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Clioquinol Ointment

DEFINITION

Clioquinol Ointment contains NLT 90.0% and NMT 110.0% of the labeled amount of clioquinol (C_9H_5ClINO) in a suitable ointment base.

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*, as obtained in the Assay, corresponds to that of the *Standard solution*.

• B.

Sample solution: Place nominally 25 mg of clioquinol from Ointment in a 100-mL volumetric flask, add 75 mL of dilute hydrochloric acid (1 in 4), and heat on a steam bath to melt the Ointment, shaking vigorously to extract the clioquinol. Cool under running water, and add dilute hydrochloric acid (1 in 4) to volume. Filter through paper, and dilute 3 mL of the filtrate with dilute hydrochloric acid (1 in 4) to 100 mL.

Acceptance criteria: The UV absorption spectrum of the *Sample solution* exhibits maxima and minima at the same wavelengths as that of a similar solution of [USP Clioquinol RS](#), concomitantly measured.

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*, and attach the cap. Heat in a water bath at 50° for 15 min, and then cool to ambient temperature.

Sample solution: Transfer nominally 150 mg of clioquinol from Ointment to a 125-mL separator. Add 75 mL of *n*-hexane, then add 15 mL of dimethylformamide, and mix for 1 min. Allow the layers to separate, and transfer the lower layer to a 50-mL volumetric flask. Repeat the extraction with separate 15- and 10-mL portions of dimethylformamide, and transfer the lower layers to the 50-mL volumetric flask. Dilute with dimethylformamide to volume. Transfer 1.0 mL of this solution to a screw-capped glass vial fitted with a septum, and evaporate at 60° under a stream of nitrogen to dryness. Add 1.0 mL of a mixture of pyridine and *n*-hexane (4:1) to the residue, 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, 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 the labeled amount of clioquinol (C_9H_5ClINO) in the portion of Ointment 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 = nominal concentration of clioquinol in the *Sample solution* (mg/mL)**Acceptance criteria:** 90.0%–110.0%**ADDITIONAL REQUIREMENTS**

- **PACKAGING AND STORAGE:** Preserve in collapsible tubes or 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 OINTMENT	Documentary Standards Support	SM12020 Small Molecules 1

Chromatographic Database Information: [Chromatographic Database](#)**Most Recently Appeared In:**

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