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CALCIUM STEARATE

Calcii stearas

DEFINITION

Mixture of calcium salts of different fatty acids consisting mainly of stearic (octadecanoic) acid [$(C_{17}H_{35}COO)_2Ca$; M_r 607] and palmitic (hexadecanoic) acid [$(C_{15}H_{31}COO)_2Ca$; M_r 550.9] with minor proportions of other fatty acids.

Content:

– *calcium*: 6.4 per cent to 7.4 per cent (A_r 40.08) (dried substance),
– *stearic acid in the fatty acid fraction*: minimum 40.0 per cent,
– *sum of stearic acid and palmitic acid in the fatty acid fraction*: minimum 90.0 per cent.

CHARACTERS

Appearance: fine, white or almost white, crystalline powder.

Solubility: practically insoluble in water and in ethanol (96 per cent).

IDENTIFICATION

First identification: C, D.

Second identification: A, B, D.

A. Freezing point (*2.2.18*): minimum 53 °C, for 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 test for fatty acid composition.
Results: the retention times of the principal peaks in the chromatogram obtained with the test solution are approximately the same as those of the principal peaks in the chromatogram obtained with the reference solution.

D. Neutralise 5 ml of solution S to *red litmus paper R* using *strong sodium hydroxide solution R*. The solution gives reaction (b) of calcium (*2.3.1*).

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*. Boil 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 5 ml, of *distilled water R*. Combine the aqueous layers, wash with 15 ml of *peroxide-free ether R* and dilute the aqueous layer to 50 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 R1*. Not more than 0.5 ml of *0.01 M hydrochloric acid* or *0.01 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*.

Sulphates (*2.4.13*): maximum 0.3 per cent.

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

Cadmium: maximum 3.0 ppm.

Atomic absorption spectrometry (*2.2.23, Method II*).

Test solution. Place 50.0 mg in a polytetrafluoroethylene digestion bomb and add 0.5 ml of a mixture of 1 volume of *hydrochloric acid R* and 5 volumes of *cadmium- and lead-free nitric acid R*. Allow to digest at 170 °C for 5 h. Allow to cool. Dissolve the residue in *water R* and dilute to 5.0 ml with the same solvent.

Reference solutions. Prepare the reference solutions using *cadmium standard solution (10 ppm Cd) R*, diluted if necessary with a 1 per cent *V/V* solution of *hydrochloric acid R*.

Source: cadmium hollow-cathode lamp.

Wavelength: 228.8 nm.

Atomisation device: graphite furnace.

Lead: maximum 10.0 ppm.

Atomic absorption spectrometry (*2.2.23, Method II*).

Test solution. Use the solution described in the test for cadmium.

Reference solutions. Prepare the reference solutions using *lead standard solution (10 ppm Pb) R*, diluted if necessary with *water R*.

Source: lead hollow-cathode lamp.

Wavelength: 283.3 nm; 217.0 nm may be used depending on the apparatus.

Atomisation device: graphite furnace.

Nickel: maximum 5.0 ppm.

Atomic absorption spectrometry (*2.2.23, Method II*).

Test solution. Use the solution described in the test for cadmium.

Reference solutions. Prepare the reference solutions using *nickel standard solution (10 ppm Ni) R*, diluted if necessary with *water R*.

Source: nickel hollow-cathode lamp.

Wavelength: 232.0 nm.

Atomisation device: graphite furnace.

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. Total viable aerobic count (*2.6.12*) not more than 10^3 micro-organisms per gram, determined by plate count. It complies with the test for *Escherichia coli* (*2.6.13*).

ASSAY

Calcium. To 0.500 g in a 250 ml conical flask add 50 ml of a mixture of equal volumes of *butanol R* and *anhydrous ethanol R*, 5 ml of *concentrated ammonia R*, 3 ml of *ammonium chloride buffer solution pH 10.0 R*, 30.0 ml of *0.1 M sodium edetate* and 15 mg of *mordant black 11 triturate R*. Heat to 45-50 °C until the solution is clear. Cool and titrate with *0.1 M zinc sulphate* until the colour changes from blue to violet. Carry out a blank titration.

1 ml of *0.1 M sodium edetate* is equivalent to 4.008 mg of Ca.

Composition of fatty acids. Gas chromatography (*2.2.28*): use the normalisation procedure.

