Treat each solution as follows. To 5.0 ml add 4 ml of *dilute sulphuric acid R*, 1 ml of *ammonium molybdate solution R*, 5 ml of *water R* and 2 ml of a solution containing 0.10 g of *4-methylaminophenol sulphate R*, 0.5 g of *anhydrous sodium sulphite R* and 20.0 g of *sodium metabisulphite R* in 100 ml of *water R*. Shake and allow to stand for 15 min. Dilute to 25.0 ml with *water R* and allow to stand for a further 15 min. Measure the absorbance (*2.2.25*) at 730 nm. Calculate the content of soluble phosphates from a calibration curve prepared using reference solutions (a), (b) and (c) after treatment.

Sulphates (*2.4.13*): maximum 0.6 per cent.

Dilute 8 ml of solution S to 100 ml with *distilled water R*. 15 ml of the solution complies with the limit test for sulphates.

Arsenic (*2.4.2*): maximum 1 ppm.

1.0 g complies with limit test A.

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

Dissolve 1.0 g in *dilute hydrochloric acid R* and dilute to 20 ml with the same acid. 12 ml of the solution complies with limit test A. Prepare the standard using *lead standard solution (1 ppm Pb) R*.

Loss on ignition. 10.0 per cent to 20.0 per cent, determined on 1.000 g at 800 ± 50 °C.

Neutralising capacity. Add 0.50 g to 30 ml of *0.1 M hydrochloric acid* previously heated to 37 °C and maintain at this temperature for 15 min while stirring. The pH (*2.2.3*) of the mixture after 15 min at 37 °C is 2.0 to 2.5.

ASSAY

Dissolve 0.400 g in 10 ml of *dilute hydrochloric acid R* and dilute to 100.0 ml with *water R*. To 10.0 ml of the solution, add 10.0 ml of *0.1 M sodium edetate* and 30 ml of a mixture of equal volumes of *ammonium acetate solution R* and *dilute acetic acid R*. Boil for 3 min, then cool. Add 25 ml of *alcohol R* and 1 ml of a freshly prepared 0.25 g/l solution of *dithizone R* in *alcohol R*. Titrate the excess of sodium edetate with *0.1 M zinc sulphate* until the colour changes to pink.

1 ml of *0.1 M sodium edetate* is equivalent to 12.20 mg of $AlPO_4$.

STORAGE

In an airtight container.

01/2008:0165

ALUMINIUM SULPHATE

Aluminii sulfas

$Al_2(SO_4)_3,xH_2O$ M_r 342.1 (anhydrous substance)

DEFINITION

Content: 51.0 per cent to 59.0 per cent of $Al_2(SO_4)_3$.

It contains a variable quantity of water of crystallisation.

CHARACTERS

Appearance: colourless, lustrous crystals or crystalline masses.

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

IDENTIFICATION

A. Solution S (see Tests) gives reaction (a) of sulphates (*2.3.1*).

B. Solution S gives the reaction of aluminium (*2.3.1*).

TESTS

Solution S. Dissolve 2.5 g in *water R* and dilute to 50 ml with the same solvent.

Appearance of solution. Solution S is not more opalescent than reference suspension III (*2.2.1*) and is colourless (*2.2.2, Method II*).

pH (*2.2.3*): 2.5 to 4.0.

Dissolve 0.5 g in *carbon dioxide-free water R* and dilute to 25 ml with the same solvent.

Alkali and alkaline-earth metals: maximum 0.4 per cent.

To 20 ml of solution S add 100 ml of *water R*, heat and add 0.1 ml of *methyl red solution R*. Add *dilute ammonia R1* until the colour of the indicator changes to yellow. Dilute to 150 ml with *water R*, heat to boiling and filter. Evaporate 75 ml of the filtrate to dryness on a water-bath and ignite. The residue weighs a maximum of 2 mg.

Ammonium (*2.4.1*): maximum 500 ppm.

Dilute 0.4 ml of solution S to 14 ml with *water R*.

Iron (*2.4.9*): maximum 100 ppm.

Dilute 2 ml of solution S to 10 ml with *water R*. Use 0.3 ml of *thioglycollic acid R* in this test.

Heavy metals (*2.4.8*): maximum 50 ppm.

Dilute 8 ml of solution S to 20 ml with *water R*. 12 ml of the solution complies with test A. Prepare the reference solution using *lead standard solution (1 ppm Pb) R*.

ASSAY

Dissolve 0.500 g in 20 ml of *water R*. Carry out the complexometric titration of aluminium (*2.5.11*).

1 ml of *0.1 M sodium edetate* is equivalent to 17.11 mg of $Al_2(SO_4)_3$.

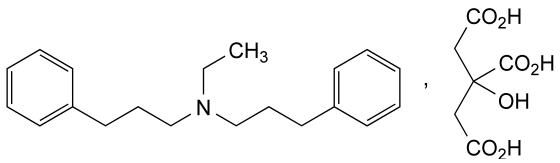
STORAGE

In an airtight container.

01/2008:2156

ALVERINE CITRATE

Alverini citras



$C_{26}H_{35}NO_7$ M_r 473.6
[5560-59-8]

DEFINITION

N-Ethyl-3-phenyl-*N*-(3-phenylpropyl)propan-1-amine dihydrogen 2-hydroxypropane-1,2,3-tricarboxylate.

