



Designation: D 29 – 98

Standard Test Methods for Sampling and Testing Lac Resins¹

This standard is issued under the fixed designation D 29; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover procedures for sampling and testing orange shellac, button lac, garnet lac, and bleached lac.

1.2 The sampling procedures and test methods appear in the following order:

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Sampling:	
Orange Shellac, Button Lac, and Garnet Lac	3
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Volatile Matter (Moisture)	14 to 15.2
Wax	16 to 16.4
Matter Soluble in Water	17 to 17.2
Ash	18 to 18.2
Color	19 to 19.4.3
Color of Orange Shellac	20 to 20.4.4
Acid Value	21 to 21.4
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Saponification Value	23 to 23.4

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Note 1.

2. Referenced Documents

2.1 ASTM Standards:

- D 304 Specification for *n*-Butyl Alcohol (Butanol)²
- D 331 Specification for 2-Ethoxyethanol²
- D 1193 Specification for Reagent Water³
- D 1544 Test Method for Color of Transparent Liquids

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D 01.33 on Polymers and Resins.

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² *Annual Book of ASTM Standards*, Vol 06.04.

³ *Annual Book of ASTM Standards*, Vol 11.01.

(Gardner Color Scale)⁴

D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method⁵

D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids⁵

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁶

SAMPLING

3. Orange Shellac, Button Lac, and Garnet Lac

3.1 *Lot Size*—For the purpose of sampling, the quantity of a lot of any one of these types of lac resin shall not exceed 500 bags or packages. The net weight of lac resin in each bag or package shall not exceed 75 kg.

3.2 *Source and Number of Samples*—Only original unopened bags or packages shall be sampled. Ten percent of the containers in every lot of lac resin shall be taken at random, but not less than 5 nor more than 25 containers shall be taken.

3.3 *Free-Flowing Lac Resins*—In sampling free-flowing lac resins, samples shall be drawn from different places in each container in double handfuls or by means of a suitable sampler such as a grain sampler. A total of approximately 2.7 kg shall be taken.

3.4 *Blocked or Matted Lac Resin*—Pieces of blocked or matted lac resin shall be chipped with an axe, pick, or other suitable instrument from each container taken for sampling. Approximately the same amount shall be taken from each container and the total amount taken shall be about 2.7 kg. The pieces of lac resin shall then be ground to pass a No. 4 (4.75-mm) sieve. All sieves referenced must conform to the requirements of Specification E 11 .

3.5 *Preparation of Samples for Observation or Analysis*—Whether free-flowing or rough ground, as in the case of blocked lac resin, the entire sample representing any lot shall be thoroughly mixed and divided into halves. The use of a mechanical mixer is recommended for mixing the resin and a riffle sampler for dividing it into quarters. When these devices are not available for use, the entire sample shall be mixed,

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ *Annual Book of ASTM Standards*, Vol 06.03.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

heaped, and quartered along two diameters that intersect at right angles and the opposite quarters combined. One half the sample, thus obtained, shall then be mixed and divided into quarters as before. Each quarter shall be placed in an airtight container, sealed, labeled (Section 5), and sent to the interested parties as the “original observation sample.” When agreed upon between the seller and the purchaser, the “original observation sample” shall be used for the determination of volatile matter (moisture) (Sections 14 to 15, as applicable). The other half of the sample shall be ground to pass a No. 10 (2.00-mm) sieve, mixed thoroughly, and divided into two equal portions A and B. Portion A shall be labeled the “reserve sample.” Portion B shall then be ground to pass a No. 25 (710- μ m) sieve, mixed thoroughly, and quartered as described above. Each quarter shall be packaged in an airtight container, sealed, labeled “prepared sample,” and sent to the testing laboratory for analysis.

4. Bleached Lac

4.1 *Lot Size*—For the purpose of sampling, the quantity of a lot shall not exceed 200 packages.

4.2 *Source and Number of Samples*—Only original unopened packages shall be sampled. Twenty percent of the containers in every lot shall be taken at random, but not less than two containers in any lot shall be taken, except in the case where the entire lot is packaged in a single container.

4.3 *Dry Bleached Lac (Free-Flowing)*—Samples shall be drawn with a scoop or suitable sampler from different parts of each container directly after the packages are opened or bored. Approximately 450 g shall be drawn from each container. The samples shall be combined, mixed thoroughly, and where larger than 1.4 kg, shall be reduced by quartering as prescribed in 3.5 to a sample of this size.

