Method Abstract

Scope

This method is used for the determination of silica in surface water and domestic and industrial wastewater according to USGS Method I-2700-85 and upon Standard Methods 4500–SiO_2 F. Additionally, this method enables silica analysis according to ISO Method 16264.

Summary

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 stannous chloride to form molybdenum blue. The absorbance is measured at 815 nm.

Interferences

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

Performance Specifications

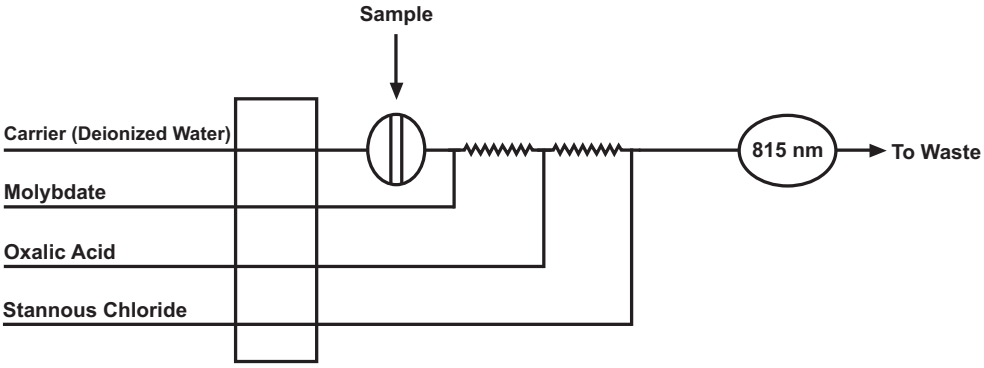
Range:	0.05–50 mg/L SiO_2
Throughput:	60 samples/hour
Precision (at 0.1 mg/L):	<1% RSD
Precision (at 1.0 mg/L):	<1% RSD
Precision (at 10.0 mg/L):	<1% RSD
Method Detection Limit (MDL):	0.005 mg/L SiO_2
ERA* QC Results	105% Recovery

*ERA (Environmental Resources Associates) WasteWatR Simple Nutrients Quality Control Sample.

Chemicals

- Ammonium Molybdate Tetrahydrate, $(NH_4)_6Mo_7O_{24}\bullet 4H_2O$
- Deionized (DI) Water (ASTM Type I or II)
- Hydrochloric Acid, concentrated, HCl
- Oxalic Acid, $C_2H_2O_4$
- Sodium Hydroxide, NaOH
- Sodium Metasilicate Pentahydrate, $Na_2SiO_3\bullet 5H_2O$
- Stannous Chloride Dihydrate, $SnCl_2\bullet 2H_2O$
- Sulfuric Acid, concentrated, H_2SO_4

Basic Flow Diagram



Selected References

Handbook for Analytical Quality Control in Water and Wastewater Laboratories; EPA–600/4–79–019; U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1979.

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.

Standard Methods for the Examination of Water and Wastewater, 21st ed.; American Public Health Association: Washington D.C., 2005.

Methods for Determination of Inorganic Substances in Water and Fluvial Sediments; Silica, Colorimetric, Dissolved; I-2700-85; US Geological Survey

