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Pharmaceutical Glaze

DEFINITION

Pharmaceutical Glaze is a specially denatured alcoholic solution of Shellac containing between 20.0% and 57.0% of anhydrous shellac and is made with either dehydrated alcohol or alcohol containing 5% water by volume. The solvent is a specially denatured alcohol approved for glaze manufacturing by the Internal Revenue Service. It contains NLT 90.0% and NMT 110.0% of the labeled amount of shellac. It may contain waxes and titanium dioxide as an opaquing agent.

IDENTIFICATION

- **A.**

Sample: Remaining sample solution retained from the *Assay*

Analysis: Pour the *Sample* from a volumetric flask onto a clean glass plate, and place the plate in a nearly vertical position. After drainage is complete, allow the resulting film to dry in a well-ventilated place at 20° for 1 h, then place the plate in an oven at a temperature of 43° for 16–24 h. Cool, and scrape the film from the plate with a sharp blade, discarding the thick edges. To 50 mg of the solid shellac from the film add a few drops of a mixture of 1 g of ammonium molybdate and 3 mL of sulfuric acid. Retain the remaining film for the tests for *Identification of Aleuritic Acid and Shellolic Acid by Thin-Layer Chromatography* and *Rosin*.

Acceptance criteria: A green color is produced, and it becomes lilac on standing for 5 min.

- **B. Identification of Aleuritic Acid and Shellolic Acid by Thin-Layer Chromatography**

Standard solution: 6 mg/mL of USP Aleuritic Acid RS in methanol, heating slightly if necessary

Solid shellac sample: 500 mg of solid shellac from the film prepared in *Identification* test *A*

Sample solution: Weigh and finely powder the *Solid shellac sample*. Transfer 500 mg of the finely powdered shellac to a test tube and heat with 4 mL of 85-mg/mL sodium hydroxide solution in a vigorously boiling water bath for 5 min. Cool, add 10 mL of ethyl acetate, and transfer the content to a separatory funnel. With stirring, add slowly 4 mL of a 120-mg/mL solution of glacial acetic acid to the funnel. Shake the solution thoroughly and withdraw the lower layer. Transfer the upper layer to a small flask, add anhydrous sodium sulfate, and pass the content through a 0.45-µm syringe filter.[1] Collect the filtrate and use it as the sample.

Chromatographic system

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

Mode: TLC

Plate: 10-cm × 20-cm, or 20-cm × 20-cm, silica gel 60 F_{254}

Application volume: 10 µL, as 8-mm bands. [Note—An automated apparatus may be used.]

Developing solvent system: Ethyl acetate, methylene chloride, methyl alcohol, and acetic acid (60:32:8:1)

Spray reagent: Prepare the anisaldehyde solution by mixing in the following order. In 0.5 mL of anisaldehyde add 10 mL of glacial acetic acid, 85 mL of methyl alcohol, and 5 mL of sulfuric acid.

Analysis

Samples: *Standard solution* and *Sample solution*

Development: Apply the *Samples* in different bands to the previously marked starting point on a TLC plate, and develop the plate two times over a path of 15 cm. Dry the plate in air.

Detection: Spray with *Spray reagent*. Heat the plate at 100°–105° for 10 min, and examine in daylight (or white light).

[Note—The principal band of aleuritic acid shows strong intensity and purple color. The retardation factor (R_F) for the principal band of aleuritic acid is 0.41. A blue-gray band with medium intensity at R_F 0.22 could be assigned to shellolic acid.]

Acceptance criteria: The chromatogram from the *Sample solution* shows several colored bands. One of the colored bands is similar in position and color to the band in the chromatogram from the *Standard solution*, and it is assigned to aleuritic acid. Below the aleuritic acid band, a blue-gray band is assigned to shellolic acid.

ASSAY

• **Procedure**

Sample solution A: When testing Glaze that does not contain titanium dioxide, transfer a quantity of Glaze containing 17 g of shellac to a 100-mL volumetric flask, and add alcohol to volume. Pipet 3 mL into a tared dish containing 10 g of washed sand and a small glass rod. Retain the remaining solution in the volumetric flask for *Identification* test *A*. The tare weight includes the combined weights of the dish, the washed sand, and the glass rod.

Stir until a uniform mixture is obtained, allow the glass rod to remain in the dish, dry at 105° for 1 h in an explosion-proof oven, cool, and weigh.

Sample solution B: When testing Glaze that contains titanium dioxide, transfer a quantity containing 10 g of solids to a beaker, and add 10 mL of alcohol. Filter off the pigment with the aid of a vacuum. Wash the filter with alcohol; transfer the combined filtrate and washing, with the aid of alcohol, to a 200-mL volumetric flask; add alcohol to volume; and mix. Pipet 6 mL into a tared dish containing 10 g of washed sand and a small glass rod. Proceed as directed in *Sample solution A*, beginning with "The tare weight...".

