Step 5.*



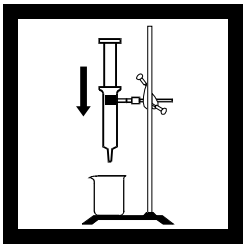
5. Mount a new Fast Column Extractor in a ring stand with a clamp. Place a 150-mL plastic beaker under the Extractor.

Note: *Fast Column Extractor is included in the LeadTrak Reagent Set.*

Note: *A new extractor is required for each test.*

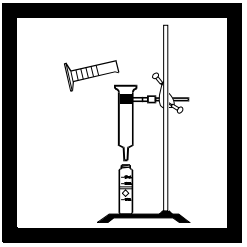


6. Pour the prepared sample slowly into the Column Extractor. Wait for the sample to flow through.

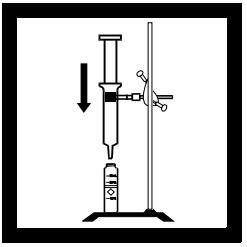


7. After the flow has stopped, fully compress the adsorbent pad in the Extractor with the plunger. Discard the contents of the beaker. Withdraw the plunger slowly from the Extractor.

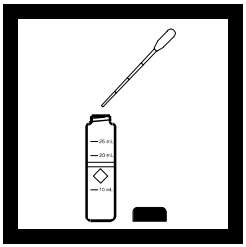
Note: *The adsorbent pad should remain at the bottom of the Extractor when the plunger is removed. Recompress with the plunger if the pad has retracted with the plunger.*



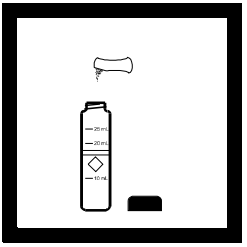
8. Place a 25-mL sample cell under the Extractor. Using a 25-mL plastic graduated cylinder, add 25 mL of pPb-3 Eluant Solution to the Extractor.



9. After the Eluant Solution has started to drip from the Extractor, insert the plunger and slowly force the remaining Eluant Solution through the Extractor. Fully compress the adsorbent pad. The volume in the sample cell should be 25 mL.

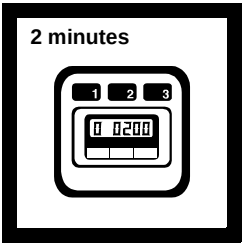


10. Using a 1-mL plastic dropper, add 1.0 mL of pPb-4 Neutralizer Solution to the sample cell. Cap and invert several times to mix. Proceed quickly to the next step.

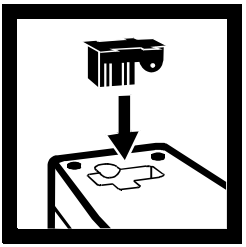


11. Add the contents of one pPb-5 Indicator Powder Pillow to the sample. Cap and invert several times to mix.

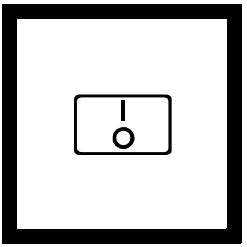
Note: *The solution will turn brown.*



12. Wait 2 minutes.

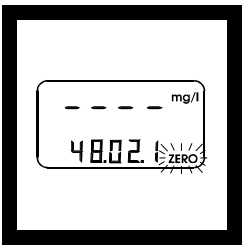


13. Install module
48.01
in a DR/700.

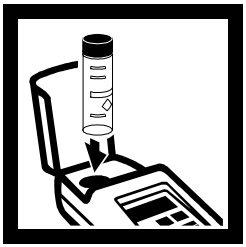


14. Press: **I/O**

The display will show
480 nm
and module number
48.01

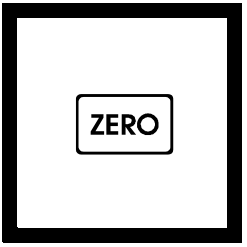


15. After 2 seconds, the display will show a program number, concentration units, decimal position and the zero prompt. If necessary press the **UP ARROW** key until the lower display shows program number
48.02.1



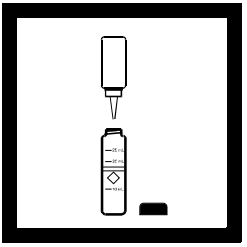
16. Place the sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



17. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 µg/L and the zero prompt will turn off.

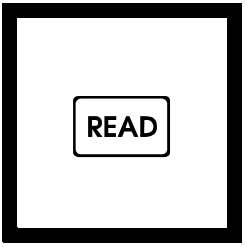


18. Remove the sample cell from the cell holder and add 6 drops of pPb-6 Decolorizer Solution to the cell. Cap and invert several times to mix.

Note: *There will be little visual difference between the blank and the sample.*

19. Replace the sample cell in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



20. Press: **READ**

The display will count down to 0. Then the display will show the results in µg/L lead (Pb).

Note: *Running a reagent blank with lead-free, reagent grade water is required for USEPA reporting purposes. Each new lot of reagents should have a reagent blank determined. The reagent blank is then subtracted from each test result.*

APPARATUS/SAMPLE PREPARATION

Because lead is very common to our environment, care must be taken to prevent sample contamination. Follow these steps for greatest test accuracy:

a) Lead-free water is necessary to minimize sample contamination when rinsing apparatus or diluting sample. The water may be either distilled or demineralized. If the water is obtained from a grocery store, verify the lead concentration is zero from the label. If the lead concentration is uncertain, determine the lead concentration with the LeadTrak test.

b) Plastic or glass sample containers and lids may be checked for contamination by rinsing with 1 mL of pPb-1 Acid Preservative. Add 100 mL of lead-free water. After 24 hours, analyze this solution using the LeadTrak test to confirm the absence of lead.

c) Rinse labware (plastic and glass) used in this test with a small amount of dilute lead-free nitric acid or pPb-1 Acid Preservative followed by rinsing with lead-free water.

d) pPb-5 Indicator may be rinsed from the glass sample cells with a few drops of pPb-1 Acid Preservative or a small amount of dilute lead-free nitric acid.

SAMPLING

Samples may be collected either from household pipes (point-of-use) or from water sources. Each source has its own sampling procedure under the proposed EPA methods. Samples may be stored up to six months.

Sampling for lead contamination in household pipes for point-of-use drinking water

a) The sample should be collected after sitting in pipes with no flow for 8 to 18 hours.

b) Add 10 mL of pPb-1 Acid Preservative to a one-liter bottle.

c) Turn on tap and collect exactly the first liter of water in the bottle containing acid preservative.

d) Cap and invert several times to mix.

c) After two minutes the sample is ready for analysis. Steps 2 and 3 are skipped in the analysis procedure. Use 100 mL of this preserved sample directly in Step 7.

Sampling for lead contamination from drinking water sources such as well water or water from main supply lines

a) Add 10 mL of pPb-1 Acid Preservative to a one-liter bottle.

b) Turn on the tap for 3-5 minutes or until the water temperature has been stable for 3 minutes.

c) Collect exactly one liter of water into the bottle containing the acid preservative.

d) Cap and invert several times to mix.

e) After two minutes the sample is ready for analysis. Steps 2 and 3 are skipped in the analysis procedure.

Notes
a) *At least one liter should be collected to obtain a representative sample. If less than one liter is collected, use 1 mL of pPb-1 Acid Preservative per 100 mL of sample.*

b) *If nitric acid is to be substituted for pPb-1 as a preservative or the sample is digested, the buffering capacity of the pPb-2 Fixer Solution may be exceeded. Adjust the sample pH to 6.7 to 7.1 pH with 5 N sodium hydroxide after Step 4.*

c) *Each sample type typically requires different sampling procedures. Consult with the appropriate regulatory agency in your area for more information about your specific sampling requirements.*

ACCURACY CHECK

Standard Additions Method

The standard additions method for checking the validity of the test results can be performed as follows:

a) Use a TenSette Pipet to add 0.1 mL of a 10 mg/L Lead Standard Solution (included in the reagent set) to a second 100-mL portion of the sample.

b) Swirl the sample to mix. Then test the sample as described in the procedure. Each 0.1-mL of standard added should increase the lead concentration determined in Step 20 by 10 µg/L.

Standard Solution Method
A 50-µg/L lead standard solution can be prepared by first pipetting 1.00 mL of Lead Standard Solution, 1000 mg/L as Pb, into a 100-mL plastic volumetric flask and diluting to the mark with lead-free water to make a 10 mg/L lead working solution (included in the reagent set). Pipet 5.00 mL of this working solution into a 1-liter plastic volumetric flask. Dilute to the mark with lead-free water. This 50-µg/L standard solution should be prepared immediately before use.

Alternatively, a 50-µg/L lead standard solution can be prepared by using a TenSette Pipet and pipetting 0.1 mL from a Lead Voluette Ampule Standard Solution, 50 mg/L as Pb, into a 100-mL plastic volumetric flask and diluting to volume with demineralized water. This solution should be prepared immediately before use.

INTERFERENCE

Interference studies were conducted by preparing a known lead solution of approximately 25 µg/L as well as the potential interfering ion. The ion was said to interfere when the resulting concentration changed by ±10%.

Ion	Interference Level
Aluminum, Al^{3+}	0.5 mg/L
Barium, Ba^{2+}	6 mg/L
Calcium, Ca^{2+}	500 mg/L
Chloride, Cl^{-}	1000 mg/L
Copper, Cu^{2+}	2 mg/L
Fluoride, F^{-}	10 mg/L
Iron, Fe^{2+}	2 mg/L
Magnesium, Mg^{2+}	500 mg/L
Manganese, Mn^{2+}	0.5 mg/L
Nitrogen, Ammonium, NH_4^{+}	500 mg/L
Nitrogen, Nitrate, NO_3^{-}	1000 mg/L
Sulfate, SO_4^{2-}	1000 mg/L
Zinc, Zn^{2+}	1 mg/L

Samples containing levels exceeding these concentration values may be diluted 1:1 and re-analyzed. Multiply the value obtained by 2 to determine the lead present in the original sample.

Every effort has been made to prevent contamination in packaging the reagents. Use of black rubber stoppers, black dropper bulbs and droppers with inked graduations may contaminate the sample and should be avoided. Use the plastic droppers provided in the reagent set.

Glassware and plastic ware should be rinsed with a dilute nitric acid solution such as 0.1 N Nitric Acid Standard Solution or a few drops of pPb-1 Acid Preservative Reagent to prevent sample contamination, especially if the previous sample had a high lead level. The sample cell walls will become colored from the pPb-5 Indicator and should be rinsed. The Extractor plunger is intended to be used for more than one test and should be rinsed as well.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched cells and two representative lots of testing reagents. Testing 100 µg/L Pb concentration samples the standard deviation was ±3.0 µg/L Pb.

Testing zero concentration samples, the limit of detection was 3.0 µg/L Pb. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** 52, 2242-2249).

SUMMARY OF METHOD

Acid soluble lead, as Pb^{2+}, in a potable water sample is first concentrated on a Fast Column Extractor. The lead is then eluted from the Extractor and determined colorimetrically with an indicator.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
LeadTrak, reagent set	1	20 tests/pkg	23750-00

REQUIRED APPARATUS

Beaker, polypropylene, 250 mL	1	each	1080-46
Beaker, polypropylene, 150 mL	1	each	1080-44
Clamp, two-prong extension	1	each	21145-00
Clamp, holder	1	each	326-00
Clippers, small	1	each	936-00
Cylinder, graduated, polypropylene, 100 mL	1	each	1081-42
Cylinder, graduated, polypropylene, 25 mL	1	each	1081-40
DR/700 Filter Module Number 48.01	1	each	46248-00
Support, ring stand	1	each	563-00

OPTIONAL REAGENTS

Lead Standard Solution, 1000 mg/L as Pb	100 mL	12796-42
Lead Standard Solution, Voluette Ampule, 50 mg/L as Pb^{2+}, 10 mL	16/pkg	14262-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Standard Solution, 0.1 N	105 mL	23328-42
pPb-1 Acid Preservative Reagent	237 mL	23685-31
pPb-2 Fixer Solution	43 mL	23685-31
pPb-3 Eluant	500 mL	23687-49
pPb-4 Neutralizer	22 mL	23688-55
pPb-5 Indicator Reagent Powder Pillows	20/pkg	23689-64
pPb-6 Decolorizer	10 mLSCDB	23748-20
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Bottle, sampling, 125 mL	each	23240-43
Bottle, sampling, 125 mL	48/pkg	23240-73

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
Bottle, sampling, 1000 mL	each	23242-53
Bottle, sampling, 1000 mL	24/pkg	23242-83
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Dropper, plastic, Squeezers	10/pkg	21247-10
Flask, volumetric, plastic, 100 mL	each	20995-42
Flask, volumetric, plastic, 1000 mL	each	20995-53
pH Meter, EC10, portable	each	50050-00
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet Filler,	each	12189-00
Pipettor, 100 µL	each	22753-00
Pipettor Tips, for Pipettor	10/pkg	22754-10
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

Module 50.01
500 nm

Hach warrants equipment it manufactures against defective materials or workmanship for at least one year from the shipping date. Warranties do not apply to limited-life electrical components such as batteries. Full warranty information is located on the reverse side of Hach invoices.

IMPORTANT NOTICE

A lamp intensity adjustment must be performed:
- Before first or initial use
- When new filter modules are installed
- On each filter module when the lamp is replaced

Refer to Lamp Intensity Adjustment in the instrument manual.

Table of Contents for 500-nm parameters

CYANURIC ACID (0 to 50 mg/L)

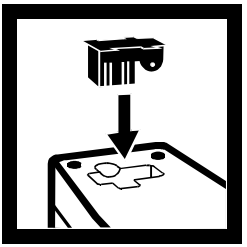
For water

Turbidimetric Method*

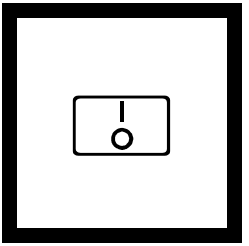
1. Fill a 25-mL cell to the 25-mL line with sample.

Note: *Filtering is required for highly colored or turbid samples. Use the labware listed under Optional Apparatus. Large amounts of color or turbidity will interfere and cause high readings.*

Note: *The DR/700 must be calibrated before sample analysis. See Calibration following these steps.*



2. Install module **50.01** in a DR/700.



3. Press: **I/O**

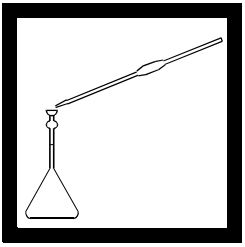
The display will show **500 nm** and module number **50.01**

*User calibration required. Range is approximate.

4. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. Press the **PROGRAM** key until the lower display shows program number

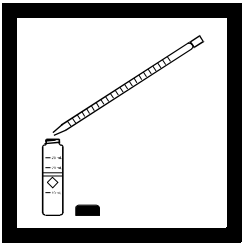
50.000

The upper display will show the S1 concentration. If desired, press the **UP ARROW** key to display the other standard.

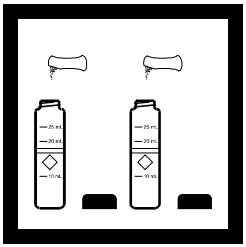


5. Make the displayed standard. For each mg/L displayed, pipet 1.00 mL from a 1000-mg/L cyanuric acid stock solution into a 1000-mL volumetric flask. Add demineralized water to the mark. Cap and invert to thoroughly mix.

Note: *To make a 25-mg/L standard, pipet 25.00 mL of a 1000-mg/L stock solution into the flask. For a zero concentration standard, use only demineralized water.*

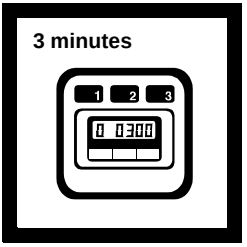


6. Fill a 25-mL cell to the 25-mL line with the displayed standard.



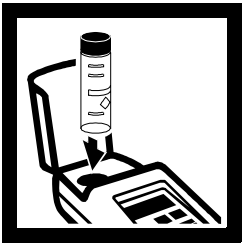
7. Add the contents of one Cyanuric Acid 2 Reagent Powder Pillow to each cell. Cap and invert several times to mix.

Note: *A white turbidity will form if cyanuric acid is present.*



8. Wait 3 minutes.

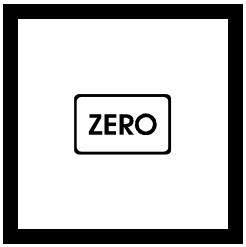
Note: *Complete Steps 9-12 within 5 minutes after the 3-minute waiting period.*



9. Place the standard treated in Step 7 in the cell holder.

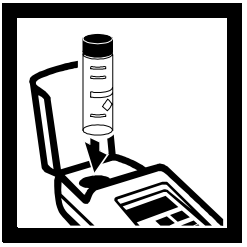
Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*

Note: *If the display is blank, repeat Steps 3 and 4.*



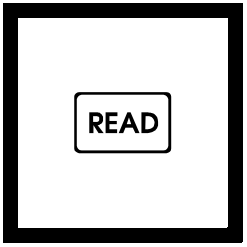
10. Press: **ZERO**

The display will count down to 0. Then the display will show the concentration of the standard and the zero prompt will turn off.



11. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



12. Within 7 minutes after the 3-minute reaction period, press: **READ**

The display will count down to 0. Then the display will show the results in mg/L cyanuric acid.

Note: *Clean sample cells with soap, water and a brush soon after each test to prevent a white film forming on the cells.*

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Samples must be analyzed within 24 hours.

CALIBRATION

A new calibration is necessary for each now manufacturing lot of Cyanuric Acid 2 Reagent Powder Pillows. A new calibration is also necessary when the water sample to be tested has a cyanuric acid concentration which is not between that of the standards used to calibrate the DR/700.

Perform the calibration as follows:

a) Dissolve 1.000 gram of cyanuric acid in l liter of demineralized water to make a 1000-mg/L solution. Fill the flask half-full with demineralized water, then agitate the flask until all the solid has dissolved. Add demineralized water to the line. Cap and invert 10 times to mix. This stock solution is stable for several weeks.

b) Make 2 cyanuric acid standards using the technique described in Step 5 of the procedure. Make one slightly less concentrated and the other slightly more concentrated than the anticipated concentration of the water sample.

c) Perform Steps 6-8 with both these standards.

d) With Filter Module 51.01 installed, perform paragraph 3.2.4.1, Calibration Using Two Prepared Standards, in the DR/700 instrument manual. Either standard can be used as Standard 1 or Standard 2. **Calibration must be completed within 7 minutes after the 3-minute reaction period.**

SUMMARY OF METHOD

The test for cyanuric acid uses the turbidimetric method. Cyanuric Acid 2 Reagent precipitates any cyanuric acid present and holds it in suspension. The amount of turbidity caused by the suspended particles is directly proportional to the amount of cyanuric acid present.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Cyanuric Acid 2 Reagent Powder Pillow	1 pillow	50/pkg	2460-66
Cyanuric Acid	1 g	25 g	7129-24
Water, demineralized	1 L	4 L	272-56

REQUIRED APPARATUS

Balance, analytical	1	each	22310-00
Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 50.01	1	each	46250-00
Flask, volumetric, 1000 mL	3	each	14574-53
Pipet, Filler, Safety Bulb	1	each	14651-00

Choose one or more based on calibration volume (Class A):

Pipet, volumetric, Class A, 1.00 mL	1	each	14515-35
Pipet, volumetric, Class A, 2.00 mL	1	each	14515-36

REQUIRED APPARATUS (continued)

Description	Quantity Per Test	Unit	Cat. No.
Pipet, volumetric, Class A, 3.00 mL	1	each	14515-03
Pipet, volumetric, Class A, 4.00 mL	1	each	14515-04
Pipet, volumetric, Class A, 5.00 mL	1	each	14515-37
Pipet, volumetric, Class A, 10.00 mL	1	each	14515-38
Pipet, volumetric, Class A, 15.00 mL	1	each	14515-39
Pipet, volumetric, Class A, 20.00 mL	1	each	14515-20
Pipet, volumetric, Class A, 25.00 mL	1	each	14515-40
Pipet, volumetric, Class A, 50.00 mL	1	each	14515-41

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells		12/pkg	24018-12
Filter Paper, folded 12.5 cm		100/pkg	1894-57
Funnel, poly, 65 mm		each	1083-67
Pipet, Mohr, 25 mL		each	20934-40
Pipet Filler, 3-valve		each	12189-00
Sample Cell, 10-mL with screw cap		6/pkg	24276-06
Sample Cell, 25-mL with screw cap		6/pkg	24019-06

For Technical Assistance, Prices and Ordering

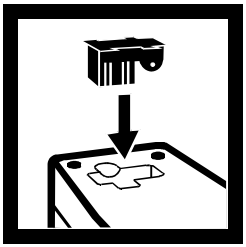
In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

IRON, FERROUS (0 to 5.00 mg/L)

For water, wastewater and seawater

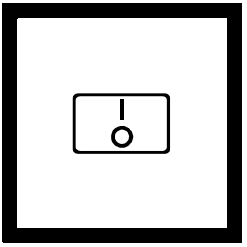
1,10 Phenanthroline Method*
(Powder Pillows or AccuVac Ampuls)

USING POWDER PILLOWS



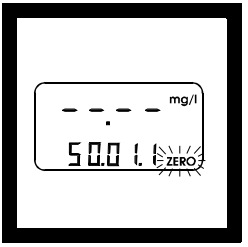
1. Install module
50.01
in a DR/700.

Note: *Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric iron, which is not measured.*



2. Press: **I/O**

The display will show
500 nm
and module number
50.01

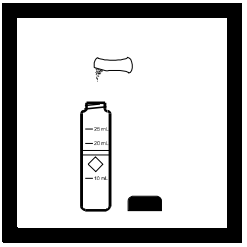


3. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
50.01.1

*Adapted from *Standard Methods for the Examination of Water and Wastewater*

4. Fill a 25-mL cell to the 25-mL line with sample.

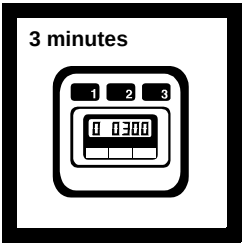
Note: *For proof of accuracy, use a 1.00 mg/L ferrous standard solution (preparation given in Accuracy Check) in place of the sample.*



5. Add the contents of one Ferrous Iron Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert to mix.

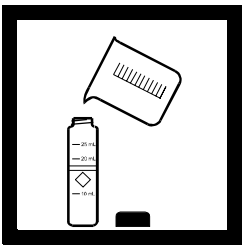
Note: *An orange color will develop if ferrous iron is present.*

Note: *Undissolved powder does not affect accuracy.*

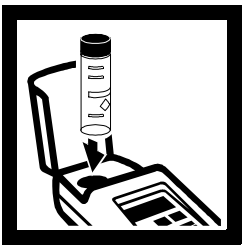


6. Wait 3 minutes.

Note: *Steps 7 and 8 can be completed during this reaction period.*

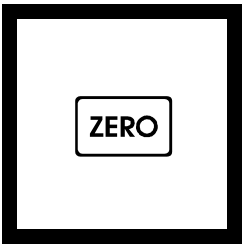


7. Fill a 25-mL cell to the 25-mL line with sample (the blank). Cap.



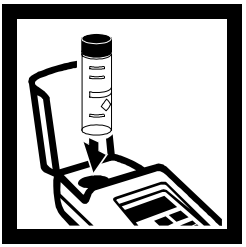
8. Place the blank in the cell holder.

Note: *In bright light, put 10 mL in a 10-mL cell, insert it and close the cell compartment cover.*



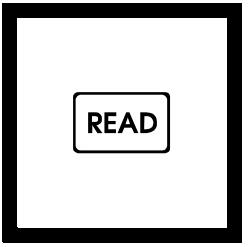
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



10. Place the prepared sample in the cell holder.

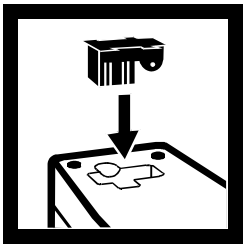
Note: *In bright light, put 10 mL in a 10-mL cell, insert it and close the cell compartment cover.*



11. Press: **READ**

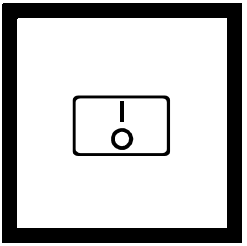
The display will count down to 0. Then the display will show the results in mg/L ferrous iron (Fe^{2+}).

USING ACCUVAC AMPULS



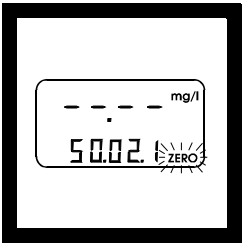
1. Install module **50.01** in a DR/700.

Note: *Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric iron, which is not measured.*



2. Press: **I/O**

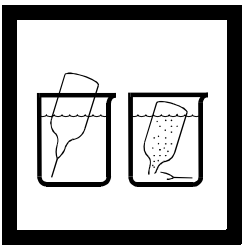
The display will show **500 nm** and module number **50.01**



3. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **50.02.1**

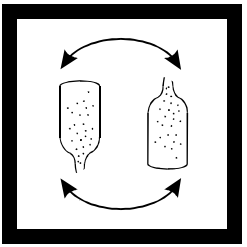
4. Fill a cell with 10 mL of sample (the blank). Cap. Collect at least 40 mL of sample in a 50-mL beaker.

Note: *In bright light use a 10-mL cell.*



5. Fill a Ferrous Iron AccuVac Ampul with sample.

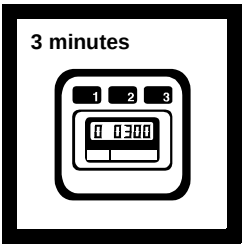
Note: *Keep the tip immersed while the ampul fills completely.*



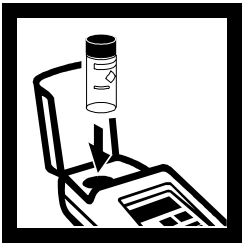
6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

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

Note: *Undissolved powder does not affect accuracy.*

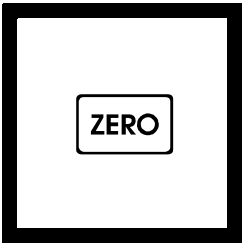


7. Wait 3 minutes.



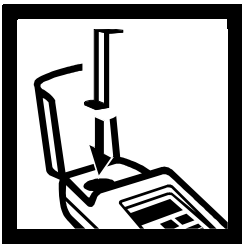
8. Place the blank in the cell holder.

Note: *In bright light close the cell compartment cover.*

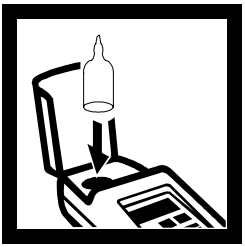


9. Press: **ZERO**

The display will count down to 0. then the display will show 0.00 mg/L and the zero prompt will turn off.

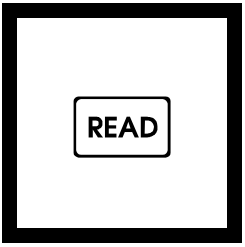


10. Insert the AccuVac Vial Adapter into the cell holder.



11. Place the prepared sample in the cell holder.

Note: *In bright light close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L ferrous iron (Fe^{2+}).

ACCURACY CHECK

Standard Solution Method

Prepare a ferrous iron stock solution (100 mg/L Fe^{2+}) by dissolving 0.7022 grams of Ferrous Ammonium Sulfate, hexahydrate, in demineralized water. Dilute to 1 liter. Prepare immediately before use. Dilute 1.0 mL of this solution to 100 mL with demineralized water to make a 1.0 mg/L standard solution. Prepare this immediately before use.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 1.50 mg/L Fe^{2+} concentration samples, the standard deviation was ±0.008 mg/L Fe^{2+}.

Testing zero concentration samples, the limit of detection was 0.007 mg/L Fe^{2+}. The limit of detection was calculated as three times the standard deviation whin testing zero concentration samples (Adapted from *Analytical Chemistry*, **1980**, 52, 2242-2249).

Using two representative lots of Ferrous Iron AccuVac Ampuls, the standard deviation was 0.014 mg/L Fe^{2+}.

SUMMARY OF METHOD

The 1,10 phenanthroline indicator in Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not interfere. The ferric iron (Fe^{3+}) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity per test	Unit	Cat. No.
Ferrous Iron Reagent			
Powder Pillows, 25-mL	1 pillow	100/pkg	1037-69

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, large, for opening pillows	1	each	968-00
DR/700 Filter Module Number 50.01	1	each	46250-00

REQUIRED REAGENTS (Using AccuVac Ampuls)

Description	Quantity per test	Unit	Cat. No.
Ferrous Iron Reagent			
AccuVac Ampuls	1 ampul	25/pkg	25140-25

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41
DR/700 Filter Module			
Number 50.01	1	each	46250-00

OPTIONAL REAGENTS

Ferrous Ammonium Sulfate,		
Hexahydrate, ACS	113 g	11256-14
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac Vial, DR/700	each	46025-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Flask, volumetric, 100 mL	each	547-42
Flask, volumetric, 1000 mL	each	547-53
Pipet, volumetric, 1 mL	each	515-35
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

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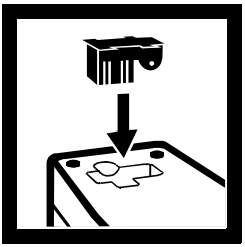
Outside the U.S.A. - Contact the Hach Office or distributor serving you.

IRON, TOTAL (0 to 5.00 mg/L)

For water, wastewater and seawater

FerroVer Method* (Powder Pillows or AccuVac Ampuls), USEPA approved for reporting (digestion - see Section 1)**

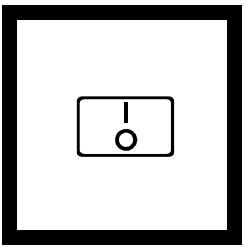
USING POWDER PILLOWS



1. Install module
50.01
in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust pH of stored samples before analysis.*

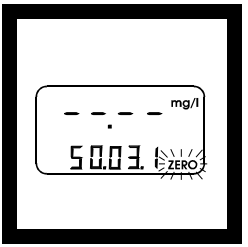
Note: *Determination of total iron requires a prior digestion; use the mild vigorous or Digesdahl digestion (Section I).*



2. Press: **I/O**

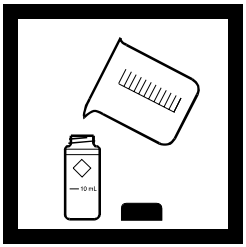
The display will show
500 nm
and module number
50.01

Note: *Determination of total iron needs a prior digestion; use the mild, vigorous or Digesdahl digestion (Section I.)*



3. After 2 seconds the display will show a program number, concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
50.03.1

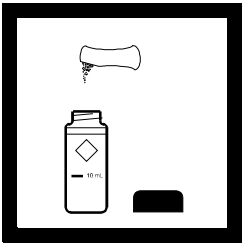
*Adapted from *Standard Methods for the Examination of Water and Wastewater.*
***Federal Register,* **1980,** *45(126),* 43459



4. Fill a 10-mL cell to the 10-mL line with sample.

Note: *A 25-mL sample can be tested by using 25-mL sample cells and optional reagents.*

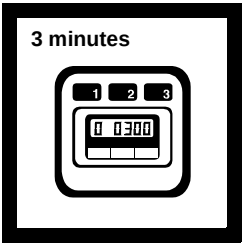
Note: *For proof of accuracy, use a 1.0 mg/L iron standard solution (preparation given in the Accuracy Check) in place of the sample.*



5. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert to mix.

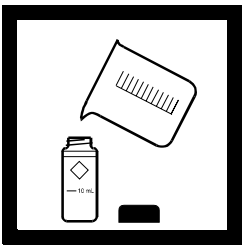
Note: *An orange color will form if iron is present.*

Note: *Accuracy is not affected by undissolved powder.*



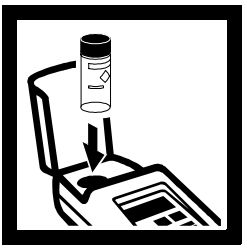
6. Wait 3 minutes.

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



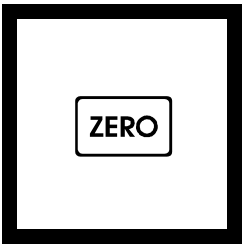
7. Fill a second 10-mL cell to the 10-mL line with sample (the blank). Cap.

Note: *For turbid samples, treat the blank with one 0.2-gram scoop of RoVer Rust Remover. Swirl to mix.*



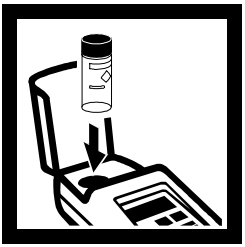
8. Within 30 minutes after the 3-minute period, place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



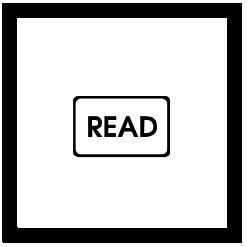
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



10. Place the prepared sample in the cell holder.

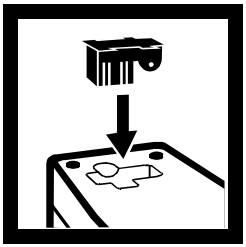
Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



11. Press: **READ**

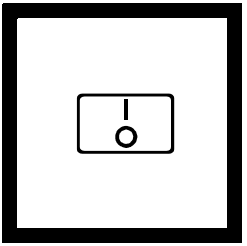
The display will count down to 0. Then the display will show the results in mg/L iron (Fe).

USING ACCUVAC AMPULS



1. Install module **50.01** in a DR/700.

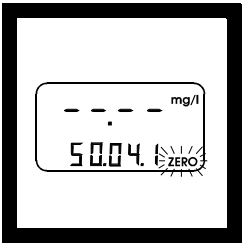
Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust pH of stored samples before analysis.*



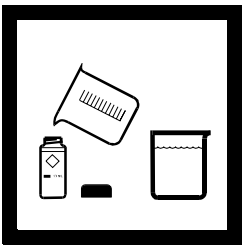
2. Press: **I/O**

The display will show **500 nm** and module number **50.01**

Note: *Determination of total iron needs a prior digestion; use the mild, vigorous or Digesdahl digestion (Section I).*

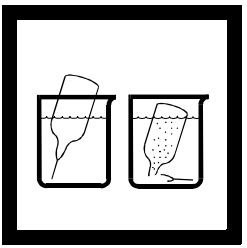


3. After 2 seconds the display will show a program number, concentration units, decimal position and the zero prompt. If necessary press the **UP ARROW** key until the lower display shows program number **50.04.1**



4. Fill a cell with 10 mL of sample (the blank). Cap. Collect at least 40 mL of sample in a 50-mL beaker.

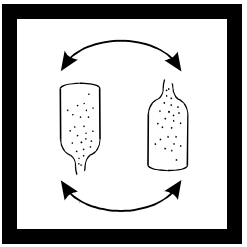
Note: *For turbid samples, treat the blank with one 0.2-gram scoop of RoVer Rust Remover. Swirl to mix.*



5. Fill a FerroVer AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*

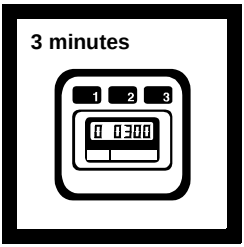
Note: *For proof of accuracy, use a 1.0 mg/L iron standard solution (preparation given in the Accuracy Check) in place of the sample.*



6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

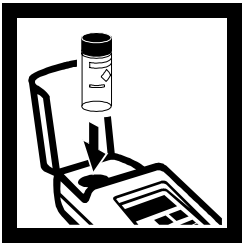
Note: *An orange color will form if iron is present.*

Note: *Accuracy is not affected by undissolved powder.*



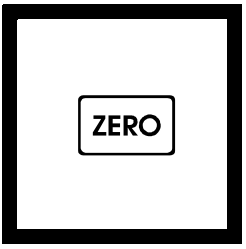
7. Wait 3 minutes.

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



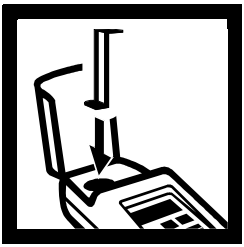
8. Within 30 minutes after the 3-minute period, place the blank in the cell holder.

Note: *In bright light close the cell compartment cover.*

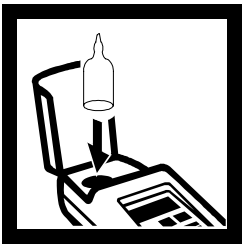


9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.

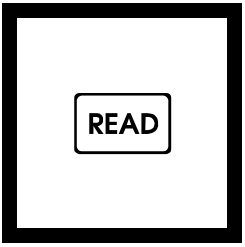


10. Insert the AccuVac Vial Adapter into the cell holder.



11. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L iron (Fe).

SAMPLING AND STORAGE

Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the sample immediately. To preserve samples, adjust the pH to 2 or less with concentrated nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Before analysis, adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution. Correct the test result for volume additions; see Sampling and Storage, Volume Additions (Section I) for more information.

If only dissolved iron is to be determined, filter the sample before acid addition using the labware listed under Optional Apparatus.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off an Iron Voluette Ampule Standard Solution, 50 mg/L.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL water samples and mix thoroughly. For AccuVac Ampuls, collect 25-mL samples in 50 mL beakers.

c) Analyze each sample as described above. The iron concentration should increase 0.2 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 1.0 mg/L iron standard by diluting 1.00 mL of Iron Standard Solution, 100 mg/L Fe, to 100 mL with demineralized water. Or, use the TenSette Pipet to dilute 1.0 mL of an Iron Voluette Ampule Standard Solution (50 mg/L) to 50 mL in a volumetric flask. Prepare this solution daily.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 1.5 mg/L Fe^{3+} concentration samples, the standard deviation was ±0.008 mg/L Fe^{3+}.

Testing zero concentration samples, the limit of detection was 0.007 mg/L Fe^{3+}. The limit of detection was calculated as three times the

standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry*, **1980,** 52, 2242-2249).

Using two representative lots of FerroVer Iron AccuVac Ampuls, the standard deviation was ±0.012 mg/L Fe^{3+} and the limit of detection was 0.014 mg/L Fe^{3+}.

INTERFERENCES

The following will not interfere below the levels shown:

Chloride	185,000 mg/L
Calcium	10,000 mg/L as $CaCO_3$
Magnesium	1000,000 mg/ as $CaCO_3$
Molybdate Molybdenum	50 mg/L as Mo

A large excess of iron will inhibit color development. A diluted sample should be tested if there is any doubt about the validity of a result.

FerroVer Iron Reagent Powder Pillows and AccuVac Ampuls contain a masking agent which eliminates potential interferences from copper.

Samples containing some forms of iron oxide require the mild, vigorous or Digesdahl digestion (Section I). After digestion, adjust the pH to between 2.5 and 5 with ammonium hydroxide.

Samples containing large amounts of sulfide should be treated as follows in a fume hood, or well ventilated area: Add 5 mL of hydrochloric acid to 100 mL of sample and boil for 20 minutes. Adjust the pH to between 2.5 and 5 with 5 N sodium hydroxide and readjust the volume to 100 mL with demineralized water. Analyze as described above.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

REAGENT STORAGE

FerroVer Reagent Powder Pillows are stable indefinitely if stored properly. A cool, dry atmosphere is recommended. The reagent can be checked by adding the contents of a pillow to about 25 mL of water containing visible rust (such as a few drops of Rust Suspension). If the orange color does not form, the reagent should be replaced.

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 the 1,10 phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat No.
FerroVer Reagent			
Powder Pillows, 10 mL	1 pillow	100/pkg	21057-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

FerroVer Iron Reagent			
AccuVac Ampuls	1 ampul	25/pkg	25070-35

REQUIRED APPARATUS (Using Powder Pillows)

DR/700 Filter Module			
Number 50.01	1	each	46250-00
Clippers, for opening			
powder pillows	1	each	968-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41
DR/700 FIlter Module			
Number 50.01	1	each	46250-00

OPTIONAL REAGENTS

Ammonium Hydroxide, ACS	500 mL	106-49
FerroVer Reagent		
Powder Pillows, 25-mL sample	50/pkg	854-66
Hydrochloric Acid		
Standard Solution, 6 N	500 mL	884-49
Iron Standard Solution, 100 mg/L	105 mL	14175-42
Iron Standard Solution,		
Voluette Ampule, 50 mg/L Fe, 10 mL	16/pkg	14254-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
RoVer Rust Remover	454 g	300-01
Rust Suspension	15 mL DB	1279-36

OPTIONAL REAGENTS

Description	Unit	Cat No.
Sodium Hydroxide Standard Solution, 5.0 N	105 mL MDB	2450-32
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac vial, DR/700	each	46025-00
Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, 25 mL	each	508-40
Dropper, calibrated, 0.5-mL mark	each	23185-37
Filter Discs, glass, 47 mm	each	2530-00
Filter Holder, membrane	100/pkg	2340-00
Filter Pump	each	2131-00
Flask, erlenmeyer, 125 mL	each	505-43
Flask, erlenmeyer, 50 mL	each	505-41
Flask, filtering, 500 mL	each	546-49
Flask, volumetric, Class A, 50 mL	each	14574-41
Flask, volumetric, Class A, 100 mL	each	14574-42
Hot Plate, 3 1/2" diameter, 120 Vac	each	12067-01
Hot Plate, 3 1/2" diameter, 240 Vac	each	12067-02
pH Indicator Paper, 1 to 11 pH	each	391-33
pH Meter, EC10, portable	each	50050-00
Pipet Filler, safety bulb	each	14651-00
Pipet, serological, 2 mL	each	532-36
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Volumetric, Class A, 0.50 mL	each	14515-34
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Sample Cell, 10-mL,	6/pkg	24276-06
Sample Cell, 25-mL, w/screw cap	6/pkg	24019-06
Spoon, measuring, 0.2 g	each	638-00
Thermometer, -20 to 105 °C	each	1877-01
Voluette Ampule Breaker Kit	each	21968-00

For Technical Assistance, Prices and Ordering Information

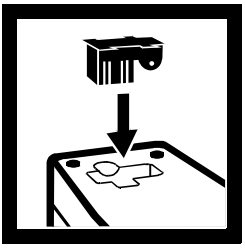
In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach Office or distributor serving you.

NITRATE, HR (0 to 30.0 mg/L NO_3^--N)

For water, wastewater and seawater*

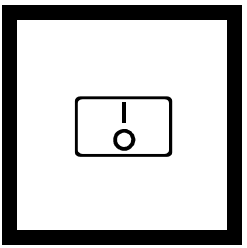
Cadmium Reduction Method (Powder Pillows or AccuVac Ampuls)

USING POWDER PILLOWS



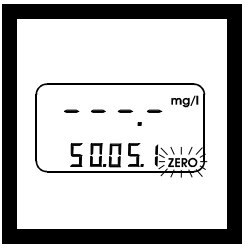
1. Install module number
50.01
in a DR/700.

Note: *If sample cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*



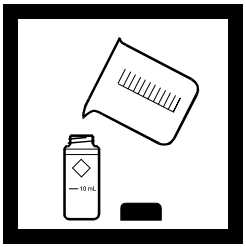
2. Press: **I/O**

The display will show
500 nm
and module number
50.01

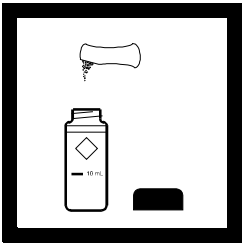


3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
50.05.1

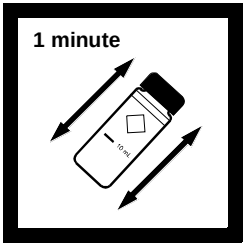
*For seawater, a manual calibration is required; see Interferences.



4. Fill a 10-mL cell to the 10-mL line with sample.



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

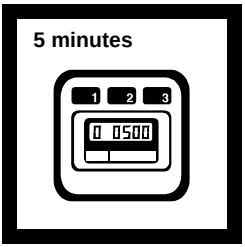


6. Shake the cell vigorously for one minute.

Note: *A deposit of unoxidized metal will remain after the NitraVer 5 Nitrate Reagent Powder dissolves. The deposit will not influence test results.*

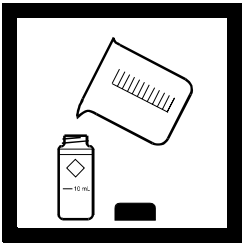
Note: *Shaking time and technique influence color development. For most accurate results, make successive tests on a 10 mg/L Nitrate Nitrogen Standard Solution (listed under Optional Reagents). Adjust the shaking time to obtain the correct result.*

Note: *An amber color will develop if nitrate nitrogen is present.*

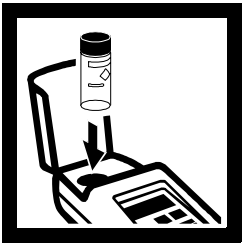


7. Set the cell down and wait 5 minutes.

Note: *Do not wait more then 15 minutes before completing Steps 8-12.*

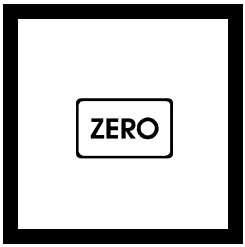


8. Fill a 10-mL cell to the 10-mL line with sample (the blank).



9. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

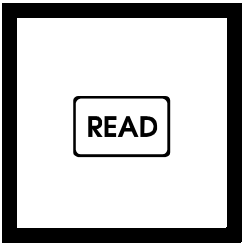


10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L and the zero prompt will turn off.

11. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



12. Press: **READ**

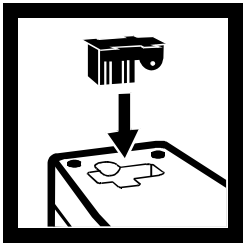
The display will count down to 0. Then the display will show the results in mg/L nitrate as nitrogen.

Note: *A reagent blank must be determined for each new lot of NitraVer 5. Repeat Steps 4 to 12, using demineralized water as the sample. Subtract this value from each result obtained with each lot of reagent.*

Note: *To convert the results to mg/L nitrate (NO_3^-), multiply by 4.4*

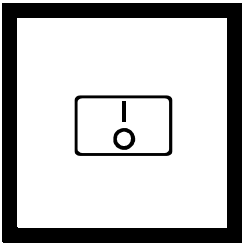
Note: *Rinse the sample cell immediately after use to remove all metal particles.*

USING ACCUVAC AMPULS



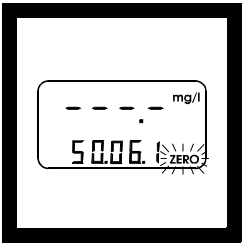
1. Install module number
50.01
in a DR/700.

Note: *If sample cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*

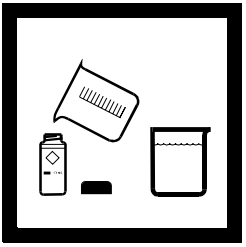


2. Press: **I/O**

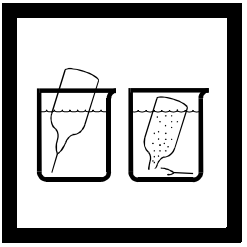
The display will show
500 nm
and module number
50.01



3. After 2 seconds the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
50.06.1



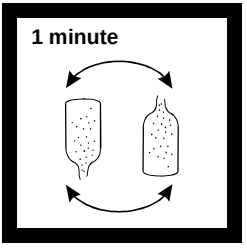
4. Fill a cell with 10 mL of sample (the blank). Cap. Collect at least 40 mL of sample in a 50-mL beaker.



5. Fill a NitraVer 5 Nitrate AccuVac Ampul with sample.

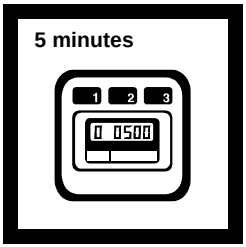
Note: *Keep the tip immersed while the ampul fills completely.*

Note: *For proof of accuracy, use a 10 mg/L Nitrate Nitrogen Standard Solution (listed under Optional Reagents) in place of the sample.*



6. Invert the ampul repeatedly for one minute to mix.

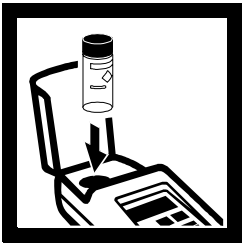
Note: *Inversion time and technique influence color development. For most accurate results, make successive tests on a 10 mg/L Nitrate Nitrogen Standard (listed under Optional Reagents). Adjust inversion time to obtain the correct result.*



7. Wait 5 minutes.

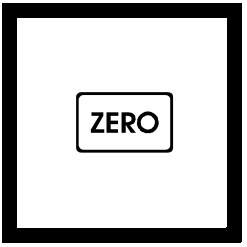
Note: *A deposit of unoxidized metal will remain after the NitraVer 5 Nitrate Reagent Powder dissolves. The deposit will not affect test results.*

Note: *An amber color will develop if nitrate nitrogen is present.*



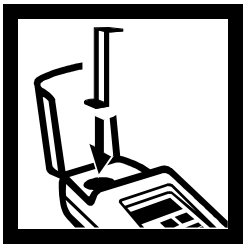
8. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

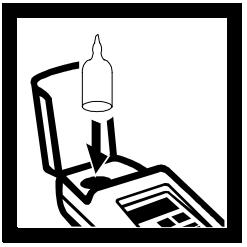


9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L and the zero prompt will turn off.

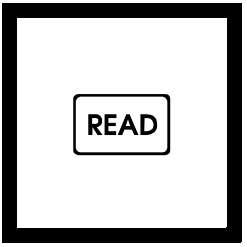


10. Place the AccuVac Vial Adapter into the cell holder.



11. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L nitrate as nitrogen.

Note: *Determine a reagent blank for each new lot of NitraVer 5 ampuls. Repeat Steps 4 - 12, using demineralized water as the sample. Subtract this value from each result obtained with each lot of reagent.*

Note: *To convert the results to mg/L nitrate (NO_3^-) multiply by 4.4*

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, up to 14 days, adjust sample pH to 2 or less with sulfuric acid, ACS, (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature. Neutralize the sample with 5.0 N Sodium Hydroxide Standard Solution. Do not use mercury compounds as preservatives. Correct test results for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a fresh High Range Nitrate Nitrogen Voluette Ampule Standard, 500 mg/L NO_3^--N.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL samples. Mix each thoroughly. (For AccuVac ampuls, use 25 mL in 50-mL beakers.)

c) Analyze each sample as described above. The nitrogen concentration should increase 2.0 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Use a 10.0 mg/L Nitrate Nitrogen Standard Solution listed under Optional reagents to check test accuracy. Or, this can be prepared by diluting 1.00 mL of solution from a High Range Nitrate Nitrogen Voluette Ampule Standard Solution, 500 mg/L NO_3^--N, to 50.0 mL with demineralized water.

PRECISION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 30.0 mg/L NO_3^--N concentration samples, the standard deviation was ±3.40 mg/L NO_3^--N.

Testing zero concentration samples, the limit of detection was 0.66 mg/L NO_3^--N. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980**, 52, 2242-2249)

Using one representative lot of AccuVacs, the standard deviation was ±2.70 mg/L NO_3^--N and the limit of detection was 0.64 mg/L NO_3^--N.

INTERFERENCES

This method registers both the nitrate and nitrite nitrogen present in the sample. If nitrite is present, the nitrite nitrogen test using program number 50.08.1 should be performed on the sample. The amount of nitrite nitrogen found should be subtracted from the results of the nitrate nitrogen test when the following pretreatment is used:

a) Add Bromine Water, 30 g/L, drop-wise to the sample in Step 4 until a yellow color remains.

b) Add one drop of Phenol Solution, 30 g/L, to destroy the color.

c) Proceed with Step 4. Report results as total nitrate and nitrite.

Strong oxidizing and reducing substances will interfere. Ferric iron causes high results and must be absent. Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride levels (i.e., seawater), but a calibration must be performed using standards spiked to the same chloride concentration. See Calibrations paragraph 3.2.4 in the DR/700 Instrument Manual for more information.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

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. This salt couples to gentisic acid to form an amber-colored product.

Nitrate can be determined directly using the Nitrate Ion Selective Electrode (Cat. No. 44560-71).

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
NitraVer 5 Nitrate Reagent Powder Pillows, 10 mL size	1 pillow	100/pkg	21061-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

NitraVer 5 Nitrate Reagent AccuVac Ampul	1 ampul	25/pkg	25110-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00

REQUIRED APPARATUS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
DR/700 Filter Module Number 50.01	1	each	46250-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41
DR/700 Filter Module Number 50.01	1	each	46250-00

OPTIONAL REAGENTS

Bromine Water, 30 g/L	29 mL*	2211-20
Nitrate Nitrogen Standard Solution, 10 mg/L NO_3^--N	500 mL	307-49
Nitrate Nitrogen Standard Solution, Voluette Ampule, 500 mg/L (NO_3^--N), 10 mL	16/pkg	14260-10
Phenol Solution, 30 g/L	29 mL	2112-20
Sodium Hydroxide Standard Solution, 5.0 N	59 mL*	2450-26
Sulfuric Acid, ACS	500 mL*	979-49
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac Vial, DR/700	each	46025-00
Cap for 10-and 25-mL sample cells	12/pkg	24018-12
Dropper, for 1-oz bottle	each	2258-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 1.0 mL	each	515-35
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

In the U.S.A.--Call 800-227-4224 toll-free for more information.

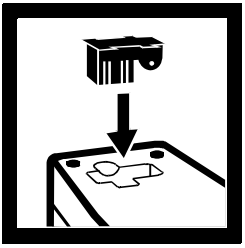
Outside the U.S.A.--Contact the Hach office or distributor serving you.

*Contact Hach for larger sizes.

NITRATE, LR (0 to 0.5 mg/L NO_3-N)

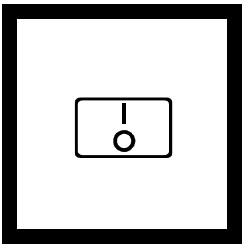
For water, wastewater and seawater*

Cadmium Reduction Method



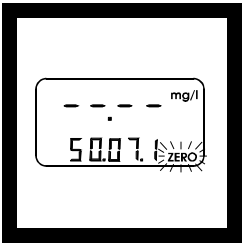
1. Install module number
50.01
in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*



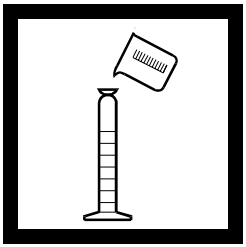
2. Press: **I/O**

The display will show
500 nm
and module number
50.01



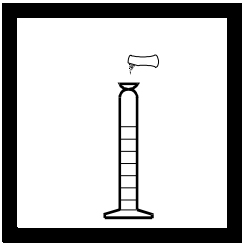
3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
50.07.1

*Seawater requires a manual calibration; see Interferences.

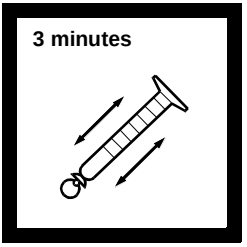


4. Fill a 50-mL graduated mixing cylinder to the 30-mL line with sample.

Note: *For proof of accuracy, use a 0.20 mg/L nitrate nitrogen standard solution (preparation given in Accuracy Check) in place of the sample.*



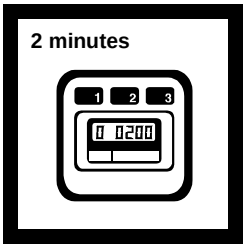
5. Add the contents of one NitraVer 6 Nitrate Reagent Powder Pillow to the cylinder. Stopper.



6. Shake the cylinder continuously for three minutes.

Note: *A deposit of unoxidized metal will remain after the NitraVer 6 Nitrate Reagent Powder dissolves. This is normal and will not affect test results.*

Note: *Shaking time and technique influence color development. For most accurate results, make successive tests on a solution containing a known amount of nitrate and adjust the shaking time to obtain the correct results. See Accuracy Check for more information.*



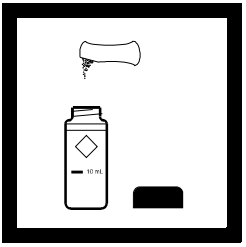
7. Wait 2 minutes.

Note: *A two-minute period allows cadmium to settle.*

8. Fill a 10-mL cell to the 10-mL line with the prepared sample.

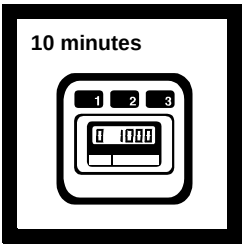
Note: *Take care not to transfer any cadmium particles.*

Note: *A 25-mL sample can be tested by using 25-mL sample cells and optional NitriVer 3 Reagent.*

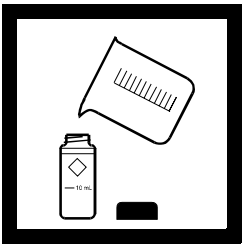


9. Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow to the sample cell (the prepared sample). Cap and shake to dissolve.

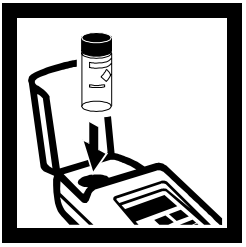
Note: *A pink color will develop if nitrate is present.*



10. Wait 10 minutes.

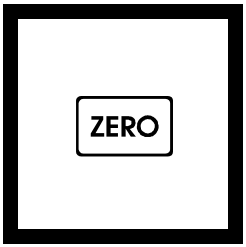


11. Fill a 10-mL cell to the 10-mL line with sample (the blank).



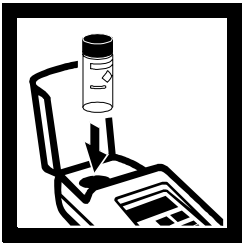
12. Place the blank in the cell holder.

Note: *In bright sunlight, it may be necessary to close the cell compartment cover.*



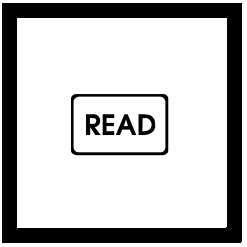
13. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



14. Place the prepared sample in the cell holder.

Note: *In bright sunlight, it may be necessary to close the cell compartment cover.*



15. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L nitrate as nitrogen (NO_3-N).

Note: *To convert the results to mg/L NO_3, multiply by 4.4.*

Note: *Rinse the sample cell immediately after use to remove all cadmium particles.*

Note: *Determine a reagent blank for each new lot of powder pillows. Repeat Steps 4 to 14 using demineralized water as the sample. Subtract this value from each result obtained with the new lot of reagent.*

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution. Do not use mercury compounds as preservatives. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard additions Method

a) Measure 30 mL of sample into three cylinders.

b) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of Nitrate Nitrogen, Voluette Ampule Standard Solution, 12 mg/L as (NO_3^--N), to the three samples. Mix well.

c) Analyze each sample as described above. The nitrate nitrogen concentration should increase 0.04 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 0.20 mg/L nitrate nitrogen standard by diluting 2.00 mL of the 10 mg/L Nitrate Nitrogen Standard Solution to 100 mL with demineralized water. Or, using the TenSette pipet, make a 0.12 mg/L nitrate nitrogen standard by diluting 1.0 mL of a Nitrate Nitrogen Voluette Ampule Standard Solution, 12 mg/L, to 100 mL with demineralized water.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 0.30 mg/L NO_3-N concentration samples the standard deviation was ±0.012 mg/L NO_3-N.

Testing zero concentration samples, the limit of detection was 0.024 mg/L NO_3-N. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52,* 2242-2249).

INTERFERENCES

This method registers both the nitrate and nitrite nitrogen present in the sample. If nitrite is present, the nitrite nitrogen test using program number 50.08.1 should be done on the sample. The amount of nitrite nitrogen found should be subtracted from the results of the nitrate nitrogen test when the following pretreatment is used:

a) Add Bromine Water drop-wise to 30-mL of sample until a yellow color persists. Mix after adding each drop.

b) Add one drop of Phenol Solution. Swirl to destroy the yellow color.

c) Continue with Step 4 of the nitrate procedure.

Calcium interferes in amounts over 100 mg/L as $CaCO_3$.

Chlorides in amounts over 100 mg/L cause low results. To determine nitrate in high chloride samples or seawater, a manual calibration must be performed. See section 3.2.4 in the DR/700 instrument manual. Prepare nitrate standard solutions with the approximate chloride concentration of the samples to be tested. Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

SUMMARY OF METHOD

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to chromotropic acid to form a pink-colored product.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
NitriVer 3 Nitrite Reagent Powder Pillows	1 pillow	100/pkg	21071-69
NitraVer 6 Nitrate Reagent Powder Pillows	1 pillow	50/pkg	14119-66

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, mixing, 50 mL	1	each	1896-41
DR/700 Filter Module Number 50.01	1	each	46250-00

OPTIONAL REAGENTS

Bromine Water	29 mL*		2211-20
Nitrate Nitrogen Standard Solution, 10 mg/L as NO_3^--N	500 mL		307-49
Nitrate Nitrogen Standard Solution, Voluette ampule, 12 mg/L as NO_3^--N, 10 mL	16/pkg		14333-10
NitriVer 3 Nitrite Reagent Powder Pillows	50/pkg		14065-66
Phenol Solution, 30 g/L	30 mL		2112-20
Pretreatment Kit, contains: (1) 2112-20, (1) 2211-20	each		2268-00
Sodium Hydroxide Standard Solution, 5.0 N	59 mL*		2450-26
Sulfuric acid, ACS	500 mL*		979-49
Water, demineralized	4 L		272-56

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells	12/pkg		24018-12
Dropper, for 1-oz bottle	each		2258-00
Flask, volumetric, 100 mL	each		547-42
pH Indicator Paper, 1 to 11 pH	5 roll/pkg		391-33
Pipet, serological, 2 mL	each		532-36
Pipet, TenSette, 0.1 to 1.0 mL	each		19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg		21856-96

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

Nitrate at these levels can be determined directly using the Nitrate Ion Selective Electrode (Cat. No. 44560-71)

For Technical Assistance, Prices and Ordering

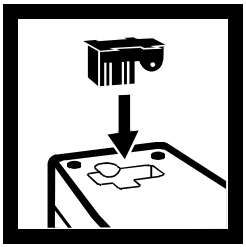
In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

NITRITE, LR (0 to 0.350 mg/L NO_2^--N)

For water, wastewater and seawater

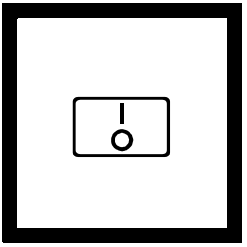
Diazotization Method (Powder Pillows or AccuVac Ampuls); USEPA accepted for reporting*

USING POWDER PILLOWS



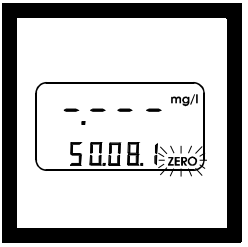
1. Install module number
50.01
in a DR/700

Note: *If sample cannot be analyzed immediately, see Sampling and Storage following these steps.*



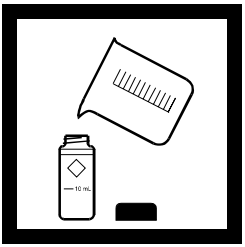
2. Press: **I/O**

The display will show
500 nm
and module number
50.01



3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
50.08.1

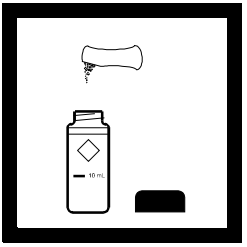
Federal Register,* **May 1, 1979, *44(85),* 25505



4. Fill a 10-mL cell to the 10-mL line with sample (the blank).

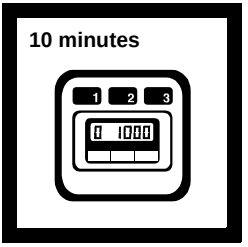
Note: *For proof of accuracy, use a 0.10 mg/L nitrite nitrogen standard solution (preparation given in Accuracy Check) in place of the sample.*

Note: *A 25-mL sample can be tested by using 25-mL sample cells and optional reagents.*

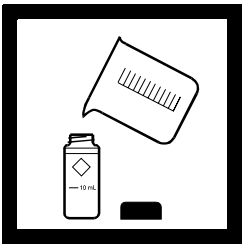


5. Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow (the prepared sample). Cap and invert to mix.

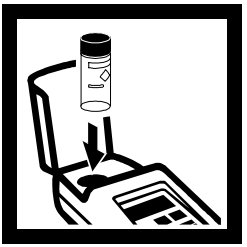
Note: *A pink color will develop if nitrite nitrogen is present.*



6. Wait 10 minutes.

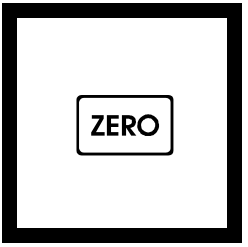


7. Fill a 10-mL cell to the 10-mL line with sample (the blank).



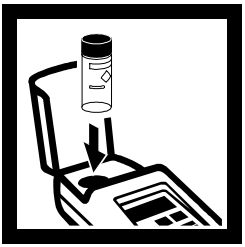
8. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



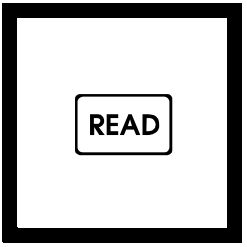
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.



10. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

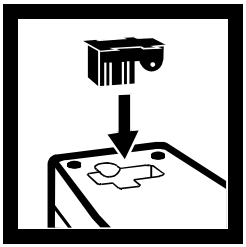


11. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L nitrite as nitrogen (NO_2^--N).

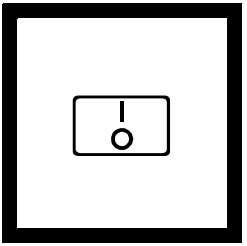
Note: *To convert the results to mg/L NO_2^-, multiply by 3.3.*

USING ACCUVAC AMPULS



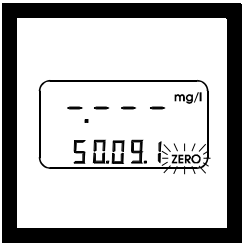
1. Install module **50.01** in a DR/700.

Note: *If sample cannot be analyzed immediately, see Sampling and Storage following these steps.*

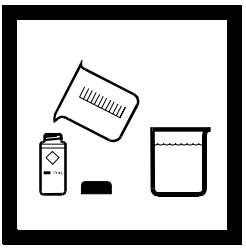


2. Press: **I/O**

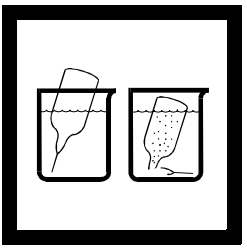
The display will show **500 nm** and module number **50.01**



3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **50.09.1**



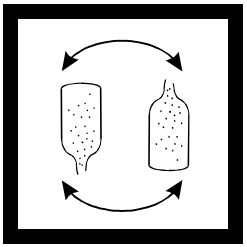
4. Fill a cell with 10 mL of the sample (the blank). Cap. Collect at least 40 mL of sample in a 50-mL beaker.



5. Fill a NitriVer 3 Nitrite AccuVac Ampul with sample.

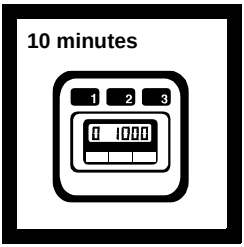
Note: *Keep the tip immersed while the ampul fills completely.*

Note: *For proof of accuracy, use a 0.10 mg/L nitrite nitrogen standard solution (preparation given in Accuracy Check) in place of the sample.*

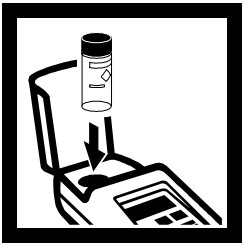


6. Invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will develop if nitrite nitrogen is present.*

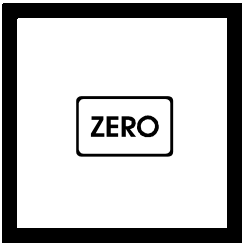


7. Wait 10 minutes.



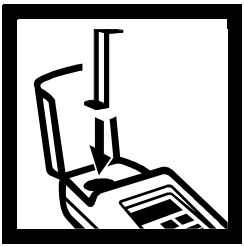
8. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

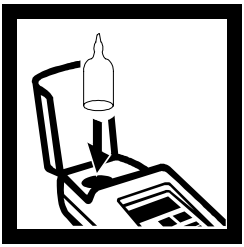


9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.

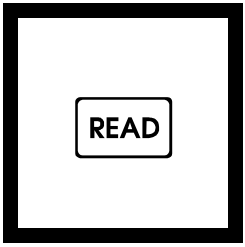


10. Insert the AccuVac Vial Adapter into the cell holder.



11. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L nitrite as nitrogen (NO_2^--N).

Note: *To convert the results to NO_2^-, multiply by 3.3.*

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, add 4.0 mL of Mercuric Chloride Solution* for each liter of sample taken and mix. Sample refrigeration is still required. **This storage method may not be used when reporting results to regulatory agencies.** Do not use acid preservatives.

*Use of mercuric chloride is not recommended due to environmental and health concerns.

ACCURACY CHECK

Standard Solution Method

Prepare a nitrite nitrogen standard solution by dissolving 0.493 grams of sodium nitrite, ACS, in 1000 mL of nitrite-free demineralized water to give a 100 mg/L nitrite nitrogen (NO_2^--N) standard solution. This solution is not stable and should be prepared daily. Use a TenSette Pipet to dilute 1.00 mL of the stock solution to 1000 mL with nitrite-free demineralized water to give a 0.10 mg/L (NO_2^--N) nitrite nitrogen standard solution. Prepare this solution immediately before use.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 0.091 mg/L NO_2^--N concentration samples the standard deviation was ±0.0010 mg/L NO_2^--N.

Testing zero concentration samples, the limit of detection was 0.0038 mg/L NO_2^--N. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52*, 2242-2249).

Using two representative lots of AccuVacs, the standard deviation was ±0.0016 mg/L NO_2^--N and the limit of detection was 0.0089 mg/L NO_2^--N.

INTERFERENCES

Strong oxidizing and reducing substances interfere. Cupric and ferrous ions cause low results. Ferric, mercurous, silver, bismuth, antimonous, lead, auric, chloroplatinate and metavanadate ions interfere by causing precipitation.

Very high levels of nitrate (100 mg/L nitrate as N or more) 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.

SUMMARY OF METHOD

Nitrite in the sample reacts with sulfanilic acid to forms an intermediate diazonium salt. This couples with chromotropic acid to produce a pink colored complex directly proportional to the amount of nitrite present.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
NitriVer 3 Nitrite Reagent Powder Pillows, 10 mL sample	1 pillow	100/pkg	21071-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

NitriVer 3 Nitrite Reagent AccuVac Ampul	1 ampul	25/pkg	25120-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 50.01	1	each	46250-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac Vial	1	each	46025-00
Beaker, 50 mL	1	each	500-41

OPTIONAL REAGENTS

Mercuric Chloride Solution	100 mL	14994-42
NitriVer 3 Nitrite Reagent Powder Pillows, 25 mL sample	50/pkg	14065-66
Sodium Nitrite, ACS	454 g	2452-01
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac Vial, DR/700	each	46025-00
Balance, analytical	each	22310-00
Caps for 10- and 25-mL Sample Cells	12/pkg	24018-12
Flask, volumetric, 1000 mL	each	547-53
Pipet, serological, 10 mL	each	532-38
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 1.0 mL	each	515-35
Pipet Filler, safety bulb	each	14651-00
Sample Cells, 10-mL with screw cap	6/pkg	24276-06
Sample Cells, 25-mL with screw cap	6/pkg	24019-06

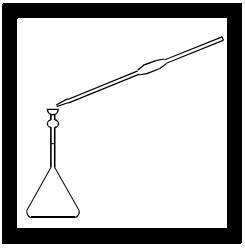
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

PLATINUM (0 to 10 g/L)

For surface finishing solutions

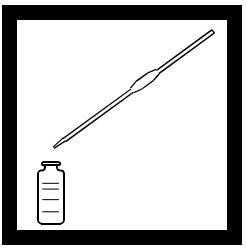
N,N'-Dimethyldithiooxamide Method*



1. Dilute the platinum bath solution (sample) by pipetting 1.0 mL of the bath solution into a 1000-mL volumetric flask. Fill the flask to the mark with demineralized water. Cap and invert at least 10 times to mix.

Note: *If the platinum bath solution has more than 10 g/L (1.2 troy oz./gal) of platinum, a larger dilution is necessary.*

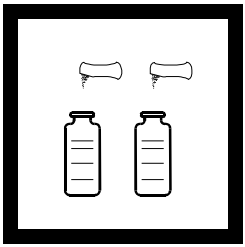
Note: *The DR/700 must be calibrated before sample measurement. See Calibration following these steps.*



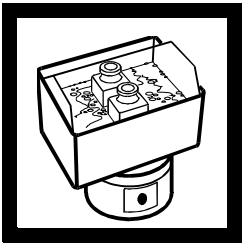
2. Pipet 15.0 mL of the diluted sample bath solution into a clean 25-mL square mixing bottle (the prepared sample).

3. Using a graduated cylinder, fill a second mixing bottle with 15 mL of demineralized water (the blank).

*User calibration required; range is approximate.

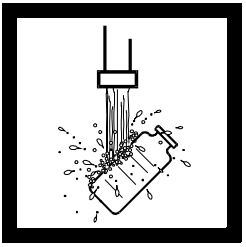


4. Add the contents of one Chromium 1 Reagent Powder Pillow to each bottle. Swirl to dissolve. Place a hollow polyethylene stopper loosely in to each bottle.

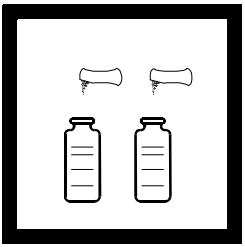


5. Place both bottles in a boiling water bath for five minutes.

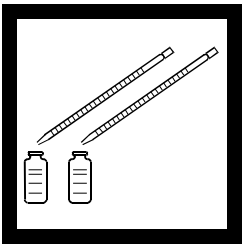
Note: *The sample may become turbid upon heating. The turbidity will clear when hydrochloric acid is added in Step 8.*



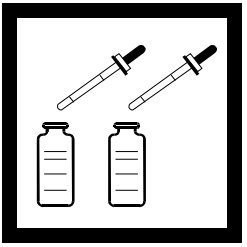
6. Cool both bottles under tap water to about room temperature.



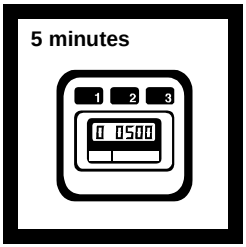
7. Add the contents of one Sodium Metabisulfite Reagent Powder Pillow to each bottle. Swirl to dissolve.



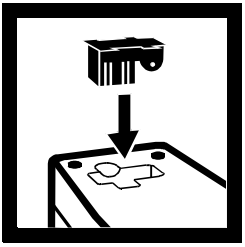
8. Using a 10-mL Mohr pipet bulb, carefully add 10 mL of concentrated hydrochloric acid to each bottle. Swirl to mix. The bottles will become warm to the touch.



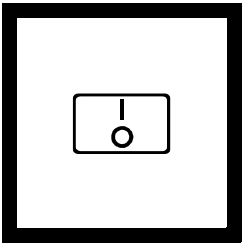
9. Using the 1-mL calibrated dropper, add 1.0 mL of N,N'-Dimethyldithiooxamide Indicator Solution to each bottle. Swirl to mix.



10. Wait 5 minutes.

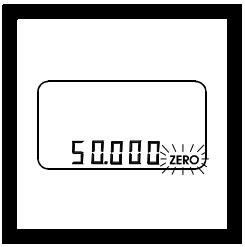


11. Install module **50.01** in a DR/700.



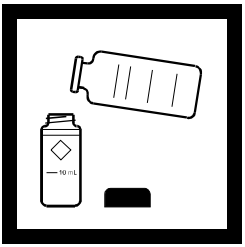
12. Press: **I/O**

The display will show **500 nm** and module number **50.01**

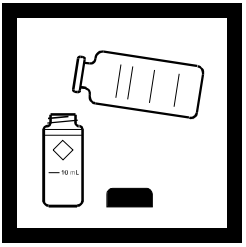


13. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. Press the **PROGRAM** key once or twice until the lower display shows program number **50.000**

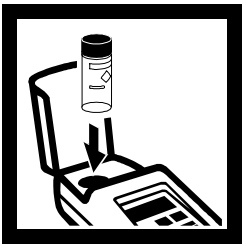
The upper display will show the S1 concentration.



