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Standard Methods of Testing POLYMERIZABLE EMBEDDING COMPOUNDS USED FOR ELECTRICAL INSULATION[1]

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1. Scope

1.1 These methods cover electrical, mechanical, and thermal tests for polymerizable compounds used for encasing or embedding electrical and electronic components or assemblies. The procedures appear in the following sections:

Procedure	Sections	ASTM Method
Sampling	3	...
Specimen Preparation and Conditioning	4	D 618
Hardness	5 to 11	D 2240
Coefficient of Linear Thermal Expansion	12 to 18	D 696
Specific Gravity	19 to 26	D 792
Coefficient of Thermal Conductivity	27 to 33	...
Thermal Shock Resistance	34 to 39	...
Dielectric Constant and Dissipation Factor	40 to 48	...
Dielectric Strength	49 to 55	...
Arc Resistance	56 to 60	D 495
Effect of High Humidity	61 to 66	...
Flame Resistance (ignition time and burning time)	67	D 229
Dielectric Strength of Embedded Electrodes	68 to 75	D 149

1.2 The compounds must cure without pressure and may or may not require heat to accomplish the reaction. They are required to electrically insulate and mechanically protect and support the part or assembly without a case, pot, or other external reinforcement after curing.

1.3 The values states in inch-pound units are to be regarded as the standard.

2. Applicable Documents

2.1 *ASTM Standards:*

D 149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies[2]

D 150 Test Methods for A-C Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials[2]

D 229 Method of Testing Rigid Sheet and Plate Materials Used for Electrical Insulation[3]

D 374 Test Methods for Thickness of Solid Electrical Insulation[2]

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

D 618 Methods of Conditioning Plastics and Electrical Insulating Materials for Testing[2]

D 696 Test Method for Coefficient of Linear Thermal Expansion of Plastics[4]

D 792 Test Methods for Specific Gravity and Density of Plastics by Displacement[4]

D 2240 Test Method for Rubber Property—Durometer Hardness[5]

E 197 Specification for Enclosures and Servicing Units for Tests Above and Below Room Temperature[6]

3. Sampling

3.1 Because of the diverse nature of the com-

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[2] *Annual Book of ASTM Standards*, Vol 10.02.

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

[4] *Annual Book of ASTM Standards*, Vol 08.01.

[5] *Annual Book of ASTM Standards*, Vol 09.01.

[6] Discontinued, see *1980 Annual Book of ASTM Standards*, Part 12.

pounds, and the various forms and packages of resins, hardeners, catalysts, etc., commercially available, no standard methods of sampling have been established. An adequate amount of material, representative of each ingredient, shall be selected from each lot to permit preparation of specimens in accordance with Section 4.

3.2 Ingredients shall not be used beyond the shelf life designated by the manufacturer.

4. Specimen Preparation and Conditioning

4.1 *Mold*—Slab specimens shall be cast in a vertical position in any suitable mold which will result in castings of a thickness which can be controlled within ±5 %. The mold may consist of two highly polished plates, ⅜ in. (9.5 mm) 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 compound will not adhere. The mold assembly shall be clamped together with sufficient pressure to prevent leakage of the compound. A mold to produce 6 by 6-in. (152 by 152 mm) specimens is conveniently handled. The resulting cast slabs shall then be cut into the size specimens required.

4.2 *Casting*—The compound ingredients shall be mixed in accordance with the manufacturer's directions, then poured into the molds. When the ingredients are mixed above ambient temperature, the molds shall be preheated approximately 10°C above the mixing temperature before the mixture is poured into the molds. In pouring, precautions shall be taken to avoid entrapping of air. Specimens shall then be cured in accordance with the manufacturer's instruction as to time and temperature. In all cases, the curing schedule used shall be reported.

4.3 *Conditioning*—Before testing, all specimens shall be conditioned 40 h at the Standard Laboratory Atmosphere in accordance with Procedure A of Methods D 618.

4.4 *Special Requirements*—Unless otherwise noted in these methods, specimens shall be prepared in accordance with the above procedure.

