

Method Abstract

Scope This method is used for the determination of silica in estuarine and coastal waters (seawater) according to USEPA Method 366.0. This method can also be used to analyze low-turbidity limnological and fresh water samples. 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 ascorbic acid to form molybdenum blue. The absorbance is measured at 815 nm. The quality of the analysis is assured through reproducible calibration and testing of the Segmented Flow Analysis (SFA).

Interferences Add oxalic acid to suppress interference from phosphate at concentrations greater than 150 $\mu\text{g/L}$. Remove hydrogen sulfide by boiling an acidified sample prior to analysis. Oxidation with bromine is also effective at removing sulfide. Large amounts of iron interfere. Interferences from fluoride at concentrations greater than 50 mg/L can be reduced by complexing with boric acid. Filter or centrifuge turbid samples prior to determination. 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 (Reference). Wash all glassware with 0.1 N HCl followed by two reagent water rinses, to prevent contamination from residual silicate.

Performance Specifications

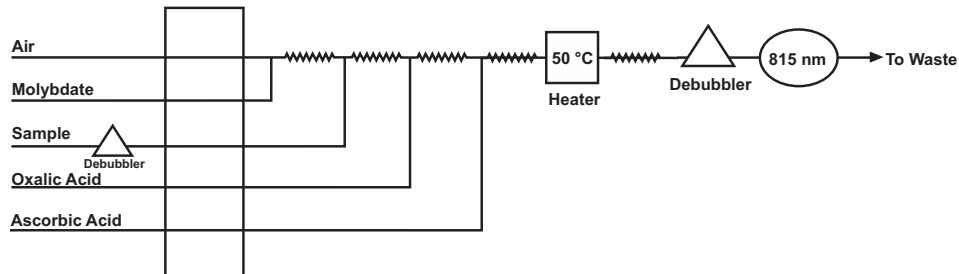
Range:	6.0–6,000 $\mu\text{g/L}$ Si
Throughput:	30 samples/hour
Precision	
30 $\mu\text{g/L}$	< 3% RSD
300 $\mu\text{g/L}$	< 3% RSD
3,000 $\mu\text{g/L}$	< 1% RSD
Method Detection Limit	1.0- $\mu\text{g/L}$ Si
Accuracy*	101%

*Undigested; ERA (Environmental Resource Associates) Wastewater Simple Nutrients Quality Control Sample.

Chemicals

Acetone, $\text{C}_3\text{H}_6\text{O}$	Sodium Bicarbonate, NaHCO_3
Ammonium Molybdate Tetrahydrate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$	Oxalic Acid, $\text{C}_2\text{H}_2\text{O}_4$
Ascorbic Acid, $\text{C}_6\text{H}_8\text{O}_6$	Sodium Chloride, NaCl
Deionized Water (ASTM Type I or II)	Sodium Metasilicate Pentahydrate, $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$
DOWFAX® 2A1 (Part #A000080)	Sulfuric Acid, concentrated, H_2SO_4
Hydrochloric Acid, concentrated, HCl	Sodium Bicarbonate, NaHCO_3
Magnesium Sulfate Heptahydrate, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	

Basic Flow Diagram



Selected References

Determination of Dissolved Silicate in Estuarine and Coastal Waters by Gas Segmented Continuous Flow Colorimetric Analysis. Methods for Chemical Analysis of Water and Wastewater; EPA/600/4-79-020; U.S. Environmental Protection Agency, Office of Research and Development, Environment Monitoring and Support Laboratory: Cincinnati, OH, 1997; Method 366.0

Keroleff, F. *Determination of Silicon, Method of Seawater Analysis*; Weinheim, Verlag Chemie, Germany, 1983, pp 174–187

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.

Methods for Determination of Inorganic Substances in Water and Fluvial Sediments; I-1700-85; U.S. Geological Survey; 552–558.

Water Quality–Determination of Soluble Silicates by Flow Analysis (FIA and CFA) and Photometric Detection. International Standard; ISO 16264:2002 (E); 1st ed; Geneva, Switzerland, 2003.