14. Fill a 10-mL cell to the 10-mL line with the blank. Cap.

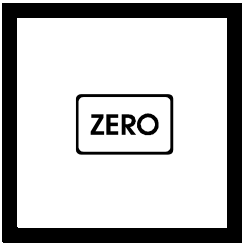


15. After the 5-minute period, fill another 10-mL cell to the 10-mL line with the prepared sample. Cap.



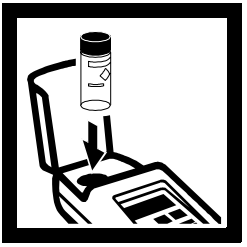
16. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



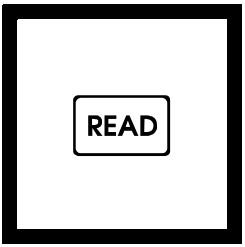
17. Press: **ZERO**

The display will count down to 0. Then the display will show zero concentration and the zero and S1 prompt will turn off.



18. Place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



19. Press: **READ**

The display will count down to 0. Then the display will show the results in g/L platinum (Pt).

Note: If necessary, correct for any dilution used in Step 1 if other than 1 mL to 1000 mL.

SAMPLING AND STORAGE

Several locations within the bath should be sampled and combined to obtain a representative sample of bath solution. Store the samples in clean plastic or glass bottles. Analyze as soon as possible after collection.

INTERFERENCES

Palladium and copper will react under the conditions of the procedure.

SUMMARY OF METHOD

The platinum in an electrolytic bath sample is first oxidized to Pt^{4+} with Chromium 1 Reagent. The excess oxidant is then destroyed and the platinum is reduced to the +2 oxidation state. Platinum ion then reacts with N,N'-Diethyldithioooxamide Indicator to form an orange complex which is proportional to the amount of platinum present.

CALIBRATION

A user calibration is required before bath sample can be analyzed. This calibration is based on a 1:1000 dilution factor being used on the bath solution.

Prepare an equivalent 10-g/L standard solution by pipetting 10.00 mL of Platinum Standard solution into a 1-L volumetric flask. Dilute to the line with demineralized water. Stopper. Invert at least 10 times to mix.

Refer to paragraph 2.2.4.1, Calibration Using Two Prepared Standards, in the DR/700 instrument manual. Follow the platinum procedure starting at Step 2 to develop the S1 and S2 standards. Use 15 mL of demineralized water in Step 3. This is Standard 1 (S1) and is equal to 0.0 g/L Pt. Use 15 mL of the prepared platinum solution above in Step 2. This is Standard 2 (S2) and is equivalent to 10.0 g/L Pt.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Chromium 1 Reagent Powder Pillows	2	100/pkg	2043-99
Hydrochloric Acid, ACS	20 mL	2.8 kg	134-06
N,N'-Dimethyldithiooxamide Indicator Solution	2 mL	100 mL	23087-32

REQUIRED REAGENTS (continued)

Description	Quantity Per Test	Unit	Cat. No.
Platinum Standard Solution, 1000 mg/L	10 mL	100 mL	23208-42
Sodium Metabisulfite Reagent Powder Pillows	2	100/pkg	7095-99
Water, demineralized	1015 mL	4 L	272-56

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508-40
Clippers, large	1	each	968-00
DR/700 Filter Module Number 50.01	1	each	46250-00
Finger Cots	1	2/pkg	14647-02
Flask, volumetric, Class A, 1000 mL	1	each	14574-53
Hot Plate, 4" circular, 120 V	1	each	12067-01
Hot Plate, 4" circular, 240 V	1	each	12067-02
Pipet, volumetric, Class A, 10.00 mL	1	each	14515-38
Pipet, volumetric, Class A, 15.00 mL	1	each	14515-39
Pipet Filler, safety bulb	1	each	14651-00
Pipet, Mohr, 10 mL	1	each	20934-38
Sample Cell, 25-mL, 1-inch, square	2	2/pkg	13537-02
Stopper, hollow, No. 1	2	6/pkg	14480-01
Water Bath and Bottle Rack (requires hot plate)	1	each	1955-55

For Technical assistance, Prices and Ordering

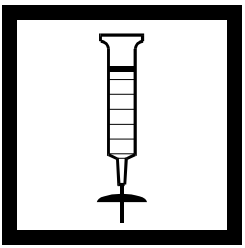
In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

POLYACRYLIC ACID (0 to 20.0 mg/L)

For water and brines

Adsorption-Colorimetric Method (as Acrysol® LMW-20, -45)

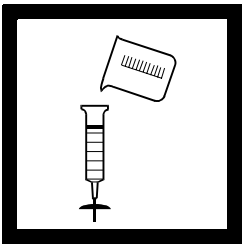
SAMPLE PREPARATION



1. Remove the syringe plunger. Attach the prefilter to the syringe barrel, twisting to lock it on.

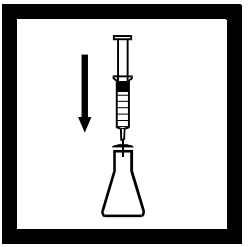
Note: *Samples should be analyzed promptly. See Sampling and Storage following these steps.*

Note: *See Interferences for pretreatment of turbid or oily samples.*

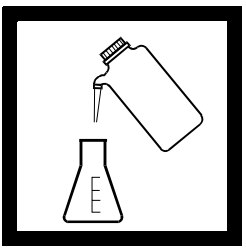


2. Rinse the syringe with the sample. Fill to the 30-cc mark.

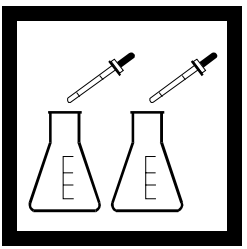
Note: *The syringe markings may wear off with continued use. They can be made more permanent by scoring with a knife.*



3. Insert the plunger and force the sample through the filter into a 50-mL erlenmeyer flask labeled "sample".



4. Fill another clean flask, labeled "reagent blank" with approximately 30 mL of demineralized water.

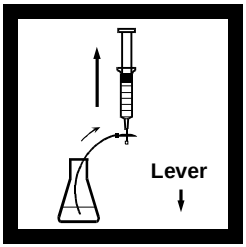


5. Add 0.5 mL of Buffer Solution, pH 2.5, to each flask. Swirl to mix.

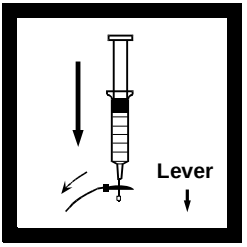
Note: *Check the sample pH with pH indicator paper. If necessary, adjust to pH 2-3 with 1:1 Nitric Acid Solution.*

6. Fill a third flask, labeled "eluant", with approximately 30 mL of Polyacrylic Acid Eluant Solution.

®Acrysol is a registered trademark of the Rohm and Haas Company

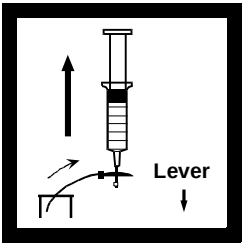


7. Assemble the syringe apparatus as shown. Place the long end of the LC cartridge on the male tip of the three-way valve. Turn the valve to the aspirate (down) position. Draw about 5 cc of blank through the tubing into the syringe. Draw in air to the 30-cc mark.



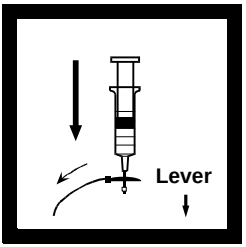
8. Rinse the syringe. Discard the solution through the tubing.

Note: *Move the plunger up and down several times to clear the tubing.*

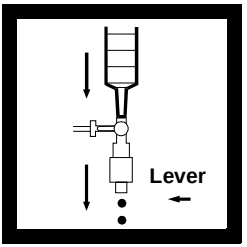


9. Draw the remaining reagent blank into the syringe through the tubing followed by a small volume of air, past the 30-cc mark.

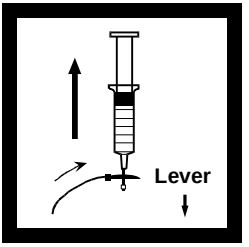
Note: *A small volume of air above the solution facilitates complete elution from the LC column.*



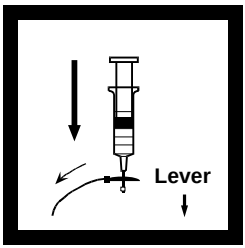
10. Push the plunger down to adjust the solution volume to exactly the 20-cc mark.



11. Rotate the valve lever to the pump (left) position and slowly force the solution through the LC cartridge over a period of at least 15 seconds, discarding the solution.

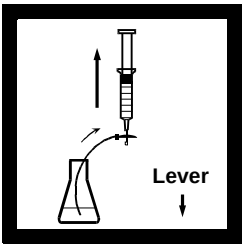


12. Again rotate the valve lever to the aspirate (down) position. Draw about 5 cc of eluant into the syringe through the tubing followed by air past the 25-cc mark.

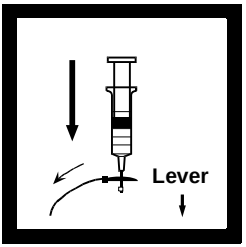


13. Rinse the syringe by shaking. Discard the eluant through the tubing.

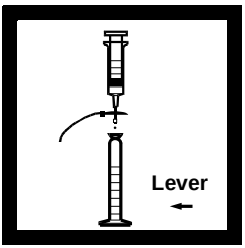
Note: *Move the plunger up and down several times to clear the tubing.*



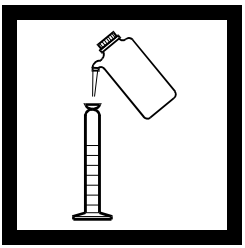
14. Draw at least 10 cc of eluant into the syringe through the tubing followed by air past the 25-cc mark.



15. Push the plunger down to adjust the eluant volume to exactly the 10-cc mark.



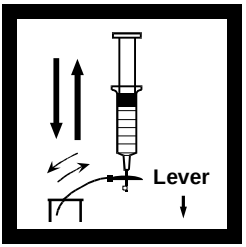
16. Rotate the valve lever to the pump (left) position. Over a period of 30 seconds force the eluant through the LC cartridge. Collect the eluant in a 25-mL tall-form graduated cylinder.



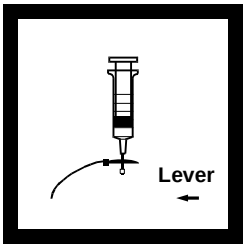
17. Fill the cylinder to exactly 25 mL with demineralized water. Stopper and invert to mix (the blank).

Note: *Up to five samples can be run with one blank.*

Note: *Volumes are critical at this point. The cylinders can be matched by pipetting 25.00 mL of demineralized water into each, and marking them at the correct volume. Tall-form cylinders must be used.*

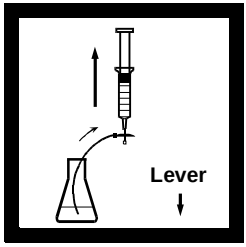


18. Clean the syringe and cartridge by drawing in 25 cc of demineralized water in the aspirate (down) position. Discard the water back through the tubing.



19. Repeat, discarding the water through the cartridge with the valve in the pump (left) position.

Note: *The cartridge must be rinsed to remove any traces of eluant, which would affect adsorption of pAA from the next sample.*

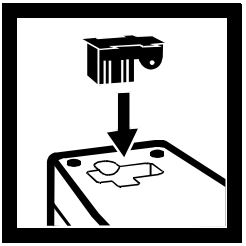


20. Return the valve to the aspirate (down) position and repeat Steps 8 through 20, using the buffered sample in place of the blank. Label the glassware "sample".

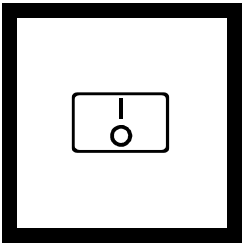
Note: *After use, rinse the LC cartridge with 2 cc of eluant solution, then demineralized water. Store the cartridge in the vial supplied with a few drops of eluant solution.*

POLYACRYLIC ACID, continued

COLORIMETRIC ANALYSIS

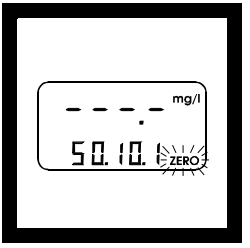


1. Install module number
50.01
in a DR/700.

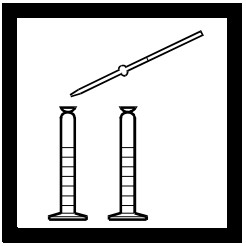


2. Press: **I/O**

The display will show 500 nm and module number
50.01

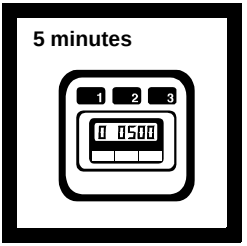


3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
50.10.1
for LMW-20 or
50.11.1
for LMW-45

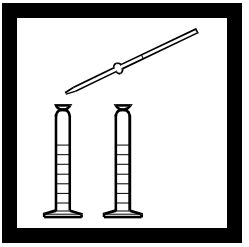


4. Pipet exactly 1.00 mL of Polyacrylic Acid 1 Reagent into each mixing cylinder. Stopper and invert to mix. Proceed immediately to Step 5.

Note: *Use a volumetric pipet or TenSette Pipet to measure this volume.*

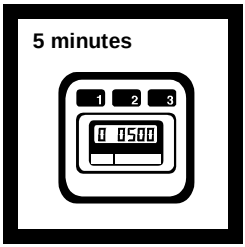


5. Wait 5 minutes.

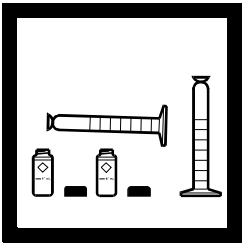


6. Add exactly 1.0 mL of Polyacrylic Acid 2 Reagent to each cylinder. Stopper and invert to mix. Proceed rapidly through Steps 7 and 8.

Note: *Use a volumetric pipet or TenSette pipet to measure this volume.*

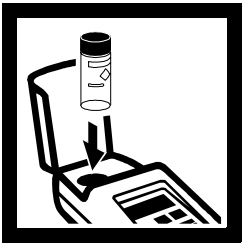


7. Immediately place the cylinders in the dark. Begin timing a 5-minute period.



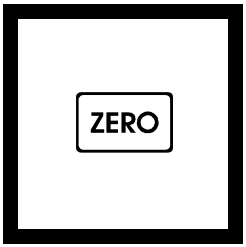
8. After the 5-minute period, fill a 10-mL cell to the 10-mL line with the blank. Cap and label the cell "reagent blank".

Fill a 10-mL cell to the 10-mL line with the prepared sample. Cap and label the cell "sample".



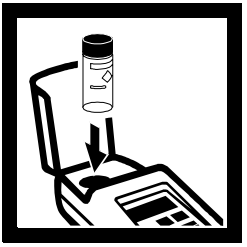
9. Place the cell labeled "reagent blank" in the cell holder.

Note: *In bright sunlight, it may be necessary to close the cell compartment cover.*



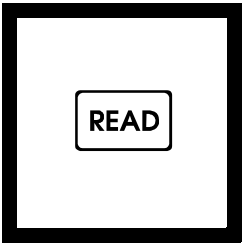
10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L and the zero prompt will turn off.



11. Place the cell labeled "sample" in the cell holder.

Note: *In bright sunlight, it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L polyacrylic acid as Acrysol (acid form, total solids basis).

Note: *If concentrations of less than 1 mg/L are being determined, see the Interference section.*

Note: *For concentrations above the test range, dilute the sample by an appropriate factor and repeat.*

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 3.4 mg/L Acrysol LMW-45, acid form, concentration samples the standard deviation was ±0.33 mg/L pAA.

Testing zero concentration samples, the limit of detection was 0.93 mg/L pAA. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52,* 2242-2249).

INTERFERENCES

Filter turbid or oily samples through glass wool or a moderately rapid paper, such as S&S No. 560, before beginning the test.

If the sample is laden with oil, do a preliminary extraction of the oil with 1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113) as follows:

a) Transfer a sample portion to a clean 125-mL separatory funnel.

b) Add 10 mL Freon 113 to the funnel. Stopper. Shake vigorously for two minutes, occasionally lifting the stopper to release pressure inside the funnel. After shaking, remove the stopper. Allow the funnel to stand undisturbed for 10 minutes while the layers separate.

c) Drain and discard the bottom (Freon) layer containing the oil. Repeat, if necessary, to remove all visible oil.

d) Continue with Step 1 of Sample Preparation.

The test works in brines having levels as high as 75,000 mg/L total dissolved solids (TDS) and 20,000 mg/L chloride. Minimize the effect of most interferences by flushing the LC cartridge after the polyacrylic acid is adsorbed on the column. Prepare 30 mL of demineralized water buffered with 0.5 mL of Buffer Solution, pH 2.5. Repeat Steps 7 to 11 in the Sample Preparation section with this solution. Continue with Step 12.

Samples with concentrations of less than 1 mg/L should be repeated. Use two 20-cc volumes (instead of one) of the sample through the cartridge in Sample Preparations Step 10 to 11. Continue with Step 12. (This will require filtering two 30-cc volumes of sample in Step 2.). Divide the resulting concentration by two. Up to five 20-cc volumes of the sample can be run through the LC cartridge. It may be necessary to flush the LC cartridge as stated above.

Avoid use of facial tissue when drying glassware or apparatus, as it may contain interfering substances. Kimwipes or Kaydry wipers are recommended.

SUMMARY OF METHOD

Polyacrylic acids (pAA) in the sample are selectively adsorbed onto a liquid chromatographic column using a technique developed by Rohm

and Haas Company. After separation from the sample, the pAA is eluted off the column and the concentration is determined colorimetrically.

Calibrations are based on Rohm and Haas Company Acrysol LMW (low molecular weight) polyacrylic acid products. Because commercially available polyacrylic acid can come in many strengths, preparation of the standards used for the calibrations was based on a 100% total solids basis and 100% active polymer. This method can be adapted for most other low molecular weight polyacrylic acids, polyacrylates or associated copolymers used as commercial scale inhibitors. For accurate work, diluted standards prepared from the product in use would be used to establish the calibration. The concentration of the polymer in solution should be reported, if possible, on a dry-weight basis.

REQUIRED REAGENTS

	Cat. No.
Polyacrylic Acid Reagent Set (30 Tests)	22252-00
Includes: (1) 22253-32, (1) 22762-42, (1) 22763-42, (1) 22256-53	

Description	Quantity Per test	Unit	Cat. No.
Buffer Solution, pH 2.5	1 mL	100 mL MDB	22253-32
Polyacrylic Acid 1 Reagent	2 mL	100 mL	22762-42
Polyacrylic Acid 2 Reagent	2 mL	100 mL	22763-42
Polyacrylic Acid Eluant Solution	30 mL	1000 mL	22256-53

REQUIRED APPARATUS

Cylinder, mixing, tall form, 25 mL	2	each	21190-40
DR/700 Filter Module Number 50.01	1	each	46250-00
Flask, erlenmeyer, 50 mL	3	each	505-41
Polyacrylic Acid Apparatus Set	1	each	22257-00
contains:			
Fitting-Tube Assembly		each	22266-00
LC Cartridge		each	22264-00

REQUIRED APPARATUS (continued)

Description	Quantity Per test	Unit	Cat. No.
Polyacrylic Acid Apparatus Set (continued)			
pH Paper		5 rolls/pkg	391-33
Prefilter, 5 µm, 25 mm		each	22261-00
Syringe, 30 cc		each	22258-00
Three-Way valve		each	22259-00
Pipet, Volumetric, Class B, 1 mL	2	each	515-35
Pipet Filler, safety bulb	1	each	14651-00

OPTIONAL REAGENTS

Nitric Acid Solution, 1:1	500 mL	2540-49
Freon 113 (1,1,2-trichloro-1,2,2-trifluoroethane)	500 mL	14348-49

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Filter Paper, folded, S&S No. 560, 12.5 cm	100/pkg	692-57
Funnel, filtering, long stem, 75 mm	each	549-68
Funnel, separatory, 125 mL	each	520-43
Kaydry Wiper, 38 X 43 cm (15 X 17")	90/box	20969-00
Kimwipe Wiper, 11 X 22 cm (5 X 8")	280/box	20970-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 25 mL	each	14515-40
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

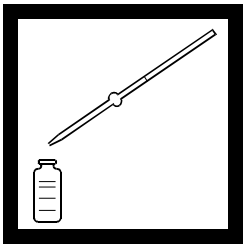
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A - Contact the Hach office or distributor serving you.

VOLATILE ACIDS (0 to 2500 mg/L)

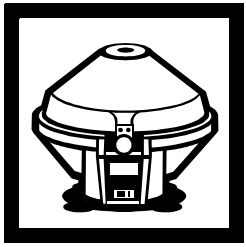
For digestor sludges

Esterification Method*

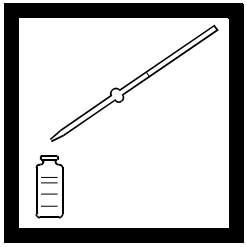


1. Pipet 0.5 mL of demineralized water into a dry 25-mL square mixing bottle (the blank).

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*

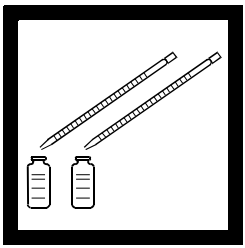


2. Filter or centrifuge 25 mL of the sample using labware listed under Required Apparatus.

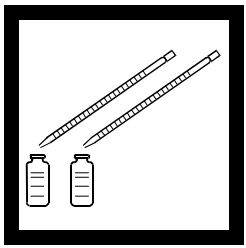


3. Pipet 0.5 mL of the filtrate or supernatant into another dry 25-mL square mixing bottles (the prepared sample).

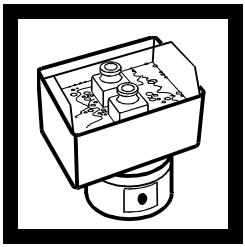
Note: *For proof of accuracy, use 0.5 mL of a 500 mg/L volatile acid solution (preparation given in Accuracy Check) in place of the sample.*



4. Pipet 1.5 mL of ethylene glycol into each sample cell. Swirl to mix.

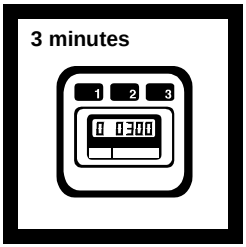


5. Pipet 0.2 mL of 19.2 N Sulfuric Acid Standard Solution into each cell. Swirl to mix.

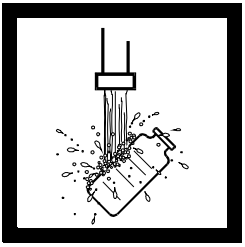


6. Place both cells into a boiling water bath.

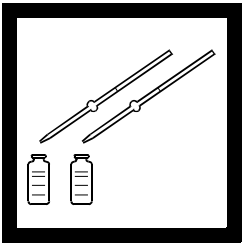
*Adapted from *The Analyst*, **1962**, *87*, 94



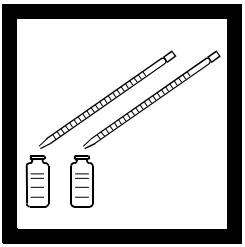
7. Wait 3 minutes.



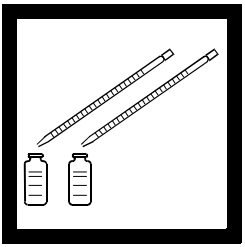
8. Cool solutions to 25 °C (until the cell feels cold) with running tap water.



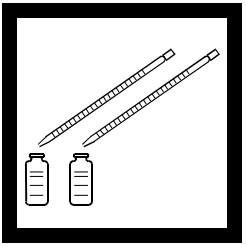
9. Pipet 0.5 mL of Hydroxylamine Hydrochloride Solution into each cell. Swirl to mix.



10. Pipet 2.0 mL of 4.5 N Sodium Hydroxide Solution into each cell. Swirl to mix.



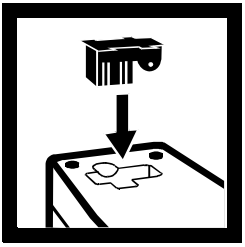
11. Add 10 mL of Ferric Chloride Sulfuric Acid Solution to each cell. Swirl to mix.



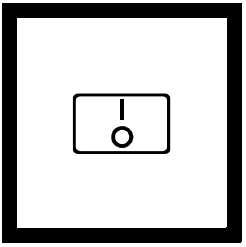
12. Add 10 mL of demineralized water to each cell. Swirl to mix.

13. Wait 3 minutes.

Note: *During the 3-minute period, complete Step 14-20 so the prepared sample is read when the timer stops.*

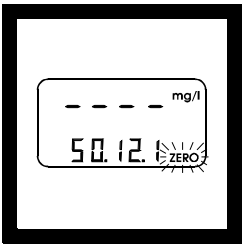


14. Install module **50.01** in a DR/700.

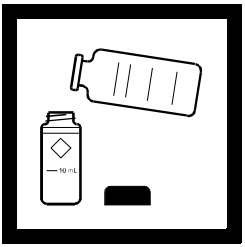


15. Press: **I/O**

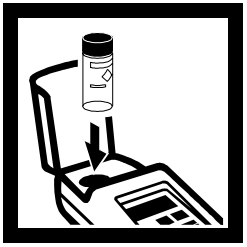
The display will show **500 nm** and module number **50.01**



16. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **50.12.1**

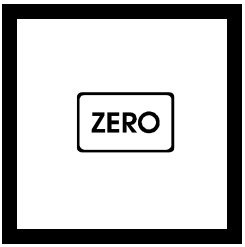


17. Pour 10 mL of the blank solution into a 10-mL round sample cell. Cap.



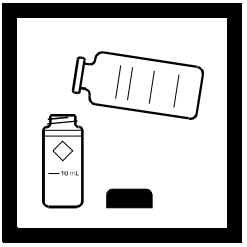
18. Place the blank in the cell holder.

Note: *In bright light, it may be necessary to close the cell compartment cover.*

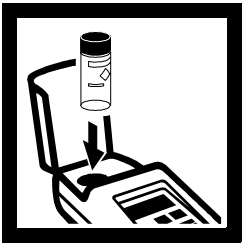


19. Press: **ZERO**

The display will count down to 0. Then the display will show 0 mg/L and the zero prompt will turn off.

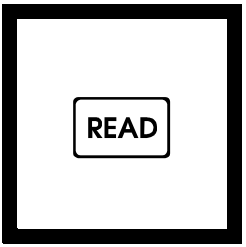


20. Pour 10 mL of the prepared sample into a 10-mL round sample cell. Cap.



21. Place the prepared sample in the cell holder.

Note: *In bright light, it may be necessary to close the cell compartment cover.*



22. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L volatile acids (HOAc).

Note: *Read the prepared sample at 3 minutes as noted in Step 13.*

SAMPLING AND STORAGE

Collect samples in plastic or glass bottles. Analyze sample as soon as possible after collection. Samples can be stored up to 24 hours by cooling to 4 °C (39 °F) or below. Warm to room temperature before running the test.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Volatile Acids Voluette Ampule Standard Solution, 62,500 mg/L as acetic acid.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL graduated mixing cylinders containing 25 mL of filtered sample. Stopper. Shake well to mix.

c) Remove a 0.5 mL aliquot of sample from each cylinder; add to a sample cell. All three samples can be analyzed along with the original test sample beginning with Step 7 of the procedure. The volatile acid concentration should increase 250 mg/L volatile acids as acetic acid for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 500 mg/L volatile acid standard by using the TenSette Pipet to add 0.8 mL of a Volatile Acids Voluette Ampule Standard Solution to a 100-mL volumetric flask. Dilute to volume with demineralized water.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 1250 mg/L as HOAc concentration samples, the standard deviation was ±4.5 mg/L HOAc.

Testing zero concentration samples, the limit of detection was 23.5 mg/L HOAc. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

VOLATILE ACIDS, continued

SUMMARY OF METHOD

The volatile acids test is designed specifically for the determination of volatile acids in digestor sludges. The method is based on esterification of the carboxylic acids present and determination of the esters by the ferric hydroxamate reaction. All volatile organic acids present are reported as their equivalent mg/L acetic acid.

REQUIRED REAGENTS

	Cat. No.
Volatile Acids Reagent Set (90 tests)	22447-00
Includes: (1) 2039-53, (2) 2042-53, (1) 818-42, (1) 2040-53, (1) 2038-32	

Description	Quantity Per Test	Unit	Cat. No.
Ethylene glycol	3 mL	1000 mL	2039-53
Ferric Chloride-Sulfuric Acid Solution	20 mL	1000 mL	2042-53
Hydroxylamine Hydrochloride Solution, 100 g/L	1 mL	100 mL	818-42
Sodium Hydroxide Standard Solution, 4.5 N	4 mL	1000 mL	2040-53
Sulfuric Acid Standard Solution, 19.2 N	0.4 mL	100 mL	2038-32
Water, demineralized	20.5 mL	4 L	272-56

REQUIRED APPARATUS

Centrifuge	1	each	22413-00
Centrifuge Tubes	varies	10/pkg	22787-39
Cots, finger	2	2/pkg	14647-02
Cylinder, graduated, 10 mL	1	each	508-38
DR/700 Filter Module Number 50.01	1	each	46250-00
Hot Plate, 4-inch circular, 3-1/2" diam.	1	each	12067-01
Pipet Filler, safety bulb	1	each	14651-00
Pipet, serological, 2 mL	2	each	532-36
Pipet, volumetric, 0.5 mL	3	each	14515-34
Sample Cell, 25-mL, square	2	2/pkg	13537-02
Water Bath and Rack	1	each	1955-55

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Volatile Acids Standard Solution, Voluette ampule, 62,500 mg/L as acetic acid, 10 mL	16/pkg	14270-10
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Caps for 10- and 25-mL sample Cells	12/pkg	24018-12
Cylinder, graduated, mixing, 25 mL	each	1896-40
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, erlenmeyer, 50 mL	each	505-41
Funnel, poly, 65 mm	each	1083-67
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

Module 52.01
525 nm

Hach warrants equipment it manufactures against defective materials or workmanship for at least one year from the shipping date. Warranties do not apply to limited-life electrical components such as batteries. Full warranty information is located on the reverse side of Hach invoices.

IMPORTANT NOTICE

A lamp intensity adjustment must be performed:
- Before first or initial use
- When new filter modules are installed
- On each filter module when the lamp is replaced

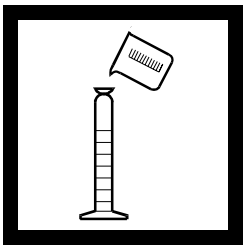
Refer to Lamp Intensity Adjustment in the instrument manual.

Table of Contents for 525-nm parameters

ALUMINUM (0 to 1.00 mg/L)

For water and wastewater

Aluminon Method*



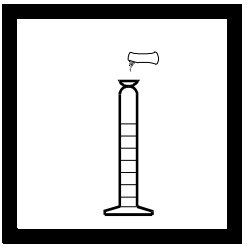
1. Fill a 50-mL graduated mixing cylinder to the 50-mL mark with sample.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage, following these steps.*

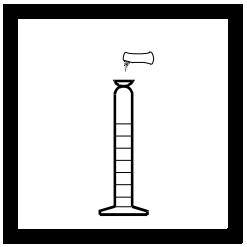
Note: *Rinse cylinder with 6N (1:1) hydrochloric acid and demineralized water before use to avoid errors due to contaminants adsorbed on the glass.*

Note: *The sample temperature must be between 20-25 °C (68-77 °F) for accurate results.*

Note: *For proof of accuracy, use a 0.4 mg/L aluminum standard solution (preparation given in Accuracy Check) in place of the sample.*



2. Add the contents of one Ascorbic Acid Powder Pillow. Stopper and invert several times to dissolve powder.

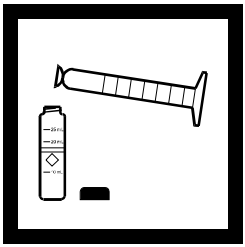


3. Add the contents of one AluVer 3 Aluminum Reagent Powder Pillow. Stopper and invert repeatedly for one minute to dissolve powder.

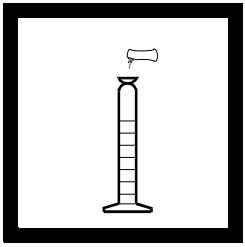
Note: *A red-orange color will develop if aluminum is present.*

Note: *Inconsistent results will be occur if any powder is undissolved.*

*Adapted from *Standard Methods for the Examination of Water and Wastewater.*

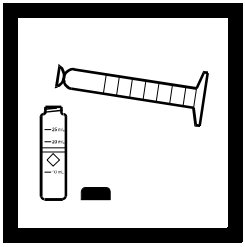


4. Fill a 25-mL cell to the 25-mL line with the mixture (the prepared sample). Cap.

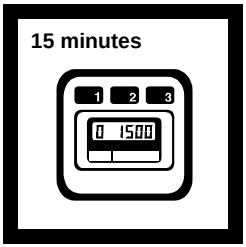


5. Add the contents of one Bleaching 3 Reagent Powder Pillow to the remaining 25 mL in the graduated mixing cylinder. Stopper and shake vigorously for 30 seconds (the blank).

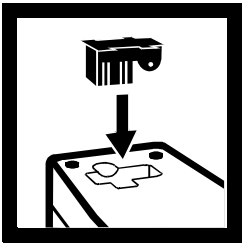
Note: *This solution should turn a light to medium orange upon bleaching. It will not become colorless.*



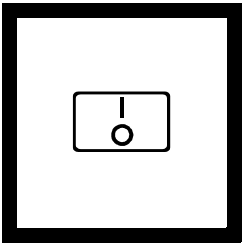
6. Pour the remaining 25 mL of bleached sample in the cylinder into a second 25-mL sample cell.



7. Begin timing a 15 minute period.

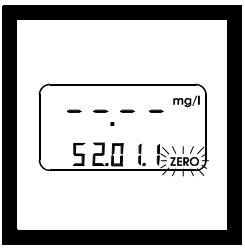


8. During the waiting period, install module
52.01
in a DR/700.

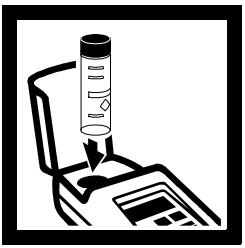


9. Press: **I/O**

The display will show
525 nm
and module number
52.01

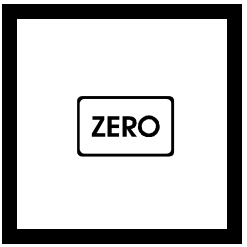


10. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
52.01.1



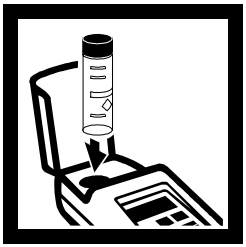
11. Within five minutes after the 15 minute waiting period, place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



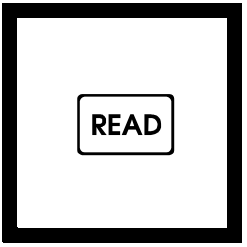
12. Press: **ZERO**

The display will count down to 0. Then the display will show
0.00 mg/L
and the zero prompt will turn off.



13. Immediately place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to 10-mL sample cells and proceed.*



14. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L aluminum.

Note: *Clean the graduated cylinder and sample cells with soap and brush immediately following the test.*

Note: *For most accurate results, determine a reagent blank (using demineralized water) for each lot of AluVer 3 Aluminum Reagent Powder Pillows. Subtract this value from each sample result obtained with this lot of reagent.*

SAMPLING AND SRORAGE

Collect samples in a cleaned glass or plastic container. To preserve the sample adjust the pH to 2 or less with nitric acid (about 1.5 mL per liter). Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the pH to 3.5 to 4.5 with 5.0 N Sodium Hydroxide. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off an Aluminum Voluette Ampule Standard Solution, 50 mg/L Al.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 50-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The aluminum concentration should increase 0.1 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 0.4-mg/L aluminum standard solution by pipetting 1.00 mL of Aluminum Standard Solution, 100 mg/L as Al^{3+}, into a 250-mL volumetric flask. Dilute to the mark with demineralized water. Prepare this solution daily. Perform the aluminum procedure as described above. The mg/L Al reading in Step 14 should be 0.4 mg/L Al.

Or, using the TenSette pipet, add 0.8 mL of solution from a Aluminum Voluette Ampule Standard Solution (50 mg/L as Al) into a 100-mL volumetric flask. Dilute to volume with demineralized water.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 0.50 mg/L Al concentration solutions, the standard deviation was ±0.009 mg/L Al.

Testing zero concentration samples, the limit of detection was

0.008 mg/L Al. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

The following do not interfere up to the indicated concentrations.

Alkalinity	1000 mg/L as $CaCO_3$
Iron	20 mg/L
Phosphate	50 mg/L

Interferences from higher alkalinity concentrations can be eliminated by the following pretreatment.

a) Add one drop of m-Nitrophenol Indicator Solution to the sample taken in Step 1. A yellow color indicates excessive alkalinity.

b) Add one drop of 5.25 N Sulfuric Acid Standard Solution. Stopper the cylinder. Invert to mix. If the yellow color persist, repeat until the sample changes to colorless. Continue with the test.

Polyphosphate interferes at all levels by causing negative errors and must not be present. Before running the test convert the polyphosphate to orthophosphate by acid hydrolysis as described under the phosphorus procedures.

Acidity interferes at greater than 300 mg/L as $CaCO_3$. Samples with greater than 300 mg/L acidity as $CaCO_3$ must be treated as follows:

a) Add one drop of m-Nitrophenol Indicator Solution to the sample taken in Step 1.

b) Add one drop of 5.0 N Sodium Hydroxide Standard Solution. Stopper the cylinder. Invert to mix. Repeat as often as necessary until the color changes from colorless to yellow.

c) Add one drop of 5.25 N Sulfuric Acid Standard Solution to change the solution from yellow back to colorless. Continue with the test.

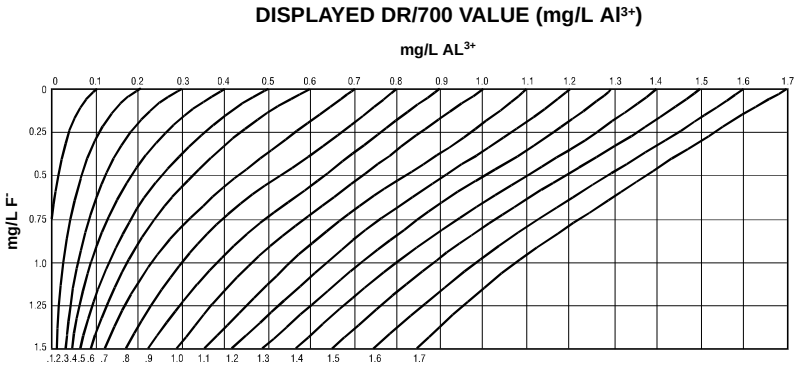
Calcium does not interfere.

Fluoride interferes at all levels by complexing with aluminum. The actual aluminum concentration can be determined using the Fluoride Interference Graph when the fluoride concentration is known. To use the Fluoride Interference Graph, select the vertical grid line along the top of the graph that represents the aluminum reading obtained in Step 14. Locate the point on the line where it intersects with the horizontal grid line that indicates how much fluoride is present in the sample. Extrapolate the true aluminum concentration by following the curved lines on either side of the intersect point down to the true aluminum concentration.

For example, if the aluminum test result was 0.7 mg/L Al and the fluoride present in the sample was 1 mg/L F^-, the point where the 0.7 grid line intersects with the 1 mg/L F^- grid line falls between the 1.2 and 1.3 mg/L Al curves. In this case, the true aluminum content would be 1.27 mg/L.

SUMMARY OF METHOD

Aluminon indicator combines with aluminum in the sample to form a red-orange color. The intensity of color is proportional to the aluminum concentration. Ascorbic acid is added to remove iron interference. The AluVer 3 Aluminum Reagent, packaged in powder form shows exceptional stability and is applicable for fresh water samples.



FLUORIDE INTERFERENCE GRAPH

REQUIRED REAGENTS

	Cat. No.
Aluminum Reagent Set (100 Tests)	22420-00
Includes: (4) 14290-68, (1) 14577-99, (1) 14294-99	

Description	Quantity Per Test	Unit	Cat. No.
AluVer 3 Aluminum Reagent Powder Pillow	1 pillow	25/pkg.	14290-68
Ascorbic Acid Powder Pillow	1 pillow	100/pkg.	14577-99
Bleaching 3 Reagent Powder Pillow	1 pillow	100/pkg.	14294-99

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinders, graduated mixing, 50 mL	1	each	1896-41
DR/700 Filter Module Number 52.01	1	each	46252-00

OPTIONAL REAGENTS

Aluminum Standard Solution, 100 mg/L	100 mL	14174-42
Aluminum Standard Solution, Voluette ampule, 50 mg/L as Al, 10 mL	6/pkg.	14792-10
Hydrochloric Acid Solution, 6N (1:1)	500 mL	884-49
m-Nitrophenol Indicator Solution, 10 g/L	100 mL	2476-32
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	50 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL	2450-32
Sodium Hydroxide Standard Solution, 5.0 N	59 mL	2450-26
Sulfuric Acid Standard Solution, 5.25 N	100 mL	2449-32
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Brush	each	690-00
Cap for 10- and 25-mL sample cells	12/pkg.	24018-12
Flask, volumetric, 250 mL	each	547-46
Flask, volumetric, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
pH Meter, EC10, portable	each	50050-00
Pipet Filler, 3-valve	each	12189-00
Pipet Filler, safety bulb	each	14651-00
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg.	21856-96
Pipet, volumetric, 1 mL	each	515-35
Sample Cell, 10-mL with screw cap	6/pkg.	24276-06
Sample Cell, 25-mL with screw cap	6/pkg.	24019-06
Thermometer, -20 to 105°C	each	1877-01

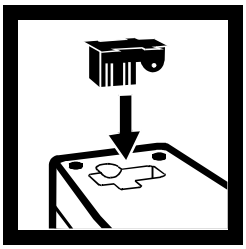
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

ALUMINUM (0 to 0.25 mg/L)

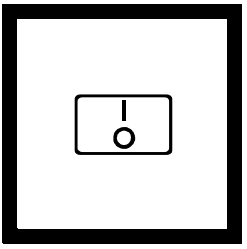
For water

Eriochrome Cyanine R Method*



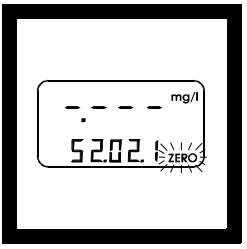
1. Install module **52.01** in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage, following these steps.*



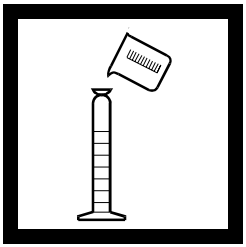
2. Press: **I/O**

The display will show **525 nm** and module **52.01**



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **52.02.1**

*Adapted from *Standard Methods for the Examination of Water and Wastewater.*

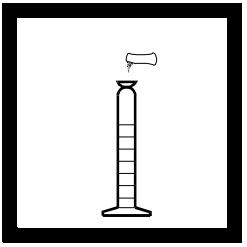


4. Fill a 50-mL graduated mixing cylinder to the 50-mL mark with sample.

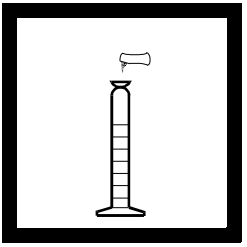
Note: *Rinse the cylinder with 6N (1:1) hydrochloric acid and demineralized water before use to avoid errors due to contaminants adsorbed on the glass.*

Note: *The sample temperature must be 20-25 °C (68-77 °F) for accurate results.*

Note: *For proof of accuracy, use a 0.1 mg/L aluminum standard solution (preparation given in Accuracy Check) in place of the sample.*

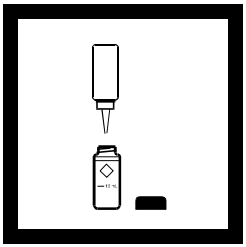


5. Add the contents of one ECR Reagent Powder Pillow. Stopper and invert several times to dissolve the powder; then wait 30 seconds.



6. Add the contents of one Hexamethylenetetramine Buffer Reagent Powder Pillow. Stopper and invert several times to dissolve powder.

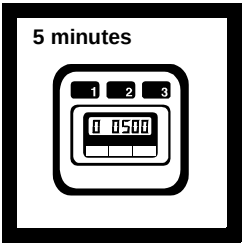
Note: *An orange to purple color will develop if aluminum is present.*



7. Put 1 drop of ECR Masking Reagent Solution into a 10-mL sample cell.

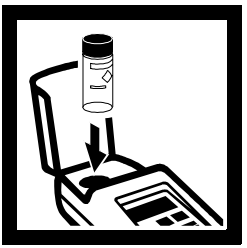
8. Fill the sample cell to the 10-mL mark with the solution from the cylinder (the blank). Cap and invert several times to mix.

Note: *The solution will start to turn yellow.*



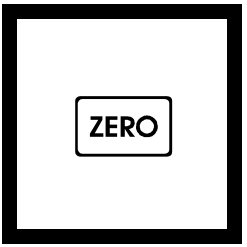
9. Wait 5 minutes.

10. Fill a second 10-mL cell to the 10-mL line with the remaining solution in the cylinder (the prepared sample). Cap.



11. Within five minutes after the 5-minute waiting period, place the blank in the cell holder.

Note: *In bright light, it may be necessary to close the cell compartment cover.*

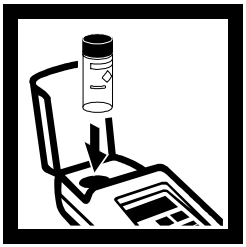


12. Press: **ZERO**

The display will count down to 0. Then the display will show

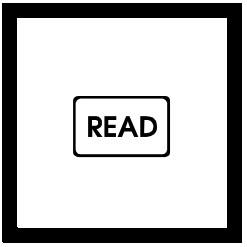
0.000 mg/L

and the zero prompt will turn off.



13. Place the prepared sample in the cell holder.

Note: In bright light, it may be necessary to close the cell compartment cover.



14. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L aluminum.

Note: *If fluoride is present, it must be measured and the actual value determined; see Table 2 in Interferences.*

SAMPLING AND STORAGE

Collect samples in a clean glass or plastic container. Preserve samples by adjusting the pH to 2 or less with nitric acid (about 1.5 mL per liter). Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the pH to 2.9 to 4.9 with 12.0 N Potassium Hydroxide Standard Solution and/or 1 N Potassium Hydroxide Solution. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Solution Method

Prepare a 0.100 mg/L aluminum standard solution by pipetting 1.00 mL of Aluminum Standard Solution, 100 mg/L as Al^{+3}, into a 1000-mL volumetric flask. Dilute to the mark with demineralized water. Prepare this solution daily. Perform the aluminum procedure as described above. The mg/L Al reading in Step 14 should be 0.10 mg/L Al.

Or, using the TenSette Pipet, add 0.2 mL of solution from an Aluminum Voluette Ampule Standard Solution (50 mg/L as Al) into a 100-mL volumetric flask. Dilute to volume with demineralized water. The mg/L Al reading in Step 14 should be 0.10 mg/L Al.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 0.200 mg/L Al concentration solutions, the standard deviation was ±0.0049 mg/L Al.

Testing zero concentration samples, the limit of detection was 0.0028 mg/L Al. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

Table 1 lists common interferences and the amount of interference that can be expected.

Table 1. Common Interferences with the Eriochrome Cyanine R Method

Constituent	Concentration	Error
Acidity	0-62 mg/L as $CaCO_3$	0%
Alkalinity	0-750 mg/L as $CaCO_3$	0%
Ca^{2+}	0-1000 mg/L as $CaCO_3$	0%
Cl^-	0-1000 mg/L	0%
Cr^{6+}	0.2 mg/L	-5% of reading
Cu^{2+}	2 mg/L	-5% of reading
Fe^{2+}	0-4 mg/L	+ mg/L Fe^{2+} x 0.0075
Fe^{3+}	0-4 mg/L	+ mg/L Fe^{3+} x 0.0075
F^-	*see Table 2*	
Hexametaphosphate	0.1 mg/L as PO_4^{3-}	-5% of reading
Mg^{2+}	0-1000 mg/L as $CaCO_3$	0%
Mn^{2+}	0-10 mg/L	0%
NO_2^-	0-5 mg/L	0%
NO_3^-	0-20 mg/L	0%
pH	2.9-4.9	0%
	7.5-11.5	0%
PO_4^{3-} (ortho)	4 mg/L	-5% of reading
SO_4^{2-}	0-1000 mg/L	0%
Zn^{2+}	0-10 mg/L	0%

A sample pH between about 4.9 and 7.5 causes dissolved aluminum to partially convert to colloidal and insoluble forms. This method measures much of that hard-to-detect aluminum without any pH adjusting pretreatment as is necessary in some other methods.

Polyphosphate interference can be reduced by converting polyphosphate to orthophosphate using the following steps:

a) Rinse a 50-mL mixing graduated cylinder and a 125-mL erlenmeyer flask containing a magnetic stir bar with 6 N Hydrochloric Acid. Rinse again with demineralized water. These rinses will remove any aluminum present.

Note: *Rinse two erlenmeyer flasks if a reagent blank is used; see Step b below.*

b) Measure 50 mL of demineralized water into the 125-mL erlenmeyer flask using the graduated cylinder. This is the reagent blank. Because of the test sensitivity, this step must be done only when any of the reagents

used in the following pretreatment are replaced, even if the new reagent has a matching lot number. When the pretreated sample has been analyzed, subtract the aluminum concentration of the reagent blank from the sample results.

c) Measure 50 mL of sample into the 125-mL erlenmeyer flask using the graduated cylinder. Use a small amount of demineralized water to rinse the cylinder contents into the flask.

d) Add 4.0 mL of 5.25 N Sulfuric Acid Solution.

e) Use a combination hot plate/stirrer to stir and boil the sample for at least 30 minutes. Add demineralized water as needed to maintain a sample volume of 20-40 mL. Do not boil dry.

f) Cool the solution to near room temperature.

g) Add 2 drops of Bromphenol Blue Indicator Solution.

h) Add 1.5 mL of 12.0 N Potassium Hydroxide Standard Solution using a calibrated plastic dropper. Swirl to mix. The solution color should be yellow or green, but not purple. If the color is purple, begin with Step a again using an additional 1 mL of Sulfuric Acid Solution in Step d.

i) While swirling the flask, add 1.0 N Potassium Hydroxide Solution, a drop at a time, until the solution turns a dirty green color.

j) Pour the solution into the graduated cylinder. Rinse the flask contents into the graduated cylinder with demineralized water to bring the total volume to 50 mL.

k) Use this solution in Step 4 of the ECR method.

Fluoride interference can be corrected by using Table 2.

Example: Is the fluoride concentration is known to be 1.00 mg/L F^- and the ECR method gives a DR/2000 reading of 0.060 mg/L aluminum, what is the true mg/L aluminum concentration?

Answer: 0.183 mg/L

Table 2. True aluminum concentration (mg/L) vs. DR/700 reading (mg/L) and fluoride concentration (mg/L) with the Eriochrome Cyanine R method

DR/700 Reading (mg/L)	Fluoride Concentration (mg/L)										
	0.00	0.20	0.40	0.60	0.80	1.00	1.20	1.40	1.60	1.80	2.00
0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
0.010	0.010	0.019	0.030	0.040	0.052	0.068	0.081	0.094	0.105	0.117	0.131
0.020	0.020	0.032	0.046	0.061	0.077	0.099	0.117	0.137	0.152	0.173	0.193
0.030	0.030	0.045	0.061	0.077	0.098	0.124	0.146	0.166	0.188	0.214	0.243
0.040	0.040	0.058	0.076	0.093	0.120	0.147	0.174	0.192	0.222		
0.050	0.050	0.068	0.087	0.109	0.135	0.165	0.188	0.217			
0.060	0.060	0.079	0.100	0.123	0.153	0.183	0.210	0.241			
0.070	0.070	0.090	0.113	0.137	0.168	0.201	0.230				
0.080	0.080	0.102	0.125	0.152	0.184	0.219					
0.090	0.090	0.113	0.138	0.166	0.200	0.237					
0.100	0.100	0.124	0.150	0.180	0.215						
0.120	0.120	0.146	0.176	0.209	0.246						
0.140	0.140	0.169	0.201	0.238							
0.160	0.160	0.191	0.226								
0.180	0.180	0.213									
0.200	0.200	0.235									
0.220	0.220										
0.240	0.240										

True Aluminum Concentration (mg/L) Al

Note: *Intermediate values can be found by interpolation. Do not use correction graphs or charts found in other publications.*

SUMMARY OF METHOD

Eriochrome Cyanine R combines with aluminum in a sample to produce an orange-red color. The intensity of color is proportional to the aluminum concentration.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
ECR Reagent Powder	1 pillow	25/pkg.	23802-68
Hexamethylenetetramine Buffer Reagent	1 pillow	25/pkg.	1878-68
ECR Masking Reagent Solution	2 drops	29 mL	23801-23

REQUIRED APPARATUS

Clippers for opening powder pillows	1	each	968-00
Cylinder, 50 mL, mixing graduated	1	each	1896-41
DR/700 Filter Module Number 52.01	1	each	46252-00

OPTIONAL REAGENTS

Aluminum Standard Solution, 100 mg/L	100 mL	14174-42
Aluminum Standard Solution, Voluette ampule, 50 mg/L as Al, 10 mL	16/pkg.	14792-10
Bromphenol Blue Indicator Solution	100 mL	14552-32
Hydrochloric Acid Solution, 6 N (1:1)	500 mL	884-49
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Potassium Hydroxide Solution, 1 N	59 mL	23144-26
Potassium Hydroxide Standard Solution, 12.0 N	100 mL	230-32
Potassium Hydroxide Standard Solution, 12.0 N	500 mL	230-49
Sulfuric Acid Standard Solution, 5.25 N	100 mL	2449-32
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Brush	each	690-00
Cap for 10- and 25-mL sample cells	12/pkg.	24018-12
Flask, erlenmeyer, glass, 125 mL	each	505-43
Flask, volumetric, 100 mL	each	14574-42
Flask, volumetric, 1000 mL	each	14574-53
Hot Plate, Stirrer, 120 V	each	23442-00
Hot Plate, Stirrer, 240 V	each	23442-02
Pad, cooling, 4" x 4"	each	18376-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10. portable	each	50050-00
Pipet Filler, safety bulb	each	14651-00
Pipet, serological, 2 mL.	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg.	21856-96
Pipet, volumetric, 1 mL	each	14515-35
Stir Bar, Octagonal, 28.6 x 7.9 mm	each	20953-52
Thermometer, -20 to 105 °C	each	1877-01

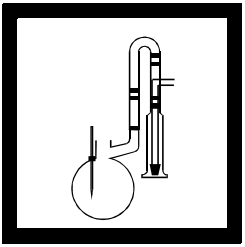
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

ARSENIC (0 to 0.200 mg/L)

For water, wastewater and seawater

Silver Diethyldithiocarbamate Method†*; USEPA accepted for reporting (distillation is required)**

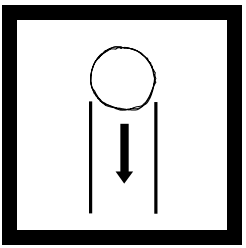


1. Prepare the distillation apparatus for arsenic recovery. Place it under a fume hood to vent toxic fumes.

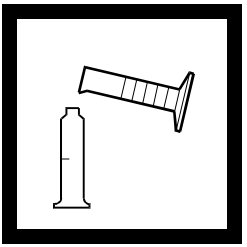
Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

Note: *See the Hach Distillation Manual for assembly instructions*

Note: *Due to potential variation between lots of arsenic absorber solution, it is necessary to perform a new calibration for each lot of this reagent. Prepare and store the calibration as directed under Using User-Programmed Method (paragraph 3.2.4) in the DR/700 Instrument Manual. Then use the following procedure.*



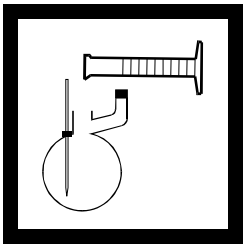
2. Dampen a cotton ball with 10% Lead Acetate Solution. Place it in the gas scrubber. Be certain the cotton seals against the glass.



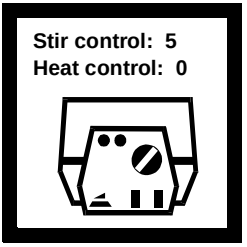
3. Measure 25 mL of prepared arsenic absorber solution into the cylinder/gas bubbler assembly with a graduated cylinder. Attach it to the distillation apparatus.

Note: *Prepare the arsenic absorber solution as directed under Reagent Preparation following these steps.*

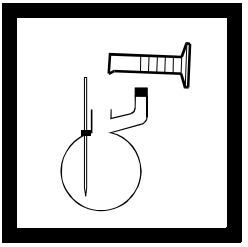
†User calibration required; range is approximate.
*Adapted from *Standard Methods for the Examination of Water and Wastewater*
**Procedure is equivalent to USEPA method 206.4 for wastewater and Standard Method 3500-As for drinking water



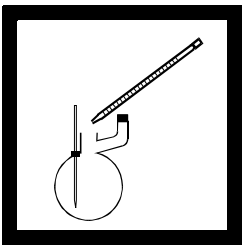
4. Measure 250 mL of sample into the distillation flask using a graduated cylinder.



5. Turn on the power switch. Set the stir control to 5. Set the heat control to 0.

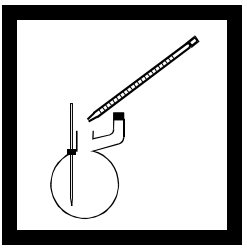


6. Measure 25 mL of hydrochloric acid, ACS, into the flask using a graduated cylinder.



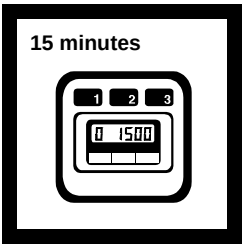
7. Measure 1 mL of Stannous Chloride Solution into the flask.

Note: *Use a serologic pipet to measure the solution.*

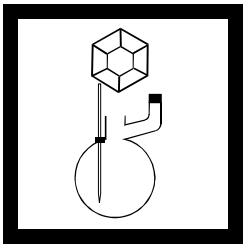


8. Add 3 mL of 20% Potassium Iodide Solution to the flask. Cap.

Note: *Use a serologic pipet to measure the solution.*

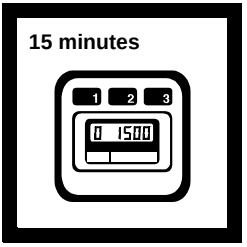


9. Wait 15 minutes.

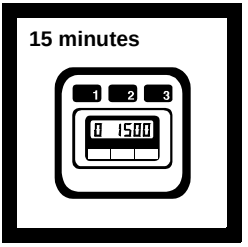


10. Add 6.0 g of 20-mesh zinc to the flask. Cap immediately.

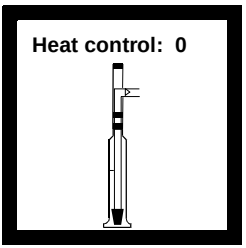
Note: *Use an analytical balance to weigh the zinc metal.*



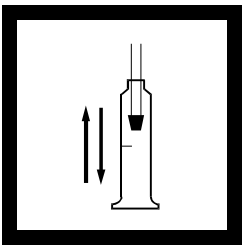
11. Set the heat control to 3. Wait 15 minutes.



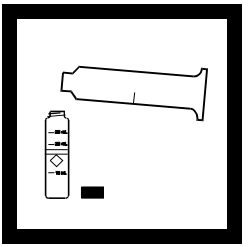
12. Set the heat control to 1. Wait 15 minutes.



13. Turn the heat off. Remove the cylinder/gas bubbler assembly as a unit.

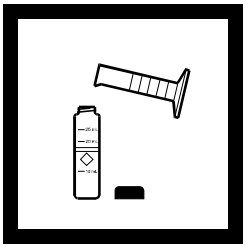


14. Rinse the gas bubbler by moving it up and down in the arsenic absorber solution.

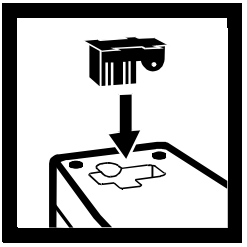


15. Fill a 25-mL cell to the 25-mL line with the reacted arsenic absorber solution (the prepared sample). Cap.

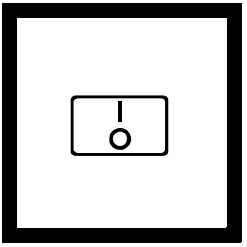
Note: *If the solution volume is less than 25 mL, add pyridine to bring the volume to exactly the 25-mL mark. Do this only in a fume hood. Cap and invert several times to mix.*



16. Fill a 25-mL cell to the 25-mL line with unreacted arsenic absorber solution (the blank). Cap the cell.

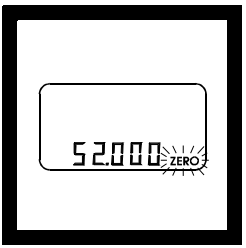


17. Install module
52.01
in a DR/700.



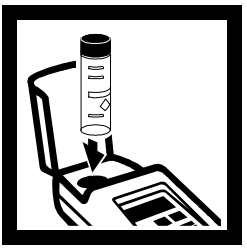
18. Press: **I/O**

The display will show
525 nm
and module number
52.01



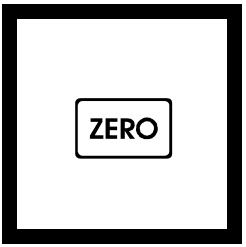
19. After 2 seconds, the display will show a program number, concentration units, and the zero prompt. Press the **PROGRAM** key until the display shows program number
52.000

The upper display will show the S1 concentration.



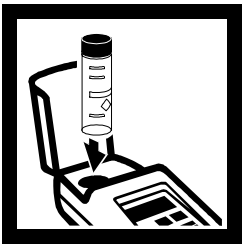
20. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



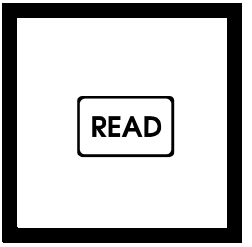
21. Press: **ZERO**

The display will count down to 0. Then the display will show the concentration of the standard and the zero and standard prompts will turn off.



22. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



23. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L arsenic (As).

REAGENT PREPARATION

Prepare the arsenic absorber solution as follows:

1. Weigh 1.00 g of silver diethyldithiocarbamate on an analytical balance.

2. Transfer the powder to a 200-mL volumetric flask. Dilute to volume with pyridine. (Use pyridine only in a fume hood.)

3. Mix well to dissolve. Store the reagent, tightly sealed, in an amber bottle. The reagent is stable for one month if stored in this manner. Larger volumes of reagent can be prepared if the reagent is used within one month.

CALIBRATION

Perform a new calibration for each lot of arsenic absorber solution prepared as follows:

a) Prepare a 10.0-mg/L arsenic working standard by pipetting 1.00 mL of Arsenic Standard Solution, 1000 mg/L As, into a 100-mL volumetric flask. Dilute to volume with demineralized water. Cap and invert 10 times to mix.

b) Prepare a 0.20 -mg/L arsenic working solution by using a 5-mL volumetric pipet to put 5.0 mL of working standard into a 250-mL volumetric flask. Dilute to volume with demineralized water. Cap the flask and invert 10 times to mix.

c) Perform Steps 1-18 of the arsenic procedure using the 0.20 mg/L arsenic standard.

d) Perform the instructions in the "Calibration Using Two Prepared Standards" section of the DR/700 Instrument Manual (paragraph 3.2.4.1).

For Standard 1, use the blank from the arsenic procedure and make the display read 00.00 mg/L.

For Standard 2, use the prepared sample from the arsenic procedure and make the display read 00.20 mg/L.

SAMPLING AND STORAGE

Collect samples in acid washed glass or plastic bottles. 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. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

INTERFERENCES

Antimony salts may interfere with color development.

SUMMARY OF METHOD

Arsenic is reduced to arsine gas by a mixture of zinc, stannous chloride, potassium iodide and hydrochloric acid in a specially equipped distillation apparatus. The arsine is passed through a scrubber containing cotton saturated with lead acetate and then into an absorber tube containing silver diethyldithiocarbamate in pyridine. The arsenic reacts to form a red complex which is read colorimetrically. This procedure requires a manual calibration.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Arsenic Standard Solution, 1000 mg/L As	varies	100 mL	14571-42
Hydrochloric Acid, ACS	25 mL	500 mL	134-49
Lead Acetate Solution, 10%	1 mL	100 mL	14580-42
Potassium Iodide Solution, 20%	3 mL	105 mL	14568-42
Pyridine	50 mL	500 mL	14469-49
Silver Diethyldithiocarbamate	1 g	25 g	14476-24
Stannous Chloride Solution	1 mL	100 mL	14569-42
Zinc, 20-mesh, ACS	6 g	454 g	795-01

REQUIRED APPARATUS

Balance, analytical	1	each	22310-00
Balls, cotton	1	100/pkg	2572-01
Boat, weighing	2	500/pkg	21790-00
Bottle, amber, 237 mL	1	6/pkg	7144-41
Cap, polypropylene	1	6/pkg	21667-06
Cylinder, graduated, 25 mL	2	each	508-40
Cylinder, graduated, 250 mL	1	each	508-46
Distillation Apparatus Arsenic Accessories	1	set	22654-00
Distillation Apparatus General Purpose Accessories	1	set	22653-00
DR/700 Module Number 52.01	1	each	46252-00
Flask, volumetric, 100 mL	1	each	14574-42
Flask, volumetric, 200 mL	1	each	14574-45
Flask, volumetric, 250 mL	4	each	14574-46
Pipet Filler, safety bulb	1	each	14651-00
Pipet, serological, 5 mL	2	each	532-37
Pipet, serological, 1 mL	1	each	532-35
Pipet, volumetric, 1.00 mL	1	each	14515-35
Pipet, volumetric, 5.00 mL	1	each	14515-37

Select one based on available voltage:

Distillation Apparatus Heater, 115 Vac, 60 Hz	each	22744-00
Distillation Apparatus Heater, 230 Vac, 50 Hz	each	22744-02

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Hydrochloric Acid, ACS	2.8 kg	134-06
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Pyridine, ACS	4 L	14469-17
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells	12/pkg	24018-12
pH Meter, EC10, portable	each	50050-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet Filler, 3-valve	each	12189-00
Pipet, serological, 2 mL	each	532-36
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

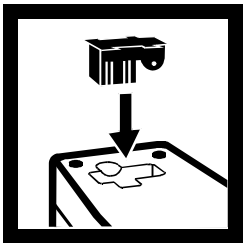
In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

BROMINE (0 to 8.00 mg/L)

For water, wastewater and seawater

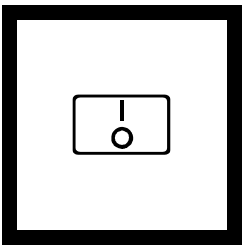
DPD Method* (Powder Pillows or AccuVac Ampuls)

USING POWDER PILLOWS



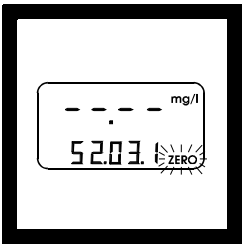
1. Install module
52.01
in a DR/700.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



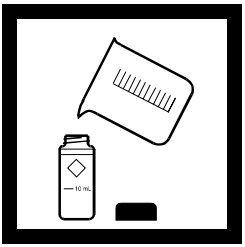
2. Press: **I/O**

The display will show
525 nm
and module number
52.01



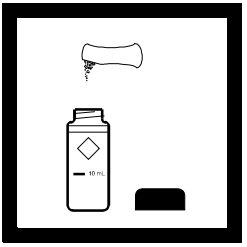
3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
52.03.1

*Adapted from *Standard Methods for the Examination of Water and Wastewater.*



4. Fill a 10-mL sample cell to the 10-mL line with sample.

Note: *A 25-mL sample can be tested by using 25-mL sample cells and optional reagents.*

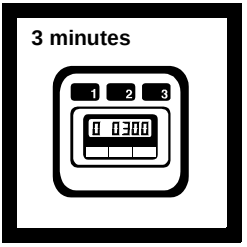


5. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Cap and shake the cell for 20 seconds.

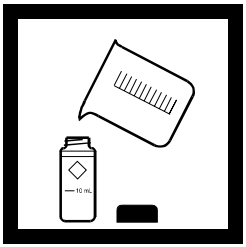
Note: *A pink color will develop if bromine is present.*

Note: *Accuracy is not affected by undissolved powder.*

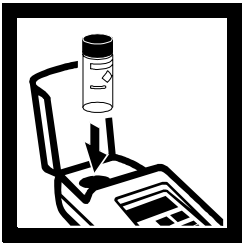
Note: *Shaking the cell dissipates bubbles which may form in samples containing dissolved gases.*



6. Wait 3 minutes.

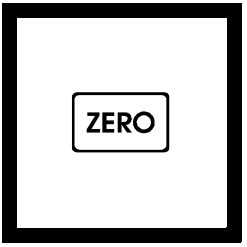


7. Fill a 10-mL cell to the 10-mL line with sample (the blank). Cap.



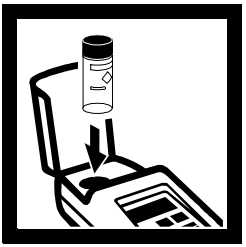
8. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



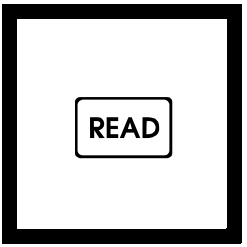
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



10. Place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*

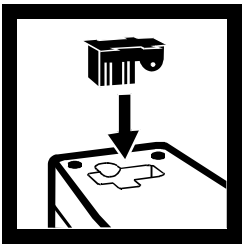


11. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L bromine (Br_2).

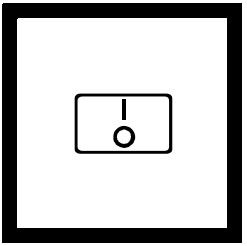
Note: *If sample temporarily turns yellow after reagent addition, or flashes the upper range limit, dilute a fresh sample and repeat the test. A slight loss of bromine may occur because of the dilution. Multiply the result by the appropriate dilution factor; see Sample Dilution Techniques (Section 1).*

USING ACCUVAC AMPULS



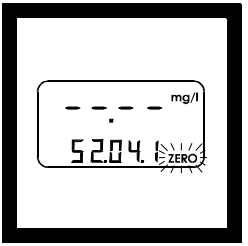
1. Install module **52.01** in a DR/700.

Note: *Sample must be analyzed immediately and cannot be preserved for later analysis.*

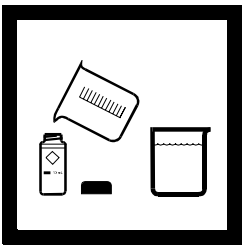


2. Press: **I/O**

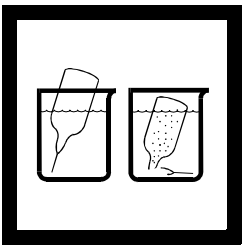
The display will show **525 nm** and module number **52.01**



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **52.04.1**

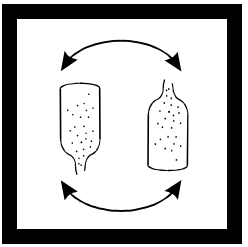


4. Fill a cell with 10 mL of sample (the blank). Cap. Collect at least 40 mL of sample in a 50-mL beaker.



5. Fill a DPD Total Chlorine Reagent AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*

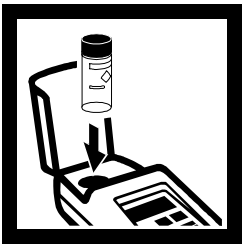


6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will develop if bromine is present.*

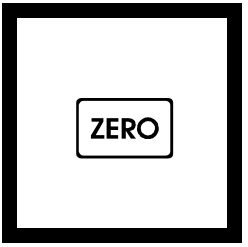
Note: *Accuracy is not affected by undissolved powder.*

7. Wait 3 minutes.



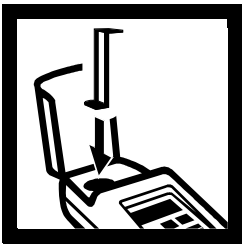
8. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*

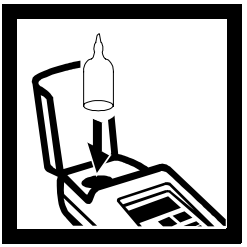


9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

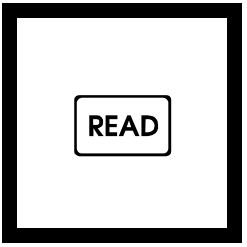


10. Insert the AccuVac Vial Adapter into the cell holder.



11. Within 3 minutes after the 3-minute period, place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L bromine (Br_2).

Note: *If sample temporarily turns yellow after reagent addition, or flashes the upper range limit, dilute a fresh sample and repeat the test. A slight loss of bromine may occur because of the dilution. Multiply the result by the appropriate dilution factor; see Sample Dilution Techniques (Section 1).*

ACCURACY CHECK

Standard Additions Method

a) Snap the top off a Chlorine Voluette Ampule Standard Solution.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 ml of standard to three 25-mL samples. Swirl gently to mix. (For AccuVac Ampuls, use 50-mL beakers.)

c) Analyze each sample as described above. Each 0.1 mL of standard will cause an incremental increase in bromine, the exact value of which depends on the chlorine concentration in the Voluette. Check the certificate enclosed with the Voluettes for the incremental chlorine value; then multiply by 2.25 to obtain the value for bromine.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 1.00 mg/L Cl_2 (equivalent to 2.25 mg/L Br_2) concentration solutions, the standard deviation was ±0.005 mg/L CL_2 (equivalent to 0.01 mg/L Br_2).

Testing zero concentration samples, the limit of detection was 0.010 mg/L Cl_2 (equivalent to 0.02 mg/L Br_2). The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

Using two representative lots of AccuVacs, the standard deviation was ±0.005 mg/L Cl_2 (equivalent to 0.01 mg/L Br_2) and the limit of detection was 0.015 mg/L Cl_2 (equivalent to 0.03 mg/L Br_2).

INTERFERENCES

Samples containing more than 300 mg/L alkalinity or 150 mg/L acidity as $CaCO_3$ may not develop the full amount of color, or it may instantly fade. Neutralize these samples to a pH of 6 to 7 with 1 N sulfuric acid or 1 N sodium hydroxide. Determine the amount required on a separate 25 mL sample. Add the same amount to the sample to be tested. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

Chlorine, iodine, ozone and oxidized forms of manganese and chromium also may react and show as bromine. Compensate for the effects of manganese (Mn^{4+}) or chromium (Cr^{6+}), by adjusting the pH to 6 to 7 as described above. Add 3 drops of 30-g/L potassium iodide to 25 mL of sample, mix and wait one minute. Add 3 drops of 5 g/L Sodium Arsenite and mix. Analyze this sample as described above. (If chromium is present, allow exactly the same reaction period with DPD for both analyses.) Subtract the result of this test from the original analysis to obtain the accurate bromine result.

DPD Total Chlorine Reagent Powder Pillows and AccuVac Ampuls contain a buffer formulation which will withstand high levels (>1000 mg/L) of hardness without interference.