4.4 *Dry Bleached Lac (Blocked or Matted)*—Samples aggregating at least 450 g shall be chopped or chiseled from different parts of each container. The composite sample from all the containers shall be quickly crushed to lumps about 25 mm square or smaller. The crushed lac resin shall be well mixed and where the amount is larger than 1.4 kg, it shall be reduced by quartering, as prescribed in 3.5, to a sample of approximately this size.

4.5 *Hanks, Bars, or Crushed Free-Ground Bleached Lac*—This material, which generally contains approximately 25 % moisture, shall be sampled by the procedures described in 4.3 or 4.4, as applicable.

4.6 *Preparation of Samples for Analysis*—The composite sample obtained as described in 4.3 or 4.4 shall be mixed thoroughly and divided into two equal portions A and B as prescribed in 3.5. Each portion shall be placed in a clean, dry glass jar provided with a rubber-sealed cap or an airtight friction-top tin can. Portion A shall be labeled “reserve sample.” Portion B obtained in accordance with 4.3 or 4.4 shall be further ground to pass a No. 20 (850- μ m) sieve, thoroughly mixed and replaced in the jar, sealed and labeled “prepared sample.” Portion B obtained in accordance with 4.5 shall be further ground to pass a No. 10 (2.00-mm) sieve, thoroughly mixed, replaced in the jar, sealed, labeled “prepared sample,” and sent to the laboratory for analysis.

5. Identification of Samples

5.1 The following information shall be legibly placed on the label, which shall be securely attached to each sample container: date of the sampling, number of bags, barrels or packages sampled, total number of containers in the lot, condition of the containers and their contents, manufacturer’s name, lot and code numbers of the containers, and the purpose identification, namely “original observation sample” or “sample for determination of volatile matter (moisture)”, “reserve sample” or “prepared sample” as may apply.

TEST METHODS

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the Specifications of the American Chemical Society, where such specifications are available.⁷ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

7. Source and Preparation of Specimens for Tests

7.1 Each portion of sample for use in a given test shall be taken from the sample of lac resin only after it has been mixed, either by rolling on paper or by rolling and tumbling in its airtight container, as the condition of the sample requires, a sufficient number of times to ensure uniformity of the specimen taken. The test specimens shall be taken from the “prepared sample” (3.5 or 4.6), as received, except in the following cases:

7.1.1 When it has been previously agreed upon between the seller and the purchaser that the “original observation sample” shall be used for the determination of volatile matter (moisture). In this case, the “original observation sample” shall be mixed, quartered, ground, and sieved in accordance with the procedure described in 3.5 for obtaining the “prepared sample.” All operations shall be done as expeditiously as possible and the test specimen taken immediately after the sieved sample has been thoroughly mixed, to avoid any possible loss by evaporation.

7.1.2 When the “prepared sample” is known to have a high moisture content, as in the case of certain forms of bleached lac (4.5), it shall be dried to a moisture content of 6 % before the test specimens are taken. The lac resin shall be dried by placing it in a thin layer in a flat-bottom dish (loosely covered to prevent dust contamination) and exposing it to the atmosphere at room temperature for 24 h and then desiccating it over anhydrous calcium chloride. The partially dried lac resin shall be kept in a clean, dry, airtight container, and shall be

⁷ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Anal. Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

thoroughly mixed by rolling and tumbling in the container before the specimens are taken for analysis.

INSOLUBLE MATTER

8. Test Method A—For Orange Shellac, Button Lac, Garnet Lac, and Regular Bleached

8.1 Apparatus:

8.1.1 *Condenser*—A four-bulb Allihn condenser of the dimensions and design shown in Fig. 1.

8.1.2 *Siphon Tube*—A Knoefler siphon tube of the dimensions shown in Fig. 1.

8.1.3 *Filter Tube*—A carbon filter tube of the dimensions shown in Fig. 1.

8.1.4 *Flask*—A borosilicate glass Erlenmeyer flask 176 ± 3 mm in height and 48 ± 1.5 mm in inside diameter at the top.

8.1.5 *Flask Support*—A suitable ring support with an iron clamp and a Nichrome or iron wire gauze square without an asbestos center.

8.1.6 *Extraction Thimble*—Extraction thimble 26 ± 1 mm in diameter and 60 ± 1 mm in height.

