Silica, colorimetric, molybdate blue, automated-segmented flow

Parameter and Code:
Silica, dissolved, I-2700-85 (mgIL as SiO_2): 00955

1. Application

This method may be used to determine concentrations of silica in surface, domestic, and industrial water in the range from 0.1 to 60 mgIL. Two analytical ranges are used: from 0.1 to 6.0 mgIL and from 1.0 to 60 mgIL.

2. Summary of method

2.1 Silica reacts with molybdate reagent in acid media to form a yellow silicomolybdate complex. This complex is reduced by ascorbic acid to form the molybdate blue color. The silicomolybdate complex may form either as an alpha or beta polymorph or as a mixture of both. Because the two polymorphic forms have absorbance maxima at different wavelengths, the pH of the mixture is kept below 2.5, a condition that favors formation of the beta polymorph (Govett, 1961; Mullen and Riley, 1955; Strickland, 1952).

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 su8pected to be present, a 1-h digestion of a 50-mL sample with 5.0 mL of 1.0*M* NaOH is suggested as a means of making all the silica available for reaction with the molybdate reagent.

3. Interferences

Interference from phosphate, which forms a phosphomolybdate complex, is suppressed by the addition of oxalic acid. Hydrogen sulfide must be removed by boiling the acidified sample prior to analysis. Large amounts of iron interfere.

4. Apparatus

4.1 *Technicon AutoAnalyzer II,* consisting of sampler, cartridge manifold, proportioning pump, colorimeter, voltage stabilizer, recorder, and printer.

4.2 With this equipment the following operating conditions have been found satisfactory for the range from 0.1 to 60 mg/L:

Absorption cell ---------- 15 mm
Wavelength ---------- 660 nm
Cam ---------- 60/h (6/1)

5. Reagents

5.1 *Ammonium molybdate solution,* 9.4 g/L: Dissolve 5 g $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ in 0.05***M*** H_2S0_4 and dilute to 500 mL with 0.05***M*** H_2S0_4. Filter and store in an amber plastic container.

5.2 *Ascorbic acid solution,* 17.6 g/L: Dissolve 17.6 g ascorbic acid in 500 mL demineralized water containing 50 mL acetone. Dilute to 1 L with demineralized water. Add 0.5 mL Levor IV solution. The solution is stable for 1 week if stored at 4°C.

5.3 *Levor IV solution:* Technicon No. 21-0332 or equivalent.

5.4 *Oxalic acid solution,* 50 g/L: Dissolve 50 g oxalic acid in demineralized water and dilute to 1 L.

5.5 *Silica standard solution,* 1.00 mL = 0.500 mg $Si0_2$: Dissolve 1.7655 g sodium metasificate ($Na_2SiO_3\cdot 5H_2O$) in demineralized water and dilute to 1,000 mL. Store in a plastic bottle.

5.6 *Silica working standards:* Prepare a blank and 1,000 mL each of a series of silica working standards by appropriate quantitative dilution of silica standard solution as follows:

Silica standard solution (mL)	Silica concentration (mg/L)
0.0	0.0
.5	0.25
1.0	.5
2.0	1.0
12.0	6.0
20.0	10
40.0	20
80.0	40
120.0	60

5.7 *Sulfuric acid, 0.05M: Cautiously* add 2.8 mL concentrated H_2S0_4 (sp gr 1.84) to demineralized water and dilute to 1 L.

6. Procedure

6.1 Set up manifold (fig. 43).

6.2 Allow colorimeter and recorder to warm up for, at least 30 min.

6.3 Adjust baseline to read zero scale divisions on the recorder with all reagents, but with demineralized water in the sample line.

6.4 Place a complete set of standards and a blank in the first positions of the first sample tray, beginning with the most concentrated standard. Place individual standards of differing concentrations in approximately every eighth position of the remainder of this and subsequent sample trays. Fill remainder of each tray with unknown samples.

6.5 Begin analysis. When the peak from the most concentrated working standard (6.0 or 60 mg/L) appears on the recorder, adjust the STD CAL control until the flat portion of the curve reads full scale.

7. Calculations

7.1 Prepare an analytical curve by plotting the height of each standard peak versus its respective silica concentration.

7.2 Compute the concentration of dissolved silica in each sample by comparing its peak height to the analytical curve. Any baseline drift that may occur must be taken into account when computing the height of a sample or standard peak.