SUMMARY OF METHOD

Bromine reacts with DPD (N,N-diethyl-p-phenylenediamine) to form a red color which is proportional to the total bromine concentration.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows, 10 mL	1 pillow	100/pkg	21056-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

DPD Total Chlorine Reagent AccuVac Ampuls	1 ampul	25/pkg	25030-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 52.01	1	each	46252-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac Vial	1	each	46025-00
Adapter, AccuVac Vial, DR/700	1	each	46025-00
Beaker, 50 mL	1	each	500-41
DR/700 Filter Module Number 52.01	1	each	46252-00

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Chlorine Standard Solution, Voluette ampule, 50-75 mg/L, 10 mL	16/pkg	14268-10
DPD Total Chlorine Powder Pillows, 25 mL	100/pkg	14064-99
Potassium Iodide Solution, 30 g/L	100 mL* MDB	343-32
Sodium Arsenite, 5 g/L	100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1 N	100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1 N	100 mL* MDB	1270-32
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, 25 mL, poly	each	1081-40
pH Meter, EC10, portable	each	50050-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Sample Cell, 10-ml with screw cap	6/pkg	24276-06
Sample Cell, 25-ml with screw cap	6/pkg	24019-06

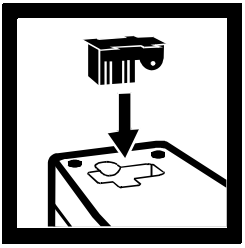
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

CADMIUM (0 to 80 µg/L)

For water and wastewater

Dithizone Method*

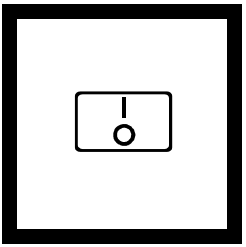


1. Install module
52.01
in a DR/700

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*

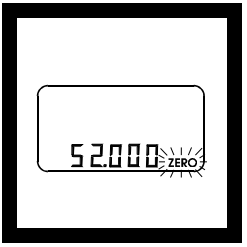
Note: *Total cadmium determination requires a prior digestion; use on the three procedures given in Digestion (Section I).*

Note: *The DR/700 must be calibrated before sample measurement. See Calibration following these steps.*



2. Press: **I/O**

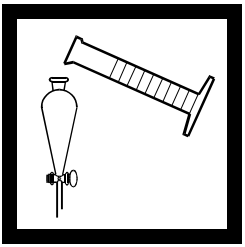
The display will show
525 nm
and module
52.01



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. Press the **PROGRAM** key until the lower display shows program number
52.000

The upper display will show the S1 concentration.

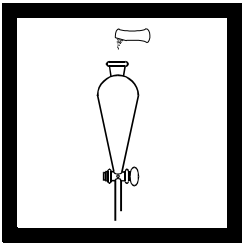
*Adapted from *Standard Methods for the Examination of Water and Wastewater.*



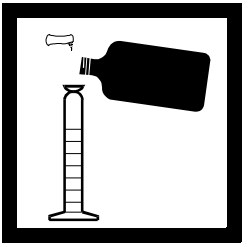
4. Fill a 250-mL graduated cylinder to the 250-mL mark with sample. Pour the sample into a 500-mL separatory funnel.

Note: *Clean all glassware with Nitric Acid Solution, 1:1. Rinse with demineralized water.*

Note: *Cloudy and turbid samples may require filtering before running the test. Report results as µg/L soluble cadmium. Use a glass membrane type filter to avoid loss of cadmium by adsorption on filter paper.*

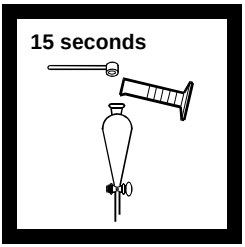


5. Add the contents of one Buffer Powder Pillow, citrate type for heavy metals. Stopper and shake to dissolve.



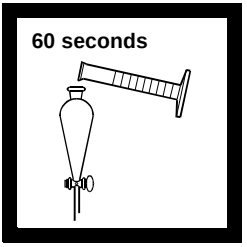
6. Add 30 mL of chloroform to a 50-mL graduated mixing cylinder. Add the contents of one DithiVer Metals Reagent Powder Pillow. Stopper the cylinder and invert several times to mix. This is DithiVer Solution.

Note: *Use adequate ventilation. The DithiVer powder will not dissolve completely in the chloroform. For further notes, see DithiVer Solution Preparation and Storage following these steps.*

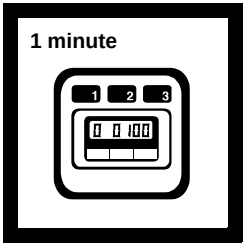


7. Add 20 mL of 50% Sodium Hydroxide Solution and the a 0.1-g scoop of potassium cyanide to the funnel. Shake vigorously for 15 seconds. Remove the stopper and let stand for one minute.

Note: *Spilled reagent will affect test accuracy and is hazardous to skin and other materials.*

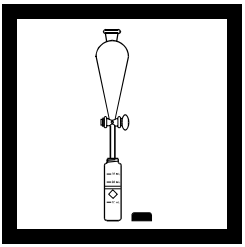


8. Add 30 mL of DithiVer Solution to the 500-mL separatory funnel. Stopper, invert, and open the stopcock slowly to vent. Close the stopcock and shake the funnel once or twice; vent again. Close the stopcock and shake the funnel vigorously for 60 seconds.



9. Let the funnel stand undisturbed for about 1 minute.

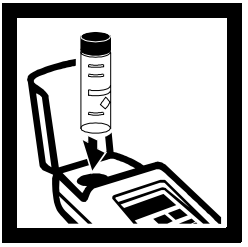
Note: *The bottom layer (chloroform) will be pink if cadmium is present.*



10. Insert a pea-sized cotton plug into the delivery tube and slowly drain the bottom layer into a dry 25-mL sample cell (the prepared sample). Cap.

Note: *The cadmium-dithizone complex is stable for hours if the sample cell is kept tightly capped and out of direct sunlight.*

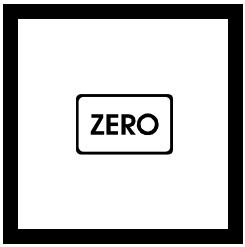
11. Fill a 25-mL cell to the 25-mL line with chloroform (the blank). Cap.



12. Place the blank in the cell holder.

Note: *If the display is blank, repeat Steps 2 and 3.*

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to 10-mL sample cells and proceed.*

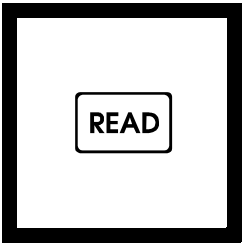


13. Press: **ZERO**

The display will count down to 0. Then the display will show 0 mg/L and the zero and S1 prompts will turn off.

14. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to 10-mL sample cells and proceed.*



15. Press: **READ**

The display will count down to 0. Then the display will show the results in µg/L cadmium (Cd).

Note: *For best results, a reagent blank should be determined for each new lot of DithiVer Metals Reagent Powder Pillows. Use demineralized water in place of the sample in Step 4 and subtract this amount from the test results obtained in Step 15.*

SAMPLING AND STORAGE

Collect samples in an acid-cleaned glass or plastic container. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Before analysis, adjust the pH to 2.5 with 5.0 N sodium hydroxide. Correct the test result for volume additions; see Sampling and Storage, Volume Additions (Section I) for more information.

DITHIVER SOLUTION PREPARATION AND STORAGE

Store DithiVer powder pillows away from light and heat. A convenient way to prepare DithiVer solution is to add the contents of 16 DithiVer Metals Reagent Powder Pillows to a 500-mL bottle of chloroform and invert several times until well mixed (carrier powder may not dissolve). Store dithizone solution in an amber glass bottle. This solution is stable for 24 hours for cadmium testing. Do not use it to run lead tests.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Cadmium Voluette Ampule Standard Solution, 25 mg/L Cd.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to three 250-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The cadmium concentration should increase 10 µg cadmium/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

CALIBRATION

The DR/700 must be calibrated in order to use the Cadmium Dithizone test procedure. To obtain most accurate results, perform a new calibration when a new lot of DithiVer Metals Reagent Powder Pillows is used.

To calibrate:

a) Prepare a 5.0 mg/L cadmium solution by pipetting 5.0 mL of 100-mg/L Cadmium Standard Solution into a 100-mL volumetric flask.

b) Dilute the solution in the flask to the mark with demineralized water.

c) Cap the flask and invert 10 times to mix. Make this solution fresh each day it is needed.

d) Pipet 4.0 mL of the 5.0-mg/L cadmium solution into a 500-mL separatory funnel.

e) Add 246 mL of demineralized water to the funnel to make an 80-µg/L cadmium solution.

f) Perform Steps 1-11 if the Cadmium Dithizone procedure, using the 80-µg/L solution in Step 4.

g) Perform paragraph 3.2.4.1, Calibration Using Two Prepared Standards, in the DR/700 Instrument Manual.

h) For Standard 1, use the blank from the cadmium procedure and make the display show 0000 µg/L.

i) For Standard 2, use the prepared sample from the cadmium procedure and make the display show 0080 µg/L.

INTERFERENCES

The following do not interfere:

Aluminum	Lead
Antimony	Magnesium
Arsenic	Manganese
Calcium	Nickel
Chromium	Tin
Cobalt	Zinc
Iron	

The following interfere causing high results when present in concentrations exceeding those listed below:

Copper	2 mg/L
Bismuth	80 mg/L
Mercury	all levels
Silver	2 mg/L

Eliminate interference from these metals by the following treatment, beginning after Step 6.

a) Measure about 5 mL of the DithiVer solution into the separatory funnel. Stopper the funnel, invert and open the stopcock to vent. Close the stopcock and shake the solution vigorously for 15 seconds. Allow the funnel to stand undisturbed until the layers separate (about 30 seconds). A yellow, red, or bronze color in the bottom (chloroform) layer confirms the presence of interfering metals. Draw off and discard the bottom (chloroform) layer.

b) Repeat extraction with fresh 5 mL portions of the DithiVer solution (discarding the bottom layer each time) until the bottom layer shows a pure dark green color for three successive extracts. Extractions can be repeated several times without appreciably affecting the amount of cadmium in the sample.

c) Extract the solution with several 2 or 3 mL portions of pure chloroform to remove any remaining DithiVer, again discarding the bottom layer each time.

d) Continue with Step 7.

e) In Step 8, substitute 28.5 mL of DithiVer solution for the 30 mL.

f) Continue with Step 9.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

WASTE DISPOSAL

Dispose of cyanide-containing wastes following the steps below:

a) Use good ventilation or a fume hood.

b) Add the waste while stirring to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochloride (household bleach).

c) Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.

d) Flush the solution down the drain with a large excess of water.

SUMMARY OF METHOD

The dithizone method is designed for the determination of cadmium in water and wastewater. The DithiVer metals reagent is a stable powder form of dithizone. Cadmium ions in basic solution react with dithizone to form a pink to red cadmium-dithizonate complex, which is extracted with chloroform.

REQUIRED REAGENTS

	Cat No.
Cadmium Reagent Set (68 Tests)	22422-00
Includes: (1) 14202-99, (1) 14458-17, (4) 12616-68, (1) 767-14, (4) 2180-49	

REQUIRED REAGENTS (continued)

Description	Quantity Per Test	Unit	Cat. No.
Buffer Powder Pillows, citrate for heavy metals	1 pillow	100/pkg	14202-99
Cadmium Standard Solution, 100 mg/L Cd	5 mL	100 mL	14024-42
Chloroform, ACS	30 mL	4 L	14458-17
DithiVer Metals Reagent Powder Pillows	1 pillow	25/pkg	12616-68
Potassium Cyanide, ACS	0.1 g	113 g	767-14
Sodium Hydroxide Solution, 50%	20 mL	500 mL	2180-49
Water, demineralized	350 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cotton Balls, absorbent	1	100/pkg	2572-01
Cylinder, graduated, 25 mL	1	each	508-40
Cylinder, graduated, 250 mL	1	each	508-46
Cylinder, mixing, graduated, 50 mL	1	each	1896-41
DR/700 Module Number 52.01	1	each	46252-00
Flask, volumetric, 100 mL	1	each	14547-42
Funnel, separatory, 500 mL	1	each	520-49
Pipet Filler, safety bulb	1	each	14651-00
Pipet, volumetric, 4.00 mL	1	each	14515-04
Pipet, volumetric, 5.00 mL	1	each	14515-37
Spoon, measuring, 0.1 g	1	each	511-00
Support Ring, 4"	1	each	580-01
Support Stand, 5" X 8"	1	each	563-00

OPTIONAL REAGENTS

Cadmium Standard Solution, Voluette ampule, 25 mg/L Cd, 10 mL	16/pkg	14261-10
Chloroform, ACS	500 mL	14458-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Nitric Acid, ACS	500 mL	152-59
Sodium Hydroxide Standard Solution, 5.0 N	59 mL	2450-26

OPTIONAL REAGENTS (continued)

Description	Unit	Cat. No.
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, 5 mL	each	508-37
Filter Discs, glass, 47 mm	100/pkg	2530-00
Flask, erlenmeyer, 500 mL	each	505-49
Flask, filtering, 500 mL	each	546-49
Flask, volumetric, 100 mL	each	547-42
Hot Plate, 3 1/2" diameter, 120 Vac	each	12067-01
Hot Plate, 3 1/2" diameter, 240 Vac	each	12067-02
Membrane Filter Holder, graduated, 500 mL, for 47 mm filter	each	2340-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet Filler, 3-valve	each	12189-00
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 2.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Tongs, crucible	each	569-00

For Technical Assistance, Prices and Ordering

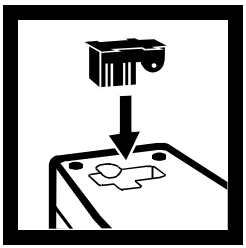
In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

CHLORINE, FREE (0 to 2.00 mg/L)

For water, wastewater and seawater

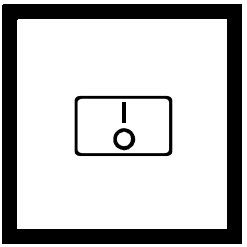
DPD Method* (Powder Pillows or AccuVac Ampuls), USEPA accepted for reporting**

USING POWDER PILLOWS



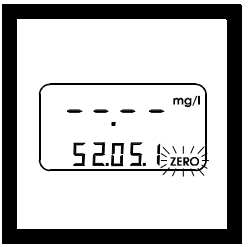
1. Install module
52.01
in a DR/700.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

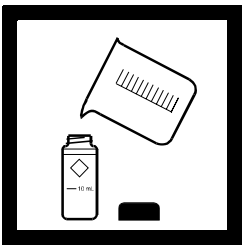


2. Press: **I/O**

The display will show
525 nm
and module number
52.01

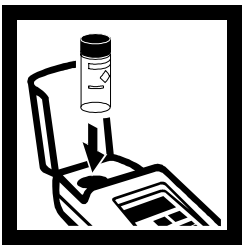


3. After 2 seconds, the display will show a program number, concentration units, and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
52.05.1



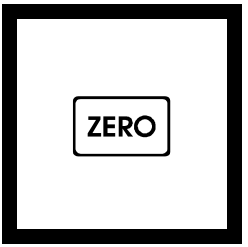
4. Fill a 10-mL cell to the 10-mL line with sample (the blank). Cap.

Note: *A 25-mL sample can be tested by using 25-mL cells and optional reagents.*



5. Place the blank in the cell holder.

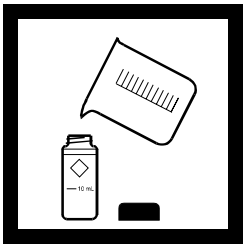
Note: *In bright light it may be necessary to close the cell compartment cover.*



6. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

*Adapted from *Standard Methods for the Examination of Water and Wastewater*
**Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-Cl G for drinking water.



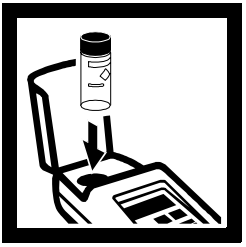
7. Fill a 10-mL cell to the 10-mL line with sample.

8. Add the contents of one DPD Free Chlorine Powder Pillow to the cell (the prepared sample). Cap the cell and shake for 20 seconds.

Note: *A pink color will develop if free chlorine is present.*

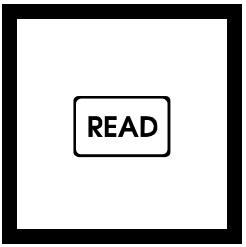
Note: *Accuracy is not affected by undissolved powder.*

Note: *Shaking the cell dissipates bubbles which may form in samples containing dissolved gases.*



9. Immediately (within 1 minute of reagent addition) place the prepared sample into the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*

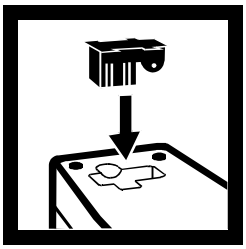


10. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L free chlorine (Cl_2)

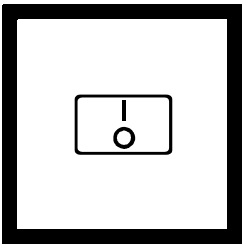
Note: *If the sample temporarily turns yellow after reagent addition, or flashes the upper range limit, dilute a fresh sample and repeat the test. A slight loss of chlorine may occur because of the dilution. Multiply the result by the appropriate dilution factor; see Sample Dilution Techniques (Section I).*

USING ACCUVAC AMPULS



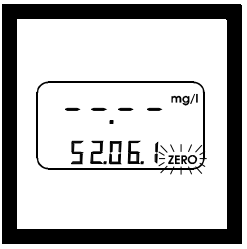
1. Install module
52.01
in a DR/700.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

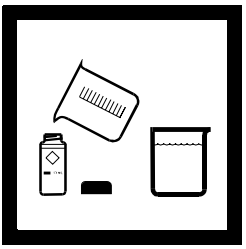


2. Press: **I/O**

The display will show
525 nm
and module number
52.01

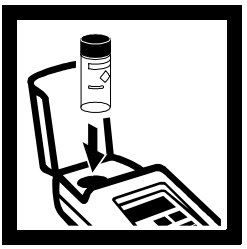


3. After 2 seconds, the display will show a program number, concentration units, and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
52.06.1



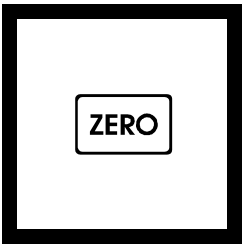
4. Fill a cell with 10 mL of sample (the blank). Cap. Collect at least 40 mL of sample in a 50-mL beaker.

Note: *In bright light it may be necessary to close the cell compartment cover.*



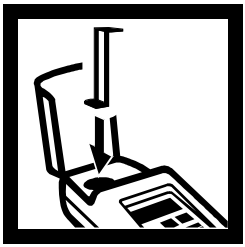
5. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*

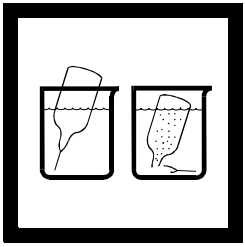


6. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

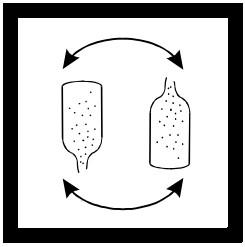


7. Insert the AccuVac Vial Adapter into the cell holder.



8. Fill a DPD Free Chlorine Reagent AccuVac Ampul with sample.

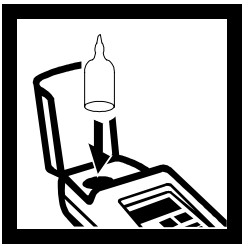
Note: *Keep the tip immersed while the ampul fills completely.*



9. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

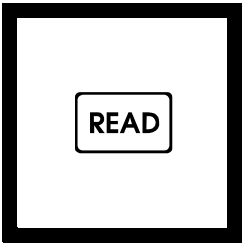
Note: *A pink color will develop if free chlorine is present.*

Note: *Accuracy is not affected by undissolved powder.*



10. Immediately (within 1 minute of sample addition) place the AccuVac ampul into the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



11. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L free chlorine (Cl_2).

Note: *If the sample temporarily turns yellow after reagent addition, or flashes the upper range limit, dilute a fresh sample and repeat the test. A slight loss of chlorine may occur because of the dilution. Multiply the result by the appropriate dilution factor; see Sample Dilution Techniques (Section I).*

ACCURACY CHECK

Standard Additions Method

a) Snap the top off a Chlorine Voluette Ampule Standard Solution.
b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to three 25-mL samples. Swirl gently to mix. (For AccuVac ampuls, use 50-mL beakers.)

c) Analyze each sample as described above. Each 0.1 mL of standard will cause an incremental increase in chlorine, the exact value of which depends on the concentration in the Voluette. Check the certificate enclosed with the Voluettes for this value.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 1.00 mg/L Cl_2 concentration solutions, the standard deviation was ±0.007 mg/L Cl_2.

Testing zero concentration samples, the limit of detection was 0.005 mg/L Cl_2. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

Using two representative lots of AccuVacs, the standard deviation was ±0.009 mg/L Cl_2. and the limit of detection was 0.010 mg/L Cl_2.

INTERFERENCES

Samples containing more than 250 mg/L alkalinity or 150 mg/L acidity as $CaCO_3$ may not develop the full amount of color, or it may instantly fade. Neutralize these samples to a pH of 6 to 7 with 1 N sulfuric acid, or 1 N sodium hydroxide. Determine the amount required on a separate 25-mL sample; then add the same amount to the sample to be tested.

Samples containing monochloramine will cause a gradual drift to higher chlorine readings. When read within one minute of reagent addition, 3.0 mg/L monochloramine will cause an increase of less than 0.1 mg/L in the free chlorine reading.

Bromine, iodine, ozone and oxidized forms of manganese and chromium also may react and show as chlorine. To compensate for the effects of manganese (Mn^{4+}) or chromium (Cr^{6+}), adjust pH to 6 to 7 as described above, then add 3 drops of potassium iodide, 30 g/L, to 25 mL of sample, mix and wait 1 minute. Add 3 drops of sodium arsenite, 5 g/L, and mix. Analyze this sample as described above. (If chromium is present, allow exactly the same reaction period with the DPD for both analyses). Subtract the result of this test from the original analysis to obtain the accurate chlorine result.

DPD Free Chlorine Reagent Powder Pillows and AccuVac Ampuls contain a buffer formulation which will withstand high (at least 1000 mg/L) levels of hardness without interference.

SUMMARY OF METHOD

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

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
DPD Free Chlorine Reagent Powder Pillows, 10 mL	1 pillow	100/pkg	21055-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

DPD Free Chlorine Reagent AccuVac Ampuls	1 ampul	25/pkg	25020-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 52.01	1	each	46252-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac vial	1	each	43784-00
Beaker, 50 mL	1	each	500-41
DR/700 Filter Module Number 52.01	1	each	46252-00

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Chlorine Standard Solution, Voluette ampule, 50-75 mg/L	16/pkg	14268-10
DPD Free Chlorine Reagent, with dispensing cap	250 tests	21055-29
DPD Free Chlorine Reagent Powder Pillows, 25-mL sample	100/pkg	14070-99
Potassium Iodide Solution, 30 g/L	100 mL* MDB	343-32
Sodium Arsenite, 5 g/L	100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1.000 N	100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N	100 mL* MDB	1270-32
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, 25 mL, poly	each	1081-40
pH Meter, EC10, portable	each	50050-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

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Outside the U.S.A. - Contact the Hach office or distributor serving you.

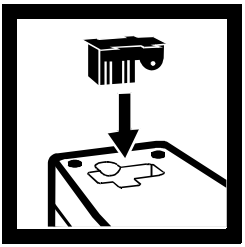
*Contact Hach for larger sizes.

CHLORINE, TOTAL (0 to 3.50 mg/L)

For water, wastewater and seawater

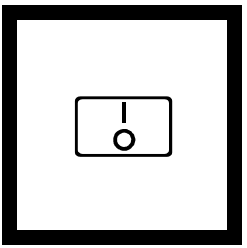
DPD Method* (Powder Pillows or AccuVac Ampuls), USEPA accepted for reporting**

USING POWDER PILLOWS



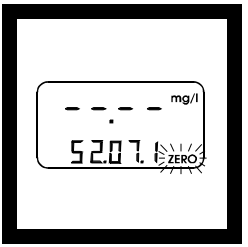
1. Install module
52.01
in a DR/700.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



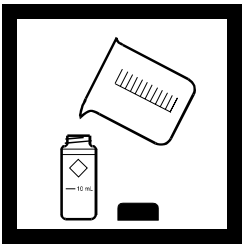
2. Press: **I/O**

The display will show
525 nm
and module number
52.01



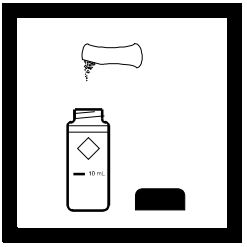
3. After 2 seconds the display will show a program number, concentration units and the zero prompt. If necessary press the **UP ARROW** key until the lower display shows program number
52.07.1

*Adapted from *Standard Methods for the Examination of Water and Wastewater.*
**Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-Cl G for drinking water



4. Fill a 10-mL cell to the 10-mL line with sample.

Note: *A 25-mL sample can be tested by using 25-mL sample cells and optional reagents.*

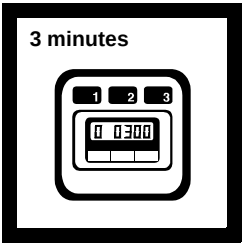


5. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the sample cell and shake for 20 seconds.

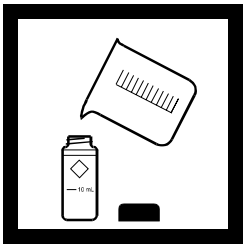
Note: *A pink color will develop if chlorine is present.*

Note: *Accuracy is not affected by undissolved powder.*

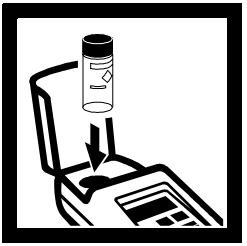
Note: *Shaking the cell dissipates bubbles which may form in samples containing dissolved gases.*



6. Wait 3 minutes.

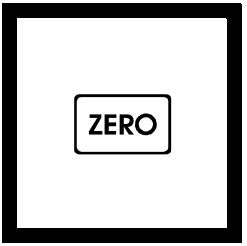


7. Fill a 10-mL cell to the 10-mL line with sample (the blank). Cap.



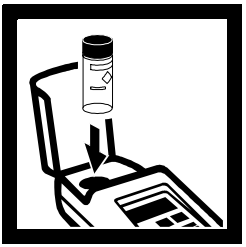
8. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



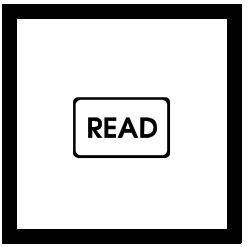
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



10. Within 3 minutes after the 3-minute period, place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*

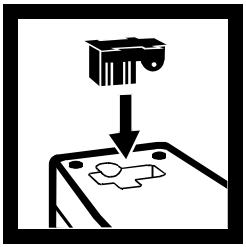


11. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L total chlorine (Cl_2).

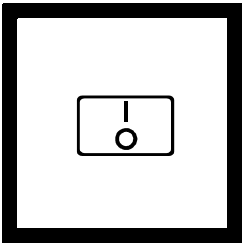
Note: *If the sample temporarily turns yellow after reagent addition, or flashes the upper range limit, dilute a fresh sample and repeat the test. A slight loss of chlorine may occur because of the dilution. Multiply the result by the appropriate dilution factor; see Sample Dilution Techniques (Section I).*

USING ACCUVAC AMPULS



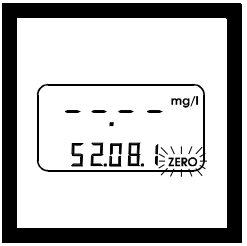
1. Install the DR/700 module number
52.01
in the DR/700.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



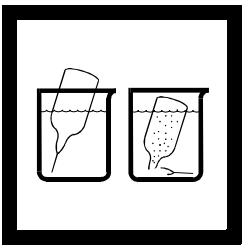
2. Press: **I/O**

The display will show
525 nm
and module number
52.01



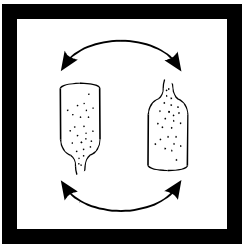
3. After 2 seconds the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
52.08.1

4. Fill a cell with 10 mL of sample (the blank). Cap. Collect at least 40 mL of sample in the 50-mL beaker.



5. Fill a DPD Total Chlorine Reagent AccuVac Ampul with sample.

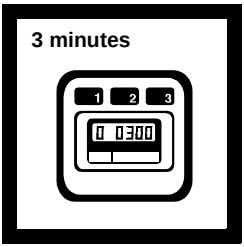
Note: *Keep the tip immersed while the ampul fills completely.*



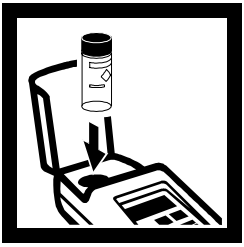
6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will form if chlorine is present.*

Note: *Accuracy is not affected by undissolved powder.*

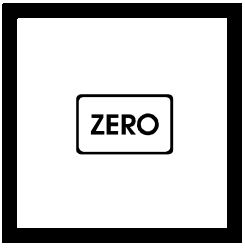


7. Wait 3 minutes.



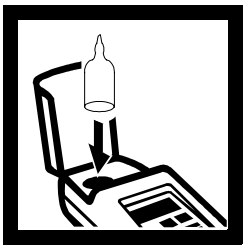
8. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



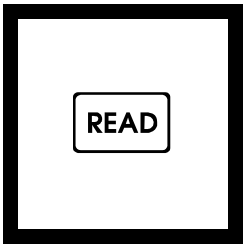
9. Press: **ZERO**
The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

10. Insert the AccuVac Vial Adapter into the cell holder.



11. Within 3 minutes after the 3-minute period, place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L total chlorine (Cl_2).

Note: *If the sample temporarily turns yellow after sample addition, or flashes the upper range limit, dilute a fresh sample and repeat the test. A slight loss of chlorine may occur because of the dilution. Multiply the result by the appropriate dilution factor; see Sample Dilution Techniques (Section I).*

ACCURACY CHECK

Standard Additions Method

a) Snap the top off the Chlorine Voluette Ampule Standard Solution.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL samples. Swirl gently to mix. (For AccuVac Ampuls, use 50-mL beakers.)

c) Analyze each sample as described above. Each 0.1 mL of standard will cause a incremental increase in chlorine, the exact value of which depends of the concentration in the Voluette. Check the certificate enclosed with the Voluettes for this value.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 1.00 mg/L Cl_2 concentration solutions, the standard deviation was ±0.005 mg/L Cl_2.

Testing zero concentration samples, the limit of detection was 0.010 mg/L Cl_2. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry,* **1980**, *52*, 2242-2249.

Using two representative lots of AccuVacs, the standard deviation was ±0.005 mg/L Cl_2. and the limit of detection was 0.015 mg/L Cl_2.

INTERFERENCES

Samples containing more than 250 mg/L alkalinity or 150 mg/L acidity as $CaCO_3$ may not develop the full amount of color, or it may instantly fade. Neutralize these samples to a pH of 6 to 7 with 1 N sulfuric acid, or 1 N sodium hydroxide. Determine the amount required on a separate 10-mL sample; then add the same amount to the sample to be tested. Bromine, iodine, ozone and oxidized forms of manganese and chromium also may react and show as chlorine. To compensate for the effects of manganese (Mn^{4+}) or chromium (Cr^{6+}), adjust pH to 6 to 7 as described above, then add 3 drops of potassium iodide, 30 g/L, to 25 mL of sample, mix and wait 1 minute. Add 3 drops of sodium arsenite,

5 g/L, and mix. Analyze this sample as described above. (If chromium is present, allow exactly the same reaction period with the DPD for both analyses). Subtract the result of this test from the original analysis to obtain the accurate chlorine result.

DPD Total Chlorine Reagent Powder Pillows and AccuVac Ampuls contain a buffer formulation which will withstand high (at least 1000 mg/L) levels of hardness without interference.

SUMMARY OF METHOD

Chlorine can be present in water as free available chlorine and as combined available chlorine. Both forms can exist in the same water and be determined together as the total available chlorine. Free chlorine is present as hypochlorous acid and/or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride and other chloro derivatives. The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N, N-diethyl-p-phenylenediamine) along with free chlorine present in the sample to form a red color which is proportional to the total chlorine concentration. To determine the concentration of combined chlorine, run a free chlorine test. Subtract the results from the results of the total chlorine test to obtain combined chlorine.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows	1 pillow	100/pkg	21056-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

DPD Total Chlorine Reagent AccuVac Ampuls	1 ampul	25/pkg	25030-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 52.01	1	each	46252-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Description	Quantity Per Test	Unit	Cat. No.
Beaker, 50 mL	1	each	500-41
DR/700 Filter Module Number 52.01	1	each	46252-00

OPTIONAL REAGENTS

Chlorine Standard Solution, Voluette ampule, 50-75 mg/L, 10 mL	16/pkg	14268-10
DPD Total Chlorine Reagent, with dispensing cap	250 tests	21056-29
Potassium Iodide Solution, 30 g/L	100 mL* MDB	343-32
Sodium Arsenite, 5 g/L	100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1 N	100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1 N	100 mL* MDB	1270-32
Water, demineralized	4 mL	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac vial	each	46025-00
Ampule Breaker Kit	each	21968-00
Caps for 10- and 25-mL Sample Cells	12/pkg	24018-12
Cylinder, graduated, 25 mL, poly	each	1081-40
Graph Paper, linear	100/pkg	22313-00
pH Meter, EC10, portable	each	50050-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

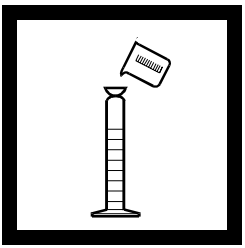
In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

*Contact Hach for larger sizes.

HARDNESS

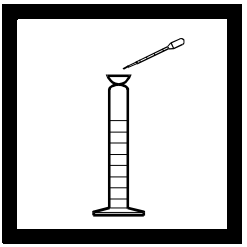
(0 to 2.00 mg/L Mg as $CaCO_3$; 0 to 2.7 mg/L Ca as $CaCO_3$)
For water, wastewater, seawater

Calcium and Magnesium; Calmagite Colorimetric Method

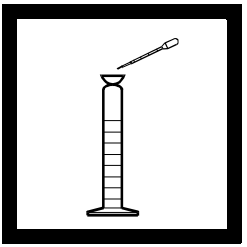


1. Pour 100 mL of water sample into a 100-mL graduated mixing cylinder.

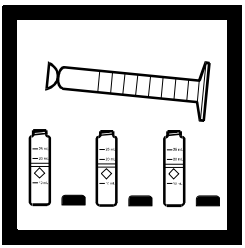
Note: *For most the most accurate magnesium test results, the sample temperature should be 21-29°C (70-84°F).*



2. Add 1.0 mL of Calcium and Magnesium Indicator Solution using a 1.0 mL measuring dropper. Stopper and invert several times to mix.

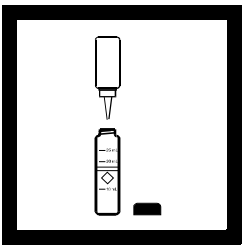


3. Add 1.0 mL of Alkali Solution for Calcium and Magnesium Test using a 1.0 mL measuring dropper. Stopper and invert several times to mix.

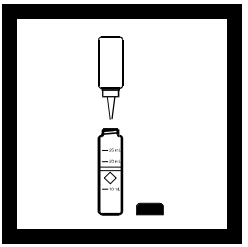


4. Pour 25 mL of this solution into each of three 25-mL sample cells.

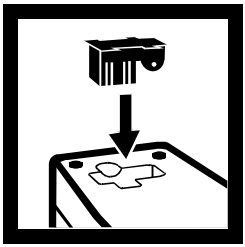
Note: *The test will detect any calcium or magnesium contamination in the mixing cylinders, measuring droppers or sample cells. To test cleanliness, repeat the test multiple times until you obtain consistent results.*



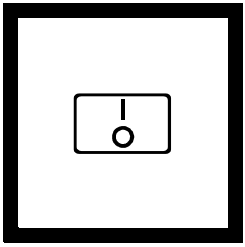
5. Add one drop of 1 M EDTA Solution to one cell (the blank). Cap and invert to mix.



6. Add one drop of EGTA Solution to another cell (the prepared sample). Cap and invert to mix.

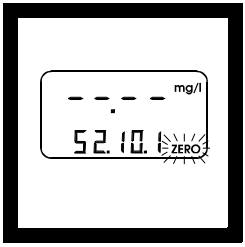


7. Install module number
52.01
in a DR/700.

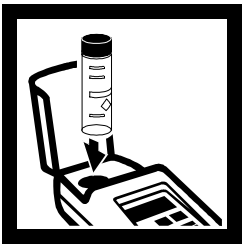


8. Press: **I/O**

The display will show
525 nm
and module number
52.01

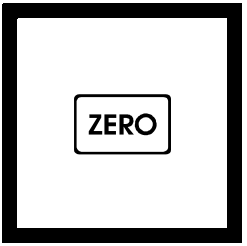


9. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
52.10.1
for magnesium as $CaCO_3$.



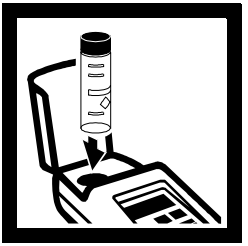
10. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



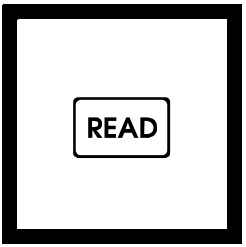
11. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



12. Place the prepared sample in the cell holder.

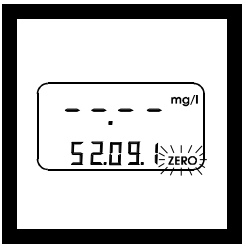
Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



13. Press: **READ**

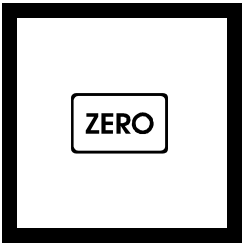
The display will count down to 0. Then the display will show the results in mg/L magnesium as $CaCO_3$.

Note: *To convert the results to mg/L Mg, multiply the result by 0.243.*



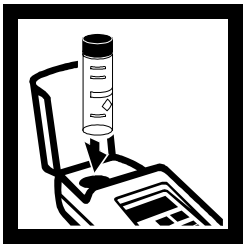
14. Press the **PROGRAM** key once and the **UP ARROW** key until the lower display shows program number **52.09.1** for calcium as $CaCO_3$.

Note: *Do not remove sample cell.*



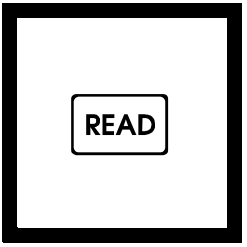
15. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



16. Place the third sample cell in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



17. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L calcium as $CaCO_3$.

Note: *To convert the results to mg/L Ca, multiply by 0.400.*

Note: *Hardness (mg/L) = mg/L Ca as $CaCO_3$ plus mg/L Mg as $CaCO_3$.*

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 1.23 mg/L Mg as $CaCO_3$ and 1.875 mg/L Ca as $CaCO_3$ concentration samples, the standard deviation was ±0.020 mg/L as $CaCO_3$ and ±0.033 mg/L Ca as $CaCO_3$, respectively.

Testing zero concentration samples, the limit of detection was 0.011 mg/L Mg as $CaCO_3$ and 0.100 mg/L Ca as $CaCO_3$. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980**, 52, 2242-2249).

INTERFERENCES

For the most accurate calcium test result, the test should be rerun on a diluted sample if the calcium is over 1.0 and the magnesium is over 0.25 mg/L as $CaCO_3$. No retesting is needed if either is below those respective concentrations.

The following cause a detectable error in test results.

Cr^{3+}	0.25 mg/L
Cu^{2+}	0.75 mg/L
EDTA, chelated	0.2 mg/L as $CaCO_3$
Fe^{2+}	1.4 mg/L
Fe^{3+}	2.0 mg/L
Mn^{2+}	0.20 mg/L
Zn^{2+}	0.050 mg/L

SUMMARY OF METHOD

The colorimetric method for measuring hardness supplements the more conventional titrimetric method through its ability to measure very low levels of calcium and magnesium. Also some interfering metals (those listed above) in the titrimetric method will be rendered inconsequential when diluting the sample to bring it within the range of this test. The indicator dye used is calmagite which forms a purplish-blue color in a strongly alkaline solution and changes to red when contacting free calcium or magnesium. Calcium and magnesium determinations are made by chelating calcium with EGTA to destroy any red color due to calcium and then chelating the calcium and magnesium with EDTA to destroy the red color due to both calcium and magnesium. By measuring the red color in the different states, calcium and magnesium concentrations are determined.

REQUIRED REAGENTS

	Cat. No.
Hardness Reagent Set (100 Tests)	23199-00
Includes: (1) 22417-37, (1) 22418-37, (1) 22419-26, (1) 22297-26	

Description	Quantity Per Test	Unit	Cat. No.
Alkali Solution for Calcium and Magnesium Test	1 mL	105 mL	22417-32
Calcium and Magnesium Indicator Solution	1 mL	105 mL	22418-32
EDTA Solution, 1 M	1 drop	59 mL	22419-26
EGTA Solution	1 drop	59 mL	22297-26

REQUIRED APPARATUS

Cylinder, 100-mL mixing	1	each	1896-42
Dropper, measuring, 1.0 mL	2	10/pkg	21247-10
DR/700 Filter Module Number 52.01	1	each	46252-00

OPTIONAL APPARATUS

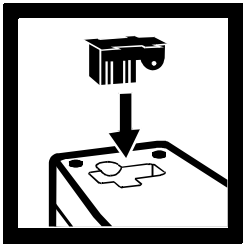
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Sample Cell, 10-mL, with screw cap	6/pkg	24276-06
Sample Cell, 25-mL, with screw cap	6/pkg	24019-06
Thermometer, -20 to 105 °C	each	1877-01

IODINE (0 to 12.50 mg/L)

For water, wastewater and seawater

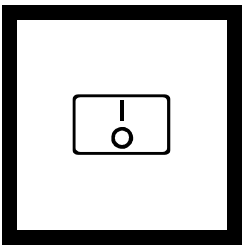
DPD Method* (Powder Pillows or AccuVac Ampuls)

USING POWDER PILLOWS



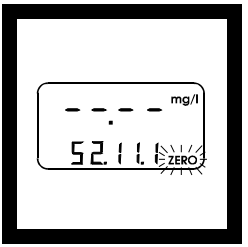
1. Install module **52.01** in a DR/700.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



2. Press: **I/O**

The display will show **525 nm** and module number **52.01**



3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **52.11.1**

*Adapted from Palin, A.T. *Inst. Water Eng*. 1967, 21(6), 537-547.

4. Fill a 10-mL cell to the 10-mL line with sample.

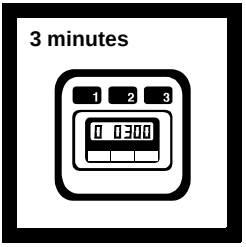
Note: *A 25-mL sample can be tested by using 25-mL sample cells and optional reagents.*

5. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Cap and shake the cell for 20 seconds.

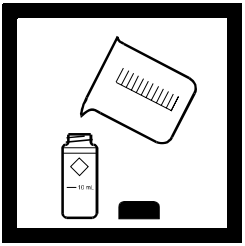
Note: *Shaking the cell dissipates bubbles which may form in samples containing dissolved gases.*

Note: *A pink color will develop if iodine is present.*

Note: *Accuracy is not affected by undissolved powder.*



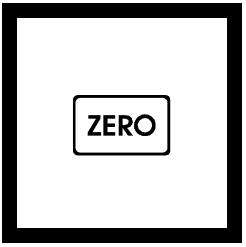
6. Wait 3 minutes.



7. Fill a 10-mL cell to the 10-mL line with sample (the blank). Cap.

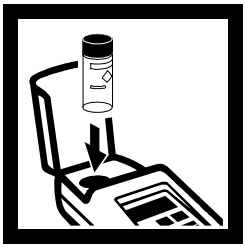
8. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



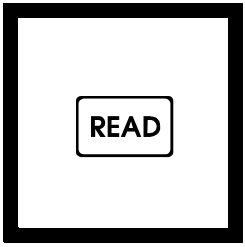
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



10. Within 3 minutes after the 3-minute period, place the prepared sample in the cell holder.

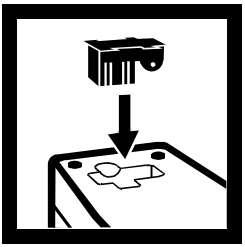
Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



11. Press: **READ**

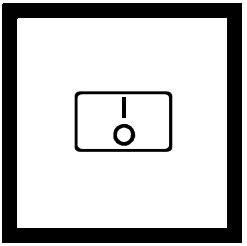
The display will count down to 0. Then the display will show the results in mg/L iodine (I_2).

USING ACCUVAC AMPULS



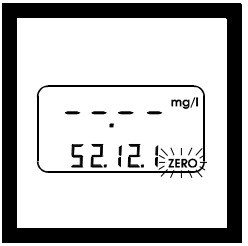
1. Install module **52.01** in a DR/700.

Note: *Sample must be analyzed immediately and cannot be preserved for later analysis.*



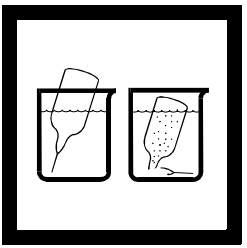
2. Press: **I/O**

The display will show **525 nm** and module number **52.01**



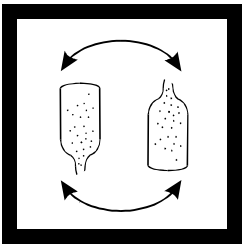
3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **52.12.1**

4. Fill a cell to the 10-mL line with 10 mL of sample (the blank). Cap. Collect at least 40 mL of sample in a 50-mL beaker.



5. Fill a DPD Total Chlorine Reagent AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*

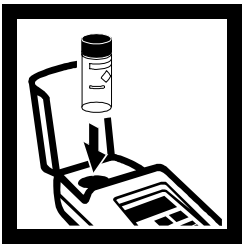


6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will form if iodine is present.*

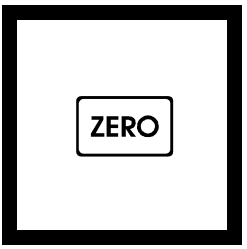
Note: *Accuracy is unaffected by undissolved powder.*

7. Wait 3 minutes.



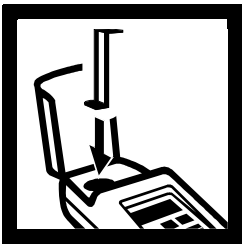
8. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

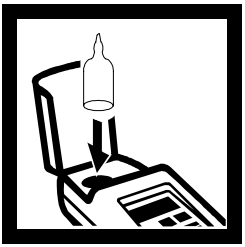


9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

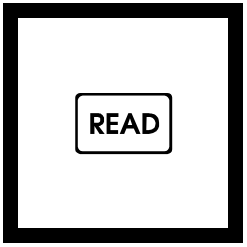


10. Insert the AccuVac Vial Adapter into the cell holder.



11. Within 3 minutes after the 3-minute period, place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L iodine (I_2).

ACCURACY CHECK

Standard Additions Method

a) Snap the top off the Chlorine Voluette Ampule Standard Solution, 50 to 75 mg/L Cl_2.

b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to three 25-mL water samples. Swirl gently to mix. (For AccuVac ampuls, use 50-mL beakers.)

c) Analyze each sample as described above. Each 0.1 mL of standard should cause an incremental increase in iodine, the exact value of which depends on the chlorine concentration in the Voluette. Check the certificate enclosed with the Voluettes for the incremental chlorine value. Multiply by 3.6 to obtain the value for iodine.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 1.00 mg/L Cl_2 (equivalent to 3.58 mg/L I_2) concentration samples, the standard deviation was ±0.005 mg/L Cl_2 (equivalent to 0.02 mg/L I_2).

Testing zero concentration samples, the limit of detection was 0.010 mg/L Cl_2 (equivalent to 0.04 mg/L I_2). The limit of detection was calculated as three times the standard deviation when tasting zero concentration samples (Adapted from *Analytical Chemistry*, **1980,** 52, 2242-2249*).*

Using two representative lots of AccuVacs, the standard deviation was ±0.005 mg/L Cl_2 (equivalent to 0.02 mg/L I_2) and the limit of detection was 0.015 mg/L Cl_2 (equivalent to 0.06 mg/L I_2)

INTERFERENCES

Samples containing more than 300 mg/L alkalinity or 150 mg/L acidity as $CaCO_3$ may not develop the full amount of color, or it may instantly fade. Neutralize these samples to a pH of 6 to 7 with 1 N sulfuric acid or 1 N sodium hydroxide. Determine the amount required on a separate 25-mL sample. Add the same amount to the sample to be tested. Correct for volume additions (See Section I).

Bromine, chlorine, ozone and oxidized forms of manganese and chromium also may react and read as iodine. To compensate for the effects of manganese (Mn^{4+}) or chromium (Cr^{6+}), adjust pH to 6 to 7 as described above. Add three drops of potassium iodide, 30 g/L, to 25 mL of sample, mix and wait one minute. Add three drops of sodium arsenite, 5 g/L, and mix. Analyze this sample as described above. This sample can be transferred to a 10-mL cell for measurement so the cell cover can be closed in bright sunlight. (If chromium, is present, allow the same reaction period with the DPD for both analyses.) Subtract the result of this test from the original analysis to obtain the accurate iodine result. Bromine, chlorine and iodine cannot be compensated for.

DPD Reagent Powder Pillows and AccuVac ampuls are formulated with a buffer which will withstand high levels (1000 mg/L) of hardness without interference.

SUMMARY OF METHOD

Iodine reacts with DPD (N, N-diethyl-p-phenylenediamine) to form a red color which is proportional to the total iodine concentration.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
DPD Total Chlorine Reagent			
Powder Pillows, 10 mL	1 pillow	100/pkg	21056-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

DPD Total Chlorine Reagent			
AccuVac Ampuls	1 ampul	25/pkg	25030-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening			
powder pillows	1	each	968-00
DR/700 Filter Module			
Number 52.01	1	each	46252-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41
DR/700 Filter Module			
Number 52.01	1	each	46252-00

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Chlorine Standard Solution Volutte Ampule, 50 to 75 mg/L Cl_2, 10 mL	16/pkg	14268-10
DPD Total Chlorine Reagent Powder Pillows, 25 mL	100/pkg	14064-99
Potassium Iodide Solution, 30 g/L	105 mL* MDB	343-32
Sodium Arsenite Solution, 5.0 g/L	105 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1 N	105 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1 N	105 mL* MDB	1270-32
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac Vial, DR/700	each	46025-00
Ampule Breaker Kit	each	21968-00
Cylinder, graduated, 25 mL, poly	each	1081-40
pH Meter, EC10, portable	each	50050-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Sample Cell, 10-mL, with screw cap	each	24276-06
Sample Cell, 25-mL, with screw cap	each	24019-06

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Outside the U.S.A--Contact the Hach office or distributor serving you.

*Contact Hach for larger sizes

LEAD (0 to 140 µg/L)

For water and wastewater

Dithizone Method†*; USEPA accepted for reporting
Digestion is required (see Section 1)**

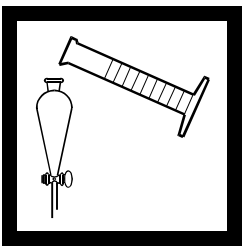
1. Fill a 250-mL graduated cylinder to the 250-mL mark with sample.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of the stored samples before analysis.*

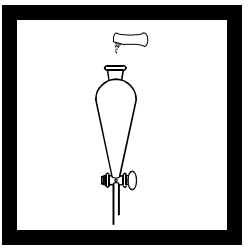
Note: *The DR/700 must be calibrated before sample measurement. See Calibration following these steps.*

Note: *Clean all glassware with a Nitric Acid Solution, 1:1. Rinse with demineralized water.*

Note: *Cloudy or turbid samples may require filtering before testing. Report results as µg/L soluble lead. Use a glass membrane filter to avoid lead adsorption on filter paper.*



2. Transfer the sample into a 500-mL separatory funnel.



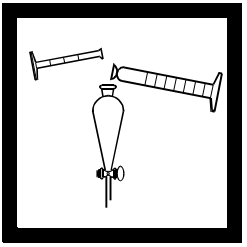
3. Add the contents of one Buffer Powder Pillow, citrate type for heavy metals. Stopper and shake to dissolve.

Note: *Spilled reagent will affect test accuracy and is hazardous to skin and other materials.*

†User calibration is required; range is approximate.
*Adapted from Snyder, L.J. *Analytical Chemistry,* **1947,** 19, 684
**Procedure is equivalent to Standard Method 3500-Pb D for wastewater.

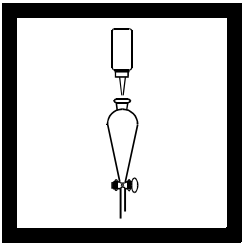
4. Add 50 mL of chloroform to a graduated mixing cylinder. Add the contents of one DithiVer Metals Reagent Powder Pillow. Stopper and invert repeatedly to mix. This is DithiVer solution.

Note: *Use adequate ventilation. The DithiVer powder will not dissolve completely in the chloroform. For further notes, see DithiVer Solution Preparation, Storage and Reagent Blank following these steps.*



5. Add 30 mL of the dithizone solution to the funnel. Stopper, invert, and open the stopcock slowly to vent. Close the stopcock. Add 5 mL of Sodium Hydroxide Standard Solution, 5.0 N. Stopper, invert, and open the stopcock to vent. Close the stopcock and shake the funnel once or twice and vent again.

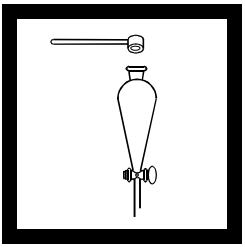
Note: *If the solution turns orange during shaking, add a few drops of 5.25 N, Sulfuric Acid Standard Solution and shake again. For the most accurate results, repeat Steps 1 through 5 and use less Sodium Hydroxide Standard Solution.*



6. Continue adding 5.0 N Sodium Hydroxide Standard Solution, dropwise, until the color of the solution changes from blue-green to orange. Then add 5 more drops of the Sodium Hydroxide Standard Solution.

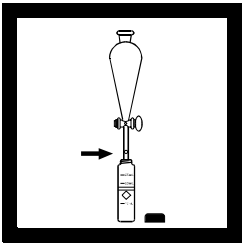
Note: *Large amounts of zinc will cause the color transition at the end point to be indistinct.*

Note: *For most accurate results, adjust the sample to pH 11.0 to 11.5 using a pH meter, omitting the five additional drops of Sodium Hydroxide Standard Solution.*



7. Add 2 heaping 1.0 g scoops of potassium cyanide, ACS, to the funnel. Stopper and shake vigorously until the potassium cyanide is all dissolved (about 15 seconds). Let the funnel stand undisturbed for 1 minute.

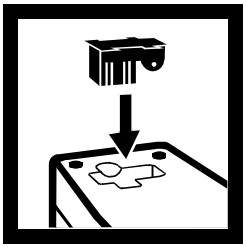
Note: *After the addition of cyanide the bottom layer will be pink if lead is present. A pink color in the bottom (chloroform) layer after Step 6 does not necessarily indicate the presence of lead.*



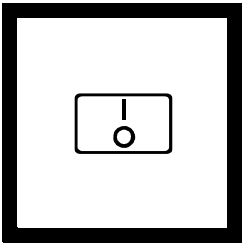
8. Insert a pea-sized cotton plug into the delivery tube of the funnel. Drain the bottom layer into a dry 25-mL sample cell (the prepared sample). Cap the cell.

Note: *The prepared sample is stable for hours if the sample cell is kept tightly capped and out of direct sunlight.*

9. Fill a 25-mL cell to the 25-mL line with chloroform (the blank). Cap.

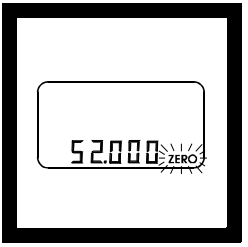


10. Install module
52.01
in a DR/700



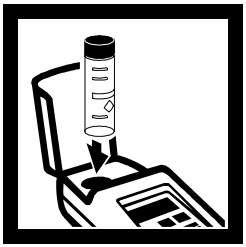
11. Press: **I/O**

The display will show
525 nm
and module
52.01



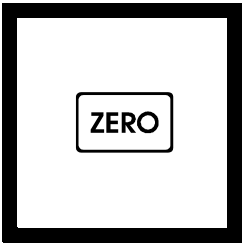
12. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. Press **PROGRAM** once of twice until the display shows program number
52.000

The upper display will show 0 µg/L.



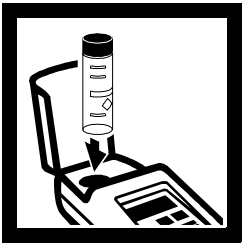
13. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the prepared sample to a 10-mL cell. If a 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



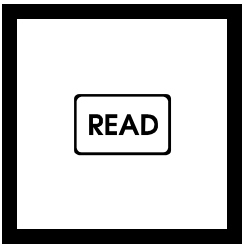
14. Press: **ZERO**

The display will count down to 0. then the display will show 0 µg/L and the zero prompt will turn off.



15. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the prepared sample to a 10-mL cell. If a 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



16. Press: **READ**

The display will count down to 0. Then the display will show the results in µg/L lead (Pb).

Note: *For best results, a reagent blank should be determined for each new lot of DithiVer Metals Reagent Powder Pillows. Use demineralized water in place of the sample in Step 2 and subtract this amount from the test results obtained in Step 16.*

DITHIVER SOLUTION PREPARATION, STORAGE AND REAGENT BLANK

Store DithiVer Powder Pillows away from light and heat. A convenient way to prepare this solution is to add the contents of 10 DithiVer Metals Reagent Powder Pillows to a 500-mL bottle of chloroform and invert several times until well mixed. Some of the powder will not dissolve. Store DithiVer solution in an amber glass bottle. This solution is usable for 24 hours for running lead tests. Do not use it to run cadmium tests.

SAMPLING AND STORAGE

Collect samples in acid-cleaned glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the pH to 2.5 to 4.5 with 5.0 N sodium hydroxide before analysis. Correct the test

result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Lead Voluette Ampule Standard Solution, 50 mg/L as Pb.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to three 250-mL samples and mix each thoroughly.

c) Analyze each sample as described above. The lead concentration should increase 20 µg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

CALIBRATION

The DR/700 must be calibrated in order to use the Lead Dithizone test procedure. To obtain the most accurate results, a new calibration should be done when a different manufacturing lot of DithiVer Metals Reagent Powder Pillows is used.

Prepare a 5.00 mg/L lead solution by pipetting 5.00 mL of 100 mg/L Lead Standard Solution into a 100-mL volumetric flask. Add demineralized water to the 100-mL mark. Cap the flask and slowly invert ten times to mix. This solution should be prepared fresh each day it is used.

Use a 4.00-mL volumetric pipet twice to pipet 8.0 mL of the 5.00 mg/L lead solution into a 500-mL separatory funnel. Add 242 mL of demineralized water to the funnel to make a 160 µg/L lead solution. Perform Steps 3 through 10 of the Lead Dithizone test procedure using the solution in the funnel.

Perform paragraph 3.2.4.1, Calibration Using Two Prepared Standards, in the DR/700 instrument manual. For Standard 1 use the Lead test procedure's "blank". Make the display show 0000 µg/L. For Standard 2 make the display show 0160 µg/L and use the Lead test procedure's "prepared sample".

INTERFERENCES

The following do not interfere.

Aluminum	Cobalt
Antimony	Iron
Arsenic	Magnesium
Cadmium	Manganese
Calcium	Nickel
Chromium	Zinc

The following interfere.

Bismuth	Silver
Copper	Tin
Mercury	

Eliminate interference from these metals by the following treatment, beginning after procedure Step 4.

a) Measure about 5 mL of the prepared DithiVer solution into the separatory funnel. Stopper the funnel, invert and open the stopcock to vent. Close the stopcock and shake the solution vigorously for 15 seconds. Allow the funnel to stand undisturbed until the layers separate (about 30 seconds). A yellow, red, or bronze color in the bottom (chloroform) layer confirms the presence of interfering metals. Draw off and discard the bottom (chloroform) layer.

b) Repeat extraction with fresh 5-mL portions of prepared DithiVer solution (discarding the bottom layer each time) until the bottom layer shows a pure dark green color for three successive extracts.

Extractions can be repeated a number of times without appreciably affecting the amount of lead in the sample.

c) Extract the solution with several 2 or 3 mL portions of pure chloroform to remove any remaining DithiVer, again discarding the bottom layer each time.

d) Continue the procedure, substituting 28.5 mL of prepared dithizone solution for the 30 mL in Step 5.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

WASTE DISPOSAL

Disposal of cyanide-containing wastes by following the steps below.

a) Use good ventilation or a fume hood

b) Add the waste, while stirring, to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochlorite (household bleach).

c) Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.

d) Flush the solution down the drain with a large excess of water.

SUMMARY OF METHOD

The DithiVer Metals Reagent is a stable powder form of dithizone. Lead ions in basic solution react with dithizone to form a pink to red lead-dithizonate complex, which is extracted with chloroform.

REQUIRED REAGENTS

	Cat. No.
Lead Reagent Set (90 Tests)	22431-00
Includes: (1) 14202-99, (2) 14458-17, (4) 12616-68, (2) 767-14, (1) 2450-53, (2) 2450-26	

Description	Quantity Per Test	Units	Cat. No.
Buffer Powder Pillows, Citrate for heavy metals	1 pillow	100/pkg	14202-99
Chloroform, ACS	50 mL	4 L	14458-17
DithiVer Metals Reagent Powder Pillows	1 pillow	25/pkg	12616-68
Potassium Cyanide, ACS	2 g	113 g	767-14
Sodium Hydroxide Solution, 5 N	5 mL	1000 mL	2450-53
Sodium Hydroxide, 5 N	drops	59 mL DB	2450-26
Water, demineralized	350 mL	4 L	272-56

REQUIRED APPARATUS

Description	Quantity Per Test	Units	Cat. No.
Clippers, for opening powder pillows	1	each	968-00
Cotton balls, absorbent	1	100/pkg	2572-01
Cylinder, mixing graduated, 50 mL	1	each	1896-41
Cylinder, graduated, 5 mL	1	each	508-37
Cylinder, graduated, 250 mL	1	each	508-46
DR/700 Filter Module Number 52.01	1	each	46252-00
Flask, volumetric, 100 mL	1	each	14574-42
Funnel, separatory, 500 mL	1	each	520-59
Pipet Filler, safety bulb	1	each	14651-00
Pipet, volumetric, 4.00 mL, Class A	1	each	14514-04
Pipet, volumetric, 5.00 mL, Class A	1	each	14515-37
Ring, support, 4"	1	each	580-01
Spoon, measuring, 1.0 g	1	each	510-00
Stand, support, 5 x 8"	1	each	563-00

OPTIONAL REAGENTS

Chloroform, ACS	500 mL	14458-49
Lead Standard Solution, 100 mg/L Pb	100 mL	12617-42
Lead Standard Solution, Voluette ampules, 50 mg/L Pb	16/pkg	14262-10
Nitric Acid Solution, 1:1	500 mL	2540-49
Nitric Acid, ACS	500 mL	152-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sulfuric Acid Standard Solution, 5.25 N	100 mL MDB	2449-32

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Caps for 10- and 25-mL sample cells	12/pkg	24018-12
Filter Discs, glass membrane, 47 mm	100/pkg	2530-00
Flask, erlenmeyer, 500 mL	each	505-49
Flask, filtering, 500 mL	each	546-49

OPTIONAL APPARATUS

Description	Units	Cat. No.
Flask, volumetric, 100 mL	each	547-42
Membrane Filter Holder, graduated, 500 mL	each	2340-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet Filler, 3-valve	each	12189-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, serological, 2 mL	each	532-36
Pipet, transfer, 2.00 mL	each	515-36
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

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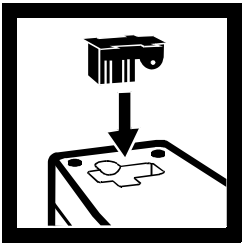
Outside the U.S.A. - Contact the Hach Office or distributor serving you.

MANGANESE, HR (0 to 20.0 mg/L)

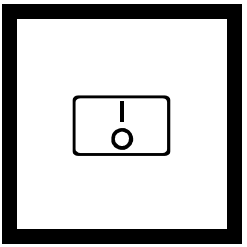
For water and wastewater

Periodate Oxidation Method*; USEPA approved for reporting†
Digestion is required; see Section 1.



1. Install module number
52.01
in a DR/700.

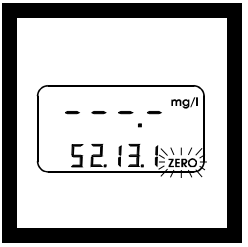
Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*



2. Press: **I/O**

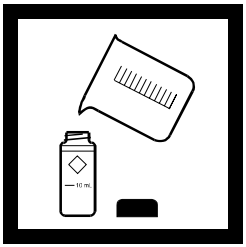
The display will show
525 nm
and module number
52.01

Note: *Total manganese determination requires prior digestion; use either the Digesdahl or mild digestion (Section 1).*



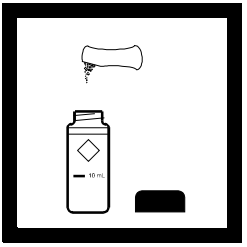
3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
52.13.1

*Adapted from *Standard Methods for the Examination for Water and Wastewater*
†*Federal Register*, **June 14, 1979** *44*(116), 34193.



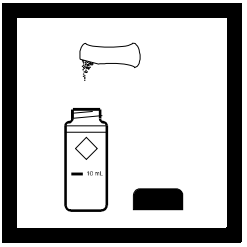
4. Fill a 10-mL cell to the 10 mL line with sample.

Note: *A 25-mL sample can be tested by using 25-mL sample cells and optional reagents.*



5. Add the contents of one Buffer Powder Pillow, citrate type. Swirl to mix.

Note: *For proof of accuracy, use a 5.0 mg/L manganese standard solution (preparation given in Accuracy Check) in place of the sample.*

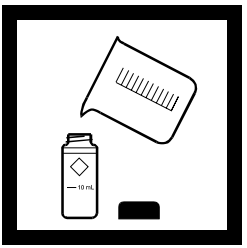


6. Add the contents of one Sodium Periodate Powder Pillow to the sample cell (the prepared sample). Cap and invert several times to mix.

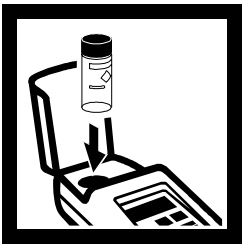
Note: *A violet color will develop if manganese is present.*

Note: *Accuracy is not affected by undissolved powder.*

7. Wait 2 minutes.

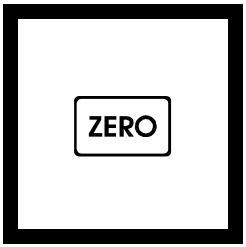


8. Fill a 10-mL cell to the 10-mL line with sample (the blank). Cap.



9. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

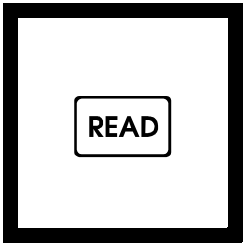


10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L and the zero prompt will turn off.

11. Within eight minutes after the 2-minute period, place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L manganese (Mn).

Note: *To convert results to other units, see Table 1.*

Table 1. Conversion Factors

To convert results from	To	Multiply by
mg/L Mn	mg/L MnO_4-	2.16
mg/L Mn	mg/L $KMnO^4$	2.88

SAMPLING AND STORAGE

Collect samples in acid-washed plastic bottles. Manganese may be lost by adsorption to glass container walls. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored at room temperature for 6 months. Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as manganese may be lost as a precipitate. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

If only dissolved manganese is to be determined, filter the sample before acid addition.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Manganese Voluette Ampule Standard Solution, High Range, 250 mg/L Mn.

b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to three 25-mL water samples. Mix thoroughly.

c) Analyze each sample as described above. The manganese concentration should increase 1.0 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section 1) for more information.

Standard Solution Method

Prepare a 5.0-mg/L manganese standard solution by pipetting 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask. Dilute to the mark with demineralized water. Or, prepare this standard by diluting 1.00 mL of the contents of a Voluette Ampule For High Range Manganese to 50 mL, using the TenSette Pipet. Prepare these solutions daily.

INTERFERENCES

The following may interfere when present in concentrations exceeding those listed below:

Calcium	700 mg/L
Chloride	70,000 mg/L

Iron	5 mg/L
Magnesium	100,000 mg/L

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 10.0 mg/L Mn concentration samples, the standard deviation was ±0.11 mg/L Mn.

Testing zero concentration samples, the limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry*, **1980,** 52, 2242-2249).

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. If only dissolved manganese is to be determined, filter the sample before acid addition.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Buffer Powder Pillows, citrate type for manganese, 10 mL	1 pillow	50/pkg	21076-69
Sodium Periodate Powder Pillows for manganese, 10 mL	1 pillow	100/pkg	21077-69

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 52.01	1	each	46252-00

OPTIONAL REAGENTS

Buffer Powder Pillows, Citrate, 25 mL (for manganese)	100/pkg	983-99
Hydrochloric Acid, 6N	500 mL	884-49

OPTIONAL REAGENTS (continued)

Description	Unit	Cat. No.
Manganese Standard Solution, 1000 mg/L Mn	100 mL*	12791-42
Manganese Standard Solution, Voluette ampule, High Range, 250 mg/L Mn, 10 mL	16/pkg	14258-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045-32
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sodium Periodate Powder Pillows, 25 mL	100/pkg	984-99
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Dropper, plastic, 0.5 and 1.0 mL marks	10/pkg	21247-10
Flask, volumetric, Class A, 50 mL	each	14574-41
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 1000 mL	each	14574-53
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, serological, 1 mL	each	532-35
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 5.0 mL	each	14515-37
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

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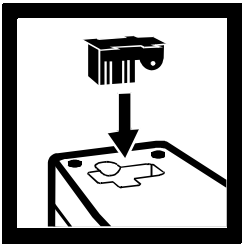
Outside the U.S.A. - Contact the Hach office or distributor serving you.

*Contact Hach for larger sizes.

OXYGEN, DISSOLVED, HR (0 to 14.0 mg/L O_2)

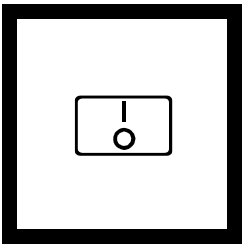
For water and wastewater

HRDO Method



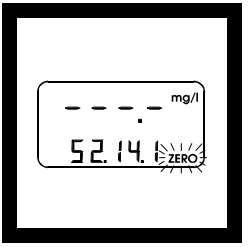
1. Install module number
52.01
in a DR/700.

Note: *Samples must be analyzed on site and cannot be stored; see Sampling and Storage following these steps.*

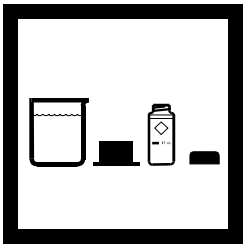


2. Press: **I/O**

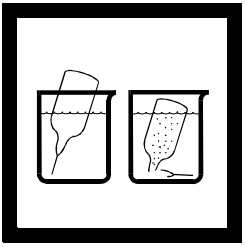
The display will show
525 nm
and module number
52.01



3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
52.14.1

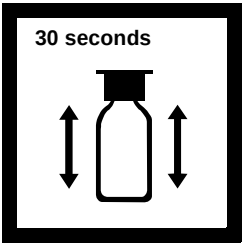


4. Fill a blue ampul cap with sample. Fill a 10-mL cell with 10 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.



5. Fill a High Range Dissolved Oxygen AccuVac Ampul with sample (the prepared sample).

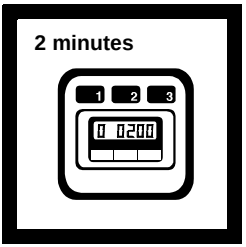
Note: *Keep the tip immersed while the ampul fills completely.*



6. Without inverting the ampul, immediately place the ampul cap that was filled with sample securely over the tip of the ampul. Shake the ampul for approximately 30 seconds.

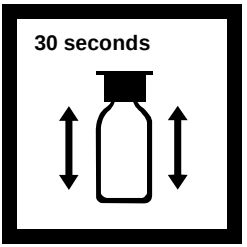
Note: *A small amount of undissolved HRDO Reagent does not affect results.*

Note: *The cap prevents contamination with atmospheric oxygen.*

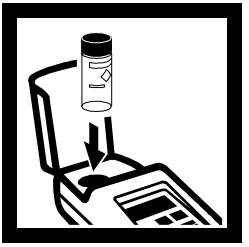


7. Wait 2 minutes.

Note: *A two-minute reaction period enables oxygen, which was degassed during aspiration, to redissolve and react.*

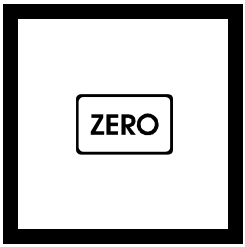


8. At the end of the two-minute period, shake the ampul for 30 seconds.



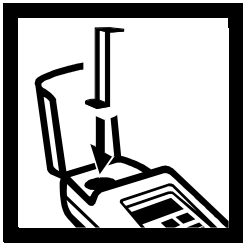
9. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

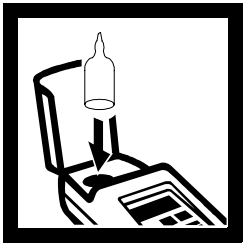


10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L and the zero prompt will turn off.

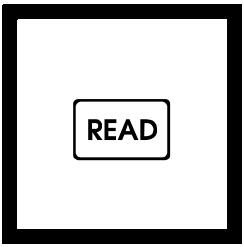


11. Insert the AccuVac Vial Adapter into the cell holder.



12. Wipe dry and place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



13. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L dissolved oxygen (0_2).

SAMPLING AND STORAGE

The foremost consideration in sampling with the High Range Dissolved Oxygen Ampul is to prevent the sample from becoming contaminated with atmospheric oxygen. This is accomplished by capping the ampul with an ampul cap in the interval between breaking open the ampul and reading the absorbance. If the ampul is securely capped, the ampul should be safe from contamination for several hours. The absorbance will decrease by approximately 3% during the first hour and will not change significantly afterwards.

Sampling and sample handling are important considerations in obtaining meaningful results. The dissolved oxygen content of the water being tested can be expected to change with depth, turbulence, temperature, sludge deposits, light, microbial action, mixing, travel time and other factors. A single dissolved oxygen test rarely reflects the accurate overall condition of a body of water. Several samples taken at different times, locations and depths are recommended for most reliable results. Samples must be tested immediately upon collection although only a small error results if the absorbance reading is taken several hours later.

ACCURACY CHECK

The results of this procedure may be compared with the results of a titrimetric procedure or dissolved oxygen meter.

STATISTICAL EVALUATION

A single operator repetitively tested samples of the same solutions, using one DR/700 and two representative lots of AccuVac Ampuls. Testing 5 mg/L O_2 concentration samples, the standard deviation was ±0.23 mg/L O_2.

Testing zero concentration samples, the limit of detection was 0.11 mg/L O_2. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980**, 52, 2242-2249).

INTERFERENCES

The following do not interfere at a level of 10 mg/L which is in excess of naturally occurring levels: Cr^{3+}, Mn^{2+}, Fe^{2+}, Ni^{2+}, Cu^{2+} and NO_2^-.

SUMMARY OF METHOD

The High Range Dissolved Oxygen AccuVac Ampul contains reagent that is vacuum sealed in a 12-mL ampul. When the AccuVac ampul is broken open in a sample containing dissolved oxygen, it forms a yellow color which turns purple. The purple color development is proportional to the concentration of dissolved oxygen.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
High Range Dissolved Oxygen AccuVac Ampuls, with 2 reusable ampul caps	1 ampul	25/pkg	25150-25

REQUIRED APPARATUS

Beaker, 50 mL	1	each	500-41
Caps, ampul, blue	varies	6/pkg	1731-06
DR/700 Filter Module Number 52.01	1	each	46252-00

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac Vial	each	43784-00
BOD bottle and stopper, 300 mL	each	621-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Dissolved Oxygen Sampler, AccuVac Model	each	24051-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

Dissolved oxygen may also be determined by titrimetric methods. Request Publication 1171 for additional information.

