4-HEXYLRESORCINOL

Prepared at the 51st JECFA (1998), published in FNP 52 Add 6 superseding specifications prepared at the 44th JECFA, published in FNP 52 Add 3 (1995). ADI "treatment of crustacea at concentrations of up to 50 mg/l, resulting in residue levels of approximately 1 mg/kg in edible portion, is not of toxicological concern", established at the 44th JECFA in 1995.

SYNONYMS 4-Hexyl-1,3-benzenediol, Hexylresorcinol

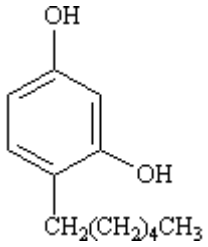
DEFINITION

Chemical names 4-Hexylresorcinol

C.A.S. number 136-77-6

Chemical formula $C_{12}H_{18}O_2$

Structural formula



Formula weight 197.24

Assay Not less than 98.0% on the dried basis

DESCRIPTION White powder

FUNCTIONAL USES Antioxidant, colour retention agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Freely soluble in ether and acetone; very slightly soluble in water

Nitric acid test To 1 ml of a saturated solution of the sample, add 1 ml of nitric acid. A light red colour appears.

Bromine test To 1 ml of a saturated solution of the sample, add 1 ml of bromine TS. A yellow, flocculent precipitate is formed. Add 2 ml of ammonia TS, and the precipitate dissolves producing a yellow solution.

Infrared absorption The infrared spectrum of the sample contained in a sodium chloride cell or between salt plates corresponds to the infrared spectrum below.

PURITY

Melting range (Vol. 4) 62 - 67º

Acidity
Not more than 0.05%
Dissolve 250 mg of the sample in water, add methyl red TS, and titrate with 0.02 N sodium hydroxide. No more than 1.0 ml is required for neutralization.

Sulfated ash (Vol. 4) Not more than 0.1%

Resorcinol and other phenols
Shake about 1 g of the sample with 50 ml of water for a few min, filter, and to the filtrate add 3 drops of ferric chloride TS. No red or blue colour is produced.

Nickel (Vol. 4)
Not more than 2 mg/kg
Dissolve 2 g of the sample in sufficient methanol to produce 20 ml and continue as directed in the Limit Test, beginning with "add 3 ml of bromine TS ...".

Mercury (Vol. 4) Not more than 3 mg/kg

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

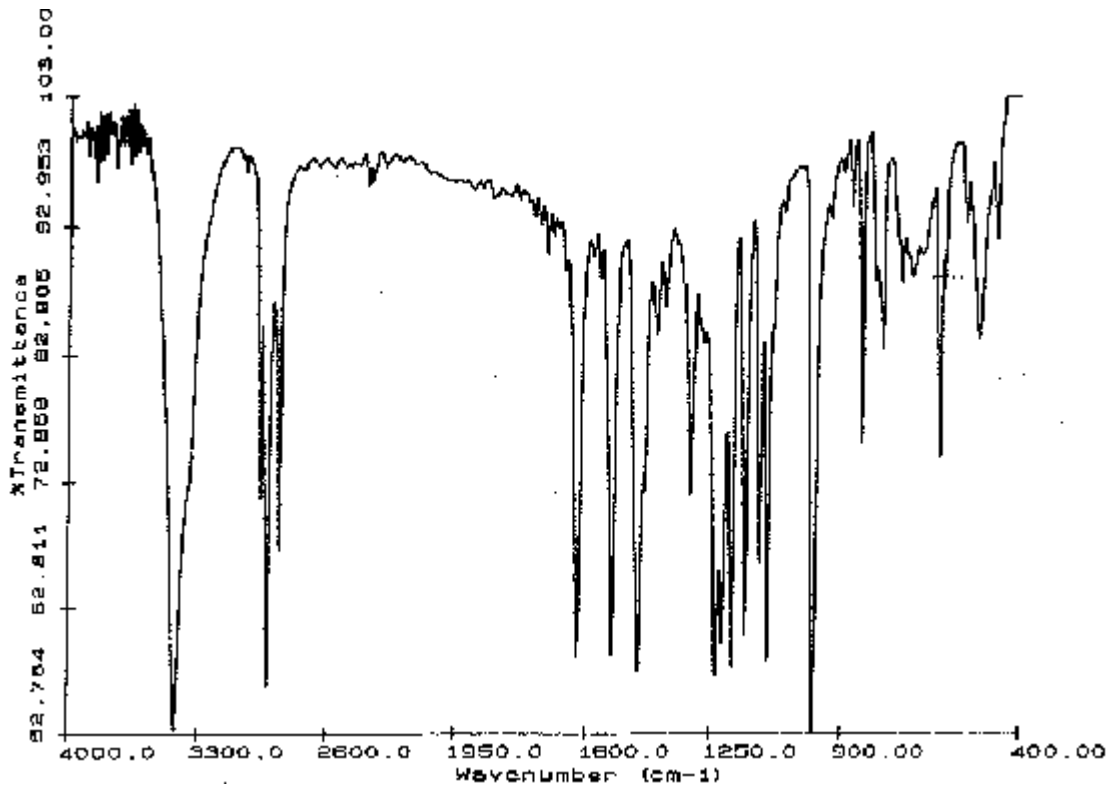
Indicator solution
Mix 1 g of soluble starch with 10 mg of mercuric iodide and sufficient cold water to make a thin paste. Add 200 ml of boiling water, and boil for 1 min with continuous stirring. Cool and use only the clear solution.

Procedure
Accurately weigh into a 250-ml iodine flask 70-100 mg of the sample, previously dried for 4 hours at room temperature over silicagel, and dissolve the sample in 10 ml of methanol. Add 30.0 ml of bromide/bromate TS, then quickly 5 ml of hydrochloric acid and insert the stopper in the flask immediately. Cool the flask under running water to room temperature, shake vigorously for 5 min, then set aside for 5 min. Add 6 ml of potassium iodide TS around the stopper, cautiously loosen the stopper, again insert the stopper tightly, and swirl gently. Add 1 ml of chloroform, and titrate the liberated iodine with 0.1N sodium thiosulfate, adding 3 ml of Indicator solution as the end point is approached. Perform a blank determination. Content of $C_{12}H_{18}O_2$ is

$$\frac{(B - S) \times 4.857 \times 100}{W} \%$$

where
B = ml of 0.1N sodium thiosulfate used for the blank
S = ml of 0.1N sodium thiosulfate used for the sample
W = mg of the weighed sample

Infrared spectrum 4-Hexylresorcinol


%Transmittance
103.00
92.953
82.905
72.858
62.811
52.764
4000.0
3300.0
2600.0
1950.0
1600.0
1250.0
900.00
400.00
Wavenumber (cm-1)