O·I·Analytical

Methodology

Total Dissolved Nitrogen by Online UV/Persulfate Digestion, Segmented Flow Analysis (SFA), and Flow Injection Analysis (FIA)

(Cartridge Part #319784)

1.0 Scope and Application

1.1 This method is used for the determination of total dissolved nitrogen in drinking water, surface water, saline water, and domestic and industrial wastes.

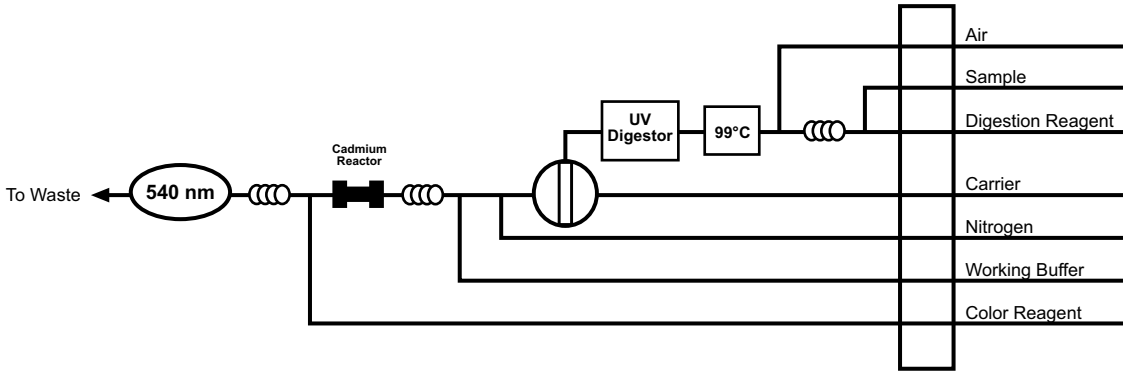
1.2 The Method Detection Limit (MDL) of this method is 0.02 mg/L nitrogen (N). The applicable range of the method is 0.10–10 mg/L nitrogen. The range may be extended to analyze higher concentrations by sample dilution.

2.0 Summary of Method

2.1 Dissolved nitrogen compounds are converted to nitrate by persulfate oxidation and subsequent UV digestion in a caustic solution of boric acid (pH 14). Nitrate is reduced quantitatively to nitrite by a cadmium-copper reductor in the form of an open tube cadmium reactor. The nitrite formed is diazotized with sulfanilamide and subsequently coupled with *N*-(1-naphthyl)ethylenediamine dihydrochloride. This reaction takes place in an acidic solution (pH 2). The resulting highly colored azo dye is colorimetrically detected at 540 nm (Reference 15.4).

2.2 The quality of the analysis is assured through reproducible calibration and testing of the Segmented Flow Analysis (SFA) and Flow Injection Analysis (FIA) system.

2.3 A general flow diagram of the SFA/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 interference from iron, copper, and other metals by using ethylenediaminetetraacetic acid (EDTA) in the buffer solution.

4.2 Norwitz and Keliher (References 15.1 and 15.2) have compiled a comprehensive study of interferences in the spectrophotometric analysis of nitrite.

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 Chloride, NH_4Cl (FW 53.50)

5.3.2 Ammonium Hydroxide, NH_4OH (FW 35.05)

5.3.3 Ammonium Sulfate, $(NH_4)_2SO_4$ (FW 132.14)

5.3.4 Boric Acid, H_3BO_3 (FW 61.83)

5.3.5 Cadmium, Cd (FW 112.40)

5.3.6 Chloroform, $CHCl_3$ (FW 119.38)

5.3.7 Cupric Sulfate Pentahydrate, $CuSO_4 \cdot 5H_2O$ (FW 249.61)

5.3.8 Ethylenediaminetetraacetic Acid, Disodium Salt Dihydrate (EDTA), $C_{10}H_{16}N_2Na_2O_8 \cdot 2H_2O$ (FW 372.24)

5.3.9 Glycine, $H_2NCH_2CO_2H$ (FW 75.07)

5.3.10 Hydrochloric Acid, concentrated, HCl (FW 36.46)

5.3.11 *N*-(1-naphthyl)ethylenediamine Dihydrochloride, $C_{12}H_{14}N_2 \cdot 2HCl$ (FW 259.18)

5.3.12 Phosphoric Acid, concentrated, H_3PO_4 (FW 98.00)

5.3.13 Potassium Nitrate, KNO_3 (FW 101.11)

5.3.14 Potassium Nitrite, KNO_2 (FW 85.11)

5.3.15 Potassium Persulfate, $K_2S_2O_8$ (FW 270.33)

5.3.16 Sodium Hydroxide, NaOH (FW 40.00)

5.3.17 Sulfanilamide, $C_6H_8N_2O_2S$ (FW 172.21)

5.3.18 Urea, H_2NCONH_2 (FW 60.06)

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 Segmented Flow Analysis (SFA)/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 540-nm optical filter

6.1.4 Data Acquisition System (PC or Notebook PC) with WinFLOW™ software

6.1.5 Total Dissolved Nitrogen Cartridge (Part #319784)

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 Chloride, NH_4Cl (FW 53.50)

7.1.2 Ammonium Hydroxide, NH_4OH (FW 35.05)

7.1.3 Ammonium Sulfate, $(NH_4)_2SO_4$ (FW 132.14)

7.1.4 Boric Acid, H_3BO_3 (FW 61.83)

7.1.5 Brij®-35, 30% w/v (Part #A21-0110-33)

7.1.6 Chloroform, $CHCl_3$ (FW 119.38)

7.1.7 Cupric Sulfate Pentahydrate, $CuSO_4 \cdot 5H_2O$ (FW 249.61)

7.1.8 Deionized Water (ASTM Type I or II)

7.1.9 Ethylenediaminetetraacetic Acid, Disodium Salt Dihydrate (EDTA), $C_{10}H_{16}N_2Na_2O_8 \cdot 2H_2O$ (FW 372.24)

7.1.10 Glycine, $H_2NCH_2CO_2H$ (FW 75.07)

7.1.11 Hydrochloric Acid, concentrated, HCl (FW 36.46)

7.1.12 *N*-(1-naphthyl)ethylenediamine Dihydrochloride, $C_{12}H_{14}N_2 \cdot 2HCl$ (FW 259.18)

7.1.13 Phosphoric Acid, concentrated, H_3PO_4 (FW 98.00)

7.1.14 Potassium Nitrate, KNO_3 (FW 101.11)

7.1.15 Potassium Nitrite, KNO_2 (FW 85.11)

7.1.16 Potassium Persulfate, $K_2S_2O_8$ (FW 270.33)

7.1.17 Sodium Hydroxide, NaOH (FW 40.00)

7.1.18 Sulfanilamide, $C_6H_8N_2O_2S$ (FW 172.21)

7.1.19 Urea, H_2NCONH_2 (FW 60.06)