HARDNESS

5. Definition

5.1 *hardness*—the indentation or penetration hardness as read on a Shore D durometer or on a Barcol hardness tester, Model GYZJ 934-1, when these instruments are operated in the manner and under the conditions specified in Section 6.

6. Significance and Use

6.1 The arbitrary hardness obtained is intended to provide a basis for comparison between two materials or between lots of a given material. It may be useful as a means of obtaining qualitative measures of the degree of polymerization, completeness of cure in the case of thermosetting materials, or degree of plasticization. It may also serve as a measure of one of the effects of addition of inert fillers.

6.2 Since for many materials the time factors involved in applications of the penetrators are very critical, it is necessary to specify exactly the time for taking the readings.

7. Apparatus

7.1 *Shore D Durometer*[7]—The Shore D durometer is similar in general to the durometers described in Test Method D 2240, except that it is provided with a comparatively sharp conical impressor point instead of the truncated conical indentor shown in Fig. 1 of Test Method D 2240.

7.2 *Barcol Impressor*,[8] Model GYZJ 934-1.

7.3 The results obtained by this test method are measures of the indentations produced by the standard penetrators supplied with commercial models of the Shore D and Barcol hardness testers, when used as specified. The Shore D durometer is employed for hardness readings up to a value of 90, while the Barcol tester is to be used when the readings of the Shore D instruments are above 80. This provides for an overlap region between the ranges of the two instruments.

NOTE 1—In this region of overlap it may be considered desirable to adopt the practice of using both the Shore D and Barcol instruments.

8. Test Specimens

8.1 Three test specimens shall be in the form of flat plates ⅛ in. (3.2 mm), thick and at least 2 by 2 in. (50 by 50 mm).

9. Conditioning

9.1 Test specimens shall be conditioned in accordance with 4.3.

[7] The Shore D durometer is manufactured by The Shore Instrument Co., Inc., Jamaica, NY.

[8] The Barcol Impressor is manufactured by the Barber-Colman Co., Rockford, IL.

10. Procedure

10.1 Conduct tests at a temperature of 23 ± 1.1°C.

10.2 Take Shore D durometer readings, using the procedure in Test Method D 2240. Take readings 10 s from the time of application of the durometer to the specimen. Take five readings on each specimen.

10.3 For the Barcol hardness tests, press the instrument firmly on the specimen and take the reading immediately (maximum reading). Take five readings on each specimen.

11. Report

11.1 The report shall include the following:

11.1 Description of the sample, including type of material, lot identification, etc.,

11.2 Specimen size and thickness,

11.3 Conditioning procedure and temperature of test, and

11.4 All observed and recorded hardness readings, and the average.

COEFFICIENT OF LINEAR THERMAL EXPANSION

12. Description of Term

12.1 *coefficient of linear thermal expansion per °C*—is a measure of the reversible linear expansion caused by changes in temperature.

13. Significance and Use

13.1 The coefficient of linear thermal expansion is of interest in these materials since differences between the embedding compound coefficient and that of the components and parts embedded may cause, for example, crushing of vacuum tubes, cracking of the embedding compound itself, or displacement and change in electrical characteristics of the parts or circuit embedded. The method is simple but should not be used where great accuracy is required. It is not applicable to very soft rubbery materials.

14. Apparatus

14.1 Apparatus shall be as described in Test Method D 696, except that the temperature bath shall be provided with a means for raising the temperature of the bath at a steady rate of 1°C/min. The bath shall be suitable for control of temperature and operation at the upper temperature to which the compound may be subjected. It shall also control and operate at a temperature sufficiently low to establish a coefficient over the range of temperatures to which the compound may be subjected.

15. Test Specimens

15.1 Three round test specimens, 2 in. (50 mm) in length, shall be prepared in accordance with Test Method D 696.

16. Conditioning

16.1 Test specimens shall first be conditioned for 1 h at 10°C above the maximum specified temperature of the run. Specimens shall then be conditioned in accordance with 4.3.

