Chloride

DOC316.53.01170

Mercuric Nitrate Method

Method 8206

10 to 8000 mg/L as Cl^-

Digital Titrator

Scope and application: For water, wastewater and seawater.

Test preparation

Before starting

The reagents that are used in this test contain mercury. Collect the reacted samples for safe disposal.
The optional TitraStir Titration Stand can hold the Digital Titrator and stir the sample.
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

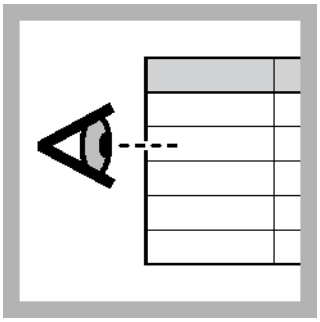
Description	Quantity
Diphenylcarbazone Indicator Powder Pillow	1
Mercuric Nitrate Titration Cartridge	1
Digital Titrator	1
Delivery tube for Digital Titrator	1
Graduated cylinder (size varies with selected sample volume), or TenSette pipet with tips	1
Erlenmeyer flask, 250-mL	1
Water, deionized	varies

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

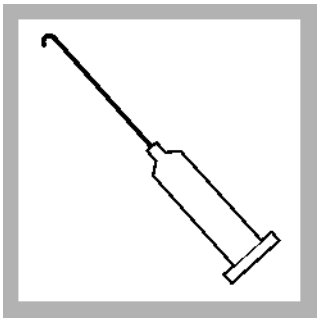
Sample collection

- Collect samples in clean glass or plastic bottles.
- If prompt analysis is not possible, keep the sample for a maximum of 7 days.

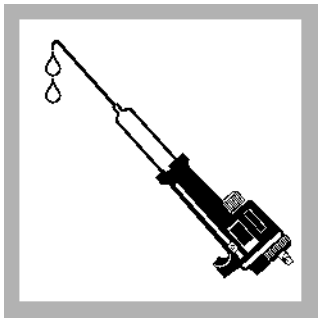
Test procedure



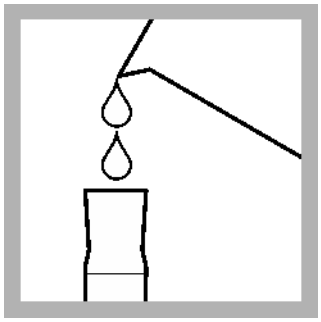
1. Select a sample volume and titration cartridge from Table 1 on page 3.



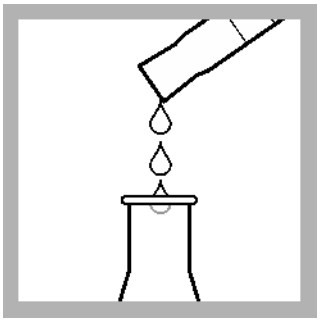
2. Insert a clean delivery tube into the digital titration cartridge. Attach the cartridge to the Digital Titrator.



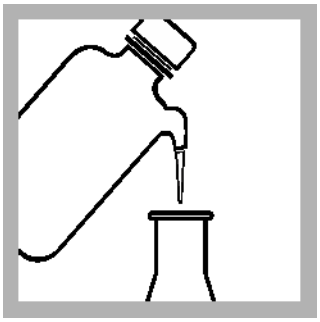
3. Hold the Digital Titrator with the cartridge tip up. Turn the delivery knob to eject air and a few drops of titrant. Reset the counter to zero and clean the tip.



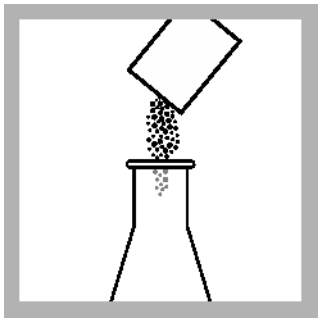
4. Use a graduated cylinder or pipet to measure the sample volume from Table 1 on page 3.



