

# Silica, colorimetric, molybdate blue

## Parameter and Code:

Silica, dissolved, I-1700-85 (mg/L as  $\text{SiO}_2$ ): 00955

### 1. Application

This method may be used to analyze water containing from 0.1 to 100 mg/L of silica. Samples containing more than 100 mg/L need to be analyzed by standard gravimetric procedures (American Society for Testing and Materials, 1984; Kolthoff and others, 1969).

### 2. Summary of method

2.1 Silica in solution as silicic acid or silicate has the property of reacting with ammonium molybdate in an acid medium to form the yellow silicomolybdate complex. The silicomolybdate complex is then reduced by sodium sulfite to form the molybdate blue color. The silicomolybdate complex may form in water as alpha and beta polymorphs (Strickland, 1952), which have absorbance maxima at different wavelengths. In order to favor the development of the beta form, the pH of the reaction mixture is reduced below 2.5 (Govett, 1961).

2.2 The possibility of having unreactive silica is greater in water containing high concentrations of silica than in water containing low concentrations of silica. When significant amounts of unreactive silica are known or suspected to be present, a 1-h digestion of 50-mL sample with 5.0 mL of 1.0M NaOH is suggested to make all the silica available for reaction with the molybdate reagent.

### 3. Interferences

Phosphate produces a similar molybdate complex under certain pH conditions. In the following determination, tartaric acid is added to suppress phosphate interference. Hydrogen sulfide and ferric and ferrous iron apparently interfere with the determination. Hydrogen sulfide may be removed by boiling an acidified

sample. The addition of disodium dihydrogen ethylenediamine tetraacetate ( $\text{Na}_2\text{EDTA}$ ) eliminates the effect of high concentrations of iron, and also complexes calcium and prevents precipitation of calcium sulfite.

### 4. Apparatus

4.1 *Spectrometer* for use at 700 nm.

4.2 Refer to the manufacturer's manual to optimize instrument.

### 5. Reagents

5.1 *Ammonium molybdate solution*: 49 g/L: Dissolve 52 g  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$  in water, adjust the pH to between 7 and 8 with 10M NaOH, and dilute to 1 L with demineralized water. Filter through 0.45- $\mu\text{m}$  membrane filter if necessary.

5.2 *Hydrochloric acid, 1.0M*: Mix 88 mL concentrated HCl (sp gr 1.19) with demineralized water and dilute to 1 L.

5.3 *Silica standard solution I*, 1.00 mL = 0.500 mg  $\text{SiO}_2$ : Dissolve 1.7655 g sodium metasilicate ( $\text{Na}_2\text{SiO}_3\cdot 5\text{H}_2\text{O}$ ) in demineralized water. Store in a plastic bottle.

5.4 *Silica standard solution II*, 1.00 mL = 0.005 mg  $\text{SiO}_2$ : Dilute 10.0 mL silica standard solution I to 1,000 mL with demineralized water. Store in a plastic bottle.

5.5  *$\text{Na}_2\text{EDTA}$  solution*, 10 g/L: Dissolve 10 g  $\text{Na}_2\text{EDTA}$  in demineralized water and dilute to 1 L.

5.6 *Sodium hydroxide solution*, 10M: Dissolve 400 g NaOH in demineralized water and dilute to 1 L.

5.7 *Sodium sulfite solution*, 170 g/L: Dissolve 170 g  $\text{Na}_2\text{SO}_3$  in demineralized water and dilute to 1 L.

5.8 *Tartaric acid solution*, 100 g/L: Dissolve 100 g  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$  in demineralized water and dilute to 1 L.

## 6. Procedure

6.1 Pipet a volume of sample containing less than 0.5 mg  $\text{SiO}_2$  (10.0 mL max) into a 50-mL beaker, and adjust the volume to 10.0 mL.

6.2 Pipet a demineralized water blank and sufficient standards into 50-mL beakers, and adjust the volume of each to 10.0 mL.

6.3 Add to each solution, with stirring, 5.0 mL 1.0M HCl, 5.0 mL  $\text{Na}_2\text{EDTA}$  solution, and 5.0 mL ammonium molybdate solution.

6.4 After 5 min add 5.0 mL tartaric acid solution and mix.

6.5 Add 10.0 mL  $\text{Na}_2\text{SO}_3$  solution and mix.

6.6 Allow to stand approx 30 min. The color is stable for several hours.

6.7 Determine the absorbance of each test sample and standards against the blank.

## 7. Calculations

7.1 Determine milligrams of silica in each sample from a plot of absorbances of standards.

7.2 Determine the concentration of dissolved silica in milligrams per liter as follows:

$$\text{SiO}_2 \text{ (mg/L)} = \frac{1,000}{\text{mL sample}} \times \text{mg SiO}_2 \text{ in sample.}$$

## 8. Report

Report silica, dissolved (00955), concentrations as follows: less than 10 mg/L, one decimal; 10 mg/L and above, two significant figures.

## 9. Precision

9.1 Precision for dissolved silica for 35 samples within the range of 2.15 to 24.4 mg/L may be expressed as follows:

$$S_T = 0.065X + 0.10$$

where

$S_T$  = overall precision, milligrams per liter, and

$X$  = concentration of silica, milligrams per liter.

The correlation coefficient is 0.8053.

9.2 Precision for dissolved silica for four of the 35 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (mg/L)	Relative standard deviation (percent)
19	2.15	16
11	9.08	5
6	10.2	15
24	24.4	7

## References

- American Society for Testing and Materials, 1984, Annual book of ASTM standards, section 11, water: Philadelphia, v. 11.01, p. 613-8.
- Govett, G. J. S., 1961, Critical factors in the colorimetric determination of silica: *Analytica Chimica Acta*, v. 25, p. 69-80.
- Kolthoff, I. M., Sandell, E. B., Meehan, E. J., and Bruckenstein, S., 1969, *Quantitative chemical analysis*, (4th ed.): New York, Macmillan, 1199 p.
- Strickland, J. D. H., 1952, The preparation and properties of silicomolybdic acid; I. The properties of alpha silicomolybdic acid: *American Chemical Society Journal*, v. 74, p. 862-7.