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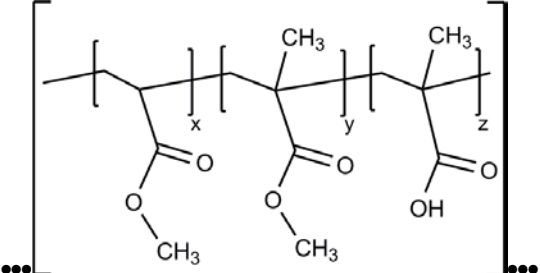
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ANIONIC METHACRYLATE COPOLYMER

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ANIONIC METHACRYLATE COPOLYMER

New specifications prepared at the 86th JECFA (2018) and published in FAO JECFA Monographs 22 (2018). No ADI was established at the 86th JECFA (2018).

SYNONYMS	E 1207, INS No. 1207, acrylates copolymers, Methyl acrylate, methyl methacrylate, methacrylic acid polymer; methacrylic acid, polymer with methyl acrylate and methyl methacrylate
DEFINITION	Anionic methacrylate copolymer is a copolymer comprised of monomers, methyl acrylate, methyl methacrylate, and methacrylic acid in the molar ratio of 7:3:1. The copolymer is manufactured by emulsion polymerization of the monomers with water soluble radical initiators. The product is purified by water vapour distillation and filtration to remove residual monomers, excess water, other volatile low-molecular weight substances and coagulum. The copolymer is standardized as a 30% aqueous dispersion. The copolymer dispersion may contain residual monomers (methyl acrylate, methyl methacrylate, and methacrylic acid).
Chemical name	Poly (methyl acrylate-co-methylmethacrylate-co-methacrylic acid) 7:3:1
C.A.S. number	26936-24-3
Chemical formula	Poly[(CH ₂ :CHCO ₂ CH ₃)-co-(CH ₂ :C(CH ₃)CO ₂ CH ₃)-co-(CH ₂ :C(CH ₃)COOH)]
Structural formula	 <p>The structural formula shows a copolymer chain with three repeating units enclosed in large square brackets with ellipses at the ends. The first unit is methyl acrylate, represented as a carbon atom bonded to a hydrogen atom, a methoxy group (-OCH₃), and a hydrogen atom, with a subscript 'x'. The second unit is methyl methacrylate, represented as a carbon atom bonded to a methyl group (-CH₃), a methoxy group (-OCH₃), and a hydrogen atom, with a subscript 'y'. The third unit is methacrylic acid, represented as a carbon atom bonded to a methyl group (-CH₃), a hydroxyl group (-OH), and a hydrogen atom, with a subscript 'z'. The units are connected by single bonds between the carbon atoms of adjacent units.</p>

The above formula is provided for illustrative purposes; in this copolymer no definitive structural unit can be defined.

Formula weight 280,000 (weight-average), 77,000 (number-average)

Assay 9.2 – 12.3 % methacrylic acid units on the dried basis

See description under Tests

DESCRIPTION Commercial form (30% aqueous dispersion) is a low viscosity, milky-white liquid.

FUNCTIONAL USES Coating agent, glazing agent.

CHARACTERISTICS

IDENTIFICATION

Viscosity (Vol. 4) Not more than 20 mPa•s

Determine viscosity using Brookfield viscometer at 20° and 30 rpm using UL adapter.

pH (Vol 4) 2.0 – 3.5

Infrared absorption (Vol. 4) The infrared absorption spectrum of a dry film of sample corresponds to the infrared spectrum in the Appendix.

Apply one drop of sample to a glass plate, cover with a water-resistant crystal disc (AgCl, KRS 5), press lightly, remove the crystal disc and dry for about 15 minutes at 60°.

