Dissolved silica determination by molybdenum blue method

This method (Hu et al., 2005) is based on the established procedure proposed by Truesdale and Smith (1976), with an improved technique to eliminate the interference of phosphate (and arsenate).

NOTE: Use acid-washed volumetric flasks for preparation of all the solutions

Reagents

Sulfuric acid solution (5 M H_2SO_4)

Prepare a 500-mL solution by adding 140 mL of concentrated H_2SO_4 to Milli-Q water (~300 mL) in a volumetric flask. Allow the solution to cool down first, and then add Milli-Q water until the calibration mark. The solution is stable indefinitely if stored at 4 °C.

Hydrochloric acid (0.25 M HCl)

Prepare a 500-mL solution by adding 10.4 mL of concentrated HCl to Milli-Q water in a volumetric flask. The solution is stable indefinitely if stored at 4 °C.

Ammonium molybdate solution (5% $(NH_4)_6M_7O_{24}{\cdot}4H_2O$)

Dissolve 5.6 g of ammonium molybdate ($NH_4M_7O_{24}{\cdot}4H_2O$) in 100 mL Milli-Q water. The solution is stable indefinitely if stored at 4 °C in a plastic container. Discard if precipitates form.

Citric acid solution

Dissolve 36.5 g of citric acid in 50 mL Milli-Q water. Stable for a month if stored at 4 °C in a plastic container. Discard if precipitates form.

Ascorbic acid solution (in 5 M H_2SO_4)*

Dissolve 0.15 g of ascorbic acid in 50 g of 5 M H_2SO_4 solution. Must be prepared fresh for each analysis.

**You can adjust the volume of this solutions based on the number of samples you will analyze (i.e., 1 mL is needed for each blank/standard/sample).*

Standards

Primary silica standard (3.78 g/L)

Dissolve 0.8934 g of sodium silicate nonahydrate ($Na_2SiO_3{\cdot}9H_2O$; must be kept in a desiccator) in 50 mL Milli-Q water in a volumetric flask (must be plastic). This solution is stable for many months at 4 °C.

Standard solutions

Prepare the standard solutions by diluting the secondary standard to a volume of 50 mL with Milli-Q water. Please use plastic volumetric flasks.

Silica calibration standards (mg/L)	Volume of silica standard (mL)
5	0.066
10	0.132
25	0.331

Sample analysis

1. Switch on the instrument 15 minutes before the measurements and set wavelength to 720 nm. This is to enable the bulbs in the spectrometer to warm up.

Prepared by Dr. Jeffrey Paulo H. Perez
E-mail: jhperez2@alum.up.edu.ph

2. Prepare the spectrophotometric cuvettes, always hold them from the opaque sides. Cuvettes must be inserted in the same orientation all the time, there is a small arrow at the top to help ensure they are used the same way around.
3. Add 3 mL of 0.25 M HCl to a plastic vial.
4. Add 0.5 mL of blank / standards / sample to the vials.
5. Add 0.4 mL 5% $(NH_4)_6M_7O_{24}{\cdot}4H_2O$ solution to the vials. Wait for 10 min.
6. Add 0.2 mL citric acid solution, and then 1 mL ascorbic acid to the vials. Wait for 40 min (full color development).
7. Transfer 1 mL of the blue colored solution to a cuvette and measure the absorbance.

Reference:

Hu C., Huang J., Fang N., Xie S., Henderson G. M. and Cai Y. (2005) Adsorbed silica in stalagmite carbonate and its relationship to past rainfall. *Geochim. Cosmochim. Acta* **69**, 2285-2292.

Truesdale V. W. and Smith C. J. (1976) The automatic determination of silicate dissolved in natural fresh water by means of procedures involving the use of either α- or β-molybdosilicic acid. *Analyst* **101**, 19-31.

Prepared by Dr. Jeffrey Paulo H. Perez
E-mail: jhperez2@alum.up.edu.ph