Designation: D4733 – 13

Standard Test Methods for Solventless Electrical Insulating Varnishes[1]

This standard is issued under the fixed designation D4733; 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 solventless varnishes used primarily as electrical, mechanical, and environmental protection for electrical equipment.

1.2 These test methods are used on solventless varnishes that are applied by dipping (at atmospheric pressure conditions or under vacuum or other certain pressure), spraying, or brushing for the purpose of impregnating or sealing electrical equipment. The following test methods are included:

1.3 The values stated in inch-pound units are to be regarded as the standard. The values in parentheses are for information only.

1.4 *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 8.3, 15.5.1, and 15.5.2.

NOTE 1—This test method is related to IEC 60455. Since both methods contain multiple test procedures, many procedures are technically equivalent while others differ significantly.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D115 Test Methods for Testing Solvent Containing Varnishes Used for Electrical Insulation

D149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies

D150 Test Methods for AC Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulation

D168 Test Method for Coke Residue of Creosote (Withdrawn 2006)[3]

D374 Test Methods for Thickness of Solid Electrical Insulation (Withdrawn 2013)[3]

D923 Practices for Sampling Electrical Insulating Liquids

D1475 Test Method For Density of Liquid Coatings, Inks, and Related Products

D1711 Terminology Relating to Electrical Insulation

D2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) Viscometer

D2519 Test Method for Bond Strength of Electrical Insulating Varnishes by the Helical Coil Test

D3056 Test Method for Gel Time of Solventless Varnishes

D3278 Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus

D3312 Test Method for Percent Reactive Monomer in Solventless Varnishes

D3487 Specification for Mineral Insulating Oil Used in Electrical Apparatus

D3636 Practice for Sampling and Judging Quality of Solid Electrical Insulating Materials

D6054 Practice for Conditioning Electrical Insulating Materials for Testing (Withdrawn 2012)[3]

E176 Terminology of Fire Standards

[1] These test methods are under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and is the direct responsibility of Subcommittee D09.01 on Electrical Insulating Varnishes, Powders and Encapsulating Compounds.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

***A Summary of Changes section appears at the end of this standard**

2.2 *IEC Standards*

IEC 60455 Resin Based Reactive Compunds Used for Electrical Insulation

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in these test methods, refer to Terminology D1711. For definitions of terms associated with fire issues, refer to Terminology E176.

3.1.2 *gel time, n—of solventless varnish*, the time required at a specified temperature for a solventless varnish to be transformed from a liquid state to a gel as measured with a suitable gel time apparatus.

3.1.3 *thixotropy, n*—the property of a material to thin upon isothermal agitation and to thicken upon subsequent rest.

3.1.4 *varnish, electrical insulating, n*—a liquid resin system that is applied to and cured on electrical components providing electrical, mechanical, and environmental protection.

3.1.4.1 *Discussion*—There are two types of electrical insulating varnish—solvent-containing and solventless. The solvent-containing varnish is solution, dispersion, or emulsion of a polymer or a mixture of polymers in a volatile, nonreactable liquid. The solventless type is a liquid resin system free of volatile, nonreactable solvents.

4. Significance and Use

4.1 The test methods referenced in these test methods are useful for control purposes during the manufacture and use of solventless varnishes.

5. Sampling

5.1 Accurate sampling, whether of the complete contents or only parts thereof, is extremely important from the standpoint of evaluating the quality of the product sampled. In most cases, the detection of contaminants that are not ordinarily dispersed uniformly through the liquid being sampled such as water or solid particles, necessitates taking samples at specific locations where the contaminants are likely to be found. For a liquid having a specific gravity less than one, water and other impurities are most likely to be found on the bottom; whereas in the case of liquids having a specific gravity greater than one, these impurities are most likely to be found on the surface.

