

THE COLORIMETRIC DETERMINATION OF SILICA IN AQUEOUS SOLUTIONS

UOP Method 792-78

SCOPE

This method is for the determination of micro amounts of silica in aqueous solutions. Not all silica species can be determined by this method, but the more common water-soluble forms can be determined (see Note 2). The concentration of silica should not exceed 0.7 micrograms per milliliter in the final colored solution.

OUTLINE OF METHOD

A portion of the aqueous solution is weighed into a tared 100-ml volumetric flask. The hydrogen ion concentration is adjusted and a blue color is developed by the addition of solutions of ammonium molybdate, oxalic acid and stannous chloride. The intensity of the blue color is determined by use of a colorimeter or spectrophotometer.

APPARATUS

Balance, analytical, Ainsworth Model A-4 or equivalent (Fisher Scientific Co.)

Beakers, 50- and 200-ml

Bottles, plastic, 100-ml

Cell, absorption, 2 cm for colorimeter

Colorimeter, analytical, Klett-Summerson or equivalent (Sargent-Welch Co.)

Filter, Klett-Summerson No. 64 (640 nm) or equivalent

Flasks, volumetric, 100-ml

Medicine droppers

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

Pipets, transfer, 1-, 2-, 3-, 5- and 7-ml

REAGENTS AND MATERIALS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society when such specifications exist, unless otherwise stated or specified. References to water mean deionized or distilled water. Unqualified references to solutions means aqueous solutions.

Ammonium molybdate, tetra hydrate, crystal

Ammonium molybdate solution, 10%. Dissolve 10 g of ammonium molybdate tetra hydrate in approximately 70 ml of water. The solution is achieved with mild heating and constant stirring. Cool the solution to room temperature. Add 1 ml of concentrated ammonium hydroxide and dilute to 100 ml. Filter out any turbidity. Store in a plastic bottle.

Hydrochloric acid, concentrated

Hydrochloric acid, 1:1 water solution. Add 50 ml of concentrated hydrochloric acid to 50 ml of water. Store in a plastic bottle.

Oxalic acid dihydrate, crystals

Oxalic acid solution, 10%. Dissolve 10 g of the oxalic acid dihydrate crystals in water and dilute to 100 ml. Store in a plastic bottle.

Silica solution, 100-ppm. Select a water glass whose silica content is accurately known, and prepare the calibrating solution by proper dilution. The diluted solution must be made basic by the addition of a small amount of sodium hydroxide to prevent polymerization of the silica. Store in a plastic bottle.

Silica solution, 10-ppm. Dilute exactly 10 ml of the 100 ppm solution to 100 ml

Stannous chloride, dihydrate, crystal

Stannous chloride solution, 25%. Weigh 25 g of the stannous chloride crystals into a 200-ml beaker. Add 25 ml of concentrated hydrochloric acid and heat until the crystals are dissolved. Dilute the solution to 100 ml with water. Store in a plastic bottle.

Stannous chloride solution, 0.25%. Pipet 1 ml of the 25% stannous chloride solution into a 100-ml volumetric flask containing approximately 70 ml of water and dilute to the mark. A fresh solution must be made on the day that an analysis is to be run.

PROCEDURE

Preparation of Calibration Curve

Pipet exactly 1, 3, 5 and 7 ml of the 10-ppm silica consecutively into each of four 100-ml volumetric flasks, retaining one 100-ml volumetric flask for the reagent blank. Dilute each of the five solutions to approximately 50 ml. Add 2 ml of the 1:1 hydrochloric acid solution to each flask. Add 3 ml of 10% ammonium molybdate solution to each flask and allow them to stand for at least 10 minutes, but no longer

than 15 minutes. Add 3 ml of the 10% oxalic acid solution to each flask and allow this to stand for at least two minutes, but no longer than five. Using a medicine dropper, add 10 drops of the 0.25% stannous chloride solution to each flask. Dilute to the mark and read the color intensity on a colorimeter at 640 nanometers using a 2-cm adsorption cell. Plot the micrograms of silica per 100 ml of solution versus the colorimeter reading.

Analysis of Sample

Weigh to the nearest 0.1 mg a portion of the aqueous sample, that contains no more than 70 μ g of silica, into a dry, tared 100-ml volumetric flask. Dilute to approximately 50 ml with water. Follow the procedure as described for the preparation of the calibration curve. From the calibration curve convert the colorimeter reading to micrograms of silica per 100 ml of solution and calculate the parts per million silica in the sample.

CALCULATIONS

$$\text{Silica, ppm} = \frac{A}{W}$$

where:

A = weight of silica from calibration curve, μ g
W = sample weight, g

NOTES

1. Develop and read the color of the standards and samples at the same time. The color stability is time dependent. Do not exceed a concentration of 0.7 μ g of silica per milliliter of final colored solution, as full color development is not achieved at greater concentrations.
2. All of the silica in the silica source selected to prepare the 100 ppm silica solution must be reactive with ammonium molybdate. If it is not, an erroneous calibration curve will result. The silica in sodium silicate solutions is in the reactive form. Other suitable silica sources are sodium silicate reagent crystals or pure silica fused with anhydrous sodium carbonate in a platinum crucible.

PRECISION

Based on 6 pairs of duplicate analyses, the estimated standard deviation is 0.6 ppm in the 5 to 50 ppm range.

TIME FOR ANALYSIS

The average elapsed time is approximately 1 hour for a single sample. When run in groups of 4, the labor requirement is 0.5 man-hours per sample.

SUGGESTED SUPPLIERS

Fisher Scientific Co., 1600 West Glenlake Ave., Itasca, IL 60143
 Sargent-Welch, 7300 North Linder, Skokie, IL 60076