E17 ETHYLCELLULOSE

Revision 3 Stage 4

DEFINITION

Ethylcellulose is a partly *O*-ethylated cellulose. It contains not less than 44.0 per cent and not more than 51.0 per cent of ethoxy ($-OC_2H_5$) groups, calculated with reference to the dried substance.

It may contain a suitable antioxidant.

IDENTIFICATION

A. Examine by infrared absorption spectrophotometry.

TESTS

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

Viscosity. 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)* and 80 g of *toluene* until the substance is dissolved. Determine the viscosity using a capillary viscometer. The viscosity, determined at 25 °C and expressed in mPa·s, is not less than 80.0 per cent and not more than 120.0 per cent of that stated on the label for a nominal viscosity greater than 6 mPa·s; and not less than 75.0 per cent and not more than 140.0 per cent of that stated on the label for a nominal viscosity not greater than 6 mPa·s.

Acetaldehyde. Introduce 3.0 g into a 250 ml conical flask with a ground-glass stopper, add 10 ml of *water* and stir mechanically for 1 h. Allow to stand for 24 h, filter and dilute the filtrate to 100.0 ml with *water*. Transfer 5.0 ml to a 25 ml volumetric flask, add 5 ml of a 0.5 g/l solution of *methylbenzothiazolone hydrazone hydrochloride* ($C_8H_{10}ClN_3S,H_2O$) and heat in a water-bath at 60 °C for 5 min. Add 2 ml of *ferric chloride-sulphamic acid reagent* and heat again at 60 °C for 5 min. Cool and dilute to 25.0 ml with *water*. 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)* to 100.0 ml with *water* (100 ppm).

Chlorides (*0.1 per cent*). Disperse 0.250 g in 50 ml of *water*, 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*. Add 1 ml of *dilute nitric acid* and pour the mixture as a single addition into a test-tube containing 1 ml of a 17 g/l solution *of silver nitrate.* Prepare a standard in the same manner using 10 ml of *chloride standard solution (5 ppm Cl)* and 5 ml of *water*. Examine the tubes laterally against a black background. After standing for 5 min

protected from light, any opalescence in the test solution is not more intense than that in the standard.

Loss on drying. Not more than 3.0 per cent, determined on 1.000 g by drying in an oven at 105 °C for 2 h.

Sulphated ash. Not more than 0.5 per cent, determined on 1.0 g.

ASSAY

Gas chromatography (*2.2.28*). *Prepare the solutions immediately before use*.

Internal standard solution. To 10 mL of *o-xylene* add 0.5 mL of n-*octane* and dilute to 100.0 mL with *o-xylene*.

Test solution. To 30.0 mg of the substance to be examined, add 60 mg of *adipic acid* in a reaction vial. Add 2.00 mL of internal standard solution and 1.0 mL of *hydroiodic acid* and close immediately with the valve. Accurately weigh the reaction vial (total mass before heating). Place the vial in an oven or heat in a suitable heater with continuous stirring, maintaining an internal temperature of about 115 ± 2 °C for 70 min. Allow to cool and weigh accurately the reaction vial (total mass after heating). If the difference of the total mass before heating to the total mass after heating is more than 10 mg, prepare a new test solution. After phase separation, pierce through the septum of the vial with a cooled syringe and withdraw a sufficient volume of the upper phase as test solution.

Reference solution. Place 60 mg of *adipic acid* and 2.00 mL of internal standard solution in a reaction vial, add 1.0 mL of *hydroiodic acid* and close immediately with a septum. Weigh accurately the vial then inject 25 µl of *iodoethane* through the septum in the vial, weigh again accurately and mix. After phase separation, pierce through the septum of the vial with a cooled syringe and withdraw a sufficient volume of the upper phase as reference solution.

Column:

— *material* : fused silica,

— *size*: *l* = 30 m, Ø = 0.53 mm,

— *stationary phase*: *poly(dimethyl)siloxane* (3 µm)[1].

Carrier gas : *helium for chromatography*.

Flow rate: 4.2 mL/min.

Split ratio: 1:40.

Temperature:

— *temperature programme* as follows:

[1] RTX-1 Restek, DB-1 or HP-1 are suitable

	Time (min)	Temperature (°C)
Column	0-3	50
	3-8	50 → 100
	8-12.3	100 → 250
	12.3-20.3	250
Injection port		250
Detector		280

Detection: flame ionisation.

Injection: 1 µl.

Relative retention with reference to *n*-octane (retention time = about 10 min): iodoethane: about = 0.6.

System suitability: reference solution:

— *resolution*: minimum 5.0 between the peaks due to *n*-octane and iodoethane;

— relative standard deviation of the response factor of the principal peak: maximum 2.0 per cent after 6 injections.

Calculate the response factor (R) from the following expression:

$$A_1 \times W_1 \times C / A_2 \times 100$$

A_1 = area of the peak due to the internal standard in the chromatogram obtained with the reference solution;

A_2 = area of the peak due to iodoethane in the chromatogram obtained with the reference solution;

W_1 = mass of iodoethane in the reference solution, in milligrams;

C = percentage content of *iodoethane*.

Calculate the percentage content *m/m* of the ethoxy groups from the following expression:

$$A_4 \times R \times M_1 \times 100 / A_3 \times W_2 \times M_2$$

A_3 = area of the peak due to the internal standard in the chromatogram obtained with the test solution;

A_4 = area of the peak due to the iodoethane in the chromatogram obtained with the test solution;

R = response factor of iodoethane;

M_1 = molar mass of ethoxy group (45.1);

M_2 = molar mass of iodoethane (156.0);

W_2 = mass of the sample (dried substance) in the test solution, in milligrams.

LABELLING

The label states:

- the nominal viscosity in millipascal seconds for a 5 per cent *m/m* solution;
- the name and concentration of any added antioxidant.

Reagents

Phenolphthalein solution.
Dissolve 0.1 g of *phenolphthalein* in 80 ml of *ethanol (96 per cent)* and dilute to 100 ml with *water*.

Methyl red solution.
Dissolve 50 mg in a mixture of 1.86 ml of *0.1 M sodium hydroxide* and 50 ml of *ethanol (96 per cent)* and dilute to 100 ml with *water*.

Ferric chloride-sulphamic acid reagent.
A solution containing 10 g/l of *ferric chloride (*$FeCl_3,6H_2O$) and 16 g/l of *sulphamic acid*.

Acetaldehyde standard solution (100 ppm C_2H_4O).
Dissolve 1.0 g of *acetaldehyde* in *water* and dilute to 100.0 ml with the same solvent. Dilute 5.0 ml of the solution to 500.0 ml with *water*. Prepare immediately before use.

Nitric acid, dilute.
Contains about 125 g/l of HNO_3 (M_r 63.0).

Chloride standard solution (5 ppm Cl).
Immediately before use, dilute with *water* to 100 times its volume a solution containing *sodium chloride* equivalent to 0.824 g of NaCl in 1000.0 ml.

Iodoethane. C_2H_5I. (M_r 155.9). [75-03-6].
Colourless or slightly yellowish liquid, darkening on exposure to air and light, miscible with ethanol (96 per cent) and most organic solvents.
d_{20}^{20}: about 1.95.
n_D^{20}: about 1.513.
boiling point: about 72 °C.
Storage: in an airtight container.
Content: minimum 99.0 per cent.