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Compound Undecylenic Acid Ointment

Change to read:

DEFINITION

Compound Undecylenic Acid Ointment contains, either individually or in any combination, undecylenic acid, calcium undecylenate, copper undecylenate, and zinc undecylenate in a suitable ointment base. It contains NLT 90.0% and NMT 110.0% ▲▲ (USP 1-Aug-2023) of total undecylenic acid ($C_{11}H_{20}O_2$), ▲which contains NLT 90.0% and NMT 110.0% of the labeled amount of zinc undecylenate ($C_{22}H_{38}O_4Zn$) and free undecylenic acid ($C_{11}H_{20}O_2$). Or it contains NLT 90.0% and NMT 110.0% of the labeled amount of calcium undecylenate ($C_{22}H_{38}O_4Ca$) or copper undecylenate ($C_{22}H_{38}O_4Cu$), if present.▲ (USP 1-Aug-2023)

Add the following:

▲IDENTIFICATION

• **A.** The retention time of the undecylenic acid peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay* for *Total Undecylenic Acid*.

• **B.** The absorbance of the *Sample solution* is similar to that of the *Standard solutions*, as obtained in the *Assay* for *Zinc Undecylenate*.▲ (USP 1-Aug-2023)

ASSAY

Change to read:

• **▲Total▲ (USP 1-Aug-2023) Undecylenic Acid**

▲Internal standard solution: 0.5 mg/mL of tridecanoic acid in *n*-heptane

Standard solution: 0.5 mg/mL of USP Undecylenic Acid RS in the *Internal standard solution* prepared as follows. Transfer 25 mg of USP Undecylenic Acid RS to a 100-mL volumetric flask, add 0.15 N hydrochloric acid to 20% of the flask volume, and heat until the sample is liquified. Cool and add a volume of the *Internal standard solution*, equivalent to 50% of the flask volume. Dilute with 0.15 N hydrochloric acid to volume and mix well. Transfer the top *n*-heptane layer to a suitable container, and dry over sodium sulfate, anhydrous. Centrifuge to clarify the mixture, and use the clear supernatant.

Sample solution: Nominally 0.5 mg/mL of undecylenic acid in the *Internal standard solution* prepared as follows. Transfer about 118 mg of Ointment, equivalent to 25 mg of undecylenic acid, to a 100-mL volumetric flask; add 0.15 N hydrochloric acid to 20% of the flask volume, and heat until the sample is liquified. Cool and add a volume of the *Internal standard solution*, equivalent to 50% of the flask volume. Dilute with 0.15 N hydrochloric acid to volume. Transfer the top *n*-heptane layer to a suitable container, and dry over sodium sulfate, anhydrous. Centrifuge to clarify the mixture, and use the clear supernatant.

[Note—Retain the bottom aqueous layer to use as the *Sample stock solution* in the *Assay* for *Zinc Undecylenate*.]

Chromatographic system

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

Mode: GC

Detector: Flame ionization

Column: 0.25-mm × 30-m fused-silica capillary; coated with a 0.25-µm film of phase G35

Temperatures

Injection port: 280°

Detector: 350°

Column: See *Table 1*.

Table 1

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
100	—	100	5
100	10	220	13
[NOTE—The relative retention times for undecylenic acid and tridecanoic acid are 1.0 and 1.1, respectively.]			15

Carrier gas: Helium
Flow rate: 0.7 mL/min
Injection volume: 1 µL
Injection type: Split, split ratio 25:1
System suitability
Sample: *Standard solution*
Suitability requirements
Resolution: NLT 10 between undecylenic acid and tridecanoic acid
Tailing factor: NMT 2.0 for undecylenic acid
Relative standard deviation: NMT 1.0% for the peak response ratio of undecylenic acid to tridecanoic acid

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the concentration (*C*) of total undecylenic acid in the *Sample solution*:

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

R_U = peak response ratio of undecylenic acid to tridecanoic acid from the *Sample solution*

R_S = peak response ratio of undecylenic acid to tridecanoic acid from the *Standard solution*

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

Calculate the percentage of total undecylenic acid ($C_{11}H_{20}O_2$) in the Ointment taken:

$$\text{Result} = \{(C \times V)/[(W \times L_Z \times F) + (W \times L_U)]\} \times 100$$

C = concentration of total undecylenic acid in the *Sample solution*

V = volume of the *Sample solution*, 50 mL

W = weight of Ointment in the *Sample solution* (mg)

L_Z = labeled percentage of zinc undecylenate

F = conversion factor, 0.8533

L_U = labeled percentage of free undecylenic acid

[NOTE—Where the definition list references a percentage, use a decimal or %/100.]

