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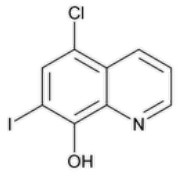
Clioquinol



C_9H_5ClINO 305.50

8-Quinolinol, 5-chloro-7-iodo-;

5-Chloro-7-iodo-8-quinolinol CAS RN®: 130-26-7; UNII: 7BHQ856EJ5.

DEFINITION

Clioquinol, dried over phosphorus pentoxide for 5 h, contains NLT 93.0% and NMT 100.5% of clioquinol (C_9H_5ClINO).

IDENTIFICATION

• **A.**

Standard solution: Prepare as directed for the *Standard solution* in the *Assay*, except use 1.0 mL of pyridine instead of the *Internal standard solution*.

Acceptance criteria: The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

Change to read:

• **B.** ▲**Spectroscopic Identification Tests ⟨197⟩, *Ultraviolet-Visible Spectroscopy*: 197U**▲ (CN 1-May-2020)

Analytical wavelength: 267 nm

Medium: 3 N hydrochloric acid

Sample solution: 5 µg/mL

Acceptance criteria: Absorptivities, calculated on the dried basis, do not differ by more than 3.0%.

• **C.**

Sample: 100 mg

Analysis: Heat the *Sample* with 5 mL of sulfuric acid.

Acceptance criteria: Copious violet vapors of iodine are evolved.

ASSAY

• **Procedure**

Internal standard solution: 2 mg/mL of pyrene in pyridine

Standard stock solution: 3 mg/mL of USP Clioquinol RS in a mixture of pyridine and *n*-hexane (4:1)

Standard solution: Transfer 1.0 mL of the *Standard stock solution* to a screw-capped glass vial fitted with a septum, add 1.0 mL of bis(trimethylsilyl)acetamide and 1.0 mL of *Internal standard solution*, attach the cap, and mix. Heat in a water bath at 50° for 15 min, and then cool to ambient temperature.

Sample stock solution: 3 mg/mL of Clioquinol, previously dried, in a mixture of pyridine and *n*-hexane (4:1)

Sample solution: Transfer 1.0 mL of the *Sample stock solution* to a screw-capped glass vial fitted with a septum, add 1.0 mL of bis(trimethylsilyl)acetamide and 1.0 mL of *Internal standard solution*, and attach the cap. Heat in a water bath at 50° for 15 min, and then cool to ambient temperature.

Chromatographic system

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

Mode: GC

Detector: Flame ionization

Column: 1.83-m × 2-mm glass; packed with 3% liquid phase G3 on 80- to 100-mesh support S1AB

Temperatures

Column: The initial temperature is 200° for a conditioning period of NLT 16 h (not connected to the detector) and is then reduced to 165°.

Injection port: 170°

Detector: 250°

Carrier gas: Helium

Flow rate: 30 mL/min for helium. Hydrogen and air are introduced into the detector at rates of 25 and 500 mL/min, respectively.

Injection volume: 1 µL

System suitability

Sample: *Standard solution*

[NOTE—The relative retention times for clioquinol and pyrene are 0.6 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 3 between the clioquinol and the internal standard peaks

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of clioquinol (C_9H_5ClINO) in the portion taken:

$$\text{Result} = (R_U/R_S) \times (C_S/C_U) \times 100$$

R_U = peak response ratio of clioquinol to the internal standard from the *Sample solution*

R_S = peak response ratio of clioquinol to the internal standard from the *Standard solution*

C_S = concentration of USP Clioquinol RS in the *Standard solution* (mg/mL)

C_U = concentration of Clioquinol in the *Sample solution* (mg/mL)

Acceptance criteria: 93.0%–100.5%

IMPURITIES

• **RESIDUE ON IGNITION ⟨281⟩:** NMT 0.5%

SPECIFIC TESTS

• **LOSS ON DRYING ⟨731⟩**

Analysis: Dry a sample over phosphorus pentoxide for 5 h.

Acceptance criteria: NMT 0.5%

• **FREE IODINE AND IODIDE**

Control solution: Dilute 2.0 mL of potassium iodide solution (1 in 6000) with water to 10 mL, add 6 mL of 2 N sulfuric acid, 1 mL of potassium dichromate TS, and 2 mL of chloroform, and shake for 15 s (0.05% of iodide).

Sample: 1.0 g

Analysis:

Shake the *Sample* with 20 mL of water for 30 s, allow to stand for 5 min, and filter. To 10 mL of the filtrate add 1 mL of 2 N sulfuric acid, then add 2 mL of chloroform, and shake: no violet color appears in the chloroform (free iodine). To the mixture add 5 mL of 2 N sulfuric acid and 1 mL of potassium dichromate TS, and shake for 15 s.

Acceptance criteria: The color of the chloroform layer is no deeper than that produced in the *Control solution*.

ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in tight, light-resistant containers.

• **USP REFERENCE STANDARDS ⟨11⟩**

USP Clioquinol RS

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

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

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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