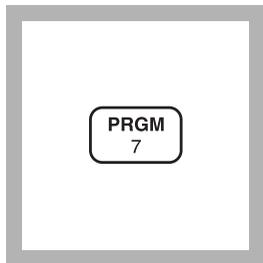


BROMINE (0 to 4.50 mg/L)

For water, wastewater, and seawater

DPD Method* (Powder Pillows or AccuVac Ampuls)**Using Powder Pillows**

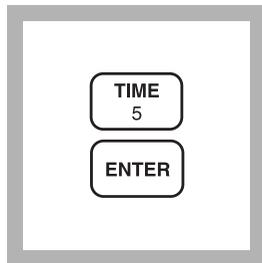
1. Enter the stored program number for bromine (Br₂)-powder pillows.

Press: **PRGM**

The display will show:

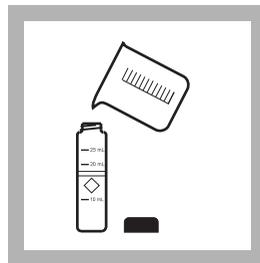
PRGM ?

Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).



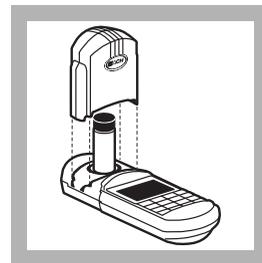
2. Press: **5 ENTER**

The display will show **mg/L, Br2** and the **ZERO** icon.



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

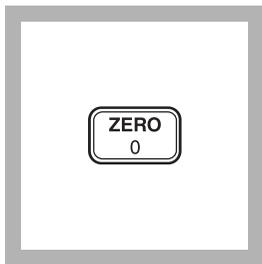
Note: Samples must be analyzed immediately and cannot be preserved for later analysis.



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

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

BROMINE, continued

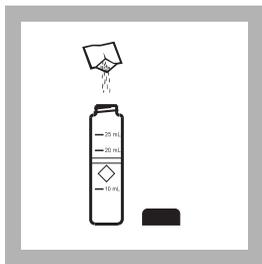


5. Press: **ZERO**

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

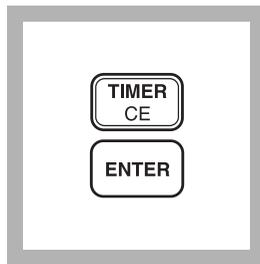
0.00 mg/L Br₂

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



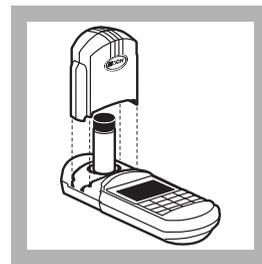
6. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and swirl vigorously to dissolve the powder.

Note: It is not necessary that all the powder dissolves. A pink color will develop if bromine is present.

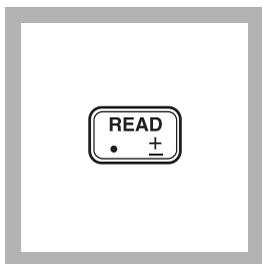


7. Press: **TIMER ENTER**

A three-minute reaction period will begin.



8. When the timer beeps, place the sample into the cell holder. Tightly cover the sample cell with the instrument cap.



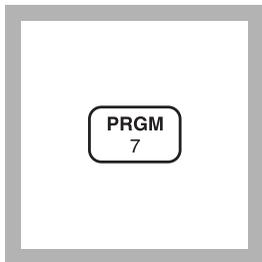
9. Press: **READ**

The cursor will move to the right, then the result in mg/L bromine will be displayed.

Note: If samples temporarily turn yellow after reagent addition, or the display flashes "limit", it is due to high bromine levels. Dilute fresh samples and repeat the test. A slight loss of bromine may occur during dilution. Multiply results by the dilution factor; see Section 1.

Note: Standard Adjust may be performed using a prepared standard (see Section 1).

Using AccuVac Ampuls



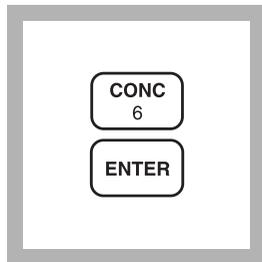
1. Enter the stored program number for bromine (Br_2) AccuVac Ampuls.