Test solution. In a conical flask fitted with a reflux condenser, dissolve 0.10 g of the substance to be examined in 5 ml of *boron trifluoride-methanol solution R*. Boil under a reflux condenser for 10 min. Add 4 ml of *heptane R* through the condenser. Boil under a reflux condenser for 10 min. Allow to cool. Add 20 ml of a *saturated sodium chloride solution R*. Shake and allow the layers to separate. Remove about 2 ml of the organic layer and dry over 0.2 g of

anhydrous sodium sulphate 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 50.0 mg of *palmitic acid CRS* and 50.0 mg of *stearic acid CRS* instead of calcium stearate.

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 0.88.

System suitability: reference solution:

– *resolution*: minimum 5.0 between the peaks due to methyl stearate and methyl palmitate.

Calculate the content of palmitic acid and stearic acid. Disregard the peak due to the solvent.

Acidity or alkalinity. Shake 1.5 g with 15 ml of *carbon dioxide-free water R* for 5 min. Allow to stand for 5 min and filter. To 10 ml of the filtrate add 0.1 ml of *phenolphthalein solution R* and 0.25 ml of *0.01 M sodium hydroxide*. The solution is red. Add 0.30 ml of *0.01 M hydrochloric acid*. The solution is colourless. Add 0.2 ml of *methyl red solution R*. The solution is reddish-orange.

Chlorides (*2.4.4*): maximum 300 ppm.

Shake 0.5 g with 15 ml of *water R* for 5 min. Allow to stand for 15 min and filter. Dilute 5 ml of the filtrate to 15 ml with *water R*.

Arsenic (*2.4.2, Method A*): maximum 10 ppm, determined on 5 ml of solution S.

Iron (*2.4.9*): maximum 100 ppm.

To 0.25 g add a mixture of 5 ml of *hydrochloric acid R* and 20 ml of *water R*. Heat to boiling, cool and filter.

Heavy metals (*2.4.8*): maximum 20 ppm.

To 2.5 g add a mixture of 2 ml of *hydrochloric acid R* and 15 ml of *water R*. Heat to boiling. Cool and then add 0.5 ml of *phenolphthalein solution R*. Cautiously add *concentrated ammonia R* until the colour changes to pink. Add 0.5 ml of *glacial acetic acid R* and dilute to 25 ml with *water R*. Filter. 12 ml of the filtrate complies with test A. Prepare the reference solution using *lead standard solution (2 ppm Pb) R*.

Loss on ignition: 18.0 per cent to 22.0 per cent, determined on 1.000 g by ignition to constant mass at 800 ± 50 °C.

ASSAY

Dissolve 0.150 g in 120 ml of *water R*. Carry out the complexometric titration of calcium (*2.5.11*).

1 ml of *0.1 M sodium edetate* is equivalent to 17.22 mg of $CaSO_4,2H_2O$.

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CALCIUM SULPHATE DIHYDRATE

Calcii sulfas dihydricus

$CaSO_4,2H_2O$ M_r 172.2
[10101-41-4]

DEFINITION

Content: 98.0 per cent to 102.0 per cent of $CaSO_4,2H_2O$.

CHARACTERS

Appearance: white or almost white fine powder.

Solubility: very slightly soluble in water, practically insoluble in ethanol (96 per cent).

IDENTIFICATION

A. Loss on ignition (see Tests).
B. Solution S (see Tests) gives reaction (a) of sulphates (*2.3.1*).
C. Solution S gives reaction (a) of calcium (*2.3.1*).

TESTS

Solution S. Dissolve 1.0 g in 50 ml of a 10 per cent *V/V* solution of *hydrochloric acid R* by heating at 50 °C for 5 min. Allow to cool.

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CALENDULA FLOWER

Calendulae flos

DEFINITION

Whole or cut, dried, and fully opened flowers that have been detached from the receptacle of the cultivated, double-flowered varieties of *Calendula officinalis* L.

Content: minimum 0.4 per cent of flavonoids, expressed as hyperoside ($C_{21}H_{20}O_{12}$; M_r 464.4) (dried drug).

IDENTIFICATION

A. The ligulate florets consist of a yellow or orange-yellow ligule, about 3-5 mm wide and about 7 mm in the middle part, with a 3-toothed apex and a hairy, partly sickle-shaped, yellowish-brown or orange-brown tube with a projecting style and a bifid stigma occasionally with a partly bent yellowish-brown or orange-brown ovary. The tubular florets, about 5 mm long, are present and consist of the yellow, orange-red or reddish-violet 5-lobed corolla and the yellowish-brown or orange-brown tube, hairy in its lower part, mostly with a partly bent yellowish-brown or orange-brown ovary.
B. Reduce to a powder (355) (*2.9.12*). The powder is yellowish-brown. Examine under a microscope using *chloral hydrate solution R*. The powder shows the following diagnostic characters: fragments of the corollas containing light yellow oil droplets, some with fairly