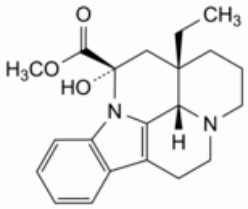
Edition: BP 2025 (Ph. Eur. 11.6 update)

Vincamine

General Notices

(Ph. Eur. monograph 1800)



$C_{21}H_{26}N_2O_3$ 354.5 *1617-90-9*

Action and use

Vasodilator.

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DEFINITION

Methyl 14-hydroxyvincane-14β-carboxylate.

Content

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

CHARACTERS

Appearance

White or almost white, crystalline powder.

Solubility

Practically insoluble in water, soluble in methylene chloride, slightly soluble in anhydrous ethanol.

IDENTIFICATION

A. Specific optical rotation (see Tests).
B. Infrared absorption spectrophotometry (*2.2.24*).

Comparison *vincamine CRS*.

TESTS

Specific optical rotation (*2.2.7*)

+ 44.3 to + 49.0 (dried substance).

Dissolve 0.1 g in *dimethylformamide R* and dilute to 20.0 mL with the same solvent.

Related substances

Liquid chromatography (*2.2.29*). *Prepare the solutions immediately before use. Dissolve the samples using sonication, while avoiding any overheating.*

Test solution Dissolve 50.0 mg of the substance to be examined in 10 mL of *tetrahydrofuran R* and dilute to 100.0 mL with the mobile phase.

Reference solution (a) Dilute 1.0 mL of the test solution to 200.0 mL with the mobile phase.

Reference solution (b) Dissolve 5 mg of *vincamine for system suitability CRS* (containing impurities A, B and C) in 1 mL of *tetrahydrofuran R* and dilute to 10 mL with the mobile phase.

Column:

— *size*: *l* = 0.25 m, Ø = 4.6 mm;

— *stationary phase*: *end-capped octadecylsilyl silica gel for chromatography with embedded polar groups R* (5 µm).

Mobile phase *tetrahydrofuran R*, *acetonitrile R*, 15.4 g/L solution of *ammonium acetate R* (17:18:65 *V/V/V*).

Flow rate 1.0 mL/min.

Detection Spectrophotometer at 272 nm.

Injection 20 µL.

Run time 3.5 times the retention time of vincamine.

Identification of impurities Use the chromatogram supplied with *vincamine for system suitability CRS* and the chromatogram obtained with reference solution (b) to identify the peaks due to impurities A, B and C.

Relative retention With reference to vincamine (retention time = about 10 min): impurity A = about 0.8; impurity B = about 0.9; impurity C = about 1.35.

System suitability Reference solution (b):

— *resolution*: minimum 2.0 between the peaks due to impurity B and vincamine.

Calculation of percentage contents:

— for each impurity, use the concentration of vincamine in reference solution (a).

Limits:

— *impurities A, C*: for each impurity, maximum 0.5 per cent;

— *unspecified impurities*: for each impurity, maximum 0.10 per cent;

— *total*: maximum 1.0 per cent;

— *reporting threshold*: 0.05 per cent.

Loss on drying (*2.2.32*)

Maximum 0.5 per cent, determined on 1.000 g by drying in an oven at 105 °C.

Sulfated ash (*2.4.14*)

Maximum 0.2 per cent, determined on 1.0 g.

ASSAY

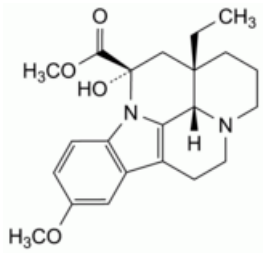
Dissolve 0.300 g in 30 mL of a mixture of 1 volume of *acetic anhydride R* and 5 volumes of *anhydrous acetic acid R*. Titrate with *0.1 M perchloric acid*, determining the end-point potentiometrically (*2.2.20*).

1 mL of *0.1 M perchloric acid* is equivalent to 35.45 mg of $C_{21}H_{26}N_2O_3$.

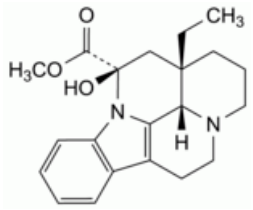
IMPURITIES

Specified impurities A, C.

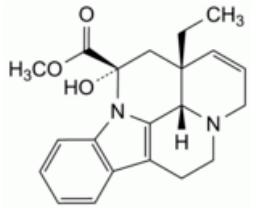
Other detectable impurities (the following substances would, if present at a sufficient level, be detected by one or other of the tests in the monograph. They are limited by the general acceptance criterion for other/unspecified impurities and/or by the general monograph Substances for pharmaceutical use (2034). It is therefore not necessary to identify these impurities for demonstration of compliance. See also 5.10. Control of impurities in substances for pharmaceutical use) B, D.



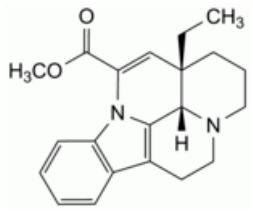
A. methyl 14-hydroxy-10-methoxyvincane-14β-carboxylate (10-methoxyvincamine),



B. methyl 14-hydroxyvincane-14α-carboxylate (14-*epi*-vincamine),



C. methyl 14-hydroxy-17,18-didehydrovincane-14β-carboxylate (17,18-didehydrovincamine),



D. methyl 14,15-didehydrovincane-14-carboxylate (apovincamine).

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