17. Procedure

17.1 Place a specimen in the dilatometer and mount the assembly in a liquid bath.

17.2 Take measurements between the temperature limits agreed upon. Maintain the assembly at the lowest temperature specified for ½ h and record the reading of the indicator.

17.3 Raise the temperature of the bath at the rate of 1°C/min and record the readings of the indicator for every 5°C rise.

17.4 When the bath reaches the maximum specified temperature, maintain the bath at this temperature for ½ h and again record the reading.

17.5 Plot a curve of length *versus* temperature, using only those readings recorded during the steady rise of temperature.

18. Calculation and Report

18.1 Using the plotted curve, calculate the coefficient of linear thermal expansion for each specimen in accordance with Test Method D 696. It will be noted that the coefficient of linear thermal expansion for some materials is known to change abruptly at certain temperatures. This will be noticeable in the length *versus* temperature curve. If such a transition is found, determine a separate coefficient of expansion for the temperature range below and above the transition point.

18.2 Calculate also the coefficient of linear thermal expansion for each specimen in accordance with Test Method D 696, using the change recorded betwen the lowest temperature and after ½ h at the highest temperature.

18.3 The report shall include those items listed in Section 7 of Test Method D 696, except for 7.1.3, 7.1.6 and 7.1.7. It shall also include the coefficient calculated for each specimen and the

average determined for each method of calculation (constant rate-of-rise and fixed points).

SPECIFIC GRAVITY

19. Definition

19.1 *specific gravity*—the ratio of the weight of a given volume of a cast sample of compound to the weight of the same volume of distilled water at 23 ± 1.1°C (73.4 ± 2°F).

20. Significance and Use

20.1 Specific gravity indicates the relative weight of different compounds and may assist in their identification in acceptance testing.

21. Apparatus

21.1 Apparatus shall be as described in 4.1 and 4.5 of Test Methods D 792.

22. Test Specimens

22.1 Three test specimens shall be measured, and each specimen shall be at least 3 in.^{2} (19.4 cm^2) in area by ⅛ in. (32 mm) in thickness. Cut edges shall be smoothed to avoid entanglement of air bubbles when the specimen is immersed in water.

23. Conditioning

23.1 Specimens shall be conditioned in accordance with 4.3.

24. Procedure

24.1 The procedure shall be in accordance with Method A-1 of Test Methods D 792.

25. Calculation

25.1 Calculate the specific gravity of the compound as in Method A-1 of Test Methods D 792.

26. Report

26.1 The report shall include the following:

26.1.1 Description of the material, including type of material, lot identification, etc.,

26.1.2 Specimen size, and

26.1.3 All recorded readings, and the average.

COEFFICIENT OF THERMAL CONDUCTIVITY

27. Definition

27.1 *thermal conductivity of an embedding compound*—the rate of heat flow under steady conditions, through unit area of unit thickness per unit temperature gradient, in the direction perpendicular to the area, using a cast sheet of material and the conditions described herein.

28. Significance and Use

28.1 The application of embedding compounds will often involve the embedment of electrical or electronic components that are heat sources during operation, and embedding compounds are usually poor thermal conductors. However, knowledge of the thermal conductivity of various resin-filler systems permits the selection of a high- or low-conductivity system, whichever is best suited to the application.

29. Apparatus

29.1 *Thermal Conductivity Apparatus*,[9] consisting of two parts, the "source" or vessel that holds boiling water and the "receiver" or receptacle containing a heat-insulated copper plug. The source is a copper vessel with heat-insulated sides and a carefully ground and nickel-plated base; the receiver is a copper plug, face-ground, nickel-plated, and carefully heat insulated. One junction of a copper-constantan thermocouple is embedded in the copper base of the source and the other junction is embedded in the copper plug of the receiver. The terminals are brought out to binding posts on the sides of the apparatus. A 5-lb (2.35-kg) weight is used on top of the source.

