01/2008:0434
corrected 6.0

SILICA, COLLOIDAL ANHYDROUS

Silica colloidalis anhydrica

SiO_2 M_r 60.1
[7631-86-9]

DEFINITION

Colloidal anhydrous silica contains not less than 99.0 per cent and not more than the equivalent of 100.5 per cent of SiO_2, determined on the ignited substance.

CHARACTERS

A light, fine, white or almost white, amorphous powder, with a particle size of about 15 nm, practically insoluble in water and in mineral acids except hydrofluoric acid. It dissolves in hot solutions of alkali hydroxides.

IDENTIFICATION

About 20 mg gives the reaction of silicates (*2.3.1*).

TESTS

pH (*2.2.3*). Shake 1.0 g with 30 ml of *carbon dioxide-free water R*. The pH of the suspension is 3.5 to 5.5.

Chlorides (*2.4.4*). To 1.0 g add a mixture of 20 ml of *dilute nitric acid R* and 30 ml of *water R* and heat on a water-bath for 15 min, shaking frequently. Dilute to 50 ml with *water R* if necessary, filter and cool. 10 ml of the filtrate diluted to 15 ml with *water R* complies with the limit test for chlorides (250 ppm).

Heavy metals (*2.4.8*). Suspend 2.5 g in sufficient *water R* to produce a semi-fluid slurry. Dry at 140 °C. When the dried substance is white, break up the mass with a glass rod. Add 25 ml of *1 M hydrochloric acid* and boil gently for 5 min, stirring frequently with the glass rod. Centrifuge for 20 min and filter the supernatant liquid through a membrane filter. To the residue in the centrifuge tube add 3 ml of *dilute hydrochloric acid R* and 9 ml of *water R* and boil. Centrifuge for 20 min and filter the supernatant liquid through the same membrane filter. Wash the residue with small quantities of *water R*, combine the filtrates and washings and dilute to 50 ml with *water R*. To 20 ml of the solution add 50 mg of *ascorbic acid R* and 1 ml of *concentrated ammonia R*. Neutralise with *dilute ammonia R2*. Dilute to 25 ml with *water R*. 12 ml of the solution complies with limit test A for heavy metals (25 ppm). Prepare the standard using *lead standard solution (1 ppm Pb) R*.

Loss on ignition. Not more than 5.0 per cent, determined on 0.200 g by ignition in a platinum crucible at 900 ± 50 °C for 2 h. Allow to cool in a desiccator before weighing.

ASSAY

To the residue obtained in the test for loss on ignition add 0.2 ml of *sulphuric acid R* and sufficient *alcohol R* to moisten the residue completely. Add 6 ml of *hydrofluoric acid R* and evaporate to dryness on a hot-plate at 95 °C to 105 °C, taking care to avoid loss from sputtering. Wash down the sides of the dish with 6 ml of *hydrofluoric acid R* and evaporate to dryness. Ignite at 900 ± 50 °C, allow to cool in a desiccator and weigh.

The difference between the mass of the final residue and the mass of the residue obtained in the test for loss on ignition gives the amount of SiO_2 in the quantity of the substance to be examined used.

01/2008:0738
corrected 6.0

SILICA, COLLOIDAL HYDRATED

Silica colloidalis hydrica

[63231-67-4]

DEFINITION

Colloidal hydrated silica contains not less than 98.0 per cent and not more than the equivalent of 100.5 per cent of SiO_2 (M_r 60.1), determined on the ignited substance.

CHARACTERS

A white or almost white, light, fine, amorphous powder, practically insoluble in water and in mineral acids, with the exception of hydrofluoric acid. It dissolves in hot solutions of alkali hydroxides.

