

Methodology



Chloride by Flow Injection Analysis (FIA)

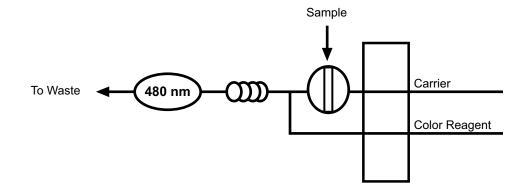
(Cartridge Part #A002601)

1.0 Scope and Application

- 1.1 This method is used for the determination of chloride in drinking water, surface water, saline water, and domestic and industrial wastes.
- 1.2 The Method Detection Limit (MDL) of this method is 0.31 mg/L chloride. The applicable range of the method is 1.0–200 mg/L chloride. The range may be extended to analyze higher concentrations by sample dilution.

2.0 Summary of Method

- 2.1 Chloride reacts with mercuric thiocyanate, liberating thiocyanate ion by the formation of soluble mercuric chloride. In the presence of ferric ion, free thiocyanate ion forms a highly colored ferric thiocyanate complex. The colored complex is measured at 480 nm (References 15.1 and 15.3).
- 2.2 The quality of the analysis is assured through reproducible calibration and testing of the Flow Injection Analysis (FIA) system.
- 2.3 A general flow diagram of the FIA system is shown below (see Section 17.0 for a detailed flow diagram).



3.0 Definitions

Definitions for terms used in this method are provided in Section 16.0, "Glossary of Definitions and Purposes."

4.0 Interferences

- 4.1 There are no significant chemical interferences for this method.
- 4.2 Filter turbid samples prior to analysis.

5.0 Safety

- 5.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been fully established. Each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level.
- 5.2 For reference purposes, a file of Material Safety Data Sheets (MSDS) for each chemical used in this method should be available to all personnel involved in this chemical analysis. The preparation of a formal safety plan is also advisable.
- 5.3 The following chemicals used in this method may be highly toxic or hazardous and should be handled with extreme caution at all times. Consult the appropriate MSDS before handling.
 - 5.3.1 Ferric Nitrate Nonahydrate, Fe(NO₂)₂•9H₂O (FW 404.00)
 - 5.3.2 Mercuric Thiocyanate, Hg(SCN)₂ (FW 316.75)
 - 5.3.3 Methanol, CH₂OH (FW 32.04)
 - 5.3.4 Nitric Acid, concentrated, HNO₂ (FW 63.01)
 - 5.3.5 Sodium Chloride, NaCl (FW 58.44)
 - 5.3.6 Thioacetamide, CH₂CSNH₂ (FW 75.13)
- 5.4 Unknown samples may be potentially hazardous and should be handled with extreme caution at all times.
- 5.5 Proper personal protective equipment (PPE) should be used when handling or working in the presence of chemicals.
- 5.6 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6.0 Apparatus, Equipment, and Supplies

- 6.1 Flow Injection Analysis (FIA) System (OI Analytical Flow Solution® IV) consisting of the following:
 - 6.1.1 Model 502 Multichannel Peristaltic Pump
 - 6.1.2 Random Access (RA) Autosampler
 - 6.1.3 Expanded Range (ER) Photometric Detector with 5-mm path length flowcell and 480-nm optical filter
 - 6.1.4 Data Acquisition System (PC or Notebook PC) with WinFLOW™ software
 - 6.1.5 Chloride by FIA Cartridge (Part #A002601)
 - 6.1.6 For FIA, Flow Solution IV must be equipped with the FIA option.
- 6.2 Sampling equipment—Sample bottle, amber glass, with polytetrafluoroethylene (PTFE)-lined cap. Clean by washing with detergent and water, rinsing with two aliquots of reagent water, and drying by baking at 110°-150°C for a minimum of one hour.
- 6.3 Standard laboratory equipment including volumetric flasks, pipettes, syringes, etc. should all be cleaned, rinsed, and dried per bottle cleaning procedure in Section 6.2.

7.0 Reagents and Calibrants

- 7.1 Raw Materials
 - 7.1.1 Brij®-35, 30% w/v (Part #A21-0110-33)
 - 7.1.2 Deionized Water (ASTM Type I or II)
 - 7.1.3 Ferric Nitrate Nonahydrate, Fe(NO₂)₂•9H₂O (FW 404.00)
 - 7.1.4 Mercuric Thiocyanate, Hg(SCN), (FW 316.75)
 - 7.1.5 Methanol, CH₃OH (FW 32.04)
 - 7.1.6 Nitric Acid, concentrated, HNO₃ (FW 63.01)
 - 7.1.7 Sodium Chloride, NaCl (FW 58.44)
 - 7.1.8 Thioacetamide, CH₂CSNH₂ (FW 75.13)