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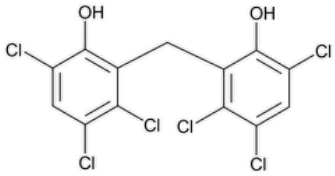
Hexachlorophene



$C_{13}H_6Cl_6O_2$ 406.90

Phenol, 2,2'-methylenebis[3,4,6-trichloro]-;

2,2'-Methylenebis[3,4,6-trichlorophenol] CAS RN®: 70-30-4; UNII: IWW5FV6NK2.

DEFINITION

Hexachlorophene contains NLT 98.0% and NMT 100.5% of hexachlorophene ($C_{13}H_6Cl_6O_2$), calculated on the dried basis.

IDENTIFICATION

Change to read:

• **A.** ▲Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: **197K**▲ (CN 1-May-2020)

• **B.**

Sample solution: 5 mg of Hexachlorophene in 5 mL of alcohol

Analysis: To the *Sample solution* add 1 drop of ferric chloride TS.

Acceptance criteria: A transient purple color is produced immediately.

ASSAY

• **Procedure**

Sample: 1.5 g of Hexachlorophene

Analysis: Dissolve the *Sample* in 25 mL of alcohol, and titrate with 0.1 N sodium hydroxide VS, determining the endpoint potentiometrically. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N sodium hydroxide is equivalent to 40.69 mg of hexachlorophene ($C_{13}H_6Cl_6O_2$).

Acceptance criteria: 98.0%–100.5% on the dried basis

IMPURITIES

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

• **Limit of 2,3,7,8-Tetrachlorodibenzo-*p*-dioxin**

[**Caution**—Because 2,3,7,8-tetrachlorodibenzo-*p*-dioxin is an extremely toxic substance, exercise all necessary precautions in the conduct of this procedure.]

Standard solution: 0.01 µg/mL of 2,3,7,8- tetrachlorodibenzo-*p*-dioxin[1]

Sample solution: Dissolve 10.0 g of Hexachlorophene in 50 mL of methanol, transfer to a 1-L separator with the aid of 25 mL of methanol, add 25 mL of 2.5 N lithium hydroxide and 225 mL of water, and extract with two 200-mL portions of freshly distilled *n*-hexane. Dry the combined *n*-hexane extracts over anhydrous sodium sulfate, filter, and evaporate on a rotary evaporator at a bath temperature not exceeding 40° to a volume of 15 mL. Transfer this solution in portions to a 12-mL centrifuge tube, concentrating each time in a gentle stream of nitrogen in a warm water bath to a volume of 1 mL. Rinse the flask with 15 mL of *n*-hexane, and evaporate similarly. Wash down the walls of the tube with 10 mL of *n*-hexane, and again evaporate to a volume of 1.0 mL.

Cool, and transfer to a microcolumn that has been prepared in the following manner. Place a small plug of glass wool in a 5-mm × 15-mm pipet, add a small quantity of sand and 1.0 g of basic alumina, tap several times to pack down the alumina, and heat in a vacuum oven at 110° for 3 h. Store under vacuum.

Elute the column with 10 mL of a mixture of *n*-hexane and methylene chloride (9:1), using a portion to rinse the tube. Collect the eluate in a 12-mL graduated centrifuge tube, and concentrate in a gentle stream of nitrogen in a warm water bath to a volume of 1.0 mL.

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: GC-MS (see *Mass Spectrometry ⟨736⟩*)

Detector: Multiple-ion

Column: 2-mm × 1-m glass; liquid phase G1 on support S1

Temperatures

Column: 250°

Injection port: 300°

Carrier gas: Helium

Flow rate: 40 mL/min

Injection volume: 2.0 µL

Analysis

Samples: *Standard solution* and *Sample solution*

Acceptance criteria: NMT 1 ppb; the sum of the peak heights at mass values of 320, 322, and 324 of the *Sample solution* is not greater than the sum of the peak heights at the same mass values of the *Standard solution*.

SPECIFIC TESTS

- **Melting Range or Temperature ⟨741⟩:** 161°–167°
- **Loss on Drying ⟨731⟩**

Analysis: Dry a sample at 105° for 4 h.

Acceptance criteria: NMT 1.0%

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight, light-resistant containers.
- **USP Reference Standards ⟨11⟩**

USP Hexachlorophene RS

[1] A solution in anisole is available commercially from KOR Isotopes, Div. of ECO, Inc., 56 Rogers St., Cambridge, MA 02142. This solution may be diluted with a mixture of *n*-hexane and methylene chloride (9:1) to the required concentration.

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

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

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. Information currently unavailable

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