

PROPYL GALLATE

Prepared at the 46th JECFA (1996), published in FNP 52 Add 4 (1996) superseding specifications prepared at the 41st JECFA (1993), published in FNP 52 Add 2 (1993). Metals and arsenic specifications revised at the 61st JECFA (2003). An ADI of 0-1.4 mg/kg bw was established at the 46th JECFA (1996)

SYNONYMS

INS No. 310

DEFINITION

Chemical names

Propyl gallate, propyl ester of gallic acid, n-propyl ester of 3,4,5-trihydroxybenzoic acid, propyl 3,4,5-trihydroxybenzoate

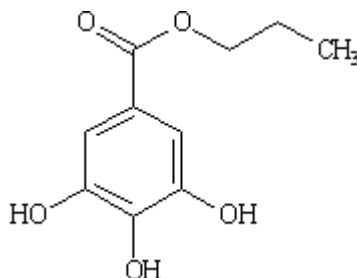
C.A.S. number

121-79-9

Chemical formula

$C_{10}H_{12}O_5$

Structural formula



Formula weight

212.20

Assay

Not less than 98.0% and not more than 102.0% on the dried basis

DESCRIPTION

White or creamy-white, crystalline odourless solid

FUNCTIONAL USES

Antioxidant

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Slightly soluble in water; freely soluble in ethanol, ether and propane-1,2-diol

Melting range (Vol. 4)

146 - 150° after drying

Gallic acid

Dissolve about 0.5 g of the sample in 10 ml of sodium hydroxide TS and boil for 30 min under nitrogen. Maintaining a stream of nitrogen, cool the mixture and acidify to pH 2-3 with sulfuric acid TS. Filter the precipitate through a sintered glass crucible, wash with a minimum amount of water and then dry at 110° for 2 h. The melting point of the gallic acid so obtained is about 240°, with decomposition.

TLC-separation of gallate esters Use a thin-layer plate prepared with silica gel G. Prepare a sample solution by dissolving 10 mg of sample in 10 ml ethanol. Prepare control solution A by dissolving 10 mg of propyl gallate in 10 ml ethanol and control solution B by dissolving 10 mg of propyl gallate and 10 mg of octyl gallate in 10 ml ethanol.

Place 5 µl of each solution on the plate. Develop the chromatogram to about 15 cm from the starting point using a developing solvent containing glacial acetic acid, petroleum ether and toluene (1:2:2).

Dry the plate in air. Spray the plate with an indicator solution, containing 20% w/v phosphomolybdic acid in ethanol until a yellow colouration persists.

Examine in daylight. After a few min there is a progressive change to blue colouration. After 5 to 10 min expose the plate to ammonia vapours until the background is white.

Examine in daylight. The principal spot of the sample solution corresponds with that for propyl gallate in the control solutions. Suitable resolution of propyl and octyl gallate spots is determined from control solution B.

PURITY

Loss on drying (Vol. 4) Not more than 0.5% (110°, 4 h)
Note: Ventilation during drying is advisable

Sulfated ash (Vol. 4) Not more than 0.1%
Test 2 g of the sample (Method I)

Chlorinated organic compounds Not more than 100 mg/kg as chlorine
Dissolve 1 g of the sample in 10 ml of 0.1N sodium hydroxide. Acidify with nitric acid TS and filter off the precipitate. Mix the precipitate with 2 g of calcium carbonate, dry the mixture and then ignite. Take up the ignition residue in 20 ml of dilute nitric acid TS and filter. Mix the solution with 0.5 ml of 0.1 N silver nitrate. The turbidity should not be more than that obtained in 20 ml of dilute nitric acid TS containing 0.5 ml of 0.1N silver nitrate and 0.3 ml of 0.01N hydrochloric acid.

Free acid Not more than 0.5% as gallic acid
To a mixture of 50 ml of carbon dioxide-free water and 50 ml of acetone, add 5 drops of bromocresol green TS and titrate with 0.005N hydrochloric acid to match a buffer (pH 5) TS containing the same amount of indicator. Dissolve 0.4 g of the sample in 50 ml of acetone and add 50 ml of carbon dioxide-free water, 5 drops of bromocresol green TS and the amount of 0.005N hydrochloric acid found in the preliminary test to bring the solvent to pH 5. Titrate the solution back to pH 5 with 0.05N sodium hydroxide, matching against the buffer (pH 5) TS. Each ml of 0.05N sodium hydroxide is equivalent to 8.506 mg of gallic acid.

Lead (Vol. 4) Not more than 2 mg/kg
Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample

preparation may be based on the principles of the method described in Volume 4, "Instrumental Methods."

METHOD OF ASSAY

Weigh accurately about 0.2 g of the dried sample into a 400-ml beaker. Add 150 ml of water and heat to boiling. Then with constant and vigorous stirring add 50 ml of bismuth nitrate TS (II). Continue stirring for a few min more until precipitation is complete, then allow the solution to cool to room temperature. Filter the yellow precipitate onto a weighed sintered-glass crucible, wash first with cold 0.05N nitric acid and then with ice-cold water, until free from acid. Dry at 110° to constant weight. Proceed as directed in the specifications for dodecyl gallate. Calculate the propyl gallate content by the formula:

$$\% \text{ Propyl gallate} = \frac{\text{Weight of precipitate} \times 48.63}{\text{Weight of sample}}$$