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Methacrylic Acid and Ethyl Acrylate Copolymer Dispersion

DEFINITION

Methacrylic Acid and Ethyl Acrylate Copolymer Dispersion is an aqueous dispersion of Methacrylic Acid and Ethyl Acrylate Copolymer. It contains, on the basis of the calculated amount of dry substance in the Dispersion, NLT 46.0% and NMT 50.6% of methacrylic acid units. It may contain suitable surface-active agents.

IDENTIFICATION

Change to read:

• **A.** ▲Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: **197K**▲ (CN 1-May-2020) **:** Proceed as directed in the chapter, except use the residue obtained in the test for *Loss on Drying* as the sample.

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

ASSAY

• **Procedure**

Sample: 2.5 g of the Dispersion

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Direct titration

Titrant: 0.1 N sodium hydroxide VS

Endpoint detection: Potentiometric

Analysis: Dissolve the *Sample* in 100 mL of neutralized acetone. Titrate the solution as directed in *Titrimetric system*. Each mL of 0.1 N sodium hydroxide is equivalent to 8.609 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:

$$\text{Result} = (V \times N)/[W \times (100 - L)] \times 860.9$$

V = volume of *Titrant* consumed (mL)

N = normality of the *Titrant*

W = weight of Dispersion taken (g)

L = percentage of the *Loss on Drying* value for the Dispersion

Acceptance criteria: 46.0%–50.6% based on the calculated amount of dry substance in the Dispersion

IMPURITIES

• **Residue on Ignition** ⟨281⟩

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 undried Dispersion basis.

• **Limit of Monomers**

Mobile phase: Add phosphoric acid dropwise to water to obtain a solution with a pH of 2.0. Prepare a mixture of this acidified water and methanol (80:20), and degas.

Sodium perchlorate solution: Dissolve 3.5 g of sodium perchlorate in 100 mL of water. This solution has a concentration of 0.25 M.

Standard solution: Dissolve 0.01 g of methacrylic acid and 0.01 g of ethyl acrylate in 5 mL of butanol, and add methanol to make exactly 100 mL. Transfer 1.0 mL of this solution to a 100-mL volumetric flask, and dilute with methanol to volume. Mix 10.0 mL of this solution with 5.0 mL of *Sodium perchlorate solution,* accurately measured. This solution contains about 0.67 µg/mL each of methacrylic acid and ethyl acrylate.

Sample solution: Transfer a quantity of Dispersion, equivalent to 3 g of solids on the dried basis, to a 50-mL volumetric flask, dilute with methanol to volume, and mix. Add 10.0 mL of this solution dropwise while continuously stirring into a beaker that contains 5.0 mL of *Sodium perchlorate solution,* accurately measured. Remove the precipitated polymer by centrifugation (e.g., NLT 5000 × *g* for NLT 5 min). Use the clear supernatant.

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Chromatographic system

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

Mode: LC

Detector: UV 202 nm

Column: 4.0-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 and ethyl acrylate are 1.0 and 2.6, respectively.]

Suitability requirements

Resolution: NLT 2.0 between methacrylic acid and ethyl acrylate

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:

$$\text{Result} = (r_U/r_S) \times (C/W) \times V_F \times D \times F \times 100$$

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

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

C = concentration of the monomer (methacrylic acid or ethyl acrylate) in the *Standard solution* (µg/mL)

W = weight of the Dispersion taken to prepare the *Sample solution* (g)

V_F = final volume of the *Sample solution*, 15 mL

D = dilution factor for preparation of the *Sample solution*, 5

F = conversion factor, 10^{-6} g/µg

Acceptance criteria: NMT 0.01% of total monomers, based on the weight of the Dispersion taken

SPECIFIC TESTS

• **Coagulum Content**

Analysis: Weigh a stainless steel sieve having 90-µm openings or a suitable single-woven wire cloth with a mesh width of 90 µm, and filter 100 g of the Dispersion through it. [Note—Suitable single-woven wire cloth mesh meets the requirements set in ISO 9044.] Wash the sieve or the cloth with distilled water until a clear filtrate is obtained, and dry the sieve or the cloth to constant weight at 110°.

Acceptance criteria: The weight of the residue does not exceed 1000 mg (1%).

• **Loss on Drying ⟨731⟩**

Analysis: Dry at 110° for 6 h.

Acceptance criteria: 68.5%–71.5%

• **Microbial Enumeration Tests ⟨61⟩** and **Tests for Specified Microorganisms ⟨62⟩:** The total aerobic microbial count does not exceed 10^3 cfu/g, and the total combined molds and yeasts count does not exceed 10^2 cfu/g.

• **pH ⟨791⟩:** 2.0–3.0

• **Viscosity—Rotational Methods, *Method II* ⟨912⟩**

Analysis: Equip a suitable rotational viscometer with an adapter comprising a cylindrical spindle rotating within an accurately machined chamber (or tube).[1] Mix the Dispersion, pipet the volume of test specimen recommended by the instrument manufacturer into the chamber (or tube), and ensure that the temperature of the test specimen is at 20 ± 0.1°. The shear rate under the test condition is NLT 1 s^{-1} and NMT 100 s^{-1}.[2] Measure the apparent viscosity following the instrument manufacturer's directions.

Acceptance criteria: The viscosity is between 2 and 15 mPa · s.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers. Store at controlled room temperature. Protect from freezing.

• **Labeling:** The label indicates the name and amount of any substance added as a surface-active agent.

• **USP Reference Standards ⟨11⟩**

USP Methacrylic Acid and Ethyl Acrylate Copolymer (1:1) RS (USP Methacrylic Acid Copolymer Type C RS)

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[1] A commercial device is available from Brookfield as an ultra-low (UL) viscosity adapter. The adapter comprises a 0.4-cm diameter shaft, an accurately machined chamber (or tube) with an internal diameter of 2.8 cm and a depth of 13.5 cm, and a cylindrical spindle 2.5 cm in diameter and 9.1 cm in height.

[2] The cylindrical spindle rotates at 30 rpm, which corresponds to a shear rate of approximately 37 s^{-1}.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
METHACRYLIC ACID AND ETHYL ACRYLATE COPOLYMER DISPERSION	Documentary Standards Support	CE2020 Complex Excipients

Chromatographic Database Information: Chromatographic Database

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