Method Abstract

Scope

This method is used for determining silica in surface water and domestic and industrial wastes according to Standard Methods 4500-SiO_2 E.

Summary

Silica in solution as silicic acid or silicate reacts with a molybdate reagent in acid media to form *ß*-molybdosilicic acid; heating converts "molybdate-unreactive" silica to "molybdate-reactive" varieties. The complex is then reduced by 1-amino-2-napthol-4-sulfonic acid to form molybdenum blue. The absorbance is measured at 815 nm.

Interferences

Filter turbid samples prior to analysis. Samples containing iron, copper, or silicate at concentrations greater than 50, 10, and 10 mg/L, respectively, interfere with this assay. Samples with background absorbance at the analytical wavelength may interfere. Residual phosphate in the flow system components and from continuous phosphate analysis may interfere. Wash the system and glassware with 0.1 N HCl to correct phosphate interferences.

Performance Specifications

Range:	0.2–20 mg/L
Throughput:	60 samples/hour
Precision (at 0.2 mg/L):	<3% RSD
Precision (at 2.0 mg/L):	<1% RSD
Precision (at 20.0 mg/L):	~1% RSD
Method Detection Limit (MDL):	0.02 mg/L
ERA QC Sample Result:	117%

Chemicals

1-Amino-2-Naphthol-4-Sulfonic Acid, $C_{10}H_9NO_4S$

Ammonium Molybdate Tetrahydrate, $(NH_4)_6Mo_7O_{24}\bullet 4H_2O$

Deionized (DI) Water, ASTM Type I or II

DOWFAX® 2A1, part number A000080

Oxalic Acid, $C_2H_2O_4$

Sodium Bisulfite, $HNaSO_3$

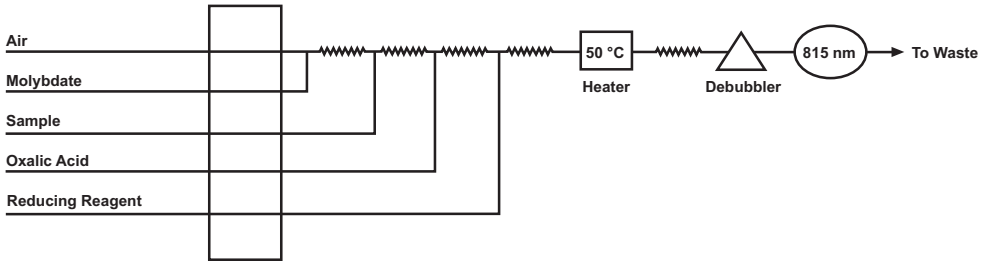
Sodium Hydroxide, NaOH

Sodium Metasilicate Pentahydrate, $Na_2SiO_3\bullet 5H_2O$

Sodium Sulfite, Na_2SO_3

Sulfuric Acid, concentrated, H_2SO_4

Basic Flow Diagram



Note

This method complies with Standard Method 4500-Silica E.

Selected Reference

Standard Methods for the Examination of Water and Wastewater, 20th ed.; American Public Health Association: Washington, D.C., 1998. Method 4500-Silica E.