Figures

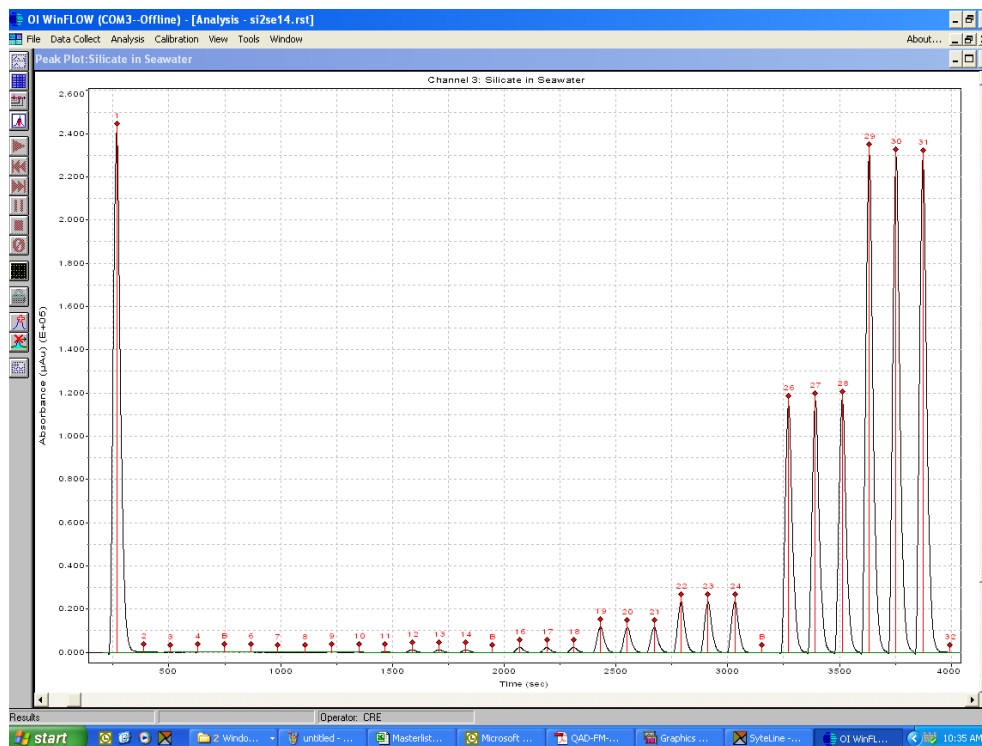


Figure 1. SiO_2 in Seawater Calibration (6.0–6,000 ppb)

