

Chloride, colorimetric, ferric thiocyanate

Parameter and Code:

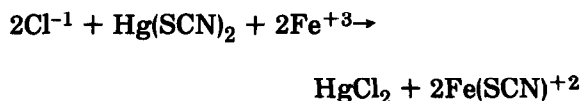
Chloride, dissolved, I-1187-85 (mg/L as Cl): 00940

1. Application

This method may be used to determine dissolved chloride in water containing from 0.1 to 10 mg/L of chloride ion. It is particularly useful for the analysis of low-dissolved- solids-content water when low chloride concentrations must be determined accurately.

2. Summary of method

2.1 Chloride is determined by measurement of the color developed by the displacement of the thiocyanate ion from mercuric thiocyanate by chloride ion in the presence of ferric ion; an intensely colored ferric thiocyanate complex is formed:



2.2 The color is stable for at least 2 h and is proportional to the chloride-ion concentration. The color has a maximum absorbance at 460 nm.

2.3 For additional information see ASTM Method D 512-81, "Standard Methods of Testing for Chloride Ion in Water" (American Society for Testing and Materials, 1984).

3. Interferences

Bromide, iodide, cyanide, thiosulfate, and nitrite interfere. Color, depending upon its spectral absorbance, may interfere with the photometric measurement.

4. Apparatus

- 4.1 *Spectrometer* for use at 460 nm.
- 4.2 Refer to manufacturer's manual to optimize instrument.

5. Reagents

5.1 *Chloride standard solution I*, 1.00 mL = 1.00 mg Cl^{-1} : Dissolve 1.648 g primary standard NaCl crystals, dried at 180°C for 1 h, in demineralized water and dilute to 1,000 mL.

5.2 *Chloride standard solution II*, 1.00 mL = 0.010 mg Cl^{-1} : Dilute 5.0 mL chloride standard solution I to 500.0 mL with demineralized water.

5.3 *Ferric ammonium sulfate solution*, 22.8 g/L: Dissolve 41.4 g $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ in 570 mL concentrated HNO_3 (sp gr 1.41) and dilute to 1 L with demineralized water.

5.4 *Mercuric thiocyanate solution*, 3 g/L: Dissolve 3 g $\text{Hg}(\text{SCN})_2$ in 1 L 95-percent ethanol (denatured alcohol formula No. 3A is also satisfactory). Stir for 1 h to saturate the solvent; allow undissolved thiocyanate to settle, and then filter through a Pyrex-wool plug or a 0.45- μm membrane filter.

6. Procedure

6.1 Pipet a volume of sample containing less than 0.250 mg of Cl^{-1} (25.0 mL max) into a 50-mL beaker and adjust the volume to 25.0 mL with demineralized water.

6.2 Prepare a demineralized-water blank and at least five standards containing from 0.0025 to 0.250 mg Cl^{-1} , and adjust the volume of each to 25.0 mL.

6.3 Add 2.0 mL $\text{FeNH}_4(\text{SO}_4)_2$ solution and stir. The samples will be essentially colorless at this point.

6.4 Add 2.0 mL $\text{Hg}(\text{SCN})_2$ solution and stir.

6.5 After at least 10 min, but within 2 h, read the absorbance of each standard and sample against the blank at 460 nm, and, when necessary, make corrections for water color.

7. Calculations

7.1 Determine the milligrams chloride from a plot of absorbances of standards containing known amounts of Cl^{-1} .

7.2 Determine the chloride concentration in milligrams per liter as follows:

$$\text{Cl}^{-1} \text{ (mg/L)} = \frac{1,000}{\text{mL sample}} \times \text{mgCl}^{-1}$$

8. Report

Report chloride, dissolved (00940), concentrations of less than 10 mg/L to the nearest 0.1 mg/L.

9. Precision

9.1 Data published by the American Society for Testing and Materials (1984) indicate the overall precision of the method to be

$$S_T = 0.054X$$

where

S_T = overall precision, milligrams per liter, and

X = concentration of Cl^{-1} , milligrams per liter.

9.2 Precision for one reference sample expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (mg/L)	Relative standard deviation (percent)
6	1.4	33

Reference

American Society for Testing and Materials, 1984, Annual book of ASTM standards, section 11, water: Philadelphia, v. 11.01, p. 392-400.