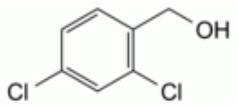
Edition: BP 2025 (Ph. Eur. 11.6 update)

2,4-Dichlorobenzyl Alcohol

General Notices

(Ph. Eur. monograph 2410)



$C_7H_6Cl_2O$ 177.0 *1777-82-8*

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DEFINITION

(2,4-Dichlorophenyl)methanol.

Content

98.0 per cent to 102.0 per cent (anhydrous substance).

CHARACTERS

Appearance

White or almost white, crystalline powder.

Solubility

Very slightly soluble in water, very soluble in ethanol (96 per cent).

mp

About 59 °C.

IDENTIFICATION

Infrared absorption spectrophotometry (2.2.24).

Comparison 2,4-dichlorobenzyl alcohol CRS.

TESTS

Related substances

Liquid chromatography (2.2.29).

Solvent mixture acetonitrile R1, water R (50:50 *V/V*).

Buffer solution Dissolve 0.68 g of potassium dihydrogen phosphate R in 900 mL of water R, adjust to pH 3.0 with phosphoric acid R and dilute to 1000.0 mL with water R.

Test solution (a) Dissolve 0.100 g of the substance to be examined in 10.0 mL of acetonitrile R1 and dilute to 50.0 mL with the solvent mixture.

Test solution (b) Dilute 5.0 mL of test solution (a) to 50.0 mL with the solvent mixture.

Reference solution (a) Dilute 1.0 mL of test solution (a) to 100.0 mL with the solvent mixture. Dilute 1.0 mL of this solution to 10.0 mL with the solvent mixture.

Reference solution (b) Dissolve 20.0 mg of 2,4-dichlorobenzyl alcohol impurity A CRS and 20.0 mg of 2,4-dichlorobenzyl alcohol impurity C CRS in 100.0 mL of acetonitrile R1. Dilute 1.0 mL of the solution to 100.0 mL with the solvent mixture.

Reference solution (c) Dissolve 0.100 g of 2,4-dichlorobenzyl alcohol CRS in 10.0 mL of acetonitrile R1 and dilute to 50.0 mL with the solvent mixture. Dilute 5.0 mL of the solution to 50.0 mL with the solvent mixture.

Column:

— *size*: *l* = 0.15 m, Ø = 4.6 mm;

— *stationary phase*: end-capped octadecylsilyl silica gel for chromatography R (5 µm);

— *temperature*: 30 °C.

Mobile phase:

— *mobile phase A*: methanol R2, acetonitrile R1, buffer solution (20:30:50 *V/V/V*);

— *mobile phase B*: acetonitrile R1;

Time (min)	Mobile phase A (per cent *V/V*)	Mobile phase B (per cent *V/V*)
0 - 7	100	0
7 - 17	100 → 20	0 → 80
17 - 30	20	80

Flow rate 1.2 mL/min.

Detection Spectrophotometer at 214 nm.

Injection 10 µL of test solution (a) and reference solutions (a) and (b).

Relative retention With reference to 2,4-dichlorobenzyl alcohol (retention time = about 7 min): impurity C = about 0.87; impurity A = about 0.91.

System suitability Reference solution (b):

— *peak-to-valley ratio*: minimum 4, where H_p = height above the baseline of the peak due to impurity C and H_v = height above the baseline of the lowest point of the curve separating this peak from the peak due to impurity A.

Calculation of percentage contents:

— for each impurity, use the concentration of 2,4-dichlorobenzyl alcohol in reference solution (a).

Limits:

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

— *total*: maximum 0.3 per cent;

— *reporting threshold*: 0.05 per cent.

Water (*2.5.32*)

Maximum 0.2 per cent, determined on 0.500 g using the evaporation technique:

— *temperature*: 120 °C.

Sulfated ash (*2.4.14*)

Maximum 0.1 per cent, determined on 1.0 g.

ASSAY

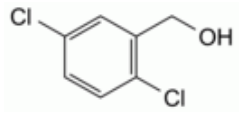
Liquid chromatography (*2.2.29*) as described in the test for related substances with the following modification.

Injection Test solution (b) and reference solution (c).

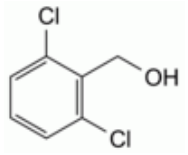
Calculate the percentage content of $C_7H_6Cl_2O$ taking into account the assigned content of *2,4-dichlorobenzyl alcohol CRS*.

IMPURITIES

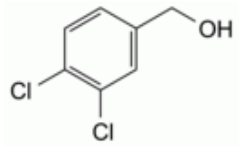
Other detectable impurities (the following substances would, if present at a sufficient level, be detected by one or other of the tests in the monograph. They are limited by the general acceptance criterion for other/unspecified impurities and/or by the general monograph Substances for pharmaceutical 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, C, D, E, F, G.



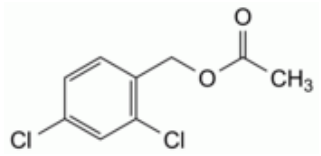
A. (2,5-dichlorophenyl)methanol,



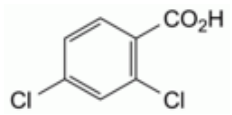
B. (2,6-dichlorophenyl)methanol,



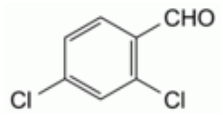
C. (3,4-dichlorophenyl)methanol,



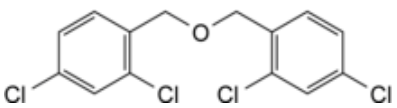
D. 2,4-dichlorobenzyl acetate,



E. 2,4-dichlorobenzoic acid,



F. 2,4-dichlorobenzaldehyde,



G. 1,1'-(oxydimethylene)bis(2,4-dichlorobenzene).

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