8.1.7 *Water Bath*—A metal container with cover of the size and design shown in Fig. 2. The container and cover shall be made of 26-oz copper sheet. The cover shall have a flanged hole 57 ± 1 mm in diameter for a 200-mL beaker and also a hole 35 ± 1 mm in diameter for the carbon filter tube. Directly below this hole in the bottom of the container shall be a flanged hole 25 ± 1 mm in diameter.

8.1.8 *Heating Device*—An electric hot plate or bunsen burner equipped with a draft shield.

8.1.9 *Weighing Bottle*—A glass-stoppered weighing bottle of the dimensions shown in Fig. 1.

8.2 *Solvent*—Specially denatured 95 % (190 proof) ethyl alcohol conforming to Formula No. 1 or No. 3A of the Alcohol, Tobacco and Firearms Division of Internal Revenue Service, U.S. Treasury Department.

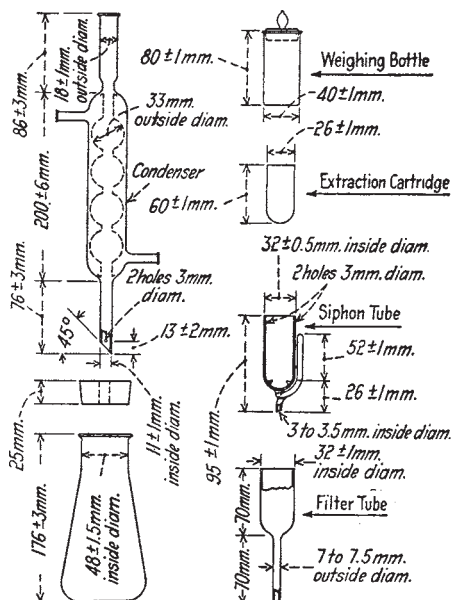


FIG. 1 Extraction Apparatus for Insoluble Matter, Test Method A

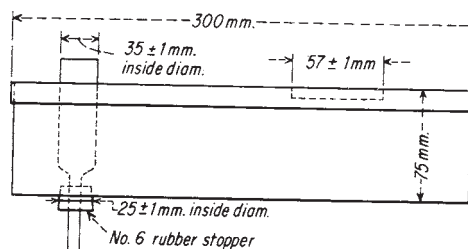


FIG. 2 Hot-Water Bath for Insoluble Matter, Test Method A

NOTE 1—Precaution: The reagents and samples used in these methods may, under some conditions, be hazardous. Refer to the supplier's Material Safety Data Sheet for specific handling and safety precautions. Safe laboratory handling procedures and all applicable OSHA regulations are to be followed.

8.3 Preparation of Extraction Thimble:

8.3.1 Pass the stem of the condenser through a hole cut in the center of a cork stopper of such size that it will tightly fit the flask. Adjust the cork on the stem so that the bottom of the cork is just above the holes in the stem. Place an extraction thimble (use new thimbles only) in the siphon tube. Suspend the siphon tube from the stem of the condenser by passing a piece of copper wire through the holes in the stem and fastening the ends of the wire through the holes in the siphon tube. The wire shall be sufficiently long to leave about 6-mm space between the tip of the condenser and the top of the siphon tube.

8.3.2 Place 125 mL of ethyl alcohol in the flask and attach the flask to the condenser by means of the cork stopper. Place the flask on an electric hot plate or a flask support. Run a steady stream of cold water through the condenser. Adjust the flame of the burner or the hot plate setting so as to give a cycle of filling and emptying of the siphon tube every 2 min, and extract the thimble for 30 min. Remove the extraction thimble from the siphon tube and allow to drain and air-dry for several minutes.

8.3.3 Place the thimble in a weighing bottle and dry in an oven for 2 h at $105 \pm 2^\circ\text{C}$. Remove and stopper the weighing bottle and cool in a desiccator. Weigh the bottle and thimble lifting the stopper momentarily before weighing. Continue drying and weighing as before after each hour of drying until the loss in weight between successive weighings does not exceed 2 mg.

8.3.4 A number of thimbles may be extracted and kept in weighing bottles or a desiccator until needed.

8.4 Procedure:

8.4.1 Weigh to 1 mg 5 ± 0.2 g of the mixed sample (Section 7) and place in a 200-mL tall-form beaker. Add 125 mL of ethyl alcohol to the beaker and place it in the hot-water bath (Fig. 2), which has been previously heated to not less than 90°C . Maintain the bath at this temperature, or above, during the solution and filtration of the sample. Boil the solution for 30 min, keeping the volume of alcohol constant to ensure complete solution of the lac resin.

