BUTYLATED HYDROXYANISOLE

Prepared at the 33rd JECFA (1988), published in FNP 38 (1988) and in FNP 52 (1992). Metals and arsenic specifications revised at the 61st JECFA (2003) An ADI of 0-0.5 mg/kg bw was established at the 33rd JECFA (1988)

SYNONYMS BHA; INS No. 320

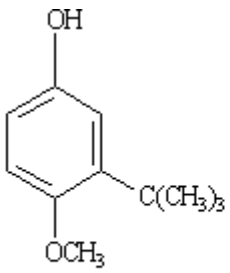
DEFINITION

Chemical names 3-Tertiary-butyl-4-hydroxyanisole, a mixture of 3- and 2-tertiary-butyl-4-hydroxyanisole

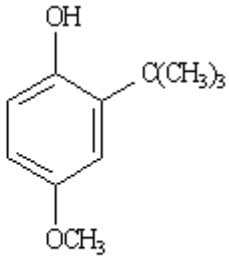
C.A.S. number 25013-16-5

Chemical formula $C_{11}H_{16}0_2$

Structural formula



3-isomer



2-isomer

Formula weight 180.25

Assay Not less than 98.5% of $C_{11}H_{16}0_2$ and not less than 85% of 3-tertiary-butyl-4-hydroxyanisole

DESCRIPTION White or slightly yellow crystals or waxy solid, with a faint characteristic odour

FUNCTIONAL USES Antioxidant

CHARACTERISTICS

IDENTIFICATION

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

Colour reaction To 5 ml of a 1 in 10,000 solution of the sample in 72% ethanol, add 2 ml of sodium borate TS and 1 ml of a 1 in 10,000 solution of 2,6-dichloroquinonechlorimide in absolute ethanol, and mix. A blue colour appears.

PURITY

Sulfated ash (Vol. 4) Not more than 0.05%
Test 5 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 at R_f 0.35) are not more intense than the major spot appearing on Chromatogram 2.

METHOD OF ASSAY

Gas Chromatographic Method

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

Standard solution:
Accurately weigh 90 mg of 3-butylated hydroxyanisole and 10 mg of 2-butylated hydroxyanisole and dissolve in Internal standard solution to make 100 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 equipped with a hydrogen flame ionization detector. Either of the following GC conditions or equivalent may be used:

A: The internal standard elutes after 3-tert-butyl-4-hydroxyanisole:
Column
- length: 1.5 m
- inner diameter: 2 mm

- material: glass
- packing: 10% XE-60 on 100-200 mesh
Temperatures
- injector: 250°
- column: 155°
- detector: 250°
Carrier gas: nitrogen
Flow rate: 30 ml/min

B: The internal standard elutes before 3-ter-butyl-4-hydroxyanisole:
Column
- length: 2 m
- inner diameter: 3 mm
- material: glass
- packing: 5% Versamide-900 on 80/100 mesh Chromosorb W-AW-DMCS
Temperatures
- injector: 225°
- column: 170°
- detector: 250°
Carrier Gas: nitrogen
Flow rate: 30 ml/min

Prepare a standard curve of 2- and 3-butylated hydroxyanisole peak height/internal standard peak height versus concentration, using internal standard solutions having various concentrations of butylated hydroxyanisole. Determine the concentrations of 2- and 3-butylated hydroxyanisole by reference to a standard curve. The sum of per cent 2-isomer and per cent 3-isomer gives per cent of total in the sample.

Alternative gas chromatographic method

Assay preparation:
Dissolve about 100 mg of the sample, accurately weighed, in Internal standard solution, and dilute with Internal standard solution to 10 ml.

Chromatographic system:
The gas chromatograph is equipped with a flame-ionization detector, and contains a 1.8-m x 2-mm stainless steel column packed with 10 percent liquid phase on the support, the column is maintained isothermally at a temperature between 175° and 185°, and helium is used as the carrier gas. Chromatograph a sufficient number of injections of the Standard preparation, and record the areas as directed under Procedure, to ensure that the relative standard deviation does not exceed 2.0% for the 3-tert-butyl-4- hydroxyanisole isomer and 6.0% for the 2-tert-butyl-4- hydroxyanisole isomer. The resolution between the isomers is not less than 1.3 and the tailing factor does not exceed 2.0.
Liquid phase: 25% 2-cyanoethyl : 75% methyl-polysiloxane.
Support:
Siliceous earth for chromatography has been fluxcalcined by mixing diatomite with Na_2CO_3 flux and calcining above 900°. The siliceous earth is acid-washed, then water-washed until neutral, but not base-washed. The siliceous earth is silanized by treating with an agent such as dimethyldichlorosilane to mask the surface silanol group.

Procedure:
Separately inject suitable portions (about 5 µl) of the Standard preparation and the Assay preparation into the gas chromatograph, and record the chromatograms. Measure the areas under the peaks for each isomer and the internal standard in each chromatogram, and calculate the quantity, in mg, of each isomer in the sample taken by the formula $10C_S(R_U/R_S)$, in which C_S is the concentration, in mg, of the isomer in the Standard solution, R_S is the ratio of the area of each isomer standard to that of the Internal standard in the chromatogram from the Standard preparation, and R_U is the ratio of the area of each isomer to that of the internal standard in the chromatogram from the Assay preparation.

Calculate the weight, in mg, of $C_{11}H_{16}0_2$ in the sample taken by adding the quantities of the two isomers.