

SILICA AND SODIUM IN WATER GLASS

UOP Method 627-85

SCOPE

This method is for determining silica and sodium, or sodium oxide, in water glass using a volumetric and gravimetric technique. The range of sodium ordinarily determined is 3 to 10%, and that of silica is 15 to 30%. Both sodium and silica may be determined at higher levels. The lower limit of detection for sodium is 0.02% and for silica 0.5%. Results are reported in mass-%.

OUTLINE OF METHOD

To determine the sodium content, a weighed sample is diluted with warm water and titrated to a methyl purple endpoint with 0.2M hydrochloric acid. The alkalinity can be calculated as sodium oxide or sodium.

Silica may be determined by a rapid control procedure or by a more precise technique. To rapidly determine the silica content, a weighed sample is treated with hydrochloric acid and then heated. The silicic acid residue is then filtered, washed, ignited and weighed. The procedure assumes that all of the residue is silica and that none of the silica is lost in the filtrate.

The more precise procedure requires that the filtrate from the previously described filtration be further treated for the recovery of additional silica. The total silica plus impurities obtained as a combined residue are further treated with hydrofluoric and sulfuric acids to volatilize the silica and thereby allow the gravimetric determination of the silica content by difference. This second procedure assumes that any impurities present are not volatilized during the heating.

APPARATUS

Balance, readability, 0.1-mg

Beakers, 400-mL

Bottle, dropping, with ground pipet stopper, 60-mL capacity, Sargent-Welch Scientific, Cat. No. S-8755-D, or equivalent

Buret, funnel top, 50-mL capacity, Teflon stopcock, Sargent-Welch Scientific, Cat. No. S-10621-C, or equivalent

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).

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Burner, Fisher, high temperature, mixed and natural gas, Sargent-Welch Scientific, Cat. No. S-12196, or equivalent

Cylinder, graduated, 100-mL

Crucibles, platinum, 40-mL capacity, Engelhard Industries, *Figure 202*, or equivalent

Crucible covers, Coors, Sargent-Welch Scientific, Cat. No. S-23720-G, or equivalent

Desiccator, Sargent-Welch Scientific, Cat. No. S-34840-L, or equivalent

Flask, volumetric, one-liter, Sargent-Welch Scientific, Cat. No. S-34840-L, or equivalent

Funnels, analytical, 60° fluted, long stem, 75-mm diameter, Sargent-Welch Scientific, Cat. No. S-35335-E, or equivalent

Furnace, muffle, capable of operation at 1000 C, Sargent-Welch Scientific, Cat. No. S-36968-F, or equivalent

Heater, infrared lamp, 250 W with receptacle, porcelain, Sargent-Welch Scientific, Cat. Nos. S-40768 and S-40769, respectively, or equivalent

Hot plate, Sargent-Welch Scientific, Cat. No. S-41000-B, or equivalent

Steam bath, Sargent-Welch Scientific, Cat. No. S-84405-A, or equivalent

Stirring apparatus, magnetic, variable speed, with Teflon stirring bar, Sargent-Welch Scientific, Cat. No. S-76491-10, or equivalent

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 are available, unless otherwise specified. References to water mean deionized or distilled water.

Filter paper, Whatman No. 42, Sargent-Welch Scientific, Cat. No. S-33175-F, or equivalent

Hydrochloric acid, concentrated, 37%

Hydrochloric acid, 1:1. Dilute the concentrated acid with an equal volume of water.

Hydrochloric acid, 0.2 M. With a graduated cylinder measure 14.5 mL of concentrated hydrochloric acid into a one-liter volumetric flask and dilute to the mark with water. Standardize with THAM, using methyl purple indicator.

Hydrofluoric acid, concentrated, 48%

Methyl purple, indicator solution, Sargent-Welch Scientific, Cat. No. SC13597, or equivalent

Sulfuric acid, concentrated, 96%

THAM, tris(hydroxymethyl)aminomethane, primary standard grade, Fisher Scientific, or equivalent

PROCEDURE

Sodium

Select an unetched 400-mL beaker and add 100 mL of warm water (50 ± 10 C) to it. Weigh by difference from a dropping bottle 2 to 5 g $\pm$ 0.1 mg of the well-mixed sample into a 400-mL beaker. Stir the solution with a glass rod, noting any signs of gel formation. If a gel is formed, this procedure is not applicable and the sample should be analyzed by UOP Method 410.

Allow the beaker and contents to cool to room temperature and add 2 to 3 drops of methyl purple indicator. Place the beaker on a stirring apparatus and titrate with 0.2-*M* hydrochloric acid, while continuously stirring, until the green color of the indicator passes through a slate color to a definite but faint purple. A few additional drops of the indicator added just prior to reaching the endpoint will make the endpoint more perceptible.

Record the volume of the 0.2-*M* hydrochloric acid required to reach the endpoint.

