

Silica

FIAlab standard method for Silica Assay using the FIAlab-2500/2600/2700 system.

Assay	Typical Throughput	Concentration Range	Notes
Silica (Mid to High))	60 samples/hour	0.5 to 300 mg /L	1 cm flow cell
Silica (Low)	60 samples/hour	0.05 to 30 mg /L	10 cm flow cell
Silica (Ultra Low)	40 samples/hour	0.02 to 6 mg /L	50 cm flow cell

Principle:

Soluble silica species react with molybdate under acidic conditions to form a yellow silicamolybdate complex. This complex is subsequently reduced with ANSA (1-amino-2-naphthol-4-sulfonic acid) and bisulfite to form a heteroploy blue complex which has an absorbance maximum at 820 nm.

Comments:

A heater is necessary, and should be set to 60 C. The flow rate of the pump should be set to 55. T. Recommended wavelengths: 800 nm for the primary and 500 nm for reference.

Make the sample loop from ten inches of 0.03" ID tubing.

Low Concentration Assays: For low concentrations (below 2 ppm), consider using the 10 cm flow cell. The sample loop size can be increased as well.

Carrier: DI Water. The quality of the DI water should be watched, since silica is often encountered as a contaminant in water. Silica contamination typically appears as the filters on the DI apparatus approach the end of their life cycle.

Interferences:

To some degree, phosphate creates a positive interferes with the silica assay.

Reagents:

Carrier:

DI Water. The quality of the DI water should be watched, since silica is often encountered as a contaminant in water. Silica contamination typically appears as the filters on the DI apparatus approach the end of their life cycle.

1-Liter Degassed DI Water.

Reagent 1: Indicator solution.

250 mL of 0.02 M Ammonium Molybdate solution.

750 mL Degassed DI Water.

A volume of ~1 M Hydrochloric Acid corresponding to 0.33 mol acid equivalents. For example, 330 mL of 1.000 N HCl, or 333 mL of 0.990 N HCl.

0.02 M Ammonium Molybdate solution: 25.0 grams Ammonium molybdate tetra-hydrate [1235.81 FW]. Dissolve in water under gentle warming and dilute to 250 mL. Filter. Make sure the filter is free of silica (do not use glass filters). **Adjust pH** to 7-8 with silica-free NaOH (approx. 7-8 mL of 50% NaOH). Store in a plastic container.

Reagent 2: Reducing Agent & Phosphate Suppressant solution.

250 mL of 0.15 M Oxalic Acid solution.

750 mL of [0.23 M ascorbic acid solution with 1.3 mL sodium dodecyl sulfate] solution.

0.15 M Oxalic Acid solution: 18.75 grams Oxalic acid dihydrate [126.07 FW]. Dissolve in water and dilute to 250 mL. Store in a plastic container.

0.23 M Ascorbic Acid with 1.3 g/L Sodium Dodecyl Sulfate: 30 grams Ascorbic acid [176.12 FW] plus 1 grams Sodium dodecyl sulfate [288.38 FW] surfactant (Sigma-Aldrich 436143-25G)

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0.75 Liter Degassed DI Water. Place the ascorbic acid into a 1-liter container and mix with 500 cc of DI water until dissolved. Add the sodium dodecyl sulfate and mix slowly (prevent foaming) until dissolved. Fill the container to 750 mL. Transfer solution into an airtight light sensitive glass bottle for maximum longevity. Minimize exposure to air and prepare fresh weekly since this solution is unstable

Standards:

500ml S65142-500 (Silica standard)
Source: 727-524-7732 - www.exaxol.com