Sample solution for Type C: Transfer 3 g of Methacrylic Acid Copolymer, Type C, to a 50-mL volumetric flask, dilute with methanol to volume, and mix. Add 5 mL of this solution dropwise, while continuously stirring, to a beaker that contains 5.0 mL of *Sodium perchlorate solution*. Remove the precipitated polymer to obtain a clear supernatant by centrifugation (e.g., NLT 5000 × *g* for NLT 5 min). Use the clear supernatant.

Chromatographic system
(See *Chromatography* ⟨621⟩*, System Suitability*.)
Mode: LC
Detector: UV 202 nm
Column: 4.0-mm × 12.5-cm analytical column; 7-µm packing L1
Flow rate: 2 mL/min
Injection size: 20 µL

For Type A or Type B
System suitability
Sample: *Standard solution for Type A or Type B*
[NOTE—The relative retention times for methacrylic acid and methyl methacrylate are 1.0 and 2.8, respectively.]
Suitability requirements
Resolution: NLT 2.0 between methacrylic acid and methyl methacrylate
Relative standard deviation: NMT 5.0%
Analysis
Samples: *Standard solution for Type A or Type B* and *Sample solution for Type A or Type B*
Calculate the percentage of each monomer (methacrylic acid or methyl methacrylate) in the portion of Methacrylic Acid Copolymer Type A or Type B taken:

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

r_U = monomer (methacrylic acid or methyl methacrylate) peak response from the *Sample solution for Type A or Type B*
r_S = monomer (methacrylic acid or methyl methacrylate) peak response from the *Standard solution for Type A or Type B*
C = concentration of the monomer (methacrylic acid or methyl methacrylate) in the *Standard solution for Type A or Type B* (µg/mL)
W = weight of Methacrylic Acid Copolymer Type A or Type B taken to prepare the *Sample solution for Type A or Type B* (g)
V_F = final volume of the *Sample solution for Type A or Type B*, 13 mL
D = dilution factor for preparation of the *Sample solution for Type A or Type B*, 16.7
F = conversion factor, 10^{-6} g/µg

Acceptance criteria: NMT 0.05% for the total amount of monomers

For Type C
System suitability
Sample: *Standard solution for Type C*
[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%
Analysis
Samples: *Standard solution for Type C* and *Sample solution for Type C*
Calculate the percentage of each monomer (methacrylic acid or ethyl acrylate) in the portion of Methacrylic Acid Copolymer Type C taken:

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

r_U = monomer (methacrylic acid or ethyl acrylate) peak response from the *Sample solution for Type C*
r_S = monomer (methacrylic acid or ethyl acrylate) peak response from the *Standard solution for Type C*
C = concentration of the monomer (methacrylic acid or ethyl acrylate) in the *Standard solution for Type C* (µg/mL)
W = weight of Methacrylic Acid Copolymer Type C taken to prepare the *Sample solution for Type C* (g)
V_F = final volume of the *Sample solution for Type C*, 10 mL
D = dilution factor for preparation of the *Sample solution for Type C*, 10
F = conversion factor, 10^{-6} g/µg

Acceptance criteria: NMT 0.01% for the total amount of monomers

SPECIFIC TESTS
- **VISCOSITY** ⟨**911**⟩
Analysis: Place 254.6 g of isopropyl alcohol and 7.9 g of water in a test flask. Add a quantity of Methacrylic Acid Copolymer, equivalent to 37.5 g of solids on the dried basis, while stirring by means of a magnetic stirrer. Close the flask, and continue stirring until the polymer has dissolved completely. Adjust the temperature to 20 ± 0.1°. Equip a rotational viscometer with an accessory.[1] The shear rate under the test condition is NLT 1 s^{-1} and NMT 100 s^{-1}. Follow the instrument manufacturer's directions to measure the apparent viscosity.
Acceptance criteria
Type A: 60–120 mPa · s
Type B: 50–200 mPa · s
Type C: 100–200 mPa · s
- **LOSS ON DRYING** ⟨**731**⟩**:** Dry a sample at 110° for 6 h: it loses NMT 5.0% of its weight.

ADDITIONAL REQUIREMENTS
- **PACKAGING AND STORAGE:** Preserve in tight containers. Store at controlled room temperature.
- **LABELING:** Label it to state whether it is Type A, B, or C. The labeling also indicates the name and quantity of any added surface-active agent.
- **USP REFERENCE STANDARDS** ⟨**11**⟩
USP Methacrylic Acid Copolymer, Type A RS
USP Methacrylic Acid Copolymer, Type B RS
USP Methacrylic Acid Copolymer, Type C RS

Methacrylic Acid Copolymer Dispersion

(Title for this monograph—not to change until May 1, 2017)
(Prior to May 1, 2017, the current practice of labeling the article of commerce with the name Methacrylic Acid Copolymer Dispersion may be continued. Use of the name Methacrylic Acid and Ethyl Acrylate Copolymer Dispersion will be permitted as of May 1, 2012, but the use of this name will not be mandatory until May 1, 2017. The 60-month extension will provide the time needed by manufacturers and users to make necessary changes.)

DEFINITION
Methacrylic Acid Copolymer Dispersion is an aqueous dispersion of Methacrylic Acid and Ethyl Acrylate Copolymer in water. 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.

