



## Post-Distillation Phenol by Segmented Flow Analysis (SFA)

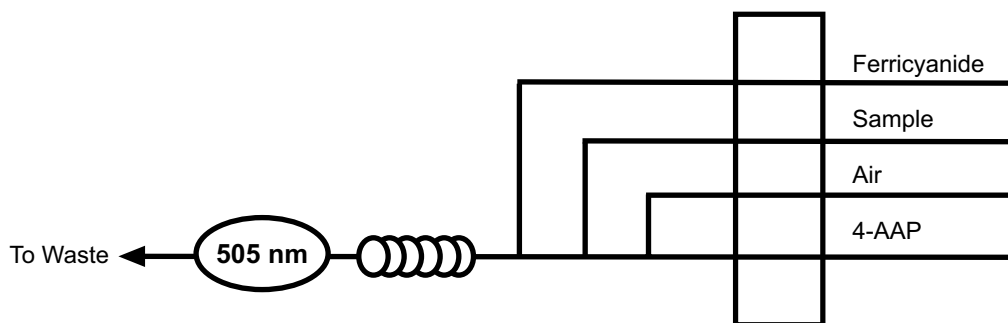
(Cartridge Part #319431)

### 1.0 Scope and Application

- 1.1 This method is used for the determination of phenolic materials in drinking, surface, and saline waters, as well as domestic and industrial wastes.
- 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, or by the use of higher order curve fitting techniques.

### 2.0 Summary of Method

- 2.1 Phenol reacts with 4-aminoantipyrine and alkaline ferricyanide to form a red complex that 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 Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of less than 4.0 with sulfuric acid, and aerating briefly by stirring and adding cupric sulfate.
- 4.2 Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of ferrous ammonium sulfate. If chlorine is not removed, the phenolic compounds may be partially oxidized, and the results may be lower than expected.
- 4.3 Background contamination from reagent and sample containers can be eliminated by using glass or acid-washed plastic 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,  $H_3BO_3$  (FW 61.84)
  - 5.3.3 Phenol,  $C_6H_5OH$  (FW 94.11)
  - 5.3.4 Phosphoric Acid, Concentrated,  $H_3PO_4$  (FW 98.00)
  - 5.3.5 Potassium Chloride, KCl (FW 74.55)
  - 5.3.6 Potassium Ferricyanide,  $K_3Fe(CN)_6$  (FW 329.25)
  - 5.3.7 Sodium Hydroxide, NaOH (FW 40.00)
  - 5.3.8 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 Post-Distillation Phenol Cartridge (Part #319431)
- 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,  $H_3BO_3$  (FW 61.84)
  - 7.1.3 Deionized Water (ASTM Type I or II)
  - 7.1.4 Dowfax® 2A1 (Part #A000080)
  - 7.1.5 Phenol,  $C_6H_5OH$  (FW 94.11)
  - 7.1.6 Phosphoric Acid, Concentrated,  $H_3PO_4$  (FW 98.00)

- 7.1.7 Potassium Chloride, KCl (FW 74.55)
- 7.1.8 Potassium Ferricyanide,  $K_3Fe(CN)_6$  (FW 329.25)
- 7.1.9 Sodium Hydroxide, NaOH (FW 40.00)
- 7.1.10 Sulfuric Acid, Concentrated,  $H_2SO_4$  (FW 98.08)