Press: **PRGM**

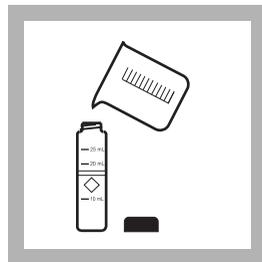
The display will show:

PRGM ?

Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).

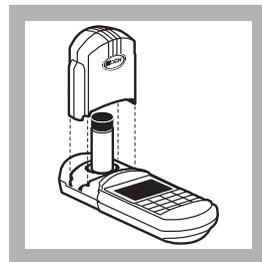


2. Press: **6 ENTER**
The display will show **mg/L, Br2** and the **ZERO** icon.

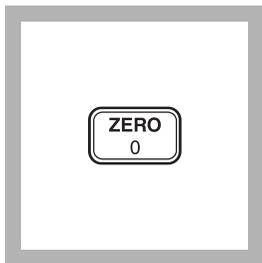


3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

Note: Samples must be analyzed immediately and cannot be preserved for later analysis.



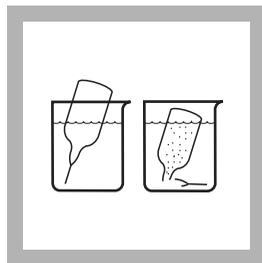
4. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**
The cursor will move to the right, then the display will show:

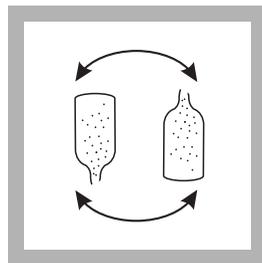
0.00 mg/L Br2

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



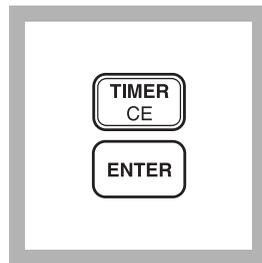
6. Fill one DPD Total Chlorine Reagent AccuVac Ampul with sample.

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



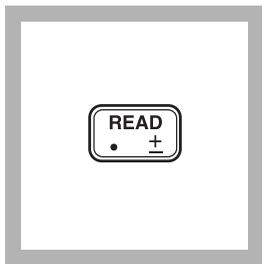
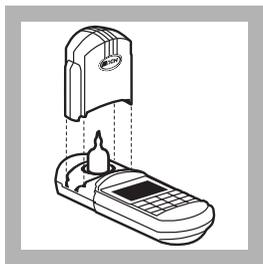
7. Quickly invert the ampule several times to mix. Wipe off any liquid or fingerprints.

Note: A pink color will form if bromine is present.



8. Press: **TIMER ENTER**
A three-minute reaction period will begin.

BROMINE, continued



9. After the timer beeps, place the AccuVac ampul into the cell holder. Tightly cover the ampule with the instrument cap.

10. Press: **READ**
The cursor will move to the right, then the result in mg/L bromine will be displayed.

Note: If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high bromine levels. Dilute a fresh sample and repeat the test. A slight loss of bromine may occur during dilution. Multiply the result by the dilution factor; see Section 1.

Note: Standard Adjust may be performed using a prepared standard (see Section 1).

Sampling and Storage

Analyze samples for bromine **immediately** after collection.

Avoid plastic containers since these may have a large bromine demand. **Pretreat glass** sample containers to remove any bromine demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for bromine is introduced when a representative sample is not obtained. If sampling from a tap, let the sample flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark.

Perform the bromine analysis immediately after collection.

Accuracy Check

Standard Additions Method (using powder pillows)

- a) Snap the top off a LR Chlorine PourRite[®] Ampule Standard Solution.
- b) Use a TenSette Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.
- c) Re-zero the instrument using the original sample (the blank).
- d) Place the spiked sample in the cell holder and press **READ**. Record the result.
- e) Calculate the equivalent concentration of mg/L bromine added to the sample:

$$\text{mg/L Bromine added} = \frac{0.1 (\text{vol. standard added}) \times \text{Label value (mg/L Chlorine)} \times 2.25}{10.1 (\text{sample} + \text{standard volume})}$$

- f) The spiked sample result (step d) should reflect the analyzed sample result + the calculated mg/L Br₂ added (step e).
- g) If this increase does not occur, see *Standard Additions in Section 1* for more information.