Analysis: The weight of shellac, W_F, in g, in the quantity of Glaze taken is obtained by subtracting the tare weight from the final weight of the dried dish and contents.

For Glaze not containing titanium dioxide calculate the percentage of shellac in the quantity of Glaze taken:

$$\text{Result} = [(W_F/V_F) \times V_I]/W \times 100$$

V_F = final volume used, 3 mL

V_I = initial volume prepared, 100 mL

W = weight of Glaze (g) for preparation of 100-mL solution

For Glaze containing titanium dioxide calculate the percentage of shellac in the quantity of Glaze taken:

$$\text{Result} = [(W_F/V_F) \times V_I]/W \times 100$$

V_F = final volume used, 6 mL

V_I = initial volume prepared, 200 mL

W = weight of Glaze (g) for preparation of 200-mL solution

Acceptance criteria: 90.0%–110.0% of the labeled amount of shellac

OTHER COMPONENTS

• **Wax**

Sample: Weigh, by difference, a quantity of Glaze containing 10 g of shellac into a 200-mL tall-form beaker.

Analysis: To the *Sample* add with stirring 150 mL of hot water containing 2.5 g of sodium carbonate, immerse the beaker in a boiling water bath, and stir until the solid is dissolved. Cover the beaker with a watch glass, and maintain the heat for 3 h more without agitation. Remove the beaker to a cold water bath. When the wax has floated to the surface, pass the solution through medium-speed quantitative ashless filter paper, transferring the wax to the paper, and wash the filter with water. Pour 5–10 mL of alcohol onto the filter to facilitate drying. Wrap the paper loosely in a larger piece of filter paper, bind with a piece of fine wire, and dry with the aid of gentle heat. Extract with chloroform in a suitable continuous extraction apparatus for 2 h, using a weighed flask to receive the extracted wax and solvent. Evaporate the solvent, and dry the wax at 105° to constant weight.

Acceptance criteria: It meets the requirements in *Table 1*.

Table 1

Type of Shellac	Wax
Orange shellac	NMT 5.5%
Refined orange shellac	NMT 0.2%
Regular bleached shellac	NMT 5.5%
Refined bleached shellac	NMT 0.2%

IMPURITIES

• **Rosin**

Sample: 2 g of the solid shellac from the film prepared in *Identification* test *A*

Analysis: Dissolve the *Sample* by shaking with 10 mL of dehydrated alcohol. Add slowly, with shaking, 50 mL of solvent hexane, wash with two successive 50-mL portions of water, filter the washed alcohol–solvent hexane solution, and evaporate to dryness. To the residue add 2 mL of a mixture of liquefied phenol, dehydrated alcohol, and solvent hexane (1:0.5:2). Stir, and transfer a portion of the solution to the cavity of a color-reaction plate. Fill an adjacent cavity with a mixture of bromine and solvent hexane (1:4), and cover both cavities with an inverted watch glass.

Acceptance criteria: No purple or deep indigo-blue color is produced in or above the liquid containing the residue.

SPECIFIC TESTS

• **Acid Value**

Sample: Weigh, by difference, a quantity of Glaze containing 2 g of shellac.

Analysis: Dissolve the *Sample* in 50 mL of alcohol that has been neutralized to phenolphthalein with 0.1 N sodium hydroxide, add additional phenolphthalein TS if necessary, and titrate with 0.1 N sodium hydroxide VS to a pink endpoint, or determine the endpoint potentiometrically (see *Titrimetry* ⟨541⟩). [Note—For Glaze containing orange shellac, titrate slowly, stirring vigorously, until a glass rod dipped into the titrated solution produces a color change when touched to a drop of thymol blue TS on a spot plate.]

Express the acid value in terms of the number of mg of potassium hydroxide required per g of dried shellac. Calculate the acid value as directed in *Fats and Fixed Oils* ⟨401⟩, *Acid Value*.

Acceptance criteria: It meets the requirements in *Table 2*.

Table 2

Type of Shellac	Acid Value (on Dried Basis)
Orange shellac	68–76
Refined orange shellac	68–79
Regular bleached shellac	73–89
Refined bleached shellac	75–91

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight, lined metal or plastic containers, protected from excessive heat, preferably at a temperature below 25°.

• **Labeling:** Label it to indicate the type of shellac. Label it also to indicate the shellac concentration, the composition of the solvent, and the quantity of titanium dioxide, if present. Where titanium dioxide or waxes are present, the label must state that the Glaze requires mixing before use.

• **USP Reference Standards** ⟨11⟩

USP Aleuritic Acid RS

[1] A 0.45-µm GHP membrane syringe filter is suitable.

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

Topic/Question	Contact	Expert Committee
PHARMACEUTICAL GLAZE	Documentary Standards Support	SE2020 Simple Excipients

Chromatographic Database Information: Chromatographic Database

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