8.4.2 Place an extracted, weighed extraction thimble (8.3) in the carbon filter tube (Fig. 2). Wet the thimble with hot alcohol and decant the boiling solution into the warm thimble until the beaker is nearly empty. Wash the remaining solution and

insoluble matter into the thimble with a stream of hot alcohol from a wash bottle using a "policeman" if necessary. Finally, wash the thimble from the top down. The transfer of the insoluble matter from the beaker and the washing down of the thimble will require at least 75 mL of hot alcohol.

8.4.3 Transfer the thimble with the insoluble matter to the siphon tube of the extraction apparatus (Fig. 1). Place 125 mL of alcohol in the flask and attach the condenser. Adjust the heating device so that a complete filling and emptying of the siphon tube with hot alcohol requires 2 min or 30 cycles per hour (Note 2), and extract for exactly 1 h (Note 3). Remove the thimble, and allow it to drain in an upright position.

NOTE 2—Occasionally, lac resins are encountered that will not yield the required 30 siphons per hour due to slow filtration. In these instances the extraction shall be continued until 30 cycles have occurred and the determination reported as abnormal or slow filtering.

NOTE 3—During the 1-h extraction, all the remaining soluble matter should be extracted by the hot alcohol, leaving only the insoluble matter in the thimble.

8.4.4 Place the thimble in the weighed weighing bottle and dry in an oven at $105 \pm 2^\circ\text{C}$ for 2 h. Remove the weighing bottle from the oven, insert its stopper, cool in a desiccator, and weigh, lifting the stopper momentarily to break the vacuum before weighing. Continue drying and weighing as before after each hour of heating until the loss in weight between successive weighings does not exceed 2 mg.

8.5 Calculation—Calculate the percent of matter insoluble in hot alcohol as follows:

$$\text{Insoluble matter, \%} = [R/S(1 - M)] \times 100 \quad (1)$$

where:

R = insoluble matter obtained, g,

S = sample used, g, and

M = volatile matter (moisture) content of the sample, expressed as a decimal fraction.

9. Test Method B—For All Grades of Lac Resins Including Refined Bleached Lac⁸

9.1 Apparatus (Fig. 3):

9.1.1 *Crucible*—A borosilicate glass Gooch crucible having a capacity of 30 mL with a fritted-glass filter disk having a medium porosity.

9.1.2 *Filter Tube*—A carbon filter tube made of borosilicate to fit the crucible.

9.1.3 *Heating Coil*—A heating coil made of 3-mm diameter copper tubing of such size and shape that the filter crucible and the large part of the carbon tube will fit snugly within it. The outside shall be insulated with sheet asbestos paper.

9.2 *Solvent*—Either of the following materials may be used as the solvent for the lac resin:

9.2.1 *2-Ethoxyethanol*, conforming to the requirements of Specification D 331.

9.2.2 *Normal Butyl Alcohol (n-Butanol)*, conforming to the requirements of Specification D 304.

9.3 Preparation of Filtering Unit:

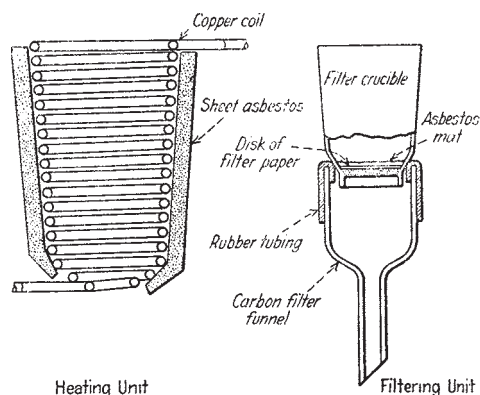


FIG. 3 Apparatus for Insoluble Matter, Test Method B

9.3.1 Cut a disk of rapid, ashless filter paper to fit inside the crucible and place it on top of the glass filter. Introduce upon the filter paper, in the customary manner, an asbestos mat approximately 3 mm in thickness. Dry the crucible at $105 \pm 2^\circ\text{C}$; then cool in a desiccator to constant weight. Weigh the prepared crucible and place it within the carbon tube, using thin rubber tubing to form an airtight connection. Place the combined filtering unit within the heating unit, attach to a suction flask, and pass a current of steam through the coil.

