Bromine

DOC316.53.01011

DPD Method[1]

Method 8016

0.05 to 4.50 mg/L Br_2

Powder Pillows or AccuVac® Ampuls

Scope and application: For testing bromine residuals (including hypobromite, hypobromous acid and bromamines) used as disinfectants in process waters, treated water, estuary water and seawater.

[1] Adapted from Standard Methods for the Examination of Water and Wastewater.

Test preparation

Instrument-specific information

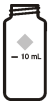
Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests. Table 2 shows sample cell and adapter requirements for AccuVac Ampul tests. The tables also show all of the instruments that have the program for this test.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for reagent addition

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606 
DR 3900	LZV846 (A)	
DR 1900	9609900 or 9609800 (C)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800 

Before starting

Samples must be analyzed immediately after collection and cannot be preserved for later analysis.
Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

Powder pillows

Description	Quantity
DPD Total Chlorine Reagent Powder Pillow, 10-mL	1
Sample cells. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to
Consumable and replacement items on page 6
for order information.

AccuVac Ampuls

Description	Quantity
DPD Total Chlorine Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Stopper for 18-mm tubes and AccuVac Ampuls	1

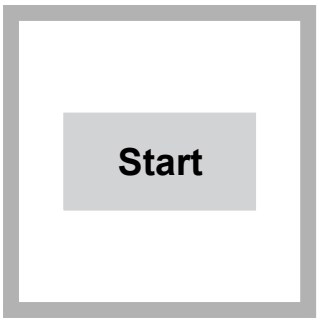
Refer to
Consumable and replacement items on page 6
for order information.

Sample collection

- Analyze samples for bromine immediately after collection.
- Bromine is a strong oxidizing agent and is unstable in natural waters. Bromine reacts quickly with various inorganic compounds and more slowly with organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence the decomposition of bromine in water.
- Collect samples in clean glass bottles. Do not use plastic containers because these can have a large bromine demand.
- Pretreat glass sample containers to remove bromine demand. Soak the containers in a weak bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse fully with deionized or distilled water. If sample containers are rinsed fully with deionized or distilled water after use, only occasional pretreatment is necessary.

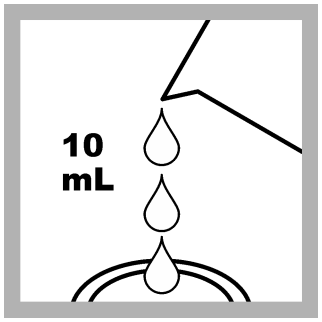
- Make sure to get a representative sample. If the sample is taken from a spigot or faucet, let the water flow for at least 5 minutes. Let the container overflow with the sample several times and then put the cap on the sample container so that there is no headspace (air) above the sample.

Powder pillow procedure

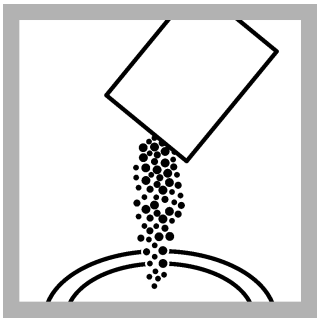


1. Start program **50 Bromine**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

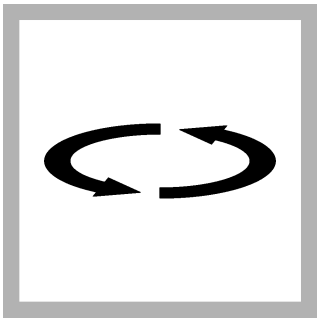
Note: *Although the program name can be different between instruments, the program number does not change.*



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



3. Prepare the sample: Add the contents of one powder pillow to the sample cell.

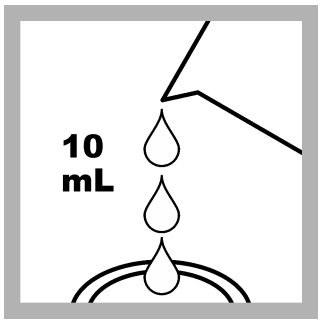


4. Swirl the sample cell for to mix.
A pink color shows if bromine is in the sample.

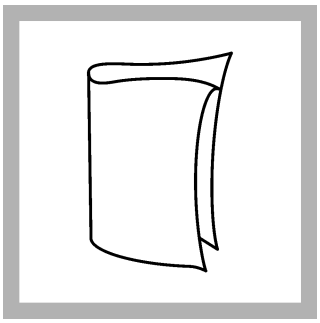


5. Start the instrument timer. A 3-minute reaction time starts.

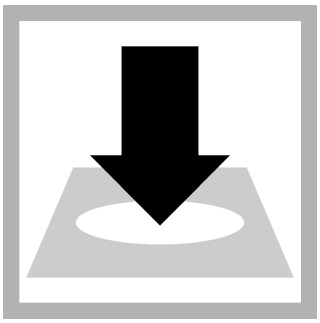
The instrument can be set to zero with the blank during the reaction timer.



