

# DODECYL 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). A temporary ADI established at the 41st JECFA (1993) was deleted at the 46th JECFA (1996) and no ADI allocated.

## SYNONYMS DEFINITION

Lauryl gallate, INS No. 312

Chemical names

Dodecyl gallate, n-dodecyl (or lauryl) ester of 3,4,5-trihydroxy-benzoic acid, dodecyl ester of gallic acid, dodecyl 3,4,5-tri-hydroxybenzoate

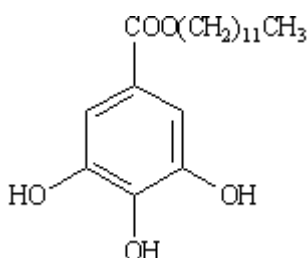
C.A.S. number

1166-52-5

Chemical formula

$C_{19}H_{30}O_5$

Structural formula



Formula weight

338.45

Assay

Not less than 98.5% on the dried basis

## DESCRIPTION

White to creamy-white crystalline odourless solid

**FUNCTIONAL USES** Antioxidant

## CHARACTERISTICS

### IDENTIFICATION

Solubility (Vol. 4)

Insoluble in water; freely soluble in ethanol and ether

Melting range (Vol. 4)

95 - 98° 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 dodecyl gallate in 10 ml ethanol, and control solution B by dissolving 10 mg of dodecyl 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 20 volumes glacial acetic acid, 40 volumes petroleum ether and 40 volumes toluene. 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 dodecyl gallate in the control solutions. Suitable resolution of dodecyl and octyl gallate spots is determined from control solution B.

#### PURITY

##### Loss on drying (Vol. 4)

Not more than 0.5% (90°, 6 h)  
Note: Ventilation during drying is advisable

##### Sulfated ash (Vol. 4)

Not more than 0.05%  
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 on 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. Calculate the dodecyl gallate content by the formula:

$$\% \text{ Dodecyl gallate} = \frac{\text{Weight of precipitate} \times 60.17}{\text{Weight of sample}}$$