9.4 Procedure:

9.4.1 Weigh to $1 \text{ mg } 5 \pm 0.2$ of the mixed sample (Section 7 and Note 4) into a 200-mL beaker. Add 75 mL of the solvent (9.29.2) and bring the solution to boiling on an electric hot plate. Keep the solution boiling slowly for 5 min to ensure complete solution.

NOTE 4—For refined bleached shellac and other shellacs having a very low insoluble matter content take a 10 to 20-g specimen using proportionally more solvent.

9.4.2 Pour about 10 mL of the boiling solvent from a wash bottle into the heated crucible. Gently apply suction and immediately pour the boiling solution into the crucible so as to retain as much as possible of the insoluble residue in the beaker. Wash the insoluble residue successively with three 20-mL portions of the solvent, boiling the solution on the electric hot plate for about 1 min before each filtration.

9.4.3 Transfer the residue from the beaker to the crucible with a stream of the boiling solvent from a wash bottle, using a policeman when necessary. Wash down the inner walls of the crucible with the boiling solvent. The total volume of the solvent used will be approximately 175 mL. It is advisable to keep the crucible covered with a small watch glass at all times, except when actually transferring the solution from the beaker to crucible, or when washing down the inside walls of the crucible to maintain a higher temperature within the crucible. Allow the crucible to remain inside the heating coil with the suction on for a few minutes, so as to suck it as dry as possible.

NOTE 5—The insoluble matter can be easily removed together with the asbestos mat and filler paper. The crucible may be used several times without further cleaning. Additional cleaning when necessary is easily accomplished by immersing the crucible in a hot mixture of sulfuric and nitric acids for a few minutes.

⁸ Hartman, C. C., "Determination of Insoluble Matter in Shellac," *Journal of Research*, Nat. Bureau Standards, Vol 7, No. 6, 1931, p. 1105.

9.4.4 Remove the crucible and wash the outside with boiling solvent. Dry in an oven at $105 \pm 2^\circ\text{C}$ for 2 h, cool in a desiccator, and weigh.

9.5 *Calculation*—Calculate the percent of matter insoluble in the hot solvent as follows:

$$\text{Insoluble, \%} = [R/S(1 - M)] \times 100 \quad (2)$$

where:

R = insoluble matter obtained, g,

S = sample used, g, and

M = volatile matter (moisture) content of the sample, expressed as a decimal fraction.

10. Iodine Value

10.1 Reagents:

10.1.1 *Potassium Iodide Solution* (100 g/L)—Dissolve 10 g of iodate-free potassium iodide (KI) in water and dilute to 100 mL.

10.1.2 *Reference Standard Shellac*—Pure shellac of known iodine value.⁹

10.1.3 *Sodium Thiosulfate, Standard Solution*—(0.1 N)—Prepare and standardize in accordance with Section 5 of Method D 1959.

10.1.4 *Starch Indicator Solution*—Prepare in accordance with Test Method D 1959.

10.1.5 *Wijs Solution*—Prepare in accordance with Test Method D 1959. The Wijs solution should be tested against an orange shellac the iodine value of which is accurately known. The iodine value thus obtained should be within ± 0.5 of the known iodine value.

10.2 Procedure:

10.2.1 Weigh to 0.1 mg about 0.2 g of the mixed sample (Section 7 and Note 6 and Note 7) into a 250-mL dry, clear glass bottle having a ground-glass stopper. Add 20 mL of glacial acetic acid and heat at 65 to 70°C on a hot-water bath, gently swirling the contents of the bottle occasionally, until solution is complete, except for the wax. This should not require more than 15 min. Add 10 mL of chloroform and cool at 21.5 to 22.5°C for $\frac{1}{2}$ h in an insulated, thermostatically controlled water bath.

NOTE 6—In the case of grossly adulterated samples, a smaller quantity (0.15 or 0.1 g) should be used instead of 0.2 g of the material in order that the excess of iodine monochloride may not be too greatly reduced, since the excess of halogen is one of the factors in determining the amount of absorption. In case less than 25 mL of the $\text{Na}_2\text{S}_2\text{O}_3$ solution is required, another test should be made using a smaller quantity of the lac resin.

NOTE 7—In weighing lac resin some difficulty is at times experienced on account of its electrical resistance properties. In very dry weather it may be found that the necessary handling to prepare it for weighing has electrostatically charged it, and that it may be necessary to leave it in the balance pan at rest for a few minutes before determining the weight.

