Edition: BP 2025 (Ph. Eur. 11.6 update)

Fluocinonide Ointment

General Notices

Action and use

Glucocorticoid.

DEFINITION

Fluocinonide Ointment contains Fluocinonide in a suitable basis.

The ointment complies with the requirements stated under Topical Semi-solid Preparations and with the following requirements.

Content of fluocinonide, $C_{26}H_{32}F_2O_7$

90.0 to 110.0% of the stated amount.

IDENTIFICATION

A. Carry out the method for *thin-layer chromatography*, Appendix III A, using the following solutions.
(1) Disperse a quantity of the ointment containing 2 mg of fluocinonide in 20 mL of *methanol (80%)* in a 100-mL separating funnel containing 50 mL of *2,2,4-trimethylpentane*. Warm the contents over a water bath and shake gently. Allow the layers to separate and transfer the lower layer to a 250-mL separating funnel containing 100 mL of *water*. Add 10 mL of *chloroform* and shake for 3 minutes. Allow the layers to separate, evaporate the lower layer to dryness on a water bath in a current of air and dissolve the residue in 1 mL of *chloroform*.
(2) 0.2% w/v of *fluocinonide BPCRS* in *chloroform*.

CHROMATOGRAPHIC CONDITIONS

(a) Use as the coating *silica gel* F_{254}.
(b) Use the mobile phase as described below.
(c) Apply 10 µL of each solution.
(d) Develop the plate to 15 cm.
(e) After removal of the plate, allow it to dry in air until the solvent has evaporated. Heat at 105° for 5 minutes and spray while hot with *alkaline tetrazolium blue solution*.

MOBILE PHASE

12 volumes of *water*, 80 volumes of *methanol*, 150 volumes of *ether* and 770 volumes of *dichloromethane*. Mix the water and the methanol before adding to the remaining components of the mobile phase.

CONFIRMATION

The principal spot in the chromatogram obtained with solution (1) corresponds in position and colour to that in the chromatogram obtained with solution (2).

B. In the Assay, the retention time of the principal peak in the chromatogram obtained with solution (1) is similar to that of the principal peak in the chromatogram obtained with solution (2).

ASSAY

Carry out the method for *liquid chromatography*, Appendix III D, using the following solutions.

(1) Disperse a quantity of the ointment containing 1.5 mg of fluocinonide in 20 mL of *methanol (80%)* in a 100-mL separating funnel containing 50 mL of *2,2,4-tri-methylpentane*. Warm the contents over a water bath and shake gently for 2 minutes. Allow the layers to separate and transfer the lower layer to a 50 mL flask. Repeat the extraction with a further 20 mL of *methanol (80%)*. Dilute the combined extracts to 50 mL with the same solvent.
(2) 0.003% w/v of *fluocinonide BPCRS* in *methanol (80%)*.

CHROMATOGRAPHIC CONDITIONS

(a) Use a stainless steel column (20 cm × 4.6 mm) packed with *octadecylsilyl silica gel for chromatography* (5 µm) (Spherisorb ODS 1 is suitable).
(b) Use isocratic elution and the mobile phase described below.
(c) Use a flow rate of 2 mL per minute.
(d) Use an ambient column temperature.
(e) Use a detection wavelength of 238 nm.
(f) Inject 20 µL of each solution.

MOBILE PHASE

0.1 volume of *glacial acetic acid*, 45 volumes of *acetonitrile* and 55 volumes of *water*.

DETERMINATION OF CONTENT

Calculate the content of $C_{26}H_{32}F_2O_7$ in the ointment using the declared content of $C_{26}H_{32}F_2O_7$ in *fluocinonide BPCRS*.