PHOSPHORUS, ACID HYDROLYZABLE

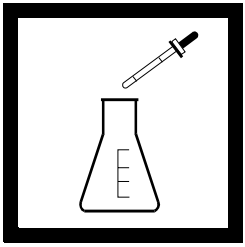
For water, wastewater, seawater

Hydrolysis to Orthophosphate Method*

1. Measure 25 mL of sample into a 50-mL erlenmeyer flask using a graduated cylinder.

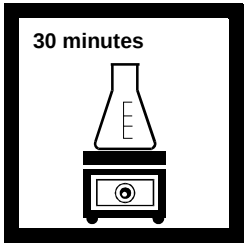
Note: *Wash all glassware with hydrochloric acid, 6 N. Rinse with demineralized water.*

Note: *If prompt analysis is impossible, see Sampling and Storage following these steps.*



2. Add 2.0 mL of Sulfuric Acid Solution, 5.25 N.

Note: *Use the 1-mL calibrated dropper provided.*

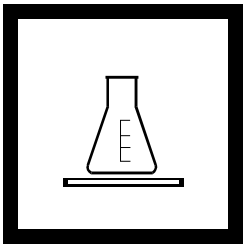


3. Place the flask (the prepared sample) on a hot plate. Boil gently for 30 minutes.

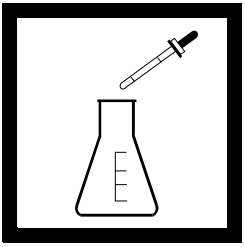
Note: *Maintain the volume near 20 mL by adding small amounts of demineralized water. Do not exceed 20 mL.*

Note: *This is more easily accomplished with a "double-boiler" technique. Place the flask in a pan or beaker of boiling water which is just deeper than the solution level in the flask. Continue boiling for 30 minutes.*

*Adapted from *Standard Methods for the Examination of Water and Wastewater*

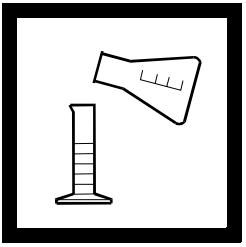


4. Cool the prepared sample to room temperature.



5. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution. Swirl to mix.

Note: *Use the 1-mL calibrated dropper provided.*



6. Pour the prepared sample into a graduated cylinder. Add demineralized water rinsings from the flask to return the volume to 25 mL. Proceed with the appropriate reactive phosphorus test.

Note: *Results of the reactive phosphorus test at this point will include the orthophosphate plus the acid-hydrolyzable (condensed) phosphate. The condensed phosphate concentration is determined by subtracting the results of a reactive phosphorus test on an untreated sample from this result.*

SAMPLING AND STORAGE

Most reliable results are obtained when samples are analyzed immediately. If prompt analysis is not possible, samples may be preserved up to 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

INTERFERENCES

If the sample is turbid, use 50 mL of sample and double the reagent volumes. Use 25 mL of the hydrolyzed sample to zero the instrument in the reactive phosphorus procedure. This compensates for any turbidity dissolved by this procedure.

SUMMARY OF METHOD

This procedure lists the necessary steps to convert condensed phosphate forms (meta-, pyro- or other polyphosphates) to orthophosphate before analysis. The procedure uses acid and heat to hydrolyze the sample. Organic phosphates are not converted to orthophosphate by this process, but a very small fraction may be unavoidably included in the result. Thus, the "acid hydrolyzable" phosphate results are primarily a measure of inorganic phosphorus. This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorous content of the sample.

The following reagents and apparatus are required in addition to those required for the reactive phosphorus test.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL* MDB	2450-32
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL* MDB	2449-32

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508-40
Flask, erlenmeyer, 50 mL	1	each	505-41

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Hydrochloric Acid, 6 N	500 mL	884-49
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Cylinder, graduated, 50 mL	each	508-41
Flask, erlenmeyer, 125 mL	each	505-43
Hot Plate, 3 $^{1}/_{2}$" diameter, 120 Vac	each	12067-01
Hot Plate, 3 $^{1}/_{2}$" diameter, 240 Vac	each	12067-02
Pad, cooling, 4" x 4"	each	18376-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00

For Technical Information Prices and Ordering

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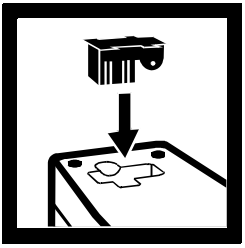
Outside the U.S.A. - Contact the Hach office or the distributor serving you.

*Contact Hach for larger sizes.

PHOSPHORUS, REACTIVE (0 to 30.00 mg/L PO_4^{3-})

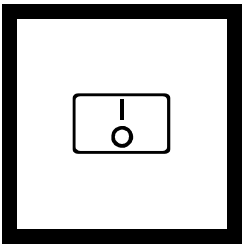
For water, wastewater, seawater

(Also called Orthophosphate) Amino Acid Method*



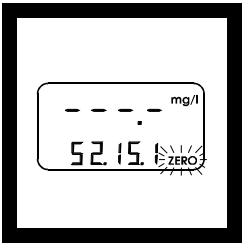
1. Install module number
52.01
in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*



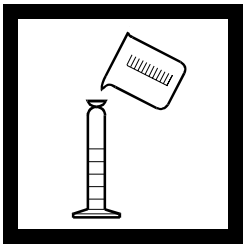
2. Press: **I/O**

The display will show
525 nm
and module number
52.01



3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
52.15.1

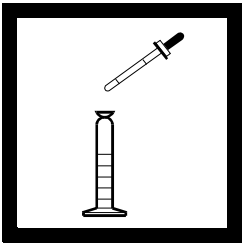
*Adapted from *Standard Methods for the Examination of Water and Wastewater.*



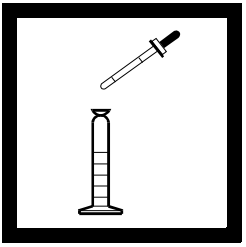
4. Fill a 25-mL graduate mixing cylinder with 25 mL of sample.

Note: *For proof of accuracy, use a 10.0 mg/L as PO_4^{3-} (3.3 mg/L as P) phosphorus standard solution (preparation given in Accuracy Check) in place of the sample.*

Note: *Run a reagent blank with each lot of reagent. Repeat the test using demineralized water as the sample. Subtract this value from each result obtained with this lot of reagent.*



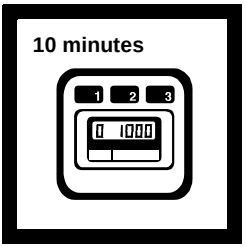
5. Add 1 mL of Molybdate Reagent using a 1-mL calibrated dropper.



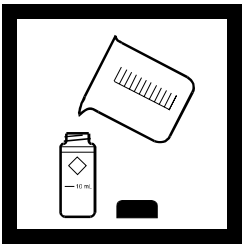
6. Add 1 mL of Amino Acid Reagent Solution. Stopper and invert several times to mix (the prepared sample).

Note: *A blue color will form if phosphate is present.*

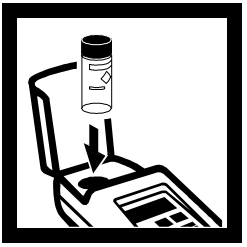
Note: *Substitute the contents of one Amino Acid Reagent Powder Pillow for 1 mL of Amino Acid Reagent Solution if desired.*



7. Wait 10 minutes.

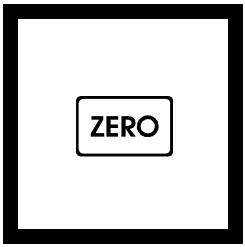


8. During the 10-minute period, fill a 10-mL cell to the 10-mL line with sample (the blank). Cap.



9. Place the blank in the cell holder.

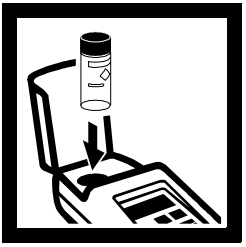
Note: *In bright sunlight, it may be necessary to close the cell compartment cover.*



10. Press: **ZERO**

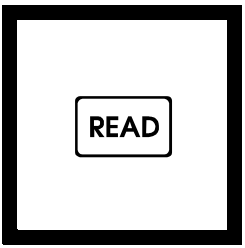
The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

11. Fill a 10-mL cell to the 10-mL line with prepared sample.



12. Place the prepared sample in the cell holder.

Note: *In bright sunlight, it may be necessary to close the cell compartment cover.*



13. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L phosphate (PO_4^{3-}).

Note: *To convert results to other units, see Table 1.*

Table 1. Conversion Factors

To convert results from	To	Multiply by
mg/L PO_4^{3-}	mg/L P_2O_5	0.747
mg/L PO_4^{3-}	mg/L P	0.326

SAMPLING AND STORAGE

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with demineralized 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 up to 24 hours by storing at 4 °C. For longer storage periods, add 4.0 mL of mercuric chloride to each liter of sample and mix. Use of mercuric chloride is discouraged due to health and environmental concerns. Samples preserve with mercuric chloride must have a sodium chloride level of 50 mg/L or higher to prevent mercury interference in the test. Spike samples low in chloride with a sodium chloride solution (5 mL of 10,246 mg/L sodium chloride solution per liter of sample).

ACCURACY CHECK

Standard Addition Method

a) Snap the neck of a Phosphate Voluette Ampule Standard, 500 mg/L PO_4^{3-}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to three 25-mL aliquots of a water samples. Mix well.

c) Analyze each sample as described in the procedure. Compare the results with the original test sample. Each 0.1-mL addition should increase the orthophosphate (PO_4^{3-}) 2.0 mg/L for stored program 485. When using store program 487, the increase should be 0.65 mg/L P for each 0.1-mL addition of standard.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 10.0-mg/L PO_4^{3-}(3.3 mg/L P) standard solution by pipetting 10.0 mL of Phosphate Standard Solution, 50 mg/L as PO_4^{3-}, into a 50-mL volumetric flask. Dilute to volume with demineralized water.

Or, prepare a 10.0-mg/L PO_4^{3-} (3.3 mg/L P) standard solution by using the TenSette Pipet to add 1.00 mL of Phosphate Voluette Ampule Standard, 500 mg/L PO_4^{3-}, into a 50-mL volumetric flask. Dilute to volume with demineralized water.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 15.0 mg/L PO_4^{3-} concentration samples, the standard deviation was ±0.10 mg/L PO_4^{3-}.

Testing zero concentration samples, the limit of detection was 0.23 mg/L PO_4^{3-}. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52,* 2242-2249).

INTERFERENCES

Samples with large amounts of turbidity may give inconsistent results. Some of the suspended particles may dissolve because of the acid used in the test. Also, results will vary because of the variable desorption of orthophosphate from the particles. For highly turbid or colored samples, add 1 mL of 10 N Sulfuric Acid Standard Solution to another 25-mL sample. Use this in place of the sample as the blank to zero the instrument in Step 10. Use a pipet and pipet filler when measuring the sulfuric acid standard.

For best results, the temperature of the sample should be 21 ±3°C (70 ±5°F).

Sulfide interferes by forming a blue color directly with the molybdate reagent. Remove sulfide interference by the following pretreatment:

a) Measure 350 mL of sample into a clean 500-mL erlenmeyer flask.

b) Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix.

c) Filter the sample through a folded filter paper and use this solution in Steps 4 and 8.

Nitrites bleach the blue color. Remove nitrite interference by adding 0.05 g of sulfamic acid to the sample. Swirl to mix. Continue with Step 5.

The following may interfere if present in concentrations exceeding those listed below:

Chloride	150,000 mg/L as Cl^-
Calcium	10,000 mg/L as $CaCO_3$
Magnesium	40,000 mg/L as $CaCO_3$

When phosphate is determined in waters containing high salt levels, low results may occur. To eliminate this interference, dilute the sample until two successive dilutions yield approximately the same results.

As the concentration of phosphate increases, the color changes from blue to green, then to yellow and finally to brown. The brown color may suggest a concentration as high as 100,000 mg/L PO_4^{3-}. If a color other than blue is formed, dilute the sample and retest.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section 1).

SUMMARY OF METHOD

In a highly acidic solution, ammonium molybdate reacts with orthophosphate to form molybdophosphoric acid. This complex is then reduced by the amino acid reagent to yield an intensely colored molybdenum blue compound.

REQUIRED REAGENTS

	Cat. No.
High Range Reactive Phosphorus Reagent Set (100 Test)	22441-00
Include: (1) 1934-32, (1) 2236-32	

Description	Quantity Per Test	Units	Cat. No.
Amino Acid Reagent	1 mL	100 mL MDB*	1934-32
Molybdate Reagent	1 mL	100 mL MDB*	2236-32

REQUIRED APPARATUS

Cylinder, 25 mL, graduated mixing	1	each	1896-40
DR/700 Filter Module Number 52.01	1	each	46252-00

OPTIONAL REAGENTS

Amino Acid Reagent Powder Pillow	100/pkg	804-99
Hydrochloric Acid Solution, 1:1 (6 N)	500 mL	884-49
Mercuric Chloride Solution	100 mL	14994-42
Phosphate Standard Solution, 50 mg/L PO_4^{3-}	599 mL	171-49

OPTIONAL REAGENTS (continued)

Description	Units	Cat. No.
Phosphate Standard Solution, Voluette ampule, 500 mg/L PO_4^{3-}, 10 mL	16/pkg	14242-10
Sodium Chloride Standard Solution, 10,246 mg/L NaCl	105 mL	23074-42
Sodium Hydroxide Standard Solution, 5.0 N	105 mL MDB*	2450-32
Sulfamic Acid, ACS	113 g	2344-14
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	2418-99
Sulfuric Acid Standard Solution, 10 N	1 L	931-53
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample Cells	12/pkg	24018-12
Clippers, for opening powder pillows	each	968-00
Cylinder, graduated, 500 mL	each	508-49
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, erlenmeyer, 500 mL	each	505-49
Flask, volumetric, Class, A, 50.00 mL	each	14574-41
Funnel, poly, 65 mm	each	1083-67
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter,EC10, portable	each	50050-00
Pipet Filler, 3-valve	each	12189-00
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 10.00 mL	each	14515-38
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Spoon, measuring, 0.05 g	each	492-00
Thermometer, -20 to 105 °C	each	1877-01

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*Contact Hach for larger sizes.

PHOSPHORUS, TOTAL

For water, wastewater and seawater

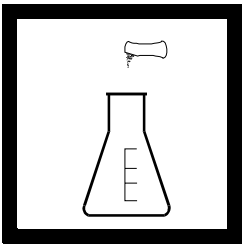
(also called Organic and Acid Hydrolyzable) Acid Persulfate Digestion Method*; USEPA accepted for reporting

1. Measure 25 mL of sample into a 50-mL erlenmeyer flask.

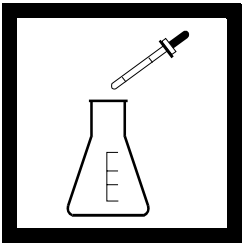
Note: *Use a graduated cylinder to measure the sample.*

Note: *Rinse all glassware with 1:1 Hydrochloric Acid Solution. Rinse again with demineralized water.*

Note: *If prompt analysis is impossible, see Sampling and Storage following these steps.*



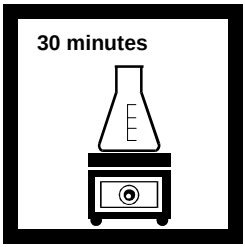
2. Add the contents of one Potassium Persulfate Powder Pillow. Swirl to mix.



3. Add 2.0 mL of 5.25 N Sulfuric Acid Solution.

Note: *Use the 1-mL calibrated dropper provided.*

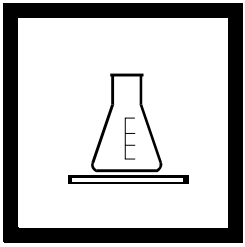
* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



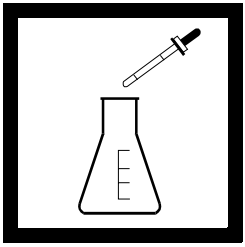
4. Place the flask on a hot plate. Boil gently for 30 minutes.

Note: *Maintain the volume near 20 mL by adding small amounts of demineralized water. Do not exceed 20 mL.*

Note: *This is more easily accomplished with a "double-boiler" technique. Place the flask in a pan or beaker of boiling water which is deeper than the solution level in the flask, Continue boiling for 30 minutes.*

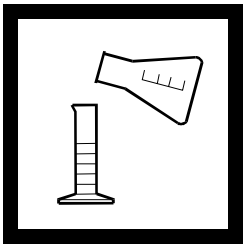


5. Cool the sample to room temperature.



6. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution. Swirl to mix.

Note: *Use the 1-mL calibrated dropper provided.*



7. Pour the sample into a 25-mL graduated cylinder. Using demineralized water rinsings from the flask, return the volume in the cylinder to 25 mL. Proceed with a reactive phosphorus test of the expected total phosphorus concentration range.

Note: *Results of the reactive phosphorus test at this point will include the organic phosphate plus the orthophosphate and the acid hydrolyzable (condensed) phosphate. The organic phosphate concentration is determined by subtracting the results of an acid hydrolyzable phosphorus test from this result. Make sure that both results are in the same units, either mg/L* PO_4^{3-} *or mg/L P before taking the difference.*

SAMPLING AND STORAGE

Most reliable results are obtained when samples are analyzed immediately. If prompt analysis is not possible, samples may be preserved up to 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

INTERFERENCES

For turbid samples, use 50 mL of sample and double the reagent quantities. Use 25 mL of the digested sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed by this procedure. For alkaline or highly buffered samples it may be necessary to use additional acid in Step 3 to drop the pH of the solution below 1.

SUMMARY OF METHOD

Phosphates present in organic and condensed inorganic forms (meta-, pyro-, or other polyphosphates) must be converted to orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate. Organically bound phosphates are thus determined indirectly by subtracting the result of an acid hydrolyzable phosphorus test from the total phosphorus result.

This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorus content of the sample. If the ascorbic acid (PhosVer 3) method is used to measure the reactive phosphorus, this method is EPA accepted for NPDES reporting.

The following reagents and apparatus are required beside those required for the reactive phosphorus test.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Potassium Persulfate Powder Pillows	1 pillow	50/pkg	2451-66
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL*MDB	2450-32

REQUIRED REAGENTS (continued)

Description	Quantity Per Test	Unit	Cat. No.
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL*MDB	2449-32
Water, demineralized	25 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, 25 mL	1	each	508-40
Flask, erlenmeyer, 50 mL	1	each	505-41

OPTIONAL REAGENTS

Hydrochloric Acid, 6 N (1:1)	500 mL	884-49
Sodium Hydroxide Solution, 5.0 N	1 L	2450-53

OPTIONAL APPARATUS

Cylinder, graduated, 50 mL	each	508-41
Flask, erlenmeyer, 125 mL	each	505-43
Hot Plate, 3 $^{1}/_{2}$-inch diameter, 120 Vac	each	12067-01
Hot Plate, 3 $^{1}/_{2}$-inch diameter, 240 Vac	each	12067-02
Pads, cooling, 4" x 4"	each	18376-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00

For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A - Contact the Hach office or distributor serving you.

Module 55.01
550 nm

Hach warrants equipment it manufactures against defective materials or workmanship for at least one year from the shipping date. Warranties do not apply to limited-life electrical components such as batteries. Full warranty information is located on the reverse side of Hach invoices.

IMPORTANT NOTICE

A lamp intensity adjustment must be performed:
- Before first or initial use
- When new filter modules are installed
- On each filter module when the lamp is replaced

Refer to Lamp Intensity Adjustment in the instrument manual.

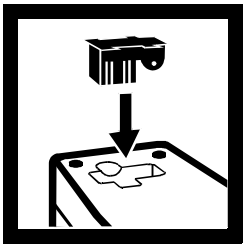
Table of Contents for 550-nm parameters

CHROMIUM, HEXAVALENT (0 to 1.000 mg/L Cr^{6+})

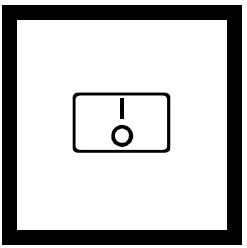
For water and wastewater

1,5-Diphenylcarbohydrazide Method* (Powder Pillows or AccuVac Ampuls), USEPA accepted for reporting**

USING POWDER PILLOWS

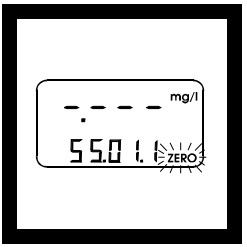


1. Install module **55.01** in a DR/700.



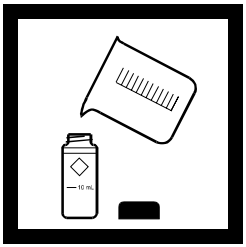
2. Press: **I/O**

The display will show **550 nm** and module number **55.01**



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **55.01.1**

* Adapted from *Standard Methods for the Examination of Water and Wastewater*
**Procedure is equivalent to USGS method I-1230-85 for wastewater



4. Fill a 10-mL cell to the 10-mL line with sample.

Note: *A 25-mL sample can be tested by using 25-mL sample cells and optional reagents.*

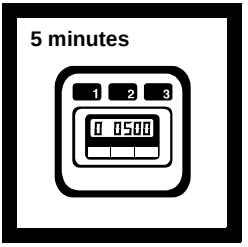
Note: *For proof of accuracy, use a 0.25 mg/L hexavalent chromium standard solution (preparation given in the Accuracy Check) in place of the sample.*

5. Add the contents of one ChromaVer 3 Reagent Powder Pillow to the cell (the prepared sample). Cap. Invert several times to mix.

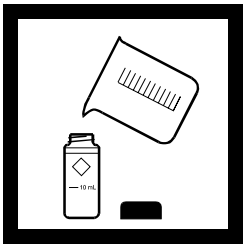
Note: *A purple color will form if hexavalent chromium is present.*

Note: *At high chromium levels a precipitate will form. Dilute sample according to Sample Dilution Techniques (Section 1).*

Note: *ChromaVer 3 Reagent should be white to tan in color. Replace if it is green or brown.*

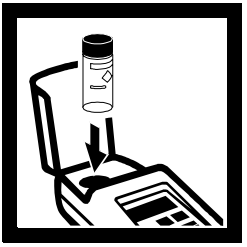


6. Wait 5 minutes.



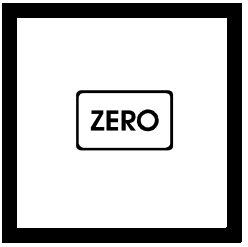
7. Fill a 10-mL cell to the 10-mL line with the sample (the blank). Cap.

Note: *For turbid samples, treat 25 mL of the blank with the contents of one Acid Reagent Powder Pillow. This will ensure any turbidity dissolved by the acid in the ChromaVer 3 Chromium Reagent also will be dissolved in the blank.*



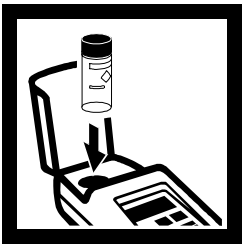
8. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



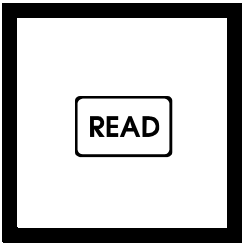
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.



10. Place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



11. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L hexavalent chromium.

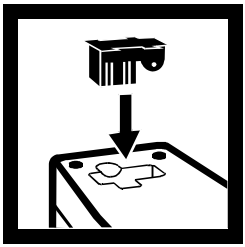
Note: *To convert results to other units, see Table 1.*

Table 1. Conversion Factors

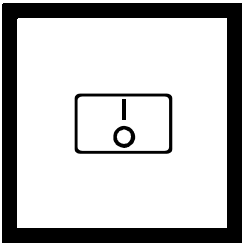
To convert results from	To	Multiply by
mg/L Cr^{6+}	mg/L CrO_4^{2-}	2.23
mg/L Cr^{6+}	mg/L Na_2CrO_4	3.12

CHROMIUM, HEXAVALENT, continued

USING ACCUVAC AMPULS

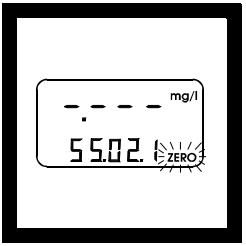


1. Install module
55.01
in a DR/700.

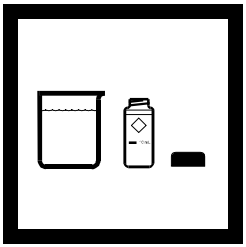


2. Press: **I/O**

The display will show
550 nm
and module number
55.01



3. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
55.02.1



4. Fill a cell with 10 mL of sample (the blank). Cap. Collect at least 40 mL of sample in a 50-mL beaker.

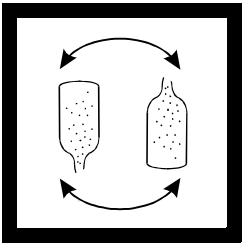
Note: *For turbid samples, treat 25 mL of the blank with the contents of one Acid Reagent Powder Pillow. This will ensure any turbidity dissolved by the acid in the ChromaVer 3 Chromium Reagent will also be dissolved in the blank.*

Note: *For proof of accuracy, use a 0.25 mg/L hexavalent chromium standard solution (preparation given in the Accuracy Check) in place of the sample.*

5. Fill a ChromaVer 3 Reagent AccuVac Ampul with sample (this is the prepared sample).

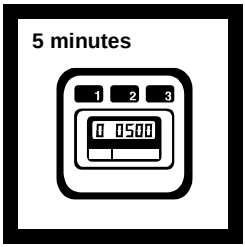
Note: *Keep tip immersed while the ampul fills.*

Note: *ChromaVer 3 should be white to tan in color. Replace if it is brown or green.*



6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

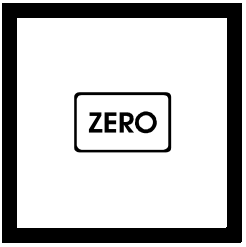
Note: *A purple color will form if hexavalent chromium is present.*



7. Wait 5 minutes.

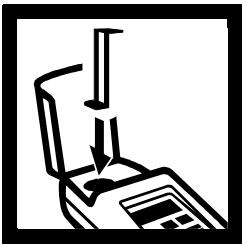
8. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*

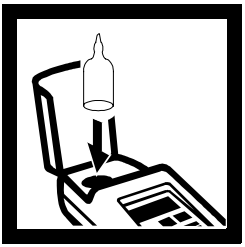


9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.

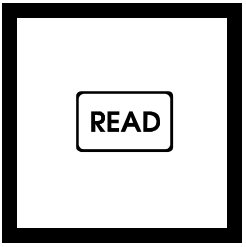


10. Insert the AccuVac Vial Adapter into the cell holder.



11. Place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L hexavalent chromium (Cr^{6+}).

Note: *To convert results to other units, see Table 1.*

Table 1. Conversion Factors

To convert results from	To	Multiply by
mg/L Cr^{6+}	mg/L CrO_4^{2-}	2.23
mg/L Cr^{6+}	mg/L Na_2CrO_4	3.12

SAMPLING AND STORAGE

Collect samples in a cleaned glass or plastic container. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored for at least 6 months at room temperature. Before analysis, adjust the pH to 4 with 5.0 N Sodium Hydroxide Standard Solution. Correct for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Chromium Voluette Ampule Standard, 12.5 mg/L Cr^{6+}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively to three 25-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The chromium concentration should increase 0.05 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 0.25-mg/L Cr^{6+} by pipetting 5.00 mL of hexavalent chromium standard solution, 50.0 mg/L Cr^{6+}, into a 1000-mL volumetric flask and diluting to the mark with demineralized water. Prepare this solution daily. Perform the chromium procedure as described above. The result should be 0.25 mg/L Cr^{6+}.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 4.00 mg/L Cr^{6+} concentration solutions, the standard deviation was ±0.0026 mg/L Cr^{6+}.

Testing zero concentration samples, the limit of detection was 0.0132 mg/L Cr^{6+}. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

Using two representative lots of AccuVacs, the standard deviation was ±0.0037 mg/L Cr^{6+} and the limit of detection was 0.0130 mg/L Cr^{6+}.

INTERFERENCES

The following do not interfere in the test up to the following concentration:

Mercurous & Mercuric Ions	Interfere slightly
Iron	1 mg/L
Vanadium	1 mg/L

Vanadium interference can be overcome by waiting ten minutes before reading.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

SUMMARY OF METHOD

Hexavalent chromium is determined by the 1,5-diphenylcarbohydrazide method using a single dry powder formulation called ChromaVer 3 Chromium Reagent. This reagent contains an acidic buffer combined with 1,5-diphenylcarbohydrazide, which reacts to give a purple color when hexavalent chromium is present.

REQUIRED REAGENTS AND APPARATUS

(Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
ChromaVer 3 Chromium Reagent Powder Pillows, for 5 and 10-mL samples	1 pillow	50/pkg	12710-99
Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 55.01	1	each	46255-00

REQUIRED REAGENTS AND APPARATUS

(Using AccuVac Ampuls)

ChromaVer 3 AccuVac ampuls	1 ampul	5/pkg	25050-25
Adapter, AccuVac Vial	1	each	46025-00
Beaker, 50 mL	1	each	500-41
DR/700 Filter Module Number 55.01	1	each	46255-00

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Acid Reagent		
Powder Pillows	50/pkg	2126-66
Chromium, Hexavalent, Standard Solution,		
50 mg/L Cr^{6+}	100 mL	810-42
Chromium, Hexavalent, Standard Solution,		
Voluette ampule, 12.5 mg/L Cr^{6+}, 10 mL	16/pkg	14256-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	473 mL	2540-49
Sodium Hydroxide Solution, 5.0 N	59 mL* SCDB	2450-26
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Flask, volumetric, Class A, 25 mL	each	14574-40
Flask, volumetric, Class A, 1000 mL	each	14574-53
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 5.00 mL, Class A	each	14515-37
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

*Contact Hach for larger sizes.

CHROMIUM, TOTAL (0 to 0.700 mg/L)

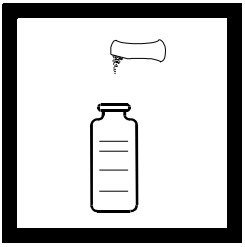
For water and wastewater

Alkaline Hypobromite Oxidation Method*

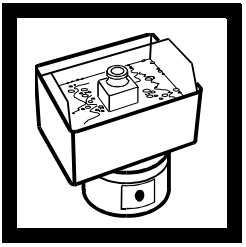
1. Fill a clean 25-mL mixing bottle with 25 mL of sample.

Note: *If sample cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*

Note: *For proof of accuracy, use a 0.25 mg/L trivalent chromium standard (preparation given in Accuracy Check) in place of the sample.*

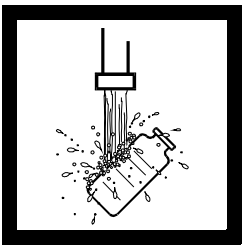


2. Add the contents of one Chromium 1 Reagent Powder Pillow (the prepared sample). Swirl to mix.



3. Place the prepared sample into a boiling water bath.

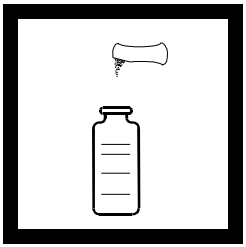
4. Wait 5 minutes.



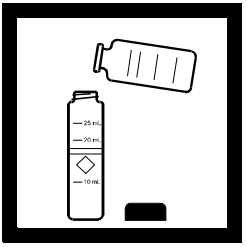
5. Remove the prepared sample from water bath. Using running tap water, cool to 25 °C.

6. Add the contents of one Chromium 2 Reagent Powder Pillow. Swirl to mix.

*Adapted from *Standard Methods for the Examination of Water and Wastewater.*

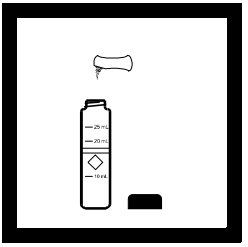


7. Add the contents of one Acid Reagent Powder Pillow. Swirl to mix.



8. Pour the prepared sample into a 25-mL cell.

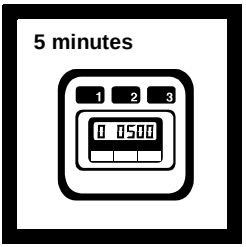
Note: *A 10-mL sample can be tested by using 10-mL sample cells and optional reagents.*



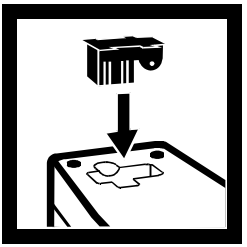
9. Add the contents of one ChromaVer 3 Chromium Reagent Powder Pillow. Swirl to mix.

Note: *A purple color will form if chromium is present.*

Note: *The color of ChromaVer 3 should be white to tan. If the color is brown to green, replace the powder. Undissolved powder does not affect accuracy.*



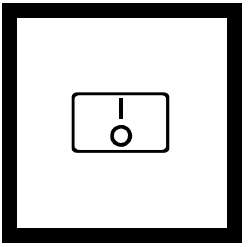
10. Wait 5 minutes.



11. During the 5 minute period, install module

55.01

in a DR/700.



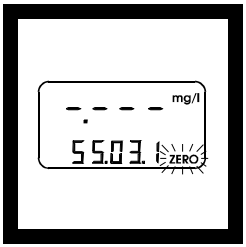
12. Press: **I/O**

The display will show

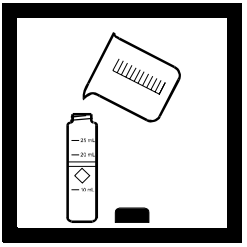
550 nm

and module number

55.01

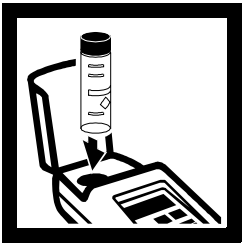


13. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **55.03.1**



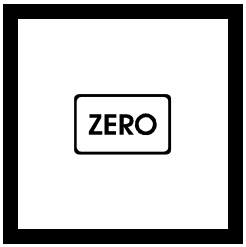
14. Fill a 25-mL cell to the 25-mL line with sample (the blank). Cap.

Note: *For turbid samples, treat the blank as described in Steps 1 through 8.*



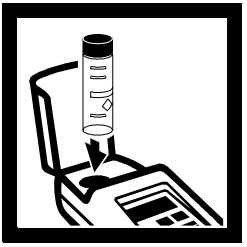
15. After the 5 minute period, place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



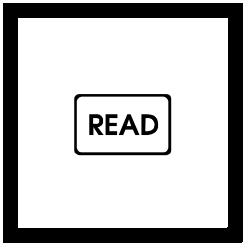
16. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.



17. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



18. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L chromium. Subtract the ChromaVer 3 Reagent blank value for final concentration; see note below.

Note: *Determine a reagent blank for each new lot of ChromaVer 3 Reagent as follows: Repeat steps 9 to 18 using demineralized water as the sample. Subtract this value from each result obtained with this lot of reagent.*

SAMPLING AND STORAGE

Collect samples in a cleaned glass or plastic container. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored for at least 6 months at room temperature. Before analysis, adjust the pH to 4 with 5.0 N Sodium Hydroxide Standard Solution. Correct for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Additions Method

a) Snap the top off a Trivalent Chromium Voluette Ampule Standard, 12.5 mg/L as Cr^{3+}.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 m/L of standard to three 25-mL water samples. Mix each thoroughly.

c) Analyze each sample as described above. The chromium concentration should increase 0.05 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 0.25 mg/L trivalent chromium standard by diluting 5.00 mL of chromium standard solution, 50 mg/L as Cr^{3+}, to 1000 mL with demineralized water. Prepare this solution daily.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 0.400 mg/L Cr concentration solutions, the standard deviation was ±0.0024 mg/L Cr.

Testing zero concentration samples, the limit of detection was 0.0072 mg/L Cr. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

Large amounts of organic material may inhibit complete oxidation of trivalent chromium. If high levels of organic material are present, see Digestion (Section I) for instruction on sample digestion. Perform the analysis, as described, on the digested sample.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

SUMMARY OF METHOD

Trivalent chromium in the sample is oxidized to the hexavalent form by hypobromite ion under alkaline conditions. The sample is acidified. The total chromium content is determined by the 1,5-diphenylcarbohydrazide method. Determine trivalent chromium by subtracting the results of a separate hexavalent chromium test from the results of the total chromium test.

REQUIRED REAGENTS

	Cat. No.
Total Chromium Reagent Set (100 Tests)	22425-00
Includes: (2) 2126-66, (2) 12066-66, (1) 2043-99, (1) 2044-99	

Description	Quantity Per Test	Unit	Cat. No.
Acid Reagent			
Powder Pillows	1 pillow	50/pkg	2126-66
ChromaVer 3 Chromium			
Reagent Powder Pillows	1 pillow	50/pkg	12066-66
Chromium 1 Reagent			
Powder Pillows	1 pillow	100/pkg	2043-99
Chromium 2 Reagent			
Powder Pillows	1 pillow	100/pkg	2044-99

REQUIRED APPARATUS

Bottle, mixing, 25 mL	1	each	17042-00
Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 55.01	1	each	46255-00
Water bath and rack	1	each	1955-99

Select one based on available voltage

Hot plate, 3 $^{1}/_{2}$" diameter, 120 Vac		each	12067-01
Hot plate, 3 $^{1}/_{2}$" diameter, 240 Vac		each	12067-02

OPTIONAL REAGENTS

ChromaVer 3 Reagent Powder Pillows, for 10 mL samples	100/pkg	12710-99

OPTIONAL REAGENTS (continued)

Description	Unit	Cat. No.
Chromium, trivalent, standard solution, 50 mg/L Cr^{3+}	100 mL	14151-42
Chromium, trivalent standard solution, Voluette ampule, 12.5 mg/L, Cr^{3+}, 10 mL	16/pkg	14257-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution 1:1	500 mL	2540-49
Sodium Hydroxide Solution 5.0 N	59 mL* DB	2450-26
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, polypropylene, 25 mL	each	1081-40
Flask, volumetric, 1000 mL	each	547-53
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 5 mL	each	515-37
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

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Outside the U.S.A. - Contact the Hach office or distributor serving you.

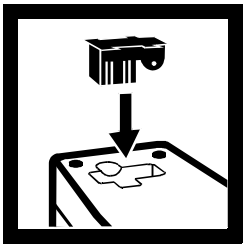
*Contact Hach for larger sizes.

COPPER (0 to 5.00 mg/L)

For water, wastewater and seawater**

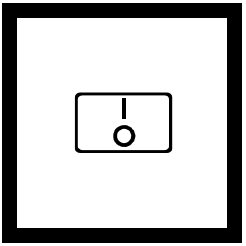
Bicinchoninate Method* (Powder Pillows or AccuVac Ampuls), USEPA Approved for reporting (digestion required; see Section 1)†**

USING POWDER PILLOWS



1. Install module **55.01** in a DR/700.

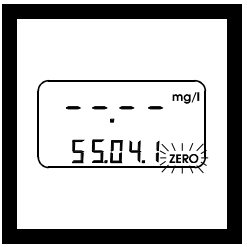
Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust pH of stored samples before analysis.*



2. Press: **I/O**

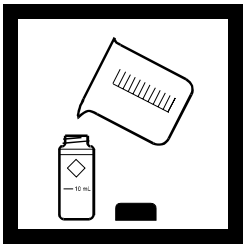
The display will show **550 nm** and module number **55.01**

Note: *Determination of total copper needs a prior digestion; see Digestion (Section 1) for digestion procedures.*



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **55.04.1**

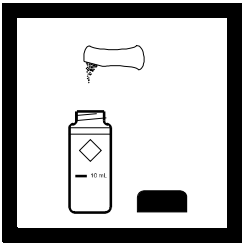
*Adapted from Nakano, S., *Yakugaku Zasshi, 82,* 486-491 [Chemical Abstracts, 58 3390e (1963)].
**Pretreatment required; see Interferences (Using Powder Pillows)
†Powder Pillows only; *Federal Register,* 45 (105) 36166 **(May 29, 1980)**



4. Fill a 10-mL sample cell to the 10-mL line with sample.

Note: *A 25-mL sample can be tested by using 25-mL sample cells and optional reagents.*

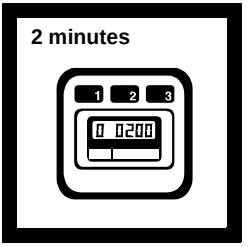
Note: *For proof of accuracy, use a 1.00 mg/L Copper Standard Solution (preparation in Accuracy Check) in place of the sample.*



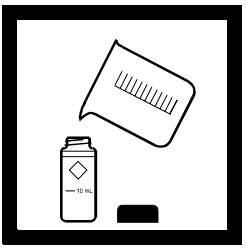
5. Add the contents of one CuVer 1 Copper Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert several times to mix.

Note: *A purple color will form if copper is present.*

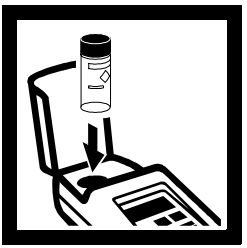
Note: *Accuracy is not affected by undissolved powder.*



6. Wait 2 minutes.

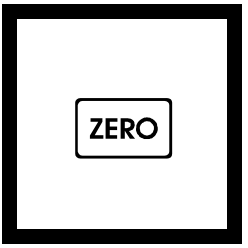


7. Fill a 10-mL cell to the 10-mL line with sample (the blank). Cap.



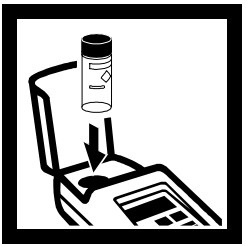
8. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



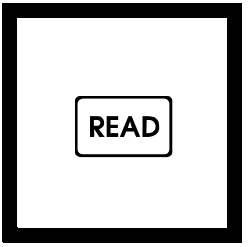
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



10. Place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*

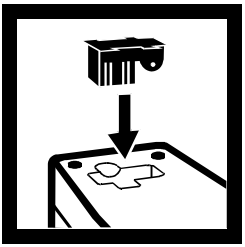


11. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L copper (Cu).

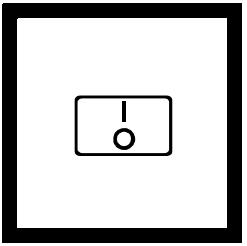
Note: *Determine a reagent blank for each new lot of reagent. Repeat Steps 4-11 using demineralized water as the sample. Subtract this value from each result obtained with this lot of reagent.*

USING ACCUVAC AMPULS



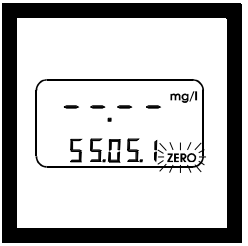
1. Install module **55.01** in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*



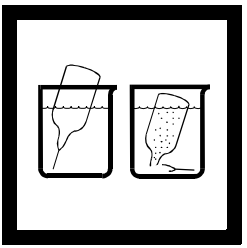
2. Press: **I/O**

The display will show **550 nm** and module number **55.01**



3. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **55.05.1**

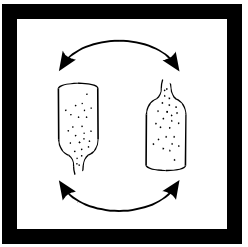
4. Fill a 10-mL cell to the 10-mL line with 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.



5. Fill a CuVer 2 AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*

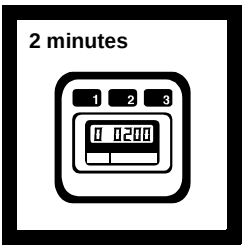
Note: *For proof of accuracy, use a 1.00 mg/L copper standard solution (preparation given in Accuracy Check) in place of the sample.*



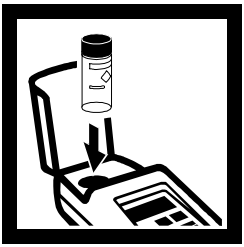
6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A purple color will form if copper is present.*

Note: *Accuracy is not affected by undissolved powder.*

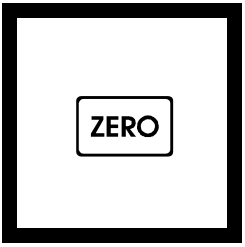


7. Wait 2 minutes.



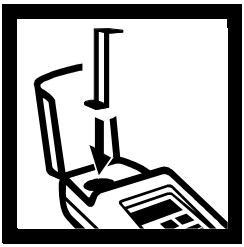
8. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*

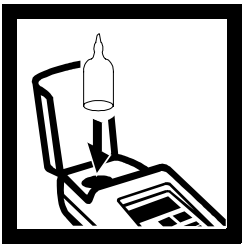


9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

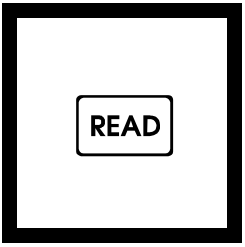


10. Insert the AccuVac Vial Adapter into the cell holder.



11. Place the prepared sample in the cell holder.

Note: In bright light it may be necessary to close the cell compartment cover.



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L copper.

Note: *Determine a reagent blank for each new lot of ampuls. Repeat Steps 4 to 11 using demineralized water as the sample. Subtract this value from each result obtained with this lot of ampuls.*

SAMPLING AND STORAGE

Collect samples in acid-cleaned glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Before analysis, adjust the pH to 4 to 6 with 8 N potassium hydroxide. Do not exceed pH 6, as copper may precipitate. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information. If only dissolved copper is to be determined, filter the sample before acid addition using the labware listed under Optional Apparatus.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off the Copper Voluette Ampule Standard Solution, 75 mg/L.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL samples. Mix each thoroughly. (For AccuVac Ampuls, use 50-mL beakers.)

c) Analyze each sample as described above. The copper concentration should increase 0.3 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Addition (Section I) for more information.

Standard Solution Method

Prepare a 1.00-mg/L copper standard by diluting 1.00 mL of Copper Standard Solution, 100 mg/L as Cu, to 100 mL with demineralized water. Prepare this solution daily.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 2.50 mg/L Cu concentration solutions, the standard deviation was ±0.012 mg/L Cu.

Testing zero concentration samples, the limit of detection was 0.014 mg/L Cu. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

Using two representative lots of AccuVacs, the standard deviation was ±0.011 mg/L Cu. and the limit of detection was 0.022 mg/L Cu.

INTERFERENCES (Using Powder Pillows)

If the sample is extremely acidic (pH 2 or less) a precipitate may form. Add 8 N Potassium Hydroxide Standard Solution drop-wise while swirling to dissolve the turbidity. Read the mg/L Cu.

If the turbidity remains and turns black, silver interference is likely. Eliminate silver interference by adding of 10 drops of saturated Potassium Chloride Solution to 75 mL of sample, followed by filtering through a fine or highly retentive filter. Use the filtered sample in the procedure.

Cyanide interferences prevent sufficient color development but can be overcome by adding 0.5 mL of formaldehyde to the sample. Wait four minutes before taking the reading. Multiply the test results by 1.02 to correct for sample dilution by the formaldehyde.

To test samples such as seawater containing high levels of hardness, iron, or aluminum, follow the powder pillow procedure using a 25-mL sample and substituting a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 pillow used in Step 5. Results obtained will include total dissolved copper (free and complexed).

To differentiate free copper from that complexed to EDTA or other complexing agents, use a Free Copper Reagent Powder Pillow in place of the CuVer 1 pillow in Step 5 and add to a 25-mL sample. Results will be free copper only. Add a Hydrosulfite Reagent Powder Pillow to the same sample and re-read the result. This result includes the total dissolved copper (free and complexed).

INTERFERENCES (Using AccuVac Ampuls)

The CuVer 2 Reagent contained in the AccuVac Ampuls is formulated to withstand high levels of calcium, iron and aluminum without interference.

Unlike CuVer 1 Reagent, CuVer 2 reacts directly with copper which is complexed by chelants such as EDTA. If free copper is to be determined separately from complexed copper, see the Powder Pillow Interference section above.

If the sample is very acidic, adjust to a pH greater than 4 before analysis. If a turbidity forms and turns black, silver interference is likely. This can be eliminated by adding 10 drops of saturated Potassium Chloride Solution to 75 mL of sample, followed by filtration through a fine filter using the labware listed under Optional Apparatus. Use the filtered sample in the procedure.

Cyanide interferences prevent sufficient color development but can be overcome by adding 0.5 mL of formaldehyde to the sample. Wait four minutes before taking the reading.

SUMMARY OF METHOD

Copper in the sample reacts with a salt of bicinchoninic acid contained in CuVer 1 or 2 Copper Reagent to form a purple colored complex in proportion to the copper concentration. This method includes procedures for both powder pillow and AccuVac reagents.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
CuVer 1 Copper Reagent			
Powder Pillows, 10 mL	1 pillow	100/pkg	21058-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

CuVer 2 Copper Reagent			
AccuVac Ampuls	1 ampul	25/pkg	25040-25

REQUIRED APPARATUS (Using Powder Pillow)

Clippers, for opening			
powder pillows	1	each	968-00
DR/700 Filter Module			
Number 55.01	1	each	46255-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac vial	1	each	46025-00
Beaker, 50 mL	1	each	500-41
DR/700 Filter Module			
Number 55.01	1	each	46255-00

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Copper Standard Solution, 100 mg/L	100 mL	128-42
Copper Standard Solution, Voluette ampule, 75 mg/L	16/pkg	14247-10
CuVer 1 Reagent Powder Pillows, 25-mL size	100/pkg	14188-99
CuVer 2 Reagent Powder Pillows, 25 mL	100/pkg	21882-99
Formaldehyde, 37%	100 mL* MDB	2059-32
Free Copper Reagent Powder Pillows	100/pkg	21186-69
Hydrochloric Acid Solution, 6 N	500 mL	884-49
Hydrosulfite Reagent Powder Pillows	100/pkg	21188-69
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Potassium Chloride Solution, saturated	59 mL SCDB	765-26
Potassium Hydroxide Standard Solution, 8.0 N	100 mL* MDB	282-32
Sodium Hydroxide Solution, 5.0 N	100 mL* MDB	2450-32
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

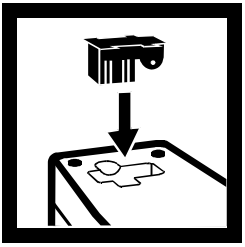
AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, polypropylene, 25 mL	each	1081-40
Cylinder, graduated, 100 mL	each	508-42
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, volumetric, 100 mL	each	547-42
Funnel, polypropylene, 65 mm	each	1083-67
Hot Plate, 3 1/2" diameter, 120 Vac	each	12067-01
Hot Plate, 3 1/2" diameter, 240 Vac	each	12067-02
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter,EC10, portable	each	50050-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

*Contact Hach for larger sizes.

DEHA (N,N-Diethylhydroxylamine) (0 to 600 µg/L)

For boiler water

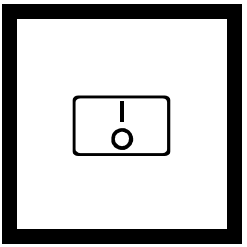
Iron Reduction Method for Oxygen Scavengers



1. Install module number
55.01
in a DR/700.

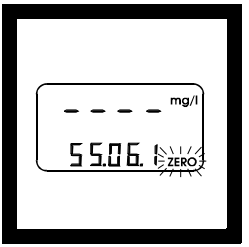
Note: *Samples must be analyzed immediately and cannot be store for later analysis.*

Note: *Other oxygen scavengers may be determined by this method if the result is multiplied by the appropriate factor. See Other Oxygen Scavengers following these steps.*



2. Press: **I/O**

The display will show
550 nm
and module number
55.01

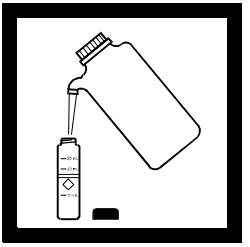


3. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
55.06.1

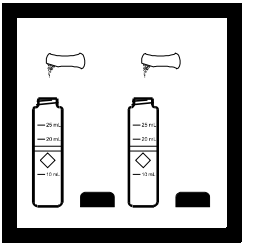
4. Fill a 25-mL cell to the 25-mL line with sample.

Note: *Rinse glassware with 1:1 Hydrochloric Acid Solution. Rinse again with demineralized water. These two steps will remove iron deposits which can cause slightly high results.*

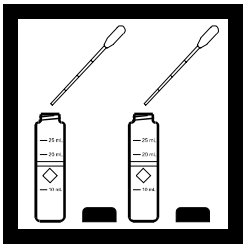
Note: *The sample temperature should be 25 ±3 °C (77 ±5 °F).*



5. Fill a second sample cell with 25 mL of demineralized water (the blank).

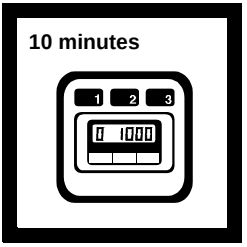


6. Add the contents of one DEHA Reagent 1 Powder Pillow to each sample cell. Cap and invert several times to mix.



7. Add exactly 0.5 mL of DEHA Reagent 2 Solution to each sample cell. Cap and invert several times to mix. Immediately place the sample cells in the dark.

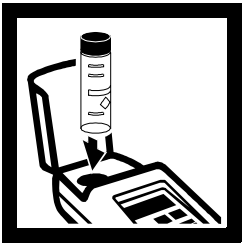
Note: *A purple color will slowly develop if DEHA is present.*



8. Immediately begin timing a 10-minute period.

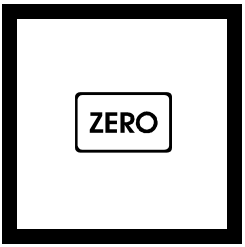
Note: *Temperature and reaction time affect the results. Be sure these factors are controlled as described.*

Note: *The sample cells must remain in the dark for the entire 10-minute period.*



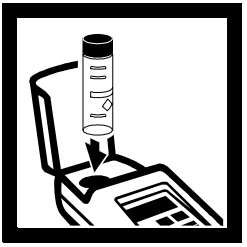
9. After the 10-minute period, place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



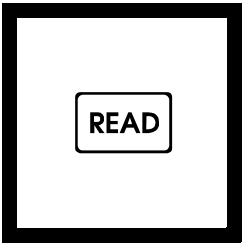
10. Press: **ZERO**

The display will count down to 0. Then the display will show 0 µg/L and the zero prompt will turn off.



11. Immediately place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in µg/L DEHA.

Note: *Repeat the above procedure, omitting Step 7, to determine the ferrous iron concentration in the sample. Subtract this result from those obtained in Step 12 to determine the actual DEHA concentration.*

OTHER OXYGEN SCAVENGERS

To determine other oxygen scavengers, perform the test as directed above; then multiply the DEHA result by the appropriate factor:

	Factor
Erythorbic Acid (Iso-ascorbic acid)	3.5
Hydroquinone	2.5
Methylethylketoxime	4.1

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 200 µg/L DEHA concentration solutions, the standard deviation was ±2.4 µg/L DEHA.

Testing zero concentration samples, the limit of detection was 3.1 µg/L DEHA. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

Substances which reduce ferric iron (such as other oxygen scavengers) will interfere. Substances which complex iron strongly may also interfere. The following may interfere when present in concentrations exceeding those listed below:

Borate (as $Na_2B_4O_7$)	500 mg/L
Cobalt	0.025 mg/L
Hardness (as $CaCO_3$)	1000 mg/L
Lignosulfonates	0.05 mg/L
Molybdenum	80 mg/L
Phosphate	10 mg/L
Phosphonates	10 mg/L
Sulfate	1000 mg/L
Zinc	50 mg/L

Light interferes with the color development.

SUMMARY OF METHOD

Diethylhydroxylamine (DEHA) or other oxygen scavengers present in the sample react with ferric iron in DEHA Reagent 2 Solution to produce ferrous ion in an amount equivalent to the DEHA concentration. This solution then reacts with DEHA 1 Reagent, which forms a purple color with ferrous iron.

Using this procedure other oxygen scavengers can be determined by multiplying the DEHA results by the appropriate multiplier.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
DEHA Reagent 1			
Powder Pillows	2 pillows	100/pkg	21679-69
DEHA Reagent 2 Solution	1 mL	500 mL	21680-49
Water, demineralized	5 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening			
powder pillows	1	each	968-00
Dropper, 0.5 and			
1.0-mL marks	1	10/pkg	21247-10
DR/700 Filter Module			
Number 55.01	1	each	46255-00

OPTIONAL REAGENTS

Hydrochloric Acid, 1:1 (6 N)	500 mL	884-49

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated,		
polypropylene, 25 mL	each	1081-40
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Thermometer, -20 to 105 °C	each	1877-01

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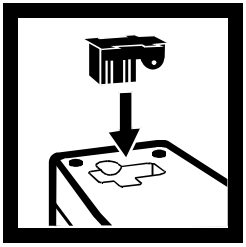
Outside the U.S.A. - Contact the Hach office or distributor serving you.

IRON (0 to 1.400 mg/L)

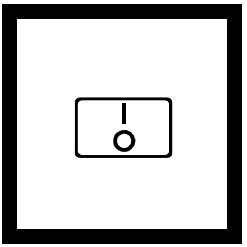
For water and seawater

FerroZine Method*



1. Install module
55.01
in a DR/700.

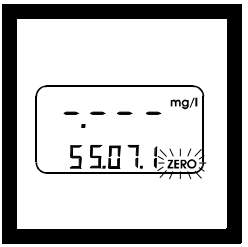
Note: *If samples cannot be analyzed immediately, see Sampling and Storage, following these steps. Adjust pH of stored samples before analysis.*



2. Press: **I/O**

The display will show
550 nm
and module number
55.01

Note: *Total iron determination needs prior digestion; use any of the three procedures given in Digestion (Section 1).*



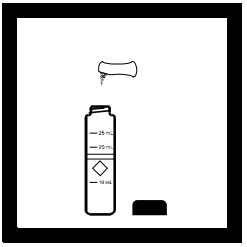
3. After 2 seconds, the display will show a program number, concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
55.07.1

*Adapted from Stookey, L.L., *Anal. Chem.*, 42 (7) 779 **(1970)**

4. Fill a 25-mL cell to the 25-mL line with sample.

Note: *Rinse glassware with a 1:1 hydrochloric acid solution. Rinse again with demineralized water. These two steps will remove iron deposits which can cause slightly higher results.*

Note: *For proof of accuracy, use a 0.4 mg/L iron standard solution (preparation given in the Accuracy Check) in place of the sample.*

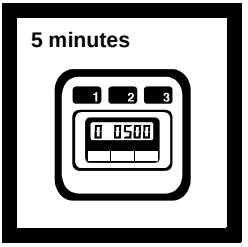


5. Add the contents of one FerroZine Iron Reagent Solution Pillow to the cell (the prepared sample). Cap and invert several times to mix.

Note: *Do not allow the clippers to come into contact with the contents of the pillow.*

Note: *0.5 mL of FerroZine Iron Reagent Solution can be used in place of the solution pillow if preferred.*

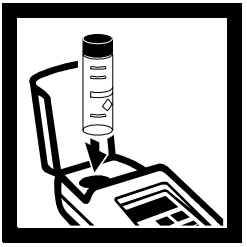
Note: *If the sample contains rust, see Interferences, below.*



6. Wait 5 minutes.

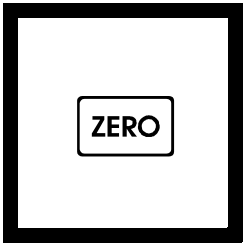
Note: *A violet color will develop if iron is present.*

7. Fill a 25-mL cell to the 25-mL line with sample (the blank). Cap.



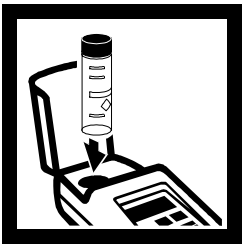
8. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



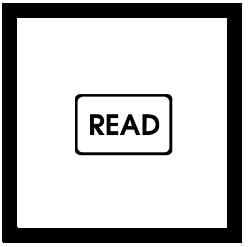
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.



10. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the sample, another 10-mL cell must be used for the blank.*



11. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L iron (Fe).

SAMPLING AND STORAGE

Collect samples in acid-washed glass or plastic bottles. To preserve samples, adjust the sample pH to 2 or less with nitric acid (about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature. If only dissolved iron is to be reported, filter sample immediately after collection and before addition of nitric acid.

Before testing, adjust the sample pH to 3 to 5 with ammonium hydroxide, ACS. Do not exceed pH 5 as iron may precipitate. Correct test results for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more detailed information.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off an Iron Volute Ampule Standard, 25 mg/L Fe.

b) Use the TenSette Pipet to add 0.1 mL of standard to the prepared sample measured in Step 11.

c) Swirl to mix and allow another five-minute reaction period, then measure the iron concentration as in Step 11.

d) Add two additional 0.1 mL standard increments, taking a concentration reading after allowing the five-minute reaction period for each.

e) Each additional 0.1 mL increment of standard added should cause a 0.1 mg/L increase in the concentration reading.

f) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

a) Pipet 1.00 mL of iron standard solution, 100 mg/L Fe, into a 250-mL volumetric flask.

b) Dilute to volume with demineralized water. This solution should be prepared daily. Analyze the working solution according to the above procedure.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 0.500 mg/L Fe concentration samples, the standard deviation was ±0.0024 mg/L Fe.

Testing zero concentration samples, the limit of detection was 0.0044 mg/L Fe. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

Copper and cobalt may interfere to give slightly high results.

EDTA interferes, use either the TPTZ or FerroVer method. The TPTZ method is suggested for low concentrations.

Any of the three digestions give in Digestion (Section I) used in place of the treatments given below will eliminate the following interferences.

If rust or hydroxides are present, the sample, with the FerroZine Iron Reagent from Step 5, should be boiled for one minute in a boiling water bath then cooled to 24 °C (75 °F) before proceeding with Step 6. The reduced sample volume should be returned to 25 mL with demineralized water.

If the sample contains magnetite (black iron oxide) or ferrites, perform the following procedure.

a) Fill a 25-mL graduated cylinder with 25 mL of sample.

b) Transfer the sample water into a 125-mL erlenmeyer flask.

c) Add the contents of one FerroZine Iron Reagent Solution Pillow and swirl to mix.

d) Place the flask on a hot plate or over a flame and bring to a boil.

e) Continue boiling gently for 20 to 30 minutes.

Note: *Do no allow to boil dry.*

Note: *A purple color will develop if iron is present.*

f) Return the boiled sample to the graduated cylinder. Rinse the erlenmeyer flask with small amounts of demineralized water and empty into the graduated cylinder.

g) Return the sample volume to the 25-mL mark with demineralized water.

h) Pour the solution into a sample cell and swirl to mix.

i) Proceed with Steps 6 through 11.

SUMMARY OF METHOD

The FerroZine Iron Reagent forms a purple-colored complex with trace amounts of iron in samples that are buffered to a pH of 3.5. This method is applicable for determining trace levels of iron in chemical reagents and glycols and can be used to analyze samples containing magnetite (black iron oxide) or ferrites.

REQUIRED REAGENTS AND APPARATUS

Description	Quantity Per Test	Unit	Cat. No.
FerroZine Iron Reagent Solution Pillows	1 pillow	50/pkg	2301-66
Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 55.01	1	each	46255-00

OPTIONAL REAGENTS

Ammonium Hydroxide, ACS	500 mL		106-49
Hydrochloric Acid Solution, 1:1 (6N)	500 mL		884-49
FerroZine Iron Reagent Solution	1000 mL		2301-53
Iron Standard Solution, 100 mg/L Fe	105 mL		14175-42
Iron Standard Solution, Volute ampule, 25 mg/L Fe, 10 mL	16/pkg		14253-10
Nitric Acid, ACS	500 mL		152-49
Nitric Acid Solution, 1:1	500 mL		2540-49
Water, demineralized	4 L		272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each		21968-00
Cap for 10 and 25-mL sample cells	12/pkg		24018-12
Cylinder, graduated, 25 mL	each		508-40
Dropper, calibrated, 0.5-mL & 1.0-mL mark	6/pkg		23185-06
Flask, erlenmeyer, 125 mL	each		505-43
Flask, erlenmeyer, 50 mL	each		505-41
Flask, volumetric, 250 mL, Class B	each		547-46
Hot plate, 3 $^1/_2$" diameter, 120 Vac	each		12067-01
Hot plate, 3 $^1/_2$" diameter, 240 Vac	each		12067-02
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg		391-33
pH Meter, EC 10, portable	each		50050-00

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 0.5 mL	each	14515-34
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Thermometer, -20 to 105 °C	each	1877-01

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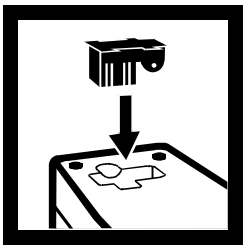
Outside the U.S.A. - Contact the Hach office or distributor serving you.

MANGANESE, LR (0 to 0.800 mg/L)

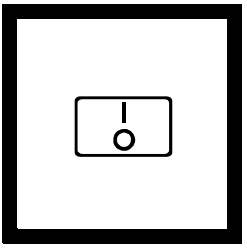
For water and wastewater

PAN Method* (Digestion required)



1. Install module
55.01
in a DR/700.

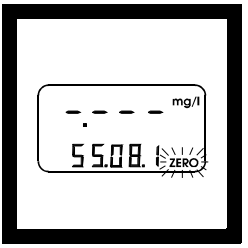
Note: *If samples cannot be analyzed immediately, see Sampling and Storage, below. Adjust the pH of stored samples before analysis.*



2. Press: **I/O**

The display will show
550 nm
and module
55.01

Note: *Total manganese determination requires a prior digestion; use any of the three digestion procedures given in Digestion (Section 1).*

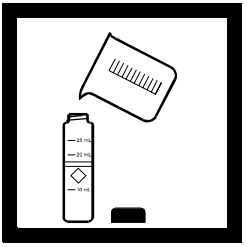


3. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
55.08.1

*Adapted from Goto, K., et al., *Talanta*, 24, 752-3 **(1977)**

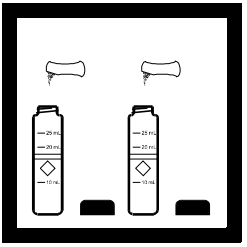
4. Pour 25 mL of demineralized water into a sample cell (the blank).

Note: *Rinse all glassware with 1:1 Nitric Acid Solution. Rinse again with demineralized water.*



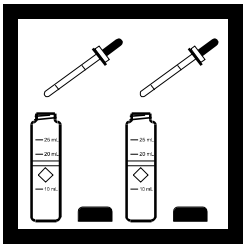
5. Pour 25 mL of sample into another sample cell (the prepared sample).

Note: *For proof of accuracy, a 0.5 mg/L standard solution (preparation given in the Accuracy Check) can be used in place of the sample.*



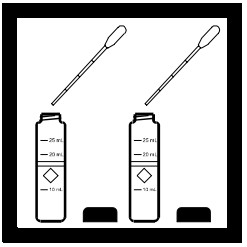
6. Add the contents of one Ascorbic Acid Powder Pillow to each cell. Cap and invert several times to mix.

Note: *For samples containing appreciable hardness (greater than 300 mg/L $CaCO_3$), add ten drops of Rochelle Salt Solution to the sample after addition of the Ascorbic Acid Powder Pillow.*



7. Add 1.0 mL of Alkaline-Cyanide Reagent Solution to each cell. Swirl to mix.

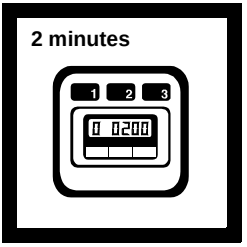
Note: *A cloudy or turbid solution may form in some samples after addition of the Alkaline-Cyanide Reagent Solution. The turbidity should dissipate after Step 8.*



8. Add 1.0 mL of 0.1% PAN Indicator Solution to each sample cell. Cap and invert several times to mix.

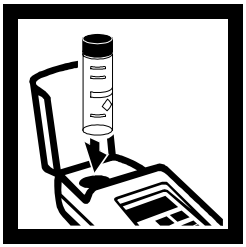
Note: *An orange color will develop if manganese is present.*

Note: *Use plastic dropper supplied because droppers with rubber bulbs may contaminate solution.*



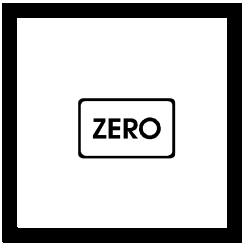
9. Wait 2 minutes.

Note: *If sample contains high amounts of iron (greater than 5 mg/L), allow 10 minutes for complete color development.*



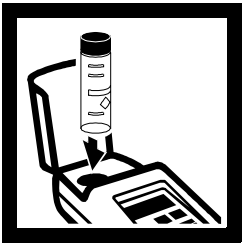
10. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If a 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



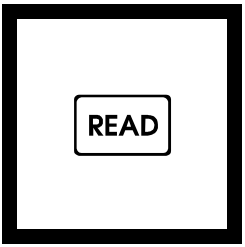
11. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



12. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If a 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



13. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L manganese.

Note: *See Waste Disposal below for proper disposal of cyanide containing wastes.*

Note: *To convert results to other units, see table.*

Table 1. Conversion Factors

To convert reading from	To	Multiply by
mg/L Mn	mg/L MnO_4-	2.16
mg/L Mn	mg/L $KMnO_4$	2.88

SAMPLING AND STORAGE

Collect samples in a clean glass or plastic container. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the pH to 4.0 to 5.0 with 5.0 N sodium hydroxide. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Manganese Voluette Ampule Standard, 25 mg/L Mn^{2+}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 25-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The manganese concentration should increase 0.1 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method
Prepare a 0.5 mg/L manganese standard solution as follows:

a) Pipet 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask.

b) Dilute to the mark with demineralized water. This solution should be prepared daily.

c) Pipet 10 mL of the above dilution into a 100-mL volumetric flask.

d) Dilute to the mark with demineralized water. This second dilution is equivalent to 0.5 mg/L Mn.

e) Perform the manganese procedure as described above. The reading in Step 12 should be 0.5 mg/L Mn.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 0.500 mg/L Mn concentration samples, the standard deviation was ±0.0029 mg/L Mn.

Testing zero concentration samples, the limit of detection was 0.0022 mg/L Mn. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980**, 52, 2242-2249)

INTERFERENCES

The following do not interfere up to the indicated concentrations:

Aluminum	20 mg/L
Cadmium	10 mg/L
Calcium	1000 mg/L as $CaCO_3$
Cobalt	20 mg/L
Copper	50 mg/L
Iron	25 mg/L
Lead	0.5 mg/L

Magnesium	300 mg/L as $CaCO_3$
Nickel	40 mg/L
Zinc	15 mg/L

WASTE DISPOSAL

Dispose of all spent cyanide-containing wastes by following the steps below.

a) Pour the waste into a large beaker and make alkaline (pH 11) with calcium hypochlorite or sodium hypochlorite (bleach). **Use good ventilation or a fume hood.**

b) Maintain an excess of sodium hydroxide and calcium hypochlorite. Let stand for 24 hours.

c) With a large excess of water, flush the solution down the drain.

SUMMARY OF METHOD

The PAN method is a highly sensitive and rapid procedure for detecting low levels of manganese. An ascorbic acid reagent is used initially to reduce all oxidized forms of manganese to Mn^{2+}. An alkaline-cyanide reagent is added to mask any potential interferences. PAN Indicator is then added to combine with the Mn^{2+} to form an orange-colored complex.

REQUIRED REAGENTS

	Cat. No.
Manganese Reagent Set (100 Tests)	22433-00
Includes: (2) 21223-37, (2) 14577-99, (2) 21224-39	

Description	Quantity Per Test	Unit	Cat. No.
Alkaline-Cyanide Reagent	2 mL	100 mL	21223-32
Ascorbic Acid Powder Pillows	2 pillows	100/pkg.	14577-99
PAN Indicator Solution, 0.1%	2 mL	118 mL	21224-39

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00

REQUIRED APPARATUS (continued)

Description	Quantity Per Test	Unit	Cat. No.
DR/700 Filter Module Number 55.01	1	each	46255-00

OPTIONAL REAGENTS

Hydrochloric Acid Solution, 1:1 (6 N)	500 mL		884-49
Manganese Standard Solution, 1000 mg/L Mn	100 mL		12791-42
Manganese Standard Solution, Voluette ampule, 25 mg/L Mn, 10 mL	16/pkg		21128-10
Nitric Acid Solution, 1:1	500 mL		2540-49
Nitric Acid, ACS	500 mL		152-49
Rochelle Salt Solution	29 mL		1725-33
Sodium Hydroxide Solution, 50%	500 mL		2180-11
Sodium Hydroxide, 5 N	1 L		2450-53
Water, demineralized	4 L		272-56

OPTIONAL APPARATUS

Beaker, glass, 1000 mL		each	500-53
Cap for 10- and 25-mL sample cells		12/pkg	24018-12
Dropper, plastic calibrated, 1.0 mL		10/pkg	21247-10
Flask, volumetric, Class A, 1000 mL		each	14574-53
Flask, volumetric, Class A, 100 mL		each	14574-42
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet, volumetric, 10.00 mL		each	14515-38
Pipet, volumetric, 5.00 mL, Class A		each	14515-37
Pipet Filler,		each	14651-00
Sample Cell, 10-mL with screw cap		6/pkg	24276-06
Sample Cell, 25-mL with screw cap		6/pkg	24019-06

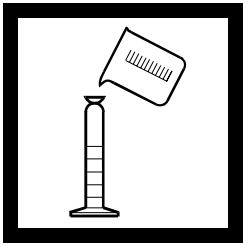
For Technical Assistance, Prices and Ordering

In the U.S.A - Call 800-227-4224 toll-free for more information.
Outside the U. S. A. - Contact the Hach office or distributor serving you.

NICKEL (0 to 1.000 mg/L)

For water and wastewater

1-(2-Pyridylazo)-2-Naphthol (PAN) Method*

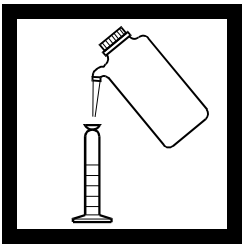


1. Measure 25 mL of sample in a mixing graduated cylinder. This will be the prepared sample.

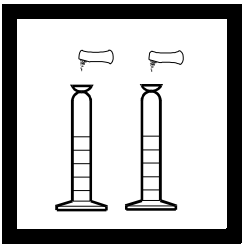
Note: *If sample is less that 10°C (50°F), warm to room temperature before analysis.*

Note: *If sample cannot be analyzed immediately, see Sampling and Storage, following these steps. Adjust pH of stored samples before analysis.*

Note: *For proof of accuracy, use a 0.5 mg/L Nickel Standard Solution (preparation given in Accuracy Check) in place of the sample.*



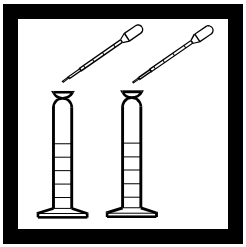
2. Measure 25 mL of demineralized water in a second cylinder (the blank).



3. Add the contents of one Phthalate-Phosphate Reagent Powder Pillow to each cylinder. Stopper. Immediately shake to dissolve.

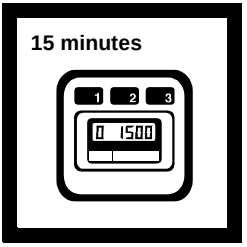
Note: *If sample contains iron (Fe^{3+}), it is important that all powder be dissolved completely before continuing with Step 4.*

* Adapted from Watanabe, H., *Talanta*, 21 295 **(1974)**



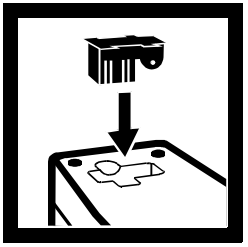
4. Add 1.0 mL of PAN Indicator Solution, 0.3%, to each cylinder. Stopper. Invert several times to mix.