Figures

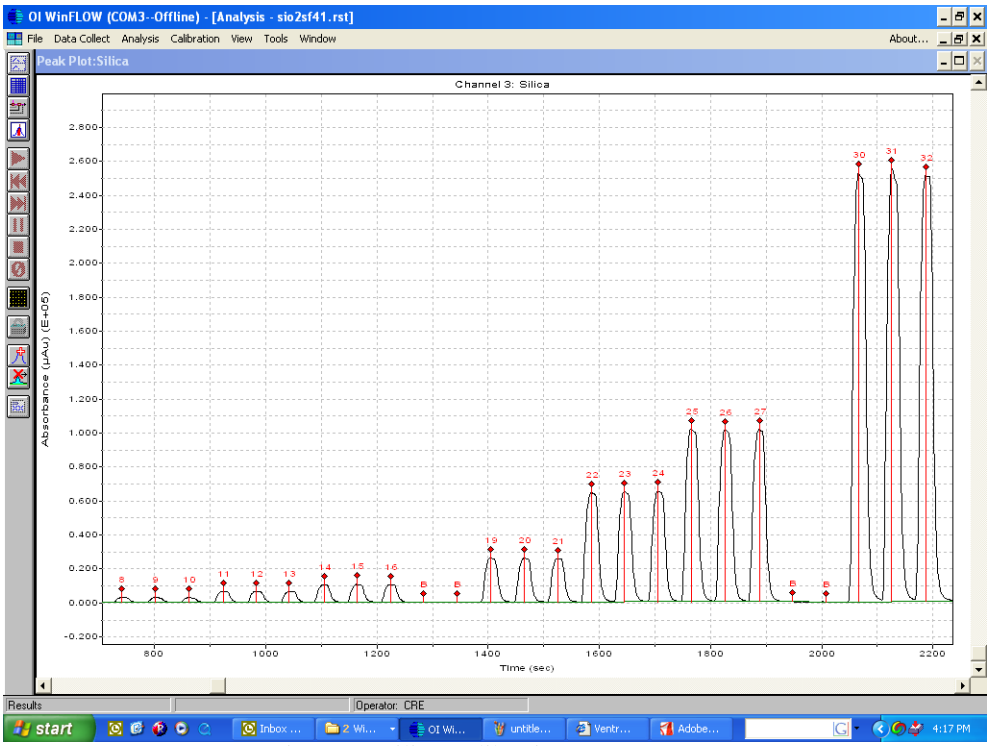


Figure 1. Silica Calibration (0.2–20 ppm)

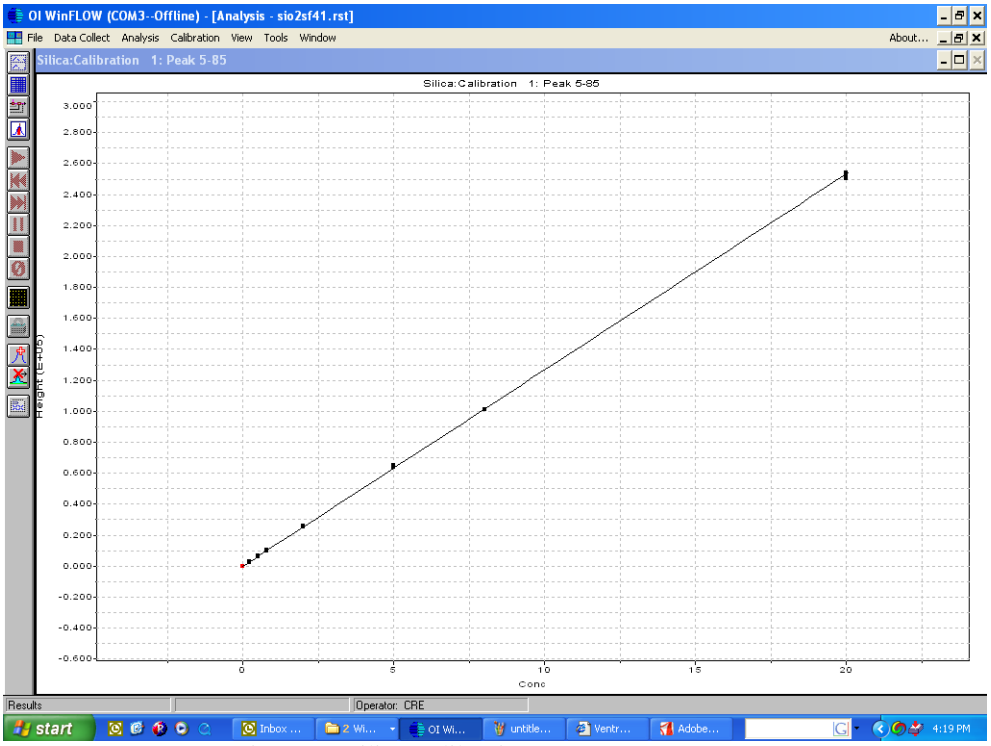


Figure 2. Silica Calibration Curve (0.2–20 ppm)

