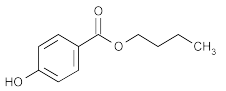
Butylparaben

Portions of the monograph text that are national *USP* text, and are not part of the harmonized text, are marked with symbols (◆◆) to specify this fact.



$C_{11}H_{14}O_3$ 194.23
Benzoic acid, 4-hydroxy-, butyl ester;
Butyl *p*-hydroxybenzoate [94-26-8].

DEFINITION

Butylparaben contains NLT 98.0% and NMT 102.0% of $C_{11}H_{14}O_3$.

IDENTIFICATION

Change to read:

- **A. Infrared Absorption** ■⟨197M⟩■2S *(NF31)*
- **B. Melting Range or Temperature** ⟨741⟩**:** 68°–71°

ASSAY

Change to read:

- **Procedure**

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

System suitability

Sample: *Standard solution B*

Suitability requirements

Relative standard deviation: NMT 0.85% for six injections

Analysis

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

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

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

P = labeled purity of USP Butylparaben RS expressed as a percentage
r_U = peak area of butylparaben from the *Sample solution*
C_S = concentration of butylparaben in *Standard solution B* (mg/mL)
r_S = peak area of butylparaben from *Standard solution B*
C_U = concentration of Butylparaben in the *Sample solution* (mg/mL)

Acceptance criteria: 98.0%–102.0%■2S *(NF31)*

IMPURITIES

- **Residue on Ignition** ⟨281⟩**:** NMT 0.1%, determined on a 1.0-g sample

Change to read:

- **Related Substances**

■Mobile phase: Methanol and a 6.8 g/L solution of potassium dihydrogen phosphate (1:1 v/v)

Sample solution: Dissolve 50.0 mg of Butylparaben 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, USP Propylparaben RS, and USP Butylparaben RS in *Mobile phase*

Standard solution B: Dissolve 50.0 mg of USP Butylparaben 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.

Standard solution D: 50 µg/mL of iso-butylparaben in *Mobile phase*

Standard solution E: *Standard solution D* in *Standard solution B* (1 in 100)

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 volume: 10 µL

Run time: About 1.5 times the retention time of butylparaben

System suitability

Sample: *Standard solutions A* and *E*

[NOTE—The retention time of butylparaben is about 22 min; the relative retention times for *p*-hydroxybenzoic acid, propylparaben, and iso-butylparaben with a reference to butylparaben are about 0.1, 0.5, and 0.9 min, respectively.]

Suitability requirements

Resolution: NLT 5.0 between the propylparaben and butylparaben peaks from *Standard solution A* and NLT 1.5 between the iso-butylparaben and butylparaben peaks from *Standard solution E*

Analysis

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

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

Acceptance criteria

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

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

Total impurities: The total peak area for all impurities from the *Sample solution* is NMT twice the area of the principal peak from *Standard solution C* (1.0%).■2S *(NF31)*

SPECIFIC TESTS

- **Acidity:** To 2 mL of *Butylparaben solution* prepared in the *Color of Solution* test add 3 mL of alcohol, 5 mL of carbon dioxide-free water, and 0.1 mL of bromocresol green TS. Titrate with 0.10 N sodium hydroxide.

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

- **Color of Solution**

Butylparaben solution: Dissolve 1 g in alcohol, and dilute with alcohol to 10 mL.

Acceptance criteria: This solution is clear and not more intensely colored than alcohol or a solution prepared immediately before use by mixing 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, and diluting 5 mL of this solution with 0.3 N hydrochloric acid to make 100 mL. Make

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

ADDITIONAL REQUIREMENTS

- **◆PACKAGING AND STORAGE:** Preserve in well-closed containers.

- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Butylparaben RS
  USP Propylparaben RS◆