Content: 99.0 per cent to 101.0 per cent (dried substance).

CHARACTERS

Appearance: white or almost white, crystalline powder.

Solubility: slightly soluble in water and in methylene chloride, sparingly soluble in ethanol (96 per cent).

mp: about 104 °C.

IDENTIFICATION

Infrared absorption spectrophotometry (*2.2.24*).

Comparison: *alverine citrate CRS*.

TESTS

pH (*2.2.3*): 3.5 to 4.5.

Dissolve 0.250 g in *carbon dioxide-free water R* and dilute to 50.0 ml with the same solvent.

Related substances. Gas chromatography (*2.2.28*): use the normalisation procedure. *Use freshly prepared solutions.*

Test solution. Dissolve 0.250 g of the substance to be examined in *water R* and dilute to 20 ml with the same solvent. Add 2 ml of *concentrated ammonia R* and shake with 3 quantities, each of 15 ml, of *methylene chloride R*. To the combined lower layers add *anhydrous sodium sulphate R*, shake, filter, and evaporate the filtrate at a temperature not exceeding 30 °C, using a rotary evaporator. Take up the residue with *methylene chloride R* and dilute to 10.0 ml with the same solvent.

Reference solution (a). Dissolve 5 mg of *alverine impurity D CRS* (impurity D citrate) in 5 ml of *water R*, add 1 ml of *concentrated ammonia R* and shake with 3 quantities, each of 5 ml, of *methylene chloride R*. To the combined lower layers add *anhydrous sodium sulphate R*, shake, filter, and evaporate the filtrate at a temperature not exceeding 30 °C, using a rotary evaporator. Take up the residue with *methylene chloride R*, add 0.2 ml of the test solution and dilute to 2 ml with *methylene chloride R*.

Reference solution (b). Dilute 1.0 ml of the test solution to 100.0 ml with *methylene chloride R*. Dilute 1.0 ml of this solution to 20.0 ml with *methylene chloride R*.

Reference solution (c). Dissolve the contents of a vial of *alverine for peak identification CRS* (containing impurities C and E) in 1 ml of *methylene chloride R*.

Column:

- *material*: fused silica;
- *size*: *l* = 25 m, Ø = 0.32 mm;
- *stationary phase*: *poly(dimethyl)(diphenyl)siloxane R* (film thickness 0.45 µm).

Carrier gas: *helium for chromatography R*.

Flow rate: 2.2 ml/min.

Split ratio: 1:11.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 7	120
	7 - 13	120 → 240
	13 - 21	240
	21 - 24	240 → 290
	24 - 39	290
Injection port		290
Detector		290

Detection: flame ionisation.

Injection: 1 µl.

Identification of impurities: use the chromatogram supplied with *alverine for peak identification CRS* and the chromatogram obtained with reference solution (c) to identify the peaks due to impurities C and E.

Relative retention with reference to alverine (retention time = about 16 min): impurity A = about 0.28; impurity B = about 0.29; impurity C = about 0.46; impurity D = about 0.97; impurity E = about 1.7.

System suitability: reference solution (a):

- *resolution*: minimum 3.0 between the peaks due to impurity D and alverine.

Limits:

- *impurities A, B*: for each impurity, maximum 0.1 per cent;
- *impurity C*: maximum 0.2 per cent;
- *impurities D, E*: for each impurity, maximum 0.3 per cent;
- *unspecified impurities*: for each impurity, maximum 0.10 per cent;
- *total*: maximum 1.0 per cent;
- *disregard limit*: the area of the principal peak in the chromatogram obtained with reference solution (b) (0.05 per cent).

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

0.5 g complies with test G. Prepare the reference solution using 1 ml of *lead standard solution (10 ppm Pb) R*.

Loss on drying (*2.2.32*): maximum 0.5 per cent, determined on 1.000 g by drying in an oven at 80 °C for 2 h.

Sulphated ash (*2.4.14*): maximum 0.1 per cent, determined on 1.0 g.

ASSAY

Dissolve 0.375 g in 50 ml of *anhydrous acetic acid R*. Titrate with *0.1 M perchloric acid*, determining the end-point potentiometrically (*2.2.20*).

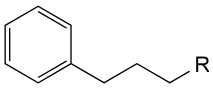
1 ml of *0.1 M perchloric acid* is equivalent to 47.36 mg of $C_{26}H_{35}NO_7$.

STORAGE

Protected from light.

IMPURITIES

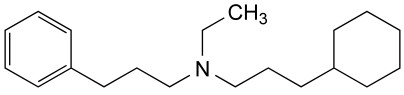
Specified impurities: A, B, C, D, E.



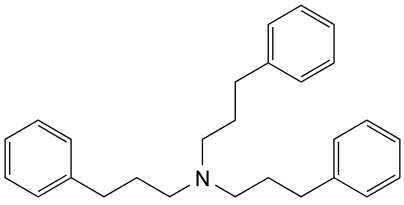
A. R = Cl: 1-chloro-3-phenylpropane,

B. R = OH: 3-phenylpropan-1-ol,

C. R = NH-C_2H_5: *N*-ethyl-3-phenylpropan-1-amine,



D. *N*-(3-cyclohexylpropyl)-*N*-ethyl-3-phenylpropan-1-amine,



E. 3-phenyl-*N*,*N*-bis(3-phenylpropyl)propan-1-amine.