



Low-Level Orthophosphate by Segmented Flow Analysis (SFA)

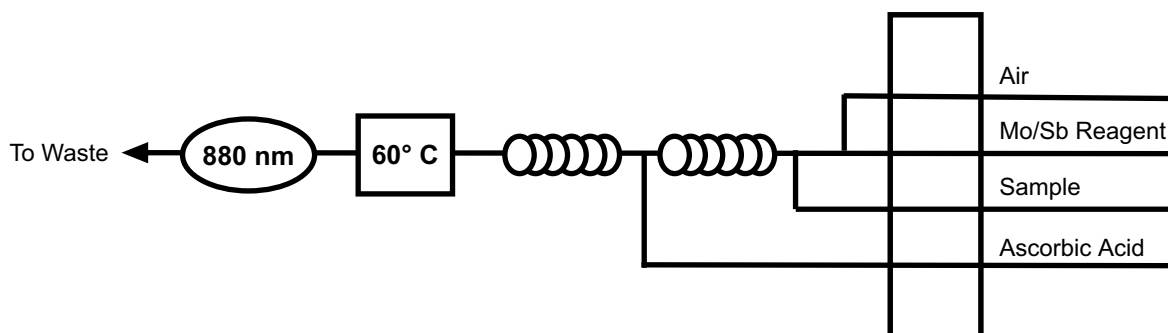
(Cartridge Part #319532)

1.0 Scope and Application

- 1.1 This method is used for the determination of low-level orthophosphate.
- 1.2 The Method Detection Limit (MDL) of this method is 0.266 ppb orthophosphate. The applicable range of the method is 1.0 ppb–1.0 ppm orthophosphate. The range may be extended to analyze higher concentrations by sample dilution (Reference 15.2).

2.0 Summary of Method

- 2.1 Orthophosphate reacts with molybdenum VI and antimony III in an acid medium to form an antimonyphosphomolybdate complex. This complex is subsequently reduced with ascorbic acid to form a blue color, and the absorbance is measured at 880 nm (Reference 15.4).
- 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 Ferric iron up to 40 mg/L, copper up to 10 mg/L, and silica up to 10 mg/L do not interfere (Reference 15.4).
- 4.2 Filter turbid samples prior to analysis.
- 4.3 Samples with background absorbance at the analytical wavelength may interfere.

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 Molybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$)
 - 5.3.2 Antimony Potassium Tartrate ($\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot 1/2\text{H}_2\text{O}$)
 - 5.3.3 Ascorbic Acid ($\text{C}_6\text{H}_8\text{O}_6$)
 - 5.3.4 Potassium Dihydrogen Phosphate (KH_2PO_4)
 - 5.3.5 Sulfuric Acid, concentrated (H_2SO_4)
- 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 10-mm path length flowcell and 880-nm optical filter
- 6.1.4 Data Acquisition System (PC or Notebook PC) with WinFLOW™ software
- 6.1.5 Low-Level Orthophosphate Cartridge (OI Analytical Part #319532)

6.2 Sampling equipment—Sample bottle, amber glass, with polytetrafluoroethylene (PTFE)-lined cap. Clean by washing with detergent and water, and rinsing with two aliquots of reagent water. Dry 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 Molybdate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ (CAS #13106-76-8, Sigma-Aldrich #27,790-8)
- 7.1.2 Antimony Potassium Tartrate, $\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot 1/2\text{H}_2\text{O}$ (Sigma-Aldrich #23,005-7)
- 7.1.3 Ascorbic Acid, $\text{C}_6\text{H}_8\text{O}_6$ (CAS #50-81-7, Sigma-Aldrich #25,556-4)
- 7.1.4 Deionized Water, ASTM Type I or II
- 7.1.5 Dowfax® 2A1 (OI Analytical Part #A000080)
- 7.1.6 Potassium Dihydrogen Phosphate, KH_2PO_4 (CAS #7778-77-0, Sigma-Aldrich #22,980-6)
- 7.1.7 Sulfuric Acid, concentrated, H_2SO_4 (CAS #7664-93-9, Sigma-Aldrich #43,558-9)