Calculate the percentage content of echinacoside using the following expression:

$$\frac{A_1 \times C_2 \times 100 \times 2.221}{A_2 \times C_1}$$

A_1 = area of the peak due to echinacoside in the chromatogram obtained with the test solution;

A_2 = area of the peak due to chlorogenic acid in the chromatogram obtained with the reference solution;

C_1 = concentration of the test solution, in milligrams per millilitre;

C_2 = concentration of chlorogenic acid in the reference solution, in milligrams per millilitre;

2.221 = peak correlation factor between chlorogenic acid and echinacoside.

STORAGE

Uncomminuted.

01/2008:1904

PALMITIC ACID

Acidum palmiticum

[57-10-3]

DEFINITION

Hexadecanoic acid ($C_{16}H_{32}O_2$; M_r 256.4), obtained from fats or oils of vegetable or animal origin.

Content: minimum 92.0 per cent.

CHARACTERS

Appearance: white or almost white, waxy solid.

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

IDENTIFICATION

A. Freezing point (see Tests).

B. Acid value (*2.5.1*): 216 to 220, determined on 0.1 g.

C. Examine the chromatograms obtained in the assay.

Results: the principal peak in the chromatogram obtained with the test solution is similar in retention time to the principal peak in the chromatogram obtained with the reference solution.

TESTS

Appearance. Heat the substance to be examined to about 75 °C. The resulting liquid is not more intensely coloured than reference solution Y_7 or BY_7 (*2.2.2, Method I*).

Acidity. Melt 5.0 g, stir for 2 min in 10 ml of hot *carbon dioxide-free water R*, cool slowly and filter. To the filtrate add 0.05 ml of *methyl orange solution R*. No red colour develops.

Freezing point (*2.2.18*): 60 °C to 66 °C.

Iodine value (*2.5.4*): maximum 1.

Stearic acid: maximum 6.0 per cent, determined as prescribed in the assay.

Nickel (*2.4.31*): maximum 1 ppm.

ASSAY

Gas chromatography (*2.4.22, Method C*). Prepare the solutions as described in the method but omitting the initial hydrolysis.

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

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

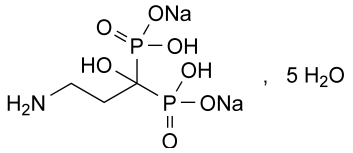
System suitability:

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

01/2008:1779

PAMIDRONATE DISODIUM PENTAHYDRATE

Dinatrii pamidronas pentahydricus



$C_3H_9NNa_2O_7P_2,5H_2O$ M_r 369.1

[109552-15-0]

DEFINITION

Disodium dihydrogen (3-amino-1-hydroxypropylidene)bisphosphonate pentahydrate.

Content: 98.0 per cent to 101.0 per cent (anhydrous substance).

CHARACTERS

Appearance: white or almost white, crystalline powder.

Solubility: soluble in water, practically insoluble in methylene chloride. It is sparingly soluble in dilute mineral acids and dissolves in dilute alkaline solutions.

IDENTIFICATION

A. Infrared absorption spectrophotometry (*2.2.24*).

Comparison: *pamidronate disodium pentahydrate CRS*.

B. Dissolve 0.5 g in 10 ml of *water R*. The solution gives reaction (a) of sodium (*2.3.1*).

TESTS

Appearance of solution. The solution is clear (*2.2.1*) and not more intensely coloured than reference solution Y_6 (*2.2.2, Method II*).

Dissolve 0.20 g in *carbon dioxide-free water R* and dilute to 10 ml with the same solvent.

pH (*2.2.3*): 7.8 to 8.8.

Dissolve 0.100 g in *carbon dioxide-free water R* and dilute to 10 ml with the same solvent.

Impurity A. Thin-layer chromatography (*2.2.27*).

Test solution. Dissolve 30 mg of the substance to be examined in *water R* and dilute to 10.0 ml with the same solvent.

Reference solution. Dissolve 15 mg of *β-alanine R* in *water R* and dilute to 100.0 ml with the same solvent. Dilute 1.0 ml of this solution to 10.0 ml with *water R*.

Plate: *TLC silica gel plate R.*

Mobile phase: *concentrated ammonia R, di-isopropyl ether R, methanol R* (4:8:9 *V/V/V*).

Application: 10 µl.

Development: over 2/3 of the plate.

Drying: in a current of warm air.

Detection: spray with a *ninhydrin solution R*. Heat at 100-105 °C for 15 min.

Limit:

– *impurity A*: any spot due to impurity A is not more intense than the spot in the chromatogram obtained with the reference solution (0.5 per cent).

Impurities B and C. Liquid chromatography (*2.2.29*).

Test solution. Dissolve 20.0 mg of the substance to be examined in *water R* and dilute to 10.0 ml with the same solvent.

