Standard solution: Dissolve 0.05 g of methacrylic acid and 0.05 g of methyl methacrylate in 5 mL of butanol, and add methanol to exactly 100 mL. Transfer 1.0 mL of this solution to a 100-mL volumetric flask. Dilute with methanol to volume. Mix 3.0 mL of this solution with 10.0 mL of *Phosphate buffer*. This solution contains 1.15 µg/mL each of methacrylic acid and methyl methacrylate.

Sample solution: Transfer 1 g of Methacrylic Acid and Methyl Methacrylate Copolymer to a 50-mL volumetric flask, dilute with methanol to volume, and mix. Add 3 mL of this solution dropwise, while continuously stirring, to a beaker that contains 10.0 mL of *Phosphate buffer*. 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

System suitability
Sample: *Standard solution*
[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* and *Sample solution*
Calculate the percentage of each monomer (methacrylic acid or methyl methacrylate) in the portion of Methacrylic Acid and Methyl Methacrylate Copolymer 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*
r_S = monomer (methacrylic acid or methyl methacrylate) peak response from the *Standard solution*
C = concentration of the monomer (methacrylic acid or methyl methacrylate) in the *Standard solution* (µg/mL)
W = weight of Methacrylic Acid and Methyl Methacrylate Copolymer taken to prepare the *Sample solution* (g)
V_F = final volume of the *Sample solution*, 13 mL
D = dilution factor for preparation of the *Sample solution*, 16.7
F = conversion factor, 10^{-6} g/µg

Acceptance criteria: NMT 0.05% 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 and Methyl Methacrylate 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
60–120 mPa · s for Methacrylic Acid and Methyl Methacrylate Copolymer having a range of 46.0%–50.6% for methacrylic acid units
50–200 mPa · s for Methacrylic Acid and Methyl Methacrylate Copolymer having a range of 27.6%–30.7% for methacrylic acid units

• **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, and store at controlled room temperature.
• **LABELING:** Label it to indicate the range of methacrylic acid units. The labeling also indicates the name and quantity of any added surface-active agent.
• **USP REFERENCE STANDARDS** ⟨11⟩
USP Methacrylic Acid and Methyl Methacrylate Copolymer (1:1) RS (USP Methacrylic Acid Copolymer, Type A RS)
USP Methacrylic Acid and Methyl Methacrylate Copolymer (1:2) RS (USP Methacrylic Acid Copolymer, Type B RS)

Methyl Alcohol

CH_4O 32.04
Methanol [67-56-1].

DEFINITION

Methyl Alcohol contains NLT 99.5% of CH_3OH.
[**CAUTION—**Methyl Alcohol is poisonous.]

IDENTIFICATION

• **A. INFRARED ABSORPTION** ⟨197F⟩
• **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

• **PROCEDURE**
System suitability solution: Dilute 1.0 mL of USP Methyl Alcohol RS and 1.0 mL of USP Acetone RS with tetrahydrofuran to 50 mL.
Internal standard solution: 2% (v/v) acetonitrile in tetrahydrofuran
Standard solution: 15.8 mg/mL of USP Methyl Alcohol RS in *Internal standard solution*
Sample solution: 15.8 mg/mL of Methyl Alcohol in *Internal standard solution*
Chromatographic system
(See *Chromatography* ⟨621⟩, *System Suitability*.)
Detector: Flame ionization
Column: 0.32-mm × 30-m fused-silica capillary column, coated with a 1.8-µm layer of phase G43
Temperature
Injector: 200°
Detector: 280°
Column: See *Table 1*.

Table 1

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
40	—	40	5
40	20	240	—

[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.

Carrier gas: Helium
Linear velocity: 35 cm/s
Injection type: Split ratio, 20:1
Injection size: 1 µL
System suitability
Samples: *System suitability solution* and *Standard solution*
[NOTE—The relative retention times for methyl alcohol, acetone, and acetonitrile are 1.0, about 1.6, and about 1.8, respectively.]
Suitability requirements
Resolution: NLT 15 between methyl alcohol and acetone, *System suitability solution*
Tailing factor: NLT 1.5 for methyl alcohol, *System suitability solution*
Relative standard deviation: NMT 2.0% for the ratio of the peak area of methyl alcohol to acetonitrile, *Standard solution*
Analysis
Samples: *Standard solution* and *Sample solution*
Calculate the percentage of methyl alcohol (CH_3OH) in the portion of Methyl Alcohol taken:

$$\text{Result} = (R_U/R_S) \times (C_S/C_U) \times 100$$

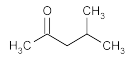
R_U = peak area ratio from the *Sample solution*
R_S = peak area ratio from the *Standard solution*
C_S = concentration of USP Methyl Alcohol RS in the *Standard solution* (mg/mL)
C_U = concentration of Methyl Alcohol in the *Sample solution* (mg/mL)
Acceptance criteria: NLT 99.5%