IDENTIFICATION

A. About 20 mg gives the reaction of silicates (*2.3.1*).

B. When heated in an oven at 100 °C to 105 °C for 2 h, it shows a loss of mass not less than 3.0 per cent.

TESTS

Solution S. To 2.5 g add 50 ml of *hydrochloric acid R* and mix. Heat on a water-bath for 30 min, stirring from time to time. Maintain the original volume by adding *dilute hydrochloric acid R*. Evaporate to dryness. Add to the residue a mixture of 8 ml of *dilute hydrochloric acid R* and 24 ml of *water R*. Heat to boiling and filter under reduced pressure through a sintered-glass filter (16) (*2.1.2*). Wash the residue on the filter with a hot mixture of 3 ml of *dilute hydrochloric acid R* and 9 ml of *water R*. Wash with small quantities of *water R*, combine the filtrate and washings and dilute to 50 ml with *water R*.

pH (*2.2.3*). Suspend 1.0 g in 30 ml of a 75 g/l solution of *potassium chloride R*. The pH of the suspension is 4.0 to 7.0.

Water-absorption capacity. In a mortar, triturate 5 g with 5 ml of *water R*, added drop by drop. The mixture remains powdery.

Substances soluble in hydrochloric acid. In a platinum dish, evaporate to dryness 10.0 ml of solution S and dry to constant mass at 100 °C to 105 °C. The mass of the residue is not more than 10 mg (2.0 per cent).

Chlorides (*2.4.4*). Heat 0.5 g with 50 ml of *water R* on a water-bath for 15 min. Dilute to 100 ml with *water R* and centrifuge at 1500 *g* for 5 min. 10 ml of the supernatant solution diluted to 15 ml with *water R* complies with the limit test for chlorides (0.1 per cent).

Sulphates (*2.4.13*). Dilute 2 ml of solution S to 100 ml with *distilled water R*. 15 ml of the solution complies with the limit test for sulphates (1 per cent).

Iron (*2.4.9*). To 2 ml of solution S add 28 ml of *water R*. 10 ml of the solution complies with the limit test for iron (300 ppm).

Heavy metals (*2.4.8*). To 20 ml of solution S add 50 mg of *hydroxylamine hydrochloride R* and 1 ml of *concentrated ammonia R*. Adjust to pH 3.5 by adding *dilute ammonia R2*, monitoring the pH potentiometrically. Dilute to 25 ml with *water R*. 12 ml of the solution complies with limit test A for heavy metals (25 ppm). Prepare the standard using *lead standard solution (1 ppm Pb) R*.

Loss on ignition. Not more than 20.0 per cent, determined on 0.200 g in a platinum crucible by heating at 100 °C to 105 °C for 1 h and then at 900 ± 50 °C for 2 h.

ASSAY

To the residue obtained in the test for loss on ignition add 0.2 ml of *sulphuric acid R* and a quantity of *alcohol R* sufficient to moisten the residue completely. Add 6 ml of *hydrofluoric acid R* and evaporate to dryness at 95 °C to 105 °C, taking care to avoid loss from sputtering. Wash the inside of the dish with 6 ml of *hydrofluoric acid R* and evaporate to dryness again. Ignite at 900 ± 50 °C, allow to cool in a desiccator and weigh. The difference between the mass of the final residue and that of the mass obtained in the test for loss on ignition corresponds to the mass of SiO_2 in the test sample.

01/2008:1562
corrected 6.0

SILICA, DENTAL TYPE

Silica ad usum dentalem

DEFINITION

Amorphous silica (precipitated, gel or obtained by flame hydrolysis).

Content: 94.0 per cent to 100.5 per cent of SiO_2 (ignited substance).

CHARACTERS

Appearance: white or almost white, light, fine amorphous powder.

Solubility: practically insoluble in water and in mineral acids. It dissolves in hydrofluoric acid and hot solutions of alkali hydroxides.

IDENTIFICATION

About 20 mg gives the reaction of silicates (*2.3.1*).

