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Clioquinol Cream

DEFINITION
Clioquinol Cream contains NLT 90.0% and NMT 110.0% of the labeled amount of clioquinol (C_9H_5ClINO) in a suitable cream 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 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 Cream, 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*, attach the cap, and mix. Heat in a water bath at 50° for 15 min, and then cool to ambient temperature.

Sample stock solution: Transfer nominally 150 mg of clioquinol from Cream to a 60-mL separator. Place the separator on its side in a vacuum oven at a pressure of 10 mm of mercury at 45° for 4 h. Remove the separator from the oven, allow to cool, add 15 mL of a mixture of pyridine and *n*-hexane (4:1), and insert a polytef stopper. Transfer the mixture to a 50-mL volumetric flask, and rinse the separator with two 15-mL portions of the same solvent, shaking each time for 30 s. Transfer both rinsings to the volumetric flask, and dilute with the same solvent to volume.

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 the labeled amount of clioquinol (C_9H_5ClINO) in the portion of Cream 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 CREAM	Documentary Standards Support	SM12020 Small Molecules 1

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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