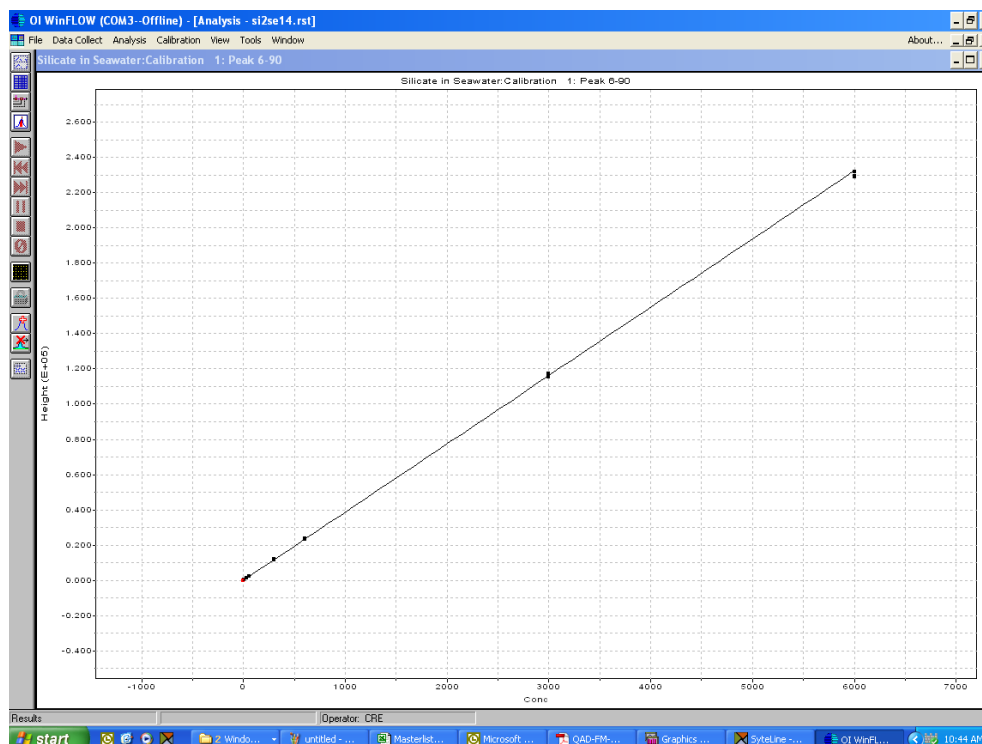


Figure 2. SiO_2 in Seawater Calibration Curve (6.0–6,000 ppb)

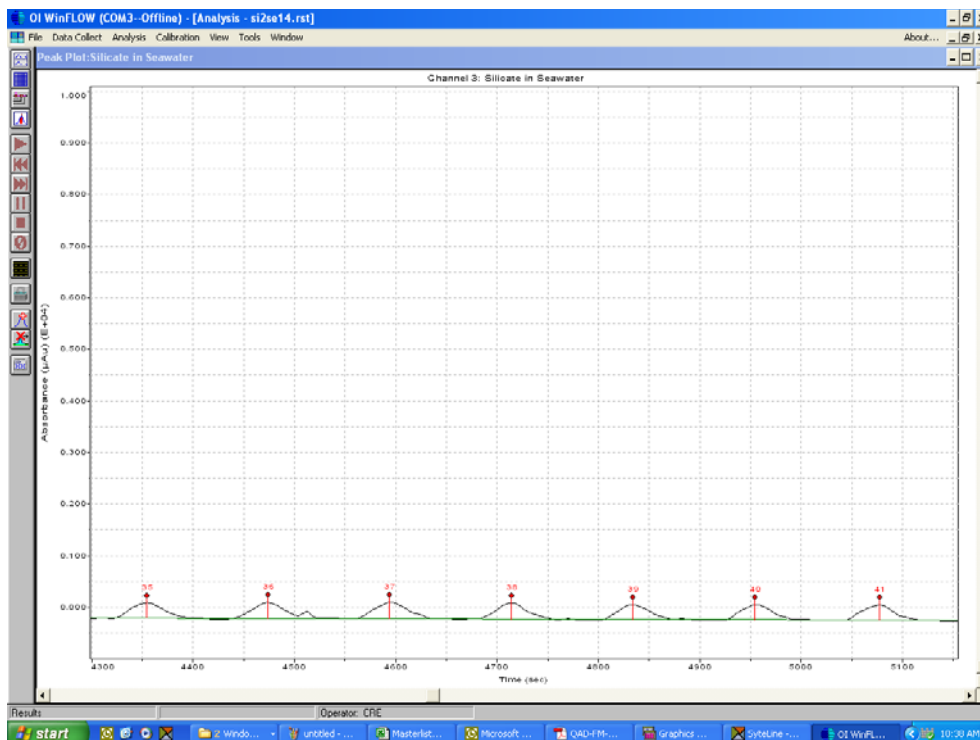


Figure 3. Method Detection Limit (at 6.0 ppb)