Standard Additions Method (using AccuVac Ampuls)

- a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.
- b) Use a graduated cylinder to measure 25 mL of sample into each of two beakers.
- c) Use a TenSette Pipet to add 0.2 mL of the standard to one of the beakers (this is the spiked sample). Swirl to mix.
- d) Fill a DPD Total Chlorine AccuVac completely from each beaker.
- e) Analyze the spiked and unspiked sample as described in the procedure.
- f) Calculate the equivalent concentration of mg/L bromine added to the sample:

BROMINE, continued

$$\text{mg/L Bromine added} = \frac{0.2 (\text{vol. standard added}) \times \text{Label value (mg/L Chlorine)} \times 2.25}{25.2 (\text{sample} + \text{standard volume})}$$

- g) The spiked sample result should reflect the analyzed sample result + the calculated mg/L Br₂ added (step f).
- h) If this increase does not occur, see *Standard Additions in Section 1* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 2.34 mg/L bromine and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.02 mg/L bromine.

In a single laboratory using a standard solution of 2.31 mg/L bromine and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation ± 0.02 mg/L bromine.

Estimated Detection Limit

The estimated detection limit for program 5 is 0.04 mg/L Br₂ and 0.03 mg/L Br₂ for program 6. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

BROMINE, continued

Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L CaCO ₃ . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Section 1, Correcting for Volume Additions</i>).
Alkalinity	Greater than 250 mg/L CaCO ₃ . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Section 1, Correcting for Volume Additions</i>).
Chlorine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as CaCO ₃
Iodine	Interferes at all levels
Manganese, Oxidized (Mn ⁴⁺ , Mn ⁷⁺) or Chromium, Oxidized (Cr ⁶⁺)	<ol style="list-style-type: none">1. Adjust sample pH to 6-7.2. Add 3 drops potassium iodide (30 g/L) to a 25-mL sample.3. Mix and wait 1 minute.4. Add 3 drops sodium arsenite (5 g/L) and mix.5. Analyze 10 mL of the treated sample as described in the procedure.6. Subtract the result from this test from the original analysis to obtain the correct bromine concentration.
Monochloramine	Interferes at all levels
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See <i>Interferences, Section 1</i> .

Summary of Method

Bromine reacts with DPD (N,N-diethyl-p-phenylenediamine) to form a magenta color which is proportional to the total bromine concentration.

BROMINE, continued

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interference will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS (USING POWDER PILLOWS)

Description	Quantity Required		Unit	Cat. No.
	Per Test			
DPD Total Chlorine Reagent Powder Pillows	1 pillow	100/pkg.....	21056-69	

REQUIRED REAGENTS (USING ACCUVAC AMPULS)

DPD Total Chlorine Reagent AccuVac Ampuls	1 ampule	25/pkg.....	25030-25
---	----------------	-------------	----------

REQUIRED APPARATUS (USING POWDER PILLOWS)

Sample Cells, 10-20-25-mL, w/ cap	6/pkg.....	24019-06
---	------------	----------

REQUIRED APPARATUS (USING ACCUVAC AMPULS)

Beaker, 50 mL	1	each.....	500-41
---------------------	---------	-----------	--------

OPTIONAL REAGENTS

Chlorine Standard Solution, PourRite ampule, 25-30 mg/L, 2 mL	20/pkg.....	26300-20
DPD Total Chlorine Reagent, SwifTest	250 Tests.....	28024-00
Potassium Iodide Solution, 30 g/L	100 mL* MDB.....	343-32
Sodium Arsenite, 5 g/L	100 mL* MDB.....	1047-32
Sodium Hydroxide Standard Solution, 1.000 N	100 mL* MDB.....	1045-32
Sulfuric Acid Standard Solution, 1 N	100 mL* MDB.....	1270-32
Water, deionized.....	4 L.....	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit.....	each.....	24052-00
PourRite Ampule Breaker.....	each.....	24846-00
Cylinder, graduated, 25 mL	each.....	508-40
pH Meter, <i>sensio</i> TM <i>I</i> , portable	each.....	51700-00
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg.....	391-33
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg.....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg.....	21856-28

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

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

* Contact Hach for larger sizes