O·I·Analytical

Methodology

On-Line Distillation Phenol by Segmented Flow Analysis (SFA)

(Cartridge Part #A002606)

1.0 Scope and Application

1.1 This method is used for the determination of phenolic compounds in drinking water, surface water, saline water, and domestic and industrial wastes (Reference 15.2).

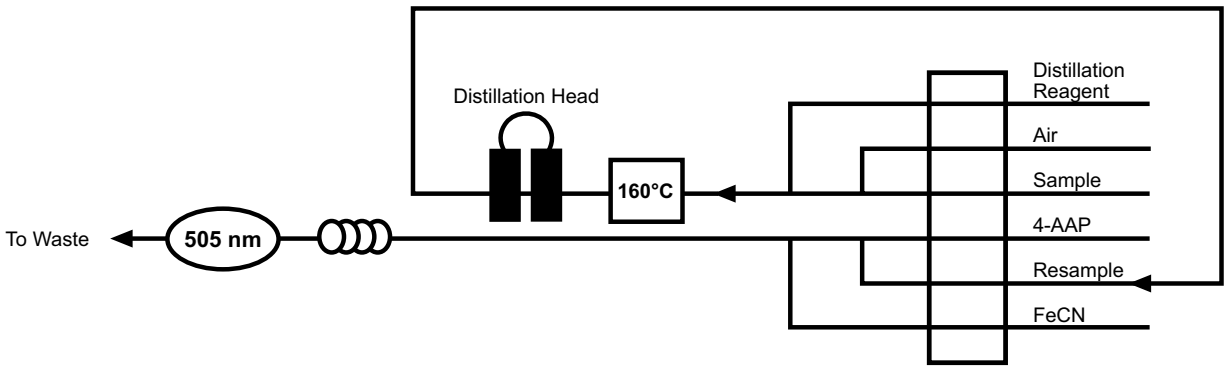
1.2 The Method Detection Limit (MDL) of this method is 1.0 µg/L phenol. The applicable range of the method is 5.0–500 µg/L phenol. The range may be extended to analyze higher concentrations by sample dilution.

2.0 Summary of Method

2.1 Phenol is distilled on-line from an acidic solution at 160°C. Phenol reacts with 4-aminoantipyrine (4-AAP) and alkaline ferricyanide (FeCN) to form a red complex. The absorbance is measured at 505 nm (Reference 15.2).

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 Eliminate interferences from sulfur compounds by acidifying the sample to a pH less than 4 with phosphoric acid, aerating briefly by stirring, and adding copper sulfate.

4.2 Remove oxidizing agents such as chlorine immediately after sampling by adding an excess of ferrous ammonium sulfate (Section 7.2.8). Oxidizing agents can be detected by the liberation of iodine upon acidification in the presence of potassium iodide. If chlorine is not removed, the phenolic compounds may be partially oxidized, and the results may be low.

4.3 Use glass tubes or acid-washed plastic cups for the samples and calibrants to eliminate background contamination from plastic tubes and sample containers.

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 4-Aminoantipyrine, $C_{11}H_{13}N_3O$ (FW 203.25)

5.3.2 Boric Acid, H_3BO_4 (FW 61.84)

5.3.3 Ferrous Ammonium Sulfate, $(NH_4)_2SO_4FeSO_4 \cdot 6H_2O$ (FW 392.13)

5.3.4 Phenol, C_6H_5OH (FW 94.11)

5.3.5 Phosphoric Acid, concentrated, 85%, H_3PO_4 (FW 98.00)

5.3.6 Potassium Chloride, KCl (FW 74.55)

5.3.7 Potassium Ferricyanide, $K_3Fe(CN)_6$ (FW 329.25)

5.3.8 Sodium Hydroxide, NaOH (FW 40.00)

5.3.9 Sulfuric Acid, concentrated, H_2SO_4 (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 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 505-nm optical filter

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

6.1.5 On-line Distillation Phenol Cartridge (Part #A002606)

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 4-Aminoantipyrine, $C_{11}H_{13}N_3O$ (FW 203.25)

7.1.2 Boric Acid, H_3BO_4 (FW 61.84)

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

7.1.4 Deionized Water (ASTM Type I or II)

7.1.5 Ferrous Ammonium Sulfate, $(NH_4)_2SO_4FeSO_4 \cdot 6H_2O$ (FW 392.13)

7.1.6 Phenol, C_6H_5OH (FW 94.11)

7.1.7 Phosphoric Acid, concentrated, 85%, H_3PO_4 (FW 98.00)

7.1.8 Potassium Chloride, KCl (FW 74.55)

7.1.9 Potassium Ferricyanide, $K_3Fe(CN)_6$ (FW 329.25)

7.1.10 Sodium Hydroxide, NaOH (FW 40.00)

7.1.11 Sulfuric Acid, concentrated, H_2SO_4 (FW 98.08)

results, store degassed reagent water under a slight vacuum when not in use.