IMPURITIES
- **NONVOLATILE RESIDUE**
Sample: 250 mL of Methyl Alcohol
Analysis: Evaporate the *Sample* in a 600-mL beaker on a steam bath, in a well-ventilated hood, until the volume is reduced to about 100 mL. Cool, transfer a portion of the liquid to a suitable, tared 50-mL platinum dish on a steam bath, and evaporate. Repeat the process until all of the liquid has been transferred, and then evaporate to dryness. Dry at 105° for 30 min, cool, and weigh.
Acceptance criteria: The weight of the residue does not exceed 2 mg, corresponding to NMT 0.001% (w/w).
- **ACETONE AND ALDEHYDES** (as acetone)
Standard solution: Dilute 1.9 mL (1.5 g) of acetone with water to 1000 mL, then dilute 1.0 mL of this solution with water to 100 mL. Dilute 2 mL of the resulting solution with water to 5 mL. The *Standard solution* contains 30 µg of acetone and is freshly prepared.
Sample solution: Dilute 1.25 mL (1 g) of Methyl Alcohol with water to 5 mL.
Analysis: Adjust to and maintain each solution at 20°. Add 5 mL of alkaline mercuric–potassium iodide TS to each of the *Standard solution* and *Sample solution.*
Acceptance criteria: Any turbidity produced in the *Sample solution* is not greater than that produced in the *Standard solution* (NMT 0.003%).
- **READILY CARBONIZABLE SUBSTANCES** ⟨**271**⟩
Sample: 5 mL
Analysis: Cool 5 mL of sulfuric acid, contained in a small conical flask, to 10°, and add the *Sample* dropwise with constant mixing, maintaining the temperature below 20° throughout the test.
Acceptance criteria: No discoloration develops.
- **READILY OXIDIZABLE SUBSTANCES**
Sample: 20 mL of Methyl Alcohol
Analysis: Cool the *Sample* to 15°, add 0.1 mL of 0.1 N potassium permanganate, and allow to stand at 15°.
Acceptance criteria: The pink color does not completely disappear within 5 min.

SPECIFIC TESTS
- **ACIDITY**
Sample solution: Mix 25 mL of water with 10 mL of alcohol and 0.5 mL of phenolphthalein TS, and add 0.02 N sodium hydroxide until a slight pink color persists after shaking for 30 s. Taking precautions to avoid absorption of carbon dioxide, add 19 mL (15 g) of Methyl Alcohol.
Analysis: Titrate the *Sample solution* with 0.020 N sodium hydroxide.
Acceptance criteria: NMT 0.45 mL of 0.020 N sodium hydroxide is required to produce a pink color.
- **ALKALINITY** (as ammonia)
Sample: 28.6 mL (22.6 g) of Methyl Alcohol
Analysis: Mix the *Sample* with 25 mL of water, add 1 drop of methyl red TS, and titrate with 0.020 N sulfuric acid.
Acceptance criteria: NMT 0.20 mL of 0.020 N sulfuric acid is required to produce a pink color (3 ppm).
- **WATER DETERMINATION,** *Method I* ⟨**921**⟩**:** NMT 0.1%

ADDITIONAL REQUIREMENTS
- **PACKAGING AND STORAGE:** Preserve in tight containers, remote from heat, sparks, and open flames.
- **USP REFERENCE STANDARDS** ⟨**11**⟩
USP Acetone RS
USP Methyl Alcohol RS

Methyl Isobutyl Ketone

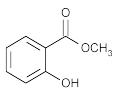


$C_6H_{12}O$ 100.16
2-Pentanone, 4-methyl-.
4-Methyl-2-pentanone [108-10-1].

» Methyl Isobutyl Ketone contains not less than 99.0 percent of $C_6H_{12}O$.

Packaging and storage—Preserve in tight containers.
Identification—The IR absorption spectrum of a thin film of it between sodium chloride crystals exhibits maxima, among others, at the following wavelengths, in µm: 5.81 (*vs*), 6.80 (*m*), 7.00 (*m*), 7.09 (*m*), 7.29 (*vs*), 7.72 (*m*), 8.06 (*m*), 8.31 (*sh*), 8.53 (*s*), and 8.91 (*m*).
Specific gravity ⟨841⟩: not more than 0.799, indicating not less than 99.0% of $C_6H_{12}O$.
Distilling range, *Method I* ⟨721⟩: between 114° and 117°, a correction factor of 0.046° per mm being applied as necessary.
Acidity—Mix 15.0 mL with 15 mL of neutralized alcohol, add phenolphthalein TS, and titrate with 0.050 N sodium hydroxide: not more than 0.40 mL is required for neutralization.
Limit of nonvolatile residue—Evaporate 50 mL in a tared porcelain dish on a steam bath, and dry at 105° for 1 hour: the weight of the residue does not exceed 4 mg (0.008%).

Methyl Salicylate



$C_8H_8O_3$ 152.15
Benzoic acid, 2-hydroxy-, methyl ester;
Methyl salicylate [119-36-8].