Edition: BP 2025 (Ph. Eur. 11.6 update)

Magnesium Aluminometasilicate

General Notices

(Ph. Eur. monograph 2854)

Action and use

Excipient.

Ph Eur

DEFINITION

Magnesium aluminometasilicate of synthetic origin exists in 2 forms (type A and type B), which differ in their apparent pH. It contains a variable quantity of water.

Content (type A and type B):

— *silicon dioxide* (SiO_2; M_r 60.1): 29.2 per cent to 35.6 per cent (dried substance);

— *aluminium oxide* (Al_2O_3; M_r 102.0): 29.1 per cent to 35.5 per cent (dried substance);

— *magnesium oxide* (MgO; M_r 40.30): 11.4 per cent to 14.0 per cent (dried substance).

CHARACTERS

Appearance

White or almost white, hygroscopic powder or granules.

Solubility

Practically insoluble in water and in ethanol (96 per cent).

IDENTIFICATION

A. To 0.5 g add 5 mL of a mixture of 1 volume of *sulfuric acid R* and 2 volumes of *water R* and heat until white fumes evolve. Cool, add 20 mL of *water R* and filter. Use the filtrate for identification tests B and C. Wash the residue with *water R*. The residue gives the reaction of silicates (*2.3.1*).

B. Neutralise the filtrate obtained in identification test A with *ammonia R*. A white, gelatinous precipitate is formed. Centrifuge and keep the supernatant for identification test C. Dissolve the precipitate in *dilute hydrochloric acid R*. Add dropwise *dilute sodium hydroxide solution R*. A white, gelatinous precipitate is formed. Filter and add a few drops of *phenolphthalein solution R* to the residue. The residue turns pink. Wash the residue with *water R* until the pink colour is completely discharged and the residue remains white upon addition of a drop of *phenolphthalein solution R*. Sprinkle a few crystals of *sodium fluoride R* on the residue. The residue, in contact with the crystals, turns pink again in a short time.

C. To 2 mL of the supernatant obtained after centrifugation in identification test B, add 1 mL of *dilute ammonia R1* and 1 mL of *ammonium chloride solution R*. Upon the addition of *dilute ammonia R1* a white precipitate may form, which dissolves after addition of the *ammonium chloride solution R*. Add 1 mL of *disodium hydrogen phosphate solution R*. A white precipitate is formed.

TESTS

Solution S

Disperse 10.0 g in 100.0 mL of *water R* and boil gently for 15 min with shaking. After cooling, dilute to 100.0 mL with *water R* and centrifuge. Dilute 50.0 mL of the clear supernatant to 100.0 mL with *water R*.

pH (*2.2.3*)

6.0 to 8.5 for type A; 8.5 to 10.5 for type B.

Disperse 2.0 g in 50 mL of *carbon dioxide-free water R*. Read the pH after the electrode has been immersed in the suspension for 2 min.

Water-soluble salts

Maximum 1.5 per cent.

Evaporate 25 mL of solution S to dryness on a water-bath and heat at 700 °C for 2 h. The residue weighs a maximum of 19 mg.

Chlorides (*2.4.4*)

Maximum 500 ppm.

Dilute 2 mL of solution S to 15 mL with *water R*.

Sulfates (*2.4.13*)

Maximum 0.5 per cent.

Dilute 10 mL of solution S to 100 mL with *distilled water R*. Dilute 6 mL of the solution to 15 mL with *distilled water R*.

Iron (*2.4.9*)

Maximum 300 ppm.

Disperse 0.10 g in 8 mL of *dilute nitric acid R*, boil for 1 min, cool and dilute to 30.0 mL with *water R*. Centrifuge and use 10 mL of the clear supernatant.

Loss on drying (*2.2.32*)

Maximum 20.0 per cent, determined on 1.000 g by drying in an oven at 110 °C for 7 h.

Neutralising capacity

Minimum 210 mL of *0.1 M hydrochloric acid* per gram of dried substance.

To 0.200 g in a glass-stoppered flask add 100.0 mL of *0.1 M hydrochloric acid*. Stopper the flask tightly, shake at 37 ± 2 °C for 1 h and filter. Titrate 50.0 mL of the filtrate with *0.1 M sodium hydroxide* to pH 3.5 while stirring thoroughly. Carry out a blank determination.

ASSAY

Silicon dioxide

In a suitable container, disperse 1.000 g (*m*) in 30 mL of *dilute hydrochloric acid R* and evaporate to dryness on a water-bath. Moisten the residue with *hydrochloric acid R* and evaporate to dryness on a water-bath. To the residue add 8 mL of *hydrochloric acid R*, stir, add 25 mL of boiling *water R* and stir again. Allow to stand, then filter the supernatant through filter paper. To the residue in the container add 10 mL of boiling *water R*, stir, allow to stand, then filter the supernatant through the filter paper. Wash the residue in the container with 3 quantities, each of 10 mL, of boiling *water R*, each time filtering the washings through the filter paper. Add 50 mL of *water R* to the residue in the container, heat on a water-bath for 15 min and filter through the filter paper. Wash the residue on the filter paper with boiling *water R* until no further precipitate is formed when 1 mL of *silver nitrate solution R1* is added to 5 mL of the washings. Transfer the residue and the filter paper to a platinum crucible that has been weighed prior to the transfer (*a*). Heat strongly to incinerate and continue to heat at 775-825 °C for 1 h. Cool and weigh (*b*).

Calculate the percentage content of silicon dioxide (SiO_2) using the following expression:

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Aluminium oxide

In a conical flask disperse 1.250 g in 15 mL of *dilute hydrochloric acid R* and 50 mL of *water R* and heat on a water-bath for 15 min. To this solution add 8 mL of *hydrochloric acid R* and heat on a water-bath for 10 min. After cooling, transfer the solution to a 250 mL volumetric flask, rinse the conical flask with *water R* and add the washings to the volumetric flask. Dilute to volume with *water R*. Centrifuge and use the supernatant as the test solution. Retain a portion for use in the assay for magnesium oxide.

Carry out the complexometric titration of aluminium (*2.5.11*). Carry out a blank determination.

1 mL of *0.1 M sodium edetate* is equivalent to 5.098 mg of Al_2O_3.

Magnesium oxide

Transfer 100.0 mL of the test solution obtained in the assay for aluminium oxide to a conical flask, add 25 mL of a 50 per cent *V/V* solution of *triethanolamine R* and shake. Add 25 mL of *ammonium chloride buffer solution pH 10.7 R* and 40 mg of *mordant black 11 triturate R*. Titrate with *0.1 M sodium edetate* until the colour changes from violet to pure blue.

1 mL of *0.1 M sodium edetate* is equivalent to 4.030 mg of MgO.

STORAGE

In an airtight container.

LABELLING

The label states the type of magnesium aluminometasilicate.

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 characteristics may be relevant for magnesium aluminometasilicate used as glidant in tablets and capsules.

Particle-size distribution (*2.9.31*)

Specific surface area (*2.9.26, Method I*)