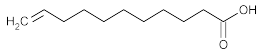
Undecylenic Acid



$C_{11}H_{20}O_2$ 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