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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Sampling and Testing Lac Resins[1]

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:

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-Ethoxyethanol[2]

D 1193 Specification for Reagent Water[3]

D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)[4]

D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method[5]

D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids[5]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[6]

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, 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

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[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.

[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.

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

[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 *Analar 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.

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.

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.

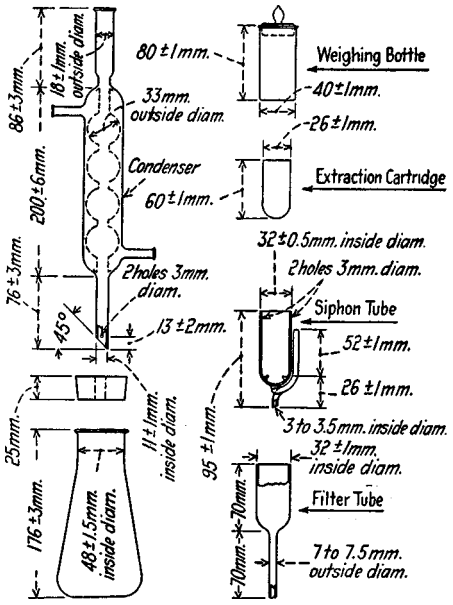

FIG. 1 Extraction Apparatus for Insoluble Matter, Test Method A

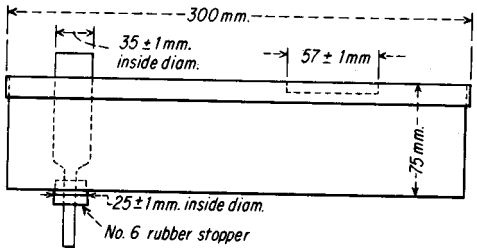

FIG. 2 Hot-Water Bath for Insoluble Matter, Test Method A

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 ± 2°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