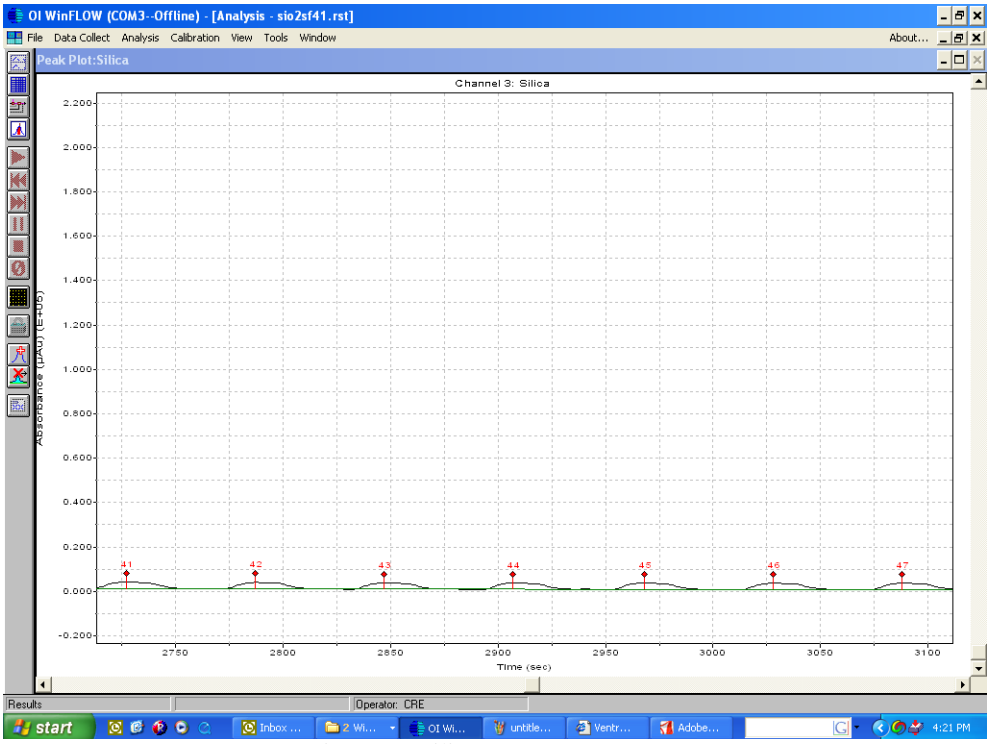


Figure 3. Silica MDL (at 0.2 ppm)

