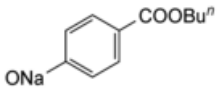
Edition: BP 2025 (Ph. Eur. 11.6 update)

Sodium Butyl Hydroxybenzoate

General Notices

Sodium Butylparaben



$C_{11}H_{13}NaO_3$ 216.2 *36457-20-2*

Action and use

Antimicrobial preservative.

DEFINITION

Sodium Butyl Hydroxybenzoate is the sodium salt of butyl 4-hydroxybenzoate. It contains not less than 99.0% and not more than 102.0% of $C_{11}H_{13}NaO_3$, calculated with reference to the anhydrous substance.

CHARACTERISTICS

A white powder; hygroscopic.

Freely soluble in *water* and in *ethanol (96%)*.

IDENTIFICATION

A. Dissolve 0.5 g in 5 mL of *water* and acidify to *litmus paper* with *hydrochloric acid*. A white precipitate is produced. Wash the precipitate with *water* and dry. The *infrared absorption spectrum* of the precipitate, Appendix II A, is concordant with the *reference spectrum* of butyl hydroxybenzoate *(RS 036)*.
B. The residue on ignition yields the reactions characteristic of *sodium salts*, Appendix VI.

TESTS

Alkalinity

pH of a 0.1% w/v solution, 9.5 to 10.5, Appendix V L.

Clarity of solution

Dissolve 1.0 g in 10 mL of *water*. The solution is *clear*, Appendix IV A.

Chloride

Dissolve 1.0 g in 100 mL of *water*, add 1 mL of *nitric acid* and filter. 15 mL of the filtrate complies with the *limit test for chlorides*, Appendix VII (330 ppm).

Sulfate

Dissolve 0.50 g in 40 mL of *water*, add 3.5 mL of *2M hydrochloric acid*, dilute to 60 mL with *water* and filter. 15 mL of the filtrate complies with the *limit test for sulfates*, Appendix VII (0.12%).

Related substances

Carry out the method for *thin-layer chromatography*, Appendix III A, using the following solutions.

(1) Dilute a 2.0% w/v solution of the substance being examined in *water* with an equal volume of *acetone*.
(2) Dilute 1 volume of solution (1) to 25 volumes with a mixture of equal volumes of *acetone* and *water*.

CHROMATOGRAPHIC CONDITIONS

(a) Use as the coating silica gel F_{254} the surface of which has been modified with chemically-bonded octadecylsilyl groups (Whatman KC18F plates are suitable).
(b) Use the mobile phase as described below.
(c) Apply 2 µL of each solution.
(d) Develop the plate to 15 cm.
(e) After removal of the plate, dry in air and examine under *ultraviolet light (254 nm)*.

MOBILE PHASE

1 volume of *glacial acetic acid*, 30 volumes of *water* and 70 volumes of *methanol*.

LIMITS

Any *secondary spot* in the chromatogram obtained with solution (1) is not more intense than the spot in the chromatogram obtained with solution (2).

Water

Not more than 5.0% w/w, Appendix IX C. Use 1 g.

ASSAY

Gently boil 0.1 g under a reflux condenser with 25 mL of 1.25M *sodium hydroxide* for 30 minutes. Allow to cool, add 25 mL of *0.0333M potassium bromate VS*, 5 mL of a 12.5% w/v solution of *potassium bromide* and 10 mL of *hydrochloric acid* and immediately stopper the flask. Shake for 15 minutes and allow to stand for 15 minutes. Add 25 mL of *dilute potassium iodide solution* and shake vigorously. Titrate the liberated iodine with *0.1M sodium thiosulfate VS* using *starch mucilage*, added towards the end of the titration, as indicator. Repeat the operation without the substance being examined. The difference between the titrations represents the amount of potassium bromate required. The volume of *0.0333M potassium bromate VS* used is equivalent to half of the volume of *0.1M sodium thiosulfate VS* required for the titration. Each mL of *0.0333M potassium bromate VS* is equivalent to 7.207 mg of $C_{11}H_{13}NaO_3$.