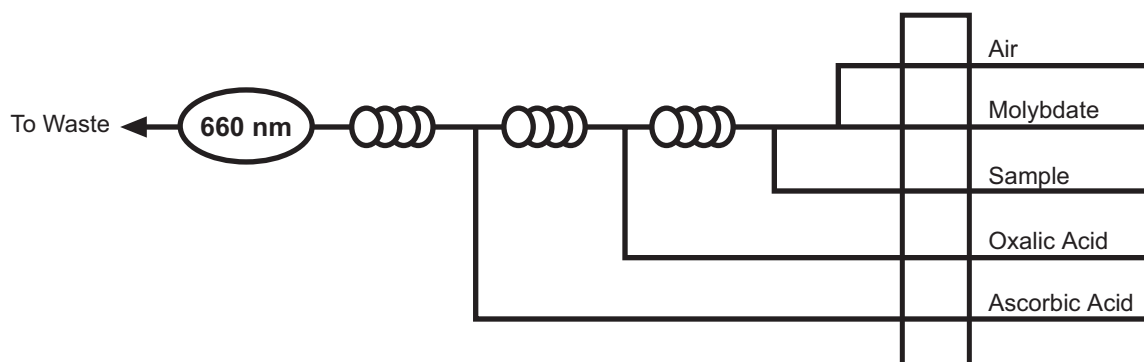


(Cartridge Part #A002600)

- 1.1 This method is used for the determination of silica in surface water and domestic and industrial wastewater.
- 1.2 The Method Detection Limit (MDL) of this method is 0.014 mg/L silica (SiO₂). The applicable range of the method is 0.10–60 mg/L silica. The range may be extended to analyze higher concentrations by sample dilution.

- 2.1 Silica in solution as silicic acid or silicate reacts with a molybdate reagent in acid media to form β -molybdosilicic acid. The complex is reduced by ascorbic acid to form molybdenum blue. The absorbance is measured at 660 nm (Reference 15.3).
- 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 Add oxalic acid to suppress interference from phosphate.
- 4.2 Remove hydrogen sulfide by boiling an acidified sample prior to analysis.
- 4.3 Large amounts of iron interfere.
- 4.4 Filter or centrifuge turbid samples prior to determination.
- 4.5 Samples with background absorbance at the analytical wavelength may interfere (References 15.3 and 15.4).
- 4.6 Avoid using borosilicate glassware for sample collection or reagent storage. Use polyethylene containers whenever possible (Reference 15.4).

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 Tetrahydrate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ (FW 1,235.95)
 - 5.3.2 Oxalic Acid, $\text{C}_2\text{H}_2\text{O}_4$ (FW 90.04)
 - 5.3.3 Sodium Hydroxide, NaOH (FW 40.00)
 - 5.3.4 Sodium Metasilicate Pentahydrate, $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ (FW 212.08)
 - 5.3.5 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 660-nm optical filter
 - 6.1.4 Data Acquisition System (PC or Notebook PC) with WinFLOW™ software
 - 6.1.5 Silica Cartridge (Part #A002600)
- 6.2 Sampling equipment—Sample bottle, high density polyethylene (HDPE) 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 Molybdate Tetrahydrate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ (FW 1,235.95)
 - 7.1.2 Ascorbic Acid, $\text{C}_6\text{H}_8\text{O}_6$ (FW 176.12)
 - 7.1.3 Deionized Water (ASTM Type I or II)
 - 7.1.4 DOWFAX® 2A1 (Part #A000080)
 - 7.1.5 Oxalic Acid, $\text{C}_2\text{H}_2\text{O}_4$ (FW 90.04)
 - 7.1.6 Sodium Hydroxide, NaOH (FW 40.00)

7.1.7 Sodium Metasilicate Pentahydrate, $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ (FW 212.08)

7.1.8 Sulfuric Acid, concentrated, H_2SO_4 (FW 98.08)