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Dioxybenzone and Oxybenzone Cream

DEFINITION

Dioxybenzone and Oxybenzone Cream is a mixture of approximately equal parts of Dioxybenzone and Oxybenzone in a suitable cream base. In each 100 g, it contains NLT 2.7 g and NMT 3.3 g each of dioxybenzone ($C_{14}H_{12}O_4$) and oxybenzone ($C_{14}H_{12}O_3$).

IDENTIFICATION

Change to read:

• **A.** ▲**Spectroscopic Identification Tests** ⟨197⟩, *Ultraviolet-Visible Spectroscopy*: **197U**▲ (CN 1-May-2020)

Sample solutions: The solutions from the Cream used for measurement of absorbance in the *Assay*

Acceptance criteria: Meets the requirements

ASSAY

• **Procedure**

Standard solution A: 12 µg/mL of USP Dioxybenzone RS in methanol

Standard solution B: 12 µg/mL of USP Oxybenzone RS in methanol

Sample solution: Dissolve a portion of Cream, equivalent to 25 mg each of dioxybenzone and oxybenzone, in methanol in a 100-mL volumetric flask, and dilute with methanol to volume. Pipet 1 mL of this solution into a 15-mL conical test tube, evaporate on a water bath just to dryness using a gentle current of air, and dissolve the residue in 200 µL of methanol.

Analysis

Prepare sheets of chromatographic paper (Whatman No. 1 or equivalent), each measuring about 23 × 28.5 cm, as follows. Immerse the sheets in a 1-in-20 solution of light mineral oil in solvent hexane, withdraw them immediately, and allow to air-dry. On one sheet, mark a starting line about 2.5 cm from the long edge, and apply the entire *Sample solution* as a uniform streak along the starting line. Use a current of air or an air blower, if necessary, to maintain the width of the streak between 5 and 10 mm. Rinse the conical test tube that contained the *Sample solution* with 100 µL of methanol, and apply the rinse to the starting line. Similarly, repeat the rinsing and streaking with two additional portions of methanol, then allow the paper to air-dry for 5 min.

Staple together the short edges of the paper to form a cylinder, and place it in a 12-cm × 25-cm cylindrical chromatographic chamber containing 40 mL of a mobile solvent consisting of a mixture of equal volumes of acetone and water. Seal the chamber, and allow the chromatogram to develop for 2 h.

Remove the paper from the chamber, air-dry, remove the staples, and view the chromatogram under short-wavelength (254 nm) UV radiation. Mark the two bands representing the separated dioxybenzone and oxybenzone, respectively.

Determine the relative position of each benzone on the chromatogram by applying suitable aliquots of each *Standard solution* to another prepared chromatographic sheet and developing the chromatogram in a manner similar to that described for the *Sample solution*.

Cut the marked bands from the sheet, then, keeping the band segments separate, cut each into several pieces to facilitate extraction. Place the pieces from each band in separate glass-stoppered 50-mL conical flasks, add 20.0 mL of methanol to each flask, and shake gently for 30 min.

Blank: To provide the chromatographic blank, treat one of the prepared chromatographic sheets in the same manner as described above, but omit the application of the *Sample solution*. From the chromatographed paper, cut the areas corresponding to the bands produced by the benzones from the *Sample solution*, and in the same manner extract the blank bands for 30 min with 20.0 mL of methanol.

Instrumental conditions

Mode: UV

Cell: 1 cm

Analytical wavelength: Maximum absorbance at about 325 nm

Analysis

Samples: Resulting solutions from *Standard solution A*, *Standard solution B*, *Sample solution*, and *Blank*

Calculate the quantity, in mg, of dioxybenzone ($C_{14}H_{12}O_4$) in the portion of Cream taken:

$$\text{Result} = [(A_U - A_B)/A_S)] \times C \times 2$$

A_U = absorbance of the dioxybenzone solution from the *Sample solution*

A_B = absorbance of the *Blank*

A_S = absorbance of *Standard solution A*

C = concentration of USP Dioxybenzone RS in *Standard solution A* (µg/mL)

Calculate the quantity, in mg, of oxybenzone ($C_{14}H_{12}O_3$) in the portion of Cream taken:

$$\text{Result} = [(A_U - A_B)/A_S)] \times C \times 2$$

A_U = absorbance of the oxybenzone solution from the *Sample solution*

A_B = absorbance of the *Blank*

A_S = absorbance of *Standard solution B*

C = concentration of USP Oxybenzone RS in *Standard solution B* (µg/mL)

Acceptance criteria: In each 100 g, NLT 2.7 g and NMT 3.3 g each of dioxybenzone ($C_{14}H_{12}O_4$) and oxybenzone ($C_{14}H_{12}O_3$)

PERFORMANCE TESTS

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

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers.

• **USP Reference Standards ⟨11⟩**

USP Dioxybenzone RS

USP Oxybenzone RS

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

Topic/Question	Contact	Expert Committee
DIOXYBENZONE AND OXYBENZONE CREAM	Documentary Standards Support	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. Information currently unavailable

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