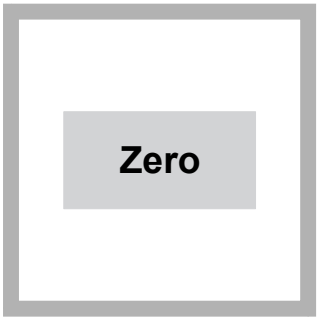
6. Prepare the blank: Fill a second sample cell with 10 mL of sample.



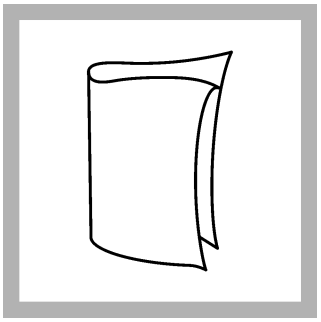
7. Clean the blank sample cell.



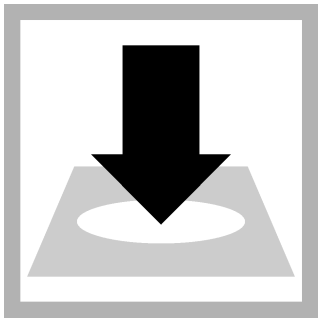
8. Insert the blank into the cell holder.



9. Push **ZERO**. The display shows 0.00 mg/L Br_2.



10. Clean the prepared sample cell.

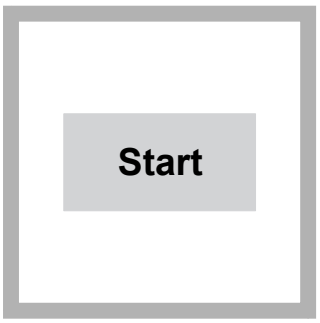


11. Within 3 minutes after the timer expires, insert the prepared sample into the cell holder.



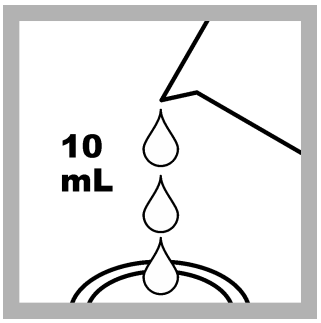
12. Push **READ**. Results show in mg/L Br_2.

AccuVac Ampul procedure

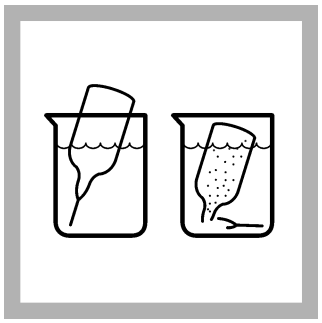


1. Start program **55 Bromine AV**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

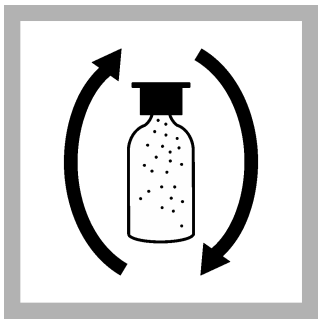
Note: *Although the program name can be different between instruments, the program number does not change.*



2. Prepare the blank: Fill the sample cell with 10 mL of sample.



3. Prepare the sample: Collect at least 40 mL of sample in a 50-mL beaker. Fill the AccuVac Ampul with sample. Keep the tip immersed while the AccuVac Ampul fills completely.

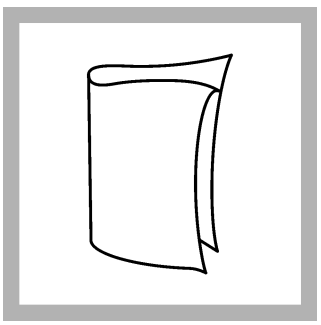


4. Quickly invert the AccuVac Ampul several times to mix.
A pink color shows if bromine is in the sample.

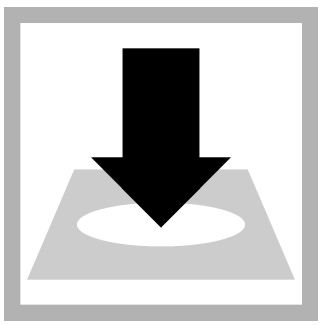


5. Start the instrument timer. A 3-minute reaction time starts.

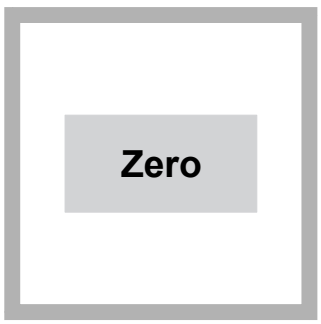
The instrument can be set to zero with the blank during the reaction timer.



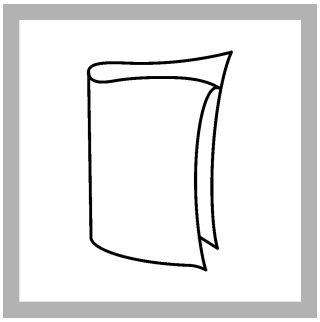
6. Clean the blank sample cell.



