BASIC BUTYLATED METHACRYLATE COPOLYMER

Copolymerum methacrylatis butylati basicum

DEFINITION
Copolymer of 2-(dimethylamino)ethyl methacrylate, butyl methacrylate and methyl methacrylate having a mean relative molecular mass of about 150 000. The ratio of 2-dimethylaminoethyl methacrylate groups to butyl methacrylate and methyl methacrylate groups is about 2:1:1.

CHARACTERS
Appearance: colourless or yellowish granules or white or almost white powder, slightly hygroscopic.

IDENTIFICATION
A. Infrared absorption spectrophotometry (2.2.24).
Comparison: Ph. Eur. reference spectrum of basic butylated methacrylate copolymer.

B. It complies with the limits of the assay.

TESTS
Solution S. Dissolve 12.5 g in a mixture of 35.0 g of acetone R and 52.5 g of 2-propanol R.

Viscosity (2.2.10): 3 mPa·s to 6 mPa·s, determined on solution S.

Apparatus: rotating viscosimeter.

Dimensions:
- spindle: diameter = 25.15 mm, height = 90.74 mm, shaft diameter = 4 mm,
- cylinder: diameter = 27.62 mm, height = 0.135 m.

Stirring speed: 30 r/min.

Volume of solution: 16 ml of solution S.

Temperature: 20 °C.

Absorbance (2.2.25): maximum 0.30 at 420 nm, determined on solution S.

Appearance of film. Spread evenly 1.0 ml of solution S on a glass plate. Upon drying a clear film is formed.

Monomers: maximum 0.3 per cent, for the sum of contents of butyl methacrylate, methyl methacrylate and 2-dimethylaminoethyl methacrylate calculated by procedures A and B.

A. Butyl methacrylate and methyl methacrylate. Liquid chromatography (2.2.29).

Test solution. Dissolve 1.00 g of the substance to be examined in phosphate buffer solution pH 2.0 R and dilute to 50.0 ml with the same buffer solution.

Reference solution. Dissolve 10.0 mg of butyl methacrylate R and 10.0 mg of methyl methacrylate R in 10.0 ml of acetonitrile R and dilute to 50.0 ml with water R. Dilute 1.0 ml of the solution to 50.0 ml with water R.

Column:
- size: l = 0.125 m, Ø = 4.6 mm,
- stationary phase: octadecylsilica gel for chromatography R (7 µm).


Flow rate: 2.0 ml/min.

Detection: spectrophotometer at 205 nm.

Injection: 50 µl.

B. 2-Dimethylaminoethyl methacrylate. Liquid chromatography (2.2.29).

Test solution. Dissolve 1.00 g of the substance to be examined in tetrahydrofuran R and dilute to 50.0 ml with the same solvent.

Reference solution. Dissolve 10.0 mg of 2-(dimethylamino)ethyl methacrylate R in tetrahydrofuran R and dilute to 50.0 ml with the same solvent. Dilute 2.0 ml of the solution to 50.0 ml with tetrahydrofuran R.

Column:
- size: l = 0.125 m, Ø = 4.6 mm,
- stationary phase: aminopropylsilyl silica gel for chromatography R (10 µm).

Mobile phase: mix 25 volumes of a 3.404 g/l solution of potassium dihydrogen phosphate R and 75 volumes of tetrahydrofuran R.

Flow rate: 2.0 ml/min.

Detection: spectrophotometer at 215 nm.

Injection: 50 µl.

Heavy metals (2.4.8): maximum 20 ppm.

2.0 g complies with limit test C. Prepare the standard using 4.0 ml of lead standard solution (10 ppm Pb) R.

Loss on drying (2.2.32): maximum 2.0 per cent, determined on 1.000 g by drying in an oven at 110 °C for 3 h.

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

ASSAY
Dissolve 0.200 g in a mixture of 4 ml of water R and 96 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 7.21 mg of CH₃N₃O₂.

STORAGE
In an airtight container.

IMPURITIES
A. R = [CH₃]₃N: butyl methacrylate,

B. R = CH₃: methyl methacrylate,

C. R = CH₂CH₂N(CH₃)₂: 2-(dimethylamino)ethyl methacrylate.