5.2 *Sampling Fluids Exhibiting Newtonian Viscosity:*

5.2.1 Sample solventless varnishes having an approximate viscosity of 25 cP (0.025 Pa·s) or less at 100°F (37.8°C) using the sampling device shown in Fig. 1 of Test Method D923. However, if the temperature of the material is below room temperature or if the humidity is above 75 % relative humidity, sample using an aspirator-type sampling device as shown in Fig. 2 of Test Method D923.

5.2.2 Sample solventless varnishes having a viscosity from 25 to 650 cP (0.025 to 0.65 Pa·s) at 100°F (37.8°C) and where contact with the atmosphere must be avoided, use a pressure-type sampling device as shown in Fig. 3 of Test Method D923.

5.2.3 Sample solventless varnishes having a viscosity above 650 cP at 100°F at 73 ± 10°F (23 ± 5°C). Open the container and mix slowly so as not to entrap air. When the material is homogeneous, take samples from two or three places on the surface and mix together.

5.3 *Sampling Fluids Exhibiting Non-Newtonian Viscosity:*

5.3.1 Sample solventless varnishes exhibiting non-Newtonian viscosity in accordance with 5.2.3.

5.4 Use sample containers and sampling procedures in accordance with Test Method D923.

5.5 When a sampling method is needed to determine whether a lot meets the requirements of a material specification, use Practice D3636 as a guide.

6. Conditioning

6.1 Condition test specimens at standard laboratory condition as specified in Practice D6054.

6.2 Deaerate specimens to be used for property evaluation before curing using the vacuum technique recommended by the varnish manufacturer.

TEST METHODS

7. Density

7.1 *Scope*—This test method covers the measurement of the density using a pycnometer. It is particularly applicable where the fluid has too high a viscosity for other methods of testing density. In this test method the density will be expressed as pounds per gallon (grams per millilitre) at 70 to 77°F (20 to 25°C).

7.2 *Procedure*—Test for density in accordance with Test Method D1475.

7.3 *Report*—Report the following information:

7.3.1 Identification of the test sample,

7.3.2 The test temperature to the nearest 0.2°F (0.1°C),

7.3.3 Density to the nearest 0.01 lb/gal (0.001 g/mL), and

7.3.4 The mean, the range, and the number of replicate determinations.

8. Flash Point

8.1 *Scope*—This test method covers the determination of the flash point of solventless varnishes. There are two types of materials involved:

8.1.1 Those varnishes having a flash point above 230°F (110°C), and

8.1.2 Those varnishes having a flash point below 230°F (110°C).

8.2 *Summary of Test Method*—A specified amount of sample is added to the instrument. The temperature of the material is increased at a constant rate until a small flame ignites the vapor of the specimen in the tester and propagates a flame across its surface. The temperature at ignition is taken as the flash point.

8.3 *This standard is used to measure and describe the response of materials, products, or assemblies to heat and flame under controlled conditions, but does not by itself incorporate all factors required for fire hazard or fire risk assessment of the materials, products or assemblies under actual fire conditions.*

8.4 *Fire testing is inherently hazardous. Adequate safeguards for personnel and property shall be employed in conducting these tests.*

8.5 *Procedure:*

8.5.1 Test the materials of 8.1.1 in accordance with Test Methods D93.

8.5.2 Test the materials of 8.1.2 in accordance with Test Method D3278.

8.6 *Report*—Report information in accordance with the report section of the test method used.

9. Gel Time

9.1 *Scope*—This test method covers the determination of gel time of a solventless varnish mixed with a reactor or catalyst and exposed to elevated temperatures.

9.2 *Significance and Use*—Knowledge of gel time is important for determining shelf life, batch uniformity, and processing characteristics.

9.3 *Procedure*—Test for gel time in accordance with Test Method D3056.

9.4 *Report*—Report the following information:

9.4.1 Identification of the material,

9.4.2 The reactor or catalyst and quantity used shall be described if the reactor or catalyst is supplied in a separate package,

9.4.3 Gel time to the nearest 0.1 min,

9.4.4 Temperature of the bath, and

9.4.5 Liquid used in the bath.

10. Monomer Content

10.1 *Scope*—This test method covers the determination of the percent monomer having a vapor pressure exceeding 0.1 Torr (13 Pa) at 77°F (25°C).

