Designation: D 1650 – 91

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St. Philadelphia, Pa 19103
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM
If not listed in the current combined index, will appear in the next edition.

Standard Test Methods of Sampling and Testing Shellac Varnish[1]

This standard is issued under the fixed designation D 1650; 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.

1. Scope

1.1 These test methods cover procedures for the sampling and testing of orange shellac and bleached lac varnishes.

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

	Sections
Sampling	5
General Requirements for Test Methods:	
Purity of Reagents	6
Preparation of Sample	7
Color	8 to 11
Drying Time	12 to 14
Nonvolatile Matter	15 to 17
Iodine Value	18 and 19
Insoluble Matter	20 to 23
Wax	24 to 27
Ash	28 and 29
Purity	30 to 34
Acid Value	35 to 37
Saponification Value	38 to 40

1.3 *This standard does not purport to address the safety problems 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.*

2. Referenced Documents

2.1 *ASTM Standards:*
D 29 Test Methods of Sampling and Testing Lac Resins[2]
D 1193 Specification for Reagent Water[3]

3. Terminology

3.1 *Description of Terms Specific to This Standard:*

3.1.1 *acid value*—the number of milligrams of potassium hydroxide required to neutralize 1 g of the nonvolatile portion of the lac varnish.

3.1.2 *lot*—For purpose of sampling, a lot shall consist of the entire amount of varnish manufactured as one batch and offered for delivery at one time.

3.1.3 *saponification value*—a measure of the alkali reactive groups in lac resins, it is expressed as the number of milligrams of potassium hydroxide that react with 1 g of the nonvolatile portion of the lac varnish.

4. Significance and Use

4.1 These test methods compiles the common measurements and procedures for characterizing shellac.

4.2 All shellac varnishes should be tested by the procedures contained in these test methods.

SAMPLING

5. Procedure

5.1 A single container shall be taken at random from each lot as representative of the whole.

5.2 In case the varnish is packaged in cans or drums of 5 gal (18.9 L) or more capacity, thoroughly mix the contents of the container and take a number of small samples from the top, bottom, and intermediate points by means of sampling tube. Mix these small samples to form a composite sample and transfer not less than 1 qt (0.94 L) to a clean, dry, glass bottle, securely stopper with a new clean cork or well-fitting cover or cap, seal, distinctly label, and transmit to the laboratory for test.

5.3 When the varnish is packaged in containers of 1 gal (3.8 L) or less capacity, send the original unopened container to the laboratory for test. When this cannot be done, thoroughly mix the contents of the container and transfer not less than 1 qt to a clean, dry, glass bottle, securely stopper with a new clean cork or well-fitting cover or cap, seal, distinctly label, and transmit to the laboratory for test.

5.4 Take precautions to ensure that the sampling apparatus and the samples themselves are neither contaminated with nor altered by any material not representative of the lot being sampled.

Test Methods

GENERAL REQUIREMENTS

6. Purity of Reagents

6.1 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 Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] 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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type IV.

---

[1] These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

Current edition approved Feb. 22, 1991. Published April 1991. Originally published as D 1650 – 59. Last previous edition D 1650 – 76 (1981).

[2] *Annual Book of ASTM Standards*, Vol 06.02.

[3] *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

[4] "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

7. Preparation of Sample

7.1 Thoroughly mix the sample of varnish received for test by shaking the container immediately before the individual portions are removed for the various tests. Keep the unused portion in a tightly closed container (preferably glass) and in a cool, dark place.

COLOR

8. Apparatus

8.1 *Glass Tubes,* as prescribed in 19.1.1 of Test Methods D 29.

8.2 *Glass Plate,* as prescribed in 19.1.2 of Test Methods D 29.

9. Solvent

9.1 *Ethyl Alcohol, Denatured,* as prescribed in 8.2 of Test Methods D 29.

10. Color Comparison Standard

10.1 A shellac varnish mutually agreed upon by the purchaser and the seller for the color comparison. The varnish shall be stored in a tightly closed glass bottle and kept in a cool dark place.

