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Ciclopirox Topical Solution

DEFINITION
Ciclopirox Topical Solution contains NLT 90.0% and NMT 110.0% of the labeled amount of ciclopirox ($C_{12}H_{17}NO_2$).

IDENTIFICATION
• **A.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY
• **Procedure**
[Note—Protect the *Standard solution* and *Sample solution* from light.]

Buffer: Transfer 5.25 g of citric acid and 25 mL of 0.1 M edetate disodium to a 1-L volumetric flask, and dilute with water to volume. Adjust with 8.5% diluted sodium hydroxide solution to a pH of 6.5.

Mobile phase: Acetonitrile and *Buffer* (35:65)

Standard solution: 0.2 mg/mL of USP Ciclopirox RS and 1 µg/mL each of USP Ciclopirox Related Compound B RS and USP Ciclopirox Related Compound C RS in methanol

Sample solution: Equivalent to 0.2 mg/mL of ciclopirox in methanol from Topical Solution. Pass through a filter of 0.45-µm pore size, and use the filtrate.

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

Mode: LC

Detector: UV 303 nm

Column: 4-mm × 12.5-cm; 5-µm packing L1

Column temperature: 30 ± 5°

Flow rate: 0.9 mL/min

Run time: 5 times the retention time of the major peak

Injection size: 20 µL

System suitability

Sample: *Standard solution*

[Note—For information only, see *Table 1* for relative retention times of impurities.]

Suitability requirements

Resolution: NLT 3.0 between ciclopirox and ciclopirox related compound B; and NLT 3.0 between ciclopirox related compound C and ciclopirox

Tailing factor: NMT 2.0 for the ciclopirox peak

Relative standard deviation: NMT 2.0% for the ciclopirox peak

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of ciclopirox ($C_{12}H_{17}NO_2$) in the portion of Topical Solution taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

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

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

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• **Minimum Fill ⟨755⟩:** Meets the requirement

IMPURITIES

• **Organic Impurities**

Buffer, Mobile phase, Standard solution, Sample solution, Chromatographic system, and **System suitability:** Proceed as directed in the *Assay*.

Analysis

Sample: *Sample solution*

Calculate the percentage of each impurity in the portion of Topical Solution taken:

$$\text{Result} = (r_U/r_T) \times (1/F) \times 100$$

r_U = peak response of each individual impurity from the *Sample solution*

r_T = sum of responses of all the peaks in the *Sample solution*

F = relative response factor (see *Table 1*)

Acceptance criteria: See *Table 1*.

Table 1

Compound	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
Ciclopirox related compound C	0.54	1.3	0.5
Ciclopirox	1.0	—	—
Ciclopirox related compound B[a]	1.87	—	—
Any unspecified individual impurity	—	1.0	0.2
Total impurities	—	—	1.2

[a] Process impurity already monitored in the drug substance.

SPECIFIC TESTS

• **Microbial Enumeration Tests ⟨61⟩** and **Tests For Specified Microorganisms ⟨62⟩:** The total aerobic microbial count does not exceed 10^2 cfu/g, and the total combined molds and yeasts count does not exceed 10^1 cfu/g.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers, protected from light. Store at controlled room temperature.

• **USP Reference Standards ⟨11⟩**

USP Ciclopirox RS

USP Ciclopirox Related Compound B RS

6-Cyclohexyl-4-methyl-2-pyrone.

$C_{12}H_{16}O_2$ 192.25

USP Ciclopirox Related Compound C RS

6-Cyclohexyl-4-methylpyridin-2(*1H*)-one.

$C_{12}H_{17}NO$ 191.27

Topic/Question	Contact	Expert Committee
CICLOPIROX TOPICAL SOLUTION	Documentary Standards Support	SM12020 Small Molecules 1

Chromatographic Database Information: Chromatographic Database

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