Calculate the conversion factor:

$$\text{Result} = (2 \times M_{r1})/M_{r2}$$

M_{r1} = molecular weight of undecylenic acid, 184.28

M_{r2} = molecular weight of zinc undecylenate, 431.92

Acceptance criteria: 90.0%–110.0% ▲(USP 1-Aug-2023)

Change to read:

• **ZINC UNDECYLENATE**

▲**Standard stock solution:** 50 µg/mL of zinc[1] in 0.15 N hydrochloric acid

Standard solution A: 1 µg/mL of zinc from the *Standard stock solution* in 0.15 N hydrochloric acid

Standard solution B: 1.5 µg/mL of zinc from the *Standard stock solution* in 0.15 N hydrochloric acid

Standard solution C: 2 µg/mL of zinc from the *Standard stock solution* in 0.15 N hydrochloric acid

Standard solution D: 2.5 µg/mL of zinc from the *Standard stock solution* in 0.15 N hydrochloric acid

Standard solution E: 3 µg/mL of zinc from the *Standard stock solution* in 0.15 N hydrochloric acid

Sample stock solution: Nominally 0.45 mg/mL of zinc undecylenate, equivalent to 0.07 mg/mL of zinc, in 0.15 N hydrochloric acid. Use the bottom aqueous layer from the *Sample solution* in the *Assay* for *Total Undecylenic Acid*.

Sample solution: Nominally 14 µg/mL of zinc undecylenate, equivalent to 2 µg/mL of zinc, from the *Sample stock solution* in 0.15 N hydrochloric acid

Instrumental conditions

(See *Atomic Absorption Spectroscopy ⟨852⟩*.)

Mode: Atomic absorption spectrophotometry

Analytical wavelength: Zinc emission line at 213.9 nm

Lamp: Zinc hollow-cathode

Flame: Air–acetylene

System suitability

Samples: *Standard solution A*, *Standard solution B*, *Standard solution C*, *Standard solution D*, and *Standard solution E*

Suitability requirements

Correlation coefficient: NLT 0.995, determined from the regression line constructed in the *Analysis*

Analysis

Samples: *Standard solution A*, *Standard solution B*, *Standard solution C*, *Standard solution D*, *Standard solution E*, and *Sample solution*

Concomitantly determine the responses for *Standard solutions A–E* and construct a linear calibration curve by plotting the absorbance values of *Standard solutions A–E* versus their corresponding concentrations, in µg/mL. The maxima absorbance should not exceed 0.2 absorbance units. Rotation of the burner may be used to reduce sensitivity, if needed. Use the calibration curve to determine the concentration (*C*), in µg/mL, for zinc in the *Sample solution*.

Calculate the percentage of the labeled amount of zinc undecylenate ($C_{22}H_{38}O_4Zn$) in the Ointment taken:

$$\text{Result} = (C_{U1}/C_{U2}) \times (M_r/A_r) \times 100$$

C_{U1} = concentration of zinc in the *Sample solution* (µg/mL)

C_{U2} = nominal concentration of zinc undecylenate in the *Sample solution* (µg/mL)

M_r = molecular weight for zinc undecylenate, 431.92

A_r = atomic weight for zinc, 65.39

Acceptance criteria: 90.0%–110.0%▲ (USP 1-Aug-2023)

Add the following:

▲• **Free Undecylenic Acid**

Analysis: Calculate the percentage of the labeled amount of free undecylenic acid ($C_{11}H_{20}O_2$) in the Ointment:

$$\text{Result} = [(\{P_T \times [(L_Z \times F) + L_U]\} - (P_Z \times L_Z \times F))/L_U] \times 100$$

P_T = percentage of total undecylenic acid, as determined in the test for *Total Undecylenic Acid*

L_Z = labeled percentage of zinc undecylenate

F = conversion factor, 0.8533

L_U = labeled percentage of free undecylenic acid

P_Z = percentage of zinc undecylenate, as determined in the test for *Zinc Undecylenate*

[Note—Where the definition list references a percentage, use a decimal or %/100.]

Acceptance criteria: 90.0%–110.0%▲ (USP 1-Aug-2023)

ADDITIONAL REQUIREMENTS

Change to read:

• **Packaging and Storage:** Preserve in tight containers, and avoid prolonged exposure to temperatures exceeding 30°. ▲Store at room temperature. Protect from freezing.▲ (USP 1-Aug-2023)

Add the following:

▲• **Labeling:** Label it to indicate the name and quantity of each active ingredient.▲ (USP 1-Aug-2023)

• **USP Reference Standards ⟨11⟩**

USP Undecylenic Acid RS

[1] From commercially available National Institute of Standards and Technology (NIST)-traceable standard solution for zinc.

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

Topic/Question	Contact	Expert Committee
COMPOUND UNDECYLENIC ACID OINTMENT	Documentary Standards Support	SM12020 Small Molecules 1
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM12020 Small Molecules 1

Chromatographic Database Information: Chromatographic Database

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