11. Procedure

11.1 Weigh, to the nearest 0.1 g, approximately 35 g of the sample (Section 7) and the color comparison standard into separate stoppered flasks. Adjust both varnishes to the same nonvolatile matter content (preferably 33.3 %) by adding the proper amounts of the alcohol to the respective flasks. Thoroughly mix the contents of each flask by shaking and then allow the flasks to stand undisturbed for ½ h. Compare the color of the sample with that of the comparison standard in accordance with the procedure described in 19.4.2 or 19.4.3 of Test Methods D 29, as agreed upon by the purchaser and the seller.

DRYING TIME

12. Apparatus

12.1 *Film Applicator*—The applicator shall be made of smoothly finished metal, preferably corrosion-resistant, and accurately constructed. It shall spread a film of uniform width and shall have such clearance that the thickness of the resulting wet film of coating applied will be 1.5 mils (38 μm).

12.2 *Glass Plate,* as prescribed in 19.1 of Test Methods D 29.

13. Solvent

13.1 *Ethyl Alcohol, Denatured,* as prescribed in 8.2 of Test Methods D 29.

14. Procedure

14.1 Varnishes having more than 36 % nonvolatile matter shall be adjusted with the alcohol to a nonvolatile content of 35 ± 1 %. Varnishes having nonvolatile contents of less than 36 % shall be tested as received.

14.2 Place the glass plate in a horizontal position and the film applicator, having a clearance to give a wet-film thickness of 1.5 mils (38 μm) unless otherwise specified, at one end of the plate. Deposit a small quantity of the sample (Section 7) on the glass plate immediately in front of the applicator. Spread the varnish the length of the panel by means of the applicator. Allow the test panel to remain in a horizontal position and dry in a well-ventilated room or chamber free from drafts and dust and in diffused light (not in direct sunlight). The temperature of the air within the room or chamber shall be between 21 and 32°C. If the time of drying is not within the specified limits of the product specification, repeat the test in a room or chamber maintained at a temperature of 223 ± 2°C and 50 ± 5 % relative humidity.

14.3 Test the film for set-to-touch and dry-hard times at points not less than 10 mm (½ in.) from its edges in accordance with 14.4 and 14.5.

14.4 *Set-to-Touch Time*—Lightly touch the test film with the tip of the finger and immediately place the finger tip against a piece of clean clear glass. Observe if any of the varnish is transferred to the glass. For the purpose of this test, the pressure of the finger shall not be greater than that required to transfer a spot of the varnish from ⅛ to 3/16 in. (3.2 to 4.8 mm) in cross section. The film shall be considered set-to-touch when it still shows a tacky condition but none of it adheres to the finger.

14.5 *Dry-Hard Time*—With one end of the thumb resting on the test film and the forefinger supporting the test panel exert a maximum downward pressure (without twisting) of the thumb on the film. Lightly polish the contacted area with a soft cloth. The film shall be considered dry-hard when any mark left by the thumb is completely removed by the polishing operation.

NONVOLATILE MATTER

15. Materials

15.1 *Ethyl Alcohol, Denatured,* as prescribed in 8.2 of Test Methods D 29.

15.2 *Sand*—Sand that has been digested in hot concentrated hydrochloric acid (HCl, sp gr 1.19) for 1 h, washed with water to remove all acid and soluble impurities, ignited, cooled in a desiccator, and stored in a clean tightly closed container.

16. Procedure

16.1 Place a portion of the sample (Section 7) in a stoppered flask or weighing pipet and weigh to the nearest 0.1 mg. Transfer between 1 and 1.5 g of the sample to a weighed flatbottom glass or metal dish (petri dish or friction-top can plug) 75 to 80 mm in diameter, containing approximately 10 g of the sand, and a small glass rod. Weigh the container again and by difference obtain the exact weight of sample transferred to the dish.

16.2 Add 1 to 2 ml of the alcohol to the dish and thoroughly mix its contents with the rod. Heat the dish and its contents in a well-ventilated convection type oven maintained at 105 ± 2°C for 1 h. Transfer the dish to a desiccator to cool and weigh.

17. Calculation

17.1 Calculate the percentage of nonvolatile matter in the sample as follows:

$$\text{Nonvolatile matter, \%} = [(x - y)/S] \times 100$$