Chlorine, Total

DOC316.53.01028

DPD Method[1] **Method 10101**

0.09 to 5.00 mg/L Cl_2 **Test 'N Tube™ Vials**

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

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

Test preparation

Instrument specific information

The table in this section shows all of the instruments that have the program for this test. Table 1 shows adapter and light shield requirements for the instruments that use them.

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

Table 1 Instrument-specific information for test tubes

Instrument	Adapters	Light shield
DR 6000	—	—
DR 5000	—	—
DR 900	4846400	Cover supplied with the instrument
DR 3900	—	LZV849
DR 3800	—	LZV646
DR 2800	—	

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.
DR 3900, DR 3800, DR 2800 and DR 2700: Install the light shield in Cell Compartment #2 before this test is started.
For best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to get the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.
For chloramination disinfection control, use one of the available Chloramine (Mono) methods.
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

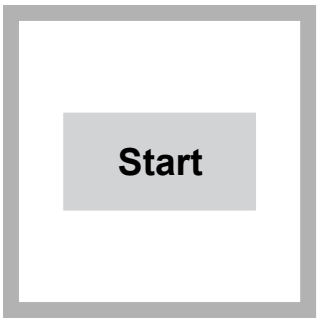
Description	Quantity
Light shield (For information about sample cells, adapters or light shields, refer to Instrument specific information on page 1.)	1
Test 'N Tube™ DPD Total Chlorine Reagent	1
Wipes, disposable	varies

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

Sample collection

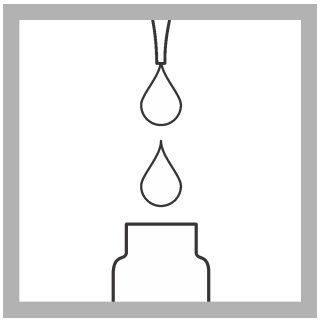
- Samples must be analyzed immediately and cannot be preserved for later analysis.
- Chlorine is a strong oxidizing agent and it is unstable in natural waters. It reacts quickly with various inorganic compounds and more slowly with organic compounds. Many factors, including reactant concentrations, sunlight, 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.

DPD method for Test N' Tubes

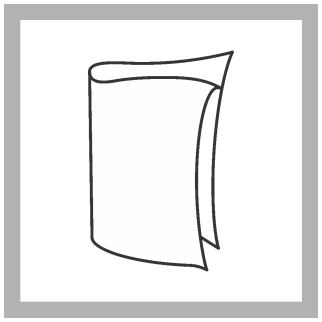


1. Start program **89 Chlorine F&T TNT**. For information about sample cells, adapters or light shields, refer to Instrument specific information on page 1.

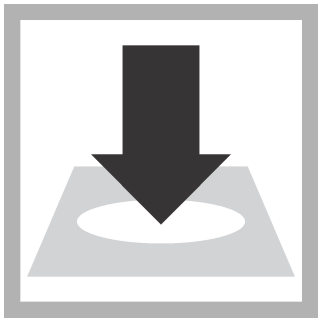
Note: *Although the program name may vary between instruments, the program number does not change.*



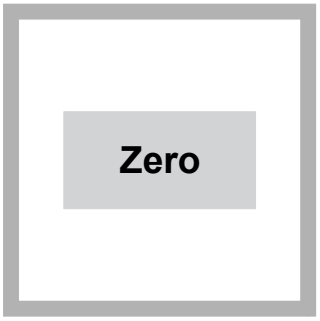
2. Prepare the blank: Fill an empty Test 'N Tube vial to the top of the label with sample.



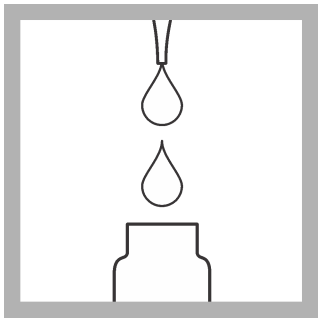
3. Clean the blank vial.



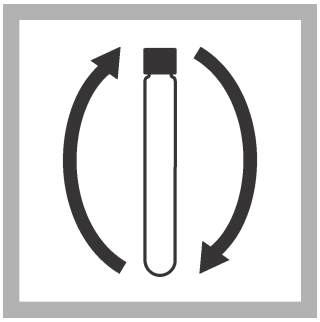
4. Insert the blank vial into the 16-mm cell holder.



5. Push **ZERO**. The display shows 0.00 mg/L Cl_2.

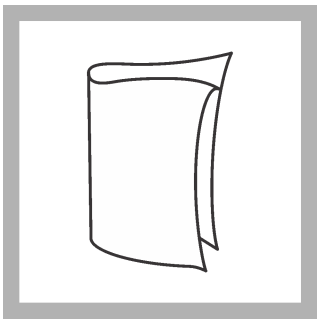


6. Prepare the sample: Remove the cap from a Total Chlorine DPD Test N' Tube. Fill the vial to the top of the label with sample.

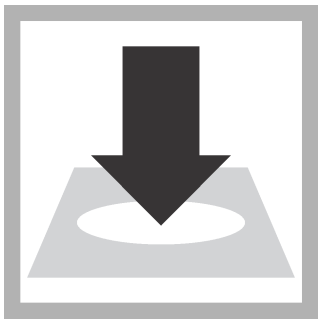


7. Put the cap on the vial. Invert to mix.

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



9. Clean the sample vial.



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

11. Push **READ**. Results show in mg/L Cl_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. 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 10-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
89	2.68 mg/L Cl_2	2.63–2.73 mg/L Cl_2	0.03 mg/L Cl_2

Summary of method

Chlorine can be present in water as free chlorine and as combined chlorine. Both forms can exist in the same water and be determined together as total 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 and free chlorine react with DPD (N,N-diethyl-p-phenylenediamine) to form a pink color which is proportional to the total chlorine concentration.

To find the concentration of combined chlorine, run a free chlorine test and a total chlorine test. Subtract the results of the free chlorine test from the total chlorine test to obtain the combined 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.
Test 'N Tube™ DPD Total Chlorine Reagent	1	50/pkg	2105645

Recommended standards and apparatus

Description	Unit	Item no.
Chlorine Standard Solution, 2-mL PourRite® Ampules, 50–75 mg/L	20/pkg	1426820
PourRite® Ampule Breaker, 2-mL	each	2484600

Optional reagents and apparatus

Description	Unit	Item no.
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
Ampule Breaker, Voluette® ampules	each	2196800
Test tube rack	each	1864100
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
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032
Chlorine Standard Solution, 10-mL Voluette® Ampule, 50–75 mg/L	16/pkg	1426810
Chlorine Standard Solution, 2-mL PourRite® Ampule, 25-30 mg/L	20/pkg	2630020

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04/2013, Edition 8