ASSAY

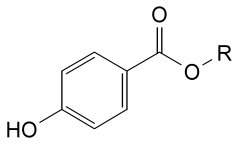
Dissolve 0.150 g in 50 ml of *anhydrous acetic acid R*. Titrate with *0.1 M perchloric acid*, determining the end-point potentiometrically (*2.2.20*).

1 ml of *0.1 M perchloric acid* is equivalent to 18.82 mg of $C_9H_9NaO_3$.

STORAGE

In an airtight container.

IMPURITIES



A. R = H: 4-hydroxybenzoic acid,

B. R = CH_3: methyl 4-hydroxybenzoate,

C. R = CH_2-CH_2-CH_3: propyl 4-hydroxybenzoate,

D. R = CH_2-CH_2-CH_2-CH_3: butyl 4-hydroxybenzoate.

01/2008:0822
corrected 6.0

ETHYLCELLULOSE

Ethylcellulosum

DEFINITION

Partly *O*-ethylated cellulose.

Content: 44.0 per cent to 51.0 per cent of ethoxy (-OC_2H_5) groups (dried substance).

CHARACTERS

Appearance: white or yellowish-white powder or granular powder, odourless or almost odourless.

Solubility: practically insoluble in water, soluble in methylene chloride and in a mixture of 20 g of ethanol (96 per cent) and 80 g of toluene, slightly soluble in ethyl acetate and in methanol, practically insoluble in glycerol (85 per cent) and in propylene glycol. The solutions may show a slight opalescence.

IDENTIFICATION

A. Infrared absorption spectrophotometry (*2.2.24*).
Comparison: *Ph. Eur. reference spectrum of ethylcellulose.*

B. It complies with the limits of the assay.

TESTS

Acidity or alkalinity. To 0.5 g add 25 ml of *carbon dioxide-free water R* and shake for 15 min. Filter through a sintered-glass filter (40) (*2.1.2*). To 10 ml of the solution add 0.1 ml of *phenolphthalein solution R* and 0.5 ml of *0.01 M sodium hydroxide*. The solution is pink. To 10 ml of the solution add 0.1 ml of *methyl red solution R* and 0.5 ml of *0.01 M hydrochloric acid*. The solution is red.

Viscosity (*2.2.9*): 80.0 per cent to 120.0 per cent of that stated on the label for a nominal viscosity greater than 6 mPa·s; 75.0 per cent to 140.0 per cent of that stated on the label for a nominal viscosity not greater than 6 mPa·s.

Shake a quantity of the substance to be examined equivalent to 5.00 g of the dried substance with 95 g of a mixture of 20 g of *ethanol (96 per cent) R* and 80 g of *toluene R* until the substance is dissolved. Determine the viscosity in mPa·s at 25 °C using a capillary viscometer.

Acetaldehyde: maximum 100 ppm.

Introduce 3.0 g into a 250 ml conical flask with a ground-glass stopper, add 10 ml of *water R* and stir mechanically for 1 h. Allow to stand for 24 h, filter and dilute the filtrate to 100.0 ml with *water R*. Transfer 5.0 ml of the filtrate to a 25 ml volumetric flask, add 5 ml of a 0.5 g/l solution of *methylbenzothiazolone hydrazone hydrochloride R* and heat in a water-bath at 60 °C for 5 min. Add 2 ml of *ferric chloride-sulphamic acid reagent R* and heat again in a water-bath at 60 °C for 5 min. Cool and dilute to 25.0 ml with *water R*. The solution is not more intensely coloured than a standard prepared at the same time and in the same manner using instead of the 5.0 ml of filtrate, 5.0 ml of a reference solution prepared by diluting 3.0 ml of *acetaldehyde standard solution (100 ppm C_2H_4O) R1* to 100.0 ml with *water R*.

Chlorides (*2.4.4*): maximum 0.1 per cent.

Disperse 0.250 g in 50 ml of *water R*, heat to boiling and allow to cool, shaking occasionally. Filter and discard the first 10 ml of the filtrate. Dilute 10 ml of the filtrate to 15 ml with *water R*.

Heavy metals (*2.4.8*): maximum 20 ppm.

1.0 g complies with test C. Prepare the reference solution using 2 ml of *lead standard solution (10 ppm Pb) R*.

Loss on drying (*2.2.32*): maximum 3.0 per cent, determined on 1.000 g by drying in an oven at 105 °C for 2 h.

Sulphated ash (*2.4.14*): maximum 0.5 per cent, determined on 1.0 g.

ASSAY

Gas chromatography (*2.2.28*).

CAUTION: hydriodic acid and its reaction by-products are highly toxic. Perform all steps for preparation of the test and reference solutions in a properly functioning hood.

Internal standard solution. Dilute 120 µl of *toluene R* to 10 ml with *o-xylene R*.

Test solution. Transfer 50.0 mg of the substance to be examined, 50.0 mg of *adipic acid R* and 2.0 ml of the internal standard solution into a suitable 5 ml thick-walled reaction vial with a pressure-tight septum-type closure. Cautiously add 2.0 ml of *hydriodic acid R*, immediately close the vial tightly and weigh the contents and the vial accurately. Shake the vial for 30 s, heat to 125 °C for 10 min, allow to cool for 2 min, shake again for 30 s and heat to 125 °C for 10 min. Afterwards allow to cool for 2 min and repeat shaking and heating for a 3rd time. Allow the vial to cool for 45 min and reweigh. If the loss is greater than 10 mg, discard the mixture and prepare another. Use the upper layer.