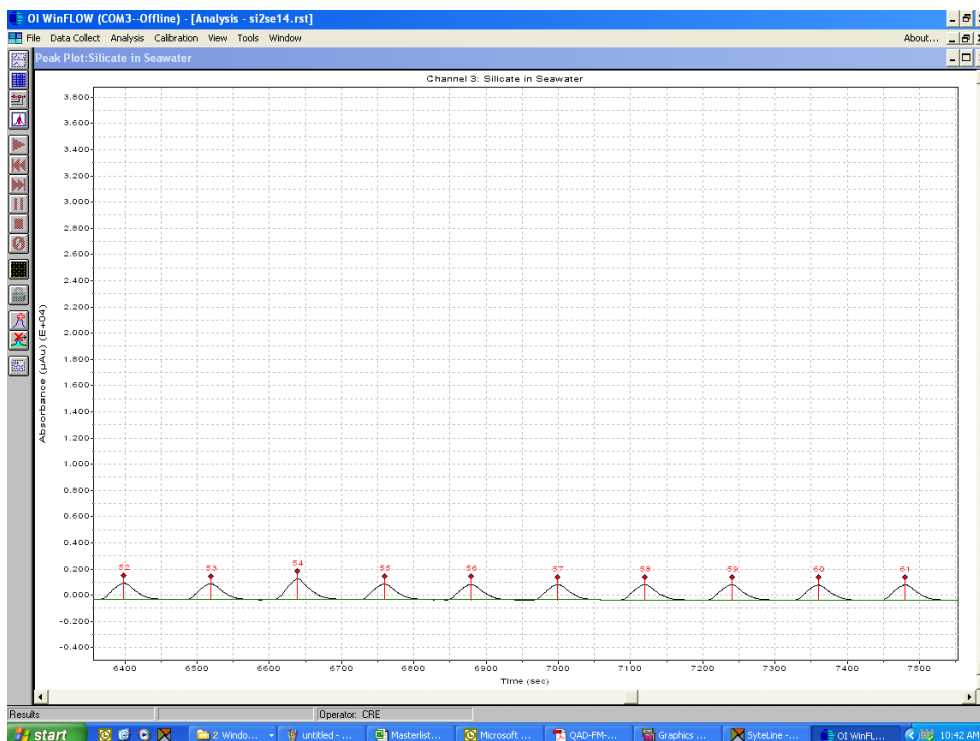


Figure 4. SiO₂ in Seawater Precision at 30 ppb (<3% RSD)

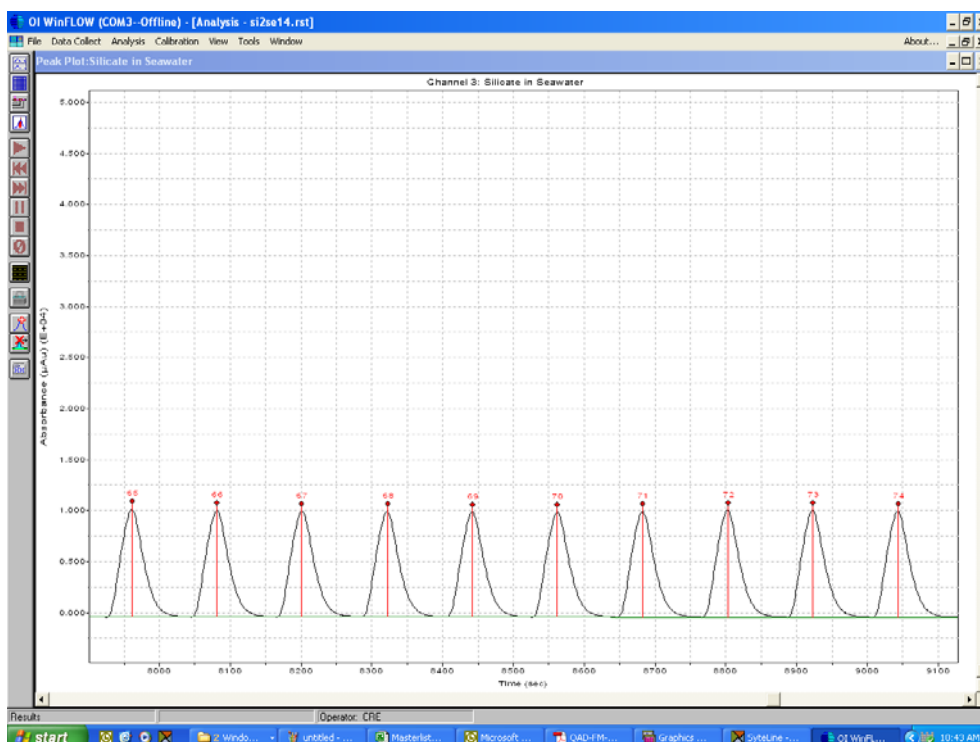


Figure 5. SiO_2 in Seawater Precision at 300 ppb (<3% RSD)