Reference solution. To 2.0 ml of a 0.3 g/l solution of *phosphoric acid R* add 2.0 ml of a 0.25 g/l solution of *phosphorous acid R* and dilute to 50.0 ml with *water R*.

Column:

– *size*: *l* = 0.10 m, Ø = 4.6 mm,

– *stationary phase*: *anion exchange resin R* (5 µm),

– *temperature*: 35 °C.

Mobile phase: to 0.5 ml of *anhydrous formic acid R* add 2500 ml of *water R*; adjust to pH 3.5 with an 80 g/l solution of *sodium hydroxide R*.

Flow rate: 1.0 ml/min.

Detection: refractometer.

Injection: 100 µl.

Relative retention with reference to pamidronate (retention time = about 13 min): impurity B = about 1.3; impurity C = about 1.6.

System suitability: reference solution:

– *resolution*: minimum 2.5 between the peaks due to impurities B and C.

Limits:

– *impurities B, C*: for each impurity, not more than the area of the corresponding peaks in the chromatogram obtained with the reference solution (0.5 per cent).

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

2.0 g complies with test C. Prepare the reference solution using *lead standard solution (2 ppm Pb) R*.

Water (*2.5.12*): 23.0 per cent to 27.0 per cent, determined on 0.100 g.

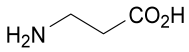
ASSAY

Dissolve 0.250 g in 70 ml of *water R*. Titrate with *0.1 M hydrochloric acid* determining the end-point potentiometrically (*2.2.20*).

1 ml of *0.1 M hydrochloric acid* is equivalent to 27.91 mg of $C_3H_9NNa_2O_7P_2$.

IMPURITIES

Specified impurities: A, B, C.



A. 3-aminopropanoic acid (β-alanine),

B. phosphoric acid,

C. phosphorous acid.

01/2008:0350

PANCREAS POWDER

Pancreatis pulvis

[53608-75-6]

DEFINITION

Pancreas powder is prepared from the fresh or frozen pancreases of mammals. It contains various enzymes having proteolytic, lipolytic and amylolytic activities.

1 mg of pancreas powder contains not less than 1.0 Ph. Eur. U. of total proteolytic activity, 15 Ph. Eur. U. of lipolytic activity and 12 Ph. Eur. U. of amylolytic activity.

CHARACTERS

A slightly brown, amorphous powder, partly soluble in water, practically insoluble in ethanol (96 per cent).

IDENTIFICATION

A. Triturate 0.5 g with 10 ml of *water R* and adjust to pH 8 with *0.1 M sodium hydroxide*, using 0.1 ml of *cresol red solution R* as indicator. Divide the suspension into 2 equal parts (suspension (a) and suspension (b)). Boil suspension (a). To each suspension add 10 mg of *fibrin congo red R*, heat to 38-40 °C and maintain at this temperature for 1 h. Suspension (a) is colourless or slightly pink and suspension (b) is distinctly more red.

B. Triturate 0.25 g with 10 ml of *water R* and adjust to pH 8 with *0.1 M sodium hydroxide*, using 0.1 ml of *cresol red solution R* as indicator. Divide the suspension into 2 equal parts (suspension (a) and suspension (b)). Boil suspension (a). Dissolve 0.1 g of *soluble starch R* in 100 ml of boiling *water R*, boil for 2 min, cool and dilute to 150 ml with *water R*. To 75 ml of the starch solution add suspension (a) and to the remaining 75 ml add suspension (b). Heat each mixture to 38-40 °C and maintain at this temperature for 5 min.

To 1 ml of each mixture add 10 ml of *iodine solution R2*. The mixture obtained with suspension (a) has an intense blue-violet colour; the mixture obtained with suspension (b) has the colour of the iodine solution.

TESTS

Fat content. In an extraction apparatus, treat 1.0 g with *light petroleum R1* for 3 h. Evaporate the solvent and dry the residue at 100-105 °C for 2 h. The residue weighs not more than 50 mg (5.0 per cent).

Loss on drying (*2.2.32*). Not more than 5.0 per cent, determined on 0.50 g by drying at 60 °C at a pressure not exceeding 670 Pa for 4 h.

Microbial contamination. Total viable aerobic count (*2.6.12*) not more than 10^4 micro-organisms per gram, determined by plate count. It complies with the tests for *Escherichia coli* and *Salmonella* (*2.6.13*).

ASSAY

Total proteolytic activity. The total proteolytic activity of pancreas powder is determined by comparing the quantity of peptides non-precipitable by a 50 g/l solution of *trichloroacetic acid R* released per minute from a substrate of casein solution with the quantity of such peptides released by *pancreas powder (protease) BRP* from the same substrate in the same conditions.

Casein solution. Suspend a quantity of *casein BRP* equivalent to 1.25 g of dried substance in 5 ml of *water R*,