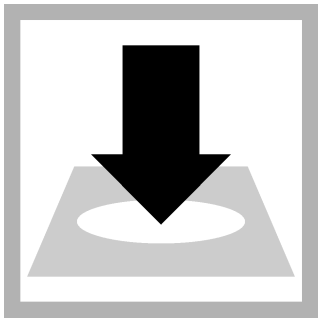
7. Insert the blank into the cell holder.



8. Push **ZERO**. The display shows 0.00 mg/L Br_2.



9. Clean the AccuVac Ampul.



10. Within 3 minutes after the timer expires, insert the prepared sample AccuVac Ampul into the cell holder.



11. Push **READ**. Results show in mg/L Br_2.

Interferences

Interfering substance	Interference level
Acidity	More than 150 mg/L $CaCO_3$. The full color may not develop or the color may fade instantly. Neutralize to pH 6–7 with 1 N Sodium Hydroxide. Measure the amount to be added on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution caused by the volume addition.
Alkalinity	More than 250 mg/L $CaCO_3$. The full color may not develop or the color may fade immediately. Neutralize to pH 6–7 with 1 N Sulfuric Acid. Measure the amount to add on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution caused by the volume addition.
Chlorine	Causes positive interference
Chlorine Dioxide	Causes positive interference
Chloramines, organic	May cause positive interference
Hardness	No effect at less than 1000 mg/L as $CaCO_3$
Iodine	Causes positive interference
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium, Oxidized (Cr^{6+})	Pre-treat the sample as follows: **1.** Adjust the sample pH to 6–7. **2.** Add 3 drops of Potassium Iodide (30-g/L) to 25 mL of sample. **3.** Mix and wait 1 minute. **4.** Add 3 drops of Sodium Arsenite[1] (5-g/L) and mix. **5.** Use the test procedure to measure the concentration of 10 mL of the treated sample. **6.** Subtract this result from the result without the treatment to obtain the correct bromine concentration.
Monochloramine	Causes positive interference
Ozone	Causes positive interference
Peroxides	May cause positive interference
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pre-treatment may be necessary. Adjust to pH 6–7 with acid (Sulfuric Acid, 1 N) or base (Sodium Hydroxide, 1 N). Correct the test result for the dilution caused by the volume additions.

[1] Samples that are treated with sodium arsenite will contain arsenic and may require special disposal consideration. Refer to the current MSDS/SDS for safe handling and disposal instructions.

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
50	2.80 mg/L Br_2	2.75–2.85 mg/L Br_2	0.05 mg/L Br_2
55	2.80 mg/L Br_2	2.71–2.89 mg/L Br_2	0.05 mg/L Br_2

Summary of method

Bromine residuals reacts with DPD (N,N-diethyl-p-phenylenediamine) to form a pink color which is proportional to the total bromine concentration. The measurement wavelength is 530 nm for spectrophotometers or 520 nm for colorimeters.

Consumable and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
DPD Total Chlorine Reagent Powder Pillow, 10-mL	1	100/pkg	2105669
OR			
DPD Total Chlorine Reagent AccuVac® Ampul	1	25/pkg	2503025

Required apparatus

Description	Quantity/Test	Unit	Item no.
AccuVac Snapper	1	each	2405200
Beaker, 50-mL	1	each	50041H
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Optional reagents and apparatus

Description	Unit	Item no.
AccuVac® Ampul vials for sample blanks	25/pkg	2677925
Ampule Breaker, 2-mL PourRite® Ampules	each	2484600
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Mixing cylinder, graduated, 25-mL	each	2088640
Mixing cylinder, graduated, 50-mL	each	189641
Chlorine Standard Solution, 2-mL PourRite® Ampules, 50–75 mg/L	20/pkg	1426820
Chlorine Standard Solution, 10-mL Voluette® Ampule, 50–75 mg/L	16/pkg	1426810
Chlorine Standard Solution, 2-mL PourRite® Ampules, 25–30 mg/L	20/pkg	2630020
DPD Total Chlorine Reagent Powder Pillows, 10-mL	1000/pkg	2105628
DPD Total Chlorine Reagent Powder Pillows, 10-mL	300/pkg	2105603
DPD Total Chlorine Reagent, 10-mL, SwifTest™ Dispenser refill vial	250 tests	2105660
Paper, pH, 0–14 pH range	100/pkg	2601300
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696

Optional reagents and apparatus (continued)

Description	Unit	Item no.
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	1000/pkg	2185628
Potassium Iodide, 30-g/L	100 mL	34332
Sodium Arsenite, 5-g/L	100 mL	104732
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	104532
SpecCheck™ Secondary Standard Kit, Chlorine DPD, 0–2.0 mg/L Set	each	2635300
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032
Water, Chlorine-demand Free	500 mL	2641549
Water, deionized	4 L	27256

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