



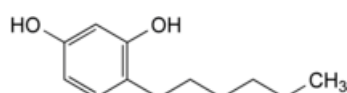
Edition: BP 2025 (Ph. Eur. 11.6 update)

## Hexylresorcinol



### [General Notices](#)

(Ph. Eur. monograph 1437)



$C_{12}H_{18}O_2$  194.3 136-77-6

### Action and use

Antihelminthic.

Ph Eur

## DEFINITION

4-Hexylbenzene-1,3-diol.

### Content

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

## CHARACTERS

### Appearance

Colourless, yellowish or reddish, crystalline powder or needles, turning brownish-pink on exposure to light or air.

### Solubility

Very slightly soluble in water, freely soluble in ethanol (96 per cent) and in methylene chloride.

It shows polymorphism ([5.9](#)).

## IDENTIFICATION

*First identification: B.*

*Second identification: A, C, D.*

A. Melting point ([2.2.14](#)): 66 °C to 68 °C, melting may occur at about 60 °C, followed by solidification and a second melting between 66 °C and 68 °C.

B. Infrared absorption spectrophotometry ([2.2.24](#)).

*Comparison* [hexylresorcinol CRS](#).

If the spectra obtained in the solid state show differences, dissolve the substance to be examined and the reference substance separately in [methanol R](#), evaporate to dryness and record new spectra using the residues.

C. Thin-layer chromatography ([2.2.27](#)).

*Test solution* Dilute 0.1 mL of solution S (see Tests) to 10 mL with [ethanol \(96 per cent\) R](#).

*Reference solution (a)* Dissolve 10 mg of [hexylresorcinol CRS](#) in [ethanol \(96 per cent\) R](#) and dilute to 10 mL with the same solvent.

*Reference solution (b)* Dissolve 10 mg of [hexylresorcinol CRS](#) and 10 mg of [resorcinol R](#) in [ethanol \(96 per cent\) R](#), then dilute to 10 mL with the same solvent.

*Plate* [TLC silica gel G plate R](#).

*Mobile phase* [methyl ethyl ketone R](#), [pentane R](#) (50:50 V/V).

*Application* 10 µL.

*Development* Over 2/3 of the plate.

*Drying* In air for 5 min.

*Detection* Spray with 3 mL of [anisaldehyde solution R](#) and heat at 100-105 °C for 5 min.

*System suitability* Reference solution (b):

— the chromatogram shows 2 clearly separated principal spots.

*Results* The principal spot in the chromatogram obtained with the test solution is similar in position, colour and size to the principal spot in the chromatogram obtained with reference solution (a).

D. Dissolve 0.1 g in 1 mL of [ethanol \(96 per cent\) R](#). Add one drop of [ferric chloride solution R1](#). A green colour is produced. Add [dilute ammonia R1](#). The solution changes to brown.

## TESTS

### Solution S

Dissolve 1.0 g in [ethanol \(96 per cent\) R](#) and dilute to 10.0 mL with the same solvent.

### Appearance of solution

Solution S is clear ([2.2.1](#)).

### Acidity

Dissolve 0.5 g in a mixture of 25 mL of [carbon dioxide-free water R](#) and 25 mL of [ether R](#) previously neutralised to [phenolphthalein solution R1](#) and titrate with [0.1 M sodium hydroxide](#), shaking vigorously after each addition. Not more than 0.4 mL is required to change the colour of the solution.

### Related substances

Liquid chromatography ([2.2.29](#)).

*Test solution* Dissolve 25 mg of the substance to be examined in the mobile phase and dilute to 25.0 mL with the mobile phase.

*Reference solution (a)* Dilute 1.0 mL of the test solution to 100.0 mL with the mobile phase. Dilute 1.0 mL of this solution to 10.0 mL with the mobile phase.

**Reference solution (b)** Dissolve the contents of a vial of [hexylresorcinol for system suitability CRS](#) (containing impurities C and D) in 1.0 mL of the mobile phase.

**Column:**

- **size:**  $l = 0.15$  m,  $\varnothing = 4.6$  mm;
- **stationary phase:** [end-capped octadecylsilyl silica gel for chromatography R](#) (3  $\mu$ m);
- **temperature:** 30 °C.

**Mobile phase** Mix 30 volumes of a 3.0 g/L solution of [glacial acetic acid R](#) previously adjusted to pH 5.9 with [dilute ammonia R1](#), and 70 volumes of [methanol R](#).

**Flow rate** 1.0 mL/min.

**Detection** Spectrophotometer at 281 nm.

**Injection** 20  $\mu$ L.

**Run time** 2.5 times the retention time of hexylresorcinol.

**Identification of impurities** Use the chromatogram supplied with [hexylresorcinol for system suitability CRS](#) and the chromatogram obtained with reference solution (b) to identify the peaks due to impurities C and D.

**Relative retention** With reference to hexylresorcinol (retention time = about 10 min): impurity C = about 0.7; impurity D = about 1.1.

**System suitability** Reference solution (b):

- **peak-to-valley ratio:** minimum 3.0, where  $H_p$  = height above the baseline of the peak due to impurity D and  $H_v$  = height above the baseline of the lowest point of the curve separating this peak from the peak due to hexylresorcinol.

**Calculation of percentage contents:**

- **correction factor:** multiply the peak area of impurity D by 0.3;
- for each impurity, use the concentration of hexylresorcinol in reference solution (a).

**Limits:**

- **impurities C, D:** for each impurity, maximum 0.2 per cent;
- **unspecified impurities:** for each impurity, maximum 0.10 per cent;
- **total:** maximum 0.5 per cent;
- **reporting threshold:** 0.05 per cent.

#### **Water** (2.5.12)

Maximum 0.5 per cent, determined on 1.00 g.

#### **Sulfated ash** (2.4.14)

Maximum 0.1 per cent, determined on 1.0 g.

### **ASSAY**

Dissolve 0.100 g in 10 mL of [methanol R](#) in a ground-glass-stoppered flask, add 30.0 mL of [0.0167 M potassium bromate](#) and 2 g of [potassium bromide R](#). Shake to dissolve the substance and add 15 mL of [dilute sulfuric acid R](#). Stopper the flask, shake and allow to stand in the dark for 15 min, stirring continuously. Add 5 mL of [methylene chloride R](#) and a solution of 1 g of [potassium iodide R](#) in 10 mL of [water R](#), allow to stand in the dark for 15 min, stirring continuously. Titrate with [0.1 M sodium thiosulfate](#), using 1 mL of [starch solution R](#), shaking thoroughly. Carry out a blank titration under the same conditions.

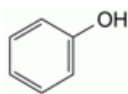
## STORAGE

In an airtight container, protected from light.

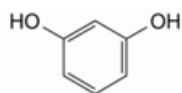
## IMPURITIES

*Specified impurities* C, D.

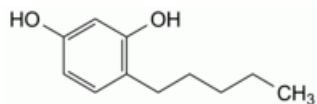
*Other detectable impurities (the following substances would, if present at a sufficient level, be detected by one or other of the tests in the monograph. They are limited by the general acceptance criterion for other/unspecified impurities and/or by the general monograph [Substances for pharmaceutical 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](#))* A, B.



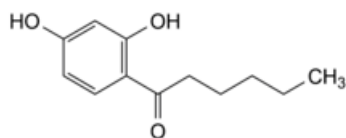
A. phenol,



B. benzene-1,3-diol (resorcinol),



C. 4-pentylbenzene-1,3-diol,



D. 1-(2,4-dihydroxyphenyl)hexan-1-one.

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