ETHYL PROTOCATECHUATE

Prepared at the 17th JECFA (1973), published in FNP 4 (1978) and in FNP 52 (1992). Metals and arsenic specifications revised at the 61st JECFA (2003). No ADI was allocated at the 17th JECFA (1973)

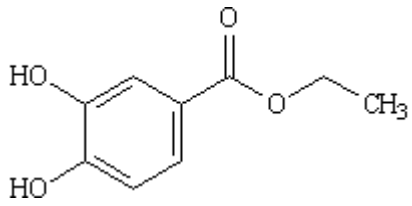
DEFINITION

Chemical names: Ethyl protocatechuate, Ethyl 3,4-dihydroxybenzoate, Ethyl ester of 3,4-dihydroxybenzoic acid

C.A.S. number: 3943-89-3

Chemical formula: $C_9H_{10}O_4$

Structural formula:



Formula weight: 182.2

Assay: Information requested

DESCRIPTION

White or pale brownish yellow, crystalline powder; odourless or has a faint phenol-like odour

FUNCTIONAL USES

Antioxidant

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4): Insoluble in water; soluble in ethanol

Melting range (Vol. 4): 132 - 135°

Test for ethanol: Dissolve 0.5 g of the sample in 10 ml of sodium hydroxide TS and distil. To 4 ml of the initial clear distillate add an equal volume of sodium hydroxide TS and 2 or 3 drops of iodine TS, and heat the solution: iodoform odour evolves.

Test for protocatechuic acid: Acidify the residue from the distillation in the Test for ethanol, to precipitate an acid which on recrystallization has a melting range of 190 - 194º with decomposition

Test for phenol: Dissolve 0.1 g of the sample in 5 ml of ethanol, and add 1 drop of diluted ferric chloride TS: a green colour develops

PURITY

Loss on drying (Vol. 4)	Not more than 0.5% (105^{o}, 2 h)
Sulfated ash (Vol. 4)	Not more than 0.05% Test 4 g of the sample
Chlorinated organic compounds	Not more than 100 mg/kg as chlorine Dissolve 1.07 g of the sample in 10 ml of 0.1 N sodium hydroxide. Acidify with nitric acid 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 be not more than that obtained by the addition of 0.5 ml of 0.1 N silver nitrate to a similar volume of dilute nitric acid TS and 0.3 ml of 0.01 N hydrochloric acid.
Free acid	Not more than 0.5%, calculated as protocatechuic acid (7.706 mg of protocatechuic acid is equivalent to 1 ml of 0.05 N NaOH) 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 0.005 N hydrochloric acid to match a buffer (pH 5.0) TS containing the same amount of indicator. Dissolve about 0.400 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 0.005 N hydrochloric acid found in the preliminary test to bring the solvent to pH 5.0. Titrate the solution back to pH 5.0 with 0.05 N sodium hydroxide, matching against the buffer pH 5.0 TS.
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	Information required