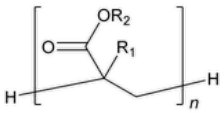
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Add the following:

▲Methyl Acrylate, Methyl Methacrylate and Methacrylic Acid (7:3:1) Copolymer 280000 Dispersion



R_1 = H; R_2 = CH_3 or
R_1 = CH_3; R_2 = CH_3 or
R_1 = CH_3; R_2 = H

Poly(methyl acrylate-*co*-methyl methacrylate-*co*-methacrylic acid) 7:3:1
CAS RN®: 26936-24-3.

DEFINITION

Methyl Acrylate, Methyl Methacrylate and Methacrylic Acid (7:3:1) Copolymer 280000 Dispersion is a 30% aqueous dispersion of a synthetic anionic copolymer based on methyl acrylate, methyl methacrylate, and methacrylic acid, with a weight-average molecular weight of approximately 280,000 g/mol. It contains, on the basis of the calculated amount of dry substance in the Dispersion, NLT 9.2% and NMT 12.3% of methacrylic acid units. It may contain suitable surface-active agents.

IDENTIFICATION

• **A. Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*:** 197F

Analysis: Place 1 drop of Dispersion on a water-resistant crystal disk (silver chloride or KRS-5).[1] After film formation, dry the crystal disk at 80° for approximately 5 min. Let the crystal disk cool down to room temperature prior to measurement.

Acceptance criteria: The IR absorption spectrum of the Dispersion exhibits maxima corresponding to the same wavelengths as those of a similar preparation of USP Methyl Acrylate, Methyl Methacrylate and Methacrylic Acid (7:3:1) Copolymer 280000 Dispersion RS treated in the same manner.

• **B.** It meets the requirements in the *Assay*.

ASSAY

• **Procedure**

Sample solution: Dissolve 5 g of the Dispersion in 10 mL of water and add 90 mL of isopropyl alcohol.

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Direct titration

Titrant: 0.5 N sodium hydroxide VS

Endpoint detection: Potentiometric

Analysis: Titrate the solution as directed in *Titrimetric system*. Each milliliter of 0.5 N sodium hydroxide VS is equivalent to 43.045 mg of methacrylic acid ($C_4H_6O_2$) units.

Calculate, on the dried basis, the percentage of methacrylic acid units in the portion of Dispersion taken:

$$\text{Result} = \frac{V \times N \times M_w}{\frac{W \times (100 - \%\,\text{LOD})}{100}}$$

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$$\times F \times 100$$

V = volume of *Titrant* consumed (mL)

N = normality of the *Titrant* (mol/L)

M_w = molecular weight of methacrylic acid, 86.09 g/mol

W = weight of Dispersion taken (g)

% LOD = the percentage value obtained from the test for *Loss on Drying*

F = conversion factor, 10^{-3} L/mL

Acceptance criteria: 9.2%–12.3% based on the calculated amount of dry substance in the Dispersion

IMPURITIES

- **Residue on Ignition ⟨281⟩**

Sample: 2.0 g of the Dispersion

Analysis: Using mild heating conditions (e.g., steam bath, sand bath) to avoid loss of material, evaporate the Dispersion to dryness prior to ignition.

Acceptance criteria: NMT 0.2% residue is obtained, calculated on the weight of the Dispersion taken

- **Organic Impurities**

Limit of Monomers

Solution A: Add phosphoric acid dropwise to water to obtain a solution with a pH of 2.0.

Solution B: Methanol and *Solution A* (70:30) (v/v)

Mobile phase: Acetonitrile and *Solution A* (10:90) (v/v)

Standard solution: Dissolve approximately 12 mg each of methacrylic acid, methyl acrylate, and methyl methacrylate, each accurately weighed, in 5 mL of isobutyl alcohol, and add acetone to make exactly 50 mL. Dilute 5.0 mL of the solution to 50 mL with acetone. Dilute 20 mL of the solution to 50 mL with acetone. Take 5.0 mL of the solution and mix with 20 mL of *Solution B*. This solution contains about 0.002 mg/mL each of methacrylic acid, methyl acrylate, and methyl methacrylate.

Sample solution: Transfer 11 g of the Dispersion to a 50-mL volumetric flask, dilute with acetone to volume, and mix. Add 5 mL of this solution dropwise while continuously stirring into a beaker that contains 20.0 mL of *Solution B*, accurately measured. Remove the precipitated polymer by centrifugation of an aliquot until the solution is clear.[2] Use the clear supernatant.

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: UV 200 nm

Column: 4.6-mm × 12.5-cm; 7-µm packing L1

Flow rate: 2 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

[Note—The relative retention times for methacrylic acid, methyl acrylate, and methyl methacrylate are 1.0, 1.2, and 3.3, respectively.]

Suitability requirements

Resolution: NLT 1.5 between methacrylic acid and methyl acrylate; NLT 8.0 between methyl acrylate and methyl methacrylate

Tailing factor: 0.8–1.5, determined for each analyte

Relative standard deviation: NMT 5.0%, determined for each analyte

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each monomer in the weight of the Dispersion taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response of the monomer (methacrylic acid, methyl acrylate, or methyl methacrylate) from the *Sample solution*

r_S = peak response of the monomer (methacrylic acid, methyl acrylate, or methyl methacrylate) from the *Standard solution*

C_S = concentration of the monomer (methacrylic acid, methyl acrylate, or methyl methacrylate) in the *Standard solution* (mg/mL)

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