TESTS

Solution S. To 2.5 g add 50 ml of *hydrochloric acid R* and mix. Heat on a water-bath for 30 min, stirring from time to time. Evaporate to dryness. Add to the residue a mixture of 8 ml of *dilute hydrochloric acid R* and 24 ml of *water R*. Heat to boiling and filter under reduced pressure through a sintered-glass filter (16) (*2.1.2*). Wash the residue on the filter with a hot mixture of 3 ml of *dilute hydrochloric acid R* and 9 ml of *water R*. Wash with small quantities of *water R*, combine the washings and the filtrate, then dilute to 50 ml with *water R*.

pH (*2.2.3*): 3.2 to 8.9.

Suspend 5 g in a mixture of 5 ml of a 7.46 g/l solution of *potassium chloride R* and 90 ml of *carbon dioxide-free water R*.

Chlorides. Liquid chromatography (*2.2.29*) as described in the test for sulphates.

Retention time: chlorides = about 4 min.

Limit:

– *chlorides*: not more than the area of the corresponding peak in the chromatogram obtained with the reference solution (0.3 per cent).

Sulphates. Liquid chromatography (*2.2.29*).

Test solution. To 0.625 g of the substance to be examined add 30 ml of *water R* and boil for 2 h. Allow to cool and quantitatively transfer to a 50 ml graduated flask. Dilute to 50.0 ml with *water R*. Dilute 5.0 ml of the supernatant to 50.0 ml with *water R* and filter through a membrane filter (nominal pore size 0.45 µm).

Reference solution. Dissolve 0.50 g of *anhydrous sodium sulphate R* and 0.062 g of *sodium chloride R* in *water R* and dilute to 1000.0 ml with *water R*. Dilute 5.0 ml of this solution to 50.0 ml with *water R*.

Column:

– *material*: non-metallic;
– *size*: *l* = 0.25 m, Ø = 4.6 mm;
– *stationary phase*: suitable anion-exchange resin (30-50 µm).

Mobile phase: dissolve 0.508 g of *sodium carbonate R* and 0.05 g of *sodium hydrogen carbonate R* in *water R*, then dilute to 1000 ml with the same solvent.

Flow rate: 1.2 ml/min.

Detection: conductivity detector.

Injection: 25 µl.

Retention time: sulphates = about 8 min.

Limit:

– *sulphates*: not more than the area of the corresponding peak in the chromatogram obtained with the reference solution (4.0 per cent, expressed as sodium sulphate).

Iron (*2.4.9*): maximum 400 ppm.

Dilute 2 ml of solution S to 40 ml with *water R*.

Heavy metals (*2.4.8*): maximum 25 ppm.

To 20 ml of solution S, add 50 mg of *hydroxylamine hydrochloride R* and 1 ml of *concentrated ammonia R*. Adjust to pH 3.5 by adding *dilute ammonia R2*, monitoring the pH potentiometrically. Dilute to 25 ml with *water R*. 12 ml of the solution complies with test A. Prepare the reference solution using *lead standard solution (1 ppm Pb) R*.

Loss on ignition: maximum 25.0 per cent, determined on 0.200 g in a platinum crucible by heating at 100-105 °C for 1 h and then at 1000 ± 50 °C for 2 h.

ASSAY

To the residue obtained in the test for loss on ignition add 0.2 ml of *sulphuric acid R* and a quantity of *ethanol (96 per cent) R* sufficient to moisten the residue completely. Add 6 ml of *hydrofluoric acid R* and evaporate to dryness at 95-105 °C, taking care to avoid loss from sputtering. Wash the inside of the crucible with 6 ml of *hydrofluoric acid R* and evaporate to dryness again. Ignite at 900 ± 50 °C, allow to cool in a desiccator and weigh. The difference between the mass of the final residue and that of the mass obtained in the test for loss on ignition corresponds to the mass of SiO_2 in the test sample.

01/2008:2208
corrected 6.0

SILICA, HYDROPHOBIC COLLOIDAL

Silica hydrophobica colloidalis

DEFINITION

Colloidal silicon dioxide partly alkylated for hydrophobation.

Content: 99.0 per cent to 101.0 per cent SiO_2 (ignited substance).

CHARACTERS

Appearance: light, fine, white or almost white, amorphous powder, not wettable by water.