Note: *Use the plastic dropper provided.*

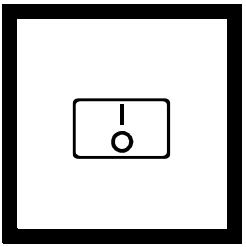


5. Wait 15 minutes. Steps 6 through 8 can be performed during this waiting period.

Note: *During color development, the sample solution color may vary from yellowish-orange to dark red, depending on the chemical make-up of the sample. The demineralized water blank should be yellow.*

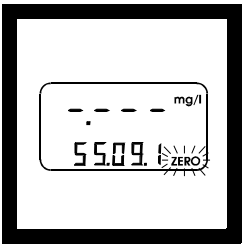


6. Install module **55.01** in a DR/700.

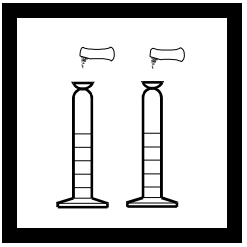


7. Press: **I/O**

The display will show **550 nm** and module number **55.01**

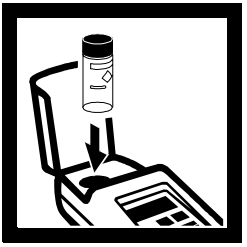


8. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **55.09.1**



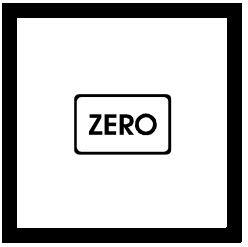
9. Add the contents of one EDTA Reagent Powder Pillow to each cylinder. Stopper and shake to dissolve.

10. Fill a 10 mL cell to the 10-mL line with the blank solution.



11. Place the blank in the cell holder.

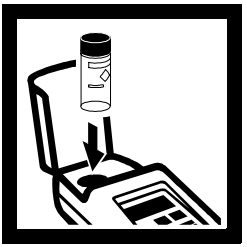
Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



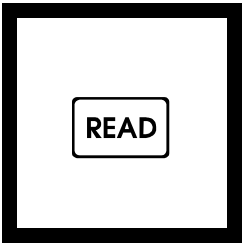
12. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.

13. Fill a second 10-mL cell to the 10-mL line with the prepared sample.



14. Place the prepared sample in the cell holder.



15. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L nickel (Ni).

SAMPLING AND STORAGE

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 5 mL per liter). Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the sample pH to between 3 to 8 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 8 as this may cause some loss of nickel as a precipitate. Correct test results for volume additions, see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Solution Method

Prepare a 0.5 mg/L nickel standard solution by diluting 10.0 mL of a 5-mg/L working stock solution to 100 mL in a 100-mL volumetric flask. The working stock solution should be prepared daily by diluting 5.00 mL of Nickel Standard Solution, 1000 mg/L as Ni, to 1000 mL with demineralized water.

Or, using the TenSette Pipet, add 0.2 mL of a Voluette Ampule Standard Solution for Nickel, 300 mg/L Ni, into a 100-mL volumetric flask. Dilute to volume with demineralized water. This is a 0.6 mg/L standard solution.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 0.600 mg/L Ni concentration samples, the standard deviation was ±0.0043 mg/L Ni.

Testing zero concentration samples, the limit of detection was 0.0043 mg/L Ni. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry*, **1980,** 52, 2242-2249).

INTERFERENCES

The following may interfere when present in concentrations exceeding those listed below:

Al^{3+}	32 mg/L
Ca^{2+}	1000 mg/L as ($CaCO^3$)
Cd^{2+}	20 mg/L
Cl^-	8000 mg/L

Cr^{3+}	20 mg/L
Cr^{6+}	40 mg/L
Cu^{2+}	15 mg/L
F^{-}	20 mg/L
Fe^{3+}	10 mg/L
Fe^{2+} and CO^{2+}	interferes directly and must not be present.
K^{+}	500 mg/L
Mg^{2+}	400 mg/L
Mn^{2+}	25 mg/L
Mo^{6+}	60 mg/L
Na^{+}	5000 mg/L
Pb^{2+}	20 mg/L
Zn^{2+}	30 mg/L

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and required sample pretreatment; see Interferences, pH (Section I).

Chelating agents, such as EDTA, interfere. Use either the Digesdahl or vigorous digestion (Section I) to eliminate this interference.

SUMMARY OF METHOD

After buffering the sample and masking any Fe^{3+} with pyrophosphate, the nickel is reacted with 1-(2-Pyridylazo)-2-Naphthol indicator. The indicator forms complexes with most metals present. After color development, EDTA is added to destroy all metal-PAN complexes except nickel and cobalt. Cobalt must be absent from samples.

REQUIRED REAGENTS

	Cat. No.
Nickel Reagent Set (100 Tests*)	22426-00
Includes: (4) 7005-66, (4) 21501-66, (2) 21502-37	

Description	Quantity Per Test	Unit	Cat. No.
EDTA Reagent			
Powder Pillows	2 pillows	50/pkg	7005-66
Phthalate-Phosphate Reagent			
Powder Pillows	2 pillows	50/pkg	21501-66

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
P.A.N. Indicator Solution, 0.3%	2 mL	100 mL	21502-32
Water, demineralized	25 mL	3.78L	272-17

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, 25 mL	2	each	20886-40
DR/700 Filter Module Number 55.01	1	each	46255-00

OPTIONAL REAGENTS

Nickel Standard Solution, 1000 mg/L Ni	100 mL	14176-42
Nickel Standard Solution, Voluette ampule, 300 mg/L Ni, 10 mL	16/pkg	14266-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL	2450-32

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 1000 mL	each	14574-53
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, serological, 1 mL	each	532-35
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 5.0 mL	each	14515-37
Pipet, volumetric, Class A, 10.0 mL	each	14515-38
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL, with screw cap	6/pkg	24276-06
Sample Cell, 25-mL, with screw cap	6/pkg	24019-06

*100 Tests equals 50 sample and 50 blanks. Contact Hach for larger sizes.

Module 57.01
575 nm

Hach warrants equipment it manufactures against defective materials or workmanship for at least one year from the shipping date. Warranties do not apply to limited-life electrical components such as batteries. Full warranty information is located on the reverse side of Hach invoices.

IMPORTANT NOTICE

A lamp intensity adjustment must be performed:
- Before first or initial use
- When new filter modules are installed
- On each filter module when the lamp is replaced

Refer to Lamp Intensity Adjustment in the instrument manual.

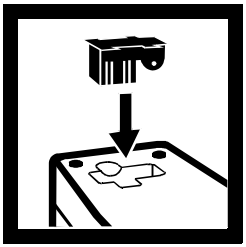
Table of Contents for 575-nm parameters

FLUORIDE (0 to 2.0 mg/L F^-)

For water, wastewater and seawater

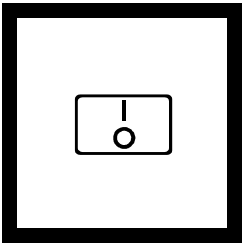
SPADNS Method* (Reagent or Ampules); USEPA accepted for reporting (distillation is required; see Section 1)**

USING SPADNS REAGENT SOLUTION



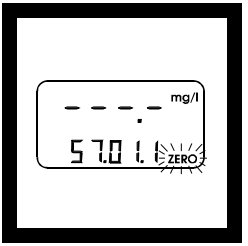
1. Install module
57.01
in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*



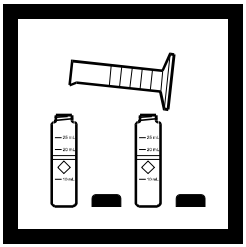
2. Press: **I/O**

The display will show
575 nm
and the module number
57.01



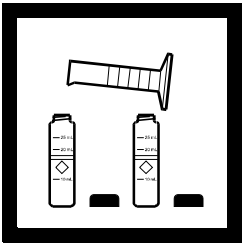
3. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows the program number
57.01.1

*Adapted from *Standard Methods for the Examination of Water and Wastewater*
**Procedure is equivalent to USEPA method 340.1 for drinking water and wastewater.



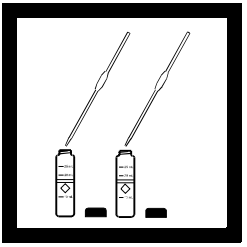
4. Using a graduated cylinder or pipet, measure 25.0 mL of sample into a dry 25-mL sample cell.

Note: *For proof of accuracy, use a 1.0 mg/L fluoride standard solution (listed under Optional Reagents) in place of the sample.*



5. Using a graduated cylinder or pipet, measure 25.0 mL of demineralized water into another dry sample cell (the blank).

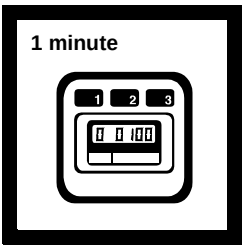
Note: *The sample and demineralized water should be at the same temperature (±1 °C). Makw temperature adjustments before or after reagent addition.*



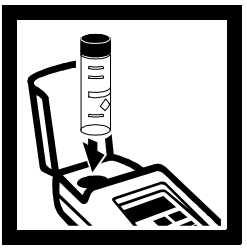
6. Pipet 5.0 mL of SPADNS Reagent into each cell. Use a pipet filler. Cap each cell. Invert each cell several times to mix.

Note: *SPADNS Reagent is toxic and corrosive; use care while measuring.*

Note: *The SPADNS Reagent must be measured accurately.*

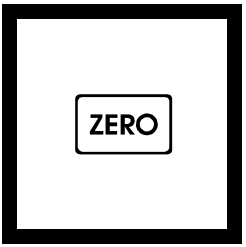


7. Wait 1 minute.



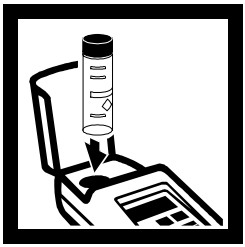
8. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*



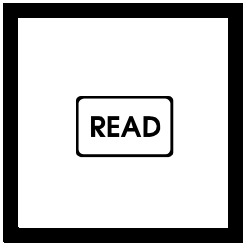
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L and the zero prompt will turn off.



10. Place the prepared sample in cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment open. In bright sunlight, it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank and 10 mL of the sample to a 10-mL sample cell and proceed.*

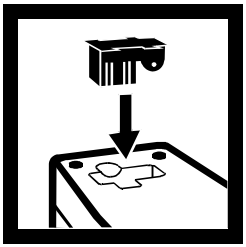


11. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L fluoride (F^-).

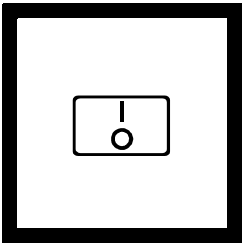
Note: *If the instrument flashes the upper range limit, dilute the sample with an equal volume of demineralized water and repeat the test, using this solution in Step 4. Multiply the result by 2.*

USING ACCUVAC AMPULS



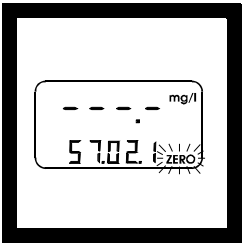
1. Install module **57.01** in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

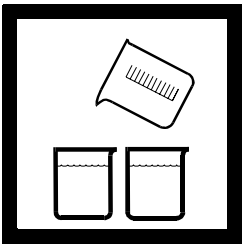


2. Press **I/O**

The display will show **575 nm** and module number **57.01**



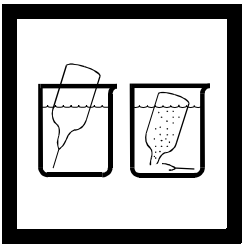
3. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **57.02.1**



4. Collect at least 40 mL of sample in a 50-mL beaker. Fill a second beaker with at least 40 mL of demineralized water.

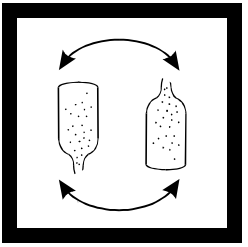
Note: *For proof of accuracy, use a 1.0 mg/L fluoride standard solution (listed under Optional Reagents) in place of the sample.*

Note: *The sample and demineralized water should be at the same temperature (±1 °C).*

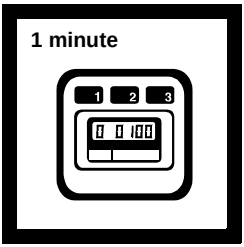


5. Fill a SPADNS Fluoride AccuVac Ampul with sample. Fill a second SPADNS Fluoride AccuVac with the demineralized water (the blank).

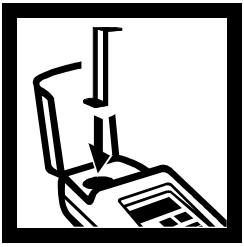
Note: *Keep tip immersed while the ampul fills completely.*



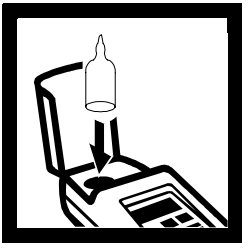
6. Quickly invert the ampuls several times to mix. Wipe off any liquid or fingerprints.



7. Wait 1 minute.

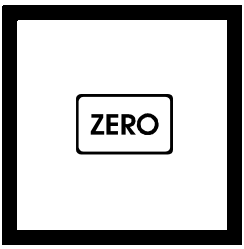


8. Insert the AccuVac Vial Adapter into the cell holder.



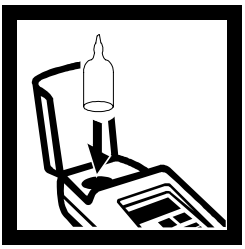
9. Place the blank in the cell holder.

Note: *In bright light, it may be necessary to close the cell compartment cover.*



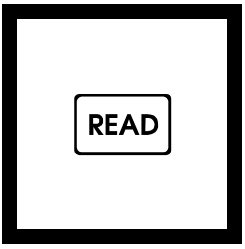
10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L and the zero prompt will turn off.



11. Place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L fluoride.

Note: *If the instrument flashes the upper range limit, dilute the sample with an equal volume of demineralized water and repeat the test, using this solution in Step 4. Multiply the result by 2.*

SAMPLING AND STORAGE

Samples may be stored in glass or plastic bottles for at least seven days when cooled to 4 °C (39 °F) or lower. Warm samples to room temperature before analysis.

ACCURACY CHECK

Standard Solution Method

A variety of standard solutions covering the entire range of the test is available from Hach. Use these in place of sample to verify technique. Minor variations between lots of reagent become measurable above 1.5 mg/L. While results in this region are usable for most purposes, better accuracy may be obtained by diluting a fresh sample 1:1 with demineralized water and retesting. Multiply the result by 2.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 2.00 mg/L F^- concentration solutions, the standard deviation was ±0.03 mg/L F^-.

Testing zero concentration samples, the limit of detection was 0.04 mg/L F^-. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

Using two representative lots of AccuVacs, the standard deviation was ±0.04 mg/L F^- and the limit of detection was 0.06 mg/L F^-.

INTERFERENCES

This test is sensitive to small amounts of interference. Glassware must be very clean. Repeating the test with the same glassware is recommended to ensure that results are accurate.

The following substances interfere to the extent shown:

	Concentration	Error
Alkalinity (as $CaCO_3$)	5000 mg/L	-0.1 mg/L F^-
Aluminum	0.1 mg/L	-0.1 mg/L F^-
Chloride	7000 mg/L	+0.1 mg/L F^-
Iron, ferric	10 mg/L	-0.1 mg/L F^-
Phosphate, ortho	16 mg/L	+0.1 mg/L F^-

Sodium Hexametaphosphate	1.0 mg/L	+0.1 mg/L F^-
Sulfate	200 mg/L	+0.1 mg/L F^-

SPADNS Reagent contains enough arsenite to eliminate interference up to 5 mg/L chlorine. For higher chlorine levels, add one drop of Sodium Arsenite Solution to 25 mL of sample for each 2 mg/L of chlorine.

To check for interferences from aluminum, read the concentration one minute after reagent addition, then again after 15 minutes. An appreciable increase in concentration suggests aluminum interference. Waiting two hours before making the final reading will eliminate the effect of up to 3.0 mg/L aluminum.

DISTILLATION PROCEDURE

Most interferences can be eliminated by distilling the sample from an acid solution as described below:

a) Set up the distillation apparatus for the general purpose distillation. See the Hach Distillation Apparatus Manual. Turn on the water and make certain it is flowing through the condenser.

b) Measure 100 mL of sample into the distillation flask. Add a magnetic stirring bar and turn on the heater power switch. Turn the stir control to 5. Cautiously measure 150 mL of StillVer Distillation Solution (2:1 Sulfuric Acid) into the flask. If high levels of chloride are present, add 5 mg silver sulfate for each mg/L chloride present.

c) Turn the heat control to setting 10, with the thermometer in place. The yellow pilot lamp shows when the heater is on.

d) When the temperature reaches 180 °C (about one hour), turn the still off. Analyze the distillate by the above method.

SUMMARY OF METHOD

The SPADNS Method for fluoride determination involves the reaction of fluoride with a red zirconium-dye solution. The fluoride combines with part of the zirconium to form a colorless complex, thus bleaching the red color in an amount proportional to the fluoride concentration. This method is approved by the USEPA for NPDES and NPDWR reporting purposes when the samples have been distilled. Seawater and wastewater samples require distillation. See Optional Apparatus for distillation apparatus listing.

REQUIRED REAGENTS (Using Solution)

Description	Quantity Per Test	Unit	Cat. No.
SPADNS Reagent for Fluoride	10 mL	500 mL	444-49
Water, demineralized	25 mL	4 L	272-56

REQUIRED APPARATUS (Using Solution)

Cylinder, graduated, 25 mL	1	each	508-40
DR/700 Filter Module Number 57.01	1	each	46257-00
Pipet Filler, safety bulb	1	each	14651-00
Pipet, volumetric, Class A, 5.00 mL	1	each	14515-37
Thermometer, -20 to 105°C	1	each	1877-01

REQUIRED REAGENTS (Using AccuVac Ampuls)

SPADNS Fluoride Reagent AccuVac Ampuls	2 ampuls	25/pkg	25060-25
Water, demineralized	varies	4 L	272-56

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac vial	1	each	43784-00
Beaker, 50 mL	2	each	500-41
DR/700 Filter Module Number 57.01	1	each	46257-00

OPTIONAL REAGENTS

Fluoride Standard Solution, 0.2 mg/L F^-	500 mL		405-02
Fluoride Standard Solution, 0.4 mg/L F^-	500 mL		405-04
Fluoride Standard Solution, 0.5 mg/L F^-	500 mL		405-05
Fluoride Standard Solution, 0.6 mg/L F^-	500 mL		405-06
Fluoride Standard Solution, 0.8 mg/L F^-	500 mL		405-08
Fluoride Standard Solution, 1.0 mg/L F^-	1 L		291-53
Fluoride Standard Solution, 1.0 mg/L F^-	500 mL		291-49
Fluoride Standard Solution, 1.2 mg/L F^-	500 mL		405-12
Fluoride Standard Solution, 1.4 mg/L F^-	500 mL		405-14
Fluoride Standard Solution, 1.5 mg/L F^-	500 mL		405-15
Fluoride Standard Solution, 1.6 mg/L F^-	500 mL		405-16
Fluoride Standard Solution, 1.8 mg/L F^-	500 mL		405-18
Fluoride Standard Solution, 2.0 mg/L F^-	500 mL		405-20

OPTIONAL REAGENTS (continued)

Description	Unit	Cat. No.
Silver Sulfate, ACS	113 g	334-14
Sodium Arsenite Solution	100 mL MDB	1047-32
StillVer Distillation Solution	500 mL	446-49

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac Vial, DR/700	each	46025-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, 100 mL	each	508-42
Cylinder, graduated, 250 mL	each	508-46
Distillation Heater and Support Apparatus Set, 115 V, 50/60 Hz	each	22744-00
Distillation Heater and Support Apparatus Set, 230 V, 50/60 Hz	each	22744-02
Distillation Apparatus General Purpose Accessories	each	22653-00
pH Meter, EC10, portable	each	50050-00
Pipet, Volumetric, 25 mL	each	515-40
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

Fluoride can also be determined directly at these levels with the Fluoride Ion Selective Electrode and pH/ISE Meter.

Fluoride ISE Analysis Package	each	13034-01
pH Meter, EC20	each	50075-00

For Technical Assistance, Prices and Ordering

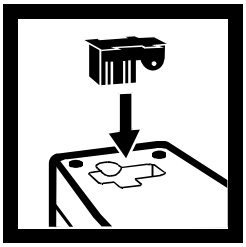
In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

IRON, TOTAL (0 to 1.00 mg/L)

For water, wastewater and seawater

TPTZ Method* (Powder Pillows or AccuVac Ampuls)

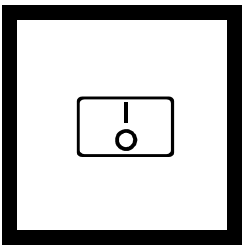
USING POWDER PILLOWS



1. Install module
57.01
in a DR/700.

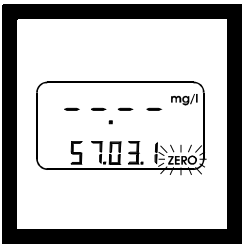
Note: *If samples cannot be analyzed immediately, see Sampling and Storage, below. Adjust pH of stored samples before analysis.*

Note: *Total iron determination needs a prior digestion. Use any of the three procedures given in Digestion (Section I).*



2. Press: **I/O**

The display will show
575 nm
and module
57.01



3. After 2 seconds the display will show a program number, concentration units, the decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
57.03.1

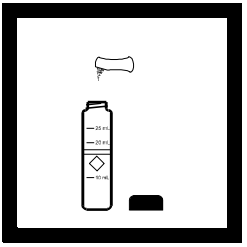
Note: *Rinse glassware with a 1:1 Hydrochloric Acid Solution. Rinse again with demineralized water. These two steps will remove iron deposits which can cause slightly high results.*

*Adapted from G. Frederick Smith Chemical Co., *The Iron Reagents,* 3rd ed. **(1980)**

4. Fill a 25-mL cell to the 25-mL line with sample.

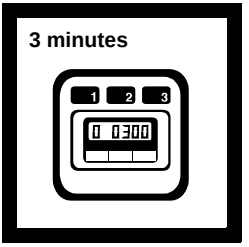
Note: *For proof of accuracy, use a 0.4 mg/L iron standard solution (preparation given in the Accuracy Check) in place of the sample.*

Note: *Sample pH is important in this test, see pH discussion in Interferences (Section 1).*



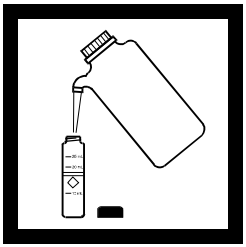
5. Add the contents of one TPTZ Iron Reagent Powder Pillow (the prepared sample). Cap. Shake for 30 seconds.

Note: *A blue color will develop if iron is present.*

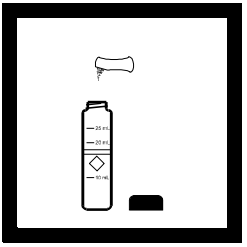


6. Wait 3 minutes.

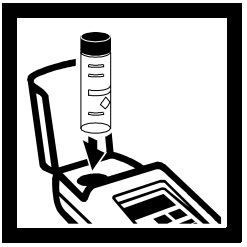
Note: *Continue with steps 7 and 8 while the timer is running.*



7. Fill a 25-mL cell to the 25-mL line with demineralized water.

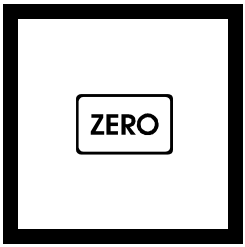


8. Add the contents of one TPTZ Iron Reagent Powder Pillow to the demineralized water (the blank). Cap. Shake for 30 seconds.



9. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it might be necessary to close the cell compartment cover. Transfer 10 mL of the prepared sample to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*

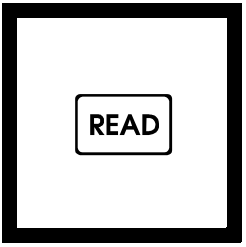


10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

11. Within 30 minutes after the 3-minute waiting period, place the prepared sample in the cell holder.

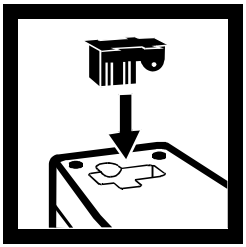
Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it might be necessary to close the cell compartment cover. Transfer 10 mL of the prepared sample to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



12. Press: **READ**

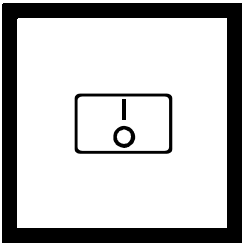
The display will count down to 0. Then the display will show the result in mg/L Iron (Fe).

USING ACCUVAC AMPULS



1. Install module
57.01
in a DR/700.

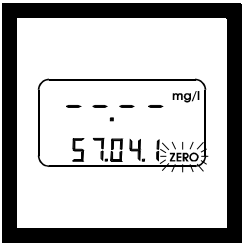
Note: *If sample cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust pH of stored samples before analysis.*



2. Press: **I/O**

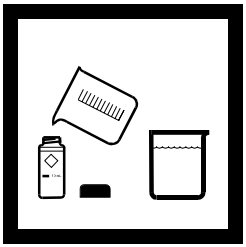
The display will show
575 nm
and module number
57.01

Note: *Total iron determination needs a prior digestion. Use any of the three procedures given in Digestion (Section 1).*



3. After 2 seconds, the display will show a program number, concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
57.04.1

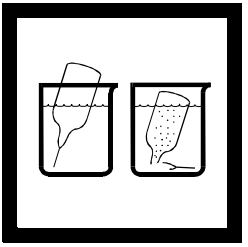
Note: *Rinse glassware with a 1:1 Hydrochloric Acid Solution. Rinse again with demineralized water. These two steps will remove iron deposits which can cause slightly high results.*



4. Fill a cell with 10 mL of sample (the blank). Cap. Collect at least 40 mL of sample in a 50 mL beaker.

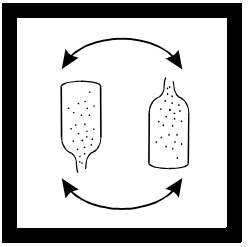
Note: *For proof of accuracy, a 0.4 mg/L iron standard solution (preparation given in the Accuracy Check) can be used in place of the sample.*

Note: *Sample pH is important in this test; see pH discussion in Interferences (Section I).*



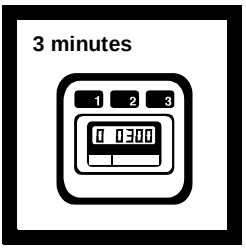
5. Fill a TPTZ Iron AccuVac Ampul with sample.

Note: *Keep tip immersed while the ampul fills completely.*

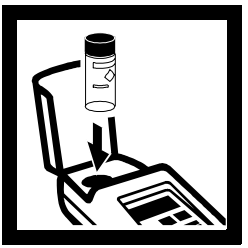


6. Invert the ampul (the prepared sample) repeatedly to mix. Wipe off any liquid or fingerprints.

Note: *A blue color will develop if iron is present.*

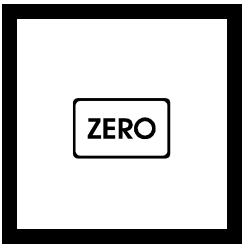


7. Wait 3 minutes.



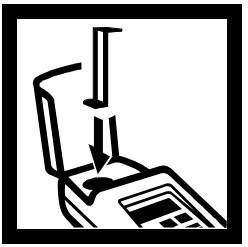
8. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

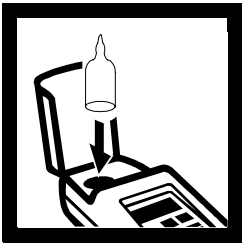


9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

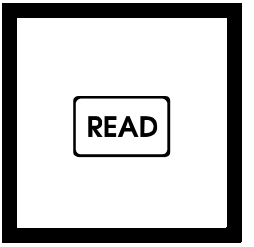


10. Insert the AccuVac Vial Adapter into the cell holder.



11. Within 30 minutes after the 3-minute waiting period, place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L iron (Fe).

Note: *For most accurate results, run the procedure using iron-free demineralized water, instead of sample, in Step 4. Subtract the value obtained in Step 12 from all later tests. Repeat for each new lot of AccuVac reagent.*

SAMPLING AND STORAGE

Collect samples in acid-washed glass or plastic bottles. Adjust the sample pH to 2 or less with Nitric Acid (about 2 mL per L). Store samples preserved in this manner up to six months at room temperature. If reporting only dissolved iron, filter the sample immediately after collection and before addition of nitric acid.

Before testing, adjust the pH of the stored sample to between 3 and 4 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 5 as iron may precipitate. Correct the test result for volume addition; see Sampling and Storage, Volume Additions (Section I).

ACCURACY CHECK

Standard Additions Method

Using AccuVac Ampuls

a) Measure 25.0 mL of sample using a graduated cylinder into each of three 50-mL beakers.

b) Snap the neck off a Volute Ampule Standard for Iron, 25 mg/L Fe.

c) Add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three 50-mL beakers using the TenSette Pipet. Swirl to mix.

d) Fill a TPTZ Iron AccuVac Ampul completely from each beaker.

e) Measure the concentration of each ampul according to the above procedure. The iron concentration reading should increase by 0.1 mg/L for each 0.1 mL addition of standard.

f) If these increases does not occur, see Standard Additions, (Section I) for more information.

Standard Solutions Method

Prepare a 0.4 mg/L iron working solution as follows:

a) Pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a 250-mL volumetric flask.

b) Dilute to volume with demineralized water. Prepare this solution fresh daily. Analyze the working solution according to the above procedure.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 0.75 mg/L Fe concentration samples, the standard deviation was ±0.035 mg/L Fe.

Testing zero concentration samples, the limit of detection was 0.080 mg/L Fe. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry*, **1980,** 52,2242-2249).

Using two representative lots of AccuVac Ampuls, the standard deviation was ±0.019 mg/L Fe and the limit of detection was ±0.035 mg/L Fe.

INTERFERENCES

In the powder pillow procedure if the sample, (without TPTZ Iron Reagent) has a color or turbidity greater than the blank of Step 8 (demineralized water plus TPTZ Iron Reagent), use the sample as the blank.

A sample pH of less than 3 or greater than 4 after the addition of reagent may inhibit color formation, cause the developed color to fade quickly or result in turbidity. Adjust the sample pH in the sample cell before the addition of reagent to between 3 to 8 by using a pH meter or pH paper and adding dropwise an appropriate amount of iron-free acid or base such as 1.0 N Sulfuric Acid Standard Solution or 1.0 N Sodium Hydroxide Standards Solution. Make a volume correction if significant volumes of acid or base are used; see Sampling and Storage, Volume Additions (Section I).

Interference tests were performed using an iron concentration of 0.5 mg/L. When interferences occurred, the color formation was inhibited or a precipitate formed. The following do not interfere with the test when present up to the levels listed:

Cadmium	4.0 mg/L
Chromium ($^{3+}$)	0.25 mg/L
Chromium ($^{6+}$)	1.2 mg/L
Cobalt	0.05 mg/L
Copper	0.6 mg/L
Cyanide	2.8 mg/L
Manganese	50.0 mg/L
Mercury	0.4 mg/L
Molybdenum	4.0 mg/L
Nickel	1.0 mg/L
Nitrite Ion	0.8 mg/L

SUMMARY OF METHOD

The TPTZ Iron Reagent forms a deep blue-purple color with ferrous iron. The indicator is combined with a reducing agent which converts precipitated or suspended iron, such as rust, to the ferrous state. The amount of ferric iron present can be determined as the difference between the results of a ferrous iron test and the concentration of total iron.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
TPTZ Iron Reagent			
Powder Pillows	2 pillows	25/pkg	22756-68
Water, demineralized	25 mL	4 L	272-56

REQUIRED REAGENTS (Using AccuVac Ampuls)

TPTZ Low Range Iron Reagent			
AccuVac Ampuls	1 ampul	25/pkg	25100-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening			
powder pillows	1	each	968-00
DR/700 Filter Module			
Number 57.01	1	each	46257-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41
DR/700 Filter Module			
Number 57.01	1	each	46257-00

OPTIONAL REAGENTS

Hydrochloric Acid Solution, 1:1 6.0 N	500 mL		884-49
Iron Standard Solution, 100 mg/L Fe	118 mL		14175-42
Iron Standard Solution, Voluette Ampule,			
25 mg/L Fe, 10 mL	16/pkg		14253-10
Nitric Acid, ACS	500 mL		152-49
Nitric Acid Solution, 1:1	473 mL		2540-49
Sodium Hydroxide			
Standard Solution, 1.0 N	100 mL	MDB	1045-32
Sodium Hydroxide			
Standard Solution, 5.0 N	100 mL	MDB	2450-32
Sulfuric Acid Standard Solution	100 mL	MDB	1270-32
Water, demineralized	4 L		272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac Vial	each	46025-00
Beaker, 50 mL	each	500-41
Cap for 10 and 25-mL sample cells	12/pkg	24018-12

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
Cylinder, graduated 25 mL	each	1081-00
Dropper, graduated, 0.5 and 1.0 mL marks	each	21247-10
Flask, volumetric, Class A, 25 mL	each	14574-40
Flask, volumetric, Class A, 50 mL	each	14574-41
Flask, volumetric, Class A, 250 mL	each	14574-46
Flask, volumetric, Class A, 100 mL	each	14575-42
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet Filler, safety bulb	each	14651-00
Pipet, serological, 2 mL	each	532-36
Pipet TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 0.50 mL	each	14515-34
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Sample Cell, 10-mL with screw cap	each	24276-06
Sample Cell, 25-mL with screw cap	each	24019-06

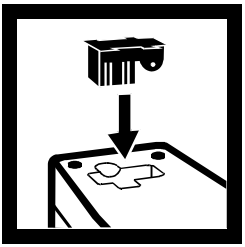
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

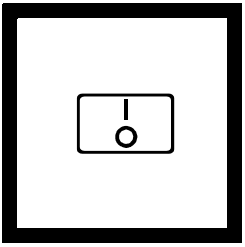
NICKEL, AUTOCATALYTIC (0 to 7.00 g/L)

For finishing baths

Photometric Method

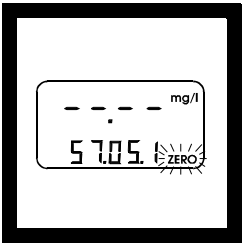


1. Install module **57.01** in a DR/700.



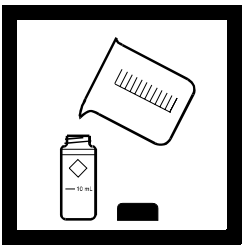
2. Press: **I/O**

The display will show **575 nm** and module number **57.01**



3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **57.05.1**

4. Fill a 10-mL cell to the 10 mL line with demineralized water (the blank).



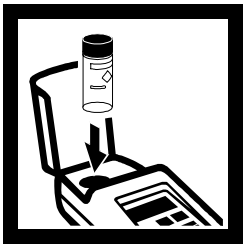
5. Fill a second 10-mL cell to the 10-mL line with bath sample.

Note: *Filter highly turbid samples.*

Note: *For proof of accuracy, use a 1000 mg/L (1.00 g/L) Nickel Standard Solution (listed under Optional Reagents) in place of the sample.*

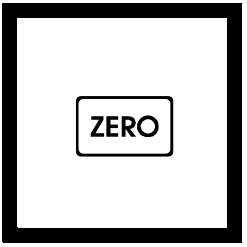
6. Add the contents of one Potassium 1 Reagent Powder Pillow to the bath sample (the prepared sample). Cap and shake to dissolve.

Note: *If a visible turbidity forms upon addition of Potassium 1 Reagent, dilute sample 1:1 with demineralized water and repeat Step 6. Multiply results obtained in Step 10 by 2.*



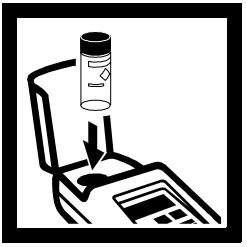
7. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



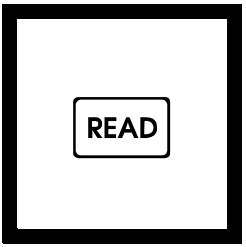
8. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 g/L and the zero prompt will turn off.



9. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



10. Press: **READ**

The display will count down to 0. Then the display will show the results in g/L nickel (Ni).

Note: *This method gives accurate results on nearly all bath formulations. If the bath formulation in use responds differently, a new manual calibration should be done on this bath formulation. See User Stored Programs (Section 1). Follow the procedures in the Operation Section of the instrument manual.*

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower.

ACCURACY CHECK

Standard Solution Method

Check accuracy with a Nickel Standard Solution, 1000 mg/L (1.00 g/L), listed under Optional Reagents. This is used in place of the sample in Step 5.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 2.19 g/L Ni concentration samples the standard deviation was ±0.019 g/L Ni.

Testing zero concentration samples, the limit of detection was 0.033 g/L Ni. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52,* 2242-2249).

INTERFERENCES

Copper will interfere by giving a similar blue color.

SUMMARY OF METHOD

A strong complexing agent chelates the nickel ions present in an "electroless" nickel bath to form a blue colored chelate. The blue color is then measured directly to give the g/L nickel present in the bath.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Potassium 1 Reagent Powder Pillows	1 pillow	25/pkg	14321-98
Water, demineralized	10 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 57.01	1	each	46257-00

OPTIONAL REAGENTS

Nickel Standard Solution, 1000 mg/L	100 mL	14176-42
Nickel Standard Solution, 1000 mg/L	500 mL	14176-49

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Funnel, poly, 65 mm	each	1083-67
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

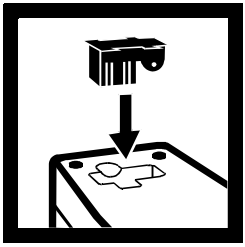
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or the distributor serving you.

NITRITE, HR (0 to 150 mg/L NO_2^-)

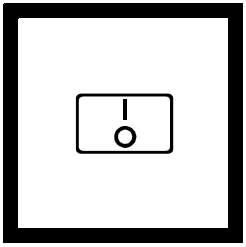
For water and wastewater

Ferrous Sulfate Method*



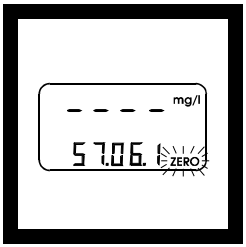
1. Install module **57.01** in a DR/700.

Note: *If sample cannot be analyzed immediately, see Sampling and Storage, below.*



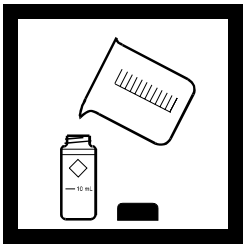
2. Press: **I/O**

The display will show **575 nm** and module **57.01**



3. After 2 seconds, the display will show a program number, concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **57.06.1**

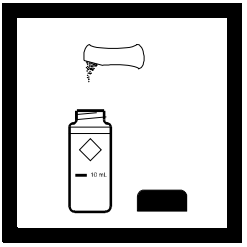
*Adapted from McAlpine, R. and Soule, B., *Qualitative Chemical Analysis*, New York 476, 575 **(1933)**



4. Fill a 10-mL sample cell to the 10-mL line with sample.

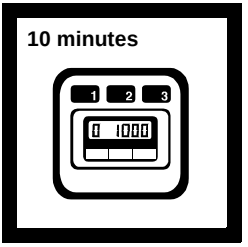
Note: *A 25-mL sample can be tested using 25-mL sample cells and optional reagents.*

Note: *For proof of accuracy, a 100 mg/L nitrite standard solution (preparation given in the Accuracy Check) can be used in place of the sample.*

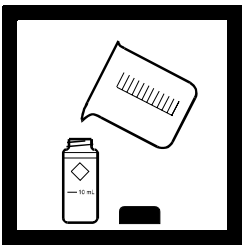


5. Add the contents of one NitriVer 2 Nitrite Reagent Powder Pillow. Cap and shake to dissolve (the prepared sample).

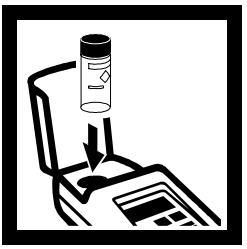
Note: *A greenish-brown color will develop if nitrite is present.*



6. Wait 10 minutes.

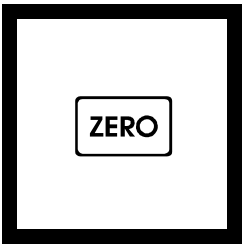


7. Fill a 10-mL cell to the 10-mL line with sample (the blank).



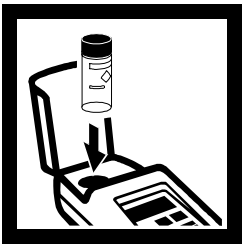
8. Place the blank in the cell holder.

Note: *In bright light close the cell compartment cover.*



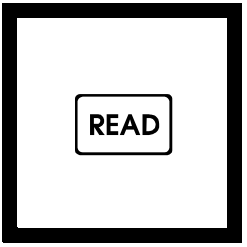
9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



10. Invert the prepared sample twice. Then place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



11. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L nitrite.

Note: *To convert results to other units, see Table 1.*

Table 1. Conversion Factors

To convert reading from	To	Multiply by
mg/L NO_2^-	mg/L NO_2-N	0.3
mg/L NO_2^-	mg/L $NaNO_2$	1.5

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles.

The following storage instructions are necessary only when prompt analysis is not possible. 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, add 4.0 mL of Mercuric Chloride Solution for each liter of sample taken and mix. Sample refrigeration is still required. Do not use acid preservatives.

ACCURACY CHECK

Standard Solution Method

Dissolve 0.150 grams of fresh sodium nitrite and dilute to 1000 mL with demineralized water to prepare a 100 mg/L nitrite standard solution. Prepare this solution daily.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells, and two representative lots of testing reagents. Testing 79.8 mg/L NO_2^- concentration samples, the standard deviation was ±3.8 mg/L NO_2^-.

Testing zero concentration samples, the limit of detection was 3.9 mg/L NO_2^-. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980**, 52, 2242-2249).

INTERFERENCES

This test does not measure nitrates nor is it applicable to glycol based samples. Dilute glycol based samples and follow the Nitrite, Low Range Procedure (DR/700 Filter Module 50.01).

SUMMARY OF METHOD

The method uses ferrous sulfate in an acidic medium to reduce nitrite to nitrous oxide. Ferrous ions combine with the nitrous oxide to form a greenish-brown complex in direct proportion to the nitrite present.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
NitriVer 2 Nitrite Reagent Powder Pillows	1 pillow	100/pkg	21075-69

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 57.01	1	each	46257-00

OPTIONAL REAGENTS

Mercuric Chloride Solution	105 mL	14994-42
NitriVer 2 Nitrite Reagent Powder Pillows	50/pkg	2219-66
NitriVer 3 Nitrite Reagent Powder Pillows	100/pkg	21071-69
Sodium Nitrite, ACS	454 g	2452-01
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Balance, analytical	each	22310-00
Cap, for 10-mL and 25-mL sample cells	12/pkg	24018-12
DR/700 Filter Module Number 50.01	each	46250-00
Flask, volumetric, 1000 mL	each	547-53
Pipet, serological, 10 mL	each	532-38
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	each	24276-06
Sample Cell, 25-mL with screw cap	each	24019-06
Spatula, micro	each	12256-00
Weighing Paper, 76 x 76 mm	500/box	14738-00

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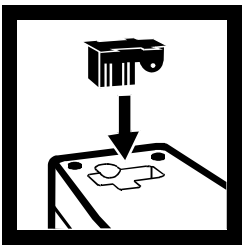
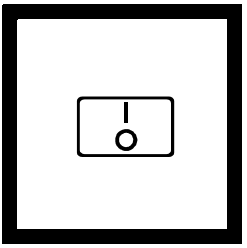
QUATERNARY AMMONIUM COMPOUNDS
(0 to 5.0 mg/L)
For water, wastewater, cooling tower and pool/spa water

Direct Binary Complex Method

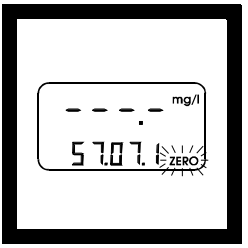


1. Install module **57.01** in a DR/700.

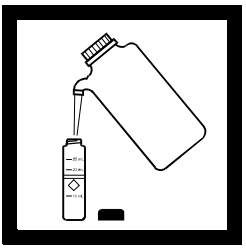


2. Press: **I/O**

The display will show **575 nm** and module number **57.01**

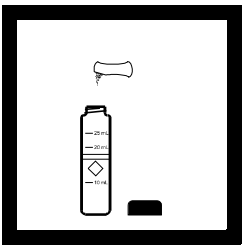


3. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **57.07.1**



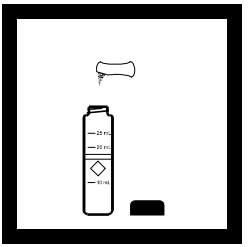
4. Fill a 25-mL cell to the 25-mL line with demineralized water (the blank).

Note: *For proof of accuracy, use a 4 mg/L cetyltrimethyl ammonium bromide (CTAB) Standard Solution (preparation in the Accuracy Check) in place of the sample.*



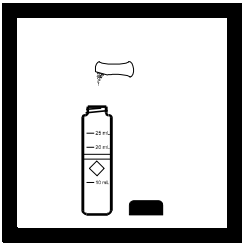
5. Add the contents of one Q.A.C. Reagent 1 powder pillow. Cap and invert to mix. Remove cap after mixing.

Note: *Shaking the cell will cause air bubble turbidity which dissipates slowly and interferes with test results.*



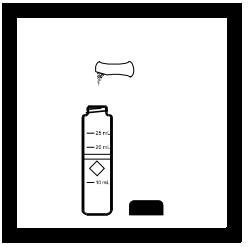
6. Add the contents of one Q.A.C. Reagent 2 powder pillow. Cap and invert to mix.

7. Fill a 25-mL cell to the 25-mL line with sample.

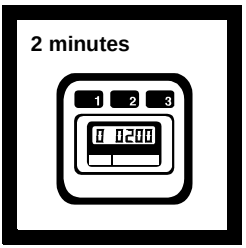


8. Add the contents of one Q.A.C. Reagent 1 powder pillow. Cap and invert to mix. Remove cap after mixing.

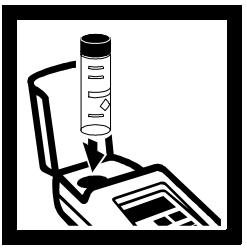
Note: *Shaking the cell will cause air bubble turbidity which dissipates slowly and interferes with test results.*



9. Add the contents of one Q.A.C. Reagent 2 powder pillow. Cap and invert to mix.

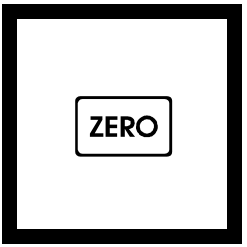


10. Wait 2 minutes.



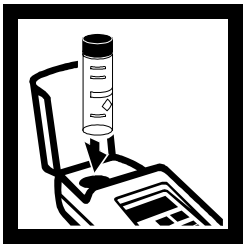
11. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



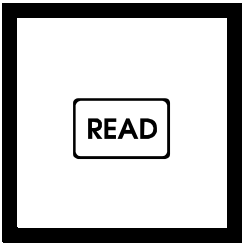
12. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L and the zero prompt will turn off.



13. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



14. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L quaternary ammonium compounds as CTAB.

SAMPLING AND STORAGE

Collect samples in glass bottles that have been rinsed several times with sample before final sample filling. Do not use plastic containers as plastic adsorbs QACs.

ACCURACY CHECK

Standard Solution Method

To assure the accuracy of the test, use a 5 mg/L CTAB Standard Solution prepared as follows:

a) Pipet 5 mL from the Q.A.C. Standard Solution, 100 mg/L as CTAB, into a 100-mL volumetric flask.

b) Dilute the solution to 100 mL with demineralized water. Mix thoroughly.

c) Analyze 25 mL of the 5 mg/L CTAB standard solution according to the preceding procedure. The result should be 5.0 ±0.1 mg/L.

Standard Additions Method
a) Use a TenSette Pipet to add 0.5, 1.0 and 1.5 mL of Q.A.C. Standard Solution, 100 mg/L as CTAB, to three 50-mL samples. Mix thoroughly.

b) Analyze 25 mL each sample according to the above procedure. The QAC concentration should increase by 1.0 mg/L CTAB for each 0.5 mL addition of standard.

INTERFERENCES

Interference studies were conducted by preparing a CTAB standard solution of approximately 3 mg/L as well as a solution of the potential interference. The constituent was said to interfere when the resulting concentration changed by 10%.

Constituent	Level Above Which Constituent Interferes (mg/L)
Positive Interferences:	
Calcium (as $CaCO_3$)	1,350
Chlorine, HOCl and OCl^-	7
Igepal nonionic surfactant	3
Iodine, I_3^-	3
Iron, Fe^{3+}	80
Liquimine 14-P, filming amine	1,825
Magnesium, Mg^{2+}	1,350
Sodium polyphosphate	1,325
Tribenzylamine	7
Triton X-100 nonionic surfactant	4
Urea	8
Negative Interferences:	
Cyanuric acid	70
Niaproof anionic surfactant	11
Polyacrylic acid	16
Sodium lauryl sulfate	8

No Interferences:	Highest Concentration Tested (mg/L)
Potassium alum, $AlKS_2O_8$	500
Silica, H_2SiO_3	400
Sodium thiosulfate, $Na_2S_2O_3$	30

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment. Adjust the sample pH to between 3 and 5 by using a pH meter or pH paper and adding, dropwise, an appropriate amount of acid or base such as 1.0 N Sulfuric Acid Standard Solution or 1.0 N Sodium Hydroxide Standard Solution. If significant volumes of acid or base are used, a volume correction should be made by dividing the total volume (sample + acid + base) by the original sample volume and then multiplying the test result by this factor.

After several samples have been analyzed, the sample cells may exhibit a build-up of a pink or purple color. A rinse with 1.0 N Sodium Hydroxide Solution followed by a Alconox detergent wash and demineralized water rinse will eliminate the build-up when it occurs.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 2.8 mg/L CTAB concentration samples, the standard deviation was ±0.12 mg/L CTAB.

Testing zero concentration samples, the limit of detection was 0.11 mg/L CTAB. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

SUMMARY OF METHOD

The test method makes use of a colorimetric chemistry in which a quaternary ammonium compound reacts with an indicator to produce a color change from pale pink to vivid purple. The test is conducted in a stabilized, acid-buffered solution containing a masking agent to eliminate potential interferences. This test is applicable to the monitoring of QACs in swimming pools and cooling towers.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Q.A.C. Reagent 1	2 pillows	50/pkg	24010-66
Q.A.C. Reagent 2	2 pillows	25/pkg	24012-68

REQUIRED APPARATUS

Description	Quantity Per Test	Unit	Cat. No.
Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 57.01	1	each	46257-00

OPTIONAL REAGENTS

Q.A.C. Standard Solution, 100 mg/L as CTAB	100 mL	24153-42
Sodium Hydroxide Standard Solution, 1.0 N	1000 mL	1045-53
Sulfuric Acid Standard Solution, 1.0 N	59 mL SCDB	270-26
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Cap for 10- and 25-mL cells	12/pkg	24018-12
Cylinder, graduated, 25 mL	each	508-40
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, TenSette 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 5 mL, Class A	each	14515-37
Pipet Filler	each	12189-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

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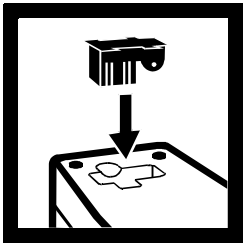
SILVER (0 to 0.600 mg/L)

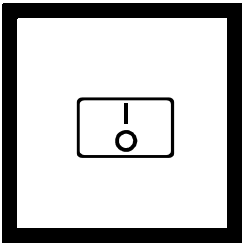
For water and wastewater

Colorimetric Method



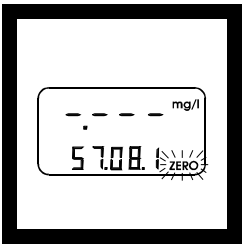
1. Install module **57.01** in a DR/700.

Note: *If sample cannot be analyzed immediately, see Sampling and Storage below. Adjust the pH of stored samples before analysis.*



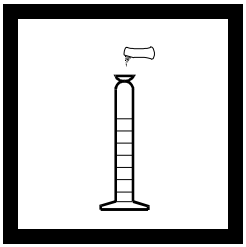
2. Press: **I/O**

The display will show **575 nm** and module number **57.01**



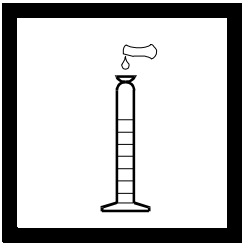
3. After 2 seconds, the display will show a program number, the concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **57.08.1.**

Note: *If cyanide is present, digest the sample; see Digestion following these steps.*



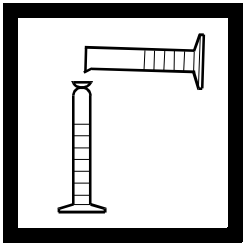
4. Add the contents of one Silver 1 Powder Pillow to a dry 50-mL mixing graduated cylinder.

Note: *If the Silver 1 Powder Pillow is wetted with water at this point, the powder will not dissolve completely, and color development will be inhibited.*



5. Add the contents of one Silver 2 Reagent Pillow to the cylinder. Swirl to completely wet the powder.

Note: *If clumps of dry powder are present when the sample is poured in, the powder will not dissolve completely, and color development will be inhibited.*

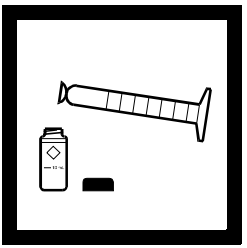


6. Using a 50-mL graduated cylinder, add 50-mL of sample (adjusted to pH 9-10) to the mixing cylinder from Step 5. Stopper and invert repeatedly for one minute to mix.

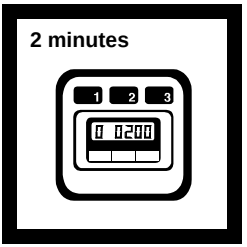
Note: *For proof of accuracy, use a 0.50 mg/L silver standard solution (preparation given in Accuracy Check) in place of the sample.*

7. Fill a 10-mL cell to the 10-mL line with the mixture (the blank). Add the contents of one Thiosulfate Powder Pillow. Swirl for 30 seconds to mix.

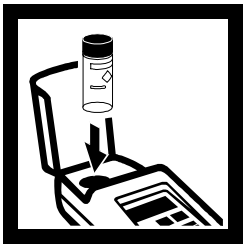
Note: *It is important to have a blank for each sample.*



8. Fill a second 10-mL cell to the 10-mL line with the mixture (the prepared sample).

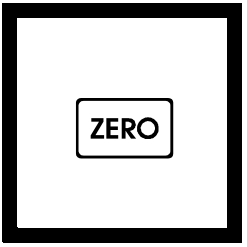


9. Wait 2 minutes.



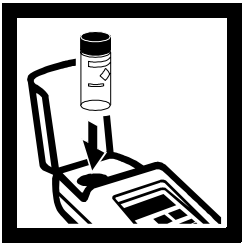
10. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



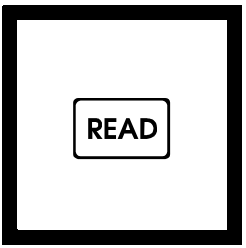
11. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.



12. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



13. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L silver (Ag).

Note: *Rinse the cells carefully between samples to avoid development of a film on the cell walls.*

SAMPLING AND STORAGE

Collect samples in acid-cleaned plastic or glass bottles. Using pH paper, adjust the pH to 2 or less with nitric acid (about 2 mL/liter). Store preserved samples at room temperature for up to six months. Adjust the pH to 9 to 10 with 5.0 N sodium hydroxide before analysis. Correct for volume additions; see Sampling and Storage, Volume Additions, (Section 1) for more information.

ACCURACY CHECK

Standard Additions Method

a) Add 5.0 mL of 1000-mg/L Silver Standard Solution to a 100-mL volumetric flask. Dilute to volume with demineralized water. Mix well. This is a 50-mg/L silver standard solution.

b) Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of this standard solution to three 50-mL samples (or sample portions diluted to 50 mL). Mix well.

c) Analyze as described above. Each 0.1 mL addition of standard should increase the silver concentration by 0.1 mg/L.

d) If these increase do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 0.50 mg/L silver standard solution as follows:

a) Use a TenSette Pipet or 1.0-mL volumetric pipet to add 1.00 mL of Silver Standard Solution, 1000-mg/L Ag, into a 100-mL volumetric flask to prepare a 10 mg/L Ag working solution.

b) Dilute to the mark with demineralized water. This solution should be prepared weekly.

c) Pipet 5.00 mL of the working standard into a 100 mL volumetric flask. Dilute to the mark with demineralized water. This 0.5 mg/L Ag solution should be prepared daily.

d) Perform the silver test as described above.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 0.500 mg/L Ag concentration samples the standard deviation was ±0.0036 mg/L Ag.

Testing zero concentration samples, the limit of detection was 0.0049 mg/L Ag. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52,* 2242-2249).

INTERFERENCES

Interfering studies were conducted by preparing a known silver solution (about 0.4 mg/L) and the potential interfering ion. The ion was said to interfere when the resulting concentration changed by ±10%.

Negative Interference:	**Interference Level**
Aluminum	30 mg/L
Ammonia	750 mg/L
Cadmium	15 mg/L
Chloride	19 mg/L
Chromium, hexavalent	90 mg/L
Copper	7 mg/L
Iron	30 mg/L
Lead	13 mg/L
Manganese	19 mg/L
Nickel	19 mg/L
Zinc	70 mg/L
Positive Interference:	
Calcium	600 mg/L
Magnesium	2000 mg/L
Mercury	2 mg/L

DIGESTION

The following digestion steps are for samples containing organic matter, thiosulfate or cyanide from wastewater, silver electroplating baths and silver strike solutions. Digestion must be carried out in a Digesdahl Digestion Apparatus.

Caution: *Poisonous hydrogen cyanide gas is generated during this digestion.*

Always wear safety glasses and use a safety shield, or operate the Digesdahl within a closed fume hood. Additional safety precautions are given in General Digesdahl Digestion (Section 1).

a) Add an appropriate size sample to the 100-mL volumetric flask of the Digesdahl. Add several boiling chips to prevent bumping.

Note: *The final concentration of the sample (after dilution to 100 mL) should be between 0 and 0.5 mg/L. Larger dilutions may be necessary for electroplating baths and silver strike solutions. This must be determined experimentally. The maximum sample size is 25 mL. Several 25-mL aliquots may be digested in succession to concentrate a very dilute sample.* ***Do not*** *exceed the maximum sample volume.*

b) Turn on the water aspirator and check to be sure there is suction in the fractionating head.

c) Add 3 mL of concentrated sulfuric acid to the sample in the volumetric flask. Immediately place the head on the volumetric flask. Never use less than 3 mL of acid.

d) Place the volumetric flask in the heater. Turn the temperature dial to 440 °C (825 °F). After the sample begins to char or the sulfuric acid reflux line becomes visible, wait 3-5 minutes. **Visually confirm the presence of acid in the flask before adding hydrogen peroxide.**

e) Add 10 mL of 50% hydrogen peroxide to the sample via the capillary funnel on the fractionating head.

f) After the hydrogen peroxide has boiled off, heat the sample until heavy white sulfuric acid fumes are present. Continue heating and reduce the sample volume to near dryness. The sample must not go completely dry at any time.

Note: *If the sample goes to dryness, turn the Digesdahl off and cool completely. Add water to flask before handling. Repeat digestion from the beginning.*

Note: *If only thiosulfate is present in the sample, proceed to Step i.*

g) Add another 3 mL of sulfuric acid via the capillary funnel. Add another 5 mL of hydrogen peroxide. Check the solution for digestion completion. If digestion is not complete, continue adding hydrogen peroxide in 5 to 10 mL portions. Several portions may be necessary.

Note: *Digestion is complete when the digestate is colorless or the color of the digestate does not change upon addition of hydrogen peroxide. Also, a completely digested sample will not foam.*

h) After digestion is complete and all the hydrogen peroxide has boiled off, reduce the volume of the digestate to near dryness. Do not allow the sample to become completely dry. Remove the flask from the heater. Wait until the flask cools to room temperature.

i) Slowly add about 25 mL of demineralized water to the cooled flask.

j) Add two drops of Phenolphthalein Indicator Solution, 1 g/L, and two drops of Thymolphthalein Indicator Solution, 1 g/L.

k) Using sodium hydroxide, adjust the pH of the solution to between 9 and 10. The solution will be pink in this pH range.

Note: *A purple color indicates a pH greater than 10. If this occurs, add a drop of sulfuric acid and 2 drops of each indicator; repeat pH adjustment. Initially, use 50% sodium hydroxide, then 1 N sodium hydroxide as the endpoint is approached.*

l) Filter turbid digestates. Quantitatively transfer the filtrate (or unfiltered sample) to a clean 100-mL volumetric flask. Dilute to the mark with demineralized water. The sample is now ready for analysis by the colorimetric method.

SUMMARY OF METHOD

Silver ions in basic solution (pH 9 to 10) react with cadion 2B to form a green to brownish to red-purple silver-cadion 2B complex. The sodium thiosulfate acts as a decolorizing agent for the blank. The contents of one Silver 1 Powder Pillow and the contents of one Silver 2 Reagent Powder Pillow incorporate the buffer, indicator and masking agents. No organic solvent extractions are required. This colorimetric method is easier to perform than the traditional dithizone method and has fewer interferences. It may also be used for electroplating and silver strike solutions.

REQUIRED REAGENTS

	Cat. No.
Silver Reagent Set (50 tests)	22966-00
Includes: (2) 22935-68, (2) 22936-68, (1) 22937-66	

Description	Quantity Per Test	Unit	Cat. No.
Silver 1 Powder Pillow	1 pillow	25/pkg	22935-68
Silver 2 Powder Pillow	1 pillow	25/pkg	22936-68
Thiosulfate Powder Pillow	1 pillow	50/pkg	11937-66

REQUIRED APPARATUS

Boiling Chips, silicon carbide	2-3	500 g	20557-34
Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, 50 mL	1	each	21179-41
Cylinder, graduated, mixing, 50 mL	1	each	1896-41
DR/700 Filter Module Number 57.01	1	each	46257-00

OPTIONAL REAGENTS

Hydrogen Peroxide, 50%	490 mL	21196-49
Nitric Acid, ACS	500 mL	152-49
Phenolphthalein Indicator Solution, 1 g/L	15 mL SCDB	1897-36
Silver Standard Solution, 1000 mg/L Ag	100 mL	14613-42
Sodium Hydroxide Solution, 1.0 N	100 mL MDB	1045-32
Sodium Hydroxide Solution, 5.0 N	100 mL MDB	2450-37
Sodium Hydroxide, 50%	500 mL	2180-49
Sulfuric Acid, ACS	4 kg	979-09
Thymolphthalein Indicator Solution, 1 g/L	15 mL SCDB	21853-36
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Digesdahl Digestion Apparatus, 115 Vac	each	23130-20
Digesdahl Digestion Apparatus, 230 Vac	each	23130-21
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, serological, 10 mL	each	532-38

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet Tips, for 19700-01	50/pkg	21856-96
Pipet Tips, for 19700-10	50/pkg	21997-96
Pipet, volumetric, Class A, 0.50 mL	each	14515-34
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, Filler, safety bulb	each	14651-00
Safety Glasses	each	18421-00
Safety Shield, for Digesdahl	each	20974-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

Module 61.01
610 nm

Hach warrants equipment it manufactures against defective materials or workmanship for at least one year from the shipping date. Warranties do not apply to limited-life electrical components such as batteries. Full warranty information is located on the reverse side of Hach invoices.

IMPORTANT NOTICE

A lamp intensity adjustment must be performed:
- Before first or initial use
- When new filter modules are installed
- On each filter module when the lamp is replaced

Refer to Lamp Intensity Adjustment in the instrument manual.

Table of Contents for 610-nm parameters

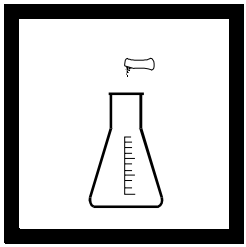
BORON (0 to 14.0 mg/L)

For water and wastewater

Carmine Method*

1. Measure 75.0 mL of sulfuric acid, ACS, using a 100-mL graduated cylinder, into a 250-mL erlenmeyer flask.

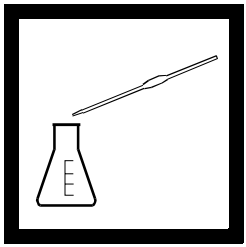
Note: *All glassware must be completely dry. Excess water will cause low results.*



2. Add the contents of one BoroVer 3 Reagent Powder Pillow to the flask. Swirl to mix.

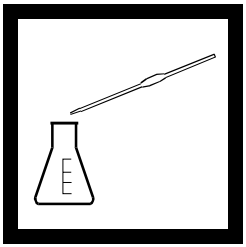
Note: *The powder will dissolve within five minutes.*

Note: *See Reagent Preparation following these steps.*



3. Accurately pipet 2.0 mL of demineralized water into a 125-mL erlenmeyer flask (the blank).

*Adapted from *Standard Methods for the Examination of Water and Wastewater.*

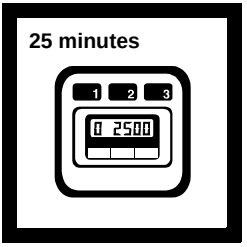


4. Accurately pipet 2.0 mL of sample into another 125-mL erlenmeyer flask (the prepared sample).

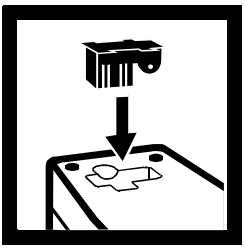
Note: *For proof of accuracy, use a 4.0 mg/L boron standard solution (preparation given in Accuracy Check) in place of the sample.*

Warning: Do not use a stoppered or capped vessel to complete Steps 4 and 5.

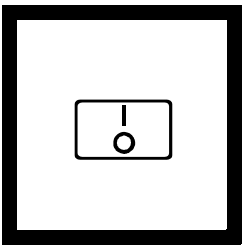
5. Add 35 mL of the BoroVer 3-sulfuric acid solution to each erlenmeyer flask using a 50-mL graduated cylinder. Swirl to mix completely.



6. Wait 25 minutes.

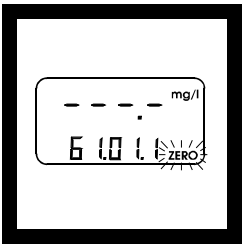


7. Install module
61.01
in a DR/700.

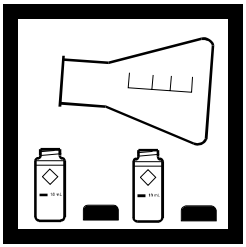


8. Press: **I/O**

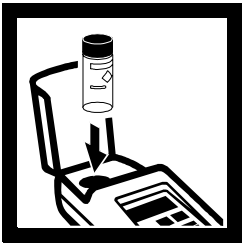
The display will show
610 nm
and module number
61.01



9. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
61.01.1

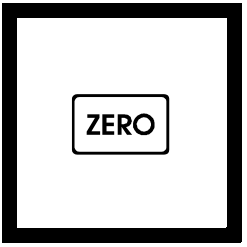


10. Pour 10 mL of the blank and 10 mL of the prepared sample into separate sample cells. Label and cap the cells



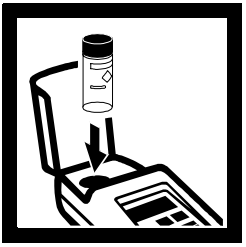
11. Place the blank in the cell holder.

Note: *In bright light, it may be necessary to close the cell compartment cover.*



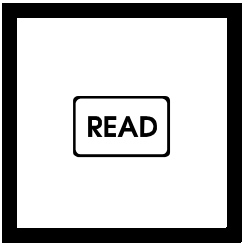
12. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L and the zero prompt will turn off.



13. Place the prepared sample in the cell holder.

Note: *In bright light, it may be necessary to close the cell compartment cover.*



14. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L boron (B).

SAMPLING AND STORAGE
Collect samples in polyethylene bottles or alkali-resistant boron-free glass.

REAGENT PREPARATION
Prepare additional BoroVer 3/sulfuric acid solution by mixing one BoroVer 3 Reagent Powder Pillow per 75 mL of sulfuric acid, ACS, adding the powder pillows individually with stirring. **Preparation of this solution generates gaseous HCl** when the indicator pillow is added to the concentrated sulfuric acid. **Use of a fume hood or other well-ventilated lab area is strongly advised.** This solution will be stable for up to 48 hours if stored in plastic containers. It should not be stored in Pyrex or Kimax (borosilicate) vessels for longer than one hour because the solution will leach boron from these containers. Use soft glass or polyethylene containers for storage.

ACCURACY CHECK
Standard Additions Method

a) Snap the neck off a Boron Voluette Ampule Standard, 250 mg/L B.

b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to three 25-mL portions of sample.

c) Perform the above procedure. The boron concentration reading should increase 1 mg/L for each 0.1 mL of standard solution added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Check the accuracy of the test using Boron Standard Solution, 4 mg/L as B, listed under Optional Reagents, below. Or, prepare this solution as follows:

a) Pipet 4.00 mL of the Boron Voluette Ampule Standard, 250 mg/L B, into a 250-mL volumetric flask.

b) Dilute to volume with demineralized water. Swirl to mix.

Analyze according to the above procedure using either of these solutions as the sample.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 8.0 mg/L B concentration solutions, the standard deviation was ±0.11 mg/L B.

Testing zero concentration samples, the limit of detection was 0.44 mg/L B. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

SUMMARY OF METHOD

Boron is determined by its reaction with carminic acid in the presence of sulfuric acid to produce a reddish to bluish color. The amount of color is directly proportional to the boron concentration.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
BoroVer 3 Boron Reagent Powder Pillows	1 pillow	50/pkg	14170-66
Sulfuric Acid, ACS	75 mL	4 Kg	979-09
Water, demineralized	2.0 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, 50 mL	1	each	508-41
Cylinder, graduated, 100 mL	1	each	508-42
DR/700 Filter Module Number 61.01	1	each	46261-00
Flask, erlenmeyer, 125 mL	2	each	505-43
Flask, erlenmeyer, 250 mL	1	each	505-46
Pipet, volumetric, 2.00 mL	2	each	14515-36

OPTIONAL REAGENTS

Boron Standard Solution, 4 mg/L as B	500 mL		1963-49
Boron Standard Solution, Voluette ampule, 250 mg/L B, 10 mL	16/pkg		14249-10

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, 500 mL	each	20885-49
Flask, erlenmeyer, 1000 mL	each	505-53
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 4.00 mL	each	14515-04
Pipet Filler, safety bulb	each	14651-00
Sample cell, 10-mL, with screw cap	6/pkg	24276-06
Sample cell, 25-mL, with screw cap	6/pkg	24019-06

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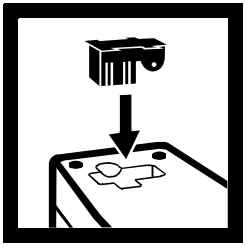
In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

COBALT (0 to 2.00 mg/L)

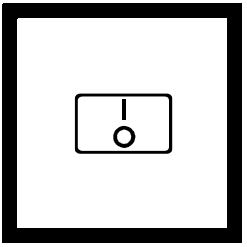
For water and wastewater

1-(2-Pyridylazo)-2-Naphthol (PAN) Method*



1. Install module
61.01
in a DR/700.

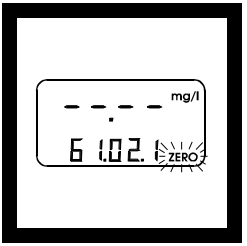
Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*



2. Press: **I/O**

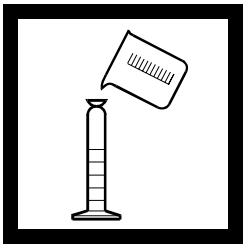
The display will show
610 nm
and module number
61.01

Note: *Total recoverable cobalt requires a prior digestion; use one of the three procedures given in Digestion (Section 1). If EDTA is present, use the vigorous digestion.*



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, the **UP ARROW** key until the lower display shows program number
61.02.1

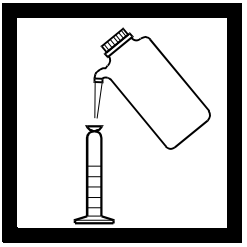
*Adapted from Watanbe, H., *Talanta*, 1974, *21*, 295



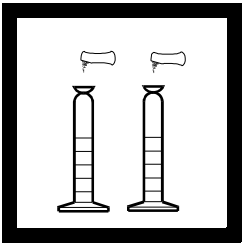
4. Measure 25 mL of sample in a 25-mL graduated mixing cylinder (the prepared sample).

Note: *If sample is less than 10 °C (50 °F), warm to room temperature prior to analysis.*

Note: *For proof of accuracy, use a 1.0 mg/L cobalt standard solution (preparation given in Accuracy Check) in place of the sample.*

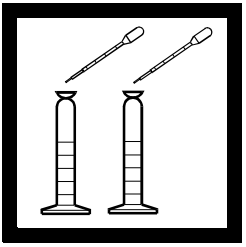


5. Measure 25 mL of demineralized water in a second 25-mL cylinder (the blank).



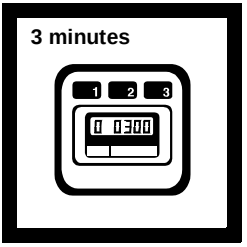
6. Add the contents of one Phthalate-Phosphate Reagent Powder Pillow to each cylinder. Stopper and immediately shake to dissolve.

Note: *If sample contains iron (Fe^{3+}), it is important that all of the powder be dissolved completely before continuing with Step 7.*



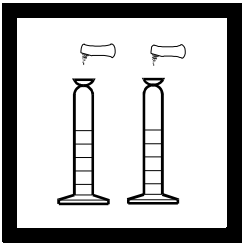
7. Add 1.0 mL of 0.3% PAN Indicator Solution to each cylinder. Stopper and invert several times to mix.

Note: *Use plastic dropper provide to add PAN solution.*



8. Wait 3 minutes.

Note: *During color development, the sample solution color may vary from green to dark red, depending on the chemical make-up of the sample. The demineralized water blank should be yellow.*

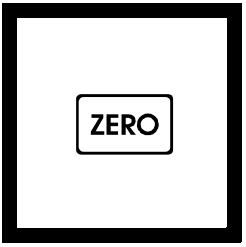


9. Add the contents of one EDTA Reagent Powder Pillow to each cylinder. Stopper and shake to dissolve.

10. Fill a 10-mL cell to the 10-mL line with the blank. Cap.

11. Place the blank in the cell holder.

Note: *In bright light, it may necessary to close the cell compartment cover.*



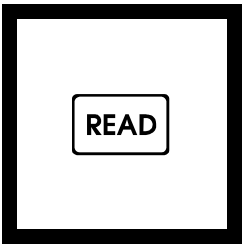
12. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

13. Fill a 10 mL cell to the 10-mL line with the prepared sample. Cap.

14. Place the prepared sample in the cell holder.

Note: *In bright light, it may necessary to close the cell compartment cover.*



15. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L cobalt (Co).

SAMPLING AND STORAGE

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 5 mL per liter). Preserved samples can be stored up to six months at room temperature. Just before analysis, adjust the sample pH between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 8 as this may cause some loss of cobalt as a precipitate. Correct test results for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Solution Method

Prepare 1.0-mg/L cobalt standard solution by diluting 10.0 mL of a 10-mg/L working stock solution to 100 mL in a volumetric flask. The working stock solution should be prepared daily by diluting 10.00 mL of cobalt Standard Solution, 1000 mg/L as Co, to 1000 mL with demineralized water. This is a 10-mg/L cobalt standard solution.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 1.0 mg/L Co concentration solutions, the standard deviation was ±0.004 mg/L Co.

