Drying: in air.

Detection: examine in ultraviolet light at 254 nm.

Results: the chromatogram obtained with the test solution does not show zones due to benzoic acid and vanillin that are more intense than the corresponding zones in the chromatogram obtained with the reference solution.

Ethanol (*2.9.10*): 95 per cent to 105 per cent of the content stated on the label.

ASSAY

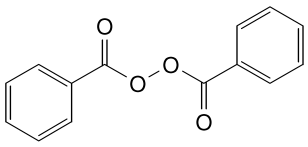
Place 3.50 g in a 250 ml borosilicate glass flask and add 15.0 ml of *0.5 M alcoholic potassium hydroxide*. Boil under a reflux condenser on a water-bath for 30 min. Allow to cool and rinse the condenser with 20 ml of *ethanol (96 per cent) R*. Titrate the excess of potassium hydroxide with *1 M hydrochloric acid*, determining the end-point potentiometrically (*2.2.20*). Carry out a blank titration.

1 ml of *0.5 M alcoholic potassium hydroxide* is equivalent to 61.05 mg of benzoic acid ($C_7H_6O_2$).

01/2008:0704
corrected 6.0

BENZOYL PEROXIDE, HYDROUS

Benzoylis peroxidum cum aqua



$C_{14}H_{10}O_4$ M_r 242.2
[94-36-0]

DEFINITION

Content:

– dibenzoyl peroxide: 70.0 per cent to 77.0 per cent,

– water: minimum 20.0 per cent.

CHARACTERS

Appearance: white or almost white, amorphous or granular powder.

Solubility: practically insoluble in water, soluble in acetone, soluble in methylene chloride with the separation of water, slightly soluble in alcohol.

It loses water rapidly on exposure to air with a risk of explosion.

Mix the entire sample thoroughly before carrying out the following tests.

IDENTIFICATION

First identification: B

Second identification: A, C, D.

A. Dissolve 80.0 mg in *alcohol R* and dilute to 100.0 ml with the same solvent. Dilute 10.0 ml of the solution to 100.0 ml with *alcohol R* (solution A). Dilute 10.0 ml of solution A to 100.0 ml with *alcohol R* (solution B). Examined between 250 nm and 300 nm (*2.2.25*), solution A shows an absorption maximum at 274 nm and a shoulder at about 282 nm. Examined between 220 nm and 250 nm, solution B shows an absorption maximum at 235 nm. The ratio of the absorbance at the maximum at 235 nm (solution B) to that at the maximum at 274 nm (solution A) is 1.17 to 1.21.

B. Infrared absorption spectrophotometry (*2.2.24*).

Comparison: *Ph. Eur. reference spectrum of hydrous benzoyl peroxide.*

C. Dissolve about 25 mg in 2 ml of *acetone R*. Add 1 ml of a 10 g/l solution of *diethylphenylenediamine sulphate R* and mix. A red colour develops which quickly darkens and becomes dark violet within 5 min.

D. To 1 g add 5 ml of *alcohol R*, 5 ml of *dilute sodium hydroxide solution R* and 10 ml of *water R*. Boil the mixture under reflux for 20 min. Cool. The solution gives reaction (c) of benzoates (*2.3.1*).

TESTS

Acidity. Dissolve a quantity of the substance to be examined containing the equivalent of 1.0 g of dibenzoyl peroxide in 25 ml of *acetone R*, add 75 ml of *water R* and filter. Wash the residue with two quantities, each of 10 ml, of *water R*. Combine the filtrate and the washings and add 0.25 ml of *phenolphthalein solution R1*. Not more than 1.25 ml of *0.1 M sodium hydroxide* is required to change the colour of the indicator. Carry out a blank test.

Related substances. Liquid chromatography (*2.2.29*). *Prepare the solutions immediately before use.*

Test solution. Dissolve a quantity of the substance to be examined containing the equivalent of 0.10 g of dibenzoyl peroxide in *acetonitrile R* and dilute to 50 ml with the same solvent.

Reference solution (a). Dilute 1.0 ml of the test solution to 100.0 ml with *acetonitrile R*. Dilute 1.0 ml of this solution to 10.0 ml with *acetonitrile R*.

Reference solution (b). Dissolve 30.0 mg of *benzoic acid R* in the mobile phase and dilute to 100.0 ml with the mobile phase. Dilute 1.0 ml of the solution to 10.0 ml with the mobile phase.

Reference solution (c). Dissolve 50.0 mg of *ethyl benzoate R* in the mobile phase and dilute to 100.0 ml with the mobile phase. Dilute 1.0 ml of the solution to 100.0 ml with the mobile phase.

Reference solution (d). Dissolve 50.0 mg of *benzaldehyde R* in the mobile phase and dilute to 100.0 ml with the mobile phase. Dilute 1.0 ml of the solution to 100.0 ml with the mobile phase.

Reference solution (e). Dissolve 30.0 mg of *benzoic acid R* and 30.0 mg of *benzaldehyde R* in the mobile phase and dilute to 100.0 ml with the mobile phase. Dilute 1.0 ml of the solution to 10.0 ml with the mobile phase.

Column:

– *size*: *l* = 0.25 m, Ø = 4.6 mm,

– *stationary phase*: *octadecylsilyl silica gel for chromatography R* (10 µm),

Mobile phase: *glacial acetic acid R, acetonitrile R, water R* (1:500:500 *V/V/V*).

Flow rate: 1 ml/min.

Detection: spectrophotometer at 235 nm.

