Chlorine, Total

DOC316.53.01154

Iodometric Method Using Sodium Thiosulfate[1]

Method 8161

0–40,000 mg/L as Cl_2

Buret Titration

Scope and application: For water and seawater.

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*, 4500-Cl B.

Test preparation

Before starting

Percent (%) chlorine = mg/L ÷ 10,000
Use these test procedures to determine the iodine or bromine concentration in a sample if chlorine is not in the sample. Refer to Conversions on page 4.
For higher concentrations, refer to the hypochlorite procedure Method 10100.
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
Dissolved Oxygen 3 Powder Pillow	1
Potassium Iodide Powder Pillow	1
Sodium Thiosulfate Standard Solution (use a concentration that is applicable to the selected sample volume)	varies
Starch Indicator Solution	1 mL
Buret, Class A, 25 mL	1
Graduated cylinder (use a size that is applicable to the selected sample volume), or TenSette pipet with tips	1
Erlenmeyer flask, 250 mL	1
Funnel, micro	1
Support stand with buret clamp	1
Water, deionized	varies

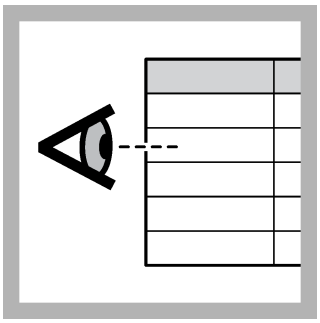
Refer to Consumables and replacement items on page 4 for order information.

Sample collection

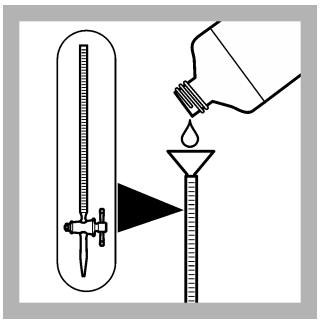
- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Chlorine is a strong oxidizing agent and is unstable in natural waters. Chlorine reacts quickly with various inorganic compounds and more slowly with organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence the decomposition of chlorine in water.
- Collect samples in clean glass bottles. Do not use plastic containers because these can have a large chlorine demand.

- Pretreat glass sample containers to remove chlorine demand. Soak the containers in a weak bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse fully with deionized or distilled water. If sample containers are rinsed fully with deionized or distilled water after use, only occasional pretreatment is necessary.
- Make 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. 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.

Test procedure

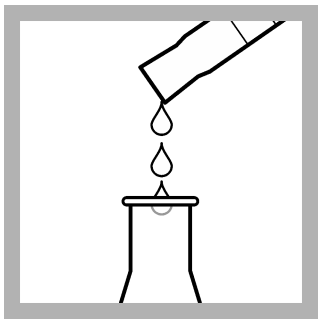


1. Select a sample volume and titrant from Table 1 on page 3.

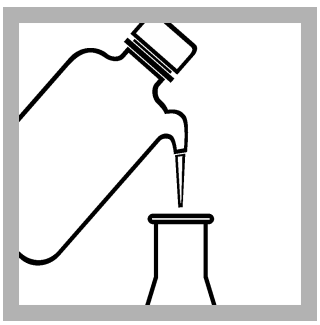


2. Fill a 25-mL buret to the zero mark with the titrant.

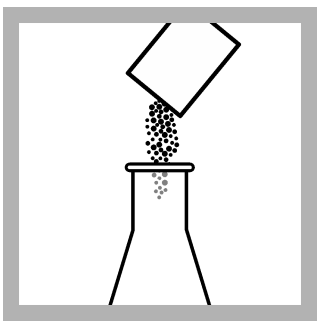
3. Use a graduated cylinder or pipet[1] to measure the sample volume from Table 1 on page 3.



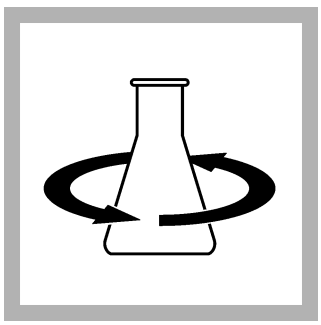
4. Pour the sample into a clean, 250-mL Erlenmeyer flask.



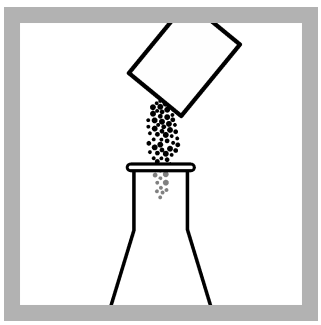
5. If the sample volume is less than 50 mL, dilute to approximately 50 mL with deionized water.



6. Add the contents of one Dissolved Oxygen 3 Powder Pillow.

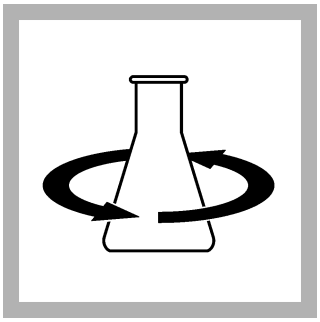


7. Swirl to mix.
The addition of the powder pillow decreases the pH to 4 or less. If the sample has a high alkaline level, make sure that the solution pH is 4 or less before the next step.

