

Flow Solution™ FS 3700 Automated Chemistry Analyzer Silica, Standard Methods by Segmented Flow Analysis (SFA)

WATER & WASTEWATER SERIES

Channel Part Number: 332386
Cartridge Part Number: 332387
Method Part Number: 332388

Scope and Application

This method is used for silica determination in surface water, as well as domestic and industrial wastes according to the Standard Method 4500-SiO₂ E.

Range	0.02 - 20 mg/L SiO ₂
Rate	60 samples/hour
Precision	~3 % RSD
Method Detection Limit (MDL)	0.02 mg/L SiO ₂

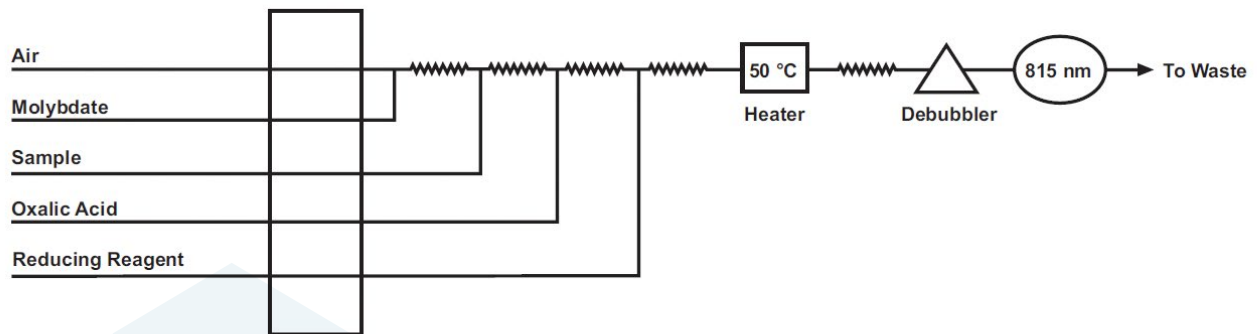


Figure 1. General flow diagram for Silica - Standard Methods by Segmented Flow Analysis Injection (SFA)

Reagents and Calibrants

Chemical Name	CAS #	Chemical Formula	Part Number
Ammonium Molybdate Tetrahydrate		$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$	
1-Amino-2-Naphthol-4-Sulfonic Acid		$\text{C}_{10}\text{H}_9\text{NO}_4\text{S}$	
Oxalic Acid		$\text{C}_2\text{H}_2\text{O}_4$	
Sodium Bisulfite		NaHSO_3	
Sodium Hydroxide		NaOH	
Sodium Metasilicate Pentahydrate		$\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$	
Sodium Sulfite		Na_2SO_3	
Sulfuric Acid, concentrated		H_2SO_4	
Water, deionized		H_2O	

Summary of Standard Methods 4500-SiO₂ E

Method

- Silica in solution as silicic acid or silicate reacts with a molybdate reagent in acid media to form β -molybdosilicic acid; heating converts "molybdate-unreactive" silica to "molybdate-reactive" varieties. The complex is then reduced by 1-amino-2-naphthol-4-sulfonic acid to form molybdenum blue. The absorbance is measured at 815 nm (Reference 4).
- Assure analysis quality through reproducible calibration and testing of the segmented flow analysis (SFA) system.

Interferences

- Add oxalic acid to suppress interference from phosphate.
- Remove hydrogen sulfide by boiling an acidified sample prior to analysis.
- Large amounts of iron interfere.
- Filter or centrifuge turbid samples prior to determination.
- Samples with background absorbance at the analytical wavelength may interfere (References 4 and 5).
- Avoid using borosilicate glassware for sample collection or reagent storage. Use polyethylene containers whenever possible.

Safety

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.
2. For reference purposes, a file of Safety Data Sheets (SDS) 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.
3. Chemicals used in this method may be highly toxic or hazardous and should be handled with extreme caution at all times. Consult the appropriate SDS before handling.
4. Unknown samples may be potentially hazardous and should be handled with extreme caution at all times.
5. Proper personal protective equipment (PPE) should be used when handling or working in the presence of chemicals.
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.

Sample Handling and Preservation

1. Collect samples in plastic or glass bottles thoroughly cleaned and rinsed with reagent water.
2. Ensure the volume of sample collected is sufficient to obtain a representative sample, analyze replicates, and minimize waste disposal.
3. Determine silica in unpreserved samples immediately upon collection.
4. If cooled immediately and stored at 4 °C, unpreserved samples may be stored for up to 28 days (Reference 6).

References

1. *Code Of Federal Regulations*, Part 136, Title 40, Appendix B, 1994.
2. Flow Solution™ 3700 Operator's Manual (part number 329998). Available from OI Analytical, P.O. Box 9010, College Station, TX, 77842-9010.
3. Flow Solution™ 3700 ACA Flow Analyzer Software Quick Start Guide (part number 327069). Available from OI Analytical, P.O. Box 9010, College Station, TX, 77842-9010.
4. Handbook for Analytical Quality Control in Water and Wastewater Laboratories; EPA-600/4-79-019; US Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1979.
5. *Less is Better: Laboratory Chemical Management for Waste Reduction*. Available from the American Chemical Society, Department of Government Regulations and Science Policy, 1155 16th Street, NW, Washington, DC, 20036.3.
6. Sample Preservation. *Methods for Chemical Analysis of Water and Wastes*; EPA-600/4-79-020; U.S.Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1984; xvii.
7. *Standard Methods for the Examination of Water and Wastewater*, 20th ed.; American Public Health Association: Washington, D.C., 1998.
8. Flow Solution™ 3700 Operator's Manual (part number 329998). Available from OI Analytical, P.O. Box 9010, College Station, TX, 77842-9010.

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