Chloride

DOC316.53.01017

Mercuric Thiocyanate Method

Method 8113

0.1 to 25.0 mg/L Cl^-

Reagent Solution

Scope and application: For 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 sample cell and orientation requirements for specific instruments.

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

Table 1 Instrument-specific information

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.	

Before starting

Filter samples that are turbid with filter paper and a funnel.
The reagents that are used in this test contain mercury. Collect the reacted samples for safe disposal.
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

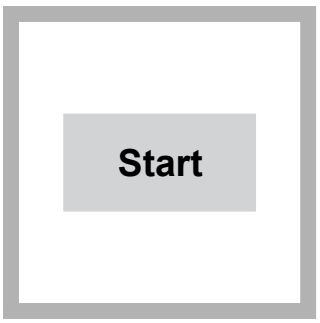
Description	Quantity
Mercuric Thiocyanate Solution	2 mL
Ferric Ion Solution	1 mL
Deionized water	10 mL
Pipet, TenSette®, 0.1–1.0 mL	1
Pipet tips, for TenSette® Pipet, 0.1–1.0 mL	2
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to Consumables and replacement items on page 4 for order information.

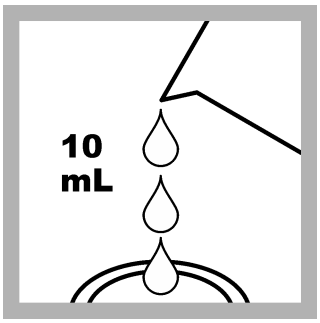
Sample collection

- Collect samples in clean glass or plastic bottles.
- If immediate analysis is not possible, keep the samples at room temperature for a maximum of 28 days.

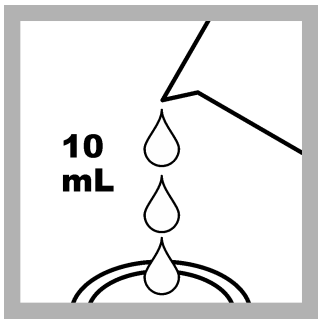
Test procedure



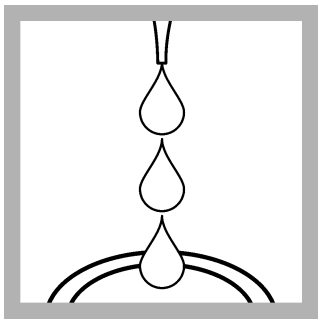
1. Start program **70 Chloride**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



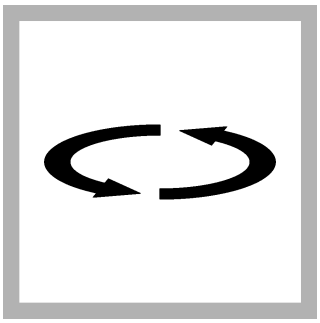
2. Prepare the blank: Fill a sample cell with 10 mL of deionized water.



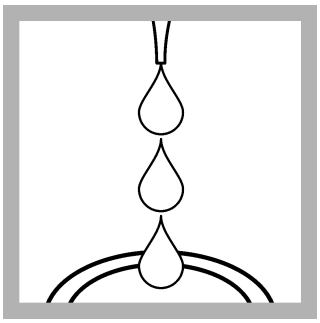
3. Prepare the sample: Fill a second sample cell with 10 mL of sample.



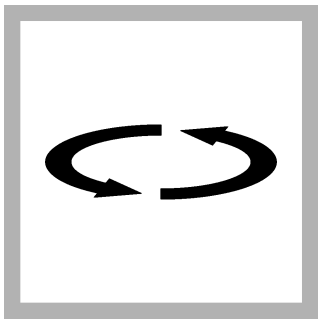
4. Use a pipet to add 0.8 mL of Mercuric Thiocyanate Solution into each sample cell.



5. Swirl to mix.

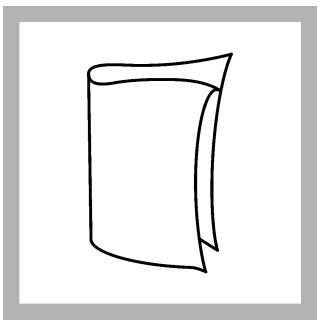


6. Use a pipet to add 0.4 mL of Ferric Ion Solution into each sample cell.

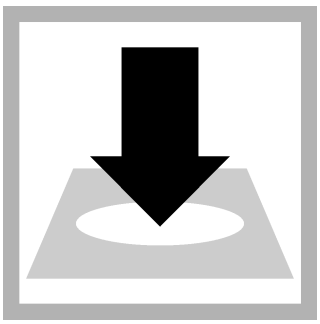


7. Swirl to mix.
The sample shows an orange color if chloride is in the sample.

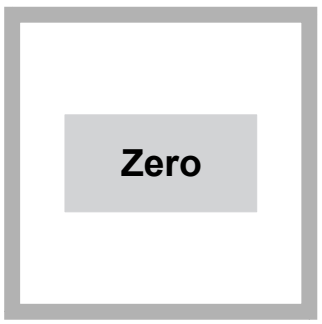
8. Start the instrument timer. A 2-minute reaction time starts.