Testing zero concentration samples, the limit of detection was 0.007 mg/L Co. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

The following may interfere in concentrations exceeding those listed below.

AL^{3+}	32 mg/L
Ca^{2+}	1000 mg/L as ($CaCO_3$)
Cd^{2+}	20 mg/L
$C1^{-}$	8000 mg/L
Cr^{3+}	20 mg/L
Cr^{6+}	40 mg/L
Cu^{2+}	15 mg/L
F^{-}	20 mg/L
Fe^{2+}	interferes directly and must not be present.

Fe^{3+}	10 mg/L
K^{+}	500 mg/L
Mg^{2+}	400 mg/L
Mn^{2+}	25 mg/L
Mo^{6+}	60 mg/L
Na^{+}	5000 mg/L
Pb^{2+}	20 mg/L
Zn^{2+}	30 mg/L

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

SUMMARY OF METHOD

After buffering the sample and masking any Fe^{3+} with pyrophosphate, the cobalt is reacted with 1-(2-Pyridylazo)-2-Naphthol indicator. The indicator forms complexes with most metals present. After color development, EDTA is added to destroy all metal-PAN complexes except nickel and cobalt. This method is unique because nickel and cobalt can be determined on the same sample.

REQUIRED REAGENTS

	Cat. No.
Cobalt Reagent Set (100 Test)	22426-00
Includes: (4) 7005-66, (4) 21501-66, (2) 21502-32	

Description	Quantity Per Test	Unit	Cat. No.
EDTA Reagent			
Powder Pillows	2 pillows	50/pkg	7005-66
Phthalate-Phosphate Reagent			
Powder Pillows	2 pillows	50/pkg	21501-66
PAN Indicator			
Solution, 0.3%	2 mL	100 mL	21502-32
Water, demineralized	25 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, mixing, graduated, 25 mL	2	each	20886-40

REQUIRED APPARATUS

Description	Quantity Per Test	Unit	Cat. No.
DR/700 Filter Module Number 61.01	1	each	46261-00

OPTIONAL REAGENTS

Cobalt Standard Solution, 1000 mg/L Co	100 m	21503-42
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL	2450-32
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 1000 mL	each	14574-53
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, serological, 1 mL	each	532-35
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 10.0 mL	each	14515-38
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

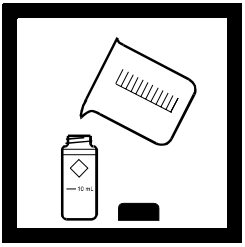
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

CYANIDE (0 to 0.200 mg/L)

For water, wastewater and seawater

Pyridine-Pyrazalone Method*

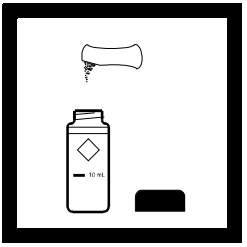


1. Fill a 10-mL cell to the 10-mL line with sample.

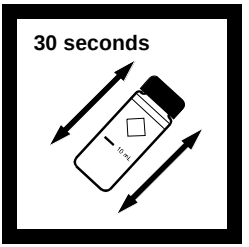
Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of the stored samples before analysis.*

Note: *A 25-mL sample can be tested using 25-mL sample cells and optional reagents.*

Note: *For proof of accuracy, use a 0.10 mg/L cyanide standard solution (preparation given in Accuracy Check) in place of the sample.*

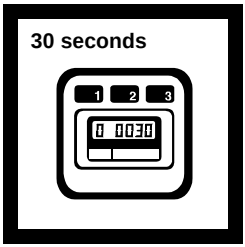


2. Add the contents of one CyaniVer 3 Cyanide Reagent Powder Pillow. Cap the sample cell.



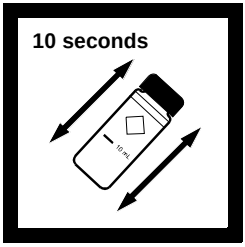
3. Shake the sample cell for 30 seconds.

*Adapted from Epstein, *J. Anal. Chem*., **1947**, *19(4)*, 272



4. Wait an additional 30 seconds, leaving the sample cell undisturbed.

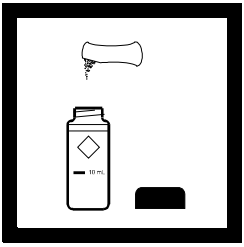
5. Add the contents of one CyaniVer 4 Cyanide Reagent Powder Pillow. Cap the sample cell.



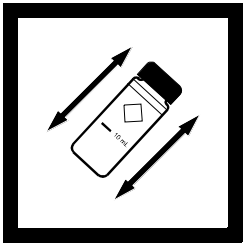
6. Shake the sample cell for 10 seconds. Proceed immediately with Step 7.

Note: *Delaying the addition of CyaniVer 5 Cyanide Reagent Powder for more than 30 seconds after the addition of the CyaniVer 4 Reagent Powder will give lower test results.*

Note: *Accuracy is not affected by undissolved CyaniVer 4 Cyanide Powder Reagent.*

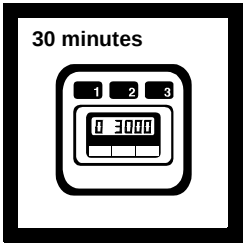


7. Add the contents of one CyaniVer 5 Cyanide Reagent Powder Pillow. Cap the sample cell.



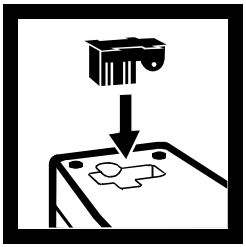
8. Shake vigorously to completely dissolve the CyaniVer 5 Reagent Powder (the prepared sample).

Note: *If cyanide is present, a pink color will develop which then turn blue after a few minutes.*

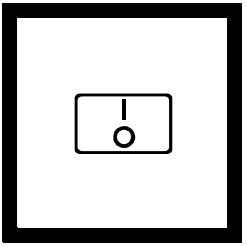


9. Wait 30 minutes.

Note: *Samples at less than 25 ^{o}C require longer reaction times and samples at greater than 25 ^{o}C give low test results.*

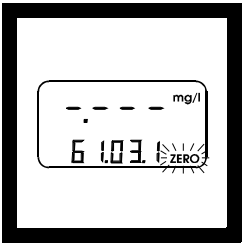


10. Install module **61.01** in a DR/700.

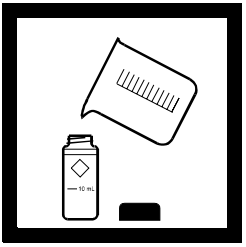


11. Press: **I/O**

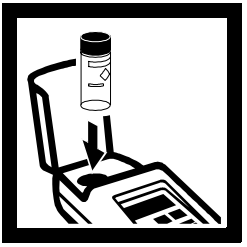
The display will show **610 nm** and module number **61.01**



12. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **61.03.1**

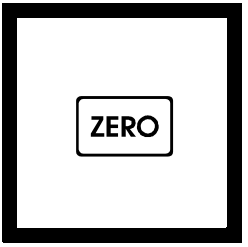


13. Fill a 10-mL cell to the 10-mL line with sample (the blank).



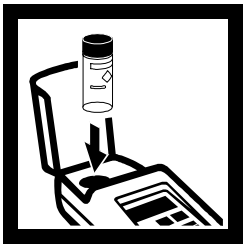
14. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



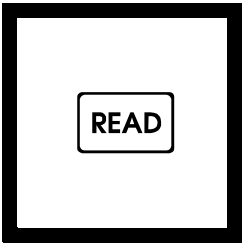
15. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.



16. Place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



17. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L cyanide (CN^-).

SAMPLING AND STORAGE

Samples collected in glass or plastic bottles should by analyzed as quickly as possible. The presence of oxidizing agents, sulfides and fatty acids can cause the loss of cyanide during sample storage. Samples containing these substances must be pretreated as described in the following procedures before preservation with sodium hydroxide. If the sample contains sulfide and is not pretreated, it must be analyzed within 24 hours.

To preserve the sample, adjust the pH above 12 by adding 4.0 mL of 5.0 N Sodium Hydroxide Standard Solution to each liter (or quart) of sample, using a glass serological pipet and pipet filler. Check the sample pH to be sure it is above 12. Four mL of sodium hydroxide is usually enough to raise the pH of most water and wastewater samples to 12. Add more 5.0 N sodium hydroxide if necessary. Store the samples at 4 °C (39 °F) or less. Samples preserved in this manner can be stored for 14 days.

Before testing, samples preserved with 5.0 N sodium hydroxide or samples that are highly alkaline due to chlorination treatment processes

or sample distillation procedures should be adjusted to approximately pH 7 with 2.5 N Hydrochloric Acid Standard Solution. When significant amounts of preservative are used, correct for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

Oxidizing Agents
Oxidizing agents such as chlorine decompose cyanides during storage. To test for their presence and to eliminate their effect, pretreat the sample as follows:

a) Take a 25-mL portion of the sample and add one drop of m-Nitrophenol Indicator Solution, 10 g/L. Swirl to mix.

b) Add 2.5 N Hydrochloric Acid Standard Solution drop-wise until the color changes from yellow to colorless. Swirl the sample thoroughly after adding each drop.

c) Add 2 drops of Potassium Iodide Solution, 30 g/L, and 2 drops of Starch Indicator Solution to the sample. Swirl to mix. The solution will turn blue if oxidizing agents are present.

d) If Step c suggests the presence of oxidizing agents, add 2 level 1-g measuring spoonfuls of ascorbic acid per liter of sample.

e) Withdraw a 25-mL portion of sample treated with ascorbic acid and repeat Steps a to c. If the sample turns blue, repeat Steps d and e.

f) If the 25-mL sample remains colorless, preserve the remaining sample to pH 12 for storage with 5 N Sodium Hydroxide Standard Solution (usually 4 mL/L).

g) Perform the procedure given under Interferences, Reducing Agents, to eliminate the effect of excess ascorbic acid, before performing the cyanide procedure.

Sulfides
Sulfides will quickly convert cyanide to thiocyanate (SCN). To test for the presence of sulfide and eliminate its effect, pretreat the sample as follows:

a) Place a drop of sample on a disc of hydrogen sulfide test paper that has been wetted with pH 4 Buffer Solution.

b) If the test paper darkens, add a 1-g measuring spoon of lead acetate to the sample. Repeat Step a.

c) If the test paper continues to turn dark, keep adding lead acetate until the sample tests negative for sulfide.

d) Filter the lead sulfide precipitate through filter paper and a funnel. Preserve the sample for storage with 5 N Sodium Hydroxide Standard Solution or neutralize to a pH of 7 for analysis.

Fatty Acids

Caution: *Perform this operation in a hood as quickly as possible.*

When distilled, fatty acids will pass over with cyanide and form soaps under the alkaline conditions of the absorber. If the presence of fatty acid is suspected, do not preserve samples with sodium hydroxide until the following pretreatment is performed. The effect of fatty acids can be minimized as follows:

a) Acidify 500 mL of sample to pH 6 or 7 with Acetic Acid Solution.

b) Pour the sample into a 1000-mL separatory funnel and add 50 mL of hexane.

c) Stopper the funnel and shake for one minute. Allow the layers to separate.

d) Drain off the sample (lower) layer into a 600-mL beaker. If the sample is to be stored, add 5 N Sodium Hydroxide Standard Solution to raise the pH to above 12.

ACCURACY CHECK

Standard Solution Method

Caution: *Cyanides and their solutions, and the hydrogen cyanide liberated by acids, are very poisonous. Both the solutions and the gas can be absorbed through the skin.*

To assure the accuracy of the test, prepare the following standard solutions.

Prepare a 100 mg/L cyanide stock solution weekly by dissolving 0.1884 grams of sodium cyanide in 10 mL of 5.0 N sodium hydroxide and diluting to 1000 mL with demineralized water.

Immediately before use prepare a 0.10 mg/L cyanide working solution by diluting 1.00 mL of the 100 mg/L stock solution to 1000 mL using demineralized water.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 0.098 mg/L CN^- concentration solutions, the standard deviation was ±0.0028 mg/L CN^-.

Testing zero concentration samples, the limit of detection was 0.0011 mg/L CN^-. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

Turbidity

Large amounts of turbidity will interfere and cause high readings. If the water sample is highly turbid, it should first be filtered before use in Steps 1 and 13. Filter using the labware listed under Optional Apparatus. Record the test results as soluble cyanide.

Oxidizing and Reducing Agents

Large amounts of chlorine in the sample will cause a milky white precipitate after the addition of the CyaniVer 5 Reagent. If chlorine or other oxidizing agents are known to be present, or if reducing agents (such as sulfide or sulfur dioxide) are known to be present, pretreat the sample before testing as follows using adequate ventilation:

Oxidizing Agents

a) Adjust a 25-mL portion of the alkaline sample to pH 7-9 with 2.5 N Hydrochloric Acid Standard Solution. Count the number of drops of acid added.

b) Add 2 drops of Potassium Iodide Solution and 2 drops of Starch Indicator Solution to the sample. Swirl to mix. The sample will turn blue if oxidizing agents are present.

c) Add Sodium Arsenite Solution drop-wise until the sample turns colorless. Swirl the sample thoroughly after each drop. Count the number of drops.

d) Take another 25-mL sample and add the total number of drops of Hydrochloric Acid Standard Solution counted in Step a.

e) Subtract one drop from the amount of Sodium Arsenite Solution added in Step c. Add this amount to the sample and mix thoroughly.

f) Continue with Step 2 of the cyanide procedure.

Reducing Agents

a) Adjust a 25-mL portion of the alkaline sample to pH 7-9 with 2.5 N Hydrochloric Acid Standard Solution. Count the number of drops added.

b) Add 4 drops of Potassium Iodide Solution and 4 drops of Starch Indicator Solution to the sample. Swirl to mix. The sample should be colorless.

c) Add Bromine Water drop-wise until a blue color appears. Swirl the sample thoroughly after each addition. Count the number of drops.

d) Take another 25 mL sample and add the total number of drops of Hydrochloric Acid Standard Solution counted in Step a.

e) Add the total number of drops of Bromine Water counted in Step c to the sample and mix thoroughly.

f) Continue with Step 2 of the cyanide procedure.

Metals

Nickel or cobalt in concentrations up to 1 mg/L do not interfere. Eliminate the interference from up to 20 mg/L copper and 5 mg/L iron by adding the contents of one HexaVer Chelating Reagent Powder Pillow to 25 mL of sample and then mixing before adding the CyaniVer 3 Cyanide Reagent powder Pillow in Step 2. Prepare a reagent blank of demineralized water and CyaniVer 3, CyaniVer 4, CyaniVer 5 reagent and HexaVer Chelating reagent to zero the instrument in Step 15.

Acid Distillation
For USEPA reporting purposes, samples must be distilled.

All samples to be analyzed for cyanide should be treated by acid distillation except when experience has shown that there is no difference in results obtained with or without distillation. With most compounds, a one-hour reflux is adequate.

If thiocyanate is present in the original sample, a distillation step is absolutely necessary as thiocyanate causes a positive interference. High concentrations of thiocyanate can yield a substantial quantity of sulfide in the distillate. The "rotten egg" smell of hydrogen sulfide will accompany the distillate when sulfide is present. The sulfide must be removed from the distillate prior to testing.

If cyanide is not present, the amount of thiocyanate can be determined. The sample is not distilled and the final reading is multiplied by 2.2. The result is mg/L thiocyanate.

The distillate can be tested and treated for sulfide after the last step of the distillation procedure by using the following lead acetate treatment procedure.

a) Place a drop of the distillate (already diluted to 250 mL) on a disc of hydrogen sulfide test paper that has been wetted with pH 4.0 Buffer Solution.

b) If the test paper darkens, add 2.5 N Hydrochloric Acid Standard Solution drop-wise to the distillate until a neutral pH is obtained.

c) Add a 1-g measuring spoon of lead acetate to the distillate and mix. Repeat Step a.

d) If the test paper continues to turn dark, continue adding lead acetate until the distillate tests negative for sulfide.

e) Filter the black lead sulfide precipitate through filter paper and funnel. Neutralize the sample to pH 7 and analyze for cyanide without delay.

Distillation Procedure
The following steps describe the distillation process using apparatus offered by Hach:

a) Set up the distillation apparatus for cyanide recovery, leaving off the thistle tube. Refer to the Hach Distillation Apparatus Manual. Turn on the water and make certain it is flowing steadily through the condenser.

b) Fill the distillation apparatus cylinder to the 50-mL mark with 0.25 N Sodium Hydroxide Standard Solution.

c) Fill a clean 250-mL graduated cylinder to the 250-mL mark with sample and pour it into the distillation flask. Place a stirring bar into the flask and attach the thistle tube.

d) Arrange the vacuum system as shown in the Hach Distillation Apparatus Manual, but do not connect the vacuum tubing to the gas bubbler. Turn on the water to the aspirator to full flow and adjust the flow meter to 0.5 SCFH.

e) Connect the vacuum tubing to the gas bubbler, making certain that air flow is maintained (check the flow meter) and that air is bubbling from the thistle tube and the gas bubbler.

f) Turn the power switch on and set the stir control to 5. Using a 50-mL graduated cylinder, pour 50 mL of 19.2 N Sulfuric Acid Standard Solution through the thistle tube and into the distillation flask.

g) Using a water bottle, rinse the thistle tube with a small amount of demineralized water.

h) Allow the solution to mix for 3 minutes; then add 20 mL of Magnesium Chloride Reagent through the thistle tube and rinse again. Allow the solution to mix for 3 more minutes.

i) Verify that there is a constant flow of water through the condenser.

j) Turn the heat control to 10.

k) It is very **important to monitor** the distillation flask at this point in the procedure. Once the sample begins to boil, slowly lower the air flow

to 0.3 SCFH. If the contents of the distillation flask begin to back up through the thistle tube, increase the air flow by adjusting the flow meter until the contents do not back up through the thistle tube. Allow the sample to boil for one hour.

l) When one hour is up, turn the still off but maintain the air flow for 15 minutes.

m) After 15 minutes, remove the rubber stopper on the 500-mL vacuum flask to break the vacuum and turn off the water to the aspirator. Turn off the water to the condenser.

n) Remove the gas bubbler/cylinder assembly from the distillation apparatus. Separate the gas bubbler from the cylinder and pour the contents of the cylinder into a 250-mL, Class A volumetric flask. Rinse the gas bubbler, cylinder and J-tube connector with demineralized water and add the washings to the volumetric flask.

o) Fill the flask to the mark with demineralized water and mix thoroughly. Neutralize the contents of the flask and analyze for cyanide.

SUMMARY OF METHOD

The pyridine-pyrazolone method used for measuring cyanide gives an intense blue color with free cyanide. A sample distillation is required to determine cyanide from transition and heavy metal cyanide complexes.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
CyaniVer 3 Cyanide Reagent Powder Pillows	1 pillow	100/pkg	21068-69
CyaniVer 4 Cyanide Reagent Powder Pillows	1 pillow	100/pkg	21069-69
CyaniVer 5 Cyanide Reagent Powder Pillows	1 pillow	100/pkg	21070-69

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 61.01	1	each	46261-00

OPTIONAL REAGENTS

	Cat. No.
Cyanide Reagent Set (100 Tests) 25-mL sample size	22428-00
Includes: (1) 14039-69, (1) 14040-99, (1) 14041-69	

Description	Unit	Cat. No.
Acetic Acid Solution, 10%	500 mL	14816-49
Ascorbic Acid	100 g	6138-26
Bromine Water	29 mL	2211-20
Buffer Solution, pH 4.0	500 mL	12223-49
CyaniVer 3 Cyanide Reagent Powder Pillows	100/pkg	14039-69
CyaniVer 4 Cyanide Reagent Powder Pillows	100/pkg	14040-99
CyaniVer 5 Cyanide Reagent Powder Pillows	100/pkg	14041-69
Hexane, ACS	3.78 L	14478-17
HexaVer Chelating Reagent Powder Pillows	100/pkg	243-99
Hydrochloric Acid Standard Solution, 2.5 N	100 mL MDB	1418-32
Lead Acetate, trihydrate, ACS	500 g	7071-34
Magnesium Chloride Solution	1 L	14762-53
m-Nitrophenol Indicator	100 mL MDB	2476-32
Potassium Iodide Solution, 30 g/L	100 mL DB	343-32
Sodium Arsenite Solution, APHA	100 mL MDB	1047-32
Sodium Cyanide, ACS	28 g	184-20
Sodium Hydroxide Standard Solution, 0.25 N	1 L	14763-53
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53
Starch Indicator Solution	100 mL MDB	349-32
Sulfuric Acid Standard Solution, 19.2 N	1 L	2038-53
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Beaker, glass, 600 mL	each	500-52
Bottle, wash, 500 mL	each	620-11
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, 25 mL	each	508-40
Cylinder, graduated, 50 mL	each	508-41
Cylinder, graduated, 250 mL	each	508-46

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
Distillation Apparatus, cyanide accessories	each	22658-00
Distillation Apparatus, general purpose accessories	each	22653-00
Distillation Apparatus Heater and Support Apparatus, 115 Vac, 60 Hz	each	22744-00
Distillation Apparatus Heater and Support Apparatus, 230 Vac, 50 Hz	each	22744-02
Dropper, plastic	each	6080-00
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, volumetric, 1000 mL	each	547-53
Flask, volumetric, Class A, 250 mL	each	14574-46
Funnel, poly, 65 mm	each	1083-67
Funnel, separatory, 500 mL	each	520-49
Hydrogen Sulfide Test Papers	100/pkg	393-33
Midi-Distillation Apparatus, 4-port	each	26384-00
Midi-Distillation Apparatus, 10-port	each	26385-00
pH Meter, EC10, portable	each	50050-00
Pipet, volumetric, 1 mL	each	515-35
Pipet, volumetric, 2 mL	each	515-36
Pipet, volumetric, 5 mL	each	515-37
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Scoop, double ended	each	12257-00
Spoon, measuring, 1.0 g	each	510-00
Support Ring, 4"	each	580-01
Support Stand	each	563-00

Cyanide can also be measured directly using the Cyanide/Iodide Ion selective electrode, Cat. No. 44410-71

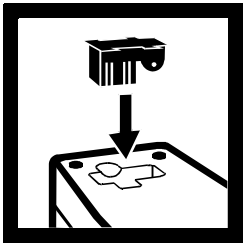
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

FORMALDEHYDE (0 to 500 µg/L)

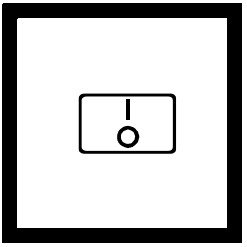
For water

MBTH Method*



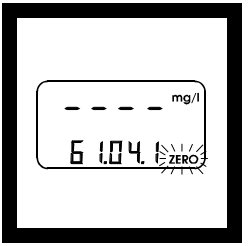
1. Install module **61.01** in a DR/700.

Note: *Samples must be analyzed immediately and cannot be stored for later analysis.*



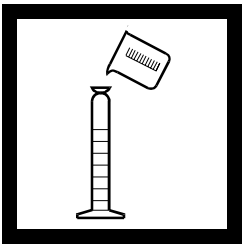
2. Press: **I/O**

The display will show **610 nm** and module number **61.01**



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **61.04.1**

*Adapted from Matthews. T.G.; Howell, T.C., *Journal of the Air Pollution Control Association,* **1981**, *31* (11), 1181-1184

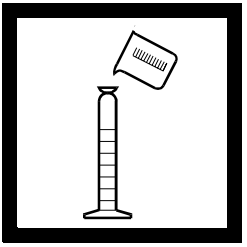


4. Accurately measure 25 mL of sample in a 50-mL mixing cylinder (the prepared sample).

Note: *Wash glassware with chromic acid cleaning solution to remove trace contaminants.*

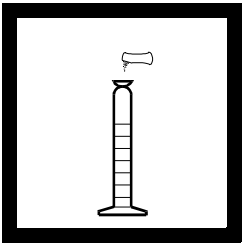
Note: *Time and temperature are very important in this test. The sample should be at 25 ±1 °C, and the times specified must be followed precisely. A temperature controlled water bath is recommended for better accuracy.*

Note: *For proof of accuracy, use a 320 µg/L formaldehyde standard solution (preparation given in Accuracy Check) in place of the sample.*

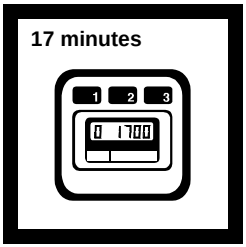


5. Accurately measure 25 mL of formaldehyde-free water in a second 50-mL mixing cylinder (the blank).

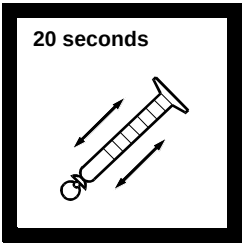
Note: *Obtain formaldehyde-free water by distilling water from alkaline permanganate (4 g sodium hydroxide, 2 g potassium permanganate per 500 mL water). Discard the first 50 to 100 mL of distillate.*



6. Add the contents of one MBTH Powder Pillow to the blank. Stopper the cylinder.

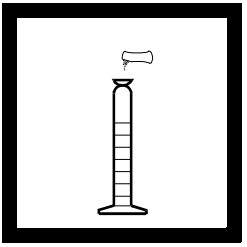


7. Set a timer for a 17-minute reaction period and proceed immediately to Step 8.

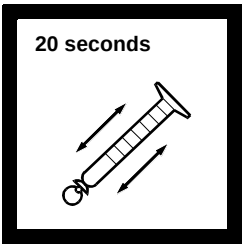


8. Immediately shake the cylinder vigorously for 20 seconds.

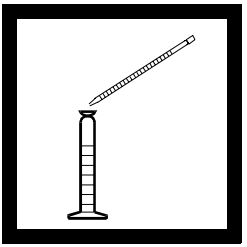
Note: *Important- begin this step immediately after setting the timer.*



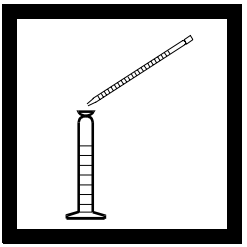
9. Wait 2 minutes (15 minutes remaining). Then add the contents of one MBTH Powder Pillow to the prepared sample. Stopper the cylinder.



10. Shake the cylinder vigorously for 20 seconds.



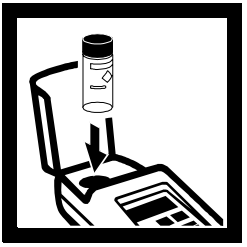
11. After 5 minutes has elapsed (12 minutes remaining) add 2.5 mL of Developing Solution For Low Range Formaldehyde to the **blank**. Stopper and invert to mix.



12. After 7 minutes has elapsed (10 minutes remaining) add 2.5 mL of Developing Solution For Low Range Formaldehyde to the **prepared sample**. Stopper and invert to mix.

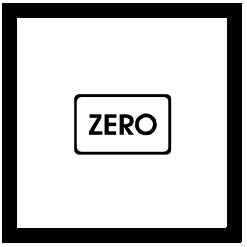
13. Just before 15 minutes has elapsed (2 minutes remaining), fill a 10-mL cell to the 10-mL line with the blank. Cap.

Note: *Pouring the solution slowly into the cell will avoid bubble formation on the cell walls. If bubbles form, swirl the cell to dislodge them.*



14. After the 15 minutes has elapsed, place the blank in the cell holder.

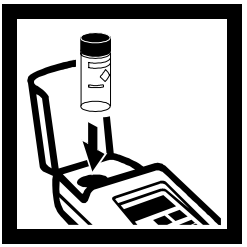
Note: *In bright light, it may be necessary to close the cell compartment cover.*



15. Press: **ZERO**

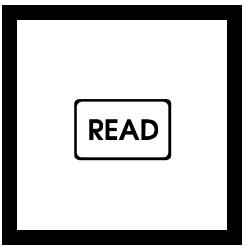
The display will count down to 0. Then the display will show 0 µg/L and the zero prompt will turn off.

16. Fill a 10-mL cell to the 10-mL line with the prepared sample. Cap.



17. Place the prepared sample in the cell holder.

Note: *In bright light, it may be necessary to close the cell compartment cover.*



18. When the 17-minute period is over, immediately press: **READ**

The display will count down to 0. Then the display will show the results in µg/L formaldehyde (CH_2O).

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Formaldehyde Voluette Ampule Standard Solution, 4000 mg/L.

b) Use the TenSette Pipet to add 0.2 mL of standard to a 100-mL volumetric flask. Dilute to volume with formaldehyde-free water. Mix well. Prepare daily. This is an 8 mg/L formaldehyde standard solution.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of diluted standard (8 mg/L) to three 25-mL water samples. Mix each thoroughly.

d) Analyze each sample as described above. The formaldehyde concentration should increase 32 µg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 320 µg/L formaldehyde standard by pipetting 1.0 mL of the 8 mg/L solution from the Accuracy Check into a 50-mL mixing cylinder. Dilute to 25.0 mL with formaldehyde-free water. Run the test directly on this sample.

INTERFERENCES

The following may interfere when present in concentrations exceeding levels listed below.

Acetate	1000 mg/L
Ammonium (as N)	10 mg/L
Aniline	10 mg/L
Bicarbonate	1000 mg/L
Calcium	3500 mg/L
Carbonate	500 mg/L
Chloride	5000 mg/L
Copper	1.6 mg/L
Cyclohexylamine	250 mg/L
Ethanolamine	33 mg/L
Ethylenediamine	1.5 mg/L
Glucose	1000 mg/L
Glycine	1000 mg/L

Iron (Fe^{3+})	12 mg/L
Lead	100 mg/L
Manganese	500 mg/L
Mercury	70 mg/L
Morpholine	0.36 mg/L
Nitrate	1000 mg/L
Nitrite	8 mg/L
Phenol	1050 mg/L
Phosphate	200 mg/L
Silica	40 mg/L
Sulfate	10000 mg/L
Urea	1000 mg/L
Zinc	1000 mg/L

Other aldehydes give a positive interference.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagent. Testing 200 µg/L concentration solutions, the standard deviation was ±2.4 µg/L CH_2O.

Testing zero concentration samples, the limit of detection was 2.9 µg/L CH_2O. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

SUMMARY OF METHOD

Formaldehyde reacts with MBTH (3-methyl-2-benzothiazoline hydrazone) and a developing solution to form a blue color in proportion to the formaldehyde concentration.

REQUIRED REAGENTS

	Cat. No.
Formaldehyde Reagent Set (100 Tests)	22577-00
Includes: (1) 22571-69, (1) 22572-49	

Description	Quantity Per Test	Unit	Cat. No.
Developing Solution For Low Range Formaldehyde	5 mL	500 mL	22572-49

REQUIRED REAGENTS (continued)

Description	Quantity Per Test	Unit	Cat. No.
MBTH Powder Pillows	2 pillow	100/pkg	22571-69

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, mixing, graduated 50 mL	2	each	1896-41
DR/700 Filter Module Number 61.01	1	each	46261-00
Pipet, serological, 5 mL	1	each	532-37
Pipet Filler, safety bulb	1	each	14651-00
Timer, 3-channel	1	each	23480-00

OPTIONAL REAGENTS

Chromic Acid Cleaning Solution	500 mL		1233-49
Formaldehyde Standard Solution, Voluette ampule, 4000 mg/L, 10 mL	16/pkg		22573-10
Potassium Permanganate, ACS	454 g		168-01
Sodium Hydroxide, pellets, ACS	500 g		187-34

OPTIONAL APPARATUS

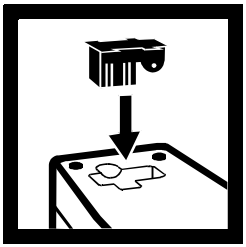
Ampule Breaker Kit		each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg		24018-12
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg		21856-96
Sample Cell, 10-mL with screw cap	6/pkg		24276-06
Sample Cell, 25-mL with screw cap	6/pkg		24019-06
Stopwatch		each	14645-00
Thermometer, -20 to 105 °C		each	1877-01
Water bath, 120 Vac, 60 Hz, 5 gal (19 L)		each	24638-00
Water bath, 240 Vac, 50 Hz, 5 gal (19 L)		each	24638-02

MOLYBDENUM, MOLYBDATE, LR

(0 to 3.00 mg/L)

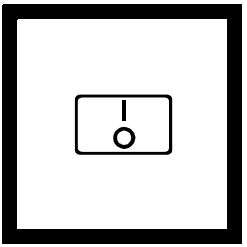
For boiler and cooling tower waters

Ternary Complex Method



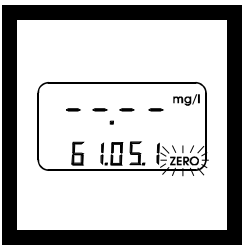
1. Install module
61.01
in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*



2. Press: **I/O**

The display will show
610 nm
and module number
61.01

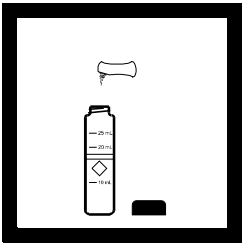


3. After 2 seconds the display will show a program number, concentration units, decimal position and the zero prompt. If necessary press the **UP ARROW** key until the lower display shows program number
61.05.1

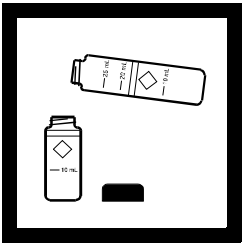
4. Fill a 25-mL sample cell to the 20-mL line with the sample.

Note: *For proof of accuracy, use a 2.0 mg/L Molybdenum Standard Solution (preparation given in Accuracy Check) in place of the sample.*

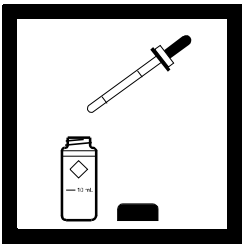
Note: *Filter turbid samples using the labware listed under Optional Apparatus.*



5. Add the contents of one Molybdenum 1 Reagent Powder Pillow. Cap Shake the sample cell to dissolve the reagents (the prepared sample).

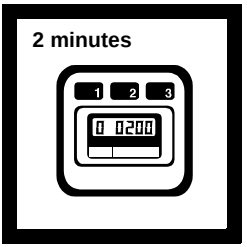


6. Fill a 10-mL cell to the 10-mL line with prepared sample.

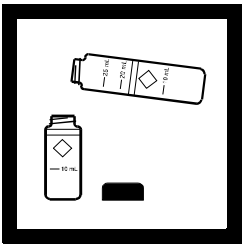


7. Add 0.5 mL of Molybdenum 2 Reagent to the 10-mL cell. Cap and invert several times to mix. This is the developed sample.

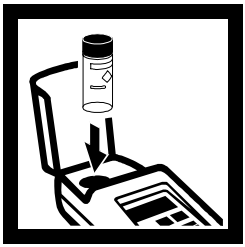
Note: *Molybdenum will cause a green color to form.*



8. Wait two minutes.

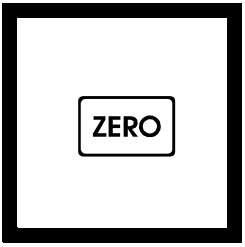


9. Fill a 10-mL cell to the 10-mL line with prepared sample (the blank). Cap.



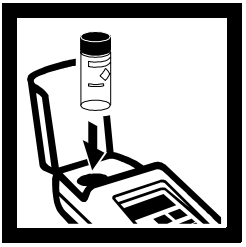
10. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



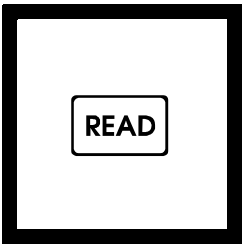
11. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



12. Place the developed sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



13. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L molybdenum.

Note: *To convert results to other units see Table 1.*

Table 1. Conversion Factors

To convert reading from	To	Multiply by
mg/L hexavalent molybdenum (Mo^{6+})	mg/L molybdate (MoO_4^{2-})	1.67
mg/L hexavalent molybdenum (Mo^{6+})	mg/L sodium molybdate (Na_2MoO_4)	2.15

SAMPLING AND STORAGE

Collect samples in glass or plastic bottles.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Molybdenum Voluette Ampule Standard Solution, 500 mg/L Mo^{6+}.

b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to three 250-mL samples. Mix thoroughly.

c) Analyze 20 mL of each sample according to the above procedure. The molybdenum concentration reading should increase by 0.2 mg/L for each 0.1 mL addition of standard.

d) If these increases do not occur, see Standard Additions (Section 1) for more information.

Standard Solution Method

To assure the accuracy of the test, use a 2-mg/L Molybdenum Standard Solution by first pipetting 10 mL of a 10-mg/L Molybdenum Standard Solution into a 50-mL graduated mixing cylinder. Next, dilute to a final volume of 50 mL using demineralized water. Mix thoroughly. Analyze 20 mL of the standard according to the above procedure.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 1.000 mg/L Mo^{6+} concentration samples, the standard deviation was ±0.008 mg/L Mo^{6+}.

Testing zero concentration samples, the limit of detection was 0.035 mg/L Mo^{6+}. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry,* **1980**, *52,* 2242-2249).

INTERFERENCES

Interference studies were conducted by preparing a molybdenum standard solution (2 mg/L Mo^{6+}) as well as a solution of the potential interfering ion. When the standard solution concentration changed by ±5% with a given ion concentration, the ion was considered an interference.

Negative Interference:

Ion	Level above which it interferes (mg/L)
Iron	200
Copper	98
Chromium (Cr^{6+})	4.5*
Chloride	1,400
AMP (Phosphonate)	15
Phosphonohydroxyacetic Acid	32
Bisulfate	3,300
Nitrite	350*
Aluminum	2
Acrylates	790
Alum	7
Lignin Sulfonate	105
Orthophosphate	4,500
Bicarbonate	5,650
EDTA	1,500
Borate	5,250
Ethylene Glycol	2% (by volume)
Sulfite	6,500
Diethanoldithiocarbamate	32

*Read the molybdenum concentration immediately after the two-minute reaction period.

Positive Interference:

Ion	Level above which it interferes (mg/L)
Carbonate	1,325
Silica	600
Benzotriazole	210

No Interference:

Ion	Highest Concentration Tested (mg/L)
Zinc	400
Calcium	720
Magnesium	8,000
Manganese	1,600
Chlorine	7.5
PBTC (phosphonate)	500
Sulfate	12,800
Bisulfite	9,600
Nickel	250

The presence of the phosphonate HEDP at concentrations up to 30 mg/L will increase the apparent molybdenum concentration reading by approximately 10% (positive interference). For these samples, multiply the value obtained in step 13 by 0.9 to obtain the actual molybdenum concentration. As the concentration of HEDP increases above 30 mg/L, a decrease in the molybdenum concentration reading occurs (negative interference).

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagent and require sample pretreatment; see pH Interference in Section I.

After a number of samples have been analyzed, the sample cells may exhibit a build-up of a slight blue color. A rinse using Hydrochloric Acid Solution, 1:1, will eliminate the build-up if it occurs.

SUMMARY OF METHOD

The ternary complex method for molybdenum determination is a method in which molybdate molybdenum reacts with an indicator and sensitizing agent to give a stable blue complex which appears green due to excess yellow reagent.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Molybdenum 1 Reagent for Low Range Molybdate Powder Pillows, for 20 mL sample size	1 pillow	50/pkg	23524-46
Molybdenum 2 Reagent for Low Range Molybdate Solution	0.5 mL	59 mL MDB	23525-12

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 61.01	1	each	46261-00

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Molybdenum 1 Reagent for Low Range Molybdate Powder Pillows, for 50-mL sample size	100/pkg	23527-66
Molybdenum 2 Reagent for Low Range Molybdate Solution, for 50-mL sample size	100 mL MDB	23525-32
Molybdenum Standard Solution, 10 mg/L Mo^{6+}	100 mL	14187-42
Molybdenum Standard Solution, Voluette ampule, 500 mg/L Mo^{6+}, 10 mL	16/pkg	14265-10
Hydrochloric Acid Solution, 1:1, 6.0 N	500 mL	884-49
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Cap for 10 & 25-mL sample cells	12/pkg	24018-12
Cylinder, mixing, graduated, 25 mL	each	1896-40
Filter Paper, folded, 12.5 cm.	100/pkg	1894-57
Funnel, poly, 65 mm	each	1083-67
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	28856-96
Pipet, volumetric, 10 mL, Class A	each	14515-38
Pipet Filler	each	14651-00
Flask, volumetric, 250 mL, Class B	each	547-46
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

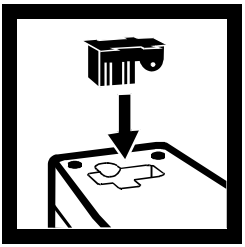
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

NITROGEN, AMMONIA (0 to 1.00 mg/L NH_3-N)

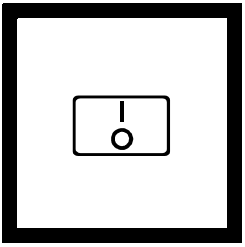
For water, wastewater, seawater

Salicylate Method*



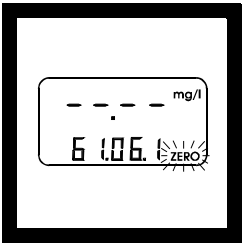
1. Install module number
61.01
in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored samples before analysis.*



2. Press: **I/O**

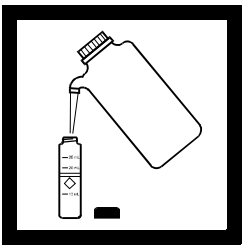
The display will show
610 nm
and module number
61.01



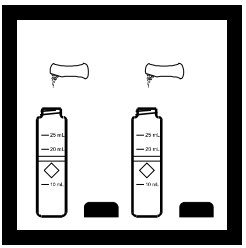
3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press UP arrow until the lower display shows program number
61.06.1

4. Fill a 25-mL cell to the 25-mL line with sample.

Note: *For proof of accuracy, use a 0.20 mg/L NH_3-N solution (preparation given in Accuracy Check) in place of the sample.*

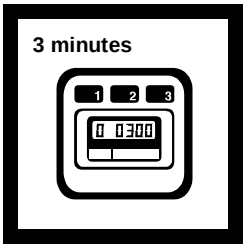


5. Fill a 25-mL cell to the 25-mL line with demineralized water (the blank).

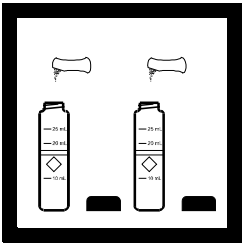


6. Add the contents of one Salicylate Reagent Powder Pillow to each cell. Cap and invert the cells several times to mix.

*Adapted from *Clin. Chim. Acta.* **1966**, *14*, *403*

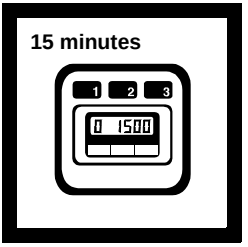


7. Wait 3 minutes.

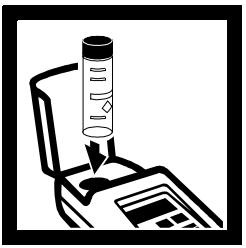


8. Add the contents of one Alkaline Cyanurate Reagent Powder Pillow to each cell. Cap and invert the cells several times to mix.

Note: *A green color will develop if ammonia nitrogen is present.*

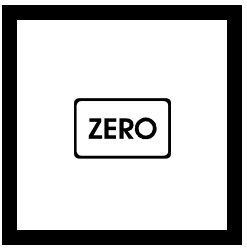


9. Wait 15 minutes.



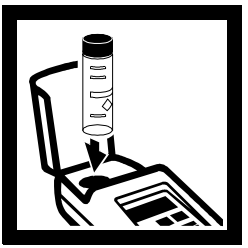
10. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



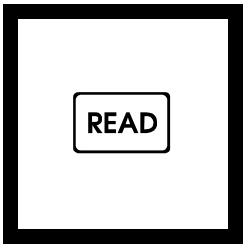
11. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



12. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



13. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L NH_3 as N.

Note: *To convert results to other units, see Table 1.*

Table 1. Conversion Factors

To convert reading from	To	Multiply by
mg/L NH_3-N	mg/L NH_4^+	1.29
mg/L NH_3-N	mg/L NH_3	1.22

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

If chlorine is known to be present, the sample must be treated immediately with sodium thiosulfate. Add one drop of 0.1 N Sodium Thiosulfate Standard Solution for each 0.3 mg of chlorine present in a one liter sample.

To preserve samples, adjust the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter). Store samples at 4 °C or less. Samples preserved in this manner can be stored up to 28 days. Just before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more detailed information.

ACCURACY CHECK

Standard Additions Method

a) Measure 25 mL of sample into three 25-mL mixing cylinders.

b) Use the TenSette Pipet to add 0.2, 0.4, and 0.6 mL of Nitrogen Ammonia Standard, 10 mg/L as NH_3-N to the three samples. Mix well.

c) Analyze each sample as described above. The ammonia nitrogen concentration should increase 0.08 mg/L for each 0.2 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method
Prepare a 0.20 mg/L ammonia nitrogen standard by diluting 2.00 mL of the Nitrogen Ammonia Standard Solution (as NH_3-N), 10 mg/L, to 100 mL with demineralized water. Or, using the TenSette Pipet, prepare a 0.20 mg/L ammonia nitrogen standard by diluting 0.4 mL of a Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as NH_3-N, to 100 mL with demineralized water.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents, Testing 0.400 mg/L NH_3 as N concentration samples, the standard deviation was ±0.006 mg/L NH_3 as N.

Testing zero concentration samples, the limit of detection was 0.029 mg/L NH_3 as N. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,***1980,** *52*, 2242-2249).

INTERFERENCES

The following ions may interfere when present in concentrations exceeding those listed below:

Calcium	1000 mg/L as $CaCO_3$
Magnesium	6000 mg/L as $CaCO_3$
Nitrite	12 mg/L as NO_2^--N
Nitrate	100 mg/L as NO_3^--N
Orthophosphate	100 mg/L as PO_4^{3-}-P
Sulfate	300 mg/L as SO_4^{2-}

Sulfide will intensify the color. Eliminate sulfide interference as follows:

a) Measure about 350 mL of sample in a 500-mL erlenmeyer flask.

b) Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix.

c) Filter the sample through a folded filter paper.

d) Use the filtered solution in Step 4.

Iron interferes with the test. Eliminate iron interference as follows:

a) Determine the amount of iron present in the sample following one of the Total Iron procedures.

b) Add the same iron concentration to the demineralized water sample in Step 5.

The interference from iron in the sample will then be successfully blanked out in Step 11.

Extremely acidic or alkaline samples should be adjusted to approximately pH 7. Use 1 N Sodium Hydroxide Standard Solution for acidic samples or 1 N Sulfuric Acid Standard Solution for basic samples.

Less common interferences such as hydrazine and glycine will cause intensified colors in the prepared sample. Turbidity and sample color will give high values. Samples with severe interferences require distillation. Albuminoid nitrogen samples also require distillation. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set. See Optional Apparatus listing. The distillation procedure is detailed in the Nitrogen, Ammonia - Nessler Method.

SUMMARY OF METHOD

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

REQUIRED REAGENTS

	Cat. No.
Nitrogen Ammonia Reagent Set (100 Tests)	22437-00
Includes: (4) 23955-68, (4) 23953-68	

Description	Quantity Per Test	Unit	Cat.No.
Ammonia Cyanurate Reagent Powder Pillows	2 pillows	25/pkg	23995-68
Ammonia Salicylate Reagent Powder Pillows	2 pillows	25/pkg	23953-68

REQUIRED APPARATUS

Clippers, large	1	each	968-00
DR/700 Filter Module Number 61.01	1	each	46261-00

OPTIONAL REAGENTS

Nitrogen Ammonia Standard Solution, 10 mg/L as (NH_3-N)	500 mL	153-49
Nitrogen Ammonia, Voluette Ampule, 50 mg/L as (NH_3-N), 10 mL	16/pkg	14791-10
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045-32
Sodium Hydroxide Standard Solution, 5.0 N	59 mL	2450-26
Sodium Thiosulfate Standard Solution, 0.10 N	100 mL MDB	323-32
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	2418-99
Sulfuric Acid, concentrated, ACS	500 mL	979-49
Sulfuric Acid Standard Solution, 1.0 N	100 mL MDB	1270-32
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, polypropylene, 500 mL	each	1081-49
Distillation Heater and Support Apparatus, 115 V	each	22744-00
Distillation Heater and Support Apparatus, 230 V	each	22744-02

OPTIONAL APPARATUS

Description	Unit	Cat.No.
Distillation Set, General Purpose	each	22653-00
Filter Paper, folded, 12.5 cm	100	1894-57
Flask, erlenmeyer, polypropylene, 500 mL	each	1082-49
Flask, volumetric, Class A, 100 mL	each	14574-42
Funnel, poly, 65 mm	each	1083-67
pH Meter, EC10, portable	each	50050-00
Pipet Filler, safety bulb	each	14651-00
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 ml	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 2.0 mL	each	14515-36
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Thermometer, -20 to 105 °C	each	1877-01

For Technical Assistance, Prices and Ordering

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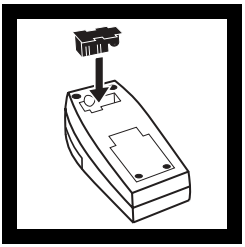
Outside the U.S.A. - Contact the Hach office or the distributor serving you.

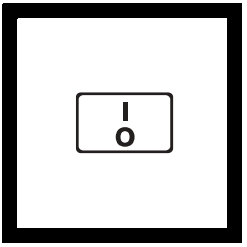
NITROGEN, MONOCHLORAMINE AND FREE AMMONIA (0 to 0.55 mg/L NH_2Cl-N or NH_3-N)

Salicylate Method **For drinking water**



1. Install module number
61.01
in a DR/700.

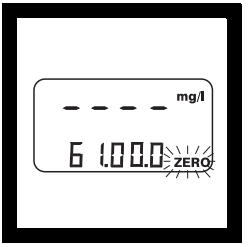
Note: *The DR/700 must be calibrated before sample analysis. See Calibration following these steps.*



2. Press: **I/O**

The display will show
610 nm
and module number
61.01

After 2 seconds, the display will show a program number, the concentration units and the zero prompt**.**



3. Press the **PROGRAM** key once or twice until the lower display shows program number
61.000

If necessary, press the **UP ARROW** until the upper display shows
0.00 mg/l

4. Fill three 10-mL round sample cells to the 10-mL line with sample.

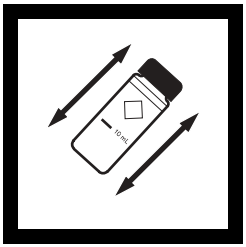
Note: *For proof of accuracy, use a 0.50 mg/L NH_3-N solution (preparation given in Accuracy Check) in place of the sample.*

5. Label the three cells "blank", "free ammonia", and "monochloramine". Cap the cell labeled "blank".

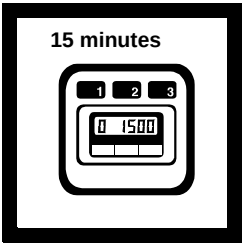
Note: *Free ammonia is the ammonia and ammonium in the sample, corrected for monochloramine.*

6. To test for free ammonia, add one drop of Hypochlorite Solution to the cell labeled free ammonia. Cap the cell and mix.

Note: *Occasionally shake the Hypochlorite Solution bottle to ensure proper dispensation.*



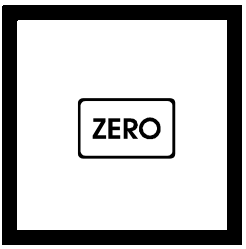
7. Promptly add the contents of one Monochloramine Reagent Pillow to the cell labeled free ammonia and one to the cell labeled monochloramine. Cap and shake to dissolve.



8. Wait 15 minutes. While waiting, wipe fingerprints, liquids, etc. from the cells.

9. Place the cell labeled blank into the cell holder.

***Note**: In bright sunlight it may be necessary to close the cell compartment cover.*



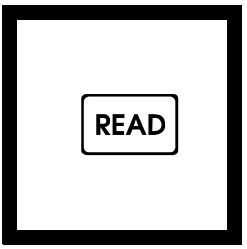
10. Press: **ZERO**

The display will count down to 0. Then the display will show

0.00 mg/L

and the zero prompt will turn off.

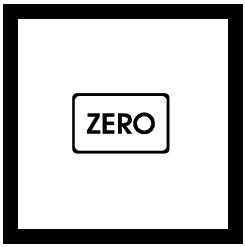
11. Place the cell labeled monochloramine in the cell holder.



12. Press: **READ**

The display will count down to 0. Then the display will show the monochloramine results in mg/L NH_3 as N.

***Note:** To convert results to other units, see Table 1.*

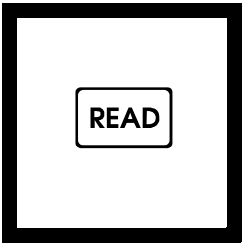


13. Leave the monochloramine cell in the cell holder. Press **ZERO** twice.

The display will show
0.00 mg/l
and the zero prompt will turn off.

14. Place the cell labeled free ammonia into the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*

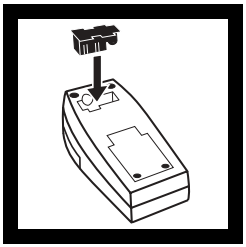


15. Press: **READ**
The display will count down to 0. Then the display will show the free ammonia result in mg/L nitrogen (NH_3-N).

Note: *To convert results to other units, see Table 1.*

Table 1. Conversion Factors

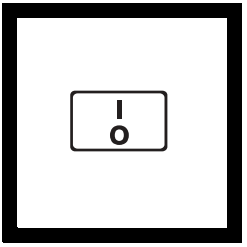
To convert reading from	To	Multiply by
mg/L NH_3-N	mg/L NH_4^+	1.29
mg/L NH_3-N	mg/L NH_3	1.22
mg/L NH_2 Cl-N	mg/L NH_2 Cl as Cl_2	5.06
mg/L NH_2 Cl-N	mg/L NH_2 Cl	3.67



Using AccuVacs

1. Install module number
61.01
in a DR/700.

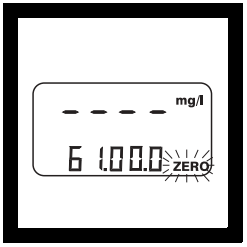
Note: *The DR/700 must be calibrated before sample analysis. See Calibration following these steps.*



2. Press: **I/O**

The display will show
610 nm
and module number
61.01

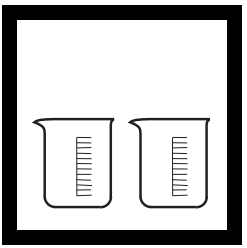
After 2 seconds, the display will show a program number, the concentration units and the zero prompt**.**



3. Press the **PROGRAM** key once or twice until the lower display shows program number
61.000

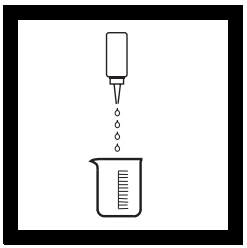
If necessary, press the **UP ARROW** until the upper display shows
0.00 mg/l

4. Fill a cell with 10 mL of sample. Cap the cell. This is the blank- nothing will be added to it.



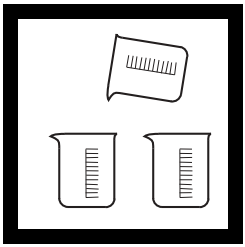
5. Label one 50-mL beaker "free ammonia" and another 50-mL beaker "monochloramine".

Note: *Free ammonia is the ammonia and ammonium in the sample, corrected for monochloramine.*



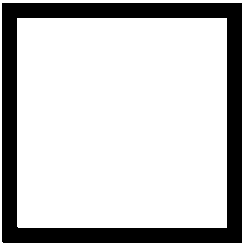
6. To test for free ammonia, add 5 drops of Hypochlorite Solution into the beaker labeled free ammonia.

Note: *Occasionally shake the Hypochlorite Solution bottle to ensure proper dispensation.*



7. Mix by vigorously adding 40-50 mL of sample to the beaker labeled free ammonia. Add 40-50 mL of sample to the beaker labeled monochloramine.

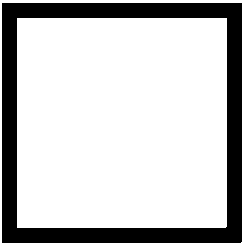
Note: *Hold the beaker 2-3 inches above the receiving beaker to create turbulence.*



8. Promptly fill a Monochloramine AccuVac ampul from each beaker.

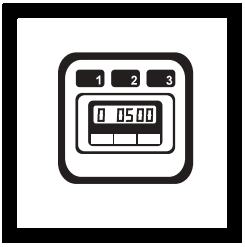
Note: *Keep tips immersed while the ampul fills.*

Note: *Label the* ***bottom*** *of each ampul with proper sample name.*



9. Invert the ampuls to dissolve the powder.

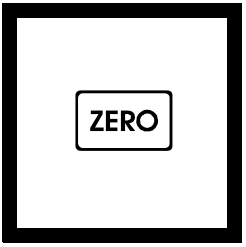
Note: *For proof of accuracy test a 0.50 mg/L NH_3-N solution (add to the free ammonia beaker). See Accuracy Check.*



10. Wait 15 minutes. While waiting, wipe fingerprints, liquids, etc. from the cells.

11. Place the cell labeled blank into the cell holder.

Note*: In bright sunlight it may be necessary to close the cell compartment cover.*



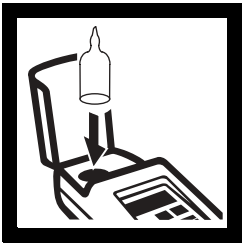
12. Press: **ZERO**

The display will count down to 0. Then the display will show

0.00 mg/L

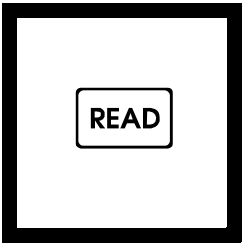
and the zero prompt will turn off.

13. Place the AccuVac Adapter in the cell holder.



14. Place the ampul filled from the beaker labeled monochloramine into the cell holder.

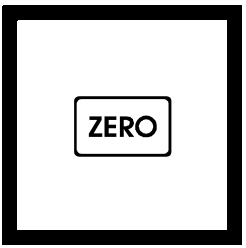
Note: *In bright light it may be necessary to close the cell compartment cover.*



15. Press: **READ**

The display will count down to 0. Then the display will show the monochloramine results in mg/L NH_3 as N.

Note: *To convert results to other units, see Table 1.*

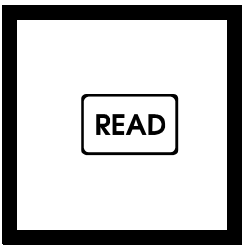


16. Leave the monochloramine ampul in the cell holder. Press **ZERO** twice.

The display will show
0.00 mg/l
and the zero prompt will turn off.

17. Place the ampul labeled free ammonia into the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



18. Press: **READ**
The display will count down to 0. Then the display will show the free ammonia result in mg/L NH_3 as N.

Note: *To convert results to other units, see Table 1.*

Note: *For best results, test monochloramine-free water and subtract the value you obtained (or subtract 0.01 mg/L) from the displayed value.*

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

ACCURACY CHECK

Dilution water is required when testing a diluted sample and when preparing standard solutions. Dilution water must be free of ammonia, chlorine and chlorine demand. A convenient source is a recirculating, deionizer system with carbon filtration which produces 18 megaohm-cm water.

Standard Additions Method

a) Measure 50 mL of sample into three 50-mL mixing cylinders.
b) Use the TenSette Pipet to add 0.3, 0.6, and 1.0 mL of Nitrogen Ammonia Standard, 10 mg/L as NH_3-N to the three samples. Mix well.
c) Analyze each sample as described above. The ammonia nitrogen concentration should increase 0.06, 0.12 and 0.20 mg/L, respectively.
d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Prepare a 0.50 mg/L ammonia nitrogen standard by diluting 5.00 mL of the Nitrogen Ammonia Standard Solution (as NH_3-N), 10 mg/L, to 100 mL with demineralized water. Or, using the TenSette Pipet, prepare a 0.50 mg/L ammonia nitrogen standard by diluting 1.0 mL of an Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as NH_3-N, to 100 mL with demineralized water.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 0.2 mg/L NH_3 as N concentration samples, the standard deviation was ±0.010 mg/L NH_3.

Testing zero concentration samples, the limit of detection was 0.009 mg/L NH_3. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52,* 2242-2249).

CALIBRATION

The test procedure may be used with a User-Programmed method and any 610-nm module. Perform Section 2.2.4.2, Calibration Using a Relative Absorbance with One Standard, in the DR/700 Instrument

Manual. Standard 1 (S1) is made by pouring 10 mL of sample into a zeroing vial or 10-mL sample cell. Cap the cell. Edit the display to show 00.00 mg/L. For standard 2 (S2), edit the display to show 00.55 mg/L and 1.124 Abs.

INTERFERENCES

Ammonia contamination from air is a common cause of high results. Ampules, beakers and other containers may require rinsing with excess sample just before use. Samples, solutions, and demineralized water will accumulate ammonia from the air.

The following ions may interfere when present in concentrations exceeding those listed below:

Calcium	3000 mg/L as $CaCO_3$
Magnesium	1600 mg/L as $CaCO_3$
pH	less than 7
Sulfate	> 900 mg/L as SO_4^{2-}

This method is not recommended for the determination of ammonia in non-chlorinated samples that have a chlorine demand greater than 2 mg/L as Cl_2.

Mixtures of small amounts of differing substances may interfere. To assess the influence of these interferences in a sample, perform the Accuracy Check, Standard Addition Method.

SUMMARY OF METHOD

This method determines "free ammonia" in the presence of monochloramine. Monochloramine (NH_2Cl) and free ammonia (NH_3 and NH_4^+) can exist in drinking water sample where chloramine disinfection is used. Hypochlorite is added to combine with free ammonia to form additional monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate in the presence of a cyanoferrate catalyst to give a green solution. Free ammonia is determined by measuring the color intensities, with and without added hypochlorite.

REQUIRED REAGENTS (Using Pillows)

Description	Quantity Per Test	Unit	Cat. No.
Free Ammonia Reagent Set (50 Tests)			26184-00
Includes: (4) 26183-68, (4) 26072-36			
Monochloramine Reagent Powder Pillows	2 pillows	25/pkg	26183-68
Hypochlorite Solution	1 drop	15 mL	26072-36

REQUIRED REAGENTS (Using AccuVac Ampuls)

DR/700 Filter Module Number 61.01	1	each	46261-00
Free Ammonia AccuVac Reagent Set (12 tests) Includes: (1) 25230-25, (1) 26072-36			25210-98
Hypochlorite Solution	5 drops	15 mL	26072-36
Monochloramine Reagent AccuVac Ampuls	2 ampuls	25/pkg	25230-25

REQUIRED APPARATUS (Using Pillows)

Clippers, large	1	each	968-00
DR/700 Filter Module Number 61.01	1	each	46261-00
Sample Cell, 10-mL with cap	3	6/pkg	24276-06

REQUIRED APPARATUS (Using AccuVac Ampuls))

Beaker, 50-mL	2	each	500-41
Vial, zeroing	1	each	21228-00

OPTIONAL REAGENTS

Nitrogen Ammonia Standard Solution, 10 mg/L as (NH_3-N)		500 mL	153-49
Nitrogen Ammonia, Voluette Ampule, 50 mg/L as (NH_3-N), 10 mL		16/pkg	14791-10

OPTIONAL APPARATUS

Ampule Breaker Kit		each	21968-00
AccuVac Drainer (to dispose of sample)		each	41036-00
AccuVac Snapper (to open AccuVac while in sample)			24052-00
Cylinder, 50 mL, mixing		each	20886-41
EasyPure System, 120 V		each	25984-00
EasyPure System, 220 V		each	25984-02
Flask, volumetric, Class A, 100 mL		each	14571-42

Pipet Filler, safety bulb	each	14651-00
Pipet, TenSette, 0.1 to 2.0 mL	each	19700-01
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Thermometer, -20 to 105 °C	each	1877-01
Wipers, disposable, Kimwipes, 30x30 cm	280/box	20970-01

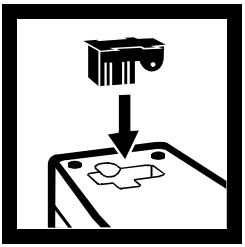
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or the distributor serving you.

OXYGEN, DISSOLVED, LR (0 to 1000 µg/L O_2)

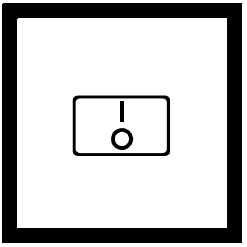
For boiler feedwater

Indigo Carmine Method (Using AccuVac Ampuls)



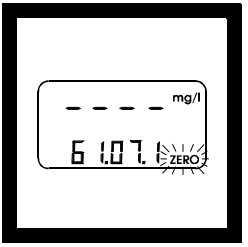
1. Install module
61.01
in a DR/700.

Note: *Samples must be analyzed on site and cannot be stored; see Sampling and Storage following this procedure.*

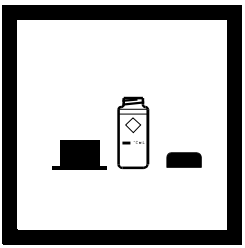


2. Press: **I/O**

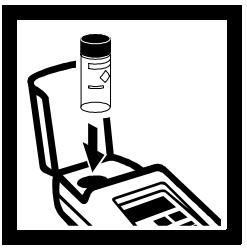
The display will show
610 nm
and module number
61.01



3. After 2 seconds, the display will show a program number, concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
61.07.1

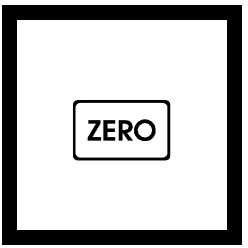


4. Fill a cell with 10 mL of sample (the blank). Cap. Fill a blue ampul cap with sample. Collect at least 40 mL of sample in a 50-mL beaker.



5. Place the blank in the cell holder.

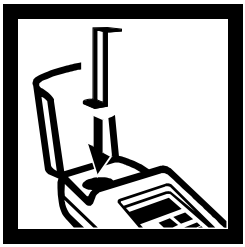
Note: *In bright light it may be necessary to close the cell compartment cover.*



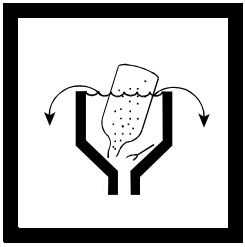
6. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 µg/L and the zero prompt will turn off.

AccuVac is a Hach Company trademark.



7. Insert the AccuVac Vial Adapter into the cell holder.

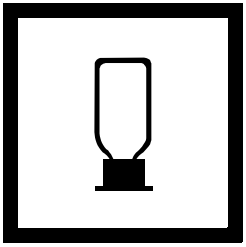


8. Fill a Low Range Dissolved Oxygen AccuVac Ampul with sample. Refer to sampling and storage for technique.

Note: *Keep tip immersed while the ampul fills completely.*

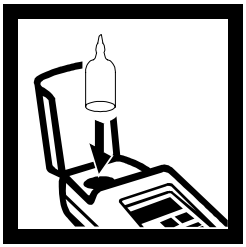
Note: *One measure of accuracy is to verify the zero concentration of the blank. Follow the steps given in the Accuracy Check.*

Note: *The ampul will contain a small piece of wire to maintain reagent quality. The solution color will be yellow.*



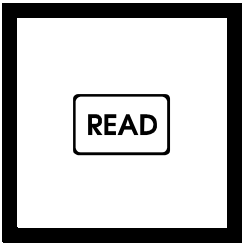
9. Without inverting the ampul, immediately place the ampul cap filled with sample securely over the ampul tip.

Note: *The cap prevents contamination with atmospheric oxygen.*



10. Wipe the ampul dry and immediately place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



11. Press: **READ**

The display will count down to 0. Then the display will show the results in µg/L dissolved oxygen.

SAMPLING AND STORAGE

The primary consideration in this procedure is to prevent contaminating the sample with atmospheric oxygen. Sampling from a stream of water that is hard plumbed to the sample source is ideal. Use a funnel to maintain a continual flow of sample and yet collect enough sample to immerse the ampul. It is important not to introduce air in place of the sample. Rubber tubing, if used, will introduce unacceptable amounts of oxygen into the sample unless the length of tubing is minimized and the flow rate is maximized. Flush the sampling system with sample for at least 5 minutes.

ACCURACY CHECK

The reagent blank for this test can be checked by following these steps:

a) Fill a 50-mL beaker with sample and add approximately 50 mg sodium hydrosulfite.

b) Immerse the tip of a Low Range Dissolved Oxygen AccuVac Ampul in the sample and break the tip. Aspirate the sample into the ampul.

c) Determine the dissolved oxygen concentration according to the preceding procedure. The result should be 0 ±1 µg/L.

INTERFERENCES

Excess amounts of sodium thioglycolate, sodium ascorbate, sodium ascorbate + sodium sulfite, sodium ascorbate + cupric sulfate, sodium nitrite, sodium sulfite, sodium thiosulfate, and hydroquinone will not reduce the oxidized form of the indicator solution; therefore, they do not cause significant interference. A 100,000-fold excess of hydrazine will begin to reduce the oxidized form of the indicator solution.

Sodium hydrosulfite will reduce the oxidized form of the indicator solution and will cause a serious interference.

STATISTICAL EVALUATION

A single operator repetitively tested samples of the same solutions, using one DR/700 and two representative lots of AccuVacs. Testing 300 µg/L O_2 samples, the standard deviation was ±27.7 µg/L O_2.

Testing zero concentration samples, the limit of detection was 16.1 µg/L O_2. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980**, 52, 2242-2249).

SUMMARY OF METHOD

The Low Range Dissolved Oxygen AccuVac Ampul contains reagent vacuum sealed in a 12-mL ampul. When the AccuVac ampul is broken open in a sample containing dissolved oxygen, the yellow solution will turn blue. The blue color development is proportional to the concentration of dissolved oxygen.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Low Range Dissolved Oxygen AccuVac Ampuls	1 ampul	25/pkg	25010-25

REQUIRED APPARATUS

Beaker, 50 mL	1	each	500-41
DR/700 Filter Module Number 60.01	1	each	46261-00

OPTIONAL REAGENTS AND APPARATUS

AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac, Vial, DR/700	each	46025-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Sodium Hydrosulfite, technical grade	500 g	294-34

For Technical Assistance Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

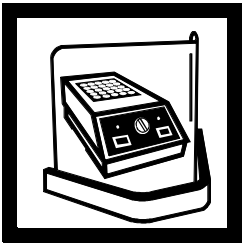
OXYGEN DEMAND, CHEMICAL (COD)

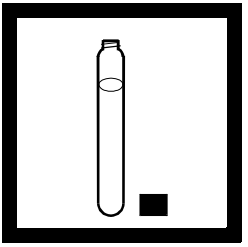
(0 to 1,500 and 0 to 15,000 mg/L)

For water, wastewater and seawater

Reactor Digestion Method*; USEPA approved for reporting (0 to 1,500 mg/L range)†

DIGESTION





1. Homogenize a 100 mL sample for 2 minutes in a blender.

Note: *For samples with high solid content, blending ensures distribution of solids and improves accuracy and reproducibility.*

Note: *Pour homogenized sample into a 250-mL beaker and stir with magnetic stirrer.*

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

2. Turn on the COD Reactor. Preheat to 150 °C. Place the plastic shield in front of the reactor.

Caution: Ensure safety devices are in place to protect analyst from splattering should reagent leaking occur.

3. Remove cap of a COD Digestion Reagent Vial of the desired range:

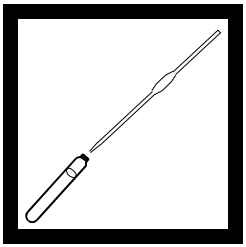
Sample Concentration Range (mg/L)	COD Digestion Reagent Vial Type
0 to 1,500	High Range
0 to 15,000	High Range Plus

Note: *The reagent mixture is light sensitive. Keep unused vials in the opaque shipping container, in a refrigerator if possible. The amount of light striking the vials during testing will not affect the results.*

Caution

Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if improperly handled or accidentally misused. Please read all warnings and the safety section of this manual. Appropriate eye protection and clothing should be used for adequate user protection. If contact occurs, flush the affected area with running water. Follow instructions carefully.

*Jirka, A.M.; Carter, M.J. *Analytical Chemistry,* **1975,** 47(8), 1397.
†*Federal Register,* **April 21, 1980,** 45(78), 26811-26812.



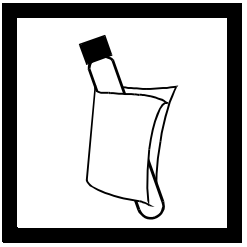
4. Hold the vial at a 45-degree angle. Pipet 2.00 mL (0.2 mL for 0 to 15,000 range) of sample into the vial.

Note: *To ensure a uniform sample aliquot, stir sample with magnetic stirrer while drawing sample aliquot into the pipet.*

Note: *For greater accuracy, three replicates should be analyzed and the results averaged. This is especially important for the High Range Plus test.*

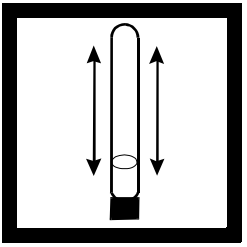
Note: *Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Do not run tests with vials which have been spilled. If some spills, wash with running water.*

Note: *For proof of accuracy, use COD standard solutions (preparation given in the Accuracy Check) in place of the sample.*



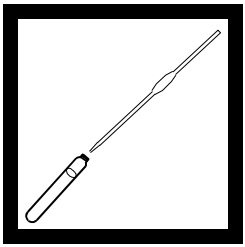
5. Replace the vial cap tightly. Rinse the COD vial with demineralized water and wipe the vial clean with a paper towel.

Note: *When using High Range Plus COD vials, tighten the vial cap with the COD cap tool provided.*



6. Hold the vial by the cap and over a sink. Invert several times to mix the contents. Place the vial in the preheated COD Reactor.

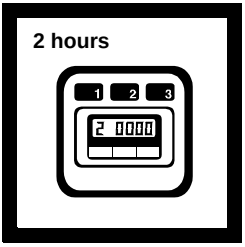
Note: *The vial will become hot during mixing.*



7. Prepare a blank by repeating Steps 3 to 6, substituting 2.00 mL (0.2 mL for 0 to 15,000 mg/L range) demineralized water for the sample.

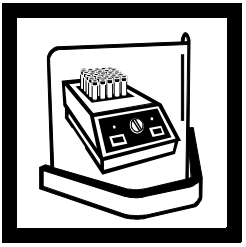
Note: *Be sure the pipet is well rinsed or use a clean pipet.*

Note: *One blank must be run with each set of samples. All tests (samples and blanks) should be run with the same lot of vials. The lot number appears on the container label.*

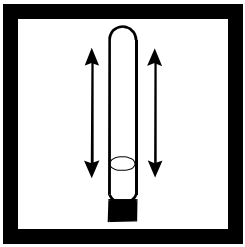


8. Heat the vials for 2 hours.

Note: *Many wastewater samples contain easily digested materials that are digested in less than 2 hours. If desired, measure the concentration (while still hot) at 15-minute intervals until it remains unchanged. At this point, the sample is completely digested. Cool vials to room temperature for final measurement.*



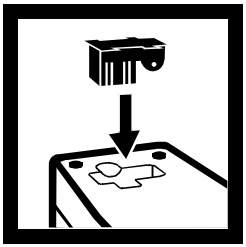
9. Turn the reactor off. Wait 20 minutes for the vials to cool to 120 °C or less.



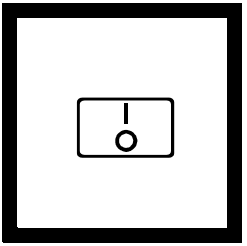
10. Invert each vial several times while still warm. Place the vials into a rack. Wait until the vials have cooled to room temperature.