29.2 *Galvanometer, D-C Milliammeter, or Potentiometer.*

29.3 *Micrometer.*

29.4 *Stop Watch.*

29.5 *Immersion Heater.*

29.6 *Solid Carbon Dioxide.*

29.7 *Mineral Oil.*

30. Test Specimens

30.1 Three specimens 3 by 3 by ⅛ in. (76.0 by 76.0 by 3.2 mm) shall be tested. Specimens shall not be warped or bent, and surfaces shall be free of major imperfections such as ridges, cavities, etc.

31. Procedure

31.1 Connect the measuring instrument by

[9] The Cenco-Fitch Apparatus, Cat. No. 77555, available from Central Scientific Co., Chicago, IL., has been found satisfactory for this purpose.

means of copper wire between the copper binding posts of the source and receiver vessels, and connect the constantan posts of each vessel with constantan wire.

31.2 Measure the test specimen for thickness, using methods described in Test Methods D 374.

31.3 Spread a few drops of mineral or other suitable oil on one face of the specimen, after which place the specimen, oiled side up, beneath the source vessel but not on the receiver. **Caution**—Do not use oil with porous materials.

31.4 Fill the source vessel with water, heated to boiling with the immersion heater.

31.5 Cool the receiver with solid carbon dioxide until its temperature is approximately −10°C. Spread a few drops of mineral oil on the receiver.

31.6 When the current differential becomes constant, as indicated by a steady deflection on the meter, place the specimen with the source vessel on the receiver. Then place the 5-lb (2.35-kg) weight on the source vessel to ensure intimate contact between source, specimen, and receiver.

31.7 Read the meter deflection every 2 min until 16 readings have been taken.

32. Calculation

32.1 Plot the logarithm of the meter readings against the time in seconds, using linear graph paper. As the receiver and source approach a common temperature, errors due to radiation losses will result in points not falling in a straight line. Using only the straight portion of the curve, draw a straight line extending to both intercepts. Label the origin B, the intercept on the abscissa A, and the intercept on the ordinate C. Then

$$m = BA/BC$$

where:

m = slope of line, s,
BA = total time, from B to A, and
BC = value at C intercept minus value at B.

32.2 Calculate the coefficient of thermal conductivity as follows:

$$K = (2.303 \times t \times M \times C)/(A \times m)$$

where:

K = coefficient of thermal conductivity,
t = thickness of specimen, cm,
M = mass of copper plug, g,
C = specific heat of copper, cal/g·°C,
A = area of copper plug, cm², and
m = slope of line, s.

NOTE 2—For a given piece of equipment M, C, and A are known; therefore, an equipment constant may be calculated.

33. Report

33.1 The coefficient of thermal conductivity, when calculated as above, shall be reported in cal·cm/s·cm²·°C.

THERMAL SHOCK RESISTANCE

34. Definition

34.1 *thermal shock resistance of an embedding compound*—the ability of four out of five specimens, each containing a hexagonal steel bar, to withstand without fracture, ten thermal cycles.

35. Significance and Use

35.1 This method is useful for screening materials but is not intended to represent all conditions of service. Electrical and electronic circuits are cast in embedding compounds, and these components may be of comparatively large mass and generally of different thermal expansion characteristics than the compound. The embedments are subjected to variations in temperature because of embedded heat sources and the wide temperature range they encounter in service. The thermal shock resistance of an embedment is a critical characteristic since cracks in a casting would negate the function of the compound. The thermal shock resistance will vary, depending on the resin-filler-curing agents used and on the curing conditions employed.

36. Apparatus

36.1 *Oven*—A forced-draft oven capable of maintaining the temperature in accordance with Grade A units of Specification E 197, and provided with a small opening in top or side so that specimens can be inserted and removed with a minimum drop in temperature. The five specimens shall be secured in a suitable mounting by the unembedded portion of the pin shown in Fig. 1. They shall be conveniently spaced so that observation can be quickly made.

36.2 *Heat Transfer Medium*—Alcohol or any heat transfer medium that remains fluid at −55°C and appreciably will not affect the compound tested. A minimum of 6 gal (22.7 L) of the liquid shall be used.

