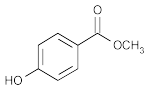
Methylparaben



$C_8H_8O_3$ 152.15
Benzoic acid, 4-hydroxy-, methyl ester;
Methyl *p*-hydroxybenzoate [99-76-3].

DEFINITION
Methylparaben contains NLT 98.0% and NMT 102.0% of $C_8H_8O_3$.

IDENTIFICATION
- **A. Infrared Absorption** ⟨197M⟩
- **B. Melting Range or Temperature** ⟨741⟩**:** 125°–128°

ASSAY
- **Procedure**

Mobile phase, Sample solution, Standard solution B, and **Chromatographic system:** Proceed as described in the procedure for *Related Substances.*

System suitability

Sample: *Standard solution B*

Suitability requirements

Relative standard deviation: NMT 0.85% for 6 injections

Analysis

Samples: *Sample solution* and *Standard solution B*

Calculate the percentage of Methylparaben in the *Sample solution*:

$$\text{Result} = P \times (r_U \times C_S)/(r_S \times C_U)$$

P = labeled purity of USP Methylparaben RS expressed as a percentage
r_U = peak area of methylparaben from the *Sample solution*
C_S = concentration of methylparaben in *Standard solution B*
r_S = peak area of methylparaben from *Standard solution B*
C_U = concentration of Methylparaben in the *Sample solution*

Acceptance criteria: 98.0%–102.0%

IMPURITIES
Inorganic Impurities
- **Residue on Ignition** ⟨281⟩**:** NMT 0.1%, determined on 1.0 g

Organic Impurities
- **Procedure: Related Substances**

Mobile phase: Methanol and a 6.8 g/L solution of potassium dihydrogen phosphate (65:35 v/v)

Sample solution: Dissolve 50.0 mg of Methylparaben in 2.5 mL of methanol, and dilute with *Mobile phase* to 50.0 mL. Dilute 10.0 mL of this solution with *Mobile phase* to 100.0 mL.

Standard solution A: 5.0 µg/mL each of *p*-hydroxybenzoic acid and USP Methylparaben RS in *Mobile phase*

Standard solution B: Dissolve 50.0 mg of USP Methylparaben RS in 2.5 mL of methanol, and dilute with *Mobile phase* to 50.0 mL. Dilute 10.0 mL of this solution with *Mobile phase* to 100.0 mL.

Standard solution C: Dilute 1.0 mL of the *Sample solution* with *Mobile phase* to 20.0 mL. Dilute 1.0 mL of this solution with *Mobile phase* to 10.0 mL.

Chromatographic system
(See *Chromatography* ⟨621⟩*, System Suitability*.)

Mode: LC
Detector: UV 272 nm
Column: 4.6-mm × 15-cm; 5-µm packing L1
Flow rate: 1.3 mL/min
Injection size: 10 µL
Run time: About 5 times the retention time of methylparaben

System suitability

Sample: *Standard solution A*

[NOTE—The retention time of methylparaben is about 2.3 min; the relative retention time for *p*-hydroxybenzoic acid is about 0.6.]

Suitability requirements

Resolution: NLT 2.0 between the *p*-hydroxybenzoic acid and methylparaben peaks

Analysis

Samples: *Sample solution* and *Standard solution C*

[NOTE—Disregard any limit that is 0.2 times the area of the principal peak in the chromatogram obtained with *Standard solution C* (0.1%).]

Acceptance criteria

***p*-Hydroxybenzoic acid:** The peak area in the *Sample solution*, multiplied by 1.4 to correct for the calculation of content, is NMT the area of the principal peak in *Standard solution C* (0.5%).

Unspecified impurities: The peak area of each impurity in the *Sample solution* is NMT the area of the principal peak in *Standard solution C* (0.5%).

Total impurities: The total peak area for all impurities in the *Sample solution* is NMT twice the area of the principal peak in *Standard solution C* (1.0%).

SPECIFIC TESTS
- **Color of Solution**

Sample solution: 100 mg/mL in alcohol

Comparison solution: Mix 2.4 mL of ferric chloride CS, 1.0 mL of cobaltous chloride CS, and 0.4 mL of cupric sulfate CS with 0.3 N hydrochloric acid to make 10 mL. Dilute 5 mL of this solution with 0.3 N hydrochloric acid to make 100 mL. [NOTE—Prepare and use this solution immediately.]

Analysis

Samples: Alcohol, *Sample solution*, and *Comparison solution*

Make the comparison by viewing the solutions downward in matched color-comparison tubes against a white surface (see *Color and Achromicity* ⟨631⟩).

Acceptance criteria: The *Sample solution* is clear and not more intensely colored than alcohol or the *Comparison solution*.

- **Acidity**

Sample solution: To 2 mL of the *Sample solution* prepared in the test for *Color of Solution*, add 3 mL of alcohol, 5 mL of carbon dioxide-free water, and 0.1 mL of bromocresol green TS.

Analysis: Titrate with 0.10 N sodium hydroxide.

Acceptance criteria: NMT 0.1 mL is required to produce a blue color.

ADDITIONAL REQUIREMENTS
- **Packaging and Storage:** Preserve in well-closed containers.
- **USP Reference Standards** ⟨11⟩
  USP Methylparaben RS

Methylparaben Sodium

» Methylparaben Sodium contains not less than 98.5 percent and not more than 101.5 percent of $C_8H_7NaO_3$, calculated on the anhydrous basis.

Packaging and storage—Preserve in tight containers.

USP Reference standards ⟨11⟩**—**
USP Methylparaben RS