DOC316.53.01029

Chlorine, Total

USEPA DPD Method¹

Method 10070

0.1 to 10.0 mg/L Cl₂ (HR)

Powder Pillows

Scope and application: For testing higher levels of total chlorine (free and combined) in drinking water, cooling water and industrial process waters.² This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.

- ¹ USEPA accepted for reporting drinking water analyses.
- ² Procedure is equivalent to USEPA and Standard Method 4500-Cl G for drinking water and wastewater.



Test preparation

Instrument specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows requirements that may vary between instruments, such as adapter and sample cell requirements. To use this table, select an instrument, then read across to find the corresponding information for this test.

Table 1 Instrument-specific information

Instrument	Adapter	Sample cell orientation	Sample cell
DR 6000	A23618	The orientation key is toward the user.	4864302
DR 5000		The orientation key is toward the user.	
DR 3900	LZV846 (A)	The orientation key is away from the user.	
DR 900	_	The orientation key is toward the user.	
DR 3800	LZV585 (B)	The 1-cm path is aligned with the arrow on the adapter.	5940506
DR 2800			
DR 2700			

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.

In bright light conditions (e.g., direct sunlight), close the cell compartment on spectrophotometers, if applicable, with the protective cover during measurements.

If the chlorine concentration is less than 2 mg/L, use Method 8167, program number 80. If the chlorine concentration is less that $500 \mu g/L$, use Method 8370 (for applicable instruments).

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used and use any recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Use the Safety Data Sheets for disposal information for unused reagents. Consult the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

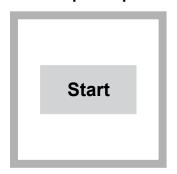
Description	Quantity
DPD Total Chlorine Reagent Powder Pillows, 25-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument specific information on page 1.)	1

Refer to Consumables and replacement items on page 5 for reorder information.

Sample collection

- Samples must be analyzed immediately and cannot be preserved for later analysis.
- Chlorine is a strong oxidizing agent and it is unstable in natural waters. It 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 chlorine in water.
- Collect samples in clean glass bottles. Avoid plastic containers since these may have a large chlorine demand.
- Pre-treat glass sample containers to remove any chlorine demand. Soak the
 containers 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 pre-treatment is necessary.
- Be 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. Then 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. If a sample cell is used, rinse the cell several times with the sample, then carefully fill to the 10-mL mark.

Powder pillow procedure



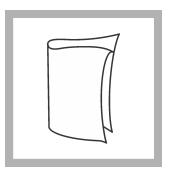
1. Start program

88 Chlorine F&T HR. For information about sample cells, adapters or light shields, refer to Instrument specific information on page 1.

Note: Although the program name may vary between instruments, the program number does not change.



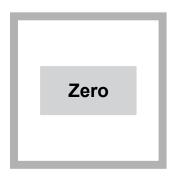
2. Prepare the blank: Fill a sample cell to the 5-mL mark with sample.



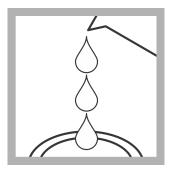
3. Clean the blank.



4. Insert the blank into the cell holder.



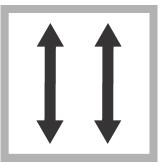
5. Push **ZERO**. The display shows 0.0 mg/L Cl₂.



6. Prepare the sample: Fill a second sample cell to the 5-mL mark with sample.



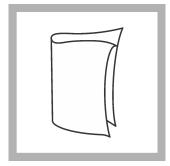
7. Add the contents of one DPD Total Chlorine Powder Pillow for 25-mL samples to the sample.



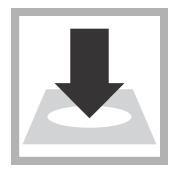
8. Close the sample cell. Shake the sample cell about 20 seconds to dissolve the reagent.



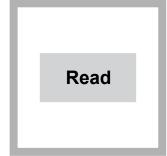
9. Start the instrument timer. A 3-minute reaction time starts.



10. Clean the sample cell.



11. When the timer expires, insert the prepared sample into the cell holder.



12. Push **READ**. Results show in mg/L Cl₂.

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. Adjust 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 from the volume addition.	
Alkalinity	More than 250 mg/L CaCO ₃ . The full color may not develop or the color may fade instantly. Adjust 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 from the volume addition.	
Bromine, Br ₂	Interferes at all levels	
Chlorine Dioxide, ClO ₂	Interferes at all levels	
Chloramines, organic	May interfere	
Hardness	No effect at less than 1000 mg/L as CaCO ₃	
Iodine, I ₂	Interferes at all levels	
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 10 mL of sample. 3. Mix and wait 1 minute. 4. Add 3 drops of Sodium Arsenite (5-g/L) and mix. 5. Use the test procedure to measure the concentration of the treated sample. 6. Subtract this result from the result without the treatment to obtain the correct chlorine concentration.	

Interfering substance	Interference level
Ozone	Interferes at all levels
Peroxides	May interfere
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pretreatment may be necessary.

Pollution prevention and waste management

If sodium arsenite was added to the sample for manganese or chromium interferences, the reacted samples will contain arsenic and must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations.

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample. Items to collect:

- Chlorine Standard Solution, 2-mL PourRite® Ampule, 50–75 mg/L (use mg/L on label)
- Ampule breaker
- Pipet, TenSette[®], 0.1–1.0 mL and tips
- 1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
- 2. Go to the Standard Additions option in the instrument menu.
- 3. Select the values for standard concentration, sample volume and spike volumes.
- 4. Open the standard solution.
- **5.** Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 5-mL portions of fresh sample. Mix well.
- **6.** Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
- 7. Select **Graph** to compare the expected results to the actual results.

Note: If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.

Method performance

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

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change	
88	5.4 mg/L Cl ₂	5.3–5.5 mg/L Cl ₂	0.1 mg/L Cl ₂	

Summary of method

The range of analysis using the DPD method for total chlorine can be extended by adding more indicator in proportion to sample volume. Thus, a larger fill powder pillow of DPD Total Chlorine Reagent is added to a 5-mL sample portion. The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N, N-diethyl-p-phenylenediamine) along with free chlorine present in the sample to form a pink color which is proportional to the total chlorine concentration. The measurement wavelength is 530 nm for spectrophotometers or 520 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
DPD Total Chlorine Reagent Powder Pillow, 25-mL	1	100/pkg	1406499

Recommended standards

Description	Unit	Item no.
Chlorine Standard Solution, 2-mL PourRite® Ampule, 50-75 mg/L	20/pkg	1426820
Chlorine Standard Solution, 10-mL Voluette® Ampule, 50–75 mg/L	16/pkg	1426810

Optional reagents and apparatus

Description	Unit	Item no.
Ampule Breaker, Voluette® ampules	each	2196800
PourRite® Ampule Breaker, 2-mL	each	2484600
Cylinder, mixing, 25-mL	each	189640
Pipet, TenSette [®] , 0.1–1.0 mL	each	1970001
Pipet tips for TenSette Pipet 1970001	50/pkg	2185696
Pipet tips for TenSette Pipet 1970001	1000/pkg	2185628
Paper, pH, 0–14 pH range	100/pkg	2601300
Potassium lodide, 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
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032
Chlorine Standard Solution, 2-mL PourRite® Ampules, 25–30 mg/L	20/pkg	2630020
DPD Total Chlorine Reagent Powder Pillows, 25-mL	1000/pkg	1406428

