Designation: D1304 – 99 (Reapproved 2020)ε1

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

This standard is issued under the fixed designation D1304; 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.

ε[1] NOTE—An editorial correction was made to 17.1 in January 2020.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see 8.2.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

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

D202 Test Methods for Sampling and Testing Untreated Paper Used for Electrical Insulation

D257 Test Methods for DC Resistance or Conductance of Insulating Materials

D495 Test Method for High-Voltage, Low-Current, Dry Arc Resistance of Solid Electrical Insulation

D897 Test Method for Tensile Properties of Adhesive Bonds

D907 Terminology of Adhesives

3. Terminology

3.1 *Definitions*—Many terms in these test methods are defined in Terminology D907.

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

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

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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.

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 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} Ω·cm (10^{19} Ω·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 D115, 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 D150.

8. Direct-Current Conductivity

8.1 *Procedure*—Determine volume resistivity and report in accordance with Test Methods D257, 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 purchase order. This resistivity, ρ_0, shall be no less than 2.0×10^{13} Ω·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 Warning). Then cool the sample in a desiccator and weigh. (**Warning—**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 D202, 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.