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Benzoic and Salicylic Acids Ointment

DEFINITION

Benzoic and Salicylic Acids Ointment is Benzoic Acid and Salicylic Acid, present in a ratio of 2:1, in a suitable ointment base. It contains NLT 90.0% and NMT 110.0% of the labeled amounts of benzoic acid ($C_7H_6O_2$) and salicylic acid ($C_7H_6O_3$).

IDENTIFICATION

• **A. Thin-Layer Chromatography**

Diluent: Mixture of chloroform and methanol (1:1)

Standard solution A: 2.4 mg/mL of USP Benzoic Acid RS in *Diluent*

Standard solution B: 1.2 mg/mL of USP Salicylic Acid RS in *Diluent*

Sample solution: Equivalent to 60 mg of benzoic acid and 30 mg of salicylic acid from Ointment, in 25 mL of *Diluent*

Chromatographic system

(See *Chromatography ⟨621⟩, Thin-Layer Chromatography*.)

Mode: TLC

Adsorbent: 0.25-mm layer of chromatographic silica gel mixture

Application volume: 5 µL of each solution at separate points 2.5 cm from the bottom edge of a 20- × 20-cm thin-layer chromatographic plate

Developing solvent system: Chloroform, acetone, isopropyl alcohol, methanol, and ammonium hydroxide (30:30:15:15:10)

Analysis

Samples: *Standard solution A*, *Standard solution B*, and *Sample solution*

Develop the chromatogram in the *Developing solvent system* until the solvent front has moved three-fourths of the length of the plate. Remove the plate from the chromatographic chamber, mark the solvent front, and allow the solvent to evaporate. View the chromatogram under short-wavelength (254 nm) UV radiation.

Acceptance criteria: The two major fluorescent spots from the *Sample solution* correspond in color and in R_F value to those from *Standard solution A* and *Standard solution B*.

ASSAY

• **Procedure**

Ferric chloride–urea reagent: On the day of use, dissolve, without heating, 18 g of urea in a mixture of 2.5 mL of ferric chloride solution (6 in 10) and 12.5 mL of 0.05 N hydrochloric acid.

Column A: Insert a small pledget of glass wool above the stem constriction of a 20- × 2.5-cm chromatographic tube. Mix 1 g of chromatographic siliceous earth with 0.5 mL of dilute phosphoric acid (3 in 10) to form a uniform, fluffy mixture; transfer to the chromatographic tube; and pack evenly over the glass wool, exerting gentle pressure. Similarly, mix 4 g of chromatographic siliceous earth with 3 mL of *Ferric chloride–urea reagent*, and pack uniformly over the first layer. Cover the column with a pad of glass wool.

Column B: Insert a small pledget of glass wool above the stem constriction of a second 20- × 2.5-cm chromatographic tube. Mix 4 g of chromatographic siliceous earth with 2 mL of sodium bicarbonate solution (1 in 12), prepared just before use, to a uniform, fluffy mixture; and pack evenly over the glass wool. Cover the column with a pad of glass wool.

Diluent: Glacial acetic acid in chloroform (3 in 100)

Standard solution A: 20 µg/mL of USP Salicylic Acid RS in *Diluent*

Standard solution B: 40 µg/mL of USP Benzoic Acid RS in *Diluent*

Sample solution: Transfer an amount of the Ointment, equivalent to 100 mg of benzoic acid and 50 mg of salicylic acid, to a 250-mL volumetric flask, and dissolve in 150 mL of chloroform by warming on a steam bath. Cool. Dilute with chloroform to volume to obtain a solution having a nominal concentration of 200 µg/mL of salicylic acid and 400 µg/mL of benzoic acid.

Analysis

Samples: *Standard solution A*, *Standard solution B*, and *Sample solution*

Mount *Column A* directly over *Column B*, then pipet 10 mL of *Sample solution* onto *Column A*, and allow it to pass into the column. Wash the columns with two 40-mL portions of chloroform, allowing the first portion to recede to the top of each column before adding the second portion. Discard the eluates, and separate the columns.

Salicylic acid content: Elute *Column A* with 95 mL of *Diluent*, collecting the eluate in a 100-mL volumetric flask. Dilute the contents of the flask with *Diluent* to volume, and mix. Concomitantly determine the absorbances of the eluate and *Standard solution A* in 1-cm cells at the wavelength of maximum absorbance at 311 nm, with a suitable spectrophotometer, using *Diluent* as the blank.

Calculate the percentage of the labeled amount of salicylic acid ($C_7H_6O_3$) in the portion of Ointment taken:

$$\text{Result} = (A_U/A_S) \times (C_S/C_U) \times F \times 100$$

A_U = absorbance of the diluted eluate from *Column A*

A_S = absorbance of *Standard solution A*

C_S = concentration of USP Salicylic Acid RS in *Standard solution A* (µg/mL)

C_U = nominal concentration of the salicylic acid in the *Sample solution* (µg/mL)

F = sample dilution factor, 10

Acceptance criteria: 90.0%–110.0%

Benzoic acid content: Elute *Column B* with 95 mL of *Diluent*, collecting the eluate in a 100-mL volumetric flask. Dilute the contents of the flask with *Diluent* to volume, and mix. Concomitantly determine the absorbances of eluate and *Standard solution B* in 1-cm cells at the wavelength of maximum absorbance at 275 nm, with a suitable spectrophotometer, using *Diluent* as the blank.

Calculate the percentage of the labeled amount of benzoic acid ($C_7H_6O_2$) in the portion of Ointment taken:

$$\text{Result} = (A_U/A_S) \times (C_S/C_U) \times F \times 100$$

A_U = absorbance of the diluted eluate from *Column B*

A_S = absorbance of *Standard solution B*

C_S = concentration of USP Benzoic Acid RS in *Standard solution B* (µg/mL)

C_U = nominal concentration of benzoic acid in the *Sample solution* (µg/mL)

F = sample dilution factor, 10

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

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

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers, and avoid exposure to temperatures exceeding 30°.

• **Labeling:** Label Ointment to indicate the concentrations of Benzoic Acid and Salicylic Acid and to indicate whether the ointment base is water-soluble or water-insoluble.

• **USP Reference Standards ⟨11⟩**

USP Benzoic Acid RS

USP Salicylic Acid RS

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

Topic/Question	Contact	Expert Committee
BENZOIC AND SALICYLIC ACIDS OINTMENT	Documentary Standards Support	SM12020 Small Molecules 1

Chromatographic Database Information: Chromatographic Database

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