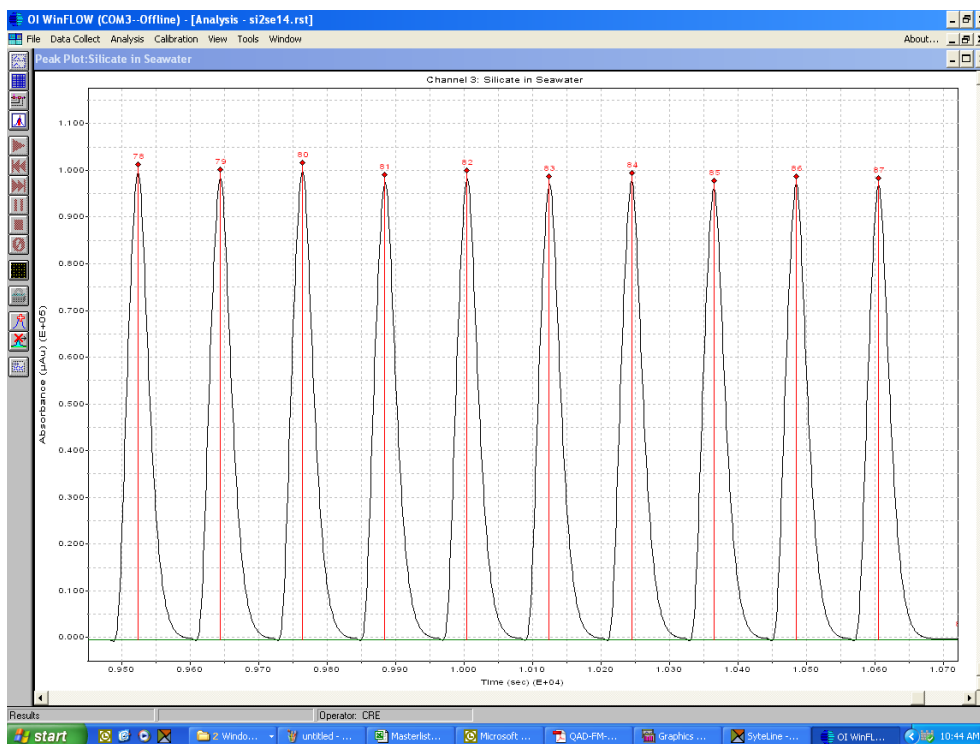


Figure 6. SiO_2 in Seawater Precision at 3,000 ppb (<1% RSD)