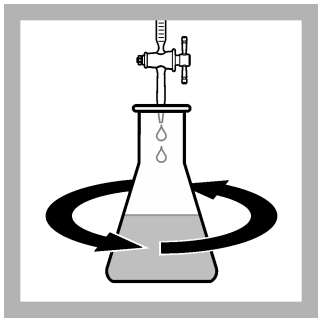


8. Add the contents of one Potassium Iodide Powder Pillow.

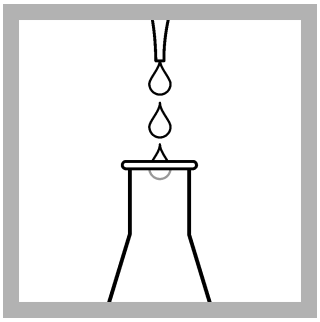
[1] Titration accuracy has a direct relation to the accuracy of the sample volume measurement. For smaller volumes, it is recommended to use a pipet to increase accuracy.



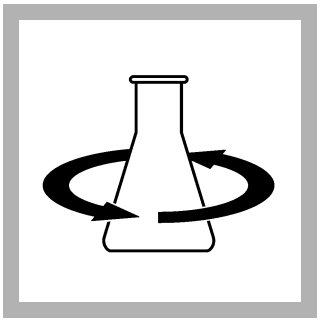
9. Swirl to mix.



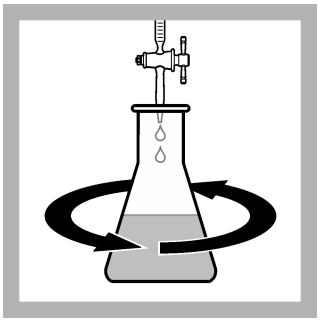
10. Put the flask under the buret. Swirl the flask. Add titrant until the color changes to pale yellow.



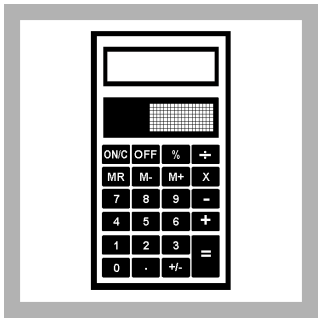
11. Add 1 mL of Starch Indicator Solution.



12. Swirl to mix.
The color of the solution changes to dark blue.



13. Put the flask under the buret. Swirl the flask. Add titrant until the color changes from dark blue to colorless.



14. Use the multiplier in Table 1 on page 3 to calculate the concentration. mL of titrant × multiplier = mg/L total chlorine as Cl_2.

Sample volumes and multipliers

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

Example: A 100-mL sample was titrated with 0.10 N titrant and 12 mL of titrant was used at the endpoint. The concentration is 12 mL × 35.5 = 426 mg/L total chlorine as Cl_2.

Table 1 Sample volumes and multipliers

Range (mg/L)	Sample volume (mL)	Titrant—sodium thiosulfate	Multiplier
0–200	100	0.025 N	8.87
100–400	50	0.025 N	17.7
200–800	100	0.10 N	35.5
400–1600	50	0.10 N	70.9
1000–4000	20	0.10 N	177
2000–8000	10	0.10 N	355
5000–20,000	4	0.10 N	887
10,000–40,000	2	0.10 N	1773

Interferences

Interfering substance	Interference level
Oxidizing agents	Oxidized manganese and other oxidizing agents causes positive interference.
Reducing agents	Reducing agents, such as organic sulfides, can interfere.

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 chlorine (Cl_2) to...	multiply results by...	Example
mg/L iodine	3.58	mg/L chlorine × 3.58 = mg/L iodine
mg/L bromine	2.25	mg/L chlorine × 2.25 = mg/L bromine

Summary of method

Total chlorine concentration is equal to the concentration of the free and the combined forms of chlorine. Free chlorine reacts with ammonia to form combined chlorine such as monochloramines. When potassium iodide is added to a sample that has chlorine at an acidic pH, free iodine is released in direct proportion to the amount of total chlorine in the sample. Then, the iodine is titrated with sodium thiosulfate to a colorless endpoint.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Dissolved Oxygen 3 Powder Pillows	1	100/pkg	98799
Potassium Iodide Powder Pillows	1	100/pkg	107799
Sodium Thiosulfate Standard Solution, 0.025 N	varies	1 L	35253
Sodium Thiosulfate Standard Solution, 0.10 N	varies	1 L	32353
Starch Indicator Solution	1 mL	100 mL MDB	34932
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	Item no.
Buret clamp, double	1	each	32800
Buret, Class A, 25 mL	1	each	2636540
Support stand	1	each	56300
Funnel, micro	1	each	2584335
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
Tensette® pipets and pipet tips—Select one or more for the sample volume:			
Pipet, TenSette®, 0.1–1.0 mL	1	each	1970001

Required apparatus (continued)

Description	Quantity/test	Unit	Item no.
Pipet tips, TenSette® Pipet, 0.1–1.0 mL	varies	50/pkg	2185696
Pipet, TenSette®, 1.0–10.0 mL	1	each	1970010
Pipet tips, TenSette® Pipet, 1.0–10.0 mL	varies	50/pkg	2199796
Flask, Erlenmeyer, 250 mL	1	each	50546

Optional apparatus

Description	Unit	Item no.
Clippers	each	96800
Stir bar, octagonal	each	2095352
TitraStir® Titration Stand, 115 VAC	each	1940000
TitraStir® Titration Stand, 230 VAC	each	1940010

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