NOTE 8—If a number of samples are being run, allow at least 5 min between the additions of the Wijs solution.

⁹The sole source of supply of Standard samples of pure orange shellac, rosin-free of accurately known iodine known to the committee at this time is the U. S. Shellac Importers Assn., Inc., 425 Park Ave., New York, NY 10022. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

10.2.2 Add 20 mL of the Wijs solution to the bottle, using a pipet. Replace the stopper and half immerse the bottle in the water bath held from 21.5 to 22.5°C for exactly 1 h (Note 8). Gently swirl the contents of the bottle occasionally during the hour. Remove the bottle from the bath and add 10 mL of the KI solution washing into the bottle any Wijs solution on the stopper with the same.

10.2.3 Immediately titrate with the standard $\text{Na}_2\text{S}_2\text{O}_3$ solution, allowing the solution to run rapidly and swirling the contents of the bottle vigorously and continuously until the solution becomes a straw color. Add 5 mL of the starch indicator solution and continue the titration dropwise until the blue color just disappears. The end point is sharp and any blue color returning after 30 s should be disregarded.

10.2.4 *Blank*—Run a blank determination on the reagents at the same time and through the entire procedure. The blank is necessary because of the effect of temperature changes on volume and possible loss in strength of the Wijs solution.

10.2.5 *Reference Standard*—Run a determination on a sample of pure shellac of known iodine value (10.1.2) with each set of test specimens.

10.3 *Calculation*—Calculate the iodine value as follows:

$$\text{Iodine value} = [(B - V)N \times 12.69]/S \quad (3)$$

where:

V = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the specimen, mL,

B = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the blank, mL,

N = normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution, and

S = sample used, g.

PURITY

11. Qualitative Test for Rosin

11.1 Reagents:

11.1.1 *Acetic Acid (Glacial)*.

11.1.2 *Ethyl Alcohol (Absolute)*.

11.1.3 *Halphen-Hicks Reagent*¹⁰—Prepare the following two solutions:

11.1.3.1 *Solution A*—One part by volume of phenol dissolved in 2 parts by volume of carbon tetrachloride.

11.1.3.2 *Solution B*—One part by volume of bromine dissolved in 4 parts by volume of carbon tetrachloride.

11.2 Procedure:

11.2.1 Weigh 2 ± 0.1 g of the mixed sample (Section 7) into a 2-L Florence flask and dissolve in 20 mL of either absolute ethyl alcohol or glacial acetic acid, warming on a steam bath if necessary. Cool, add 100 mL of petroleum ether, and mix thoroughly.

11.2.2 Add sufficient water to bring the petroleum ether layer into the neck of the flask when separated. Add the water portionwise, shaking the flask between additions to prevent coagulation of the precipitated lac resin. Allow to stand until the petroleum ether separates into a distinct layer.

¹⁰For more detailed information, reference may be made to Hicks, E. F., "New Color Reactions for Some of the Resins with Halphen's Reagent for Colophony," *Industrial and Engineering Chemistry*, Vol 3, 1911, p. 86.

11.2.3 Siphon off at least 50 mL of the petroleum ether solution, filter if cloudy, and heat on an electric hot plate or steam bath until the petroleum ether is completely removed.

11.2.4 Dissolve the residue in several millilitres of Solution A, and transfer 1 to 2 mL of the mixture to one of the cavities of a white porcelain spot plate. Immediately fill an adjacent cavity with Solution B. Cover the plate with an inverted watch glass and note the color developed in the Solution A mixture by the bromine vapors from Solution B. The development of a fugitive violet color, best observed on the flat portion of the spot plate, indicates the presence of rosin. A control sample containing rosin should be run simultaneously, as a guide in judging the color developed with the test specimen.

12. Qualitative Test for Copal

12.1 *Reagents:*

12.1.1 *Ethyl Alcohol, Denatured*—(See 8.2).

12.1.2 *Methyl Alcohol (99 %)*.

12.2 *Procedure:*

12.2.1 Weigh to 0.1 g about 15 g of the mixed sample (Section 7) into an Erlenmeyer flask. Add twice its weight of the denatured alcohol, stopper the flask and let stand with periodic shaking until the sample is in the solution. Filter the solution through a folded filter paper, discarding the first 5 mL of filtrate.

12.2.2 Transfer 10 mL of the filtrate to a large test tube (6 by 3/4 in.) (150 by 20 mm) and nearly fill the tube with methyl alcohol. Stopper the tube and mix its contents thoroughly. Immediate formation of a cloudiness or precipitate indicates the presence of copal. Lac resin free of copal should remain clear.