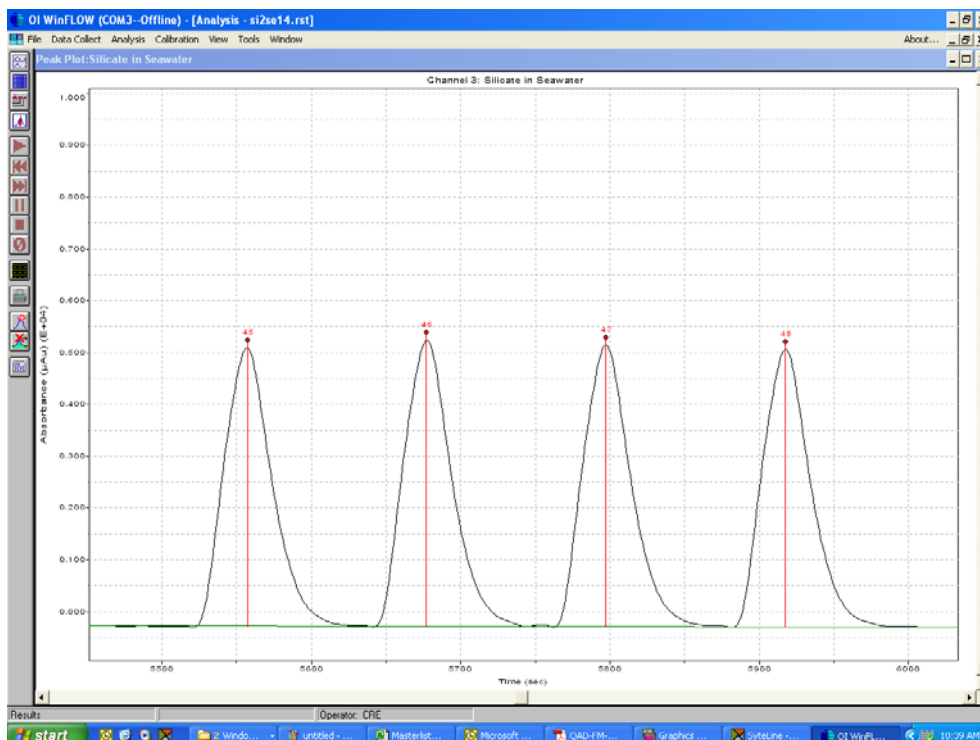


Figure 7. SiO₂ in Seawater ERA QC (140 ppb)

OI WinFLOW (COM3--Offline) - [Analysis - si2se14.rst]

Silicate in Seawater:Calibration 1: Peak 6-90

Name	Conc	Height
* Cal 0.00 ppb	0.000000	6.432583
* Cal 0.00 ppb	0.000000	-44.310345
* Cal 0.00 ppb	0.000000	-1.016330
* Cal 6.00 ppb	6.000000	304.501984
* Cal 6.00 ppb	6.000000	267.275757
* Cal 6.00 ppb	6.000000	307.375488
* Cal 30.0 ppb	30.000000	1293.65051
* Cal 30.0 ppb	30.000000	1291.14147
* Cal 30.0 ppb	30.000000	1266.82678
* Cal 60.0 ppb	60.000000	2369.75952
* Cal 60.0 ppb	60.000000	2357.18627
* Cal 60.0 ppb	60.000000	2327.85766
* Cal 300 ppb	300.000000	11898.7253
* Cal 300 ppb	300.000000	11694.2792
* Cal 300 ppb	300.000000	11648.2923
* Cal 600 ppb	600.000000	23412.4570
* Cal 600 ppb	600.000000	23319.5234
* Cal 600 ppb	600.000000	23591.9414
* Cal 3000 ppb	3000.000000	115120.421
* Cal 3000 ppb	3000.000000	116424.507
* Cal 3000 ppb	3000.000000	117352.873
* Cal 6000 ppb	6000.000000	231707.623
* Cal 6000 ppb	6000.000000	229261.013
* Cal 6000 ppb	6000.000000	228698.656

Calib Coef:	
y=bx+a	
a: (intercept)	7.2576e+01
b:	3.8728e+01
Corr Coef:	0.999959
Carryover:	0.08154
No Drift Peaks	

Figure 8. SiO₂ in Seawater Calibration Results (6.0–6,000 ppb)

Method Abstract

Table 1. Silica in Seawater Validation Results Table

Parameter	Calibrant 6.0 µg/L	Calibrant 30 µg/L	Calibrant 300 µg/L	Calibrant 3,000 µg/L	ERA QC Standard 140 µg/L
Rep 1	5.7681	29.9033	322.4898	3358.9480	136.8627
Rep 2	6.0669	29.8176	328.7360	3351.9590	140.7245
Rep 3	6.4683	29.4735	320.0856	3351.5459	138.6000
Rep 4	6.2505	29.0486	311.1887	3322.9573	136.4531
Rep 5	5.6430	28.6056	314.5254	3314.4287	—
Rep 6	5.7398	28.3165	309.6150	3387.8044	—
Rep 7	5.7597	28.2472	308.9940	3348.8958	—
Rep 8	—	28.1580	306.2426	3353.9912	—
Rep 9	—	27.7733	303.7422	3331.0391	—
Rep 10	—	28.1745	301.7883	3348.5505	—
Average	5.9566004	28.751816	312.74074	3347.012	138.1601
Standard Deviation	0.3109246	0.7586194	8.6756188	20.55232	1.946496
%RSD	5.2198337	2.6385095	2.7740609	0.6140498	1.40887
MDL	0.9763034	—	—	—	—
%Accuracy	—	—	—	—	101.09