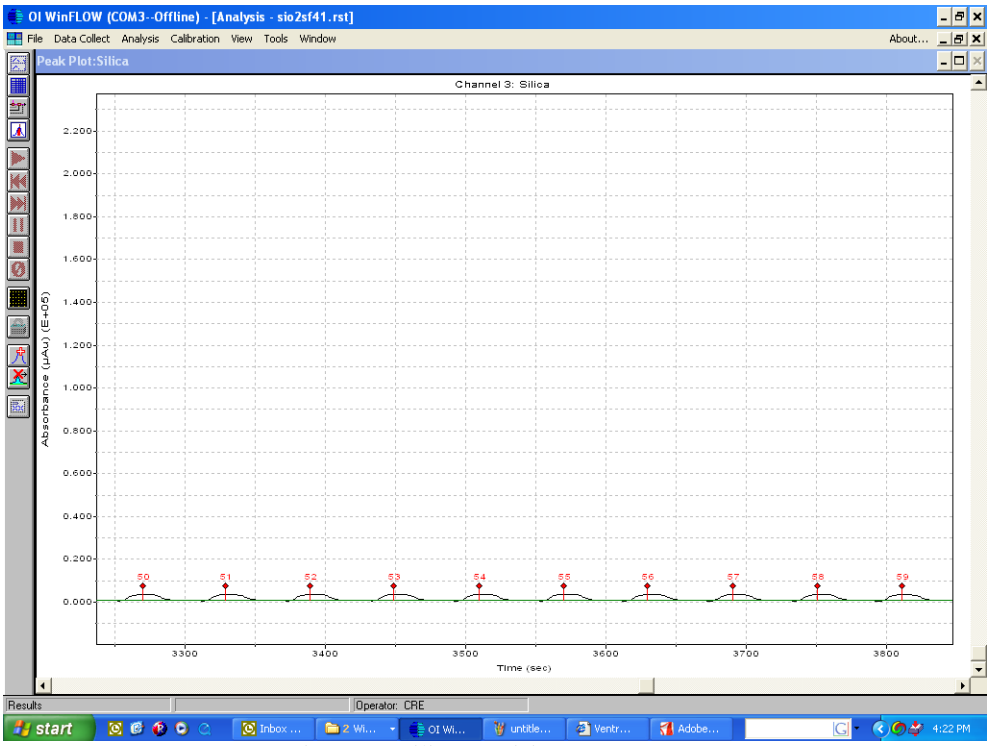


Figure 4. Silica Precision (at 0.2 ppm)

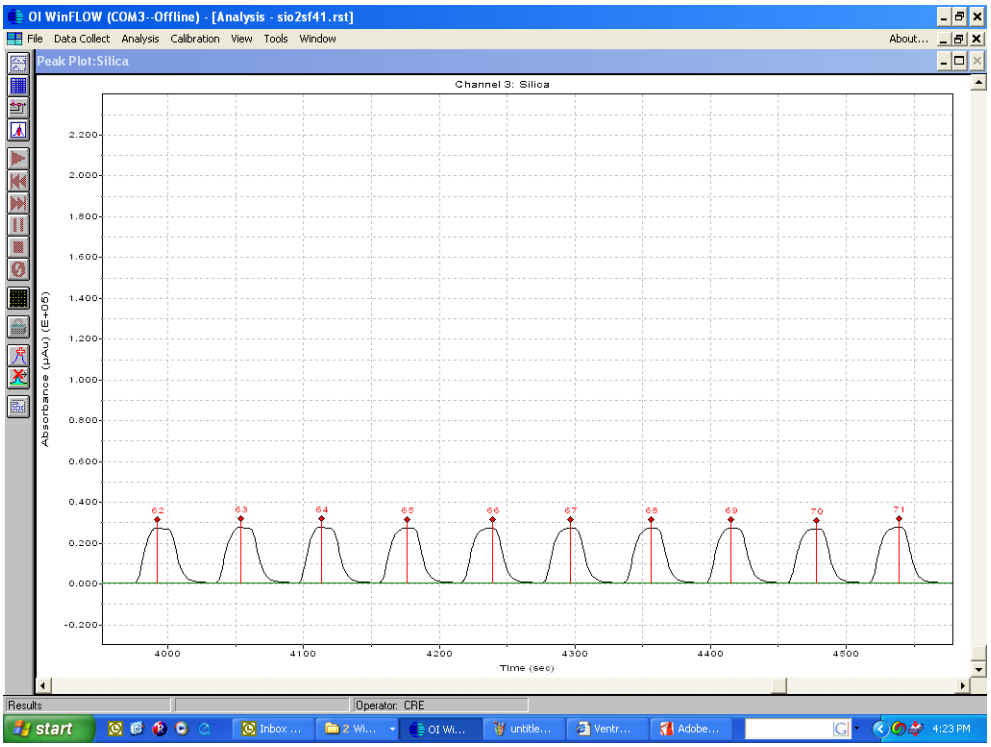


Figure 5. Silica Precision (at 2.0 ppm)