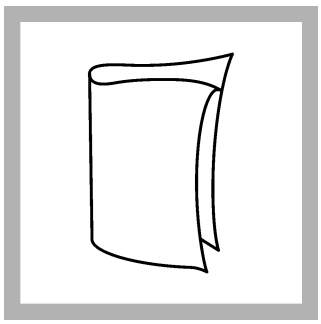
9. When the timer expires, clean the blank sample cell. Complete the remaining steps within 5 minutes.



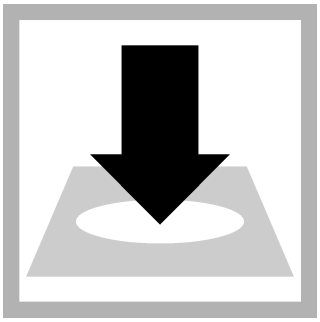
10. Insert the blank into the cell holder.



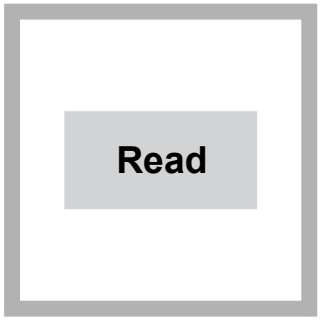
11. Push **ZERO**. The display shows 0.0 mg/L Cl^-.



12. Clean the prepared sample cell.



13. Within 5 minutes after the timer expires, insert the prepared sample into the cell holder.



14. Push **READ**. Results show in mg/L Cl^-.

Interferences

Table 2 Interfering substances

Interfering substance	Interference level
Extreme pH	The expected pH after the reagents are added is approximately 2. If the sample is strongly acidic or alkaline, adjust a portion of sample before testing to a pH of approximately 7. Use 5 N Sodium Hydroxide Standard Solution[1]. Measure the pH with pH paper because most pH electrodes will contaminate the sample with chloride.

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:

- 1000-mg/L Chloride Standard Solution
- 50-mL cylinders, graduated mixing (3)
- 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 50-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.*

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

[1] Refer to Optional reagents and apparatus on page 5.

Items to collect:

- 1000-mg/L Chloride Standard Solution
- 500-mL volumetric flask, Class A
- 10.00-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 20.0-mg/L chloride standard solution as follows:
   a. Use a pipet to add 10.00 mL of a 1000-mg/L chloride standard solution into the volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the test procedure to measure the concentration of the prepared standard solution.
3. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.*

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
70	20.0 mg/L Cl^-	17.9–22.1 mg/L Cl^-	0.1 at 1.0 mg/L, 0.3 at 10.0 mg/L, 0.6 at 20.0 mg/L Cl^-

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. The measurement wavelength is 455 nm.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Chloride Reagent Set, includes:	—	250 tests/pkg[2]	2319800
Ferric Ion Solution	1 mL	100 mL	2212242
Mercuric Thiocyanate Solution	2 mL	200 mL	2212129
Water, deionized	varies	100 mL	27242

Required apparatus

Description	Quantity/test	Unit	Item no.
Pipet, TenSette®, 0.1–1.0 mL	1	each	1970001
Pipet tips, for TenSette® Pipet, 0.1–1.0 mL	2	50/pkg	2185696

[2] The number of tests refers to any combination of samples and reagent blanks.

Recommended standards

Description	Unit	Item no.
Chloride Standard Solution, 1000-mg/L Cl^-	500 mL	18349

Optional reagents and apparatus

Description	Unit	Item no.
Mixing cylinder, graduated, 50 mL	each	2088641
Filter paper, folded, 3–5-micron, 12.5-cm	100/pkg	69257
Flask, volumetric, Class A, 500 mL, glass	each	1457449
Funnel, poly, 75 mm	each	108368
Paper, pH, 1.0–11.0	5 rolls/pkg	39133
Pipet, volumetric, Class A, 10 mL	each	1451538
Pipet filler, safety bulb	each	1465100
Safety goggles, vented	each	2550700
Sodium Hydroxide Solution, 5 N	50 mL	245026

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