Ammonio Methacrylate Copolymer

» Ammonio Methacrylate Copolymer is a fully polymerized copolymer of acrylic and methacrylic acid esters with a low content of quaternary ammonium groups. The assay requirements differ for the two types as set forth in the accompanying table.

Type	Ammonio methacrylate units, dried basis (%)	
	Min.	Max.
A	8.85	11.96
B	4.48	6.77

Packaging and storage— Preserve in tight containers, and store at a temperature not exceeding 30°.

Labeling— Label it to state whether it is Type A or Type B.

USP Reference standards 〈 11 〉— *USP Ammonio Methacrylate Copolymer, Type A RS*. *USP Ammonio Methacrylate Copolymer, Type B RS*.

Identification—

A: *Infrared Absorption* 〈 197K 〉.

B: Pour a few mL of the solution prepared for the *Viscosity* test onto a polytef sheet or glass plate, and allow the solvent to evaporate: a clear film results.

Viscosity 〈 911 〉— Place 52.5 g of isopropyl alcohol and 35.0 g of acetone in a conical flask having a ground-glass joint. Add a quantity of Ammonio Methacrylate Copolymer, accurately weighed and equivalent to 12.5 g of solids on a dried basis, while stirring until the polymer has dissolved completely. Equip a suitable rotational viscosimeter with an adapter system consisting of a measuring cylinder and a spindle. The measuring cylinder has an internal diameter of 2.762 cm and a depth of 13.50 cm; the spindle is 2.515 cm in diameter, 9.074 cm in height, and has a shaft 0.40 cm in diameter. Transfer 16 mL of the solution into the measuring cylinder, and adjust the temperature of the solution and the adapter to 20 ± 0.1°. With the spindle rotating at 30 rpm, immediately observe and record the scale reading. Convert the scale reading to centipoises by multiplying the reading by the constant for the viscosimeter, adapter system, and speed employed. The viscosity is not more than 15 centipoises.

Loss on drying 〈 731 〉— Dry it in vacuum at 80° for 5 hours: it loses not more than 3.0% of its weight.

Residue on ignition 〈 281 〉: not more than 0.1%.

Heavy metals, *Method II* 〈 231 〉: 0.002%.

Limit of monomers—

Sodium perchlorate solution— Dissolve 3.5 g of sodium perchlorate ($NaClO_4 \cdot H_2O$) in 100 mL of water.

Mobile phase— Dilute phosphoric acid with water to obtain a solution having a pH of 2.0. Mix four volumes of this solution with one volume of methanol, filter, and degas. Make adjustments if necessary (see *System Suitability* under *Chromatography* 〈 621 〉).

Standard solution— Dissolve 80 mg of ethyl acrylate and 10 mg of methyl methacrylate in 50 mL of methanol.

Standard preparation— Dilute 1 mL of the *Standard solution* with methanol to 100 mL. Add 10 mL of this solution to 5 mL of *Sodium perchlorate solution*.

Test preparation— Dissolve 5 g of Ammonio Methacrylate Copolymer in methanol, and dilute with the same solvent to 50 mL. Add 5 mL of *Sodium perchlorate solution* dropwise to 10 mL of this solution while continuously stirring. Remove the precipitated polymer by centrifugation. Use the clear supernatant.

Chromatographic system (see *Chromatography* 〈 621 〉)— The liquid chromatograph is equipped with a 202-nm detector and a 4.6-mm × 12-cm column that contains packing L1. The flow rate is about 2 mL per minute. Chromatograph the *Standard preparation,* and record the peak responses as directed for *Procedure:* the resolution, *R,* of the pair of analytes is not less than 1.0; the capacity factors, *k '*, for ethyl acrylate and methyl methacrylate are about 9.8 and 11.3, respectively; and the relative standard deviation for replicate injections is not more than 2.0% for each analyte.

Procedure— Separately inject equal volumes (about 50 µL) of the *Standard preparation* and the *Test preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity, in mg, of ethyl acrylate in the portion of Ammonio Methacrylate Copolymer taken by the formula:

$$0.8(r_U / r_S),$$

in which r_U and r_S are the peak responses of ethyl acrylate obtained from the *Test preparation* and the *Standard preparation,* respectively. Calculate the quantity, in mg, of methyl methacrylate in the portion of *Ammonio Methacrylate Copolymer* taken by the formula:

$$0.1(r_U / r_S),$$

in which r_U and r_S are the peak responses of methyl methacrylate obtained from the *Test preparation* and the *Standard preparation,* respectively. Not more than 0.005% of methyl methacrylate and not more than 0.025% of ethyl acrylate are found.

Residual solvents 〈 467 〉 **:** meets the requirements.
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Assay— Dissolve about 1 g of *Ammonio Methacrylate Copolymer Type A*, or 2 g of *Type B*, previously dried and accurately weighed, in a mixture of 96 mL of glacial acetic acid and 4 mL of water. Add 5 mL of mercuric acetate TS and titrate with 0.1 N perchloric acid VS, determining the endpoint potentiometrically. Perform a blank determination and make any necessary correction. Each mL of 0.1 N perchloric acid is equivalent to 20.772 mg of ammonio methacrylate ($C_9H_{18}ClNO_2$) units.