O·I·Analytical

Methodology

On-Line Distillation Cyanide by UV Digestion and Segmented Flow Analysis (SFA)

(Cartridge Part #A002599)

1.0 Scope and Application

1.1 This method is used for the determination of cyanide in drinking water, surface water, and domestic and industrial wastes.

Note: This method is not approved for the National Pollution Discharge Elimination System (NPDES).

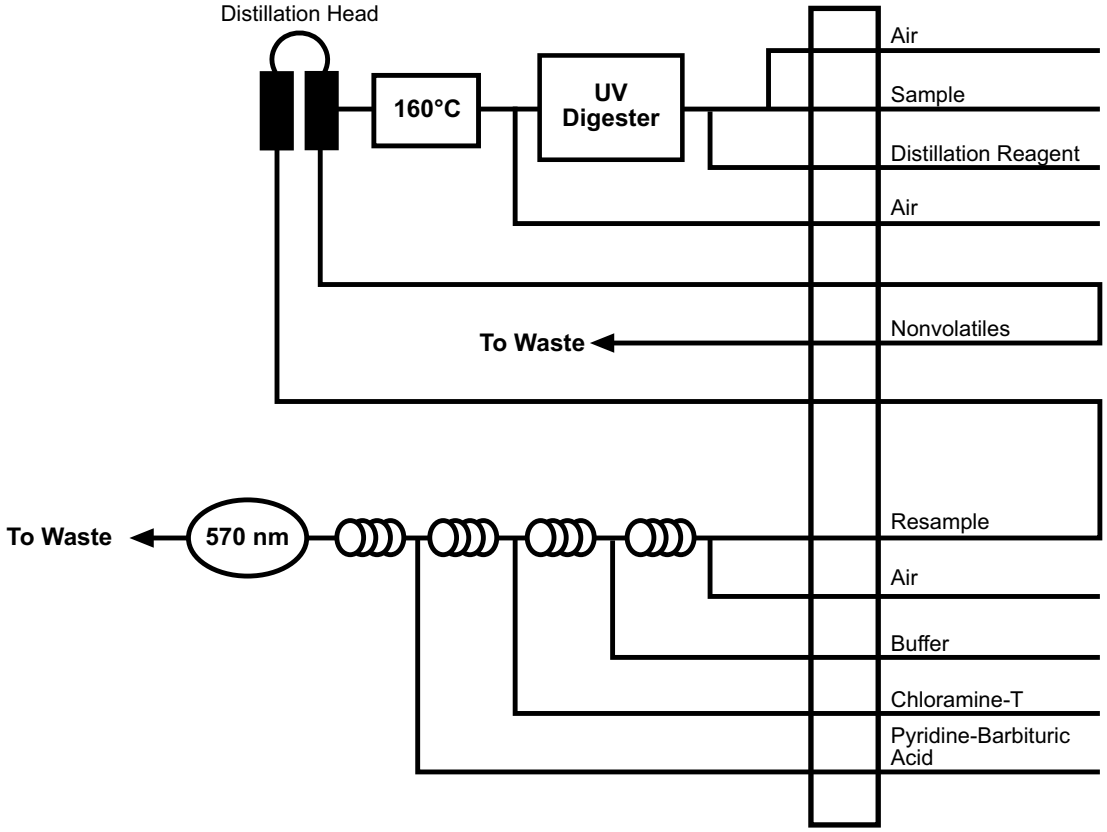
1.2 The Method Detection Limit (MDL) of this method is 1.5 µg/L cyanide. The applicable range of the method is 5.0–500 µg/L cyanide. The range may be extended to analyze higher concentrations by sample dilution.

2.0 Summary of Method

2.1 Cyanide is released from cyanide complexes by UV digestion and distillation. The liberated hydrogen cyanide is converted to cyanogen chloride by reaction with chloramine-T trihydrate at a pH of less than 8. The cyanogen chloride then reacts with the pyridine-barbituric acid reagent to form a red colored complex, which is measured at 570 nm (References 15.3 and 15.5).

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

2.3 A general flow diagram of the SFA 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 Interferences are eliminated or reduced prior to color formation by distillation.

4.2 Treat samples containing sulfide by adding cadmium nitrate powder (Section 8.6).

4.3 Remove oxidizing agents that decompose cyanides by adding ascorbic acid (Section 8.4).

4.4 Thiocyanates produce a positive interference when they are decomposed to cyanide by ultraviolet radiation (References 15.3 and 15.5).

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 Barbituric Acid, $C_4H_4N_2O_3$ (FW 128.09)

5.3.2 Chloramine-T Trihydrate, $CH_3C_6H_4SO_2NNaCl{\bullet}3H_2O$ (FW 281.69)

5.3.3 Hydrochloric Acid, concentrated, HCl (FW 36.46)

5.3.4 Hypophosphorous Acid, 50% w/v, H_3PO_2 (FW 66.00)

5.3.5 Phosphoric Acid, concentrated, H_3PO_4 (FW 98.00)

5.3.6 Potassium Cyanide, KCN (FW 65.12)

5.3.7 Potassium Phosphate Monobasic, KH_2PO_4 (FW 136.09)

5.3.8 Pyridine, C_5H_5N (FW 79.10)

5.3.9 Sodium Hydroxide, NaOH (FW 40.00)

5.3.10 Sodium Phosphate Dibasic, Na_2HPO_4 (FW 141.96)

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) 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 On-Line Distillation Cyanide Cartridge (Part #A002599)

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 Barbituric Acid, $C_4H_4N_2O_3$ (FW 128.09)

7.1.2 Chloramine-T Trihydrate, $CH_3C_6H_4SO_2NNaCl \cdot 3H_2O$ (FW 281.69)

7.1.3 Deionized Water (ASTM Type I or II)

7.1.4 DOWFAX® 2A1 (Part #A000080)

7.1.5 Hydrochloric Acid, concentrated, HCl (FW 36.46)

7.1.6 Hypophosphorous Acid, 50% w/v, H_3PO_2 (FW 66.00)

7.1.7 Phosphoric Acid, concentrated, H_3PO_4 (FW 98.00)

7.1.8 Potassium Cyanide, KCN (FW 65.12)

7.1.9 Potassium Phosphate Monobasic, KH_2PO_4 (FW 136.09)

7.1.10 Pyridine, C_5H_5N (FW 79.10)

7.1.11 Sodium Hydroxide, NaOH (FW 40.00)

7.1.12 Sodium Phosphate Dibasic, Na_2HPO_4 (FW 141.96)