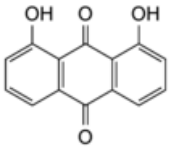
Edition: BP 2025 (Ph. Eur. 11.6 update)

Dantron

General Notices



$C_{14}H_8O_4$ 240.2 *117-10-2*

Action and use

Anthraquinone stimulant laxative.

Preparation

Co-danthrusate Capsules

DEFINITION

Dantron is mainly 1,8-dihydroxyanthraquinone. It contains not less than 98.0% and not more than 102.0% of total phenols, calculated as $C_{14}H_8O_4$ and with reference to the dried substance.

CHARACTERISTICS

An orange, crystalline powder.

Practically insoluble in *water*; slightly soluble in *ether*; very slightly soluble in *ethanol (96%)*. It dissolves in solutions of alkali hydroxides.

IDENTIFICATION

A. The *infrared absorption spectrum*, Appendix II A, is concordant with the *reference spectrum* of dantron *(RS 083)*.
B. The *light absorption*, Appendix II B, in the range 230 to 350 nm of a 0.001% w/v solution in *dichloromethane* exhibits maxima at 255 nm and 285 nm and a less well-defined maximum at 275 nm. The *absorbance* at the maximum at 255 nm is about 0.82 and at the maximum at 285 nm is about 0.48, each calculated with reference to the dried substance.
C. Dissolve 5 mg in 5 mL of 1M *sodium hydroxide*. A clear red solution is produced immediately.

TESTS

Mercury

To 0.50 g in a Kjeldahl flask add 2.5 mL of *nitric acid* and allow to stand until the initial vigorous reaction has subsided. Add 2.5 mL of *sulfuric acid* and heat until dense white fumes are evolved. Cool, add 2.5 mL of *nitric acid* and heat until fumes are again evolved. Repeat the procedure with a further 2.5 mL of *nitric acid*, cool, add 50 mL of *water*, boil the solution until the volume has been reduced to about 25 mL and cool. Transfer to a separating funnel using *water*, dilute to about 50 mL with *water* and add 50 mL of 0.5M *sulfuric acid*. Add 100 mL of *water*, 2 g of *hydroxylamine hydrochloride*, 1 mL of 0.05M *disodium edetate*, 1 mL of *glacial acetic acid* and 5 mL of *dichloromethane*, shake, allow to separate and discard the dichloromethane layer. Titrate the aqueous layer with a 0.0008% w/v solution of *dithizone* in *dichloromethane*, shaking vigorously after each addition, allowing the layers to separate and discarding the dichloromethane layer, until the dichloromethane layer remains green. Repeat the operation using a solution prepared by diluting 1 mL of *mercury standard solution (5 ppm Hg)* to 100 mL with 0.5M *sulfuric acid* and beginning at the words 'Add 100 mL of *water*…'. The volume of the dithizone solution required by the substance being examined does not exceed that required by the mercury standard solution.

Related substances

Carry out the method for *liquid chromatography*, Appendix III D, using the following solutions.

(1) Dissolve 50 mg of the substance being examined in 20 mL of *tetrahydrofuran* and dilute to 100 mL with the mobile phase.
(2) Dilute 1 volume of solution (1) to 50 volumes with the mobile phase.
(3) Dissolve 50 mg of *dantron impurity standard BPCRS* in 20 mL of *tetrahydrofuran* and dilute to 100 mL with the mobile phase.

CHROMATOGRAPHIC CONDITIONS

(a) Use a stainless steel column (25 cm x 4.6 mm) packed with *octadecylsilyl silica gel for chromatography* (5 µm) (Nucleosil C18 is suitable).
(b) Use an isocratic system using the mobile phase described below.
(c) Use a flow rate of 1 mL per minute.
(d) Use an ambient column temperature.
(e) Use a detection wavelength of 254 nm.
(f) Inject 20 µL of each solution.
(g) Allow the chromatography to proceed for 1.5 times the retention time of the principal peak.

MOBILE PHASE

A mixture of 2.5 volumes of *glacial acetic acid*, 40 volumes of *tetrahydrofuran* and 60 volumes of *water*.

SYSTEM SUITABILITY

The test is not valid unless, in the chromatogram obtained with solution (3):

— — a peak due to 1-hydroxyanthraquinone appears immediately before the principal peak, as indicated in the reference chromatogram supplied with *dantron impurity standard BPCRS*;

— the height of the trough separating the two peaks is not greater than one third of the height of the peak due to 1-hydroxyanthraquinone.

LIMITS

In the chromatogram obtained with solution (1):

— the area of any peak corresponding to 1-hydroxyanthraquinone is not greater than 2.5 times the area of the principal peak in the chromatogram obtained with solution (2) (3.3% taking into account the correction factor of the impurity);

— — the sum of the areas of any other *secondary peaks* is not greater than the area of the principal peak in the chromatogram obtained with solution (2) (2%);

— disregard any peak with a retention time less than one third of that of the principal peak.

Loss on drying

When dried to constant weight at 105°, loses not more than 0.5% of its weight. Use 1 g.

ASSAY

Dissolve 0.2 g in 50 mL of *anhydrous pyridine* and carry out Method II for *non-aqueous titration*, Appendix VIII A, using 0.1 M *tetrabutylammonium hydroxide VS* as titrant and determining the end point potentiometrically. Each mL of 0.1M *tetrabutylammonium hydroxide VS* is equivalent to 24.02 mg of total phenols, calculated as $C_{14}H_8O_4$.