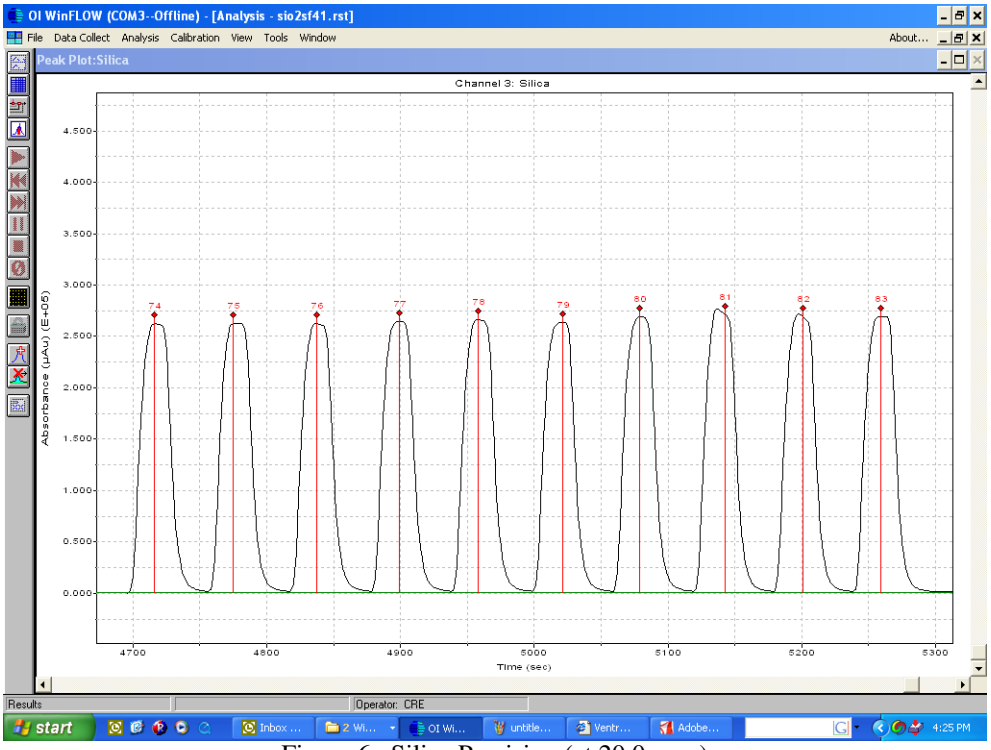


Figure 6. Silica Precision (at 20.0 ppm)

