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Dental-type Silica

General Notices

(Ph. Eur. monograph 1562)

Action and use

Excipient.

Ph Eur

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, and 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 sulfates.

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).

Sulfates

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 sulfate 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 the 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* and dilute to 1000 mL with the same solvent.

Flow rate 1.2 mL/min.

Detection Conductivity detector.

Injection 25 µL.

Retention time Sulfates = about 8 min.

Limit:

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

Iron (2.4.9)

Maximum 400 ppm.

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

Loss on ignition

Maximum 25.0 per cent, determined on 0.200 g by heating in a platinum crucible 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 *sulfuric 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.

FUNCTIONALITY-RELATED CHARACTERISTICS

This section provides information on characteristics that are recognised as being relevant control parameters for one or more functions of the substance when used as an excipient (see chapter 5.15). Some of the characteristics described in the Functionality-related characteristics section may also be present in the mandatory part of the monograph since they also represent mandatory quality criteria. In such cases, a cross-reference to the tests described in the mandatory part is included in the Functionality-related characteristics section. Control of the characteristics can contribute to the quality of a medicinal product by improving the consistency of the manufacturing process and the performance of the medicinal product during use. Where control methods are cited, they are recognised as being suitable for the purpose, but other methods can also be used. Wherever results for a particular characteristic are reported, the control method must be indicated.

The following characteristic may be relevant for dental type silica used as abrasive.

Specific surface area (*2.9.26, Method I*)

Determine the specific surface area in the P/P_0 range of 0.05 to 0.30.

Sample outgasing 60 min at 160 °C.