Figures

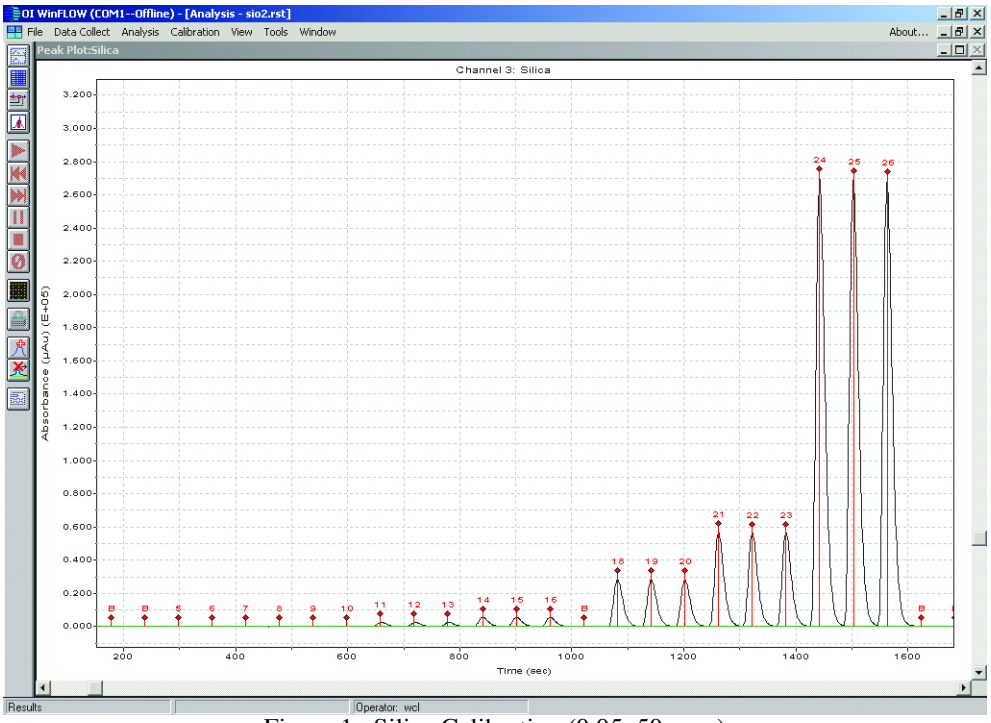


Figure 1. Silica Calibration (0.05–50 ppm)

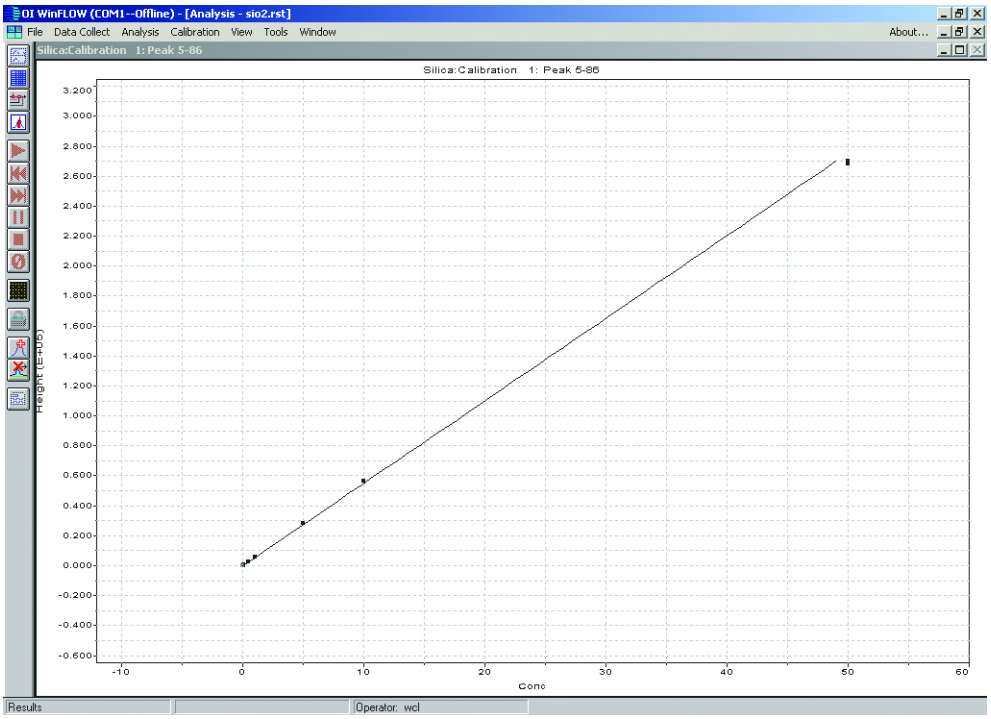
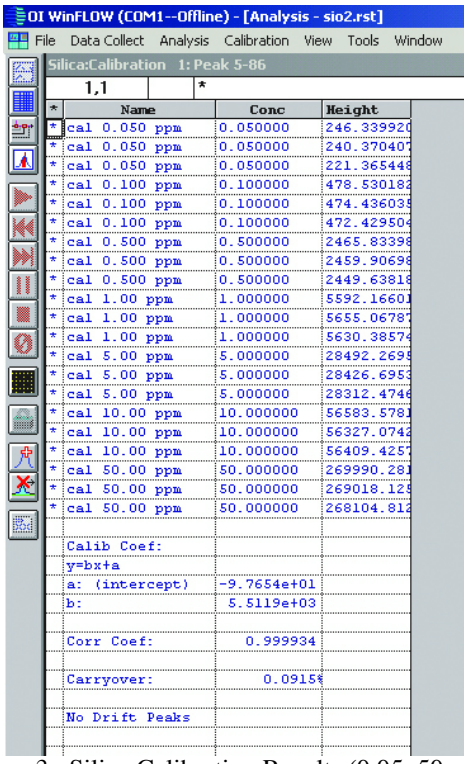