10.2 *Significance and Use*—The percent of reactive monomer in a solventless varnish will affect the viscosity, handling, and processing properties of the uncured resin. The amount of reactive monomer also has the potential to affect the physical and electrical properties of the cured resin.

10.3 *Procedure*—Test for the monomer content in accordance with Test Method D3312.

10.4 *Report*—Report the following information:

10.4.1 Identification of the solventless varnish, and

10.4.2 Percent reactive monomer.

11. Rheological Properties (Non-Newtonian)

11.1 *Scope*—This test method covers the determination of the rheological properties of solventless varnishes that are non-newtonian in character.

11.2 *Significance and Use*—This test method is used to test solventless varnishes that are thixotropic in nature and require specific treatment of the sample in order to determine this property.

11.3 *Procedure*—Test for thixotropy in accordance with Test Method D2196, Method B or C.

11.4 *Report*—Report information as specified in Test Method D2196.

12. Brookfield Viscosity

12.1 *Scope*—This test method covers the determination of viscosity for liquids having flow properties approaching those of the newtonian solutions.

12.2 *Apparatus:*

12.2.1 *Brookfield Viscometer.*

12.2.2 *Thermometer.*

12.2.3 *Water Bath.*

12.2.4 *Stirrer.*

12.3 *Procedure:*

12.3.1 Bring the specimen to a temperature of 77 ± 2°F (25 ± 1.0°C) using a water bath and stirrer if necessary. Allow the specimen to sit in the water bath at 77 ± 2°F until no bubbles are visible in the specimen.

12.3.2 Make all measurements with the Brookfield viscometer at 77 ± 2°F (25 ± 1.0°C).

12.4 *Report*—Report information as specified in Test Method D2196.

12.5 *Precision and Bias:*

12.5.1 *Precision*—See Test Method D2196 for a statement of precision.

12.5.2 *Bias*—See Test Method D2196 for a statement of bias.

13. Bond Strength

13.1 *Scope*—This test method covers the determination of the bond strength of an electrical insulating varnish when applied to a helical coil.

13.2 *Significance and Use*—Values obtained by flexural test can provide information with regard to the bond strength of a particular varnish in combination with a particular wire when measured under the prescribed conditions.

13.3 *Procedure*—Perform the test in accordance with Test Method D2519.

13.4 *Report*—Report information in accordance with Test Method D2519.

13.5 *Precision and Bias:*

13.5.1 *Precision*—See Test Method D2519 for a statement of bias.

13.5.2 *Bias*—See Test Method D2519 for a statement of bias.

14. Coating Thickness

14.1 *Scope:*

14.1.1 This test method is used to determine the thickness of a coating obtained with a solventless varnish.

14.1.2 The shear history of the varnish immediately preceding the test has the potential to have an appreciable effect on the coating thickness obtained. This test method specifies the degree of shear of the sample and the rate of withdrawal of the specimen in order to arrive at a reproducible buildup on a test specimen.

14.2 *Apparatus:*

14.2.1 *Device,* capable of withdrawing a specimen at approximately 4 in./min (100 mm/min).

14.2.2 *Water Bath,* or conditioned room controlled to a temperature of 75 ± 2°F (24 ± 1°C).

14.2.3 *Thermometer,* with a range of 32 to 212°F (0 to 100°C).

14.2.4 *Test Panel,* consisting of a steel or aluminum strip 3 in. (75 mm) in width, 5 in. (125 mm) in length, and 0.005–0.032 in. (0.12–0.81 mm) in thickness.

14.2.4.1 The thickness is determined by measuring the thickness at 2 in. (50 mm) below the top and at 4 in. (100 mm) below the top. An average of these two is the thickness of the bare panel. Thickness measurements shall be made in accordance with Test Methods D374.

14.3 *Procedure:*

14.3.1 Bring the test specimen to a temperature of 75 ± 2°F (24 ± 1°C) and pour slowly into a suitable container. Do not trap air in the varnish.

