Designation: D 1304 – 99

Standard Test Methods for Adhesives Relative to Their Use as Electrical Insulation[1]

This standard is issued under the fixed designation D 1304; 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 testing adhesives in liquid, highly viscous, solid, or set states, that are intended to be cured by electronic heating, or that are intended to provide electrical insulation, or that are intended for use in electrical apparatus.

1.2 The procedures appear in the following order:

(1) Procedure for Testing Adhesives Before Use:

	Section
Power Factor and Dielectric Constant of Liquid Adhesives	7
Direct-Current Conductivity	8
Extract Conductivity	9
Acidity and Alkalinity	10
pH Value	11

(2) Procedures for Testing Properties of Adhesives As Used:

	Section
Power Factor and Dielectric Constant of a Dried or Cured Adhesive Film	12
Dielectric Strength	13
Volume and Surface Resistivity	14
Arc Resistance	15

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*. For a specific hazard statement, see 8.2.

2. Referenced Documents

2.1 *ASTM Standards:*

D 115 Test Methods for Testing Solvent Containing Varnishes Used for Electrical Insulation[2]

D 150 Test Methods for AC Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulation[2]

D 202 Test Methods for Sampling and Testing Untreated Paper Used for Electrical Insulation[2]

D 257 Test Methods for DC Resistance or Conductance of Insulating Materials[2]

D 495 Test Method for High-Voltage, Low-Current, Dry Arc Resistance of Solid Electrical Insulation[2]

D 897 Test Method for Tensile Properties of Adhesive Bonds[3]

D 907 Terminology of Adhesives[3]

3. Terminology

3.1 *Definitions*—Many terms in these test methods are defined in Terminology D 907.

4. Significance and Use

4.1 Insulating materials are used to isolate components of an electrical system from each other and from ground, as well as to provide mechanical support for the components. For this purpose, it is generally desirable to have the insulation resistance as high as possible, consistent with acceptable mechanical, chemical, and heat-resisting properties. Since insulation resistance or conductance combines both volume and surface resistance or conductance, its measured value is most useful when the test specimen and electrodes have the same form as is required in actual use. Surface resistance or conductance changes rapidly with humidity, while volume resistance or conductance changes slowly although the final change may eventually be greater.

4.2 Resistivity or conductivity is used to predict, indirectly, the low-frequency dielectric breakdown and dissipation factor properties of some materials. Resistivity or conductivity is often used as an indirect measure of moisture content, degree of cure, mechanical continuity, and deterioration of various types. The usefulness of these indirect measurements is dependent on the degree of correlation established by supporting theoretical or experimental investigations. A decrease of surface resistance will result either in an increase of the dielectric breakdown voltage because the electric field intensity is reduced, or a decrease of the dielectric breakdown voltage because the area under stress is increased.

4.3 All the dielectric resistances or conductances depend on the length of time of electrification and on the value of applied voltage (in addition to the usual environmental variables). These must be known to make the measured value of resistance or conductance meaningful.

4.4 Volume resistivity or conductivity is used as an aid in designing an insulator for a specific application. The change of resistivity or conductivity with temperature and humidity may be great, and must be known when designing for operating

[1] These test methods are under the jurisdiction of ASTM Committee D-14 and are the direct responsibility of Subcommittee D14.80 on Metal Bonding Adhesives

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[2] *Annual Book of ASTM Standards*, Vol 10.01.

[3] *Annual Book of ASTM Standards*, Vol 15.06.

conditions. Volume resistivity or conductivity determinations are often used in checking the uniformity of an insulating material, either with regard to processing or to detect conductive impurities that affect the quality of the material and that may not be readily detectable by other methods.

4.5 Volume resistivities above $10^{21}\Omega\cdot$cm ($10^{19}\Omega\cdot$m), obtained on specimens under usual laboratory conditions, are of doubtful validity, considering the limitations of commonly used measuring equipment.

4.6 Surface resistance or conductance cannot be measured accurately, only approximated, because some degree of volume resistance or conductance is always involved in the measurement. The measured value is also affected by the surface contamination. Surface contamination, and its rate of accumulation, is affected by many factors including electrostatic charging and interfacial tension. These, in turn, may affect the surface resistivity. Surface resistivity or conductivity can be considered to be related to material properties when contamination is involved but is not a material property in the usual sense.

5. General Considerations

5.1 Definitions, theory, and measuring equipment pertaining to this method shall be in accordance with test methods already established for the property under consideration.