PURITY

Loss on drying (Vol. 4) 68.5 – 71.5% (110°, 5 h)

Sulfated ash (Vol. 4) Not more than 0.2%

Test 5 g of the sample (Method I)

Methanol (Vol. 4)

Not more than 1,000 mg/kg

Residual monomers

Methyl acrylate: Not more than 1 mg/kg

Methyl methacrylate: Not more than 3 mg/kg

Methacrylic acid: Not more than 1 mg/kg

See description under TESTS

Lead (Vol. 4)

Not more than 1.0 mg/kg in the dispersion

Determine using a method appropriate to the specified level. The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4 (under “General Methods, Metallic Impurities”).

Microbiological criteria
(Vol. 4)

Total plate count: Less than 1,000 cfu/g

Yeast and moulds: Less than 100 cfu/g

Coliforms: Negative in 10 g

TESTS

IDENTIFICATION
TESTS

Residual monomers

Determined by liquid chromatography (Vol. 4)

Standards and Reagents:

- Acetonitrile: HPLC grade with UV absorption: A_{\max} of 1% at 190 nm
- Acetone, methanol, isobutanol and deionized water: HPLC grade
- Phosphoric acid solution (pH 2): Adjust phosphoric acid (85 %) with an appropriate volume of deionized water to pH 2.

- Standards: methyl acrylate, methyl methacrylate and methacrylic acid (>99%)

Preparation of mixed standard solutions:

Stock mixed standard solution:

Pipette 5 ml of isobutanol into a 50 ml volumetric flask. Accurately weigh approximately 10 mg of methyl acrylate, 12 mg of methyl methacrylate and 11 mg of methacrylic acid, add to isobutanol and dilute to volume with acetone.

Intermediate mixed standard solution-1:

Dilute 5.0 ml of stock mixed standard solution to 50 ml with acetone in a volumetric flask.

Intermediate mixed standard solution-2: Dilute 20.0 ml of intermediate mixed standard solution-1 to 50 ml with acetone in a volumetric flask.

Working mixed standard solution:

Dilute 5 ml of Intermediate mixed standard solution-2 to 25 ml with methanol:phosphoric acid-pH 2 (70:30) in a volumetric flask.

Preparation of sample solution:

Accurately weigh approximately 11 g of sample, dissolve in acetone and dilute to 50 ml in a volumetric flask. Add 5.0 ml of the solution dropwise (precipitation of the polymer should be slow to avoid entrapment of monomer in the precipitate) to 20 ml methanol and phosphoric acid- pH 2 (70:30 v/v). Centrifuge until the supernatant is clear and use the supernatant as the sample solution.

Procedure:

- Use an HPLC with diode array or UV detector at 200 nm
- Column: Octadecylsilane chemically bonded to porous silica (125 cm x 4.6 mm i.d.x 7 µm)
- Injection volume: 20 µl
- Mobile phase: Acetonitrile:Phosphoric acid-pH 2 (10:90 v/v)
- Flow rate: 2 ml/min

Inject separately 20 µl each of working mixed standard solution and sample solution. Calculate the amount of each monomer in the sample from the peak areas obtained in the chromatograms of working mixed standard solution (rR) and sample solution (rS);

amount of standard (R, mg), weight of sample (W, g) and dilution factor (40).

$$\text{Conc. monomer } [\mu\text{g/g}] = \frac{rS \times R \times 40}{rR \times W}$$

Total monomers in the sample ($\mu\text{g/g}$) = Sum of monomers in the sample and correct the results for recovery.

METHOD OF ASSAY Accurately weigh about 5 g sample and dissolve completely in 90 ml isopropyl alcohol and 10 ml water. Titrate with 0.5 N sodium hydroxide standard solution to a potentiometric endpoint. Perform a blank titration under the same conditions. One ml 0.5 N NaOH corresponds to 43.045 mg methacrylic acid units.

$$\text{Methacrylic acid units (\%w/w, on the dried basis)} = \frac{\text{ml of 0.5 N NaOH} \times 430.45}{\text{sample weight (g)} \times \% \text{ dry substance in sample}}$$

Appendix: Infrared spectrum of anionic methacrylate copolymer