13. Estimation of Adulteration

13.1 Since the variation between the highest and lowest iodine values of a pure lac resin is not great, it is recommended that the following assumptions (Note 9) be made:

Type of Lac Resin	Assumed Iodine Value
Rosin-free and copal-free shellac, button lac, and garnet lac	15
Rosin-free and copal-free bleached lac	10
Rosin	228
Copal	130

13.2 Calculate the percent of adulteration as follows:

Percent of rosin in orange shellac, button lac, and garnet lac

$$= [(x - 15)/(228 - 15)] \times 100 \tag{4}$$

Percent of rosin in bleached lac = $[(x - 10)/(228 - 10)] \times 100$

Percent of copal in orange shellac, button lac, and garnet lac

$$= [(x - 15)/(130 - 15)] \times 100$$

Percent of copal in bleached lac = $[(x - 10)/(130 - 10)] \times 100$

where:

x = iodine value of the sample under test, determined in accordance with 10.2.

NOTE 9—The results obtained by assuming the values of 15 and 10 as the iodine value of orange and bleached shellac, respectively, and 228 as the iodine value of rosin may give a slightly lower percent rosin, under

some circumstances, than that which is actually present.

VOLATILE MATTER (MOISTURE)

14. Test Method A—For Orange Shellac, Button Lac, Garnet Lac, and Dry Bleached Lac

14.1 *Procedure:*

14.1.1 Weigh to 0.1 mg approximately 2 g of the mixed sample from the air-tight container (7.1.1 or 7.1.2) into a weighed, clean, dry, flat-bottom glass dish about 4 in. (100 mm) in diameter and provided with a ground-glass cover. Place the dish, with the cover removed, in a well-ventilated oven maintained at $40 \pm 1^\circ\text{C}$ for 6 h.

14.1.2 Transfer the dish and cover to a vacuum desiccator containing concentrated sulfuric acid (H_2SO_4 , sp gr 1.84). Immediately evacuate the desiccator and keep the specimen uncovered in the vacuum for 18 h. Release the vacuum, replace the cover on the dish, and weigh immediately.

14.2 *Calculation*—Calculate the percent volatile matter (moisture) in the sample as follows:

$$\text{Volatile matter (moisture), \%} = [1 - (S_2/S_1)] \times 100 \tag{5}$$

where:

S_1 = sample used, g, and

S_2 = dried specimen, g.

15. Test Method B—For Bleached Lac in Form of Hanks, Bars, and Crushed Fresh-Ground

15.1 *Procedure:*

15.1.1 Thoroughly mix the “original observation” sample in its original airtight container by rolling and tumbling. Transfer 25 to 50 g to a mortar and crush as rapidly as possible into fine granules. Keep the mortar covered as well as possible to avoid any loss of moisture. Immediately transfer approximately 10 g of the crushed lac to a weighed, clean, dry, flat-bottom glass dish about 100 mm in diameter, provided with a ground-glass cover, and weigh to 0.1 mg. Record the weight of the lac taken for use as S_1 .

15.1.2 Place the dish and contents in a vacuum desiccator containing H_2SO_4 (sp gr 1.84). Remove the cover from the dish and immediately evacuate the desiccator. Keep the dish in the vacuum at room temperature for 18 to 24 h. Replace the cover on the dish, remove from the desiccator, and weigh. Record the weight of the partially dried lac for use as S_2 .

15.1.3 Grind the partially dried lac resin until it entirely passes a No. 40 (425- μm) sieve. Thoroughly mix the sieved lac on a mixing sheet (7.1). Transfer approximately a 2-g specimen of it from the mixing sheet to a weighed, covered dish of the type described in 15.1.1, and weigh to 0.1 mg. Record the weight of the specimen taken for use as S_3 .

15.1.4 Heat the dish with cover removed in a well-ventilated oven at $40 \pm 1^\circ\text{C}$ for 18 h. Replace the cover on the dish, cool in a desiccator, and weigh. Record the weight of dried lac obtained for use as S_4 .

15.2 *Calculation*—Calculate the percent of volatile matter (moisture) in the original lac sample as follows:

$$\text{Volatile matter (moisture), \%} = [1 - (S_4 \times S_2)/(S_3 \times S_1)] \times 100 \tag{6}$$