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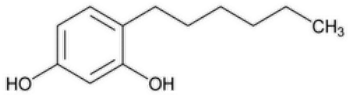
Hexylresorcinol



$C_{12}H_{18}O_2$ 194.27

1,3-Benzenediol, 4-hexyl-;

4-Hexylresorcinol CAS RN®: 136-77-6; UNII: R9QTB5E82N.

DEFINITION

Hexylresorcinol, dried over silica gel for 4 h, contains NLT 98.0% and NMT 100.5% of hexylresorcinol ($C_{12}H_{18}O_2$).

[**Caution—**Hexylresorcinol is irritating to the oral mucosa and respiratory tract and to the skin, and its solution in alcohol has vesicant properties.]

IDENTIFICATION

- **A.**

  Sample solution: 1 mL of a saturated solution

  Analysis: To the *Sample solution*, add 1 mL of nitric acid.

  Acceptance criteria: A light red color appears.

- **B.**

  Sample solution: 1 mL of a saturated solution

  Analysis: To the *Sample solution*, add 1 mL of bromine TS.

  Acceptance criteria: A yellow, flocculent precipitate is formed. Add 2 mL of 6 N ammonium hydroxide. The precipitate dissolves, producing a yellow solution.

ASSAY

- **Procedure**

  Sample solution: Dissolve 70–100 mg of Hexylresorcinol, previously dried over silica gel for 4 h and weighed, in 10 mL of methanol in a 250-mL iodine flask. Add 30.0 mL of 0.1 N bromine VS, then add 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.

  Titrimetric system

  (See *Titrimetry* ⟨541⟩.)

  Mode: Residual titration

  Titrant: 0.1 N bromine VS

  Back-titrant: 0.1 N sodium thiosulfate VS

  Endpoint detection: Visual

  Analysis: Titrate the liberated iodine with *Back-titrant*, adding 3 mL of starch TS as the endpoint is approached. Perform a blank determination.

  Calculate the percentage of hexylresorcinol ($C_{12}H_{18}O_2$) in the sample taken. Each milliliter of *Titrant* is equivalent to 4.857 mg of hexylresorcinol ($C_{12}H_{18}O_2$).

  Acceptance criteria: 98.0%–100.5%, dried over silica gel for 4 h

IMPURITIES

Delete the following:

▲• **Mercury**

Select all reagents for this test to have as low a content of mercury as practicable, and store all reagent solutions in containers of borosilicate glass. Glassware used in this test shall be specially cleaned by being soaked in warm 8 N nitric acid for 30 min and rinsed with water. Keep flasks for this determination separate from other flasks, and use only for mercury determinations.

Standard solution: Transfer 34.0 mg of mercuric chloride to a 250-mL volumetric flask. Add 1 drop of hydrochloric acid, add water to dissolve, and dilute with water to volume. Transfer 1.0 mL of this solution to a 100-mL volumetric flask, add 1 drop of hydrochloric acid, and dilute with water to volume. Transfer 1.0 mL of this solution to a 500-mL volumetric flask, add 1 drop of hydrochloric acid, and dilute with water to volume.

Sample solution: Transfer 134 mg to a 250-mL beaker, and cautiously add 10 mL of 11 N nitric acid and 10 mL of 18 N sulfuric acid. Digest, with the aid of heat, in a well-ventilated hood until the evolution of brown fumes ceases. Cautiously add an additional 10 mL of 11 N nitric acid, and continue heating until no more fumes are evolved. Cool, transfer to a 200-mL volumetric flask, and dilute with water to volume.

Instrumental conditions

Mode: Atomic absorption spectrophotometry

Analytical wavelength: 253.65 nm

Lamp: Mercury hollow-cathode (and an absorption cell that permits the flameless detection of mercury)

Analysis: Transfer 100 mL of *Standard solution* to a 300-mL mercury analysis reaction vessel, and add 2 drops of potassium permanganate solution (1 in 20) (the solution should be purple; add additional permanganate solution dropwise, if necessary). Add 5 mL of 11 N nitric acid, stir, and allow to stand for NLT 15 s. Add 5 mL of 18 N sulfuric acid, stir, and allow to stand for NLT 45 s. Add 5 mL of hydroxylamine hydrochloride solution (3 in 200), stir, and allow to stand until the solution turns light yellow or colorless. Add 5 mL of stannous chloride solution (1 in 10). Disregard the presence of insoluble matter in this solution. Mix before use. Immediately insert the aerator connected to the air pump. Connect in a closed system, with a circulating air pump, a calcium chloride drying tube and an aerator inserted in a 300-mL reaction vessel so that air passed through the treated solution contained in the reaction vessel evaporates any metallic mercury present. In a similar manner, treat 100 mL of the *Sample solution* and 100 mL of water (reagent blank), and determine the maximum absorbances at the same wavelength.

Check the zero setting of the instrument frequently.

Acceptance criteria: 3 ppm; the absorbance of the solution from the *Sample solution* does not exceed that of the solution from the *Standard solution*.▲ (USP 1-May-2022)

• **Residue on Ignition ⟨281⟩:** NMT 0.1%

• **Resorcinol and Other Phenols**

Sample: 1 g

Analysis: Shake the *Sample* with 50 mL of water for a few min, filter, and to the filtrate add 3 drops of ferric chloride TS.

Acceptance criteria: No red or blue color is produced.

SPECIFIC TESTS

• **Acidity**

Sample: 250 mg

Analysis: Dissolve the *Sample* in 500 mL of water, add methyl red TS, and titrate with 0.020 N sodium hydroxide.

Acceptance criteria: NMT 1.0 mL is required for neutralization.

• **Melting Range or Temperature ⟨741⟩, *Class I*:** 62°–67°

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight, light-resistant containers.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
HEXYLRESORCINOL	Documentary Standards Support	SM12020 Small Molecules 1

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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