Chloride DOC316.53.01171

Silver Nitrate Method

Method 8207

10 to 10,000 mg/L as CI-

Digital Titrator

Scope and Application: For water, wastewater and seawater.



Test preparation

Before starting the test:

mg/L sodium chloride = mg/L chloride x 1.65

meq/L chloride = mg/L chloride / 35.45

For added convenience when stirring, use the TitraStir® stirring apparatus.

Collect the following items:

Description	Quantity
Chloride 2 Indicator Powder Pillow	1 pillow
Silver Nitrate titration cartridge (see Range-specific information)	1 cartridge
Digital titrator	1
Delivery tube for digital titrator	1
Graduated cylinder	1
Erlenmeyer flask, 250-mL	1

See Consumables and replacement items for reorder information.

Chloride



1. Select a sample volume and titration cartridge from the *Range-specific information* table.



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator.



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



4. Use a graduated cylinder or pipet to measure the sample volume from the *Rangespecific information* table into a 250 mL Erlenmeyer flask.

Chloride (continued)



5. Transfer the sample into a clean, 250-mL Erlenmeyer flask. If the sample volume is less than 100 mL, dilute to approximately 100 mL with deionized water.



6. Add the contents of one Chloride 2 Indicator Powder Pillow. Swirl to mix.

Results will still be accurate if a small amount of powder does not dissolve.



7. Place the delivery tube into the solution and swirl the flask. Turn the knob on the titrator to add titrant to the solution. Continue to swirl the flask and add titrant until the color changes from yellow to red-brown.

Write down the number of digits displayed on the counter.



8. Use the multiplier in the *Range-specific information* table to calculate the concentration:

digits x multiplier = mg/L Cl⁻

Example: 100 mL of sample was titrated with the 0.2256 N cartridge and 250 digits were used to reach the endpoint. The concentration is 250 x 0.1 = 25 mg/L CI

Table 85 Range-specific information

Range (mg/L as CI ⁻)	Sample volume (mL)	Titration cartridge (N AgNO ₃)	Multiplier
10–40	100	0.2256	0.1
25–100	40	0.2256	0.25
100–400	50	1.128	1.0
250–1000	20	1.128	2.5
1000–4000	5	1.128	10.0
2500-10,000	2	1.128	25.0

Sample collection, preservation and storage

Collect samples in clean plastic or glass bottles. The sample can be stored for up to 7 days before the analysis.

Interferences

Interfering substances lists substances that can interfere with this test.

Table 86 Interfering substances

Interfering substance	Interference level
Bromide	Interferes directly and is included in the test result.
Cyanide	Interferes directly and is included in the test result.
Iron	Concentrations above 10 mg/L mask the end point.

Table 86 Interfering substances (continued)

Interfering substance	Interference level
Iodide	Interferes directly and is included in the test result.
Orthophosphate	Concentrations above 25 mg/L will precipitate the silver.
рН	Neutralize strongly alkaline or acidic samples to a pH of 2 to 7 with 5.25 N sulfuric acid or 5.0 N sodium hydroxide. If a pH meter is used in the pH adjustment, use a separate sample to find the correct amount of acid or base to use. Then add the same amount of acid or base to the sample to be tested. pH electrodes will contaminate the sample.
Sulfide	Complete the following steps to remove sulfide interference: 1. Add the contents of one Sulfide Inhibitor Reagent Powder Pillow to approximately 125 mL of sample. 2. Mix for one minute. 3. Filter through folded filter paper. 4. Use the filtered sample in the chloride test procedure.
Sulfite	Concentrations above 10 mg/L interfere with this method. Eliminate sulfite interference by adding three drops of Hydrogen Peroxide, 30%, to the sample before the test is started.

Accuracy check

Use the standard additions method to determine whether the sample has an interference and confirm analytical technique.

Standard additions method (sample spike)

Required for accuracy check:

- Chloride Voluette® Ampule Standard Solution, 12,500-mg/L Cl-
- · Ampule breaker
- TenSette Pipet, 0.1–1.0 mL
- 1. Open the standard solution ampule.
- 2. Use the TenSette Pipet to add 0.1 mL of the standard to the titrated sample. Swirl to mix.
- 3. Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
- 4. Use the TenSette Pipet to add 0.2 mL of standard to the titrated sample. Swirl to mix.
- 5. Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
- 6. Use the TenSette Pipet to add 0.3 mL of standard to the titrated sample. Swirl to mix.
- Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
- Each 0.1 mL of standard that was added will use approximately 12.5 digits of the 2.256 N titration cartridge or 25 digits of the 1.128 N titration cartridge to reach the endpoint.

If more or less titrant was used, the problem can be due to user technique, an interference (see *Interferences*) or a problem with reagents or apparatus.

Summary of method

The sample is titrated with Silver Nitrate Standard Solution in the presence of potassium chromate (from the Chloride 2 Indicator Powder). The silver nitrate reacts with the chloride present to produce insoluble white silver chloride. After all the chloride has been precipitated, the silver ions react with the excess chromate present to form a red-brown silver chromate precipitate, marking the end point of the titration.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Chloride Reagent Set (approximately 100 tests):	<u> </u>		2288000
(2) Chloride 2 Indicator Powder Pillows	1 pillow	50/pkg	105766
(1) Silver Nitrate Titration Cartridge, 0.2256 N	varies	each	1439601
(1) Silver Nitrate Titration Cartridge, 1.128 N	varies	each	1439701

Required apparatus

Description	Quantity/Test	Unit	Catalog number
Digital Titrator	1	each	1690001
Flask, Erlenmeyer, graduated, 250-mL	1	each	50546
Graduated cylinder—select one or more based on range:			
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
Delivery tubes w/ 180° hook	1	each	1720500
Delivery tubes w/ 90° hook	1	each	4157800

Recommended standards

Description	Unit	Catalog number
Chloride Standard Solution, Voluette® Ampule, 12,500-mg/L Cl⁻, 10-mL	16/pkg	1425010
Voluette breaker	_	2196800

Optional reagents and apparatus

Description	Unit	Catalog number
Filter paper, 12.5 cm	100/pkg	69257
Funnel, analytical, poly, 65 mm	each	108367
Hydrogen Peroxide, 30%, ACS	473 mL	14411
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Stir bar, octagonal 28.6 mm x 7.9 mm	each	2095352
Sulfide Inhibitor Reagent Powder Pillow	100/pkg	241899
Sulfuric Acid Standard Solution, 5.25 N	100 mL MDB	244932
TenSette Pipet, 0.1 to 1.0 mL	each	1970001
TitraStir Stir Plate, 115 VAC	each	1940000
TitraStir Stir Plate, 230 VAC	each	1940010
Water, deionized	500 mL	27249
pH Test Strip, 0–14 pH	100/pkg	2601300
Pipet tips	100/pkg	2185628
Pipet tips	50/pkg	2185696
Chloride standard solution, 1000 mg/L	500 mL	18349
Sampling bottle	250 mL	2087076
Dropper, glass	5/pkg	1419705