6. Test Specimens

6.1 For tests that are to be performed upon the adhesive itself, the specimens shall consist of an adequate representative sample which, until required, shall be kept at room temperature in a nearly filled, tightly sealed container to avoid contamination or escape of solvents.

6.2 For tests that are to be performed upon the adhesive as a film, the test specimens shall be prepared in accordance with Test Methods D 115, with exceptions as noted.

7. Power Factor and Dielectric Constant of Liquid Adhesives

7.1 *Procedure*—Test the adhesive and report in accordance with the procedure specified for liquids in Test Methods D 150.

8. Direct-Current Conductivity

8.1 *Procedure*—Determine volume resistivity and report in accordance with Test Methods D 257, except as follows: Make volume resistivity measurements with an electrification time of 1 min, a maximum potential gradient of 15 V/mil, and with the material at a temperature of 73.4 ± 2°F. Determine the volume resistivity of a weighed amount of suitable reagent grade solvent as selected or agreed upon by the manufacturer and user or as specified in the specification or puchase order. This resistivity, ρ_0, shall be no less than $2.0 \times 10^{13}\Omega\cdot$cm. Without removing the solvent from the container, add sufficient adhesive to result in a 2.0 ± 0.1 weight percent total solids content solution. Weigh adhesives in solid sheet or film form directly. For adhesives in a liquid state determine the total solids content by the following method before adding to the above solvent.

8.2 Pour approximately 10 g of the sample into a low-form weighing bottle, cover, and weigh. After removing the cover apply heat at 70°C until the sample reaches constant weight (see Note 1). Then cool the sample in a desiccator and weigh.

NOTE 1—**Caution**—Care should be exercised when volatile materials are of such a nature as to constitute toxic, fire, or explosive hazard.

8.3 *Calculations*— Calculate the percentage of total solids as follows:

$$\text{Total solids, percent} = \text{wt of residue/wt of sample} \times 100 \quad (1)$$

8.4 Stir the dried adhesive into the solvent with a clean glass rod until all the adhesive is dissolved. Return the cell to the solution and raise and lower it in the solution several times. Determine volume resistivity of the solution ρ_1 and calculate conductivity, σ, as follows:

$$\sigma(\text{ micromho cm}) = 1/\rho_0 - 1/\rho_1 \quad (2)$$

9. Extract Conductivity

9.1 *Procedure*—Determine the extract conductivity of an adhesive and report in accordance with the procedure specified for Aqueous Extract Conductivity in Test Methods D 202, except use a 5-g sample of adhesive in a solid state, or sufficient sample to be equivalent to 5 g of solids based on determining percentage total solids in accordance with 4.2. For adhesives soluble in a solvent (not water) add a suitable reagent grade solvent as selected or agreed upon by the manufacturer and user, or as specified in the specification or purchase order, until the total volume is 100 ml. Stir and warm gently until the sample is completely dissolved or diluted. To the solution add slowly with constant stirring 100 ml of boiling distilled water. Gently warm with constant stirring on an electric hot plate until the solvent has been distilled off, which can be detected by the absence of its characteristic odor, and the solution has been reduced to 100 ml or less. For adhesives soluble in water, add boiling distilled water until the total volume is 100 ml. Stir and warm gently until the sample is completely dissolved or diluted.

9.2 After filtering, wash the residue with 100 ml of hot distilled water. Adjust the final volume to 250 ml with hot distilled water. The conductivity, based on a sample-to-water ratio of 1 g to 100 ml, is equal to:

$$0.5\,K/R_2 - K/R_3 \times 10^6 \text{ micromhos/cm} \quad (3)$$

where:

K = cell constant ($C \times R$),
R_2 = resistance in ohms of extract solution at 25 ± 0.5°C, and
R_3 = resistance in ohms of water blank at 25 ± 0.5°C.

10. Acidity and Alkalinity

10.1 *Procedure*—Determine the acidity or alkalinity of an adhesive and report in accordance with Test Methods D 202, except that the extraction procedure shall be as prescribed in Section 6 of these methods.

11. pH Value

11.1 *Procedure*—Determine the pH value of an adhesive and report in accordance with Test Methods D 202, except that the extraction procedure shall be as prescribed in Section 9 of these test methods.