Figure 2. Silica Calibration Curve (0.05–50 ppm)



OI WinFLOW (COM1--Offline) - [Analysis - sio2.rst]

File Data Collect Analysis Calibration View Tools Window

Silica:Calibration 1: Peak 5-86

1,1 *

*	Name	Conc	Height
*	cal 0.050 ppm	0.050000	246.339920
*	cal 0.050 ppm	0.050000	240.370407
*	cal 0.050 ppm	0.050000	221.365448
*	cal 0.100 ppm	0.100000	478.530182
*	cal 0.100 ppm	0.100000	474.436035
*	cal 0.100 ppm	0.100000	472.429504
*	cal 0.500 ppm	0.500000	2465.83398
*	cal 0.500 ppm	0.500000	2459.90698
*	cal 0.500 ppm	0.500000	2449.63818
*	cal 1.00 ppm	1.000000	5592.16601
*	cal 1.00 ppm	1.000000	5655.06787
*	cal 1.00 ppm	1.000000	5630.38574
*	cal 5.00 ppm	5.000000	28492.2695
*	cal 5.00 ppm	5.000000	28426.6953
*	cal 5.00 ppm	5.000000	28312.4746
*	cal 10.00 ppm	10.000000	56583.5781
*	cal 10.00 ppm	10.000000	56327.0742
*	cal 10.00 ppm	10.000000	56409.4257
*	cal 50.00 ppm	50.000000	269990.281
*	cal 50.00 ppm	50.000000	269018.125
*	cal 50.00 ppm	50.000000	268104.812
	Calib Coef:		
	y=bx+a		
	a: (intercept)	-9.7654e+01	
	b:	5.5119e+03	
	Corr Coef:	0.999934	
	Carryover:	0.0915%	
	No Drift Peaks		

Figure 3. Silica Calibration Results (0.05–50 ppm)

Table 1. Silica Precision Calculations

	0.10 mg SiO_2/L	1.00 mg SiO_2/L	10.0 mg SiO_2/L	50.0 mg SiO_2/L	0.05 mg SiO_2/L	ERA P127-739B
Rep 1	0.106	1.03	10.3	48.6	0.066	17.3
Rep 2	0.106	1.03	10.4	48.5	0.064	16.8
Rep 3	0.106	1.04	10.2	48.2	0.063	16.7
Rep 4	0.105	1.04	10.2	48.1	0.065	16.7
Rep 5	0.106	1.04	10.2	48.3	0.066	—
Rep 6	0.104	1.05	10.4	49.0	0.068	—
Rep 7	0.106	1.04	10.3	48.9	0.065	—
Rep 8	—	1.04	10.2	48.7	—	—
Rep 9	—	—	10.2	48.8	—	—
Rep 10	—	—	10.1	48.1	—	—
Mean	0.106	1.04	10.2	48.5	0.065	16.9
Standard Deviation	0.00079	0.004785	0.093124	0.33275	0.001691	0.285185
% RSD	0.75	0.46	0.91	0.68	2.59	1.69
% Recovery	106	104	102	97	130	108
MDL	—	—	—	—	**0.005**	—

