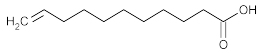
Undecylenic Acid



$C_{11}H_{20}O_2$ 184.28
10-Undecenoic acid.
10-Undecenoic acid [112-38-9].

» Undecylenic Acid contains not less than 97.0 percent and not more than 100.5 percent of $C_{11}H_{20}O_2$.

Packaging and storage—Preserve in tight, light-resistant containers.

Identification—

A: To 1 mL add potassium permanganate TS, dropwise: the permanganate color is discharged.

B: Place 3 mL of it and 3 mL of freshly distilled aniline in a tall test tube, and heat for 10 minutes at a rate such that the ring of condensate remains just below the mouth of the tube. Cool, add 10 mL of alcohol and 10 mL of ether, and transfer to a separator. Wash the ether solution with four 20-mL portions of water, and discard the water washings. Heat on a steam bath until the odor of ether no longer is perceptible, then add a few mg of activated carbon, mix, and filter. Evaporate the filtrate nearly to dryness, and recrystallize the residue from 70 percent alcohol: the anilide so obtained melts between 66° and 67.5°.

Specific gravity ⟨841⟩: between 0.910 and 0.913.

Congealing range ⟨651⟩: not lower than 21°.

Refractive index ⟨831⟩: between 1.447 and 1.448.

Residue on ignition ⟨281⟩: not more than 0.15%.

Water-soluble acids— Shake 5 mL with 5 mL of water, and filter the water layer through a filter paper previously moistened with water. Add 1 drop of methyl orange TS, and titrate with 0.01 N sodium hydroxide VS: not more than 1.0 mL of 0.010 N sodium hydroxide is required to match the color produced by 1 drop of methyl orange TS in 5 mL of water.

Heavy metals, *Method II* ⟨231⟩: 0.001%.

Iodine value ⟨401⟩: between 131 and 138.

Assay— Dissolve about 750 mg of Undecylenic Acid, accurately weighed, in 50 mL of alcohol, add 3 drops of phenolphthalein TS, and titrate with 0.1 N sodium hydroxide VS to the first pink color that persists for not less than 30 seconds. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N sodium hydroxide is equivalent to 18.43 mg of $C_{11}H_{20}O_2$.

Compound Undecylenic Acid Ointment

» Compound Undecylenic Acid Ointment contains undecylenic acid, calcium undecylenate, copper undecylenate, or zinc undecylenate, individually or in any combination, in a suitable ointment base. It contains not less than 90.0 percent and not more than 110.0 percent of the labeled amount of total undecylenic acid ($C_{11}H_{20}O_2$).

Packaging and storage— Preserve in tight containers, and avoid prolonged exposure to temperatures exceeding 30°.

USP Reference standards ⟨11⟩**—**
USP Undecylenic Acid RS

Assay for zinc undecylenate—

Standard preparations—Prepare a solution of freshly ignited zinc oxide in dilute hydrochloric acid (1 in 60) to obtain the equivalent of 1.0 mg of zinc per mL. Dilute quantitatively with water to obtain separate solutions containing the equivalent of 15 and 30 µg of zinc per mL.

Assay preparation—Transfer about 1.0 g of Ointment, accurately weighed, to a 100-mL beaker. Add 25 mL of dilute hydrochloric acid (1 in 20), swirl, and heat carefully until the mixture is liquefied. Cool, and transfer the mixture to a 250-mL separator. Complete the transfer of the waxy residue by thoroughly rinsing the beaker with 50 mL of water and two 50-mL portions of chloroform and adding the rinsings to the separator. Equilibrate the mixture, and transfer the chloroform extract to a 500-mL separator. Extract the aqueous phase with another 100-mL portion of chloroform, combine the second chloroform extract with the main extract in the 500-mL separator, and transfer the aqueous phase to a 200-mL volumetric flask. Wash the combined chloroform extracts with three 25-mL portions of water, add the aqueous washings to the 200-mL volumetric flask, dilute with water to volume, and mix to obtain a specimen stock solution. [NOTE—Retain the chloroform extract for the *Assay for undecylenic acid*.] Transfer 15.0 mL or other suitable volume (see *Procedure*) of this specimen stock solution to a 100-mL volumetric flask, dilute with water to volume, and mix.

