BUTYLATED HYDROXYTOLUENE

Prepared at the 37th JECFA (1990), published in FNP 52 (1992) superseding specifications prepared at the 30th JECFA (1986), published in FNP 37 (1986). Metals and arsenic specifications revised at the 61st JECFA (2003) An ADI of 0-0.3 mg/kg bw was established at the 44th JECFA (1995)

SYNONYMS BHT; INS No. 321

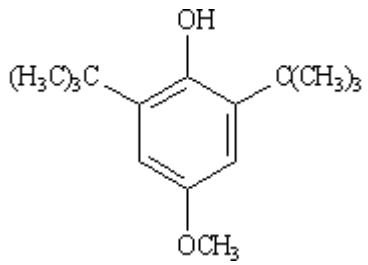
DEFINITION

Chemical names 2,6-Ditertiary-butyl-p-cresol, 4-methyl-2,6-ditertiary-butyl-phenol

C.A.S. number 128-37-0

Chemical formula $C_{15}H_{24}O$

Structural formula



Formula weight 220.36

Assay Not less than 99.0%

DESCRIPTION White, crystalline or flaked solid, odourless or having a characteristic faint aromatic odour

FUNCTIONAL USES Antioxidant

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Insoluble in water and propane-1,2-diol; freely soluble in ethanol

Melting range (Vol. 4) 69^{o} - 72^{o}

Spectrophotometry (Vol. 4) The absorption in the range 230 to 320 nm of a 2 cm layer of a 1 in 100,000 solution in dehydrated ethanol exhibits a maximum only at 278 nm

Colour reaction To 10 ml of a 1 in 100,000 solution of the sample in methanol add 10 ml of water, 2 ml of sodium nitrite solution (3 in 1000) and 5 ml of dianisidine dihydrochloride solution (200 mg of 3,3-dimethoxy-benzidine dihydrochloride dissolved in a mixture of 40 ml of methanol and 60 ml of 1 N hydrochloric acid). An orange red colour develops within 3 min. Add 5 ml of chloroform, and shake. The chloroform layer exhibits a purple or magenta colour that fades

when exposed to light.

PURITY

Solidification (Vol. 4) — Not lower than 69.2°

Sulfated ash (Vol. 4) — Not more than 0.005%
Test 20 g of the sample (Method I)

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.”

Phenolic impurities — Not more than 0.5%
See description under TESTS

TESTS

PURITY TESTS

Phenolic impurities — Determine by *Thin-Layer Chromatography*, (see Volume 4) using silica gel G plates.

Solution 1: Dissolve 0.25 g of the sample in 10 ml of ether.
Solution 2: Dilute 1 ml of Solution 1 to 10 ml with ether, and then dilute 1 ml of the resulting solution to 20 ml with ether. Use the final dilution as solution 2.

Procedure
Spot 2 µl each of Solution 1 and of Solution 2 on separate TLC plates. Place each plate in a developing chamber containing chloroform as solvent, and allow the solvent front to ascend to a point 15 cm above the sample spots. Develop the chromatograms by spraying with an aqueous mixture of equal volumes of 2% ferric chloride solution and 1% potassium ferricyanide solution mixed prior to use. The blue colours produced may be intensified by spraying with 2N hydrochloric acid. Any blue spots appearing (other than the major spot and the spot) are not more intense than the major spot appearing on Chromatogram 2.

METHOD OF ASSAY

Gas Chromatography Method (see Volume 4)

Internal standard solution (diphenylamine or 4-tertiary butylphenol): Accurately weigh 500 mg, dissolve in acetone and make up to 250 ml with acetone.
Standard solution: Accurately weigh 100 mg of butylated hydroxytoluene and dissolve in acetone to make 50 ml.

Procedure:
Dissolve 10 mg of the sample, accurately weighed, in the internal standard solution to make 50 ml. Inject aliquots of the solution into a gas chromatograph, using the following conditions:
Column
- length: 1.5 m

- inner diameter: 3 mm
- material: glass
- packing: 10% XE-60 on 100-200 mesh

Temperatures
- injector: 225°
- column: 155°
- detector: 250°

Carrier gas: nitrogen
Flow rate: 30 ml/min
Detector type: FID

Prepare a standard curve of butylated hydroxytoluene peak height/internal standard peak height versus concentration, using internal standard solutions having various concentrations of butylated hydroxytoluene. Determine the concentration of butylated hydroxytoluene in the sample from the standard curve.