12. Power Factor and Dielectric Constant of a Dried or Cured Adhesive Film

12.1 *Test Specimens*— Prepare test specimens in accordance with the procedure specified for determining the dielectric strength of dried varnish film in Test Methods D 115. If cure is required, the time and temperature shall be in accordance with recommendations of the manufacturer of the adhesive.

12.2 *Procedure*—Determine the power factor and dielectric constant and report in accordance with the procedure specified for films in Test Methods D 150.

13. Dielectric Strength of a Dried or Cured Adhesive Film

13.1 *Test Specimens*— Prepare test specimens in accordance with 12.1.

13.2 *Procedure*—Determine dielectric strength and report in accordance with the procedure specified for determining the dielectric strength of dried varnish film in Test Methods D 115.

14. Volume and Surface Resistivity

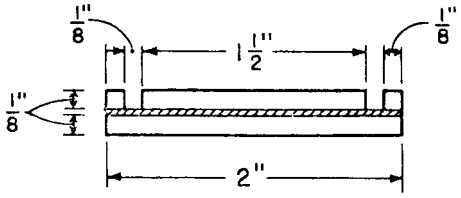
14.1 *Test Specimens*— Test specimens for use in determining the volume and surface resistivity of solid adhesives shall consist of two metal disks, one ⅛ by 2 in. (3 by 51 mm) in diameter, the other ⅛ by 1.5 in. (3 by 38 mm) diameter, and a metal ring ⅛ by 2 in. in outside diameter by 1.75 in. (3 by 51 by 44 mm) in inside diameter, bonded together by the sample adhesive as shown in Fig. 1. The specimen shall be bonded by the application of heat and pressure, if necessary, by means of parallel platens in a suitable press. Preparation of areas to be cemented, and gluing, shall be in accordance with the recommendations of the manufacturer of the adhesive and with Test Method D 897. Thickness of the glue line shall be not greater than 0.060 in. (1.52 mm) nor less than 0.0020 in. (0.051 mm).

14.2 *Procedure*—Determine the volume and surface resistivity of solid adhesives and report in accordance with Test Methods D 257. The voltage applied shall not exceed 30 V/mil. Read at the end of 1 min electrification.

15. Arc Resistance

15.1 *Test Specimens*— Prepare test specimens for use in determining the arc resistance of adhesives in accordance with Section 12, except use a 4 by 4 in. sheet of XXX grade flat phenolic laminate for the base material, and the thickness of the adhesive film shall be between 0.002 (0.05 mm) and 0.005 in (0.13 mm).

15.2 *Procedure*—Determine the arc resistance of solid adhesives and report in accordance with Test Method D 495.



Metric Equivalents			
in.	⅛	1½	2
°mm	3	38	51

FIG. 1 Test Specimen for Use in Determining Volume and Surface Resistivity.

16. Report

16.1 Report the following information:

16.1.1 Complete identification of the adherend and adhesive materials tested, including adhesive type, source, and manufacturer's code number.

16.1.2 Curing conditions used.

16.1.3 Conditioning environment and test environment.

16.1.4 Number of specimens tested per sample.

16.1.5 Dimensions of each test specimen including glue line thickness.

16.1.6 Corrected resistances of each test specimen.

16.1.7 Volume resistivity, in ohm-centimeters, of each test specimen.

16.1.8 Average volume resistivity of the sample, when more than one test specimen per sample is used.

16.1.9 Standard deviation of the volume resistivity measurements when more than one test specimen per sample is used.

16.1.10 Power factor as in Test Methods D 150.

16.1.11 Direct-current conductivity as in Test Methods D 257.

16.1.12 Extract conductivity as in Test Methods D 202.

16.1.13 Acidity and alkalinity as in Test Methods D 202.

16.1.14 pH value as in Test Methods D 202.

16.1.15 Volume and surface resistivity as in Test Methods D 257.

16.1.16 Arc resistance as in Test Method D 495.

17. Precision and Bias

17.1 *Precision*—Precision and Bias for this test method is beng determined and will be available by September 2004.

17.2 *Bias*—A statement of bias cannot be made because of the lack of a standard reference material.

17.3 The precision and bias of this test method are a function of the adhesive system, surface preparation, substrates, test temperature, cyclic stress level, and other factors related to the test apparatus, laboratory, and operator variabilities. Precision shall be reported in terms of the standard deviation of the data and the standard error of the mean.

18. Keywords

18.1 adhesives; arc resistivity; d-c conductivity; dielectric strength; electrical insulation; power factor; surface resistivity; volume resistivity