14.3.2 Slowly lower the test panel into the varnish.

14.3.3 Place the container with the varnish and panel in a constant-temperature water bath or in a conditioned room held at 75 ± 2°F (24 ± 1°C). Allow it to stand in this condition without vibration for 60 to 65 min.

14.3.4 At the end of 60 min move the specimen under a dip coater that has been previously adjusted to withdraw the specimen at a rate of approximately 4 in./min (100 mm/min). Attach the specimen without disturbing the jar or panel.

14.3.5 After the panel has been raised out of the varnish, stop the dip coater and allow the panel to stand for 30 min. Move the container away from the specimen so that the specimen can hang free without detriment to the coating.

14.3.6 Place the specimen in an oven and bake in accordance with the manufacturer's instructions. Be careful not to vibrate the specimen in the early part of the baking.

14.4 *Report*—Report the following information:

14.4.1 Average thickness of the coated panel,

14.4.2 Average thickness of the uncoated panel,

14.4.3 The thickness of the coating on both sides of the panel is the difference between the coated and uncoated panel.

14.4.4 The coating thickness (the thickness of the coating obtained in 14.4.3 divided by 2).

14.5 *Precision and Bias:*

14.5.1 *Precision*—It is not practicable to specify the precision of this procedure because it has not been determined.

14.5.2 *Bias*—Since there is no acceptable reference material suitable for determining the bias of this test method, no statement of bias is being made.

15. Dielectric Strength

15.1 *Significance and Use:*

15.1.1 Dielectric strength of an insulating varnish is an important indication of its ability to withstand electric stress without failure. The value determined by this test is not a measure of dielectric strength expected in service, but is a numerical value which is acceptable for use for purchase by specification as an indication of quality, for comparison of different varnishes, and, to limited degree, for design work when coupled with experience.

15.1.2 See the Significance and Use section of Test Methods D149 for further information on this subject.

15.2 *Apparatus:*

15.2.1 Electrical apparatus is as described in Test Method D149.

15.2.2 *Electrodes* are opposing cylinders 50 mm (2 in.) in diameter and 25 mm (1 in.) in length, with the edges rounded to a radius of 6.4 mm (¼ in.). Use clean insulating oil meeting the requirements of Specification D3487, Type I or Type II as the surrounding medium for test.

15.3 *Test Specimens:*

15.3.1 Cut five specimens, 100 by 100 by 3 mm (4 by 4 by ⅛ in.) from cast slabs. Cast the slabs in the vertical position in any suitable mold which will result in castings of thickness which can be controlled within ±5 %. The mold shall consist of two highly polished plates, 9.5 mm (⅜ in.) thick, coated with a suitable release agent and spaced to the desired specimen thickness with a U-shaped spacer of any suitable material to which the varnish will not adhere. Clamp the mold assembly with sufficient pressure to prevent leakage of the compound. Cure specimens in accordance with manufacturer's instructions.

15.4 *Conditioning:*

15.4.1 Condition the specimens in accordance with Procedure A of Practice D6054.

15.5 *Procedure:*

15.5.1 **Caution:***It is possible that lethal voltages will be present during this test. It is essential that the test apparatus, and all associated equipment potentially electrically connected to it, be properly designed and installed for safe operation. Solidly ground all electrically conductive parts that any person might come in contact with during the test. Provide means for use at the completion of any test to ground any parts which: were at high voltage during the test; will potentially have acquired an induced charge during the test; will potentially retain a charge even after disconnection of the voltage source. Thoroughly instruct all operators in the proper way to conduct tests safely. When making high voltage tests, particularly in compressed gas or in oil, it is possible that the energy released at breakdown will be sufficient to result in fire, explosion, or rupture of the test chamber. Design test equipment, test chambers, and test specimens so as to minimize the possibility of such occurrences and to eliminate the possibility of personal injury.*