Procedure—Aspirate each *Standard preparation* and the *Assay preparation* into the flame of a suitable atomic absorption spectrophotometer, and determine the absorbances of the solutions at 214 nm. Typically, an acetylene–air mixture is adjusted to obtain a blue flame about 7 mm in height with a suitable burner that is rotated to a position perpendicular to the light path. [NOTE—If the absorbance of the *Assay preparation* is outside the central 70% of the range between the absorbances of the *Standard preparations*, discard the *Assay preparation* and prepare another by diluting the specimen stock solution quantitatively as necessary to obtain a suitable absorbance.] Calculate the percentage of zinc undecylenate in the Ointment taken by the formula:

$$(M_r/A_r)(0.2/W)C_{S2}+\frac{(C_{S1}-C_{S2})(A_U-A_{S2})}{(A_{S1}-A_{S2})}$$

in which 431.94 is the molecular weight of zinc undecylenate; 65.39 is the atomic weight of zinc; *W* is the weight, in g, of Ointment taken; A_U, A_H, and A_L are the absorbances of the *Assay preparation* and the high- and low-concentration *Standard preparations*, respectively; and C_H and C_L are the concentrations, in µg per mL, of the high- and low-concentration *Standard preparations*, respectively.

Assay for undecylenic acid—

Internal standard solution—Prepare a solution in chloroform containing 10 mg of tridecanoic acid in each mL.

Standard preparation—Dissolve an accurately weighed quantity of USP Undecylenic Acid RS in chloroform to obtain a solution having a known concentration of about 3.8 mg per mL. Transfer 5.0 mL of this solution to a 50-mL volumetric flask, add 3.0 mL of *Internal standard solution*, dilute with chloroform to volume, and mix.

Assay preparation—Pass the chloroform extract prepared from the Ointment as directed in *Assay for zinc undecylenate* through phase-separating filter paper into a 250-mL volumetric flask. Rinse the separator with three 15-mL portions of chloroform, passing the rinsings through the filter and combining them with the main chloroform solution, add chloroform to volume, and mix. Transfer 20.0 mL of this solution to a 50-mL volumetric flask, add 3.0 mL of *Internal standard solution*, dilute with chloroform to volume, and mix.

Chromatographic system—Under typical conditions, the gas chromatograph is equipped with a flame-ionization detector and contains a 2-mm × 1.8-m glass column packed with 3% liquid phase G1 on 100- to 200-mesh support S1A. The column is maintained at a temperature of about 165°. Dry helium is

used as the carrier gas at a flow rate of about 30 mL per minute.

System suitability—Chromatograph five injections of the silylated *Standard preparation*, and record the peak responses as directed for *Procedure*: the resolution, *R* (see *Chromatography* ⟨621⟩), is not less than 3.0.

Procedure—Transfer 1.0-mL portions of the *Standard preparation* and the *Assay preparation* to separate, stoppered test tubes. To each tube add 50 µL of bis(trimethylsilyl)trifluoroacetamide, insert the stopper, mix, and allow to stand for 30 minutes. Inject a suitable portion (2 to 5 µL) of the *Standard preparation* into a suitable gas chromatograph, and record the chromatogram so as to obtain not less than 50% of maximum recorder response. Similarly inject a suitable portion of the *Assay preparation*, and record the chromatogram. Measure the peak responses for the first (undecylenic acid) and second (tridecanoic acid) peaks of the chromatograms. [NOTE—Relative retention times are, approximately, 0.43 for undecylenic acid and 1.0 for tridecanoic acid.] Calculate the percentage of total undecylenic acid in the Ointment taken by the formula:

$$62.5(C / W)(R_U / R_S)$$

in which *C* is the concentration, in mg per mL, of USP Undecylenic Acid RS in the *Standard preparation; W* is the weight, in g, of Ointment taken; and R_U and R_S are the ratios of the peak responses of undecylenic acid to those of tridecanoic acid from the *Assay preparation* and the *Standard preparation*, respectively. The difference between the total undecylenic acid and the undecylenic acid equivalent to the determined zinc undecylenate (the weight of zinc undecylenate multiplied by 0.8533 gives the equivalent of undecylenic acid), both expressed as a percentage of the Ointment, gives the percentage of free undecylenic acid in the Ointment.

Urea

CH_4N_2O 60.06
Urea.
Carbamide [57-13-6].

» Urea contains not less than 99.0 percent and not more than 100.5 percent of CH_4N_2O.