Reference solution. Transfer 100.0 mg of *adipic acid R*, 4.0 ml of the internal standard solution and 4.0 ml of *hydriodic acid R* into a suitable 10 ml thick-walled reaction vial with a pressure-tight septum-type closure. Close the vial tightly and weigh the vial and contents accurately. Afterwards inject 50 µl of *iodoethane R* through the septum with a syringe, weigh the vial again and calculate the mass of iodoethane added, by difference. Shake well and allow the layers to separate. Use the upper layer.

Column:

– *material*: stainless steel,
– *size*: *l* = 5.0 m, Ø = 2 mm,
– *stationary phase*: *diatomaceous earth for gas chromatography R* (150-180 µm) impregnated with 3 per cent *m/m* of *poly(dimethyl)siloxane R*.

Carrier gas: *nitrogen for chromatography R*.

Flow rate: 15 ml/min.

Temperature:

– *column*: 80 °C,

– *injection port and detector*: 200 °C.

Detection: flame ionisation.

Injection: 1 µl.

Relative retention with reference to toluene: iodoethane = about 0.6; *o*-xylene = about 2.3.

System suitability: reference solution:

– *resolution*: minimum 2.0 between the peaks due to iodoethane and toluene.

Calculate the percentage content of ethoxy groups using the following expression:

$$\frac{Q_1 \times m_2 \times 45.1 \times 100 \times 100}{2 \times Q_2 \times m_1 \times 156.0 \times (100 - d)}$$

Q_1 = ratio of iodoethane peak area to toluene peak area in the chromatogram obtained with the test solution,

Q_2 = ratio of iodoethane peak area to toluene peak area in the chromatogram obtained with the reference solution,

m_1 = mass of the substance to be examined used in the test solution, in milligrams,

m_2 = mass of iodoethane used in the reference solution, in milligrams,

d = percentage loss on drying.

LABELLING

The label states the nominal viscosity in millipascal seconds for a 5 per cent *m/m* solution.

01/2008:1421

ETHYLENE GLYCOL MONOPALMITOSTEARATE

Ethylenglycoli monopalmitostearas

DEFINITION

Mixture of ethylene glycol mono- and diesters of stearic (octadecanoic) and palmitic (hexadecanoic) acids, produced from the condensation of ethylene glycol and stearic acid 50 of vegetable or animal origin (see *Stearic acid (1474)*).

Content: minimum of 50.0 per cent of monoesters.

CHARACTERS

Appearance: white or almost white, waxy solid.

Solubility: practically insoluble in water, soluble in acetone and in hot alcohol.

IDENTIFICATION

A. It complies with the test for melting point (see Tests).

B. It complies with the test for composition of fatty acids (see Tests).

C. It complies with the assay (monoesters content).

TESTS

Melting point (*2.2.15*): 54 °C to 60 °C.

Acid value (*2.5.1*): maximum 3.0, determined on 10.0 g.

Iodine value (*2.5.4*): maximum 3.0.

Saponification value (*2.5.6*): 170 to 195, determined on 2.0 g.

Composition of fatty acids (*2.4.22, Method A*). The fatty acid fraction has the following composition:

– *stearic acid*: 40.0 per cent to 60.0 per cent,

– *sum of contents of palmitic acid and stearic acid*: minimum 90.0 per cent.

Free ethylene glycol: maximum 5.0 per cent, determined as prescribed under Assay.

Total ash (*2.4.16*): maximum 0.1 per cent, determined on 1.0 g.

ASSAY

Size-exclusion chromatography (*2.2.30*).

Test solution. Into a 15 ml flask, weigh about 0.2 g (*m*), to the nearest 0.1 mg. Add 5.0 ml of *tetrahydrofuran R* and shake to dissolve. Heat gently, if necessary. Reweigh the flask and calculate the total mass of solvent and substance (*M*).

Reference solutions. Into four 15 ml flasks, weigh, to the nearest 0.1 mg, about 2.5 mg, 5.0 mg, 10.0 mg and 20.0 mg of *ethylene glycol R*. Add 5.0 ml of *tetrahydrofuran R* and shake to dissolve. Weigh the flasks again and calculate the concentration of ethylene glycol in milligrams per gram for each reference solution.

Column:

– *size*: *l* = 0.6 m, Ø = 7 mm,

– *stationary phase*: *styrene-divinylbenzene copolymer R* (particle diameter 5 µm and pore size 10 nm).

Mobile phase: *tetrahydrofuran R*.

Flow rate: 1 ml/min.

Detection: differential refractometer.

Injection: 40 µl.

Relative retention with reference to ethylene glycol: diesters = about 0.76, monoesters = about 0.83.

Limits:

– *free ethylene glycol*: from the calibration curve obtained with the reference solutions, determine the concentration (*C*) in milligrams per gram in the test solution and calculate the percentage content in the substance to be examined using the following expression:

$$\frac{C \times M}{m \times 10}$$

– *monoesters*: calculate the percentage content of monoesters using the following expression:

$$\frac{A}{A + B} \times (100 - D)$$

A = area of the peak due to the monoesters,

B = area of the peak due to the diesters,

D = percentage content of free ethylene glycol + percentage content of free fatty acids which may be determined using the following expression: $\frac{I_A \times 270}{561.1}$

I_A = acid value.

STORAGE

Protected from light.