Injection: 20 µl loop injector.

Run time: 2 times the retention time of dibenzoyl peroxide.

Relative retention with reference to dibenzoyl peroxide (retention time = about 28.4 min): impurity B = about 0.15; impurity A = about 0.2; impurity C = about 0.4.

System suitability: reference solution (e):

– *resolution*: minimum 6 between the peaks corresponding to benzoic acid and benzaldehyde.

Limits:

– *impurity A*: not more than the area of the principal peak in the chromatogram obtained with reference solution (d) (0.25 per cent),

– *impurity B*: not more than the area of the principal peak in the chromatogram obtained with reference solution (b) (1.5 per cent),

– *impurity C*: not more than the area of the principal peak in the chromatogram obtained with reference solution (c) (0.25 per cent),

– *any other impurity*: not more than the area of the principal peak in the chromatogram obtained with reference solution (a) (0.1 per cent),

– *disregard limit*: 0.2 times the area of the principal peak in the chromatogram obtained with reference solution (a) (0.02 per cent).

Chlorides (*2.4.4*): maximum 0.4 per cent.

Dissolve a quantity of the substance to be examined containing the equivalent of 0.5 g of dibenzoyl peroxide in 15 ml of *acetone R*. Add, while stirring, 50 ml of *0.05 M nitric acid*. Allow to stand for 10 min and filter. Wash the residue with 2 quantities, each of 10 ml, of *0.05 M nitric acid*. Combine the filtrate and the washings and dilute to 100 ml with *0.05 M nitric acid*. 2.5 ml of the solution diluted to 15.0 ml with *water R* complies with the limit test for chlorides.

ASSAY

Solution (a). Dissolve 2.500 g immediately before use in 75 ml of *dimethylformamide R* and dilute to 100.0 ml with the same solvent.

Dibenzoyl peroxide. To 5.0 ml of solution (a) add 20 ml of *acetone R* and 3 ml of a 500 g/l solution of *potassium iodide R* and mix. Allow to stand for 1 min. Titrate with *0.1 M sodium thiosulphate* using 1 ml of *starch solution R*, added towards the end of the titration, as indicator. Carry out a blank titration.

1 ml of *0.1 M sodium thiosulphate* is equivalent to 12.11 mg of $C_{14}H_{10}O_4$.

Water (*2.5.12*). Carry out the semi-micro determination of water, using 5.0 ml of solution (a). Use as the solvent a mixture of 20.0 ml of *anhydrous methanol R* and 3.0 ml of a 100 g/l solution of *potassium iodide R* in *dimethylformamide R*. After adding solution (a), stir for 5 min before starting the titration. Carry out a blank determination.

Calculate the percentage content of water using the expression:

$$\frac{(n_1 - n_2) \times w \times 2}{m} + (p \times 0.0744)$$

n_1 = number of millilitres of *iodosulphurous reagent R* used in the sample determination,

n_2 = number of millilitres of *iodosulphurous reagent R* used in the blank determination,

w = water equivalent of *iodosulphurous reagent R* in milligrams of water per millilitre of reagent,

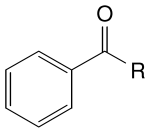
m = mass of the substance to be examined used for the preparation of solution (a) in grams,

p = percentage content of dibenzoyl peroxide.

STORAGE

In a container that has been treated to reduce static discharge and that has a device for release of excess pressure, at a temperature of 2 °C to 8 °C, protected from light.

IMPURITIES



A. R = H: benzaldehyde,

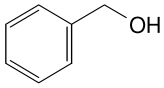
B. R = OH: benzoic acid,

C. R = O-CH_2-CH_3: ethyl benzoate.

01/2008:0256

BENZYL ALCOHOL

Alcohol benzylicus



C_7H_8O M_r 108.1

[100-51-6]

DEFINITION

Phenylmethanol.

Content: 98.0 per cent to 100.5 per cent.

CHARACTERS

Appearance: clear, colourless, oily liquid.

Solubility: soluble in water, miscible with ethanol (96 per cent) and with fatty and essential oils.

Relative density: 1.043 to 1.049.

IDENTIFICATION

Infrared absorption spectrophotometry (*2.2.24*).

Comparison: *benzyl alcohol CRS*.

TESTS

Appearance of solution. Shake 2.0 ml with 60 ml of *water R*. It dissolves completely. The solution is clear (*2.2.1*) and colourless (*2.2.2, Method II*).

Acidity. To 10 ml add 10 ml of *ethanol (96 per cent) R* and 1 ml of *phenolphthalein solution R*. Not more than 1 ml of *0.1 M sodium hydroxide* is required to change the colour of the indicator to pink.

Refractive index (*2.2.6*): 1.538 to 1.541.

Peroxide value (*2.5.5*): maximum 5.

Related substances. Gas chromatography (*2.2.28*).

Test solution. The substance to be examined.

Standard solution (a). Dissolve 0.100 g of *ethylbenzene R* in the test solution and dilute to 10.0 ml with the same solution. Dilute 2.0 ml of this solution to 20.0 ml with the test solution.

Standard solution (b). Dissolve 2.000 g of *dicyclohexyl R* in the test solution and dilute to 10.0 ml with the same solution. Dilute 2.0 ml of this solution to 20.0 ml with the test solution.

Reference solution (a). Dissolve 0.750 g of *benzaldehyde R* and 0.500 g of *cyclohexylmethanol R* in the test solution and dilute to 25.0 ml with the test solution. Add 1.0 ml of