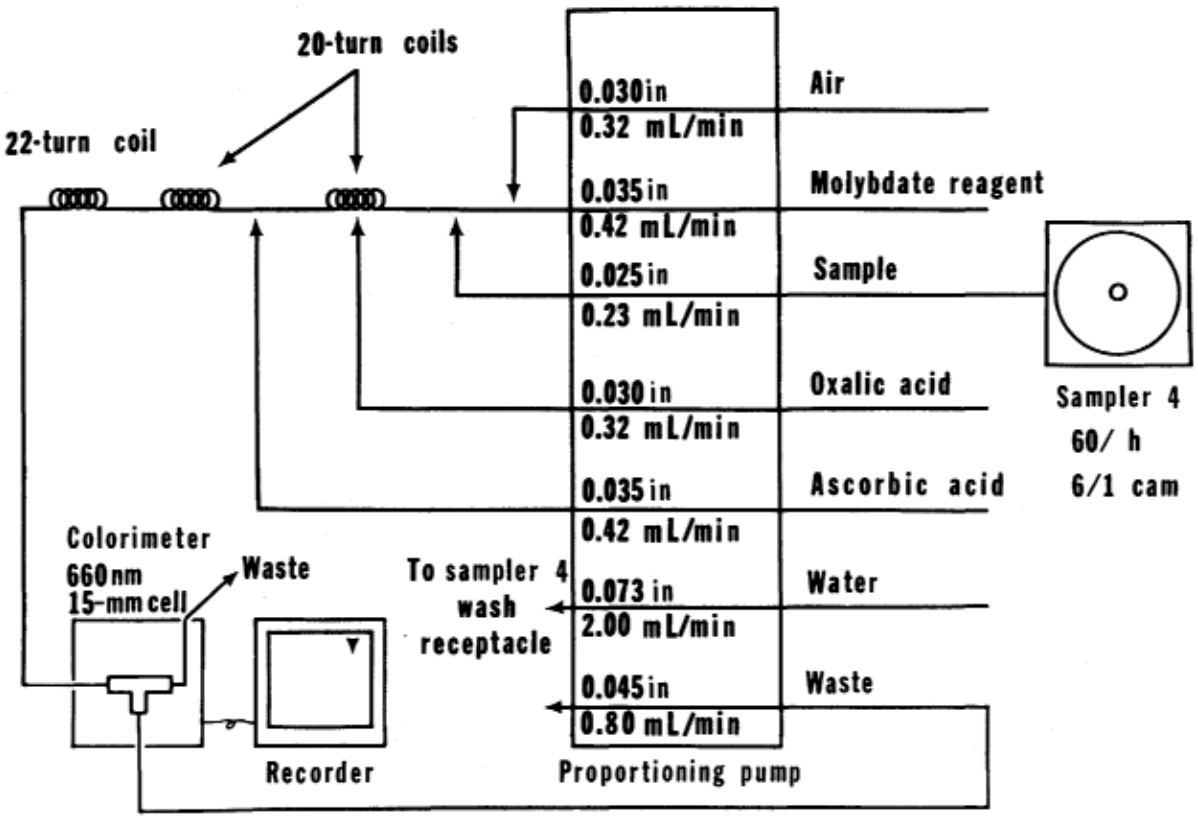

Figure 43.—Silica, molybdate manifold

8. Report

Report silica, dissolved (00955), concentrations as follows: less than 10 mg/L, nearest 0.1 mg/L; 10 mg/L and above, two significant figures.

9. Precision

9.1 The standard deviation for dissolved silica within the range of 4.70 to 17.4 mg/L for 20 samples was found to be independent of concentration. The 95-percent confidence interval for the average standard deviation of 1.10 mg/L ranged from 0.97 to 1.26 mg/L.

9.2 Precision for dissolved silica for four of the 20 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (mg/L)	Relative standard deviation (percent)
5	4.70	7
8	8.95	23
6	10.6	6
5	17.4	7

References

Govett, G. J. S., 1961, Critical factors in the colorimetric determination of silica: Analytica Chimica Acta, v. 25, p. 69-80.

Mullen, J. B., and Riley, J. P., 1955, The colorimetric determination of silicate with special reference to sea and natural waters: Analytica Chimica Acta, v. 12, p. 162-76.

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.