36.3 *Temperature Control*—A low-temperature cabinet or any other device that will maintain the temperature of the liquid in accordance

with Grade A units of Specification E 197.

36.4 *Specimen Mold,* as illustrated in Fig. 1 and described in 37.2.

36.5 *Specimen Embedment*—The embedment shall be a ¾-in. (19-mm) hexagonal bar, 1 in. (25.4 mm) long (cold-drawn low-carbon steel) polished with a No. "0" emery cloth and washed with a 50:50 xylol-alcohol mixture. A ⅛-in. (3.2-mm) diameter cold-drawn low-carbon steel pin, ⅝ in. (15.9 mm) long shall be press fitted into a hole drilled in the center of one end of the hexagonal bar to a depth of ⅛ in. (3.2 mm).

37. Test Specimens

37.1 Five specimens shall be cast in accordance with the manufacturer's curing instructions and to the dimensions shown in Fig. 1. Each specimen shall contain an embedment positioned as shown in Fig. 1.

37.2 A suggested mold which has been found satisfactory is shown in Fig. 2. The mold is assembled by inserting the pin of the hexagonal bar *F* into bushing *E*, which holds it snugly. The hexagonal bar is thus perpendicular to bottom plate *D*. The brass tube *C* is then set in the cavity of the bottom plate. The ends of the tube and the bottom plate are machined exactly so that the inside wall of the tube is parallel to the hexagonal bar. The shredded lead *G* is then packed loosely around the outside of the tube in the cavity of the bottom plate. Jig *B* is then placed over the tube, fitted into the cavity of the bottom plate, and forced downward with a hand press. In this operation the shredded lead is compressed into a gasket *H*, which seals the tube to the bottom plate and automatically centers the tube so that the hexagonal bar is centrally located within the tube.

37.3 The liquid mixture is poured into the open end of the tube. The cast specimens are easily removed by pushing them out of the wide end of the tube.

38. Procedure

38.1 Place the specimens in the oven for 30 min at the specified temperature (90, 105, 130, 155, or 180°C, depending on the type compound or service anticipated), remove and quickly observe, then plunge into the low-temperature bath at −55 ± 2°C for 10 min. Remove, wipe the liquid from the specimens, quickly observe, then start the next cycle. Cycle until the specimen fails or until ten cycles have been completed without failure. Failure will be evidenced by the development of cracks or fissures in the specimen.

39. Report

39.1 The report shall include the following:

39.1.1 The curing schedule used,

39.1.2 Temperature of the oven to the nearest °C, and

39.1.3 Number of failures and cycles in which failures occurred. A one half cycle shall be reported when specimens fail at the elevated temperatures.

DIELECTRIC CONSTANT AND DISSIPATION FACTOR

40. Scope

40.1 This method specifies equipment and procedures for determining the dielectric constant and dissipation factor of an embedding compound from 60 Hz to 100 kHz over a range of temperatures, and from 1 to 100 MHz at room temperature.

41. Definitions

41.1 Dielectric constant and dissipation factor are fully defined in Test Methods D 150. Briefly, the dielectric constant of an embedding compound is the ratio of the capacitance between two conductors using the compound as the dielectric to the capacitance between the same configuration of conductors in vacuum (or air). The dissipation factor is the ratio of the resistive to the capacitive current flowing to a capacitor using the compound as the dielectric. The product of dielectric constant and dissipation factor is the loss index (loss factor).

42. Significance and Use

42.1 The dielectric constant has a direct effect on the distributed capacitance of coils and on the capacitance between parts of circuits embedded in the compound. The rate of energy dissipation in the compound under an applied alternating electric field is proportional to the loss index. Heat generated by dielectric loss must be considered for components operating at high voltage or high frequency, or which have inadequate means of heat removal. Hence a knowledge of the dielectric constant and dissipation factor, and of their changes with frequency and temperature, is helpful in selecting embedding and encapsulating