ETHYL p-HYDROXYBENZOATE


SYNONYMS
Ethylparaben, ethyl p-oxybenzoate, INS No. 214

DEFINITION

Chemical names
Ethyl p-hydroxybenzoate, ethyl ester of p-hydroxybenzoic acid

C.A.S. number
120-47-8

Chemical formula
C_9H_{10}O_3

Structural formula

\[
\begin{align*}
\text{O} & \quad \text{O} \\
\text{H} & \quad \text{CH}_3 \\
\end{align*}
\]

Formula weight
166.18

Assay
Not less than 99.0% on the dried basis

DESCRIPTION
Almost odourless, small, colourless crystals or a white, crystalline powder

FUNCTIONAL USES
Preservative

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)
Freely soluble in ethanol, ether and propylene glycol.

Melting range (Vol. 4)
115 - 118°

Test for p-hydroxybenzoate
Melting range of p-hydroxybenzoic acid derived from the sample is 212-217°
To 0.5 g of the sample add 10 ml of sodium hydroxide TS. Boil for 30 min and concentrate to about 5 ml. Cool, acidify with dilute sulfuric acid TS, collect the precipitate on a filter, and wash thoroughly with water. Dry in a desiccator over sulfuric acid. Determine the melting range of p-hydroxybenzoic acid so obtained.

PURITY

Loss on drying (Vol. 4)
Not more than 0.5% (80°, 2 h)
Sulfated ash (Vol. 4) Not more than 0.05%
Test 2 g of the sample (Method I)

Acidity
Heat 750 mg of the sample with 15 ml of water at 80° for 1 min, cool, and filter. The filtrate should be acid or neutral to litmus. To 10 ml of the filtrate add 0.2 ml of 0.1 N sodium hydroxide and 2 drops of methyl red TS. The solution should be yellow without even a light cast of pink.

p-Hydroxybenzoic acid and salicylic acid
Dissolve 0.5 g of the sample, accurately weighed, in 30 ml of ether, add 20 ml of a 1 in 100 sodium hydrogen carbonate solution, shake, and separate the water layer. Wash the water layer with two 20 ml portions of ether, add 5 ml of dilute sulfuric acid and 30 ml of ether, and shake. Separate the ether layer, and shake with about 10 ml of water. Filter the ether layer, and wash the vessel and the filter with a small amount of ether. Combine the washings and the filtrate, evaporate ether on a water bath, and dry the residue over sulfuric acid to constant weight. The weight of the residue should not exceed 5 mg. Dissolve any residue in 25 ml of water, heat to about 70°, filter, and add a few drops of dilute ferric chloride TS. No violet to reddish violet colour should be produced.

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, to the nearest mg, 2 g of the dried sample and transfer into a flask. Add 40 ml of N sodium hydroxide and rinse the sides of the flask with water. Cover with a watch glass, boil gently for 1 h and cool. Add 5 drops of bromothymol blue TS and titrate the excess sodium hydroxide with N sulfuric acid, containing the same proportion of indicator. Perform a blank determination with the reagents and make any necessary correction. Each ml of N sodium hydroxide is equivalent to 166.18 mg of C₉H₁₀O₃.