

Methodology



Ammonia Nitrogen, USEPA by Flow Injection Analysis (FIA)

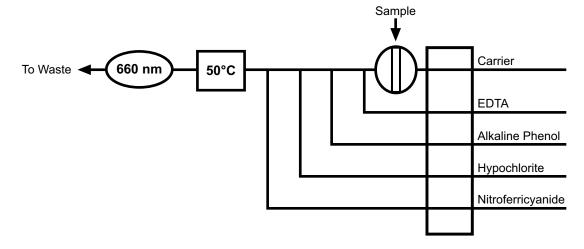
(Cartridge Part #319800)

1.0 Scope and Application

- 1.1 This method is used for the determination of ammonia in drinking water, surface water, and domestic and industrial wastes, according to USEPA Method 350.1 (Reference 15.4).
- 1.2 The Method Detection Limit (MDL) of this method is 0.002 mg/L ammonia as nitrogen (N). The applicable range of the method is 0.01–25 mg/L ammonia as nitrogen. The range may be extended to analyze higher concentrations by sample dilution.

2.0 Summary of Method

- 2.1 Ammonia reacts with alkaline phenol and hypochlorite to form indophenol blue in an amount that is proportional to the ammonia concentration. The blue color is intensified with sodium nitroferricyanide. The absorbance is measured at 660 nm (Reference 15.5).
- 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 Eliminate precipitation of calcium and magnesium hydroxides by adding ethylenediaminetetraacetic acid (EDTA).
- 4.2 Filter turbid samples prior to analysis.
- 4.3 Samples with background absorbance at the analytical wavelength may interfere (References 15.5 and 15.7).
- 4.4 Color intensity is sensitive to pH. Standardize samples at pH 5–7 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 Ammonium Sulfate, (NH₄)₂SO₄ (FW 132.14)
 - 5.3.2 Chloroform, CHCl₂ (FW 119.38)
 - 5.3.3 Ethylenediaminetetraacetic Acid, Disodium Salt Dihydrate (EDTA), C₁₀H₁₆N₂Na₂O₈•2H₂O (FW 372.24)
 - 5.3.4 KleenflowTM Acidic (Part #A001251)
 - 5.3.5 Kleenflow Basic (Part #A001252)
 - 5.3.6 Phenol, solid or liquified, 88%, C₅H₅OH (FW 94.11)
 - 5.3.7 Sodium Hydroxide, NaOH (FW 40.00)
 - 5.3.8 Sodium Hypochlorite, 5.25% available chlorine (household bleach), NaOCl (FW 74.44)
 - 5.3.9 Sodium Nitroferricyanide Dihydrate, Na₂Fe(CN)₅NO•2H₂O (FW 297.95)

- 5.3.10 Sulfuric Acid, concentrated, H₂SO₄ (FW 98.08)
- 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 660-nm optical filter
 - 6.1.4 Data Acquisition System (PC or Notebook PC) with WinFLOW™ software
 - 6.1.5 Ammonia Nitrogen/TKN, USEPA Cartridge (Part #319800)
 - 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 Ammonium Sulfate, $(NH_A)_2SO_A$ (FW 132.14)
 - 7.1.2 Brij®-35, 30% w/v (Part #A21-0110-33)
 - 7.1.3 Chloroform, CHCl₂ (FW 119.38)
 - 7.1.4 Deionized Water (ASTM Type I or II)

Part #319435 Flow Solution IV

- 7.1.5 Ethylenediaminetetraacetic Acid, Disodium Salt Dihydrate (EDTA), C₁₀H₁₆N₂Na₂O₈•2H₂O (FW 372.24)
- 7.1.6 Kleenflow Acidic (Part #A001251)
- 7.1.7 Kleenflow Basic (Part #A001252)
- 7.1.8 Phenol, solid or liquified, 88%, C₆H₅OH (FW 94.11)
- 7.1.9 Sodium Hydroxide, NaOH (FW 40.00)
- 7.1.10 Sodium Hypochlorite, 5.25% available chlorine (household bleach), NaOCl (FW 74.44)
- 7.1.11 Sodium Nitroferricyanide Dihydrate, Na₂Fe(CN)₅NO•2H₂O (FW 297.95)
- 7.1.12 Sulfuric Acid, H₂SO₄ (FW 98.08)