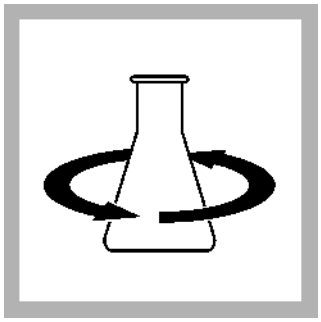
5. Pour the sample into a clean, 250-mL Erlenmeyer flask.



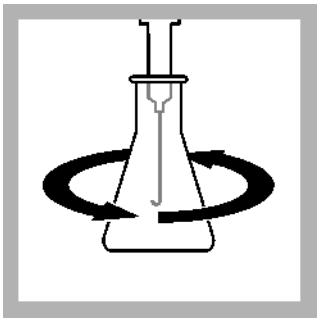
6. If the sample volume is less than 100 mL, dilute to approximately 100 mL with deionized water.



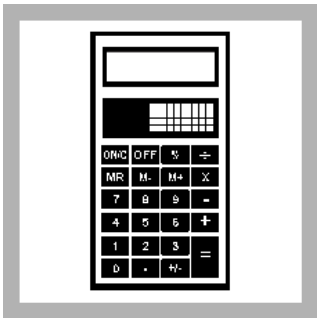
7. Add the contents of one Diphenylcarbazone Indicator Powder Pillow.



8. Swirl to mix.
A small amount of undissolved powder will not have an effect on the results.



9. Put the end of the delivery tube fully into the solution. Swirl the flask. Turn the knob on the Digital Titrator to add titrant to the solution. Continue to swirl the flask. Add titrant until the color changes from yellow to light pink. Record the number of digits on the counter.



10. Use the multiplier in Table 1 on page 3 to calculate the concentration. Digits used × digit multiplier = mg/L Cl^-.

Sample volumes and digit multipliers

Select a range in Table 1, then read across the table row to find the applicable information for this test. Use the digit multiplier to calculate the concentration in the test procedure.

Example: A 100-mL sample was titrated with the 0.2256 N Mercuric Nitrate Titration Cartridge and the counter showed 250 digits at the endpoint. The concentration is 250 digits x 0.1 = 25 mg/L Cl^-.

Table 1 Sample volumes and digit multipliers

Range (mg/L as Cl^-)	Sample volume (mL)	Titration cartridge	Digit multiplier
10–40	100	0.2256 N $Hg(NO_3)_2$	0.1
40–160	25	0.2256 N $Hg(NO_3)_2$	0.4
100–400	100	2.256 N $Hg(NO_3)_2$	1.0
200–800	50	2.256 N $Hg(NO_3)_2$	2.0
500–2000	20	2.256 N $Hg(NO_3)_2$	5.0
1000–4000	10	2.256 N $Hg(NO_3)_2$	10.0
2000–8000	5	2.256 N $Hg(NO_3)_2$	20.0

Conversions

To change the units or chemical form of the test result, multiply the test result by the factor in Table 2.

Table 2 Conversions

mg/L chloride (Cl^-) to...	multiply by...	Example
mg/L sodium chloride (NaCl)	1.65	1000 mg/L chloride x 1.65 = 1650 mg/L NaCl
meq/L chloride (Cl^-)	0.02821	1000 mg/L chloride x 0.02821 = 28.21 meq/L Cl^-