(800) 653-1711
(979) 690-1711
www.oico.com
oimail@oico.com

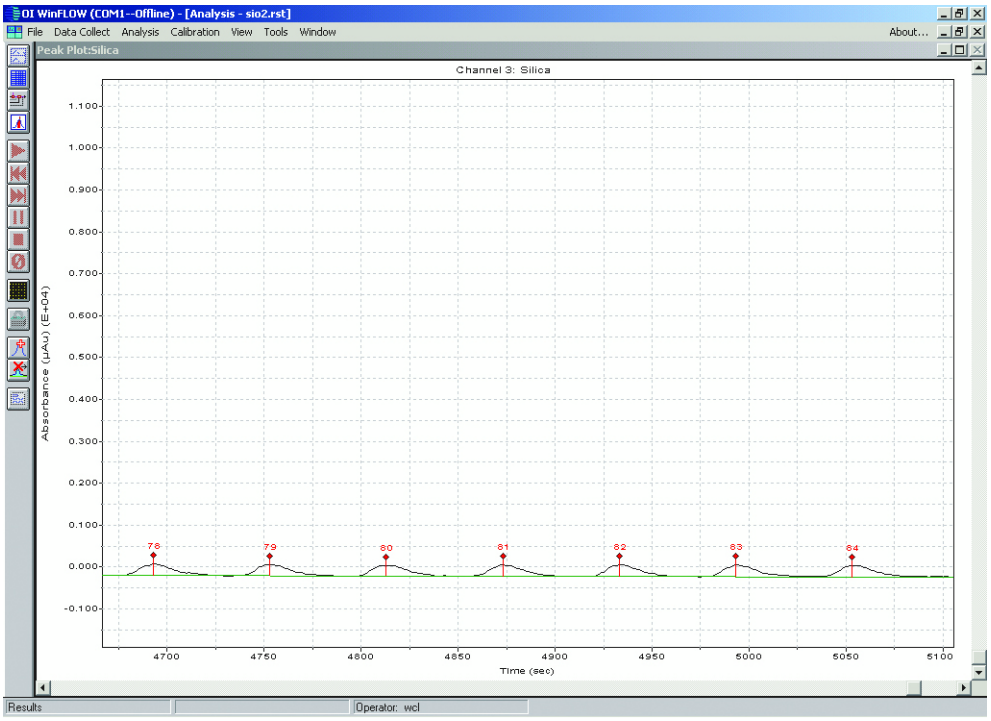


Figure 4. Silica Method Detection Limit (at 0.05 ppm)