Note: *If a pure green color appears in the reacted sample, the reagent capacity may have been exceeded. Measure the COD and if necessary, repeat the test with a diluted sample or use a higher range digestion vial.*

COLORIMETRIC DETERMINATION, 0 to 1,500 and 0 to 15,000 mg/L COD

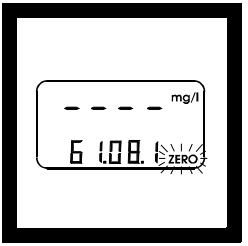


1. Install module **61.01** in a DR/700.

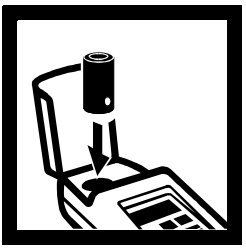


2. Press: **I/O**

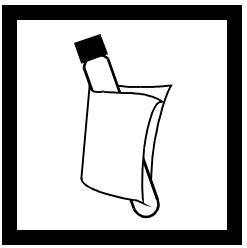
The display will show **610 nm** and module number **61.01**



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **61.08.1**

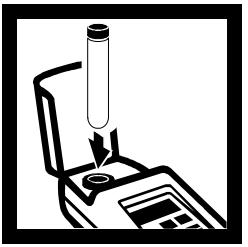


4. Fully insert a COD Vial Adapter into the cell holder with the tabs in the square slot.



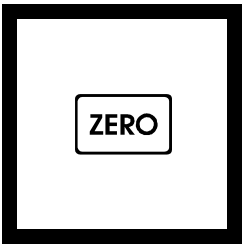
5. Clean the outside of the blank with a towel.

Note: *Wiping with a damp towel followed by a dry one will remove fingerprints or other marks.*



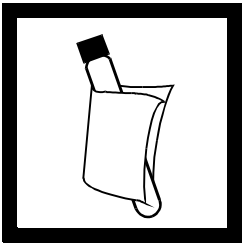
6. Place the blank into the adaptor with the Hach logo facing the front of the instrument.

Note: *Avoid bright light when making measurements.*



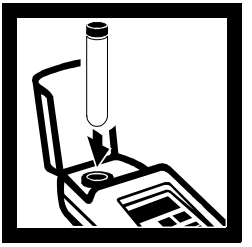
7. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L, and the zero prompt will turn off.

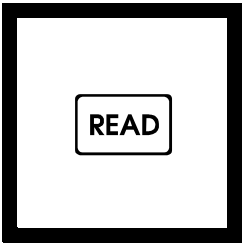


8. Clean the outside of the sample vial with a towel.

Note: *Wiping with a damp towel, followed by a dry one will remove fingerprints or other marks.*



9. Place the sample vial in the adapter with the Hach logo facing the front of the instrument.



10. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L COD.

0 to 15,000 mg/L Note: *When High Range Plus COD Digestion Reagent Vials are used, multiply the displayed value by 10.*

Note: *For most accurate results with samples near 1,500 mg/L COD or 15,000 mg/L COD, repeat the analysis with a diluted sample.*

SAMPLING AND STORAGE

Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days. Correct results for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information.

ACCURACY CHECK

Standard Solution Method

Check the accuracy of the 0 to 1,500 mg/L range by using either a 300 mg/L or 1000 mg/L COD Standard Solution. Use 2 mL of one of these solutions as the sample volume; the expected result will be 300 or 1000 mg/L COD respectively.

Or, prepare a 500 mg/L standard by dissolving 425 mg of dried (120 °C, overnight) KHP. Dilute to 1 liter with demineralized water.

Check the accuracy of the 0 to 15,000 mg/L range by using a 10,000 mg/L COD standard solution. Prepare the 10,000 mg/L solution by dissolving 8.500 g of dried (120 °C, overnight) KHP in 1 liter of demineralized water. Use 0.2 mL of this solution as the sample volume; the expected result will be 10,000 mg/L COD.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagents.

For 0-1,500 range, a 1200 mg/L COD sample was tested and the standard deviation was ±29 mg/L COD. Multiply by 10 for the 0-15,000 range.

Testing zero concentration samples, the limit of detection was 65 mg/L COD. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

Chloride is the primary interference when determining COD concentration. Each COD vial contains mercuric sulfate that will eliminate chloride interference up to the level specified in column 1 in the table below. Dilute samples with higher chloride concentrations. Dilute the sample enough to reduce the chloride concentration to the level given in column 2.

Vial Type Used	(1) Maximum Cl^- concentration in sample (mg/L)	(2) Maximum Cl^- concentration of diluted samples (mg/L)	(3) Maximum Cl^- concentration in sample when 0.50 $HgSO_4$ added (mg/L)
High Range	2000	1000	4000
High Range Plus	20,000	10,000	40,000

If sample dilution will cause the COD concentration to be too low for accurate determination, add 0.50 g of mercuric sulfate ($HgSO_4$) to each COD vial before the sample is added. The additional mercuric sulfate will raise the maximum chloride concentration allowable to the level given in column 3.

BLANKS FOR COLORIMETRIC DETERMINATION

The blank may be used repeatedly for measurements using the same lot of vials. Store it in the dark. Monitor decomposition by measuring the absorbance at the appropriate wavelength (420 or 620 nm). Zero the instrument in the absorbance mode, using a vial containing 5 mL of demineralized water and measure the absorbance of the blank. Record the value. Prepare a new blank when the absorbance has changed by about 0.01 absorbance units.

SUMMARY OF METHOD

The mg/L COD results are defined as the mg of O_2 consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion ($Cr_2O_7^{2-}$) to green chromic ion (Cr^{3+}). When the 0-1,500 mg/L or 0-15,000 mg/L colorimetric method is used, the amount of Cr^{3+} produced is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex chloride.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Select the appropriate COD Digestion Reagent Vial:			
High Range, 0 to 1,500 mg/L COD	1 to 2 vials	25/pkg	21259-25
High Range Plus, 0 to 15,000 mg/L COD	1 to 2 vials	25/pkg	24159-25
Water, demineralized	varies	4 L	272-56

REQUIRED APPARATUS

Cap Tool, COD	1	each	45587-00
COD Reactor, 120/240 Vac	1	each	45600-00
COD Vial Adapter, DR/700	1	each	46008-00
DR/700 Filter Module Number 61.01	1	each	46261-00
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet, volumetric, Class A, 2.00 mL	1	each	14515-36
Pipet Filler, safety bulb	1	each	14651-00
Pipet Tips, for 19700-01 Tensette Pipet	1	50/pkg*	21856-96
Test Tube Rack	1 to 2 racks	each	18641-00

OPTIONAL REAGENTS

Description	Unit	Cat. No.
COD Digestion Reagent Vials, 0 to 1,500 mg/L COD	150/pkg	21259-15
COD Standard Solution, 300 mg/L	236 mL	12186-31
COD Standard Solution, 1000 mg/L	236 mL	22539-31
Potassium Acid Phthalate, ACS	500 g	315-34
Sulfuric Acid, ACS	500 mL*	979-49
Mercuric Sulfate, ACS	28 g*	1915-20

OPTIONAL APPARATUS

Beaker, 250 mL	each	500-46
Blender		obtain locally
Cylinder, graduated, 5 mL	each	508-37
Electromagnetic Stirrer, 120 V, with electrode stand	each	45300-01

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Electromagnetic Stirrer, 230 V, with electrode stand	each	45300-02
Flask, volumetric, Class A, 1000 mL	each	14574-53
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, serological, 5 mL	each	532-37
Pipet, volumetric, Class A, 10 mL	each	14515-38
Safety shield, for COD reactor	each	23810-00
Spoon, measuring, 0.5 g	each	907-00
Stir Bar, 22.2 x 4.76 mm ($^{7}/_{8}$" x $^{3}/_{16}$")	each	45315-00
Stir Bar Retriever	each	15232-00

RELATED LITERATURE

Ask for your copy by literature code number.

Title	Literature Code No.
COD Disposal Information Brochure	4144

For Technical Assistance, Prices and Ordering

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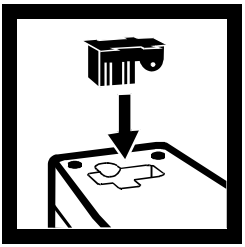
Outside the U.S.A. - Contact the Hach office or distributor serving you.

*Contact Hach for larger sizes.

OZONE

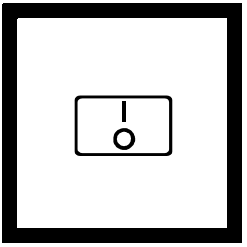
(0 to 0.25 mg/L O_3, 0 to 0.75 mg/L O_3 or 0 to 1.50 mg/L O_3)
For water

Indigo Method (Using AccuVac Ampuls)



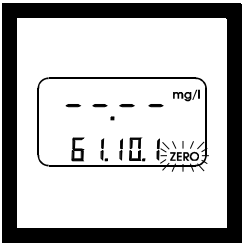
1. Install module
61.01
in a DR/700.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

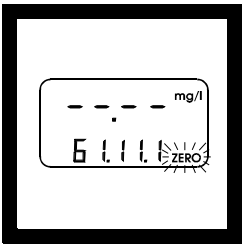


2. Press: **I/O**

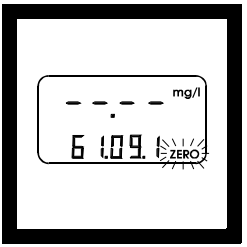
The display will show
610 nm
and module number
61.01



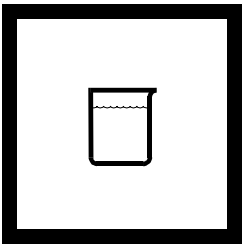
3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the display shows
61.10.1
for low range
(0-0.25 mg/L).



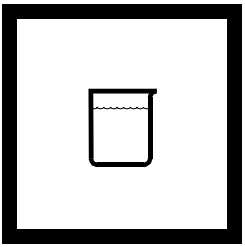
OR
Press the **UP ARROW** key until the display shows
61.11.1
for mid range
(0-0.75 mg/L).



OR
Press the **UP ARROW** key until the display shows
61.09.1
for high range
(0-1.50 mg/L).

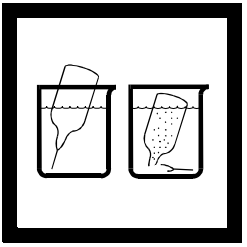


4. Gently collect at least 40 mL of sample in a 50-mL beaker.



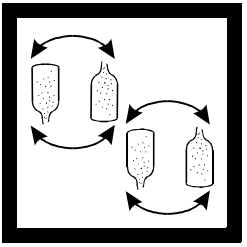
5. Collect at least 40 mL of ozone-free water (blank) in another 50-mL beaker.

Note: *Ozone-free water used for the blank may be demineralized water or tap water.*



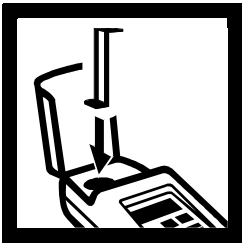
6. Fill one Indigo Ozone Reagent AccuVac Ampul with the sample and one ampul with the blank.

Note: *Keep the tip immersed while the ampul fills completely.*

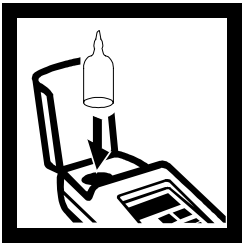


7. Quickly invert both ampuls several times to mix. Wipe off any liquid or fingerprints.

Note: *Part of the blue color will be bleached if ozone is present.*



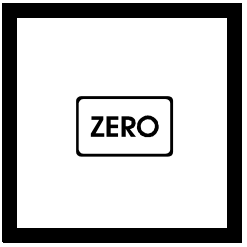
8. Insert the AccuVac Vial Adapter into the cell holder.



9. Place the **sample** AccuVac Ampul into the cell holder.

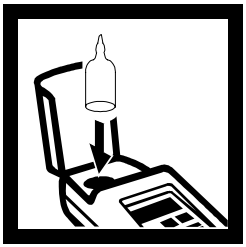
Note: *Standardization for this procedure is intentionally reversed.*

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



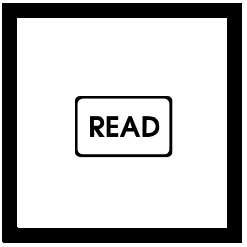
10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 and the zero prompt will turn off.



11. Place the AccuVac ampul containing the **blank** into the cell holder.

Note: *In bright sunlight, it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the result in mg/L ozone (O_3).

SAMPLING

The chief consideration when collecting a sample is to prevent the escape of ozone from the sample. The sample should be collected gently and analyzed immediately. Warming the sample, or disturbing the sample by stirring or shaking, will result in ozone loss. After collecting the sample, do not transfer it from one container to another unless absolutely necessary.

STABILITY OF INDIGO REAGENT

Indigo is light-sensitive. Therefore, the AccuVac ampuls should be kept in the dark at all times. However, the indigo solution decomposes slowly under room light after filling with sample. The blank ampul can be used for multiple measurements during the same day.

SUMMARY OF METHOD

The reagent formulation adjusts the sample pH to 2.5 after the ampul has filled. The indigo reagent reacts immediately and quantitatively with ozone.

The blue color of indigo is bleached in proportion to the amount of ozone present in the sample. Other reagents in the formulation prevent

chlorine interference. No transfer of sample is needed in the procedure. Therefore, ozone loss due to sampling is eliminated.

STATISTICAL EVALUATION

Low Range

A single operator repetitively tested samples of the same solutions, using one DR/700 and two representative lots of AccuVacs. Testing 0.15 mg/L O_3 samples, the standard deviation was ±0.009 mg/L O_3.

Testing zero concentration samples, the limit of detection was 0.024 mg/L.

Medium Range

A single operator repetitively tested samples of the same solutions, using one DR/700 and two representative lots of AccuVacs. Testing 0.28 mg/L O_3 samples, the standard deviation was ±0.018 mg/L O_3.

Testing zero concentration samples, the limit of detection was 0.030 mg/L.

High Range

A single operator repetitively tested samples of the same solutions, using one DR/700 and two representative lots of AccuVacs. Testing 0.96 mg/L O_3 samples, the standard deviation was ±0.024 mg/L O_3.

Testing zero concentration samples, the limit of detection was 0.037 mg/L. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** 52, 2242-2249).

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Select one or more based on range:			
Ozone AccuVac Ampuls			
0-0.25 mg/L	2 ampuls	25/pkg	25160-25
0-0.75 mg/L	2 ampuls	25/pkg	25170-25
0-1.50 mg/L	2 ampuls	25/pkg	25180-25

REQUIRED APPARATUS

Beaker, 50 mL	2	each	500-41
DR/700 Filter Module			
Number 61.01	1	each	46261-00

OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit	each	24052-00
Adapter, AccuVac Vial, DR/700	each	46025-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

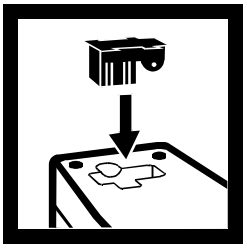
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information,
Outside the U.S.A. - Contact the Hach office or distributor serving you.

SULFIDE (0 to 0.600 mg/L S^{2-})

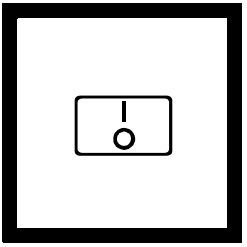
For water, wastewater and seawater

Methylene Blue Method*; USEPA accepted for reporting**



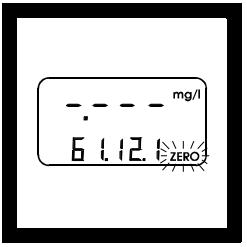
1. Install module **61.01** in a DR/700.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis. Avoid excessive agitation.*



2. Press: **I/O**

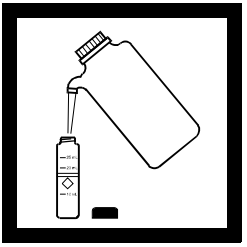
The display will show **610 nm** and module number **61.01**



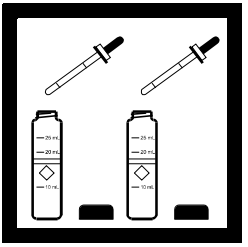
3. After 2 seconds, the display will show a program number, concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **61.12.1**

4. Fill a 25-mL sample cell to the 25 mL line with sample (the prepared sample).

Note: *For turbid samples, see Interferences following these steps for pretreatment instructions.*

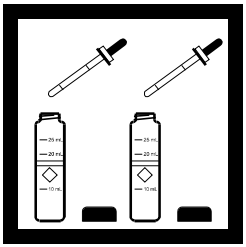


5. Fill another 25-mL cell to the 25 mL line with demineralized water (the blank).



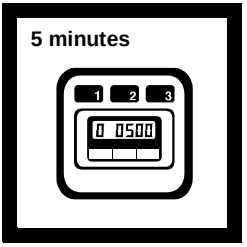
6. Add 1.0 mL of Sulfide 1 Reagent to each cell. Swirl to mix.

*Adapted from *Standard Methods for the Examination of Water and Wastewater.*
**Procedure is equivalent to USEPA method 376.2 and Standard Method 4500-S^{2-}D for wastewater.

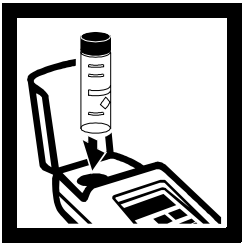


7. Add 1.0 mL of Sulfide 2 Reagent to each cell and cap. Immediately swirl to mix.

Note: *A pink color will develop. Then the solution will turn blue if sulfide is present.*

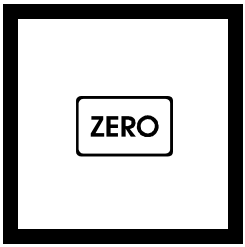


8. Wait 5 minutes.



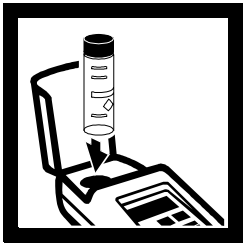
9. Place the blank in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover, Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



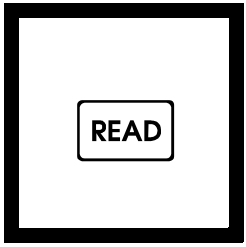
10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.



11. Immediately place the prepared sample into the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight, it may be necessary to close the cell compartment cover, Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L sulfide (S^{2-}).

ACCURACY CHECK

Standard Solution Method

Sulfide standard solutions are very unstable and should be prepared from sodium sulfate and standardized as described in *Standard Methods for the Examination of Water and Wastewater,* 17th ed., page 4-195.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions using one DR/700, matched sample cells and two representative lots of testing reagents, Testing 0.595 mg/L S^{2-} concentration samples, the standard deviation was ±0.008 mg/L S^{2-}.

Testing zero concentration samples, the limit of detection was 0.010 mg/L S^{2-}. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52,* 2242-2249).

INTERFERENCES

For turbid samples, prepare a sulfide-free blank as follows. Use it in place of the demineralized water blank in the procedure.

a) Measure 25 mL of sample into a 50-mL erlenmeyer flask.

b) Add Bromine Water dropwise with constant swirling until a yellow color remains.

c) Add Phenol Solution dropwise until the yellow color just disappears. Use of this blank solution will compensate for turbidity in the sample.

Strong reducing substances such as sulfite, thiosulfate and hydrosulfite interfere by reducing the blue color or preventing its development. High concentrations of sulfide may inhibit full color development and require sample dilution. Some sulfide loss may occur when the sample is diluted.

SUMMARY OF METHOD

Hydrogen sulfide and acid-soluble metal sulfides react with N,N-dimethyl-p-phenylenediamine oxalate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration.

High sulfide levels in oil field waters may be determined after proper dilution.

Determine soluble sulfides by centrifuging the sample in completely filled, capped tubes and analyzing the supernatant. Insoluble sulfides are then estimated by subtracting the soluble sulfide concentration from the total sulfide result.

REQUIRED REAGENTS

	Cat. No.
Sulfide Reagent Set (100 tests)	22445-00
Includes: (2) 1816-14, (2) 1817-14	

Description	Quantity Per Test	Unit	Cat. No
Sulfide 1 Reagent	2 mL	100 mL MDB	1816-32
Sulfide 2 Reagent	2 mL	100 mL MDB	1817-32

REQUIRED APPARATUS

Description	Quantity Per Test	Unit	Cat. No
DR/700 Filter Module Number 61.01	1	each	46261-00

OPTIONAL REAGENTS

Bromine Water, 30 g/L		29 mL	2211-20
Phenol Solution, 30 g/L		29 mL	2112-20
Sodium Sulfide, ACS		114 g	785-14

OPTIONAL APPARATUS

Cap for 10- and 25- mL sample cells		12/pkg	24018-12
Cylinder, graduated, 25 mL		each	508-40
Dropper, for 1 oz bottle		each	2258-00
Flask, erlenmeyer, 50 mL		each	505-41
Sample Cell, 10-mL with screw cap		6/pkg	24276-06
Sample Cell, 25-mL with screw cap		6/pkg	24019-06
Standard Methods for the Examination of Water and Wastewater, 18th ed.		each	22708-00

For Technical Assistance, Prices and Ordering

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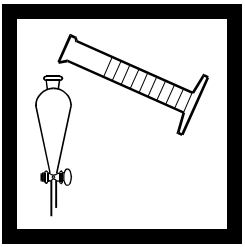
Outside the U.S.A. - Contact the Hach office or the distributor serving you.

SURFACTANTS, ANIONIC (0 to 0.300 mg/L)

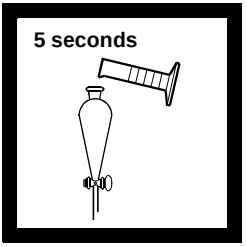
For water, wastewater and seawater

(Also called Detergents) Crystal Violet Method*

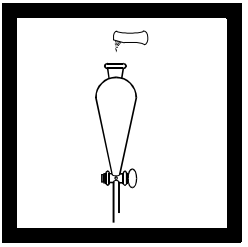


1. Measure 300 mL of sample in a 500-mL separatory funnel.

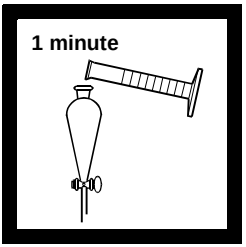
Note: *If sample cannot be analyzed immediately, see Sampling and Storage following these steps.*



2. Add 10 mL of Sulfate Buffer Solution. Stopper and shake the funnel for five seconds.



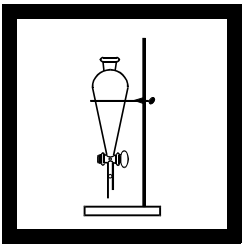
3. Add the contents of one Detergent Reagent Powder Pillow. Stopper and shake to dissolve.



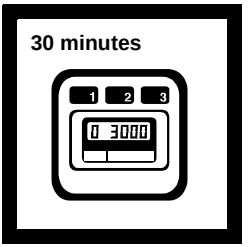
4. Add 30 mL of benzene to the separatory funnel. Stopper and shake gently for one minute.

Note: *Spilled reagent will affect test accuracy and is hazardous to skin and other materials.*

Note: *Use benzene only with adequate ventilation.*



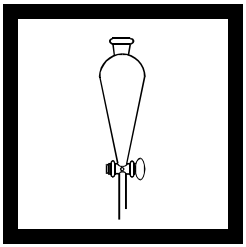
5. Place the separatory funnel in a support stand.



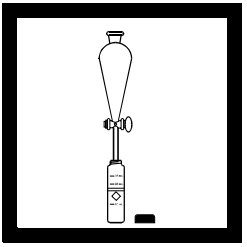
6. Wait 30 minutes.

Note: *Excessive agitation may cause an emulsion to form, requiring a longer time for phase separation. If this occurs, remove most of the lower water layer and gently agitate the separatory funnel with a clean inert object (such as a Teflon-coated magnetic stirring bar) in the funnel.*

*Adapted from *Analytical Chemistry*, **1966**, *38*, 791

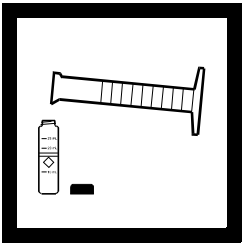


7. Remove the stopper from the separatory funnel. Drain the water layer off the bottom and discard it.

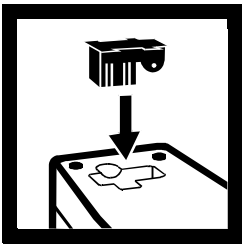


8. Fill a 25-mL cell to the 25-mL line with the top benzene layer (the prepared sample). Cap.

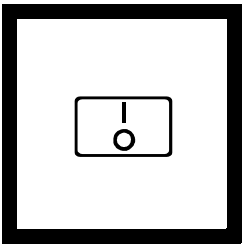
Note: *The benzene extract cannot be filtered before color measurement. Filtration will remove the color.*



9. Fill a 25-mL cell to the 25-mL line with pure benzene (the blank). Cap.

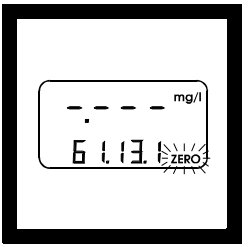


10. Install module number
61.01
in a DR/700.

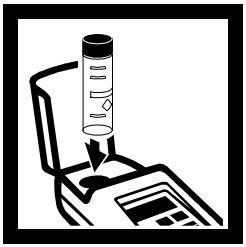


11. Press: **I/O**

The display will show
610 nm
and module number
61.01

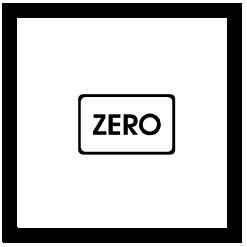


12. After 2 seconds, the display will show a program number, the concentration units, decimal position, and zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
61.13.1



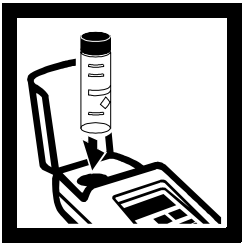
13. Place the blank into the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



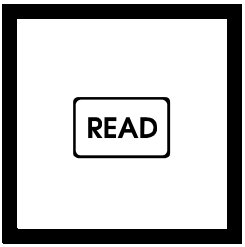
14. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.



15. Place the prepared sample in the cell holder.

Note: *Typical indoor lighting permits the DR/700 to operate with the cell compartment cover open. In bright sunlight it may be necessary to close the cell compartment cover. Transfer 10 mL of the blank solution to a 10-mL cell. If the 10-mL cell is used for the blank, another 10-mL cell must be used for the sample.*



16. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L anionic surfactants.

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Analyze sample as soon as possible after collection. If prompt analysis is not possible, store samples up to 24 hours by cooling to 4 °C (39 °F) or below. Warm to room temperature before analysis.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Detergent Voluette Ampule Standard Solution, 60 mg/L as LAS.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 300-mL samples. Mix well.

c) Analyze and compare the results with the sample. The anionic surfactants reading should increases 0.02 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 0.180 mg/L as LAS concentration samples, the standard deviation was ±0.0036 mg/L as LAS.

Testing zero concentration samples, the limit of detection was 0.0008 mg/L as LAS. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

Perchlorate and periodate ions will interfere. High concentrations of chloride, such as those levels found in seawater and brines, will cause low results.

SUMMARY OF METHOD

Surfactants, ABS (alkyl benzene sulfonate) or LAS (linear alklyl sulfonate) are determined by ion association with crystal violet dye and extraction of the ion pair complex into benzene.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Benzene, ACS	55 mL	500 mL	14440-49
Buffer Solution, sulfate, type	10 mL	500 mL	452-49
Detergent Reagent Powder Pillows	1	25/pkg	1008-68

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, 25 mL, graduated	1	each	2172-49
Cylinder, 50 mL, graduated	1	each	508-41
Cylinder, 500 mL, graduated	1	each	508-49
DR/700 Filter Module Number 61.01	1	each	46261-00
Funnel, 500 mL separatory	1	each	520-49
Ring, support, 4 inch	1	each	580-01
Stand support, 127 x 203 mm (5 x 8")	1	each	563-00

OPTIONAL REAGENTS

Acetone, ACS	500 mL	14429-49
Detergent Standard Solution, Voluette Ampule, 60 mg/L as LAS, 10 mL	16/pkg	14271-10

OPTIONAL APPARATUS

Ampule Breaker	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

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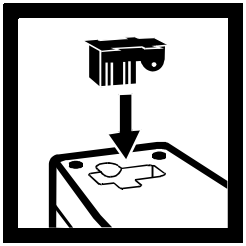
Outside the U.S.A. - Contact the Hach office or distributor serving you.

ZINC (0 to 3.00 mg/L)

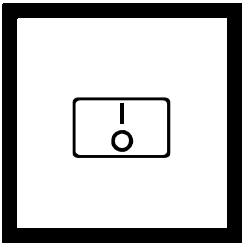
For water and wastewater

Zincon Method*; USEPA approved for reporting (digestion is required; see Section 1)†.



1. Install module
61.01
in a DR/700.

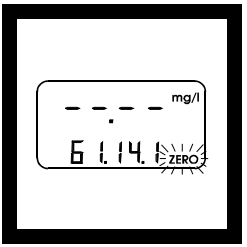
Note: *If sample cannot be analyzed immediately, see Sampling and Storage following these steps. Adjust the pH of stored sample before analysis.*



2. Press: **I/O**

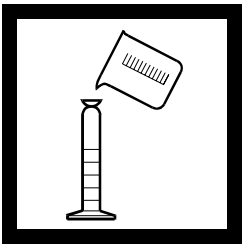
The display will show
610 nm
and module number
61.01

Note: *Total zinc determination requires a prior digestion; use either the Digesdahl or mild digestion (Section 1). Adjust the digested sample to pH 4 to 5; see Sampling and Storage following these steps.*



3. After 2 seconds, the display will show a program number, concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
61.14.1

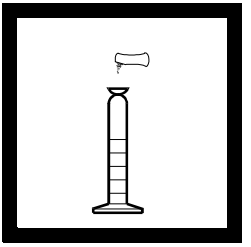
*Adapted from *Standard Methods for the Examination of Water and Wastewater.*
†*Federal Register*, **May 1980**, *45(105)*, 36166



4. Fill a 25-mL graduated mixing cylinder to the 20-mL mark with sample.

Note: *For proof of accuracy, use a 0.5 mg/L zinc standard solution (preparation given in Accuracy Check) in place of the sample.*

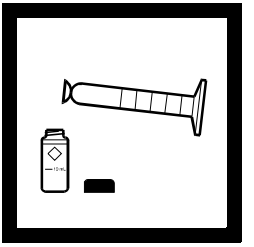
Note: *Perform the test using the cells described. Using different containers in Steps 7 and 8 may affect results.*



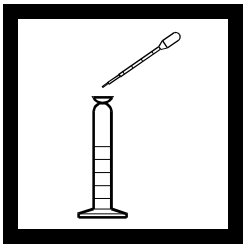
5. Add the contents of one ZincoVer 5 Reagent Powder Pillow. Cap. Invert several times until the powder dissolves completely.

Note: *Inconsistent readings may result if all the powder is not dissolved.*

Note: *At this point the sample color should be orange. If the color is brown or blue, dilute the sample and repeat the test. Either the zinc concentration is too high or an interfering metal is present.*

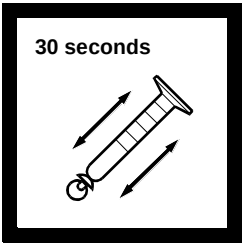


6. Measure 10 mL of the solution into a 10-mL sample cell (the blank). Cap the cell.



7. Add 0.5 mL of cyclohexanone to the remaining solution in the cylinder.

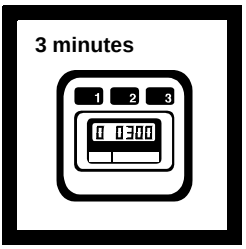
Note: *Use a plastic dropper because rubber bulbs may contaminate the cyclohexanone.*



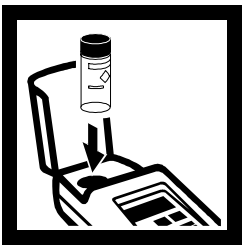
8. Stopper the cylinder (the prepared sample). Shake for 30 seconds.

Note: *The sample color will be reddish-brown, brown or blue depending on the zinc concentration.*

9. Pour the prepared sample into a 10-mL sample cell. Cap the cell.

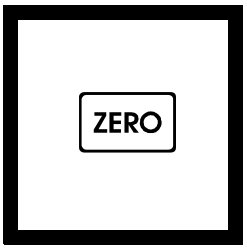


10. Wait 3 minutes.



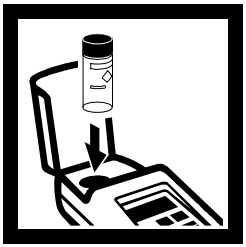
11. Place the blank in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



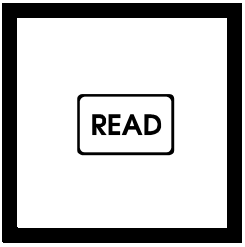
12. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



13. Within 10 minutes of the three-minute period, place the prepared sample in the cell holder.

Note: *In bright light it may be necessary to close the cell compartment cover.*



14. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L zinc (Zn).

Note: *Determine a reagent blank for each lot of reagent by running the procedure on demineralized water. Subtract this value from all following results obtained in Step 14.*

SAMPLING AND STORAGE

Collect samples in acid-washed plastic bottles. For storage, adjust the pH to 2 or less with nitric acid (about 2 mL per liter). The preserved samples can be stored for up to six months at room temperature. Adjust the pH to 4 to 5 with 5.0 N Sodium Hydroxide before analysis. Do not exceed pH 5, as zinc may be lost as a precipitate. Correct the test result for volume additions; see Sampling and Storage, Volume Additions, (Section I) for more information. If only dissolved zinc is to be determined, filter the sample before acid addition.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Zinc Voluette Ampule Standard Solution, 25 mg/L.

b) Use the TenSette pipet to add 0.2, 0.4, and 0.6 mL of standard to three 50-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The zinc concentration should increase 0.1 mg/L for each 0.2 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method
Prepare a 0.5 mg/L zinc standard solution by diluting 0.50 mL of zinc standard solution, 100 mg/L as Zn, to 100 mL with demineralized water. Prepare this solution daily.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 0.80 mg/L Zn concentration samples, the standard deviation was ±0.0068 mg/L Zn.

Testing zero concentration samples, the limit of detection was 0.022 mg/L Zn. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

The following may interfere when present in concentration exceeding those listed below:

Aluminum	6 mg/L
Cadmium	0.5 mg/L
Copper	5 mg/L
Iron (ferric)	7 mg/L
Manganese	5 mg/L
Nickel	5 mg/L

Large amounts of organic material may interfere. Perform the mild digestion (Section I), to eliminate this interference.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

WASTE DISPOSAL

ZincoVer 5 Reagent contains cyanide. Dispose of it safely as follows:

a) Use good ventilation or a fume hood.

b) Add the waste while stirring to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochlorite (household bleach).

c) Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.

d) Flush the solution down the drain with a large excess of water.

SUMMARY OF METHOD

Zinc and other metals in the sample are complexed with cyanide. The addition of cyclohexanone causes a selective release of zinc. The zinc then reacts with 2-carboxy-2'-hydroxy-5'-sulfoformazyl benzene (zincon) indicator. The zinc concentration is proportional to the resulting blue color.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Cyclohexanone	0.5 mL	100 mL MDB	14033-37
ZincoVer 5 Reagent Powder Pillows	1 pillow	100/pkg	21066-69

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, mixing, 25 mL	1	each	1896-40
DR/700 Filter Module Number 61.01	1	each	46261-00
Dropper, plastic, 0.5 and 1.0 mL marks	1	10/pkg	21247-10

OPTIONAL REAGENTS

Bleach, household	1 gal	obtain locally
Hydrochloric Acid, 6 N	500 mL	884-49
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	59 mL* SCDB	2450-26
Sodium Hydroxide, 50% w/w	500 mL	2180-49
Water, demineralized	4 L	272-56
Zinc Standard Solution, 100 mg/L	100 mL	2378-42
Zinc Standard Solution, Voluette ampule, 25 mg/L as Zn, 10 mL	16/pkg	14246-10

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Aspirator, vacuum	each	2131-00
Beaker, glass, 1000 mL	each	500-53
Caps for 10- and 25-mL sample cells	12/pkg	24018-12
Cylinder, graduated, 100 mL	each	508-42
Dropper, plastic, 0.5 & 1.0 mL	10/pkg	21247-10
Filter Discs, glass, 47 mm	100/pkg	2530-00
Filter Holder, 47 mm	each	2340-00
Flask, erlenmeyer, 250 mL	each	505-46
Flask, volumetric, Class A, 100 mL	each	14574-42
Hot Plate, micro, 115 V	each	12067-0
Hot Plate, micro, 230 V	each	12067-02
pH Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet Filler, safety bulb	each	14651-00
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet, TenSette, tips for 19700-01	50/pkg	21856-96
Pipet, volumetric, 5 mL	each	515-37
Pipet, volumetric, Class A, 0.5 mL	each	14515-34
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

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Outside the U.S.A. - Contact the Hach office or distributor serving you.

*Contact Hach for larger sizes.
†100 Tests equals 100 samples and 100 blanks.

Module 69.01
690 nm

Hach warrants equipment it manufactures against defective materials or workmanship for at least one year from the shipping date. Warranties do not apply to limited-life electrical components such as batteries. Full warranty information is located on the reverse side of Hach invoices.

IMPORTANT NOTICE

A lamp intensity adjustment must be performed:
- Before first or initial use
- When new filter modules are installed
- On each filter module when the lamp is replaced

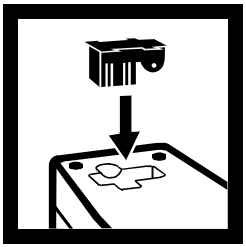
Refer to Lamp Intensity Adjustment in the instrument manual.

Table of Contents for 690-nm parameters

OXYGEN, DISSOLVED, SHR (0 to 40.0 mg/L O_2)

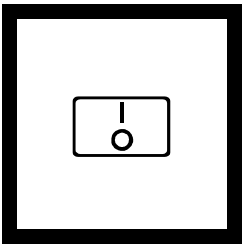
For aquaculture

Super High Range Method



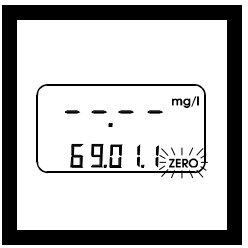
1. Install module number
69.01
in a DR/700.

Note: *The sample must be analyzed on site and cannot be stored; see Sampling and Storage below.*



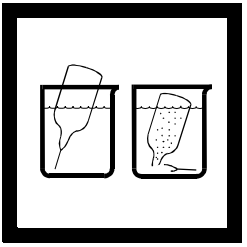
2. Press: **I/O**

The display will show
690 nm
and module number
69.01



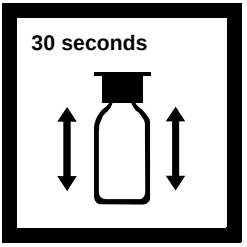
3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
69.01.1

4. Fill a blue ampul cap with sample. Then fill a cell with 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.



5. Fill a High Range Dissolved Oxygen AccuVac Ampul with sample.

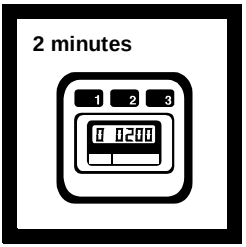
Note: *Keep the tip immersed while the ampul fills completely.*



6. Without inverting the ampul, immediately place the ampul cap that has been filled with sample securely over the tip of the ampul. Shake the ampul for approximately 30 seconds.

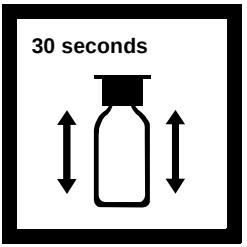
Note: *A small amount of undissolved HRDO Reagent does not affect results.*

Note: *The cap prevents contamination with atmospheric oxygen.*

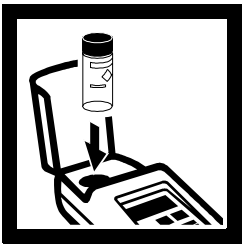


7. Wait 2 minutes.

Note: *A two-minute reaction period enables oxygen, which was degassed during aspiration, to redissolve and react.*

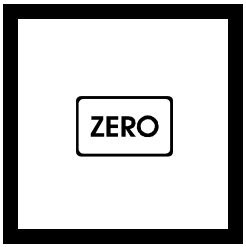


8. After the two minute period, shake the ampul for 30 seconds.



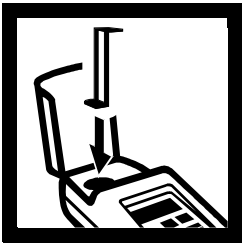
9. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

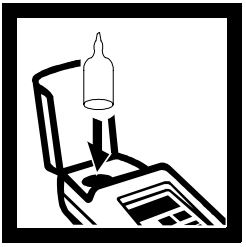


10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

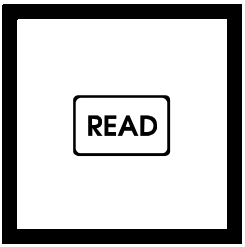


11. Insert the AccuVac Vial Adapter into the cell holder.



12. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



13. Press: **READ**

The display will count down to 0. Then the display will show the result in mg/L dissolved oxygen (O_2).

SAMPLING AND STORAGE

The foremost consideration in sampling with the High Range Dissolved Oxygen AccuVac Ampul is to prevent the sample from becoming contaminated with atmospheric oxygen. This is accomplished by capping the ampul with an ampul cap in the interval between breaking open the ampul and reading the absorbance. If the ampul is securely capped, the ampul should be safe from contamination for several hours. The absorbance will decrease by approximately 3% during the first hour and will not change significantly afterwards.

Sampling and sample handling are important considerations in obtaining meaningful results. The dissolved oxygen content of the water being tested can be expected to change with depth, turbulence, temperature, sludge deposits, light, microbial action, mixing, travel time and other factors. A single dissolved oxygen test rarely reflects the accurate overall condition of a body of water. Several samples taken at different times, locations and depths are recommended for most reliable results. Samples must be tested immediately upon collection although only a small error results if the absorbance reading is taken several hours later.

ACCURACY CHECK

The results of this procedure may be compared with the results of a titrimetric procedure or dissolved oxygen meter.

STATISTICAL EVALUATION

A single operator repetitively tested samples of the same solutions, using one DR/700 and two representative lots of AccuVac Ampuls. Testing 24.0 mg/L O_2 samples, the standard deviation was ±1.08 mg/L O_2.

Testing zero concentration samples, the limit of detection was 1.60 mg/L O_2. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52,* 2242-2249).

INTERFERENCES

The following do not interfere at a level of 10 mg/L which is in excess of naturally occurring levels of Cr^{3+}, Mn^{2+}, Fe^{2+}, Ni^{2+}, Cu^{2+} and NO_2^-.

SUMMARY OF METHOD

The High Range Dissolved Oxygen AccuVac Ampul contains reagent vacuum sealed in a 12-mL ampul. When the AccuVac Ampul is broken open in a sample containing dissolved oxygen, it forms a yellow color which turns purple. The purple color development is proportional to the concentration of dissolved oxygen.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
High Range Dissolved Oxygen AccuVac Ampuls, with 2 reusable ampul caps	1 ampul	25/pkg	25150-25

REQUIRED APPARATUS

Adapter, AccuVac Vial	1	each	46025-00
Beaker, 50 mL	1	each	500-41
Caps, Ampul, blue	1	6/pkg	1731-06
DR/700 Filter Module Number 69.01	1	each	46269-00

OPTIONAL APPARATUS

Dissolved oxygen may also be determined by titrimetric methods. Request Publication 8042 for additional information.

AccuVac Snapper Kit	each	24052-00
AccuVac Dissolved Oxygen Sampler	each	24051-00
BOD Bottle and Stopper, 300 mL	each	621-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06

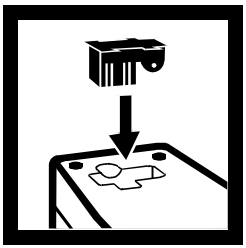
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or the distributor serving you.

TANNIN AND LIGNIN (0 to 9.0 mg/L)

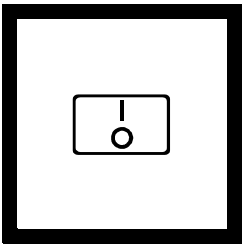
For water, wastewater, boiler water

Tyrosine Method*



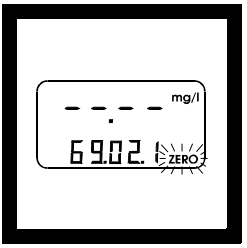
1. Install module **69.01** in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*



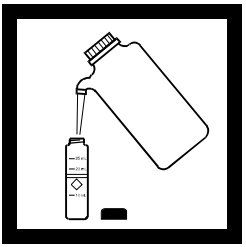
2. Press: **I/O**

The display will show **690 nm** and module number **69.01**

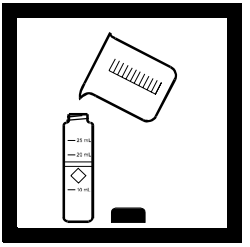


3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number **69.02.1**

*Adapted from Kloster, M.B., *Journal American Water Works Association*, 1974, Vol *66, No. 1*, p 44.



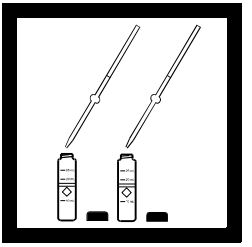
4. Fill a 25-mL cell to the 25-mL line with demineralized water (the blank).



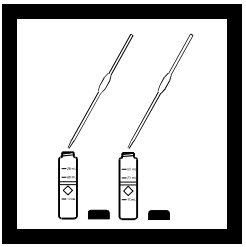
5. Fill another 25-mL cell to the 25-mL line with sample (the prepared sample).

Note: *Filter samples that are highly turbid. Report results as mg/L soluble tannic acid.*

Note: *For proof of accuracy, use a 2.0 mg/L tannic acid standard solution (preparation given in Accuracy Check) in place of the sample.*

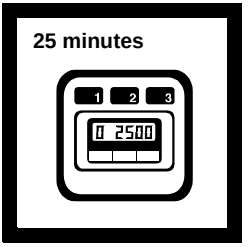


6. Pipet 0.5 mL of TanniVer 3 Tannin-Lignin Reagent into each cell. Cap and invert several times to mix.

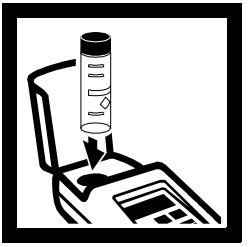


7. Pipet 5.0 mL of Sodium Carbonate Solution into each cell. Cap and invert several times to mix.

Note: *A blue color will develop if tannins and/or lignins are present.*

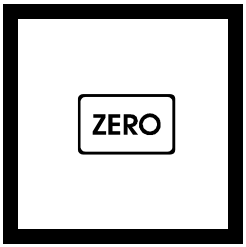


8. Wait 25 minutes.



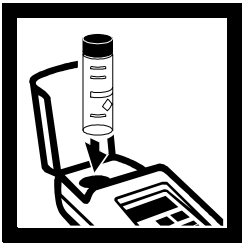
9. Place the blank in the cell holder.

Note: *Use a 10-mL cell for the blank and the sample in bright sunlight.*



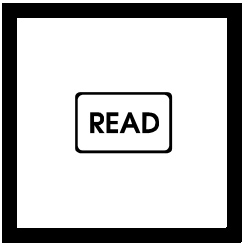
10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.



11. Place the prepared sample in the cell holder.

Note: *Use a 10-mL cell for the blank and the sample in bright sunlight.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L tannic acid.

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles.

ACCURACY CHECK

Standard Solution Method

Prepare a 200-mg/L tannic acid stock solution by dissolving 0.200 grams of tannic acid in demineralized water and diluting to 1000 mL. Prepare this solution monthly. Make a 2.0-mg/L tannic acid standard by diluting 10.00 mL of the stock solution to 1000 mL with demineralized water. Prepare this standard daily.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 6.0 mg/L tannic acid concentration samples, the standard deviation was ±0.042 mg/L tannic acid.

Testing zero concentration samples, the limit of detection was 0.031 mg/L tannic acid. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (adapted from *Analytical Chemistry*, **1980**, *52*, 2242-2249).

INTERFERENCES

Sulfite interference can be removed by adding 1 mL of formaldehyde to the sample before running the test.

Ferrous iron causes a positive interference. Two mg/L of ferrous iron produce a color equivalent to about 1 mg/L of tannic acid. Add one 0.2-g scoop of sodium pyrophosphate to the sample before running the test to eliminate interference due to levels up to 20 mg/L of ferrous iron.

SUMMARY OF METHOD

This test method registers all hydroxylated aromatic compounds, including tannin, lignin, phenol and cresol. The tyrosine method produces a blue color proportional to the amount of these compounds present. The results are reported as total tannin and lignin present expressed as mg/L tannic acid.

REQUIRED REAGENTS

	Cat. No.
Tannin and Lignin Reagent Set (up to 100 tests)	22446-00
Includes: (1) 675-49, (1) 2560-42	

Description	Quantity Per Test	Unit	Cat. No.
Sodium Chromate Solution for Tannin-Lignin	10 mL	500 mL	675-49
TanniVer 3 Tannin-Lignin Reagent	1 mL	100 mL	2560-42
Water, demineralized	25 mL	4 L	272-56

REQUIRED APPARATUS

Description	Quantity Per Test	Unit	Cat. No.
DR/700 Filter Module Number 69.01	1	each	46269-00
Pipet, volumetric, 5.00 mL	1	each	515-37
Pipet, volumetric, Class A, 0.5 mL	1	each	14515-34

OPTIONAL REAGENTS

Formaldehyde	100 mL	2059-32
Sodium Pyrophosphate, ACS	50 g	784-25
Tannic Acid	113 g	791-14

OPTIONAL APPARATUS

Balance, analytical	each	22310-00
Cap for 10- and 25-mL sample cells	6/pkg	24018-12
Cylinder, graduated, 25 mL	each	508-40
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask,. volumetric, 1000 mL	each	547-53
Funnel, poly, 65 mm	each	1083-67
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 1.0 mL	each	515-35
Pipet, volumetric, 10.00 mL	each	14515-38
Pipet Filler, Safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Spoon, measuring, 0.2 g	each	638-00

For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

Module 81.01
810 nm

Hach warrants equipment it manufactures against defective materials or workmanship for at least one year from the shipping date. Warranties do not apply to limited-life electrical components such as batteries. Full warranty information is located on the reverse side of Hach invoices.

IMPORTANT NOTICE

A lamp intensity adjustment must be performed:
- Before first or initial use
- When new filter modules are installed
- On each filter module when the lamp is replaced

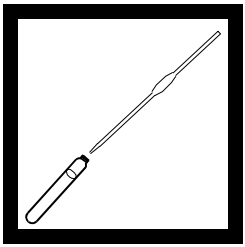
Refer to Lamp Intensity Adjustment in the instrument manual.

Table of Contents for 810-nm parameters

COPPER, AUTOCATALYTIC (0 to 3.00 g/L)

For finishing baths

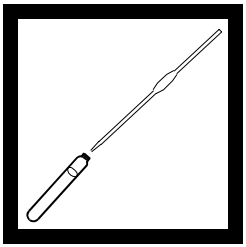
Colorimetric Method*



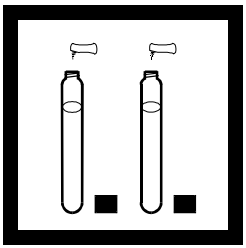
1. Pipet 5.0 mL of demineralized water into a 16-mm screw-top culture tube (the blank).

Note: *Because of variation between plating bath formulations, perform a new calibration for each bath. Prepare and store the calibration as directed under Calibration following these steps.*

Note: *Analyze samples immediately for best results.*

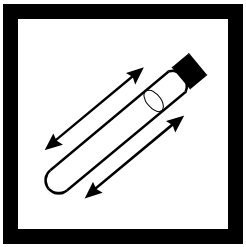


2. Pipet 3.0 mL of bath sample and 2.0 mL of demineralized water into a second 16-mm screw-top culture tube (the prepared sample).

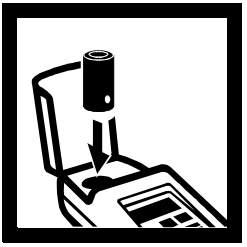


3. Add the contents of one Acid Reagent Powder Pillow to each tube. Mix gently.

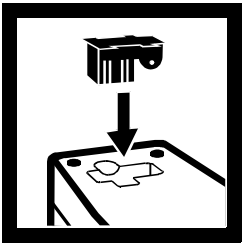
*User calibration required; range is approximate.



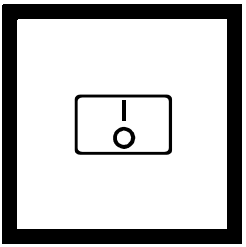
4. Wait until any foaming (effervescence) stops. Cap and shake to dissolve all the powder.



5. Full insert a COD Vial Adapter into the cell holder with the tabs in the square slot.

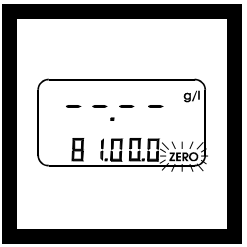


6. Install module
81.01
in a DR/700.



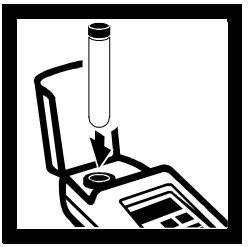
7. Press: **I/O**

The display will show
810 nm
and module number
81.01

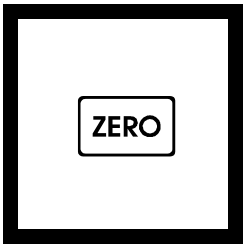


8. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. Press the **PROGRAM** key once or twice until the lower display shows program number
81.000

The upper display will show the S1 concentration.

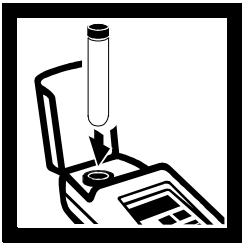


9. Place the blank into the adapter with the Hach logo facing the front of the instrument.



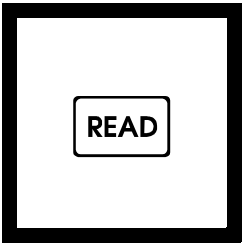
10. Press: **ZERO**

The display will count down from 0. Then the display will show 0.00 g/L and the zero and S1 prompts will turn off.



11. Place the prepared sample into the adapter with the Hach logo facing the front of the instrument.

Note: *Save the blank for future determinations.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in g/L copper (Cu).

CALIBRATION

Perform a calibration for each bath formulation.

1) Prepare a "known sample" using the plating bath of a known, full-strength copper concentration or titrate a sample of the bath to determine its concentration.

2) Perform Steps 1 through 6 of the Autocatalytic Copper test procedure. In Step 2 use the known standard as the bath sample.

3) Perform the Operator Program: Calibrating Procedure Using Two Standards (paragraph 3.2.4.1 in the Instrument Manual). Use the processed demineralized water (the blank) as Standard 1. Make the display read 00.00 of whatever concentration units you would like to use (i.e., g/L, %, mg/L).

4) Use the prepared known sample as Standard 2. Make the display show the known copper concentration.

This procedure provides good readability on baths up to 3 g/L. If the bath in use is more concentrated, dilute the sample and apply an appropriate correction factor; see Sample Dilution Techniques (Section I) for more information.

ACCURACY CHECK
Check for accuracy by titrating a sample of the bath in use to assure that the calibration is correct.

INTERFERENCES
Other blue-colored bath components will interfere to cause high results.

SUMMARY OF METHOD
Bath samples are acidified to destroy the color of the alkaline copper complexes. The remaining color of the acidic copper solution is then measured directly. Since different bath formulations cause slight response variations, a calibration must be performed for the bath in use.

REQUIRED REAGENTS

Descriptions	Quantity Per Test	Unit	Cat. No.
Acid Reagent Powder Pillows	2 pillows	50/pkg	1042-66
Water, demineralized	varies	4 L	272-56

REQUIRED APPARATUS

Cap, tube, for 22758-00	3	25/pkg	14238-25
Clippers, for opening powder pillows	1	each	968-00
COD Vial Adapter	1	each	46008-00
DR/700 Filter Module Number 81.01	1	each	46281-00
Pipet, serological, 5 mL	1	each	532-37
Pipet Filler, safety bulb	1	each	14651-00
Tube, culture, 16 x 100 mm	3	each	22758-00

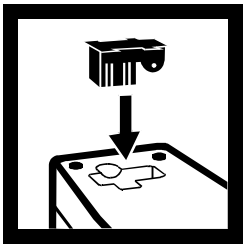
OPTIONAL APPARATUS

Pipet, TenSette, 1 to 10 mL	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet	50/pkg	21997-96

PHOSPHONATES (0-2.5 to 0-125 mg/L)

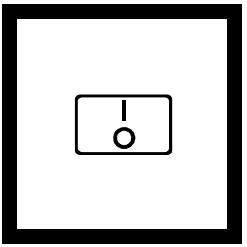
For water, wastewater and seawater

Persulfate UV Oxidation Method*



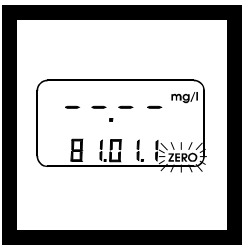
1. Install module number
81.01
in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*



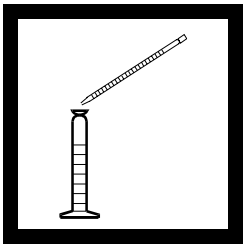
2. Press: **I/O**

The display will show
810 nm
and module number
81.01

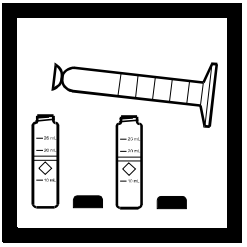


3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
81.01.1

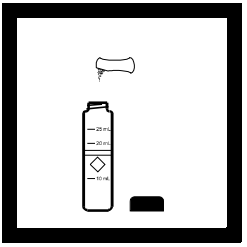
*Adapted from Blystone, P.; Larson, P. A., *Rapid Method for Analysis of Phosphonate Compounds*, International Water Conference, Pittsburgh, PA, October 1981



4. Choose a sample size from Table 1 below. Pipet the chosen sample volume into a 50-mL graduate mixing cylinder. Dilute the sample to 50 mL with demineralized water. Mix well.



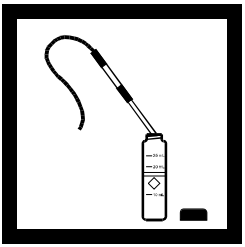
5. Split the diluted sample by pouring 25 mL into each of two sample cells.



6. Add the contents of one Potassium Persulfate For Phosphonate Powder Pillow to one of the sample cells (the prepared sample). Cap and invert several times to mix.

Table 1. Sample Volume

Expected Phosphonate Range (mg/L)	Sample Volume (mL)
0-2.5	50
0-5	25
0-12.5	10
0-25	5
0-125	1

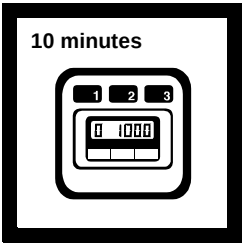


7. Insert the ultraviolet (UV) lamp into the prepared sample. The other sample is the blank.

Note: *UV safety goggles should be worn while the lamp is on.*

Note: *Do no handle the lamp surface. Fingerprints will etch the glass. Wipe lamp clean with a soft, clean tissue between samples. Do not use phosphate detergents to wash glassware.*

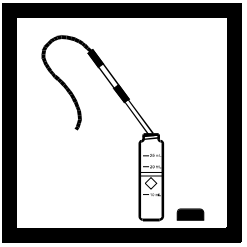
Note: *A specially designed cord adapter (Cat. No. 19584-00) is available for performing two digestions with a single power supply. A second UV lamp (Cat. No. 20823-00) is also required.*



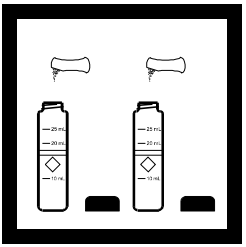
8. Turn the lamp on. Wait for 10 minutes.

Note: *Phosphonates are converted to orthophosphate in this step.*

Note: *The digestion step should normally be completed in less than ten minutes. However, a contaminated sample or a weak lamp could result in incomplete conversion to phosphate. Check conversion efficiency by running a longer digestion and seeing if readings increase.*



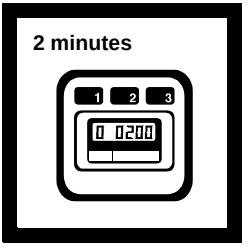
9. Turn the UV lamp off and remove from the sample cell.



10. Add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to each sample cell. Cap and invert several times to mix.

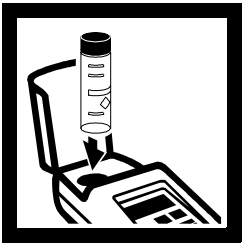
Note: *A blue color will develop if phosphate, produced by the digestion, is present.*

Note: *A 10-mL sample can be tested by using 10-mL sample cells and optional reagents.*



11. Wait 2 minutes.

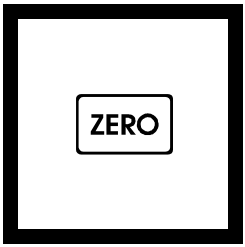
Note: *If the sample is colder than 15 °C, 4 minutes are required for color development.*



12. Place the blank in the cell holder.

Note: *Use a 10-mL cell for the blank and sample in bright sunlight and close the cell compartment cover.*

Note: *Perform Steps 13 and 14 within three minutes after the two-minute period.*

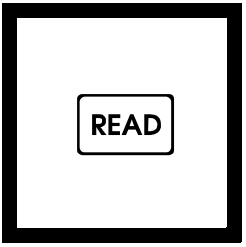


13. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L and the zero prompt will turn off.

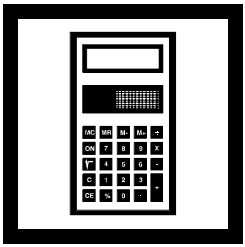
14. Place the prepared sample in the cell holder.

Note: *Use a 10-mL cell for the blank and sample in bright sunlight and close the cell compartment cover.*



15. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L phosphonate.



16. Multiply the result by the appropriate value in Table 2 to obtain the actual concentration of the sample.

Note: *Results may be expressed in terms of active phosphonic acid by picking the appropriate conversion factor and using the equation in Table 3.*

Table 2. Concentration Conversions

Sample volume (mL)	Multiplier
50	0.1
25	0.2
10	0.5
5	1.0
1	5.0

mg/L Phosphonate = Instrument Reading x Multiplier

Table 3. Phosphonate Activity Conversion Factors

Type of Phosphonic Acid	Conversion Factor
Bayhibit AM, PBTC	2.84
Dequest 2000, Wayplex NTP, ATMP	1.050
Dequest 2010, Wayplex HEDPA-60, HEDP	1.085
Dequest 2041, EDTMPA	1.148
Dequest 2051, HMDTMPA	1.295
Dequest 2060, DETPMPA	1.207

Active Phosphonic Acid, mg/L = Phosphonic Acid Value from Step 15 x Conversion Factor

SAMPLING AND STORAGE

Collect sample in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with demineralized water. Do not use a commercial detergent. If prompt analysis is impossible, adjust the pH to 2 or less with about 2 mL of sulfuric acid, ACS, per liter of sample. Store the sample at 4 °C (39 °F) or below. Preserved samples can be stored at least 24 hours. See Section 1 for more information on dilution factors, cleaning instructions, etc.

INTERFERENCES

When testing a 5-mL sample volume, the following may interfere when present in concentrations exceeding those listed below:

Aluminum	100 mg/L
Benzotriazole	10 mg/L
Bicarbonate	1000 mg/L
Bromide	100 mg/L
Calcium	5000 mg/L
CDTA	100 mg/L
Chloride	5000 mg/L
Chromate	100 mg/L
Copper	100 mg/L
Cyanide* 1	100 mg/L
Diethanoldithiocarbamate	50 mg/L
EDTA	100 mg/L
Iron	200 mg/L
Nitrate	200 mg/L
NTA	250 mg/L
Orthophosphate	15 mg/L
Silica	500 mg/L
Silicate	100 mg/L
Sulfate	2000 mg/L
Sulfite	100 mg/L
Thiourea	10 mg/L

*The UV digestion should be increased to 30 minutes.

The interference levels will decrease as the sample size increases. For example, copper does not interfere at or below 100 mg/L for a 5.00 mL sample. If the sample volume is increased to 10.00 mL, copper will begin to interfere above 50 mg/L.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

Arsenate and sulfide interfere directly. Phosphates and organo-phosphorous compounds other than phosphonates react quantitatively. Meta and polyphosphates do not interfere.

SUMMARY OF METHOD

This method is directly applicable to boiler and cooling tower samples. The procedure is based on a UV catalyzed oxidation of phosphonate to orthophosphate. Range may be as low as 0 to 2.5 mg/L or as high as 0 to 125 mg/L.

REQUIRED REAGENTS

	Cat. No.
Phosphonates Reagent Set (100 Tests)	22440-00
Includes: (2) 2125-99, (1) 20847-69	

Description	Quantity Per Test	Unit	Cat. No
PhosVer 3 Phosphate Reagent Powder Pillows	2 pillows	100/pkg	2125-99
Potassium Persulfate Powder Pillow for Phosphonate	1 pillow	100/pkg	20847-69
Water, demineralized	varies	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	20658-00
Cylinder, mixing, graduated, 50 mL	1	each	1896-41
Goggles, UV safety	1	each	21134-00
DR/700 Filter Module Number 61.01	1	each	46261-00
Pipet, volumetric, Class A, 5.00 mL	1	each	14515-37

REQUIRED APPARATUS

Description	Quantity Per Test	Unit	Cat. No
UV Lamp with power supply, 115 Vac	1	each	20828-00
OR			
UV Lamp with power supply, 230 Vac	1	each	20828-02

OPTIONAL REAGENTS

Hydrochloric Acid, 6.0N (1:1)	500 mL	884-49
Sulfuric Acid, ACS	500 mL	979-49

OPTIONAL APPARATUS

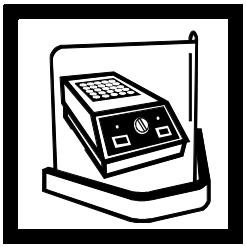
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
Cord Adapter, single to dual UV lamp	each	19485-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, serological, 2 mL	each	532-36
Pipet, Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
UV Lamp	each	20823-00

For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

PHOSPHORUS, ACID HYDROLYZABLE
(0 to 5.00 mg/L)
For water, wastewater and seawater

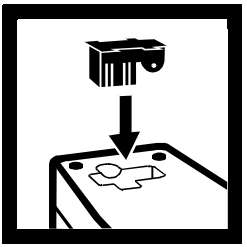
PhosVer 3 with Acid Hydrolysis; Test 'N Tube™ Procedure



1. Turn on the COD Reactor. Heat to 103 to 106°C. Place the plastic shield in front of the reactor.

Note: *Ensure safety devices are in place to protect the analyst from splattering should leakage occur.*

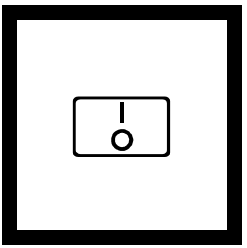
Note: *See COD Reactor manual for temperature adjustment instructions.*



2. Install module
81.01
in a DR/700

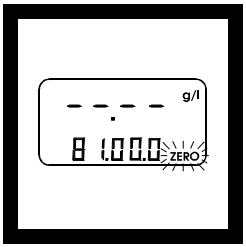
Note: *Refer to Calibration following these steps.*

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse again with demineralized water. Do not use detergents containing phosphate to clean glassware.*



3. Press: **I/O**

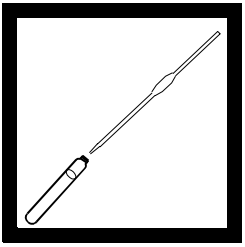
The display will show
810 nm
and module
81.01



4. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. Press the **PROGRAM** key until the lower display shows program number

81.000

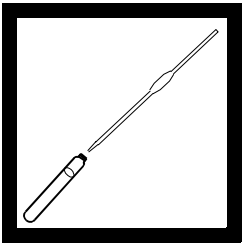
The upper display will show the S1 concentration.



5. Fill a Test 'N Tube Cuvette vial with 5 mL of sample.

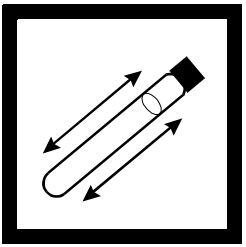
Note: *For proof of accuracy, use a 1.0 mg/L Phosphorus as phosphate (0.33 mg/L P) Standard Solution (see Optional Reagents) in place of the sample.*

Note: *Run a reagent blank for this test. Use demineralized water in place of the sample. Subtract this result from all test results run with this lot of PhosVer 3.*

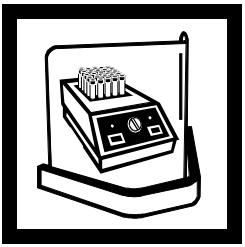


6. Add 2 mL of 1.00 N H_2SO_4 to the sample vial.

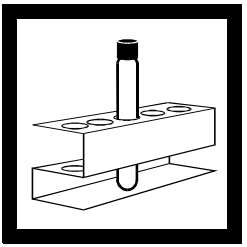
Note: *A 2-mL Repipet and 1 liter bottle of H_2SO_4 (1270-53) can be used for convenience when analyzing multiple samples.*



7. Cap tightly and shake to mix.

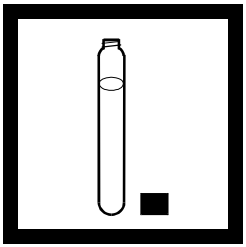


8. Heat the vial for 30 minutes.

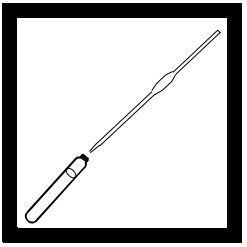


9. Carefully remove the vial from the reactor. Place it in a test tube rack and allow to cool to room temperature.

Note: *Tubes will be hot.*

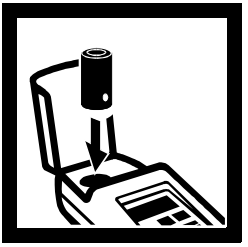


10. Remove the cap from the vial.

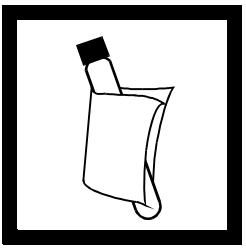


11. Add 2 mL of 1.00 N sodium hydroxide to the vial. Cap tightly and shake to mix.

Note: *A 2-mL Repipet and 900-mL bottle of NaOH can be used for convenience when analyzing multiple samples.*

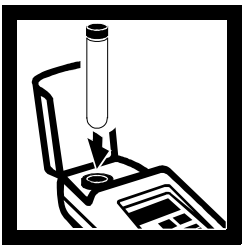


12. Fully insert a COD Vial Adapter into the cell holder with the tabs in the square slot.

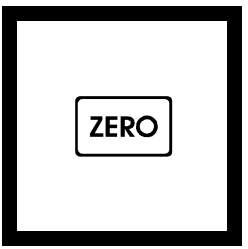


13. Clean the outside of the vial with a towel.

Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



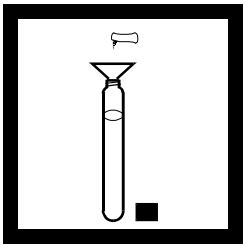
14. Place the sample vial in the adapter with the Hach logo facing the front of the instrument.



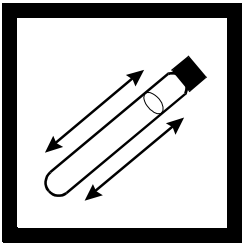
15. Press: **ZERO**

The display will count down to 0. Then the display will show 0.000 mg/L, and the zero prompt will turn off.

Note: *For multiple samples, zero on the first sample and record the blank value. Subtract the blank value from each sample result.*



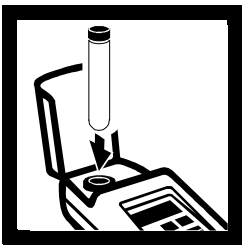
16. Remove the vial from the instrument. Remove the cap from the vial. Using a funnel, add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow for 10 mL samples to the vial.



17. Shake the vial for 10-15 seconds.

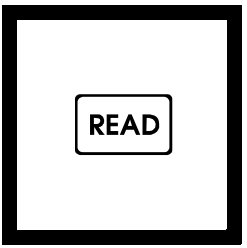
Note: *The powder will not completely dissolve.*

18. Wait 2 minutes. Read the sample between 2 and 8 minutes.



19. Clean the outside of the sample vial with a towel. Place the prepared sample into the adapter with the Hach logo facing the front of the instrument.

Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



20. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L PO_4.

CALIBRATION PROCEDURE

1) If the DR/700 is on and in a pre-programmed method, proceed to Step 3. If it is off, press **I/O**. The upper display will show the wavelength and the lower display will show the module installed and the software version number. Verify that filter module **81.01** is installed.

2) After 2 seconds, the display will show a concentration format, a program number and the ZERO prompt.

3) Press **PROGRAM** to obtain program number 81.000. The ZERO annunciator will flash.

4) Press **CAL**. The S1 annunciator and the first digit on the left will flash.

5) Press the **UP ARROW** key to set the first digit to 0. When correct, press the **RIGHT ARROW** key to select the next digit. Repeat this procedure until all four digits are set to 0.

6) Press the **RIGHT ARROW** key until the decimal point flashes. Press the **UP ARROW** key to move the decimal point so the display reads 0.000 for PO_4^{3-}.

7) Press the **RIGHT ARROW** key. A units indicator will flash. Press the **UP ARROW** until mg/L is displayed.

8) Prepare a demineralized water blank in a sample vial. Cap the vial and put it into the DR/700.

9) Press **ZERO**. The display will count down and then show another four-digit concentration value. The S2 annunciator will begin flashing.

10) Press the **RIGHT ARROW** key. The left digit will begin flashing. Press the arrow keys to edit the display to read 5.000 for PO_4^{3-}.

11) Press **READ**. The display will count down and then show a relative absorbance near zero. This step enters the upper limit (S2) value. The S2 annunciator will flash.

Note: *The colorimeter will display measurements above the upper limit, but the accuracy is questionable.*

12) Press the **RIGHT ARROW** key. The left digit and the S2 annunciator will flash. Use the arrow keys to enter the correct absorbance difference. If a negative number appears, continue to press the **UP ARROW** key until a positive 0.570 appears.

13) Press **READ**. This enters the calibration curve. The S2 annunciator will flash.

14) Press **CAL**. The calibration is automatically completed and stored in the filter module memory under program number 81.000. The display will show the concentration value for demineralized water and the ZERO indicator will be flashing.

SAMPLING AND STORAGE

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with demineralized 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 up to 24 hours by storing at 4 °C. For longer storage periods, add 4.0 mL of mercuric chloride to each liter of sample and mix (use of mercuric chloride is discouraged due to health and environmental concerns). Sample refrigeration is still necessary. Samples preserved with mercuric chloride must have a sodium chloride level of 50 mg/L or higher to prevent mercury interference in the test. Spike samples low in chloride with 0.1 g sodium chloride per liter of sample.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck of a Phosphate Voluette Ampule Standard, 25 mg/L as P.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to three 5-mL aliquots of a water samples. Mix well.

c) Analyze each sample as described in the procedure. The concentration should increase 1.5 mg/L, 3.0 mg/L, 4.3 mg/L, respectively.

d) If these increases do not occur, see Standard Additions for more information.

INTERFERENCES

Large amounts of turbidity may cause inconsistent results in the test because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.

The PhosVer 3 Phosphate Reagent Powder Pillows should be stored in a cool, dry environment.

The following may interfere when present in concentrations exceeding these listed below:

Aluminum	200 mg/L
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Zinc	80 mg/L

Arsenate and hydrogen sulfide do interfere.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.

PRECISION

DR/700: In a single laboratory, using a standard solution of 4.20 mg/L PO_4^{3-} and two lots of reagent with a DR/700, a single operator obtained a standard deviation of ±0.11 mg/L PO_4^{3-}.

