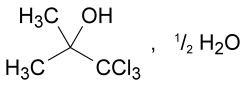
01/2008:0383
corrected 6.0

CHLOROBUTANOL HEMIHYDRATE

Chlorobutanolum hemihydricum



$C_4H_7Cl_3O,{}^1/_2H_2O$ M_r 186.5
[6001-64-5]

DEFINITION

1,1,1-Trichloro-2-methylpropan-2-ol hemihydrate.

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

CHARACTERS

Appearance: white or almost white, crystalline powder or colourless crystals, sublimes readily.

Solubility: slightly soluble in water, very soluble in ethanol (96 per cent), soluble in glycerol (85 per cent).

mp: about 78 °C (without previous drying).

IDENTIFICATION

A. Add about 20 mg to a mixture of 1 ml of *pyridine R* and 2 ml of *strong sodium hydroxide solution R*. Heat in a water-bath and shake. Allow to stand. The pyridine layer becomes red.

B. Add about 20 mg to 5 ml of *ammoniacal silver nitrate solution R* and warm slightly. A black precipitate is formed.

C. To about 20 mg add 3 ml of *1 M sodium hydroxide* and shake to dissolve. Add 5 ml of *water R* and then, slowly, 2 ml of *iodinated potassium iodide solution R*. A yellowish precipitate is formed.

D. Water (see Tests).

TESTS

Solution S. Dissolve 5 g in *ethanol (96 per cent) R* and dilute to 10 ml with the same solvent.

Appearance of solution. Solution S is not more opalescent than reference suspension II (*2.2.1*) and not more intensely coloured than reference solution BY_5 (*2.2.2, Method II*).

Acidity. To 4 ml of solution S add 15 ml of *ethanol (96 per cent) R* and 0.1 ml of *bromothymol blue solution R1*. Not more than 1.0 ml of *0.01 M sodium hydroxide* is required to change the colour of the indicator to blue.

Chlorides (*2.4.4*): maximum 100 ppm.

To 1 ml of solution S add 4 ml of *ethanol (96 per cent) R* and dilute to 15 ml with *water R*. When preparing the standard, replace the 5 ml of *water R* by 5 ml of *ethanol (96 per cent) R*.

Water (*2.5.12*): 4.5 per cent to 5.5 per cent, determined on 0.300 g.

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

ASSAY

Dissolve 0.100 g in 20 ml of *ethanol (96 per cent) R*. Add 10 ml of *dilute sodium hydroxide solution R*, heat in a water-bath for 5 min and cool. Add 20 ml of *dilute nitric acid R*, 25.0 ml of *0.1 M silver nitrate* and 2 ml of *dibutyl phthalate R* and shake vigorously. Add 2 ml of *ferric ammonium sulphate solution R2* and titrate with *0.1 M ammonium thiocyanate* until an orange colour is obtained.

1 ml of *0.1 M silver nitrate* is equivalent to 5.92 mg of $C_4H_7Cl_3O$.

STORAGE

In an airtight container.

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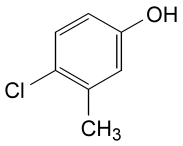
CHLOROCRESOL

Chlorocresolum



C_7H_7ClO M_r 142.6
[59-50-7]

DEFINITION

4-Chloro-3-methylphenol.

Content: 98.0 per cent to 101.0 per cent.

CHARACTERS

Appearance: white or almost white, crystalline powder or compacted crystalline masses supplied as pellets or colourless or white crystals.

Solubility: slightly soluble in water, very soluble in ethanol (96 per cent), freely soluble in fatty oils. It dissolves in solutions of alkali hydroxides.

IDENTIFICATION

A. Melting point (*2.2.14*): 64 °C to 67 °C.

B. To 0.1 g add 0.2 ml of *benzoyl chloride R* and 0.5 ml of *dilute sodium hydroxide solution R*. Shake vigorously until a white, crystalline precipitate is formed. Add 5 ml of *water R* and filter. The precipitate, recrystallised from 5 ml of *methanol R* and dried at 70 °C, melts (*2.2.14*) at 85 °C to 88 °C.

C. To 5 ml of solution S (see Tests) add 0.1 ml of *ferric chloride solution R1*. A bluish colour is produced.

TESTS

Solution S. To 3.0 g, finely powdered, add 60 ml of *carbon dioxide-free water R*, shake for 2 min and filter.

Appearance of solution. The solution is clear (*2.2.1*) and not more intensely coloured than reference solution BY_6 (*2.2.2, Method II*).

Dissolve 1.25 g in *ethanol (96 per cent) R* and dilute to 25 ml with the same solvent.

Acidity. To 10 ml of solution S add 0.1 ml of *methyl red solution R*. The solution is orange or red. Not more than 0.2 ml of *0.01 M sodium hydroxide* is required to produce a pure yellow colour.

Related substances. Gas chromatography (*2.2.28*): use the normalisation procedure.

Test solution. Dissolve 1.0 g of the substance to be examined in *acetone R* and dilute to 100 ml with the same solvent.

Column:

– *material*: glass;

– *size*: *l* = 1.80 m, Ø = 3-4 mm;