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Ciclopirox Olamine Cream

» Ciclopirox Olamine Cream contains not less than 90.0 percent and not more than 110.0 percent of the labeled amount of ciclopirox olamine ($C_{12}H_{17}NO_2 \cdot C_2H_7NO$).

Packaging and storage—Preserve in collapsible tubes, and store at controlled room temperature.

USP Reference standards ⟨11⟩—

USP Benzyl Alcohol RS
USP Ciclopirox Olamine RS

Identification— Dilute 4 mL of the *Assay preparation* obtained as directed in the *Assay* with a mixture of methanol and 6.25 N sodium hydroxide (123:2) to make 100 mL: the UV absorption spectrum of the solution so obtained exhibits maxima and minima at the same wavelengths as that of a similar solution prepared from the *Standard preparation* obtained as directed in the *Assay,* concomitantly measured.

Minimum fill ⟨755⟩**:** meets the requirements.

pH ⟨791⟩—Add 15 mL of boiling water, previously adjusted with 0.1 N hydrochloric acid or 0.1 N sodium hydroxide to a pH of 6 to 7, to 3.5 g of Cream in a 50-mL centrifuge tube. Place a cap on the tube, and shake vigorously until an emulsion is formed. Loosen the cap, and heat the tube on a steam bath for 10 minutes. Allow to cool, centrifuge, and determine the pH of the aqueous phase: the pH is between 5.0 and 8.0.

Content of benzyl alcohol *(if present)*—

Solvent mixture—Mix chloroform and methanol (4:1).

Internal standard solution—Prepare a solution of 1-nonyl alcohol in *Solvent mixture* containing about 1.75 mg per mL.

Standard preparation—Dilute an accurately weighed quantity of USP Benzyl Alcohol RS, quantitatively and stepwise, with *Solvent mixture* to obtain a solution having a known concentration of about 2 mg per mL. Transfer 5.0 mL of this solution and 5.0 mL of *Internal standard solution* to a 50-mL volumetric flask, dilute with *Solvent mixture* to volume, and mix.

Test preparation—Transfer 1.0 g of Cream to a 50-mL volumetric flask, add about 30 mL of *Solvent mixture,* and mix. Add 5.0 mL of *Internal standard solution,* dilute with *Solvent mixture* to volume, and mix to obtain a clear solution.

Chromatographic system (see *Chromatography* ⟨621⟩)—The gas chromatograph is equipped with a flame-ionization detector and contains a 4-mm × 2-m glass column packed with 3% phase G3 on 100- to 120-mesh support S1AB. The column is maintained at a temperature of about 100°, the injection port and detector temperatures are maintained at about 315°, and nitrogen is used as the carrier gas at a flow rate of about 45 mL per minute. Chromatograph the *Standard preparation,* and record the peak responses as directed for *Procedure:* the resolution, *R,* between the peaks is not less than 1.6; the tailing factor for the benzyl alcohol peak and the internal standard peak is not greater than 3.5; and the relative standard deviation for replicate injections is not more than 3%.

Procedure—Separately inject equal volumes (about 2 µL) of the *Standard preparation* and the *Test preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. [Note—After six injections, raise the column temperature to about 300° for about 5 minutes, then cool to 100°.] Calculate the percentage of benzyl alcohol in the Cream taken by the formula:

$$C(R_U/R_S),$$

in which C is the concentration, in mg per mL, of benzyl alcohol in the *Standard preparation;* and R_U and R_S are the peak response ratios of the benzyl alcohol peak to the internal standard peak obtained from the *Test preparation* and the *Standard preparation,* respectively: between 90.0% and 110.0% of the claimed amount is present.

Assay—

Ferrous sulfate solution—Transfer 600 mg of ferrous sulfate to a 25-mL volumetric flask. Add 0.6 mL of glacial acetic acid, dilute with water to volume, and mix.

Standard preparation—Dissolve an accurately weighed quantity of USP Ciclopirox Olamine RS in methanol to obtain a solution having a known concentration of about 0.2 mg per mL.

Assay preparation—Transfer an accurately weighed quantity of Cream, equivalent to about 10 mg of ciclopirox olamine, to a 50-mL volumetric flask, add 25 mL of methanol, and shake by mechanical means for about 10 minutes. Dilute with methanol to volume, mix, centrifuge, and use the supernatant.

Procedure—Transfer 4.0 mL of the *Standard preparation,* 4.0 mL of the *Assay preparation,* and 4.0 mL of methanol to provide a blank, to separate 25-mL volumetric flasks. Add 15 mL of methanol to each flask, and mix. Then to each flask add 1.0 mL of *Ferrous sulfate solution,* mix, dilute with methanol to volume, and mix. Store the flasks in the dark for 1 hour. Concomitantly determine the absorbances of the solutions from the *Assay preparation* and the *Standard preparation* against the blank in 1-cm cells at the wavelength of maximum absorbance at about 440 nm, with a suitable spectrophotometer. Calculate the quantity, in mg, of ciclopirox olamine ($C_{12}H_{17}NO_2 \cdot C_2H_7NO$) in each g of the Cream taken by the formula:

$$50(C/W)(A_U/A_S)$$

in which C is the concentration, in mg per mL, of USP Ciclopirox Olamine RS in the *Standard preparation; W* is the weight, in g, of Cream taken; and A_U and A_S are the absorbances of the solutions from the *Assay preparation* and the *Standard preparation,* respectively.

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

Topic/Question	Contact	Expert Committee
CICLOPIROX OLAMINE CREAM	Documentary Standards Support	SM12020 Small Molecules 1

Chromatographic Database Information: Chromatographic Database

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