Silica: Rapid Control Procedure A

From a dropping bottle weigh by difference 0.8 to 1.5 g $\pm$ 0.1 mg of the well-mixed sample into an unetched 400-mL beaker. Add 40-mL of 1:1 hydrochloric acid and place the beaker on a steam bath. Use an infrared lamp to hasten drying as the contents approach dryness. Allow the beaker and contents to cool and then moisten with 5 mL of concentrated hydrochloric acid. Evaporate to dryness again. Allow to cool, then add 5 mL of concentrated hydrochloric acid and 50 mL of water, and bring just to a boil. Allow to cool and then filter the mixture. Wash the precipitate using at least 200 mL of water in small increments.

Place the filter paper and contents in a platinum crucible weighed to the nearest 0.1 mg and dry on a hot plate. Char the paper in the threshold of a muffle furnace and finally ignite at 1000 C for one hour.

Remove the crucible from the furnace and place it directly in a desiccator. Cover with the crucible cover and allow to cool for 20 minutes. Remove the crucible from the desiccator, uncover and weigh the crucible containing the silica to the nearest 0.1 mg.

Silica: Precise Method Procedure B

Proceed as described for A but retain the precipitate collected on the filter paper. Concentrate the filtrate to 20-40 mL by boiling. Add 20 mL of concentrated hydrochloric acid and evaporate to dryness. Allow to cool, moisten the residue with 5 mL of concentrated hydrochloric acid and again evaporate to dryness. Add 5 mL of concentrated hydrochloric acid and 50 mL of water, and bring the solution just to a boil. Allow the beaker and contents to cool, filter, and wash well with water.

Place the filter paper from this and the previous filtration in a platinum crucible and dry on a hot plate. Char the papers in the threshold of a muffle furnace and finally ignite at 1000 C for one hour.

Remove the crucible from the furnace and place it directly in a desiccator. Cover with the crucible cover and allow to cool for 20 minutes. Remove the crucible from the desiccator, uncover and weigh the crucible containing the impure silica to the nearest 0.1 mg.

Add several milliliters of water, 4 to 5 drops of concentrated sulfuric acid and 10 mL of concentrated hydrofluoric acid to the crucible and heat on a hot plate to near dryness. Repeat the additions of hydrofluoric acid and heating two more times. When the crucible content reaches dryness after the last addition of hydrofluoric acid, heat the crucible with care over a burner until the last traces of sulfuric acid are removed. Ignite the crucible and contents in a muffle furnace for one hour at 1000 C, cool in the desiccator and weigh the crucible as described previously.

CALCULATIONS

Sodium or Sodium Oxide

$$\text{Sodium, mass-\%} = \frac{2.299 A M}{W}$$

$$\text{Sodium, Oxide, mass-\%} = \frac{3.099 A M}{W}$$

where:

A = volume of 0.2 M hydrochloric acid used for titration, mL

M = molarity of the hydrochloric acid

W = mass of sample, g

2.299 = milliequivalent weight of sodium multiplied by 100 to convert to percent

3.099 = milliequivalent weight of sodium oxide multiplied by 100 to convert to percent

Silica

Procedure A

$$\text{SiO}_2, \text{ mass-\%} = \frac{100(S_2 - S_1)}{W_1}$$

where:

S₁ = mass of crucible, g

S₂ = mass of crucible plus silica, g

W₁ = mass of sample, g

100 = factor to convert fraction to mass-percent

Procedure B

$$\text{SiO}_2, \text{ mass-\%} = \frac{100(S_3 - S_4)}{W_1}$$

where:

S₃ = mass of crucible and contents before hydrofluoric acid treatment, g

S₄ = mass of crucible and contents after hydrofluoric acid treatment, g

W₁ and 100 = previously defined

PRECISION

Based on the number of replicates shown, the estimated standard deviations (esd) were calculated for the compounds shown in the table below. Duplicate results by the same operator should not differ by more than the allowable difference shown (95% probability).

Compound	Number of Replicates	Concentration, mass-%	esd, mass-%	Allowable Difference, mass-%
Sodium oxide	6	4.8	0.03	0.10
Silica: Rapid Control, Procedure A	5	14.9	0.09	0.36
Silica: Precise, Procedure B	5	15.3	0.07	0.26

TIME FOR ANALYSIS

The elapsed time and labor requirement for each procedure is shown in the following table:

	Labor Requirement	Elapsed Time, Hours
Sodium oxide	0.4	0.4
Silica: Rapid Control, Procedure A	0.8	4
Silica: Precise, Procedure B	1.2	8

REFERENCE

UOP Method 410

SUGGESTED SUPPLIERS

Engelhard Industries, 700 Blair Road, Carteret, NJ 07008
 Fisher Scientific Co., 1600 W. Glenlake Ave., Itasca, IL 60143
 Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60077