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Hydrophobic Colloidal Silica

CAS RN®: 68611-44-9.

DEFINITION

Hydrophobic Colloidal Silica is prepared by partial alkylation for hydrophobation. It contains NLT 99.0% and NMT 101.0% of silicon dioxide (SiO_2), calculated on the ignited basis.

IDENTIFICATION

• A.

Sample: 25 mg

Analysis: Add the *Sample* to a platinum crucible, and ignite at 900° for 2 h. Using a copper wire, mix the ignited substance with 10 mg of sodium fluoride and a few drops of sulfuric acid to give a thin slurry. Cover the crucible with a thin, transparent plate of plastic under which a drop of water is suspended, and warm gently.

Acceptance criteria: Within a short time, a white ring is rapidly formed around the drop of water.

• B. It meets the requirements for *Water-Dispersible Substances* in *Specific Tests*.

ASSAY

• PROCEDURE

Sample: The residue obtained in the test for *Loss on Ignition*

Analysis: To the *Sample* add sufficient alcohol to moisten the residue completely, and then add 0.2 mL of sulfuric acid. Add 6 mL of hydrofluoric acid, and evaporate to dryness on a hot plate at about 100°, taking care to avoid loss from sputtering. Wash down the sides of the platinum crucible with 6 mL of hydrofluoric acid, and evaporate to dryness. Ignite at 900° for 2 h, cool in a desiccator, and weigh. The difference between the weight of the residue obtained in the test for *Loss on Ignition* and the weight of the final residue gives the amount of silicon dioxide (SiO_2) in the quantity of the substance to be examined.

Acceptance criteria: 99.0%–101.0% on the ignited basis

IMPURITIES

• [Loss on Ignition \(733\)](#)

Sample: 0.2 g

Analysis: Ignite the *Sample* in a platinum crucible at 900° for 2 h. Cool in a desiccator before weighing. [NOTE—It is advisable to place the crucible in a cold oven and then heat up the oven.]

Acceptance criteria: NMT 6.0%

• LIMIT OF LEAD

Select reagents having as low a lead content as practicable, and store all solutions in containers of borosilicate glass. Rinse all glassware thoroughly with warm, dilute nitric acid (1 in 2) followed by water.

Ammonium acetate buffer solution, pH 3.5: Weigh 25.0 g of ammonium acetate, and dissolve in 25 mL of water. Add 38.0 mL of dilute hydrochloric acid. Adjust the pH, if necessary, with dilute hydrochloric acid or weak ammonia solution (containing 460 mL/L of strong ammonia solution). Dilute with water to 100.0 mL.

Thioacetamide solution: Prepare immediately before use. To 0.2 mL of thioacetamide TS add 1 mL of a mixture of 5 mL of water, 15 mL of 1 N sodium hydroxide, and 20 mL of 85% glycerol. Heat in a water bath for 20 s.

Sample solution: Suspend 2.5 g of Hydrophobic Colloidal Silica in 30 mL of methanol, stir, and add 30 mL of weak ammonia solution (containing 460 mL/L of strong ammonia solution). With frequent stirring, evaporate on a water bath, and dry the residue in an oven at 140°. When the dried substance is white, break up the mass with a glass rod. Reduce the residue to a powder, and add 15 mL of methanol and 25 mL of 1 N hydrochloric acid. Boil gently for 5 min, stirring frequently with the glass rod. Centrifuge for 20 min, and pass the supernatant

through a membrane filter. To the residue in the centrifuge tube add 3 mL of dilute hydrochloric acid and 9 mL of water, and bring to a boil. Centrifuge for 20 min, and pass the supernatant through the same membrane filter. Wash the residue with small quantities of water, combine the filtrates and washings, and dilute with water to 50 mL. To 20 mL of this solution add 50 mg of ascorbic acid and 1 mL of strong ammonia solution. Neutralize with diluted weak ammonia solution (containing 160 mL/L of strong ammonia solution). Dilute with water to 25 mL.

Reference solution: Pipet 10 mL of standard lead solution TS, and mix with 2 mL of the *Sample solution*.

Blank solution: A mixture of 10 mL of water and 2 mL of the *Sample solution*

Analysis: To the *Sample solution*, *Reference solution*, and *Blank solution* add 2 mL of *Ammonium acetate buffer solution, pH 3.5*. Mix, and add 1.2 mL of *Thioacetamide solution*. Mix immediately. Examine the solutions after 2 min. The test is invalid if the *Reference solution* does not show a slight brown color compared to the *Blank solution*.

Acceptance criteria: The brown color in the *Sample solution* is not more intense than that in the *Reference solution* (25 µg/g). [NOTE—If the result is difficult to judge, pass the solutions through a membrane filter of 3-µm pore size, and carry out the filtration slowly and uniformly. Compare the spots on the filters obtained with the different solutions.]

• **LIMIT OF CHLORIDE**

Standard solution: Add 10 mL of 0.15 mM sodium chloride and 5 mL of water. Add 1 mL of dilute nitric acid, and pour into a test tube containing 1 mL of silver nitrate TS.

Sample solution: To 1 g of Hydrophobic Colloidal Silica add 30 mL of methanol and 20 mL of dilute nitric acid. Heat on a water bath for 15 min with frequent stirring. Cool, dilute with water to 50 mL, and filter. Dilute 10 mL of the filtrate with water to 15 mL. Add 1 mL of dilute nitric acid, and pour into a test tube containing 1 mL of silver nitrate TS.

Analysis: Examine the tubes laterally against a black background.

Acceptance criteria: After standing for 5 min protected from light, any opalescence in the *Sample solution* is not more intense than that in the *Standard solution* (0.025%).

SPECIFIC TESTS

• **WATER-DISPERSIBLE SUBSTANCES**

Sample: 0.4 g

Analysis: Place the *Sample* in a 500-mL separating funnel, add 100 mL of water, and shake for 1 min. Allow to stand for 1 h. Allow 90 mL of the aqueous phase to run out dropwise without filtration into a suitable dish dried at 140°, and cool in a desiccator. Evaporate to dryness at 140°, starting at a low temperature to avoid splashing. Cool in a desiccator.

Acceptance criteria: NMT 3.0%; the weight of the residue does not exceed 12 mg.

ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in well-closed containers. No storage requirements specified.

Auxiliary Information - Please [check for your question in the FAQs](#) before contacting USP.

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