Packaging and storage—Preserve in well-closed containers. Store at 25°, excursions permitted between 15° and 30°.

Labeling—Where it is intended for use in preparing injectable dosage forms, the label states that it is sterile or must be subjected to further processing during the preparation of injectable dosage forms.

USP Reference standards ⟨11⟩**—**
USP Endotoxin RS

Identification—

A: Heat about 500 mg in a test tube: it liquefies, and ammonia is evolved. Continue the heating until the liquid becomes turbid, then cool. Dissolve the fused mass in a mixture of 10 mL of water and 1 mL of sodium hydroxide solution (1 in 10), and add 1 drop of cupric sulfate TS: the solution acquires a reddish-violet color.

B: Dissolve 100 mg in 1 mL of water, and add 1 mL of nitric acid: a white crystalline precipitate of urea nitrate is formed.

Melting range ⟨741⟩**:** between 132° and 135°.

Residue on ignition ⟨281⟩**:** not more than 0.1%.

Alcohol-insoluble matter—Dissolve 5.0 g in 50 mL of warm alcohol, and if any insoluble residue remains, filter the solution on a tared filter, wash the residue and the filter with 20 mL of warm alcohol, and dry at 105° for 1 hour: the weight of the residue does not exceed 2 mg (0.04%).

Chloride ⟨221⟩**—**A 2.0-g portion shows no more chloride than corresponds to 0.20 mL of 0.020 N hydrochloric acid (0.007%).

Sulfate ⟨221⟩**—**A 2.0-g portion shows no more sulfate than corresponds to 0.20 mL of 0.020 N sulfuric acid (0.010%).

Heavy metals ⟨231⟩**—**Dissolve 1.0 g in 20 mL of water, and add 5 mL of 0.1 N hydrochloric acid: the limit is 0.002%.

Other requirements—Where the label states that Urea is sterile, it meets the requirements for *Sterility Tests* ⟨71⟩ and for *Bacterial endotoxins* under *Urea for Injection*. Where the label states that Urea must be subjected to further processing during the preparation of injectable dosage forms, it meets the requirements for *Bacterial endotoxins* under *Urea for Injection*.

Assay—Transfer about 500 mg of Urea, accurately weighed, to a 200-mL volumetric flask, dissolve in and dilute with water to volume, and mix. Pipet 2 mL of this solution into a micro-Kjeldahl digestion flask, and proceed as directed under *Nitrogen Determination, Method II* ⟨461⟩, beginning with "Add 1 g of a powdered mixture." [NOTE—In this procedure, continue heating the flask until fuming begins, then heat for 1 additional hour.] Each mL of 0.01 N acid is equivalent to 0.3003 mg of CH_4N_2O.

Urea for Injection

» Urea for Injection is Urea suitable for parenteral use.

Packaging and storage— Preserve in *Containers for Sterile Solids* as described under *Injections* ⟨1⟩.

USP Reference standards ⟨11⟩**—**
USP Endotoxin RS

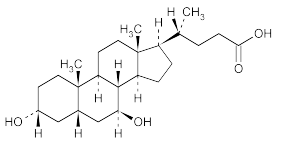
Completeness of solution ⟨641⟩**—** A 1.0-g portion dissolves in 10 mL of carbon dioxide-free water to yield a clear solution.

Constituted solution— At the time of use, it meets the requirements for *Constituted Solutions* under *Injections* ⟨1⟩.

Bacterial endotoxins ⟨85⟩**—** It contains not more than 0.003 USP Endotoxin Unit per mg of urea.

Other requirements—It responds to the *Identification* tests and meets the requirements for *Melting range*, *Residue on ignition*, *Alcohol-insoluble matter*, *Chloride*, *Sulfate*, *Heavy metals*, and *Assay* under *Urea.* It meets also the requirements for *Sterility Tests* ⟨71⟩, *Uniformity of Dosage Units* ⟨905⟩, and *Labeling* under *Injections* ⟨1⟩.

Ursodiol



$C_{24}H_{40}O_4$ 392.57
Cholan-24-oic acid, 3,7-dihydroxy-, (3α,5β,7β)-.
3α,7β-Dihydroxy-5β-cholan-24-oic acid [128-13-2].

» Ursodiol contains not less than 98.5 percent and not more than 101.5 percent of $C_{24}H_{40}O_4$, calculated on the dried basis.

Packaging and storage— Preserve in tight containers.