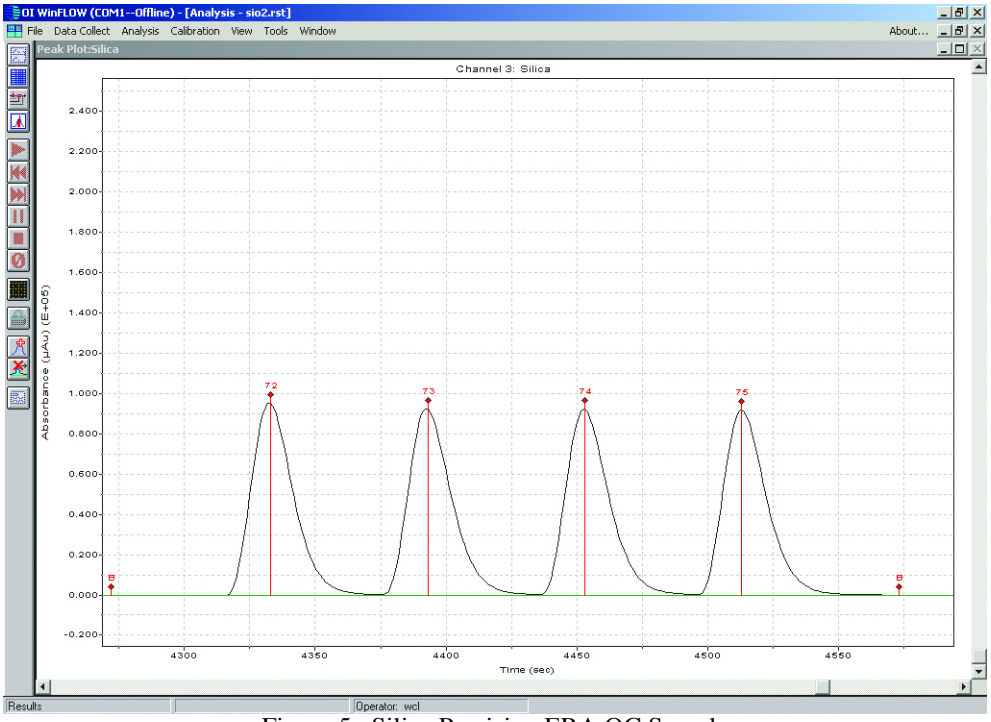


Figure 5. Silica Precision ERA QC Sample

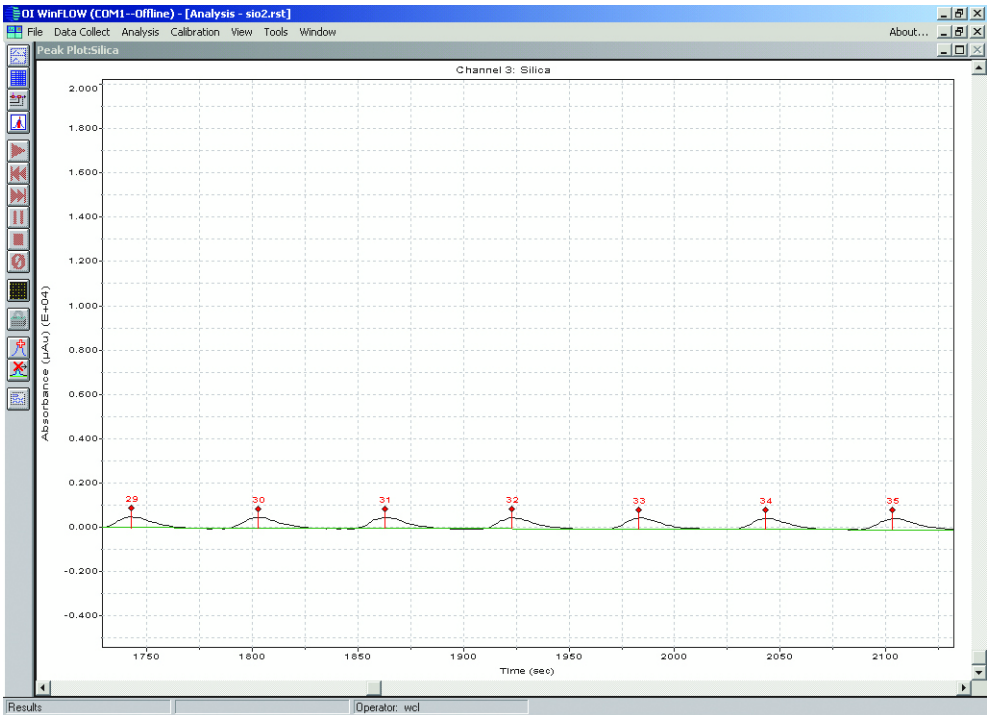


Figure 6. Silica Precision (at 0.1 ppm)

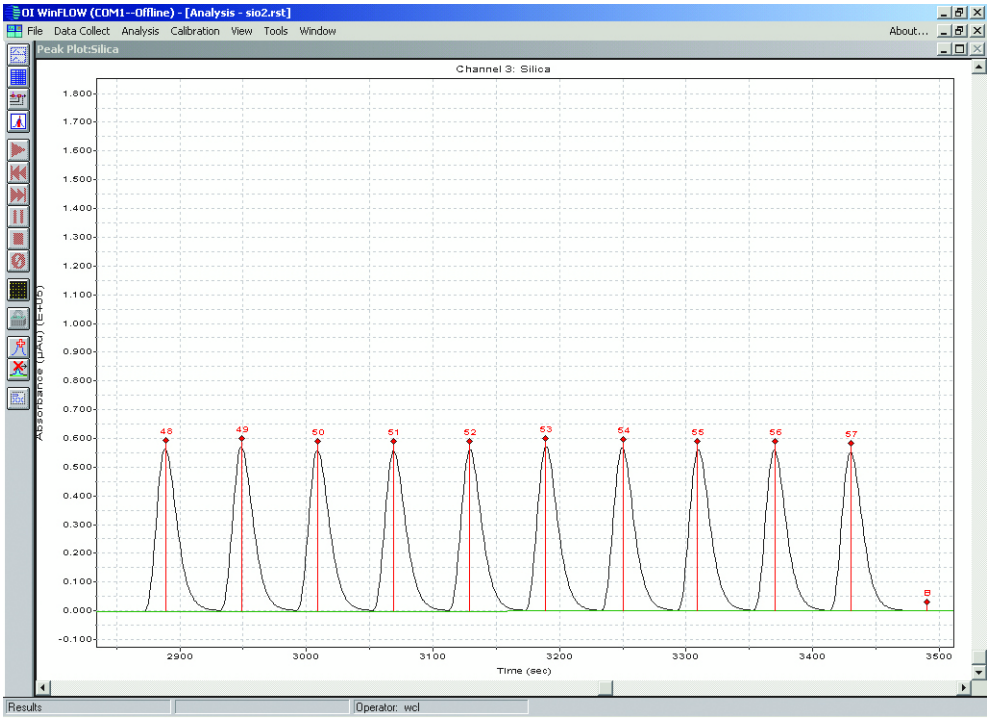


Figure 7. Silica Precision (at 10.0 ppm)