15.5.2 **Caution:***Ozone is a physiologically hazardous gas at elevated concentrations. The exposure limits are set by governmental agencies and are usually based upon recommendations made by the American Conference of Governmental Industrial Hygienists.*[4] Ozone is likely to be present whenever voltages exist which are sufficient to cause partial, or complete, discharges in air or other atmospheres that contain oxygen. Ozone has a distinctive odor which is initially discernible at

[4] American Conference of Governmental Industrial Hygienists, Inc. (ACGIH), 1330 Kemper Meadow Dr., Cincinnati, OH 45240, http://www.acgih.org.

low concentrations but sustained inhalation of ozone can cause temporary loss of sensitivity to the scent of ozone. Because of this it is important to measure the concentration of ozone in the atmosphere, using commercially available monitoring devices, whenever the odor of ozone is persistently present or when ozone generating conditions continue. Use appropriate means, such as exhaust vents, to reduce ozone concentrations to acceptable levels in working areas.

15.5.3 Measure the thickness of each specimen at the center by the dead-weight dial micrometer, Method C of Test Methods D374.

15.5.4 Place specimen between the electrodes and add surrounding medium, if necessary. Raise the voltage at the rate of 500 v/s until failure.

15.5.5 Calculate the dielectric strength in v/mil (kV/mm) for each specimen by dividing the breakdown voltage by the specimen thickness.

15.6 *Report:*

15.6.1 Report the following information:

15.6.1.1 Average thickness of five specimens,

15.6.1.2 Individual breakdown voltages, and

15.6.1.3 Average dielectric strength

15.7 *Precision and Bias:*

15.7.1 *Precision*—This test method has been in use for many years, but no statement of precision has been made and no activity is planned to develop such a statement.

15.7.2 *Bias*—A statement of bias is not possible due to the lack of a standard reference material.

16. Dielectric Strength (Using Metal Panels)

16.1 Follow the procedure for determining dielectic strength of dried varnish film in accordance with Test Methods D115, with the following exceptions:

16.1.1 Aluminum or steel panels, 3 in. by 6 in. by 0.005 to 0.032 in., (7.6 cm by 15.2 cm by 0.013 to 0.08 cm) are acceptable substitutes for copper.

16.1.2 Do not adjust the varnish.

16.1.3 Dip in varnish only once.

16.2 Report information for dielectric breakdown of dried varnish film in accordance with Test Methods D115.

17. Permittivity and Dissipation Factor

17.1 *Procedure*—Test for permittivity and dissipation factor in accordance with Test Methods D150. Cast a slab of the material ⅛ in. (3 mm) in thickness and test a specimen 2 in. (50 mm) in diameter. Use a two-terminal micrometer electrode system capable of heating the electrodes. Select test temperature, voltage, and frequency based upon the application of the varnish.

17.2 *Report*—Report information in accordance with Test Methods D150.

17.3 *Precision and Bias:*

17.3.1 *Precision*—This test method has been in use for many years, but no statement of precision has been made and no activity is planned to develop such a statement.

17.3.2 *Bias*—A statement of bias is not possible due to the lack of a standard reference material.

18. Build

18.1 Follow the copper strip procedure in accordance with the section entitled “Build” of Test Methods D115, except that aluminum or steel panels, 3 in. by 6 in. by 0.005 to 0.032 in. (7.6 cm by 15.2 cm by 0.013 to 0.08 cm) are acceptable substitutes for copper strips.

18.2 Report information in accordance with the section entitled “Build” of Test Methods D115.

19. Keywords

19.1 bond strength; coating thickness; density; dielectric strength; dissipation factor; flash point; gel time; monomer content; permittivity; solventless varnishes; varnishes; viscosity

SUMMARY OF CHANGES

Committee D09 has identified the location of selected changes to these test methods since the last issue, D4733–03 (2009), that may impact the use of these test methods. (Approved April 1, 2013)

(1) Terminology E176 added to Referenced Documents section.

(2) Correct fire caveats added to 8.3 and 8.4.

(3) Eliminated non-mandatory language throughout.