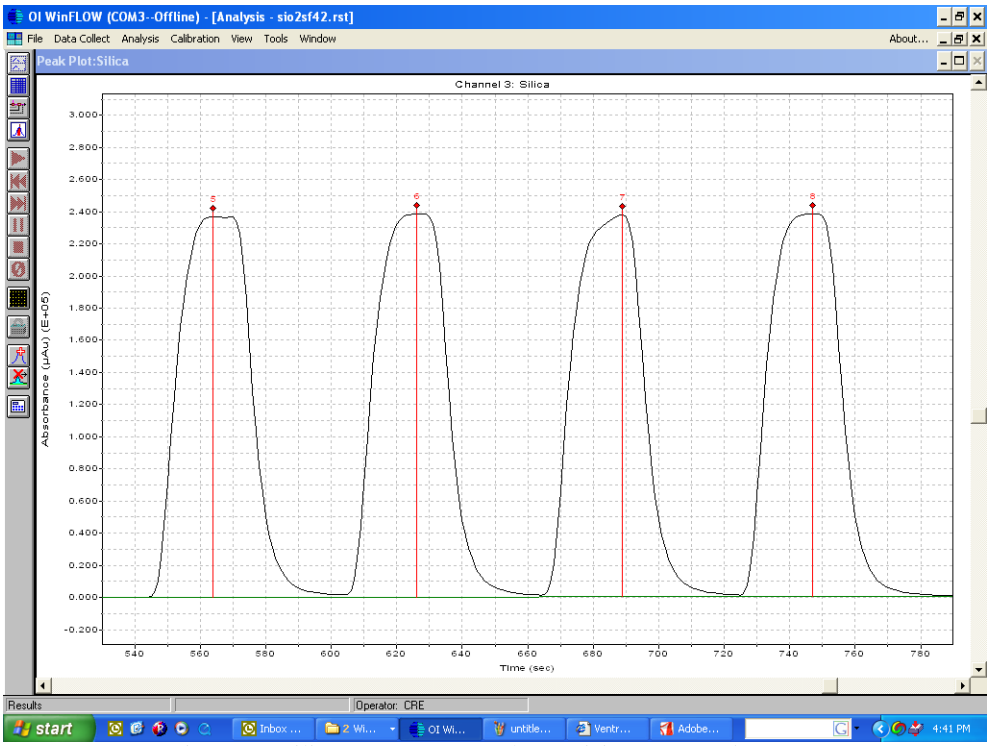


Figure 7. Silica ERA QC Sample Precision (ERA 15.9 ppm)

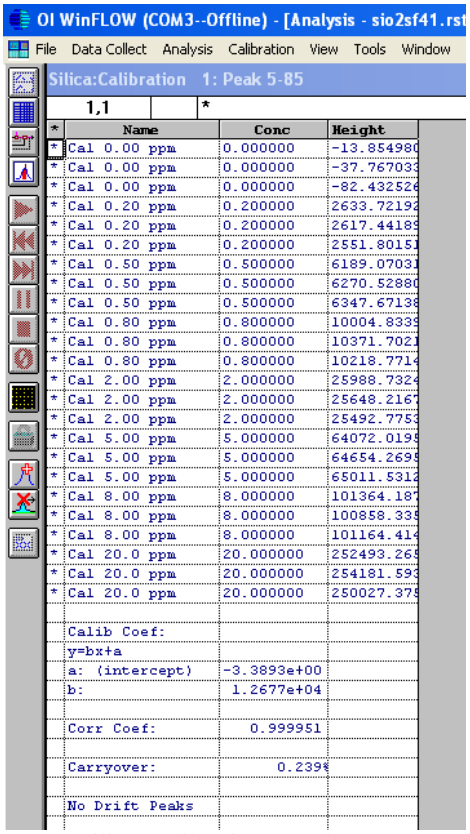


Figure 8. Silica Calibration Results (0.2–20 ppm)

Table 1. Silica Method Data

Parameter	Calibrant 0.2 mg/L	Calibrant 0.2 mg/L	Calibrant 2.0 mg/L	Calibrant 20.0 mg/L	ERA QC Standard 15.9 mg/L
Rep 1	0.2250	0.2074	2.1098	20.6590	18.6827
Rep 2	0.2158	0.2166	2.1317	20.6133	18.7346
Rep 3	0.2032	0.2118	2.1315	20.6234	18.7206
Rep 4	0.2086	0.2218	2.1083	20.7777	18.7433
Rep 5	0.2087	0.2197	2.1128	20.9041	—
Rep 6	0.2108	0.2252	2.1113	20.6465	—
Rep 7	0.2185	0.2240	2.1124	21.1473	—
Rep 8	—	0.2215	2.1104	21.2323	—
Rep 9	—	0.2268	2.0955	21.0734	—
Rep 10	—	0.2194	2.1501	21.1043	—
Average	0.2129	0.2194	2.1174	20.8781	18.7203
Standard Deviation	0.0073	0.0060	0.0157	0.2437	0.0755
% RSD	3.44	2.76	0.74	1.17	0.40
MDL	0.0230	—	—	—	—
% Accuracy	—	—	—	—	117.74%

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