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NEUTRAL METHACRYLATE COPOLYMER (TENTATIVE)

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NEUTRAL METHACRYLATE COPOLYMER (TENTATIVE)

New tentative specifications prepared at the 86th JECFA (2018) and published in FAO JECFA Monographs 22 (2018). An ADI of "not specified" was established at 86th JECFA (2018).

Information required on:

- *A validated method for the assay of neutral methacrylate copolymer (e.g., quantitative IR)*
- *Performance characteristics (method validation data) of the assay method*
- *Assay and monomers data on at least five batches of products currently available in commerce*

SYNONYMS

E 1206, INS No. 1206, Ethyl acrylate methyl methacrylate polymer, Ethyl acrylate methyl methacrylate polymer; Ethyl acrylate polymer with methyl methacrylate, Methyl methacrylate ethyl acrylate polymer, Methyl methacrylate polymer with ethyl acrylate.

DEFINITION

Neutral Methacrylate Copolymer is a copolymer comprised of the monomers ethyl acrylate and methyl methacrylate in the molar ratio of 2:1. 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 with polyethylene glycol monostearyl ether. The copolymer dispersion may contain the residual monomers (methyl methacrylate and ethyl acrylate).

Chemical name

Poly(ethyl acrylate-co-methylmethacrylate)

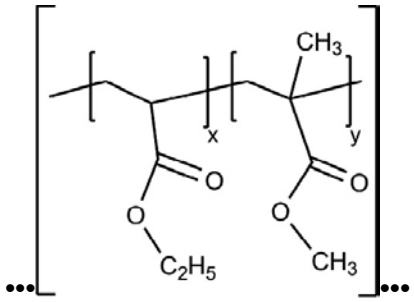
C.A.S. number

9010-88-2

Chemical formula

Poly[(CH_2:$CHCO_2CH_2CH_3$)-co-(CH_2:$C(CH_3)CO_2CH_3$)]

Structural formula



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

Formula weight 600,000 (weight-average), 220,000 (number-average)

Assay Information required

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

FUNCTIONAL USES Coating agent, binding agent, glazing agent

CHARACTERISTICS

IDENTIFICATION

Viscosity (Vol. 4) Not more than 50 mPa.s

Determine viscosity using Brookfield viscometer at 20° and 300 pm using UL adapter.

pH (Vol 4) 5.5 – 8.6

Infrared absorption 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°, 3 h)
Sulfated ash (Vol. 4)	Not more than 0.4% Test 5 g of the sample (Method I)
Residual solvents (Vol. 4)	Methanol: Not more than 100 mg/kg
Residual monomers	Ethanol: Not more than 1,000 mg/kg Methyl methacrylate: Not more than 50 mg/kg Ethyl acrylate: Not more than 20 mg/kg See description under TESTS
Lead (Vol. 4)	Not more than 1.0 mg/kg 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: Not more than 1,000 cfu/g Yeast and moulds: Not more than 100 cfu/g Coliforms: Negative in 10 g

TESTS

PURITY TESTS

Residual monomers — Determined by liquid chromatography (Vol. 4)

Standards and Reagents:

- Acetonitrile: UV absorption: A_{max} of 1% at 190 nm
- Tetrahydrofuran and deionized water:
- Sodium perchlorate (35g/l)
- Standards: Ethyl acrylate and methyl methacrylate (>99%)

Preparation of mixed standard solutions

Stock mixed standard solution (200 µg/ml):
Accurately weigh about 10 mg each of ethyl acrylate and methyl methacrylate, dissolve in tetrahydrofuran and make up to 50 ml with tetrahydrofuran in a volumetric flask.

Intermediate mixed standard solution-1 (20 µg/ml):
Dilute 1.0 ml of stock mixed standard solution to 10 ml with tetrahydrofuran in a volumetric flask.

Intermediate mixed standard solution-2 (2 µg/ml):
Dilute 1.0 ml of intermediate mixed standard solution-1 to 10 ml with tetrahydrofuan in a volumetric flask.

Working mixed standard solution (0.67 µg/ml):
To 10 ml of Intermediate mixed standard solution-2 add 5 ml of sodium perchlorate and mix. Dilute 5 ml of this mixture to 10 ml with deionized water.

Preparation Sample Solution
Accurately weigh approximately1.0 g of sample, dissolve in tetrahydrofuran and dilute to 50.0 ml in a volumetric flask. To 5 ml of sodium perchlorate solution, add 10 ml of sample solution drop wise, whilst stirring continuously. Centrifuge and filter the clear supernatant. Dilute 5 ml of this mixture to 10 ml with deionized water.

Procedure
Use a HPLC with diode array/UV detector at 205 nm
Column: Octadecylsilyl silica gel (12 cm x 4.6 mm i.d., 5-10 µm.)
Injection volume: 50 µl
Mobile phase: Acetonitrile:Water (15:85)
Flow rate: 2 ml/min

Inject separately 50 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 in the injected solution (R, µg) and weight of sample in injected sample solution (W, g)

$$\text{Amount of each monomer } (\mu g/g) = \frac{rS \times R}{rR \times W}$$

Total monomers in the sample (µg/g) = Sum of monomers in the sample

METHOD OF ASSAY Information Required

Appendix: Infrared spectrum of neutral methacrylate copolymer