SAMPLE DISPOSAL INFORMATION

The phosphate vials contain only materials that are non-regulated for disposal. Neutralize the vial solutions to a pH of 6-9 and dispose of as a non-regulated material.

SUMMARY OF METHOD

Orthophosphate reacts with molybdate in an acid medium to produce a Phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows, 10 mL	1 Pillow	100/pkg	21060-69
Sulfuric Acid Standard Solution, 1.000 N	2 mL	100 mL	1270-32
Sodium Hydroxide Standard Solution, 1.000 N	2 mL	100 mL	1045-32

REQUIRED APPARATUS

Test 'N Tube Cuvette Vials	1	25/pkg	25831-25
COD Reactor, 120/240 Vac	1	each	45600-00
COD Vial Adapter, DR/700	1	each	46008-00
Funnel, micro	1	each	25843-35
Pipet, volumetric, Class A, 5 mL	1	each	14515-37
Pipet Filler, safety bulb	1	each	14651-00
Safety Shield, laboratory bench	1	each	23810-00
Test Tube Rack	1-3	each	18641-00

OPTIONAL REAGENTS

Hydrochloric Acid Standard Solution, 6.0 N (1:1)		500 mL	884-49
Mercuric Chloride Solution, 10 g/L		100 mL	14994-42
Phosphate Standard Solution, 1 mg/L as PO_4^{3-}		500 mL	2569-42
Phosphate Standard Solution, Voluette ampule, 50 mg/L as PO_4, 10 mL		16/pkg	171-10
Sodium Chloride, ACS		454 g	182-01
Sodium Hydroxide Standard Solution, 5.0 N		900 mL	2450-53
Sodium Hydroxide Standard Solution, 1.000 N		900 mL	1045-53
Sulfuric Acid Standard Solution, 1.000 N		1 L	1270-53
Water, demineralized		4 L	272-56

OPTIONAL APPARATUS

Dispenser, Repipet Jr., 2 mL		each	22307-01
pH Indicator Paper, 1 to 11 pH units		5 rolls/pkg	391-33

OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
pH Meter, EC10, portable	each	50050-00
Pipet, TenSette, 1 to 10 mL	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet	50/pkg	21997-06

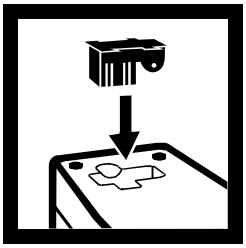
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

PHOSPHORUS, REACTIVE (0 to 5.00 mg/L)

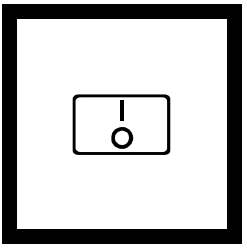
For water, wastewater and seawater

PhosVer 3 Method; Test 'N Tube™ Procedure



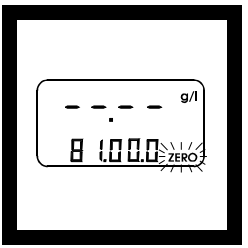
1. Install module
81.01
in a DR/700

Note: *Refer to Calibration following these steps.*



2. Press: **I/O**

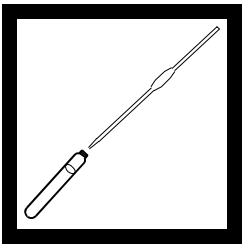
The display will show
810 nm
and module
81.01



3. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. Press the **PROGRAM** key until the lower display shows program number
81.000

The upper display will show the S1 concentration.

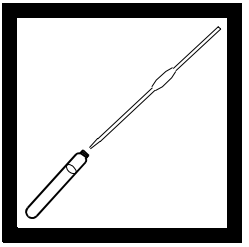
Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse again with demineralized water. Do not use phosphate detergents to clean glassware.*



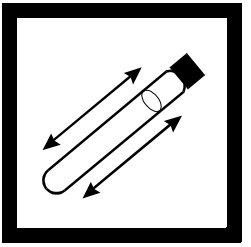
4. Fill a Test 'N Tube Cuvette vial with 5 mL of sample.

Note: *For proof of accuracy, use a 1.0 mg/L Phosphorus as phosphate (0.33 mg/L P) Standard Solution (see Optional Reagents) in place of the sample.*

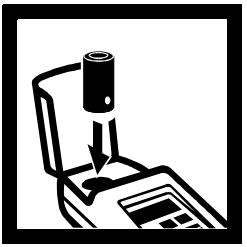
Note: *Run a reagent blank for this test. Use demineralized water in place of the sample. Subtract this result from all test results run with this lot of PhosVer.*



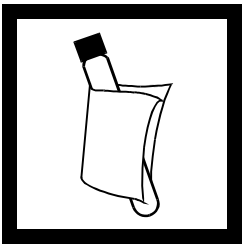
5. Add 4 mL of demineralized water to the sample vial.



6. Cap tightly and shake to mix.

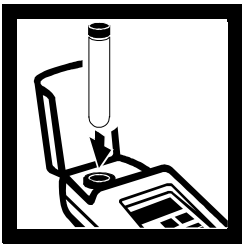


7. Fully insert a COD Vial Adapter into the cell holder with the tabs in the square slot.

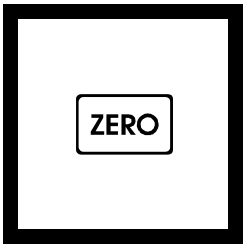


8. Clean the outside of the sample vial with a towel.

Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



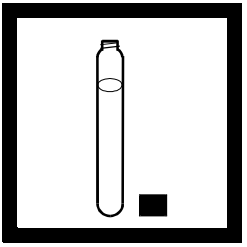
9. Place the sample vial in the adapter with the Hach logo facing the front of the instrument.



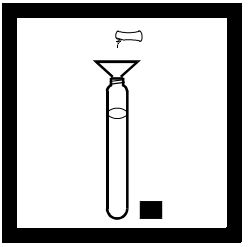
10. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L, and the zero prompt will turn off.

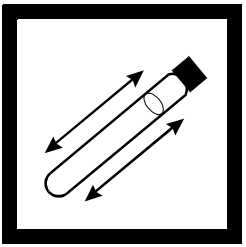
Note: *For multiple samples, zero on the first sample and record the blank value. Subtract this value from each sample result.*



11. Remove the cap from the vial.

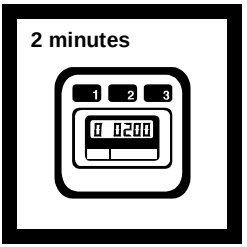


12. Using a funnel, add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow for 10-mL samples to the vial.

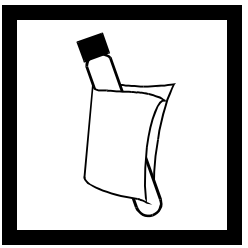


13. Cap tightly and shake for 10-15 seconds.

Note: *The powder will not dissolve completely.*

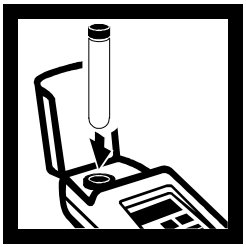


14. Wait 2 minutes

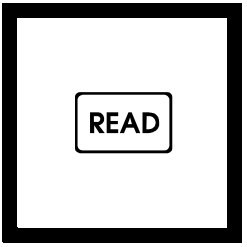


15. Clean the outside of the sample vial with a towel.

Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



16. Place the prepared sample into the adapter with the Hach logo facing the front of the instrument.



17. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L PO_4^{3-}.

CALIBRATION PROCEDURE

1) If the DR/700 is on and in a pre-programmed method, proceed to Step 3. If it is off, press the **I/O** key. The upper display will show the wavelength and the lower display will show the module installed and the software version number. Verify that filter module **81.01** is installed.

2) After 2 seconds, the display will show a concentration format, a program number and the ZERO prompt.

3) Press the **PROGRAM** key to obtain program number 81.000. The ZERO annunciator will flash.

4) Press the **CAL** key. The S1 annunciator and the first digit on the left will flash

5) Press the **UP ARROW** key to set the first digit to 0. When correct, press the **RIGHT ARROW** key to select the next digit. Repeat this procedure until all four digits are set to 0.

6) Press the **RIGHT ARROW** key until the decimal point flashes. Press the **UP ARROW** key to move the decimal point so the display reads 0.000 for PO_4^{3-}.

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9) Press **ZERO**. The display will count down and then show another four-digit concentration value. The S2 annunciator will begin flashing.

10) Press the **RIGHT ARROW** key. The left digit will begin flashing. Press the arrow keys to edit the display to read 5.000 for PO_4^{3-}.

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13) Press **READ**. This enters the calibration curve. The S2 annunciator will flash.

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a) Snap the neck of a Phosphate Voluette Ampule Standard, 25 mg/L as P.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to three 5-mL aliquots of a water samples. Mix well.

c) Analyze each sample as described in the procedure. The concentration should increase 1.5 mg/L, 3.0 mg/L, 4.3 mg/L, respectively.

d) If these increases do not occur, see Standard Additions for more information.

INTERFERENCES

Large amounts of turbidity may cause inconsistent results in the test because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.

The PhosVer 3 Phosphate Reagent Powder Pillows should be stored in a cool, dry environment.

The following may interfere when present in concentrations exceeding these listed below:

Aluminum	200 mg/L
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Zinc	80 mg/L

Arsenate and hydrogen sulfide do interfere.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.

PRECISION

DR/700: In a single laboratory, using a standard solution of 4.20 mg/L PO_4^{3-} and two lots of reagent with a DR/700, a single operator obtained a standard deviation of ±0.11 mg/L PO_4^{3-}.

SAMPLE DISPOSAL INFORMATION

The phosphate vials contain only materials that are non-regulated for disposal. Neutralize the vial solutions to a pH of 6-9 and dispose as a normal non-regulated material.

SUMMARY OF METHOD

Orthophosphate reacts with molybdate in an acid medium to produce a Phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows, 10 mL	1 Pillow	100/pkg	21060-69

REQUIRED APPARATUS

Test 'N Tube Cuvette Vials	1	25/pkg	25831-25
COD Reactor, 120/240 Vac	1	each	45600-00
COD Vial Adapter, DR/700	1	each	46008-00
DR/700 Filter Module 81.00	1	each	46281-00
Funnel, micro	1	each	25843-35
Pipet, volumetric, Class A, 2 mL	1	each	14515-36
Pipet, volumetric, Class A, 4 mL	1	each	14515-04
Pipet, volumetric, Class A, 5 mL	1	each	14515-37
Pipet Filler, safety bulb	1	each	14651-00
Safety Shield, laboratory bench	1	each	23810-00
Test Tube Rack	1-3	each	18641-00

OPTIONAL REAGENTS

Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Mercuric Chloride Solution, 10 g/L	100 mL	14994-42

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Phosphate Standard Solution, 1 mg/L as PO_4^{3-}	500 mL	2569-42
Phosphate Standard Solution, Voluette ampule, 50 mg/L as PO_4, 10 mL	16/pkg	171-10
Sodium Chloride, ACS	454 g	182-01
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53
Sodium Hydroxide Standard Solution, 1.000 N	900 mL	1045-53
Sulfuric Acid Standard Solution, 1.000 N	1 L	1270-53
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Dispenser, Repipet Jr., 2 mL	each	22307-01
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, TenSette, 1 to 10 mL	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet	50/pkg	21997-06

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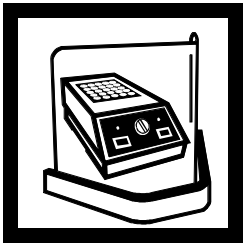
In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

PHOSPHORUS, TOTAL (0 to 3.50 mg/L)

For water, wastewater and seawater

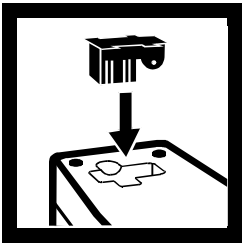
PhosVer 3 and Acid Persulfate Digestion; Test 'N Tube™ Procedure



1. Turn on the COD Reactor. Heat to 103 to 106ºC. Place the plastic shield in front of the reactor.

Note: *Ensure safety devices are in place to protect the analyst from splattering should leakage occur.*

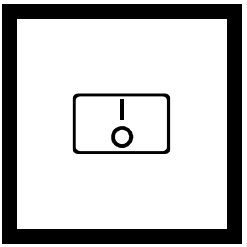
Note: *See COD Reactor manual for temperature adjustment instructions.*



2. Install module **81.01** in a DR/700.

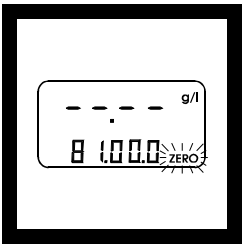
Note: *Refer to Calibration following these steps.*

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse again with demineralized water. Do not use detergents containing phosphates to clean glassware.*



3. Press: **I/O**

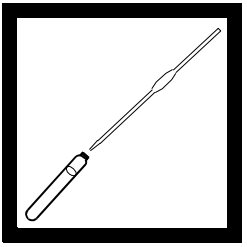
The display will show **810 nm** and module **81.01**



4. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. Press the **PROGRAM** key until the lower display shows program number

81.000

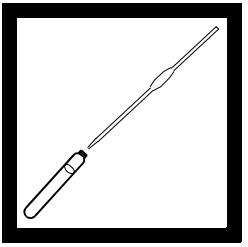
The upper display will show the S1 concentration.



5. Fill a Test 'N Tube Cuvette vial with 5 mL of sample.

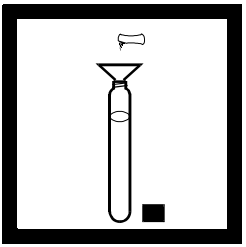
Note: *For proof of accuracy, use a 1.0 mg/L Phosphorus as phosphate (0.33 mg/L P) Standard Solution (see Optional Reagents) in place of the sample.*

Note: *Run a reagent blank for this test. Use demineralized water in place of the sample. Subtract this result from all test results run with this lot of PhosVer 3.*

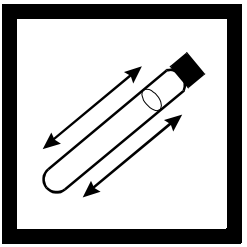


6. Add 2 mL of 1.00 N H_2SO_4 to the sample vial.

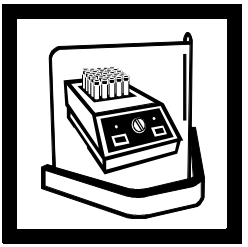
Note: *A 2-mL Repipet and 1 liter bottle of sulfuric acid(1270-53) can be used for convenience when analyzing multiple samples.*



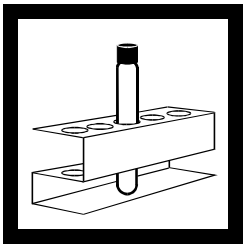
7. Using a funnel, add the contents of one Potassium Persulfate Powder Pillow for Phosphonate to the vial.



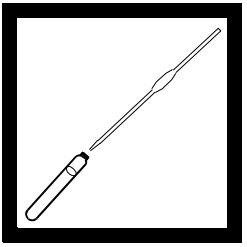
8. Cap tightly and shake to mix.



9. Heat the vial for 30 minutes.

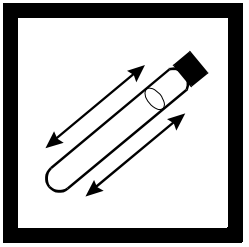


10. Carefully remove the vial from the reactor. Place it in a test tube rack and allow to cool to room temperature.

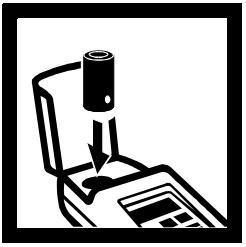


11. Remove the cap from the vial. Add 2 mL of 1.00 N sodium hydroxide to the vial.

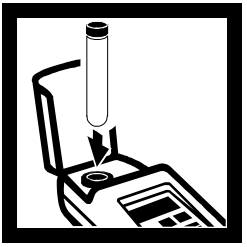
Note: *A 2-mL Repipet and 900-mL bottle of NaOH can be used for convenience when analyzing multiple samples.*



12. Cap tightly and shake to mix.

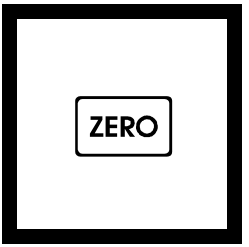


13. Fully insert a COD Vial Adapter into the cell holder with the tabs in the square slot.



14. Clean the outside of the vial with a towel. Place the sample vial in the adapter with the Hach logo facing the front of the instrument.

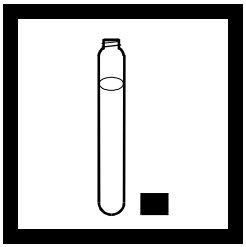
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



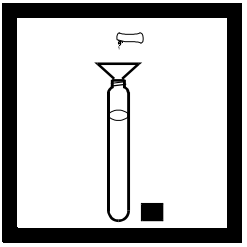
15. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/L, and the zero prompt will turn off.

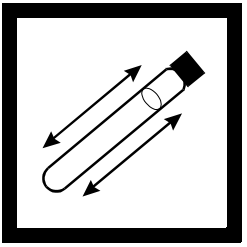
Note: *For multiple samples, zero on the first sample and record the blank value. Subtract the blank value from each sample result.*



16. Remove the vial from the instrument. Remove the cap from the vial.

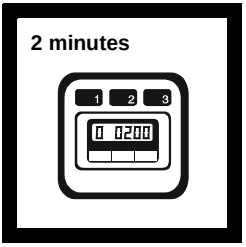


17. Using a funnel, add the contents of one PhosVer 3 Phosphate Reagent Powder Pilow for 10-mL samples Powder Pillow to the vial.

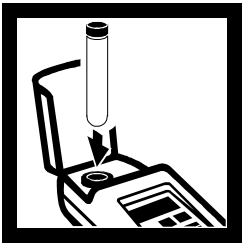


18. Cap tightly and shake for 10-15 seconds.

Note: *The powder will not dissolve completely.*

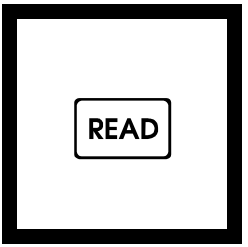


19. Wait 2 minutes. Read sample between 2 and 8 minutes.



20. Clean the outside of the sample vial with a towel. Place the prepared sample into the adapter with the Hach logo facing the front of the instrument.

Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



21. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L PO_4.

CALIBRATION PROCEDURE

1) If the DR/700 is on and in a pre-programmed method, proceed to Step 3. If it is off, press the **I/O** key. The upper display will show the wavelength and the lower display will show the module installed and the software version number. Verify that filter module **81.01** is installed.

2) After 2 seconds, the display will show a concentration format, a program number and the ZERO prompt.

3) Press the **PROGRAM** key to obtain program number 81.000. The ZERO annunciator will flash.

4) Press the **CAL** key. The S1 annunciator and the first digit on the left will flash.

5) Press the **UP ARROW** key to set the first digit to 0. When correct, press the **RIGHT ARROW** key to select the next digit. Repeat this procedure until all four digits are set to 0.

6) Press the **RIGHT ARROW** key until the decimal point flashes. Press the **UP ARROW** key to move the decimal point so the display reads 0.000 for PO_4^{3-}.

7) Press the **RIGHT ARROW** key. A units indicator will flash. Press **UP ARROW** until mg/L is displayed.

8) Prepare a demineralized water blank in a sample vial. Cap the vial and put it into the DR/700.

9) Press **ZERO**. The display will count down and then show another four-digit concentration value. The S2 annunciator will begin flashing.

10) Press the **RIGHT ARROW** key. The left digit will begin flashing. Press the arrow keys to edit the display to read 5.00 for PO_4^{3-}.

11) Press **READ**. The display will count down and then show a relative absorbance near zero. This step enters the upper limit (S2) value. The S2 annunciator will flash.

Note: *The colorimeter will display measurements above the upper limit, but the accuracy is questionable.*

12) Press the **RIGHT ARROW** key. The left digit and the S2 annunciator will flash. Use the arrow keys to enter the correct absorbance difference. Press the **UP ARROW** key until a positive 0.570 appears.

13) Press **READ**. This enters the calibration curve. The S2 annunciator will flash.

14) Press **CAL**. The calibration is automatically completed and stored in the filter module memory under program number 81.000. The display will show the concentration value for demineralized water and the ZERO indicator will be flashing.

SAMPLING AND STORAGE

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with demineralized 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 up to 24 hours by storing at 4 °C. For longer storage periods, add 4.0 mL of mercuric chloride to each liter of sample and mix (use of mercuric chloride is discouraged due to health and environmental concerns). Sample refrigeration is still necessary. Samples preserved with mercuric chloride must have a sodium chloride level of 50 mg/L of higher to prevent mercury interference in the test. Spike samples low in chloride with 0.1 g sodium chloride per liter of sample.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck of a Phosphate Voluette Ampule Standard, 25 mg/L as P.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to three 5-mL aliquots of a water samples. Mix well.

c) Analyze each sample as described in the procedure. The concentration should increase 1.5 mg/L, 3.0 mg/L, 4.3 mg/L, respectively.

d) If these increases do not occur, see Standard Additions for more information.

INTERFERENCES

Large amounts of turbidity may cause inconsistent results in the test because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.

The PhosVer 3 Phosphate Reagent Powder Pillows should be stored in a cool, dry environment.

The following may interfere when present in concentrations exceeding these listed below:

Aluminum	200 mg/L
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Zinc	80 mg/L

Arsenate and hydrogen sulfide do interfere.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.

PRECISION

DR/700: In a single laboratory, using a standard solution of 4.20 mg/L PO_4^{3-} and two lots of reagent with a DR/700, a single operator obtained a standard deviation of ±0.11 mg/L PO_4^{3-}.

SAMPLE DISPOSAL INFORMATION

The phosphate vials contain only materials that are non-regulated for disposal. Neutralize the vial solutions to a pH of 6-9 and dispose of as a normal non-regulated material.

SUMMARY OF METHOD

Orthophosphate reacts with molybdate in an acid medium to produce a Phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows, 10 mL	1 Pillow	100/pkg	21060-69
Reagents for Total Phosphorus			
Potassium Persulfate for Phosphonate Powder Pillows	1	100/pkg	20847-69
Sulfuric Acid Standard Solution, 1.000 N	2 mL	2 x 100 mL	1270-32
Sodium Hydroxide Standard Solution, 1.000 N	2 mL	2 x 100 mL	1045-32

REQUIRED APPARATUS

Test 'N Tube Vials Cuvette Vials	1	25/pkg	25831-25
COD Reactor, 120/240 Vac	1	each	45600-00
COD Vial Adapter, DR/700	1	each	46008-00
DR/700 Filter Module Number 81.01	1	each	46281-00
Funnel, micro	1	each	25843-35
Pipet, volumetric, Class A, 5 mL	1	each	14515-37
Pipet Filler, safety bulb	1	each	14651-00
Safety Shield, laboratory bench	1	each	23810-00
Test Tube Rack	1-3	each	18641-00

OPTIONAL REAGENTS

Hydrochloric Acid Standard Solution, 6.0 N (1:1)		500 mL	884-49
Mercuric Chloride Solution, 10 g/L		100 mL	14994-42
Phosphate Standard Solution, 1 mg/L as PO_4^{3-}		500 mL	2569-42
Phosphate Standard Solution, Voluette ampule, 50 mg/L as PO_4, 10 mL		16/pkg	171-10
Sodium Chloride, ACS		454 g	182-01
Sodium Hydroxide Standard Solution, 1.000 N		900 mL	1045-53
Sodium Hydroxide Standard Solution, 5.0 N		1 L	2450-53

OPTIONAL REAGENTS (continued)

Description	Unit	Cat. No.
Sulfuric Acid		
Standard Solution, 1.000 N	1 L	1270-53
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Dispenser, Repipet Jr., 2 mL	each	22307-01
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, TenSette, 1 to 10 mL	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet	50/pkg	21997-06

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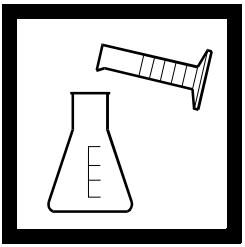
In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.

PHOSPHORUS, ACID HYDROLYZABLE

For water, wastewater, seawater

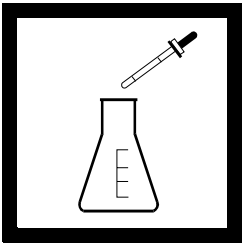
Hydrolysis to Orthophosphate Method*



1. Measure 25 mL of sample into a 50-mL erlenmeyer flask using a graduated cylinder.

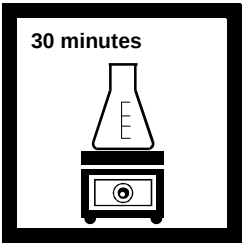
Note: *Wash all glassware with hydrochloric acid, 6 N. Rinse with demineralized water.*

Note: *If prompt analysis is impossible, see Sampling and Storage following these steps.*



2. Add 2.0 mL of Sulfuric Acid Solution, 5.25 N.

Note: *Use the 1-mL calibrated dropper provided.*

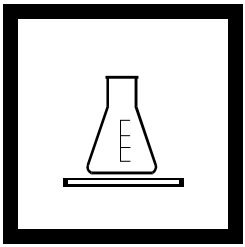


3. Place the flask (the prepared sample) on a hot plate. Boil gently for 30 minutes.

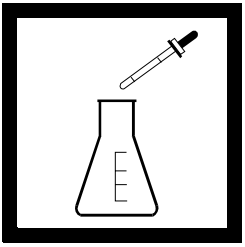
Note: *Maintain the volume near 20 mL by adding small amounts of demineralized water. Do not exceed 20 mL.*

Note: *This is more easily accomplished with a "double-boiler" technique. Place the flask in a pan or beaker of boiling water which is just deeper than the solution level in the flask. Continue boiling for 30 minutes.*

*Adapted from *Standard Methods for the Examination of Water and Wastewater*

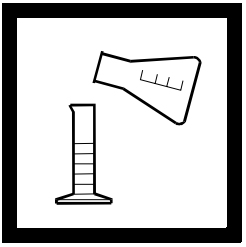


4. Cool the prepared sample to room temperature.



5. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution. Swirl to mix.

Note: *Use the 1-mL calibrated dropper provided.*



6. Pour the prepared sample into a graduated cylinder. Add demineralized water rinsings from the flask to return the volume to 25 mL. Proceed with the appropriate reactive phosphorus test.

Note: *Results of the reactive phosphorus test at this point will include the orthophosphate plus the acid-hydrolyzable (condensed) phosphate. The condensed phosphate concentration is determined by subtracting the results of a reactive phosphorus test on an untreated sample from this result.*

SAMPLING AND STORAGE

Most reliable results are obtained when samples are analyzed immediately. If prompt analysis is not possible, samples may be preserved up to 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

INTERFERENCES

If the sample is turbid, use 50 mL of sample and double the reagent volumes. Use 25 mL of the hydrolyzed sample to zero the instrument in the reactive phosphorus procedure. This compensates for any turbidity dissolved by this procedure.

SUMMARY OF METHOD

This procedure lists the necessary steps to convert condensed phosphate forms (meta-, pyro- or other polyphosphates) to orthophosphate before analysis. The procedure uses acid and heat to hydrolyze the sample. Organic phosphates are not converted to orthophosphate by this process, but a very small fraction may be unavoidably included in the result. Thus, the "acid hydrolyzable" phosphate results are primarily a measure of inorganic phosphorus. This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorous content of the sample.

The following reagents and apparatus are required in addition to those required for the reactive phosphorus test.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL* MDB	2450-32
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL* MDB	2449-32

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508-40
Flask, erlenmeyer, 50 mL	1	each	505-41

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Hydrochloric Acid, 6 N	500 mL	884-49
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Cylinder, graduated, 50 mL	each	508-41
Flask, erlenmeyer, 125 mL	each	505-43
Hot Plate, 3 $^{1}/_{2}$" diameter, 120 Vac	each	12067-01
Hot Plate, 3 $^{1}/_{2}$" diameter, 240 Vac	each	12067-02
Pad, cooling, 4" x 4"	each	18376-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00

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*Contact Hach for larger sizes.

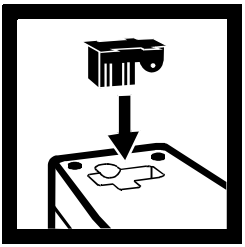
PHOSPHORUS, REACTIVE

(0 to 2.50 mg/L PO_4^{3-})

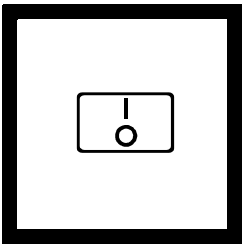
For water, wastewater, seawater

(also called: Orthophosphate) PhosVer 3 (Ascorbic Acid) Method* (Powder Pillows or AccuVac Ampuls), USEPA accepted for reporting**

USING POWDER PILLOWS

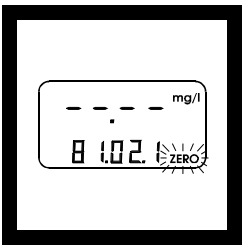


1. Install DR/700 module
81.01
in the instrument.



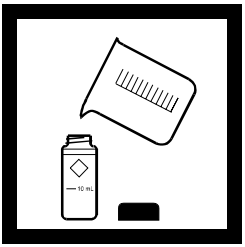
2. Press: **I/O**

The display will show
810 nm
and module number
81.01



3. After 2 seconds, the display will show a program number, concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
81.02.1

*Adapted from *Standard Methods for the Examination of Water and Wastewater.*
**Procedure is equivalent to USEPA method 365.2 and Standard Method 4500-P-E for wastewater.

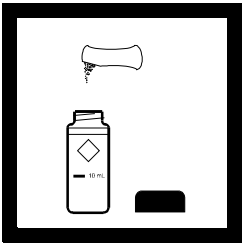


4. Fill a 10-mL cell to the 10-mL line with sample.

Note: *For proof of accuracy, use a 1.0 mg/L Phosphate (0.33 mg/L P) Standard Solution (listed under Optional Reagents) following these steps, in place of the sample.*

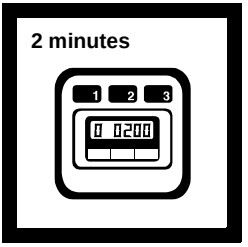
Note: *Run a reagent blank for this test. Use demineralized water in place of the sample in Step 4. Subtract this result from all test results run with this lot of PhosVer.*

Note: *Optional 25-mL reagents sample may be used (see Optional Reagents).*



5. Add the contents of one PhosVer 3 Phosphate Powder Pillow to the sample cell (the prepared sample). Cap and invert several times to mix.

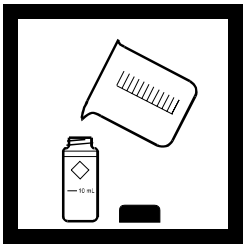
Note: *A blue color will form if phosphate is present.*



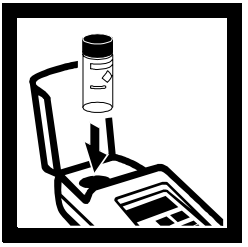
6. Wait two minutes.

Note: *An 8-10 minute reaction period should be used if determining total phosphate following the acid-persulfate digestion.*

Note: *If the sample temperature is less than 15 °C (59 °F), allow 9 minutes of reaction time.*

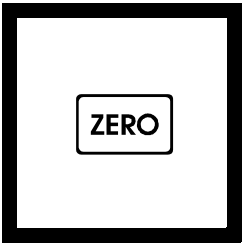


7. Fill a 10-mL cell to the 10-mL line with sample (the blank).



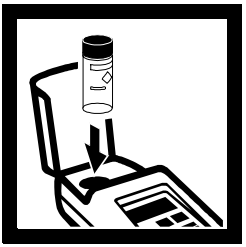
8. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



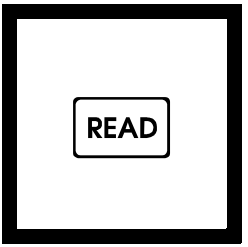
9. Press: **ZERO**

The display will count down to 0 Then the display will show 0.00 mg/L, and the zero prompt will turn off.



10. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



11. Press: **READ**

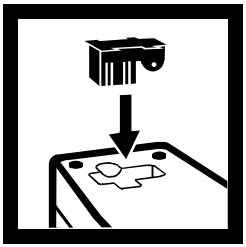
The display will count down to 0. Then the display will show the results in mg/L phosphate (as PO_4).

Note: *To convert results to other units, see Table 1.*

Table 1. Conversion Factors

To convert reading from	To	Multiply by
mg/L PO_4	mg/L P_2O_5	0.747
mg/L PO_4	mg/L P	0.326

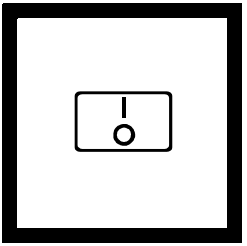
USING ACCUVAC AMPULS



1. Install DR/700 module

81.01

in the instrument.



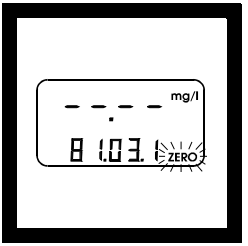
2. Press: **I/O**

The display will show

810 nm

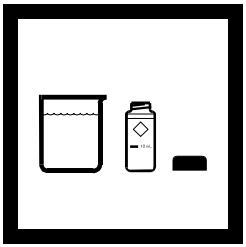
and module number

81.01



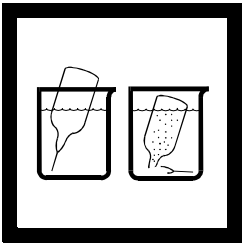
3. After 2 seconds, the display will show a program number, concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number

81.03.1



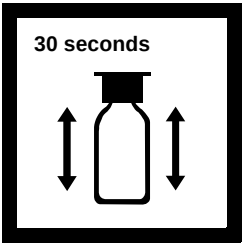
4. Fill a cell with 10 mL of sample (the blank). Cap. Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Run a reagent blank for this test. Use demineralized water in place of the sample in Step 4. Subtract this result from all test results run with this lot of PhosVer.*



5. Fill a PhosVer 3 Phosphate AccuVac Ampul with sample.

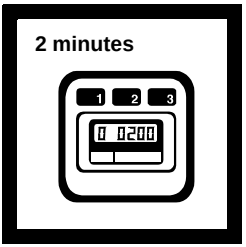
Note: *Keep the tip immersed while the ampul fills completely.*



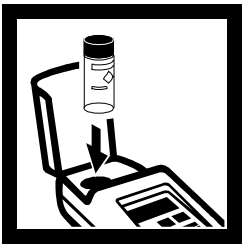
6. Place an ampul cap securely over the tip of the ampul. Shake the ampul for approximately 30 seconds. Wipe off any liquid and finger prints.

Note: *A blue color will form if phosphate is present.*

Note: *Accuracy is unaffected by undissolved powder.*

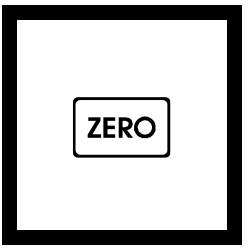


7. Wait two minutes.



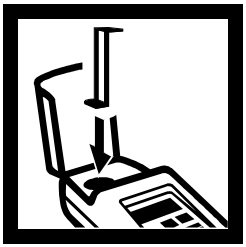
8. Place the blank in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

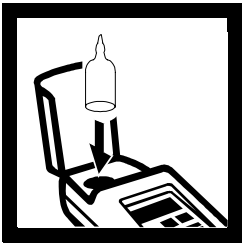


9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.0 mg/l, and the zero prompt will turn off.

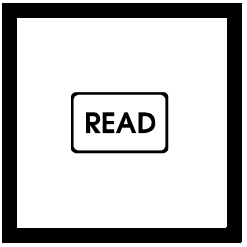


10. Insert the AccuVac Vial Adapter into the cell holder.



11. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L phosphate (as PO_4).

Note: *To convert results to other units, see Table 2.*

Table 2. Conversion Factors

To convert reading from	To	Multiply by
mg/L PO_4	mg/L P_2O_5	0.747
mg/L PO_4	mg/L P	0.326

SAMPLING AND STORAGE

Collect sample in plastic or glass bottles that have cleaned with 1:1 Hydrochloric Acid Solution and rinsed with demineralized water. Do not use commercial detergents containing phosphate for cleaning glassware used in phosphate analysis.

Most reliable results are obtained when samples are analyzed as soon as possible after collection. if prompt analysis is impossible, preserve samples up to 24 hours by storing at or below 4 °C. For longer storage periods, add 4.0 mL of Mercuric Chloride Solution to each liter of sample taken and mix. Use of mercuric chloride is discouraged whenever possible for health and environmental considerations. Sample refrigeration is still required. Samples preserved with mercuric chloride must have a sodium chloride level of 50 mg/L or more to prevent mercury interference. Samples low in chloride should be spiked with 0.1 g sodium chloride per liter of sample.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Phosphate Voluette Ampule Standard Solution, 50 mg/L PO_4^{-}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 25-mL water sample. Mix each thoroughly. (For AccuVac Ampuls use 50-mL Beakers.)

c) Analyze each sample as described above. The phosphate concentration should increase 0.2 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section 1) for more information.

INTERFERENCES

Large amounts of turbidity may cause inconsistent results in the phosphate tests because the acid present in the powder pillow may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles. For highly turbid or colored samples, add the contents of one Phosphate Pretreatment Powder Pillow to 25 mL of sample. Mix well. Use this solution to zero the instrument.

The PhosVer 3 Reagent Powder Pillows should be stored in a cool, dry environment.

The following may interfere when present in concentrations exceeding these listed below:

Copper	10 mg/L
Iron	100 mg/L
Silica	50 mg/L
Silicate	10 mg/L

Arsenate and hydrogen sulfide do interfere.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section 1).

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 1.60 mg/L PO_4^{3-} concentration samples, the standard deviation was ±0.007 mg/L PO_4^{3-}.

Testing zero concentration samples, the limit of detection was 0.019 mg/L PO_4^{3-}. The limit of detection was calculated as three times the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52,* 2242-2249.

Using two representative lots of AccuVacs, the standard deviation was ±0.008 mg/L PO_4^{3-} and the limit of detection was 0.021 mg/L PO_4^{3-}.

SUMMARY OF METHOD

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent			
Powder Pillows	1 Pillow	100/pkg	21060-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

PhosVer 3 Phosphate Reagent			
AccuVac Ampuls	1 ampul	25/pkg	25080-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening			
powder pillows	1	each	968-00
DR/700 Filter Module			
Number 81.01	1	each	46281-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41
Cap, ampul, blue	1	25/pkg	1731-25
DR/700 Filter Module			
Number 81.01	1	each	46281-00

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Hydrochloric Acid		
Standard Solution, 6.0 N (1:1)	500 mL	884-49
Mercuric Chloride Solution, 10 g/L	100 mL	14994-42
Phosphate Pretreatment Powder Pillows	50/pkg	14501-66
Phosphate Standard Solution,		
1 mg/L as PO_4	500 mL	2569-42
Phosphate Standard Solution, Voluette ampul,		
50 mg/L as PO_4, 10 mL	16/pkg	171-10
PhosVer 3 Phosphate Reagent		
Powder Pillows, 25 mL sample	100 pkg	2125-99
Sodium Chloride, ACS	454 g	182-01
Sodium Hydroxide		
Standard Solution, 5.0 N	100 mL* MDB	2450-32
Water, demineralized	4 L	272-56

OPTIONAL APPARATUS

Adapter, AccuVac Vial, DR/700	each	46025-00
Ampule Breaker Kit	each	21968-00
Cap for 10- and 25-mL sample cells	12/pkg	24018-12
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, 2 mL serological	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01	50/pkg	21856-96
Pipet Filler, safety bulb	each	14651-00
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Spoon, measuring, 0.1 g	each	511-00

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PHOSPHORUS, TOTAL

For water, wastewater and seawater

(also called Organic and Acid Hydrolyzable) Acid Persulfate Digestion Method*; USEPA accepted for reporting

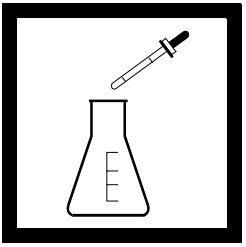
1. Measure 25 mL of sample into a 50-mL erlenmeyer flask.

Note: *Use a graduated cylinder to measure the sample.*

Note: *Rinse all glassware with 1:1 Hydrochloric Acid Solution. Rinse again with demineralized water.*

Note: *If prompt analysis is impossible, see Sampling and Storage following these steps.*

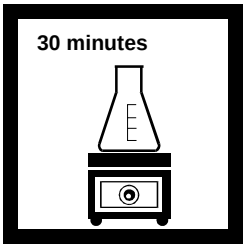
2. Add the contents of one Potassium Persulfate Powder Pillow. Swirl to mix.



3. Add 2.0 mL of 5.25 N Sulfuric Acid Solution.

Note: *Use the 1-mL calibrated dropper provided.*

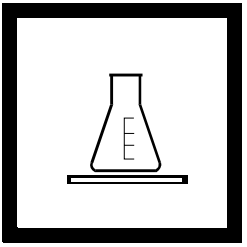
* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



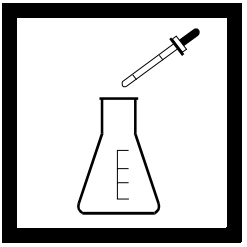
4. Place the flask on a hot plate. Boil gently for 30 minutes.

Note: *Maintain the volume near 20 mL by adding small amounts of demineralized water. Do not exceed 20 mL.*

Note: *This is more easily accomplished with a "double-boiler" technique. Place the flask in a pan or beaker of boiling water which is deeper than the solution level in the flask, Continue boiling for 30 minutes.*

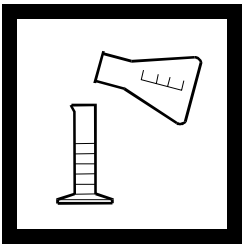


5. Cool the sample to room temperature.



6. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution. Swirl to mix.

Note: *Use the 1-mL calibrated dropper provided.*



7. Pour the sample into a 25-mL graduated cylinder. Using demineralized water rinsings from the flask, return the volume in the cylinder to 25 mL. Proceed with a reactive phosphorus test of the expected total phosphorus concentration range.

Note: *Results of the reactive phosphorus test at this point will include the organic phosphate plus the orthophosphate and the acid hydrolyzable (condensed) phosphate. The organic phosphate concentration is determined by subtracting the results of an acid hydrolyzable phosphorus test from this result. Make sure that both results are in the same units, either mg/L* PO_4^{3-} *or mg/L P before taking the difference.*

SAMPLING AND STORAGE

Most reliable results are obtained when samples are analyzed immediately. If prompt analysis is not possible, samples may be preserved up to 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

INTERFERENCES

For turbid samples, use 50 mL of sample and double the reagent quantities. Use 25 mL of the digested sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed by this procedure. For alkaline or highly buffered samples it may be necessary to use additional acid in Step 3 to drop the pH of the solution below 1.

SUMMARY OF METHOD

Phosphates present in organic and condensed inorganic forms (meta-, pyro-, or other polyphosphates) must be converted to orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate. Organically bound phosphates are thus determined indirectly by subtracting the result of an acid hydrolyzable phosphorus test from the total phosphorus result.

This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorus content of the sample. If the ascorbic acid (PhosVer 3) method is used to measure the reactive phosphorus, this method is EPA accepted for NPDES reporting.

The following reagents and apparatus are required beside those required for the reactive phosphorus test.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No.
Potassium Persulfate Powder Pillows	1 pillow	50/pkg	2451-66
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL*MDB	2450-32

REQUIRED REAGENTS (continued)

Description	Quantity Per Test	Unit	Cat. No.
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL*MDB	2449-32
Water, demineralized	25 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, 25 mL	1	each	508-40
Flask, erlenmeyer, 50 mL	1	each	505-41

OPTIONAL REAGENTS

Hydrochloric Acid, 6 N (1:1)	500 mL	884-49
Sodium Hydroxide Solution, 5.0 N	1 L	2450-53

OPTIONAL APPARATUS

Cylinder, graduated, 50 mL	each	508-41
Flask, erlenmeyer, 125 mL	each	505-43
Hot Plate, 3 $^{1}/_{2}$-inch diameter, 120 Vac	each	12067-01
Hot Plate, 3 $^{1}/_{2}$-inch diameter, 240 Vac	each	12067-02
Pads, cooling, 4" x 4"	each	18376-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00

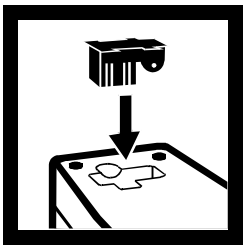
For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A - Contact the Hach office or distributor serving you.

SILICA, LR (0 to 2.00 mg/L)

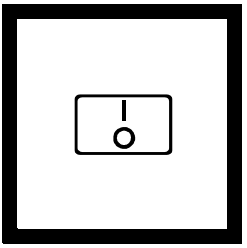
For water and seawater

Heteropoly Blue Method*



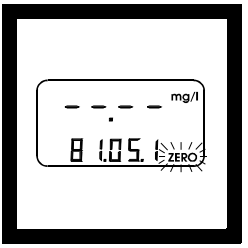
1. Install module
81.01
in a DR/700.

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*



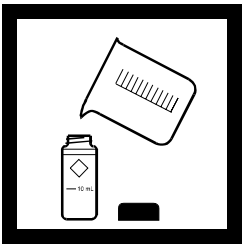
2. Press: **I/O**

The display will show
810 nm
and module number
81.01



3. After 2 seconds, the display will show a program number, concentration units, decimal position and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
81.05.1

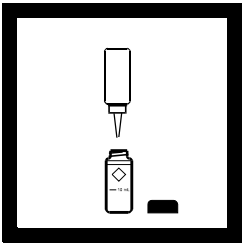
*Adapted from *Standard Methods for the Examination of Water and Wastewater.*



4. Fill a 10-mL cell to the 10-mL line with sample.

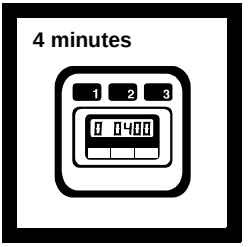
Note: *A 25-mL sample can be tested by using 25-mL sample cells and the reagents listed under Optional Reagents.*

Note: *For proof of accuracy, use a 0.5 or 1.0 mg/L Silica Standard Solution (listed under Optional Reagents) in place of the sample.*



5. Add 10 drops of Molybdate 3 Reagent to the cell. Cap and invert several times to mix.

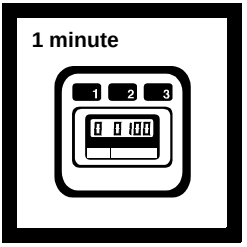
Note: *If 25-mL cells are used, add 0.5 mL of Molybdate 3 Reagent from the dropper supplied with the 100-mL bottle listed under Optional Reagents.*



6. Wait 4 minutes and remove cap.

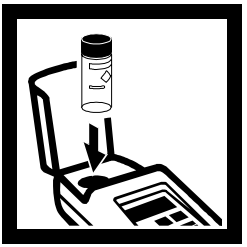
Note: *Reaction time depends on sample temperature. The time given is for samples at 20 °C (68 °F). If sample temperature is 10 °C (50 °F), wait 8 minutes. If at 30 °C (86 °F), wait 2 minutes.*

7. Add the contents of one Citric Acid Reagent Powder Pillow to the sample cell. Cap and invert several times to mix. This is the blank.



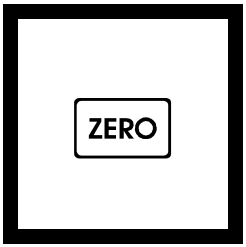
8. Wait 1 minute.

Note: *The one-minute reaction period is for samples at 20 °C (68 °F). If sample temperature is at 10°C (50 °F), wait two minutes. If sample temperature is at 30 °C (86 °F), wait 30 sec.*



9. Place the blank in the cell holder

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*

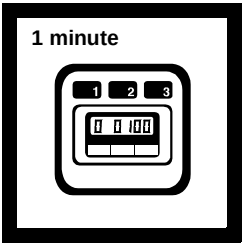


10. Press: **ZERO**

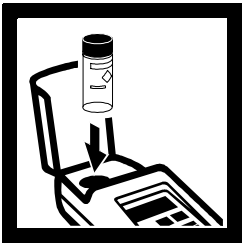
The display will count down to 0. Then the display will show 0.000 mg/L and the zero prompt will turn off.

11. Add the contents of one Amino Acid F Reagent Powder Pillow to the cell. This is the prepared sample. Cap and invert several times to mix.

Note: *A blue color will develop if silica is present.*

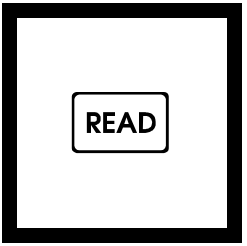


12. Wait 1 minute.



13. Place the prepared sample in the cell holder.

Note: *In bright sunlight it may be necessary to close the cell compartment cover.*



14. Press: **READ**

The display will count down 0. Then the display will show the results in mg/L silica (SiO_2).

SAMPLING AND STORAGE

Collect samples in clean plastic bottles. Analyze samples as soon as possible after collection. If prompt analysis is not possible, store samples for up to 7 days by cooling to 4 °C (39 °F) or below. Warm samples to room temperature before analysis.

ACCURACY CHECK

Standard Additions Method

a) Open a Low Range Silica Voluette Ampule Standard Solution, 50 mg/L SiO_2.

b) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of standard, respectively, to three 25-mL samples. Mix each thoroughly.

c) Analyze each of the spiked samples according to the above procedure. The silica concentration should increase 0.2 mg/L for each 0.1 mL of standard added.

d) If these increase do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Silica Standard Solutions of 0.500 mg/L and 1.000 mg/L SiO_2 are listed under Optional Reagents. Use these in place of the sample and analyze according to the above procedure.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of testing reagents. Testing 0.750 mg/L SiO_2 concentration samples the standard deviation was ±0.0161 mg/L SiO_2.

Testing zero concentration samples, the limit of detection was 0.0144 mg/L SiO_2. The limit of detection was calculated as three time the standard deviation when testing zero concentration samples (Adapted from *Analytical Chemistry,* **1980,** *52,* 2242-2249).

INTERFERENCES

Color and turbidity interferences are eliminated by zeroing the instrument with the original sample.

Sulfides and large amounts of iron interfere.

There is no interference from phosphate below 50 mg/L PO_4. At 60 mg/L PO_4, an interference of minus 2% is observed. At 75 mg/L the interference is minus 11%.

Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these "molybdate-unreactive" forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the Examination of Water and Wastewater,* Silica, Digestion with Sodium Bicarbonate. A longer reaction time of sample with the Molybdate 3 Reagent, before addition of citric acid, is often helpful in lieu of the bicarbonate pretreatment.

SUMMARY OF METHOD

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid preferentially destroys the phosphate complexes. An amino acid is then added to reduce the yellow silicomolybdate to an intense blue color, which is proportional to the silica concentration.

REQUIRED REAGENTS

Description	Quantity Per Test	Unit	Cat. No
Amino Acid F Reagent Powder Pillows	1 pillow	100/pkg	22540-69
Citric Acid Powder Pillows	2 pillows	100/pkg	21062-69
Molybdate 3 Reagent	1.0 mL	50 mL SCDB	1995-26

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
DR/700 Filter Module Number 81.01	1	each	46281-00

OPTIONAL REAGENTS

Amino Acid F Reagent Powder Pillows, 25 mL sample size	100/pkg	22538-69

OPTIONAL REAGENTS

Description	Unit	Cat. No
Citric Acid Powder Pillows		
25 mL sample size	100/pkg	14548-99
Molybdate 3 Reagent, 25 mL sample size	100 mL MDB	1995-32
Silica Standard Solution, 0.500 mg/L SiO_2	3.78 L	21008-17
Silica Standard Solution, 1.000 mg/L SiO_2	500 mL	1106-49
Silica Voluette Ampule		
Standard Solution, 50 mg/L	16/pkg	1117-10
Sodium Bicarbonate, ACS	454 g	776-01
Sulfuric Acid Standard Solution, 1.0000 N	1000 mL	1270-53

OPTIONAL APPARATUS

Caps for 10- and 25-mL sample cells	12/pkg	24018-12
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Sample Cell, 10-mL with screw cap	6/pkg	24276-06
Sample Cell, 25-mL with screw cap	6/pkg	24019-06
Standard Methods for the Examination of Water and Wastewater, 18th edition	each	22708-00
Thermometer, - 20 to 105 °C	each	1877-01

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Outside the U.S.A. - Contact the Hach office or the distributor serving you.

SUSPENDED SOLIDS (0 to 750 mg/L)

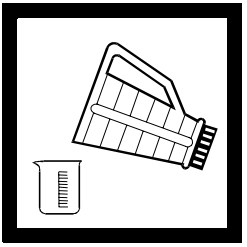
For water and wastewater

(Also called Nonfilterable Residue) Photometric Method*

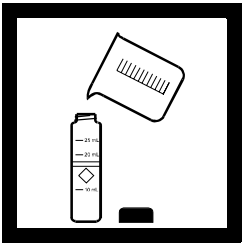
1. Blend 500 mL of sample in a blender at high speed for exactly two minutes.

Note: *If sample cannot be analyzed immediately, see Sampling and Storage following these steps.*

Note: *Obtain blender locally. All other apparatus is available from Hach.*

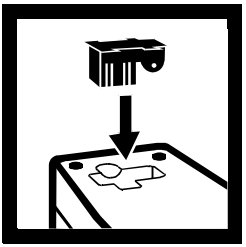


2. Pour the blended sample into a 600-mL beaker.

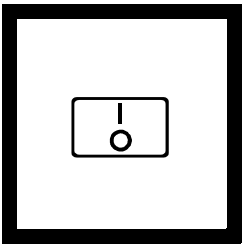


3. Stir the sample and immediately pipet 25 mL of the blended sample from the center of the beaker into a sample cell (the prepared sample). Cap.

Note: *Use a pipet filler or bulb.*

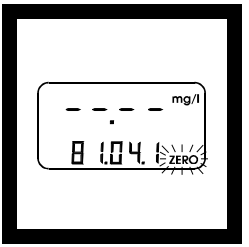


4. Install module
81.01
in a DR/700.



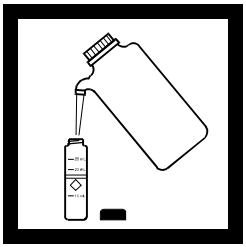
5. Press: **I/O**

The display will show
810 nm
and module number
81.01



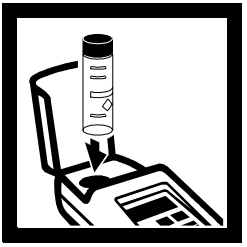
6. After 2 seconds, the display will show a program number, the concentration units and the zero prompt. If necessary, press the **UP ARROW** key until the lower display shows program number
81.04.1

* Adapted from *Sewage and Industrial Waste, 31,* 1159 (**1959**)



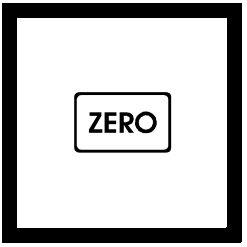
7. Fill a 25-mL cell to the 25-mL line with tap or demineralized water (the blank).

Note: *Remove gas bubbles in the tap water by swirling or tapping the bottom of the cell on a table top.*



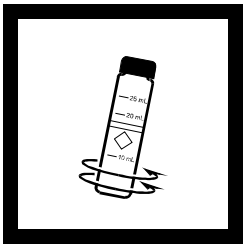
8. Place the blank in the cell holder.

Note: Use 10-mL cells for the blank and the prepared sample in bright sunlight. Close the cell compartment cover.

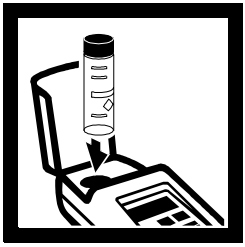


9. Press: **ZERO**

The display will count down to 0. Then the display will show 0.00 mg/L and the zero prompt will turn off.

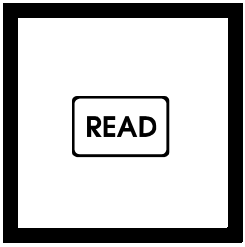


10. Slowly and gently invert and swirl the prepared sample to uniformly suspend any residue. Avoid vigorous mixing which would introduce gas bubbles into the sample.



11. Place the prepared sample into the cell holder.

Note: Use 10-mL cells for the blank and the prepared sample in bright sunlight. Close the cell compartment cover.



12. Press: **READ**

The display will count down to 0. Then the display will show the results in mg/L nonfilterable residue.

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Analyze as soon as possible after collection. Samples may be stored for 7 days by cooling to 4 °C (39 °F).

INTERFERENCES

Calibration for this test is based on parallel samples using the gravimetric technique on sewage samples form a municipal sewage plant. For most samples, this calibration will provide satisfactory results. When higher accuracy is required, it is recommended that parallel spectrophotometric and gravimetric determinations be run using portions of the same sample. The new calibration should be made on your particular sample using a gravimetric technique as a basis.

STATISTICAL EVALUATION

A single operator repetitively tested samples of two laboratory prepared solutions, using one DR/700, matched sample cells and two representative lots of reagents. Testing 325 mg/L Nonfilterable Residue (Suspended Solids) samples the standard deviation was ±3 mg/L Nonfilterable Residue (Suspended Solids).

SUMMARY OF METHOD

The photometric method of determining suspended solids is a simple, direct measurement which does not require the filtration or ignition and weighing steps necessary in gravimetric procedures. The stored program has been calibrated using samples from a municipal sewage treatment plant. The USEPA specifies the gravimetric method for solids determinations, while the photometric method is often used for checking in-plant processes.

REQUIRED APPARATUS

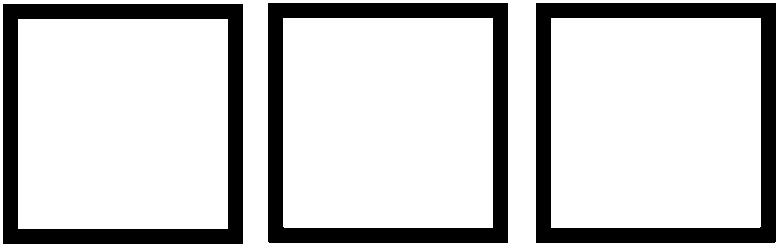
Description	Quantity Per Test	Unit	Cat. No.
Beaker, 600 mL, poly	1	each	1080-52
Blender	1	each	obtain locally
DR/700 Filter Module Number 81.01	1	each	46281-00

OPTIONAL APPARATUS

Cap for 10- and 25-mL sample cells		12/pkg	24018-12
Cylinder, 500 mL, graduated, poly		each	1081-49
Stirring Rod, glass		3/pkg	1770-01
Sample Cell, 10-mL with screw cap		6/pkg	24276-06
Sample Cell, 25-mL with screw cap		6/pkg	24019-06

For Technical Assistance, Prices and Ordering

In the U.S.A. - Call 800-227-4224 toll-free for more information.
Outside the U.S.A. - Contact the Hach office or distributor serving you.



SECTION III TECHNICAL SUPPORT

TECHNICAL TRAINING WORKSHOPS

Free technical training workshops are provided for water and wastewater analysis featuring demonstrations and hands-on experiences. Attendance is limited to keep classes small and personalized. Each workshop is two days. Workshops are available for:

•Water Analysis
•Wastewater Analysis
•Boiler/Cooling Water
•Science Educators Workshop
•Heavy Metals and Sludge Analysis
•Microbiology
•Various Food and Agriculture Topics

Location and Schedule

Workshops are held at our Technical Training Center in Loveland, Colorado. Loveland is a mid-sized town about 45-minutes north of Denver.

Workshops are scheduled for Monday-Tuesday and Thursday-Friday so travel one way can be on a weekend.

To obtain the latest workshop listing and schedule, call toll-free (800) 227-4224 and ask for the Training Center Administrator or contact the Hach office or distributor serving you.

TECHNICAL AND APPLICATION ASSISTANCE

A staff of trained technical service representatives and chemists are available to handle your specific questions and make application recommendations. Telephone numbers, fax numbers and mailing addresses are given below.

Hach Company, World Headquarters
P.O. Box 389
Loveland, Colorado, 80539 U.S.A.
Telephone (970) 669-3050
FAX: (970) 669-2932

ORDERING INFORMATION

Prices for the reagents/apparatus in the procedures can be found in the Hach Products for Analysis Catalog (PFA). Please call 800-227-4224 if you cannot locate an item.

TECHNICAL PUBLICATIONS

This information is grouped into the following categories:

- General Information
- Agricultural Information
- Biochemical Oxygen Demand
- Digestion
- Food, Feed and Beverage
- General Chemistry
- Microbiology
- pH Measurement
- Plating and Surface Finishing
- Process Analysis
- Sludge Analysis
- Spectrophotometric Analysis
- Turbidity
- Water/Wastewater Treatment

To request a specific publication, circle the appropriate number on the literature request form at the end of this section. These publications are free. If possible, please limit your requests to six publications.

General Information

Hach's free newsletter provides up-to-date information for the analyst. Published three times per year, *News and Notes for the Analyst* contains updates on federal regulations, in-depth discussions of chemical methods, application notes for instrument or test procedures and a Hach product update.

Products for Analysis catalog describes laboratory, portable, and on-line instruments, including microbiological testing products, spectrophotometers, pH/ISE electrodes and meters, digestion apparatus and test kits, as well as labware, chemicals and reagents.

Agricultural Analysis
Systems for Agricultural Analysis. This catalog describes chemical analysis systems for soil, greenhouse media, sap, plant tissue and fluid fertilizer.

Biochemical Oxygen Demand
"A Comparison of the Graphical and Standard Methods for the Determination of Biochemical Oxygen Demand". By Stephen L. Woodring and Dennis A. Clifford. Reprinted from *Journal Water Pollution Control Federation*, Vol. 60, No. 4, April, 1988.

The Graphical method was found to increase reliability in BOD data and give more confidence in very low BOD levels obtained in testing secondary and tertiary effluent of biological processes.

"Graphical Method for Calculating Biochemical Oxygen Demand". By Robert J. Klein Jr. and Charles R. Gibbs. Reprinted from *Journal Water Pollution Control Federation*, Vol. 51, No. 9, September, 1979.

Presents a graphical technique for handling BOD test results that automatically corrects for blank and seed, permits uses of dilution water with a higher demand concentration, does not require an initial DO determination and provides added information.

Introduction to BOD
This brochure describes Hach's biochemical oxygen demand system. A method to make dilution water quickly is included.

Introduction to Biochemical Oxygen Demand. By Clifford Hach, Robert L. Klein, Jr. and Charles R. Gibbs, 1988.

Provides answers to the questions: What is BOD? What is the significance of BOD? How is BOD measured? How reliable is the BOD test?

Chemical Oxygen Demand
Hach COD system for Wastewater Testing. Describes a semi-micro COD monitoring system with ready-to-use sample vials. Includes a cost comparison.

Introduction to Chemical Oxygen Demand. By Charles R. Gibbs, 1987.

Discusses methods for determining oxygen demand, digestion procedures, measurement procedures and appropriate reagents and apparatus.

Digestion
Acid Digestion Using the Hach Digesdahl Digestion Apparatus. Sample Preparation for Protein and Elemental Analysis. By Scott V. Brayton, 1988.

Focuses on digestion using sulfuric acid and hydrogen peroxide, a modification of the Kjeldahl method, which is suitable for determining a range of metals and nonmetals including nitrogen. The digestion was integrated into a total analytical system based on spectrophotometric analysis. Analytes determined include: Ag, Al, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, N, Ni, Pb, P, S, and Zn. Circle number 7016.

"Accuracy in Kjeldahl Protein Analysis". By Y.S. Chen, et al. Reprinted from *American Laboratory*, June, 1988.

Discusses the basis for the accuracy of the Kjeldahl method and describes what has been achieved in establishing the absolute accuracy for nitrogen determination using a modified Kjeldahl procedure.

Food, Feed and Beverage
"Golden Cheese Streamlines Quality Control Testing". Reprinted from *Prepared Foods*.

Describes a Quality control program that provides rapid, accurate, economical results with little personal training for a food processor.

General Chemistry
"Measurement of Color and Insoluble Matter in Reagent Chemicals". By Clifford C. Hach. Reprinted from *American Laboratory*, June, 1983.

When results from colorimetric and turbidimetric tests are compared with traditional gravimetric measurements, the colorimetric tests are shown to offer more sensitive and useful information about insoluble and color-related impurities.

"Spectrophotometric Determination of Chlorine Dioxide". By Daniel L. Harp, R.L. Klein Jr., and D.J Schoonover. Reprinted from *Journal American Water Works Association*, Vol. 73, No. 7, July, 1981.

Spectrophotometric procedures based on the oxidation of chlorophenol red, a pH indicator, can determine trace amounts of chlorine dioxide in water.

Microbiology
The Use of Indicator Organisms to Assess the Safety of Public Water. By Sharon Sloat and Carol Ziel, 1987.

Describes how indicator organisms are used as the basis for most microbiological quality standards. The topics covered are: indicator organisms, drinking water standards, methods for the enumeration of indicator and opportunistic organisms, media formulations and preparation and references.

pH Measurement
"A Common Source of Error in pH Measurement". By John A Illingsworth. Reprinted from *Biochemical Journal*, Vol. 195, pages 259-262, 1981.

Studies the reference junction as a source of error in pH measurements. The reference junctions of 30 electrodes selected at random from seven laboratories were compared to the ideal of 7.06 ±0.01 pH. Twenty-four of the electrodes failed,

"Low Ionic Strength pH Measurement". By Alan Kopelove, Stanley Franklin and Gale McGaha Miller. Reprinted from *American Laboratory*, June, 1989.

"pH Measurement Problems Solved". By Gale McGaha Miller. Reprinted from *American Laboratory News Edition,* October, 1988.

This article compares the conventional reference junction used in most pH electrodes and the free diffusion reference junction used in Hach pH electrodes.

Plating and Surface Finishing
"Heavy Metal Removal Using Dithiocarbamates".
By R.E. Wing and W.E. Rayford. Reprinted from *Plating & Surface Finishing*, January, 1982.

Dithiocarbamates effectively precipitate heavy metals from plating rinse water over a wide pH range. Concentrations of residual metal are well below established EPA discharge limits.

"Know Your Water Quality: Influent and Effluent". By Tom Haukebo. Reprinted from *Plating & Surface Finishing*, November, 1986.

Discusses how knowledge of the quality of water at both ends of the surface finishing process is critical for product quality, cost control and legal reasons. Methods are available for the simple and reliable determination of water quality without the need for outside services and within the requirements of environmental regulations.

"Sodium Hypophosphite Analysis in Electroless Nickel". By Diane M. Tramontana. Reprinted from Metal Finishing, December, 1986.

In the smaller job shop, where expensive analytical equipment cannot be justified, wet analytical methods are recommended for monitoring the sodium hypophosphite concentrations used in electroless nickel plating.

Process Analysis
Introduction to: *The Pump-Colorimeter Analyzer.* By Paul Larson, Charles Gibbs, Clifford Hach and David Schnoover, 1987.

Describes development of a continuous-reading on-line analyzer for water and wastewater, Topics covered are: design objectives, mechanical operation, optical design, and electronics. Also covered are field test results for free chlorine in drinking water, total chlorine in sewage, and orthophosphate in sewage.

"Keeping Tabs on Excess Chlorine". By Charles W. Ingrahm. Reprinted from *Water Engineering & Management*, April, 1983.

This article describes the use of an on-line instrument for measuring chlorine in effluent at a 12 mgd facility.

"Nonradioactive Silica Can Be Monitored On-line". By Clifford Hach. Reprinted from *Power*, Vol. 128, No. 1, 1984.

Combining turbidity measurement with on-line measurement of reactive silica provides a much needed tool for protection of high-pressure-system components.

Spectrophotometric Analysis

DR/2000. This brochure describes the capabilities of the DR/2000 spectrophotometer. This instrument combines microprocessor technology, sophisticated optics and prepackaged reagents to make colorimetric analysis easier than ever before. Over 120 preprogrammed calibrations are stored n the instrument's memory.

DR/3000. This brochure describes the capabilities of the DR/3000 spectrophotometer. This instrument is Hach's top-of-the-line spectrophotometer and features advanced, single-beam optics. Over 100 preprogrammed calibrations are stored in the instrument's memory.

Turbidity

Introduction to the Ratio Turbidimeter. By Richard D. Vanous, 1986.

Describes the design objectives and applications for the Ratio Turbidimeter in laboratory and process control settings, The unique optics of this instrument permit turbidity measurement in virtually any liquid, regardless of color.

Principals of Surface Scatter Turbidimetry Measurement. By Clifford Hach, 1986.

Addresses the problems of upper turbidity range limit, stray light interference and loss of sensitivity due to dirty optical surfaces. A design that minimizes these adverse effects is also presented.

"Turbidimeter Standards, Calibration, and Practice". By John M. Heer. Reprinted from *Waterworld News*, Vol. 3, No. 2, 1987.
Topics covered include: primary standards for turbidity, using formazin standards, secondary standards, calibration frequency and technique, calibration tips, and zero turbidity check.

Turbidity Standards. By Clifford Hach, 1986.

Describes the development of a primary turbidity standard, using formazin standards, secondary standards and appropriate reagents and apparatus.

Turbidity Measurement
"Turbidimetric Measurements of High Purity Water". By Clifford C. Hach and Lawrence M. Liggett. Reprinted from *Ultrapure Water,* Vol. 2 No. 4, July/August, 1985.

Describes a very sensitive instrument technique for detecting particles in liquids, including particles too small to be detected by the most sensitive particle counters.

"Understanding Nephelometric Instrumentation". By Richard D. Vanous. Reprinted from *American Laboratory,* July, 1978.

Reviews the theory of nephelometric instrumentation and discusses the variables that influence turbidity measurements.

Understanding Turbidity Measurement. By Clifford Hach, Richard D. Vanous, and John M. Heer, 1989.

Provides an overview to turbidity measurement. The topics covered are: Introduction and Definition, Modern Instruments, Practical Aspects of Turbidity Measurement, Innovative Approaches to Turbidity Measurement.

Water/Wastewater Treatment

"Backwash Turbidimeter Lowers Operating Costs". By Josephine W. Boyd. Reprinted from *Opflow,* Vol. 11, No. 3, March 1985.

Describes how the use of a backwash turbidimeter results in a significant drop in consumption of both finished water and power. This instrument is easier to use and more reliable than attempting to control the process by visual measurement.

"Get More Information from Water Analysis". By Don Hartman. Reprinted from *Water Conditioning & Purification Magazine*, September, 1988.

Discusses the most common inorganic tests that can be done by a layman in the field or at a small, in-store laboratory. Some data manipulation techniques are offered that add more value to the collected data.

"Low-cost Lab Work". By J.W. Boyd and T.W. Farrill. Reprinted from *Operations Forum,* December, 1986.

Plant operators are discovering colorimetric test methods for metal ions offer a way to comply with the U. S. Environmental Protection Agency's pretreatment and National Pollution Discharge Elimination System testing requirements and still stay within budget.

"Weigh the Advantages of Hach's Prepared Reagents". Compares the cost of using reagents prepared by a manufacturer to the cost of using do-it-yourself reagents for chlorine analysis.

"Water Hardness Above 2 ppm Alerted by Hardness Monitor". By Ken Wilson and Dr. Samuel Lee. Reprinted from *Chemical Processing,* September, 1985.

Regeneration of zeolite beds used in softening water has been cut by two-thirds by the use of hardness monitors.

HOW TO ORDER

By Phone:
6:00 a.m. to 5:00 pm. MST Monday through Friday
800-227-HACH (800-227-4224) (U.S.A. only)
(970) 669-3050

By Mail:
Hach Company World Headquarters
P.O. Box 389
Loveland, Colorado, U.S.A. 80539-038

By Fax:
970-669-2932 (Hach Loveland)

Information Required:
- Hach account number (if available)
- Billing Address
- Shipping address
- Your name and phone number
- Purchase order number
- Catalog number
- Brief description or model number
- Quantity

On orders over $500, we require a written or faxed copy of the purchase order or purchase requisition, complete with authorized signature and your terms and conditions.

Outside the United States
Hach maintains a network of dealers and distributors throughout the world.

Obtain assistance from a nearby Hach distributor or:
Hach Company World Headquarters
P.O. Box 389
Loveland, Colorado, U.S.A. 80539-0389
Telephone: (970) 669-3050
Fax: (970) 669-2932

In Canada:
Hach Sales & Service Canada Ltd.
1313 Border Street, Unit 34
Winnipeg, Manitoba R3H 0X4
Telephone: (204) 632-5598
Fax: (204) 694-5134

Information presented on these pages applies only to Hach products manufactured for use within the United States. Exportation of these products renders these terms void.

Shipment
Hach products are shipped FOB from the shipping point. If you do not specify a method of shipment, our staff will determine the best means of transportation consistent with Department of Transportation and postal service regulations. Motor freight shipments will be sent freight collect unless specified otherwise at the time of order. Limited quantities of Class B poisonous and "Dangerous When Wet" materials may be shipped by United Parcel Service without additional charge. This shipment avoids slow and costly motor freight shipments. Although Hach ships your order as quickly as possible, we cannot guarantee delivery dates. Also, it is your responsibility to insure your order during shipment.

Claims and Returns
We take extreme care to fill and pack orders properly. If your freight appears to be damaged when you receive it, do not accept it from the carrier. If you have accepted a delivery and find hidden damages, call a Hach customer service representative immediately. Be sure to keep all containers and packing materials.

AUTHORIZATION MUST BE OBTAINED from Hach when returning items for any reason. Call 800-227-4224; Alaska and Hawaii call 970-669-3050 collect. Sorry, but all "freight collect" shipments of returned merchandise must be refused.

If you need to return items, please follow the procedure below:

1. Within 30 days after receiving the merchandise, contact Hach customer service to get Return Authorization.

2. Hach will send you Return Authorization labels, documentation and instructions.

3. Attach Return Authorization labels and documents to the outside of the shipping cartons and ship to Hach within 15 days. Hach will refuse return shipments without these.

To receive credit, returned shipments must meet the following requirements:

1. Chemicals must be in original, unopened containers. Products that have been used or defaced may not be returned. For kits and portable laboratories, a refurbishing fee will be charged for non-returnable chemicals.

2. Freight charges associated with returned merchandise are the customer's responsibility. Hach will not accept "freight collect" shipments unless you get prior authorization.

3. All returns must be shipped within 15 days after receiving the authorization. Returns not shipped within this time need to be reauthorized.

4. All chemicals authorized for return must be packaged and shipped to comply with U.S. Department of Transportation regulations.

Prices and Terms
Prices are subject to change without notice. All prices are FOB from the shipping point (usually Ames, Iowa). Hach offers instant credit up to $200 on Net 30 Day terms. Larger orders are subject to credit review. Customers may send remittance with orders or we can ship C.O.D. if preferred.

Warranty
Hach warrants its products to be of high quality, to be free of material defects on the date of shipment and to be as specified. Full warranty information is on the back of Hach invoices.

Limits of Usage
Our chemicals and reagents are offered for laboratory and manufacturing use ONLY. They may not be used as drugs, cosmetics or food additives.

MSDS
Hach Material Safety Data Sheets, among the most complete and informative in the industry, provide comprehensive safety data essential for day-to-day operations and safety training.

An MSDS accompanies all Hach chemical products including test kits. For an additional $15.00 per item, we will print copies of Material Safety Data Sheets on your own forms.

Label Information
Labels on Hach chemicals and reagents supply the following:

- Product Name—Printed in French, German, Italian and Spanish as well as English on all but the smallest-size labels.
- Hach Catalog Number—Makes reordering easy and helps match the appropriate MSDS.
- Storage Information and Lot Numbers—Lot numbers made up of letters and numbers indicate an extended shelf life; a four-digit number indicates items should be rotated and checked with a standard to confirm performance. The lot number is essential if you call for technical assistance or with questions about reagent performance.