[1] A suitable accessory is available from Brookfield Engineering as the LV1 spindle, a cylindrical spindle 1.9 cm in diameter and 6.5 cm high attached to a shaft 0.3 cm in diameter. The spindle rotates at 30 rpm at an immersion depth of 8.15 cm.

IDENTIFICATION

• **A. Infrared Absorption ⟨197K⟩:** Proceed as directed, except to use the residue obtained in the test for *Loss on Drying* as the sample.

Change to read:

• **B.** ▲It meets the requirements of the *Assay*.▲*NF30*

ASSAY

Change to read:

• **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▲*NF30*

Analysis: Dissolve the *Sample* in 100 mL of neutralized acetone. ▲Titrate the solution as directed in *Titrimetric system*.▲*NF30* 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

Inorganic Impurities

• **Residue on Ignition ⟨281⟩:** Using mild heating conditions (e.g., steam bath, sand bath) to avoid loss of material, evaporate the Dispersion to dryness prior to ignition: NMT 0.2% residue is obtained, calculated on the undried Dispersion basis.

• **Heavy Metals,** *Method II* **⟨231⟩:** Using mild heating conditions (e.g., steam bath, sand bath) to avoid loss of material, evaporate the Dispersion to dryness prior to wetting with sulfuric acid and ignition: the color of the solution from the *Test Preparation* is not darker than that of the solution from the *Standard Preparation* (20 ppm).

Change to read:

Organic Impurities

• **Limit of Monomers**

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

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

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 5.0 mL of this solution with 5.0 mL of *Sodium perchlorate solution,* accurately measured. This solution contains about 0.5 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 5 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).▲*NF30* Use the clear supernatant.

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 size: 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%,▲*NF30* determined for each analyte

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each monomer in the solid portion of the Dispersion taken:

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

r_U = monomer (methacrylic acid or ethyl acrylate) peak response from the *Sample solution*
r_S = monomer (methacrylic acid or ethyl acrylate) peak response from the *Standard solution*
C = concentration of the monomer (methacrylic acid or ethyl acrylate) in the *Standard solution* (µg/mL)
W = solid weight of the Dispersion, calculated on the dried basis, taken to prepare the *Sample solution* (g)
V_F = final volume of the *Sample solution*, 10 mL
D = dilution factor for preparation of the *Sample solution*, 10
F = conversion factor, 10^{-6} g/µg

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

SPECIFIC TESTS

• **Coagulum Content:** 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°: the weight of the residue does not exceed 1000 mg (1%).

• **Loss on Drying ⟨731⟩:** Dry a sample at 110° for 6 h: it loses 68.5%–71.5% of its weight.

Add the following:

▲• **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.▲*NF30*

• **pH ⟨791⟩:** 2.0–3.0

• **Viscosity ⟨911⟩:** 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),

[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.

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.

Change to read:

- **USP REFERENCE STANDARDS** ⟨**11**⟩
  ▲USP Methacrylic Acid and Ethyl Acrylate Copolymer (1:1) RS (USP Methacrylic Acid Copolymer, Type C RS)▲*NF30*

Add the following:

▲Methacrylic Acid and Ethyl Acrylate Copolymer Dispersion

(Title for this monograph—to become official May 1, 2017)
(Prior to May 1, 2017, the current practice of labeling the article of commerce with the name Methacrylic Acid Copolymer Dispersion may be continued. Use of the name Methacrylic Acid and Ethyl Acrylate Copolymer Dispersion will be permitted as of May 1, 2012, but the use of this name will not be mandatory until May 1, 2017. The 60-month extension will provide the time needed by manufacturers and users to make necessary changes.)

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
- **A. INFRARED ABSORPTION** ⟨**197K**⟩**:** Proceed as directed, except to use the residue obtained in the test for *Loss on Drying* as the sample.
- **B.** It meets the requirements of 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
Inorganic Impurities
- **RESIDUE ON IGNITION** ⟨**281**⟩**:** Using mild heating conditions (e.g., steam bath, sand bath) to avoid loss of material, evaporate the Dispersion to dryness prior to ignition: NMT 0.2% residue is obtained, calculated on the undried Dispersion basis.
- **HEAVY METALS,** *Method II* ⟨**231**⟩**:** Using mild heating conditions (e.g., steam bath, sand bath) to avoid loss of material, evaporate the Dispersion to dryness prior to wetting with sulfuric acid and ignition: the color of the solution from the *Test Preparation* is not darker than that of the solution from the *Standard Preparation* (20 ppm).

Organic Impurities
- **LIMIT OF MONOMERS**

Sodium perchlorate solution: Dissolve 3.5 g of sodium perchlorate in 100 mL of water. This solution has a concentration of 0.25 M.
Mobile phase: Add phosphoric acid dropwise to water to obtain a solution having a pH of 2.0. Prepare a mixture of this acidified water and methanol (80:20), and degas.
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 5.0 mL of this solution with 5.0 mL of *Sodium perchlorate solution,* accurately measured. This solution contains about 0.5 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 5 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.
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 size: 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 solid portion of the Dispersion taken:

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

r_U = monomer (methacrylic acid or ethyl acrylate) peak response from the *Sample solution*
r_S = monomer (methacrylic acid or ethyl acrylate) peak response from the *Standard solution*
C = concentration of the monomer (methacrylic acid or ethyl acrylate) in the *Standard solution* (µg/mL)
W = solid weight of the Dispersion, calculated on the dried basis, taken to prepare the *Sample solution* (g)

[2] The cylindrical spindle rotates at 30 rpm.