Interferences

Interfering substance	Interference level
Bromide	Interferes directly and is included in the test result.
Chromate	Concentrations that are more than 10 mg/L interfere with this method.
Iodide	Interferes directly and is included in the test result.
Iron, ferric	Concentrations that are more than 10 mg/L interfere with this method.
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment (of the sample) by the reagents. Sample pretreatment may be necessary. Adjust strongly alkaline or acidic samples to a pH of 2 to 7 with 5.25 N sulfuric acid or 5.0 N sodium hydroxide. Do not use a pH meter directly for the pH adjustment because the pH electrode will contaminate the sample. Collect a separate sample to find the correct quantity of acid or base to add. Then, add the same quantity of acid or base to the sample that is used in the test procedure.
Sulfide	Remove sulfide interference as follows: **1.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow to approximately 125 mL of sample. **2.** Mix for 1 minute. **3.** Pour the solution through folded filter paper in a funnel. **4.** Use the filtered sample in the chloride test procedure.
Sulfite	Concentrations that are more than 10 mg/L interfere with this method. To remove sulfite interference, add 3 drops of 30% Hydrogen Peroxide to the sample, then start the test.

Accuracy check

Standard additions method (sample spike)

Use the standard additions method to validate the test procedure, reagents, apparatus, technique and to find if there is an interference in the sample.

Items to collect:

- Chloride Voluette Ampule Standard Solution, 12,500-mg/L Cl^-
- Ampule Breaker
- Pipet, TenSette, 0.1–1.0 mL and pipet tips

1. Use the test procedure to measure the concentration of the sample.
2. Use a TenSette pipet to add 0.1 mL of the standard solution to the titrated sample.
3. Titrate the spiked sample to the endpoint. Record the number of digits on the counter.
4. Add one more 0.1-mL addition of the standard solution to the titrated sample.
5. Titrate the spiked sample to the endpoint. Record the number of digits on the counter.
6. Add one more 0.1-mL addition of the standard solution to the titrated sample.
7. Titrate the spiked sample to the endpoint. Record the number of digits on the counter.
8. Compare the actual result to the correct result. The correct result for this titration is 125 digits of the 0.2256 N Mercuric Nitrate Titration Cartridge or 12.5 digits of the 2.256 N Mercuric Nitrate Titration Cartridge for each 0.1-mL addition of the standard solution. If much more or less titrant was used, there can be a problem with user technique, reagents, apparatus or an interference.

Summary of method

Mercuric ions in the titrant react with chloride in the sample to form mercuric chloride. After all of the chloride is in the form of mercuric chloride, the mercuric ions react with diphenylcarbazone indicator to form a pink-purple complex, which shows the endpoint of the titration. The reagent powder contains the indicator and a buffer.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Chloride Reagent Set (approximately 100 tests)	—	each	2272600
Diphenylcarbazone Indicator Powder Pillows	1	100/pkg	83699
Mercuric Nitrate Titration Cartridge, 0.2256 N	varies	each	1439301
Mercuric Nitrate Titration Cartridge, 2.256 N	varies	each	92101
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	Item no.
Graduated cylinders—Select one or more for the sample volume:			
Cylinder, graduated, 5-mL	1	each	50837
Cylinder, graduated, 10-mL	1	each	50838
Cylinder, graduated, 25-mL	1	each	50840
Cylinder, graduated, 50-mL	1	each	50841
Cylinder, graduated, 100-mL	1	each	50842
Digital Titrator	1	each	1690001

Required apparatus (continued)

Description	Quantity/test	Unit	Item no.
Delivery tube for Digital Titrator, J-hook tip	1	5/pkg	1720500
Flask, Erlenmeyer, 250-mL	1	each	50546

Recommended standards

Description	Unit	Item no.
Chloride Standard Solution, 12,500 mg/L as Cl^-, 10-mL Voluette ampules	16/pkg	1425010

Optional reagents and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Filter paper, folded, 3–5-micron, 12.5-cm	100/pkg	69257
Funnel, poly, 65-mm	each	108367
Hydrogen Peroxide Solution, 30%, ACS	473 mL	14411
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Stir bar, octagonal	each	2095352
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	241899
Sulfuric Acid Standard Solution, 5.25 N	100 mL	244932
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
TitraStir Titration Stand, 115 VAC	each	1940000
TitraStir Titration Stand, 230 VAC	each	1940010
Delivery tube for Digital Titrator, 90-degree bend for use with TitraStir Titration Stand	5/pkg	4157800

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