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Aluminium Stearate

General Notices

(Ph. Eur. monograph 1663)

Ph Eur

DEFINITION

Aluminium salts of a mixture of solid organic acids consisting mainly of variable proportions of aluminium stearate [$(C_{17}H_{35}COO)_3Al$; *Mr* 877] and aluminium palmitate [$(C_{15}H_{31}COO)_3Al$; *Mr* 793]. The organic acids are obtained from sources of vegetable or animal origin.

Content

— *aluminium* (Al; *Ar* 26.98): 3.0 per cent to 9.0 per cent (dried substance);

— *stearic acid in thefattyacidfraction* : minimum 35.0 per cent;

— *sum of stearic acidandpalmiticacidinthefattyacidfraction*: minimum 90.0 per cent.

CHARACTERS

Appearance

White or almost white, very fine, light powder.

Solubility

Practically insoluble in water and in anhydrous ethanol.

IDENTIFICATION

First identification: C, D.

Second identification: A, B, D.

A. Freezing point (2.2.18): minimum 53 °C, determined on the residue obtained in the preparation of solution S (see Tests).
B. Acid value (2.5.1): 195 to 210.
Dissolve 0.200 g of the residue obtained in the preparation of solution S in 25 mL of the prescribed mixture of solvents.

C. Examine the chromatograms obtained in the assay of stearic acid and palmitic acid.

Results The 2 principal peaks in the chromatogram obtained with the test solution are similar in retention time to the 2 principal peaks in the chromatogram obtained with the reference solution.

D. 1 mL of solution S gives the reaction of aluminium (2.3.1). The addition of 0.5 mL of *dilute hydrochloric acid R* described in the general method is omitted.

TESTS

Solution S

To 5.0 g add 50 mL of *peroxide-free ether R*, 20 mL of *dilute nitric acid R* and 20 mL of *distilled water R* and heat gently under a reflux condenser until dissolution is complete. Allow to cool. In a separating funnel, separate the aqueous layer and shake the ether layer with 2 quantities, each of 4 mL, of *distilled water R*. Combine the aqueous layers, wash with 15 mL of *peroxide-free ether R* and dilute to 50.0 mL with *distilled water R* (solution S). Evaporate the ether layer to dryness and dry the residue at 100-105 °C. Keep the residue for identification tests A and B.

Acidity or alkalinity

To 1.0 g add 20 mL of *carbon dioxide-free water R* and boil for 1 min with continuous shaking. Cool and filter. To 10 mL of the filtrate add 0.05 mL of *bromothymol blue solution R4*. Not more than 0.05 mL of *0.1 M hydrochloric acid* or *0.1 M sodium hydroxide* is required to change the colour of the indicator.

Chlorides (2.4.4)

Maximum 0.1 per cent.

Dilute 0.5 mL of solution S to 15 mL with *water R*.

Sulfates (2.4.13)

Maximum 0.5 per cent.

Dilute 0.3 mL of solution S to 15 mL with *distilled water R*.

Loss on drying (2.2.32)

Maximum 6.0 per cent, determined on 1.000 g by drying in an oven at 105 °C.

Microbial contamination

TAMC: acceptance criterion 103 CFU/g (2.6.12).

TYMC: acceptance criterion 102 CFU/g (2.6.12).

Absence of *Escherichia coli* (2.6.13).

Absence of *Salmonella* (2.6.13).

ASSAY

Aluminium

To 0.250 g in a 250 mL conical flask add 20 mL of *methanol R* and, slowly, 2 mL of *sulfuric acid R*. Heat the solution for 30 min under reflux on a water-bath, swirling frequently. Allow to cool. Add 100 mL of *water R* and adjust to about pH 1 by adding approximately 12 mL of *dilute sodium hydroxide solution R*. Add 20.0 mL of *0.1 M sodium edetate* and adjust to between pH 5 and pH 6 by the addition of *sodium acetate R*. Add 70 mg of *xylenol orange triturate R* and titrate immediately and quickly with *0.1 M zinc sulfate* until the colour changes from yellow to pinkish-violet.

1 mL of *0.1 M sodium edetate* is equivalent to 2.698 mg of Al.

Stearic acid and palmitic acid

Gas chromatography (2.2.28): use the normalisation procedure.

Test solution In a conical flask fitted with a reflux condenser, dissolve 0.100 g of the substance to be examined in 5 mL of borontrifluoride-methanolsolution R . Boil under a reflux condenser for 10 min. Add 4 mL of heptane R through the condenser and boil again under a reflux condenser for 10 min. Allow to cool. Add 20 mL of saturated sodium chloride solution RShake and allow the layers to separate. Dry the organic layer over 0.1 g of anhydrous sodium sulfate R previously washed with heptane R. Dilute 1.0 mL of the solution to 10.0 mL with heptane R.

Reference solution Prepare the reference solution in the same manner as the test solution using 50 mg of palmitic acidCRS and 50 mg of stearic acid CRS instead of the substance to be examined.

Column:

— *material*: fused silica;

— *size*: *l* = 30 m, Ø = 0.32 mm;

— *stationary phase*: macrogol 20 000 R (film thickness 0.5 µm).

Carrier gas helium for chromatography R.

Flow rate 2.4 mL/min.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 2	70
	2 - 36	70 → 240
	36 - 41	240
Injection port		220
Detector		260

Detection Flame ionisation.

Injection 1 µL.

Relative retention With reference to methyl stearate: methyl palmitate = about 0.9.

System suitability Reference solution:

— resolution: minimum 5.0 between the peaks due to methyl palmitate and methyl stearate;

— *repeatability*: maximum relative standard deviation of 3.0 per cent for the areas of the peaks due to methyl palmitate and methyl stearate, determined on 6 injections; maximum relative standard deviation of 1.0 per cent for the ratio of the areas of the peaks due to methyl palmitate to the areas of the peaks due to methyl stearate, determined on 6 injections.