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An American National Standard

Standard Test Method for Fluid and Grease Resistance of Thermoset Encapsulating Compounds Used in Electronic and Microelectronic Applications[1]

This standard is issued under the fixed designation F 677; 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 This test method determines the resistance of encapsulating compounds to fluids or greases by measuring changes in weight (Note 1) and volume under defined conditions of time and temperature.

NOTE 1—To provide consistency with the usage in other ASTM test methods concerned with determining the properties of plastic materials, the terms "weight" and "weigh" are used in this test method although the units of measurement are those of mass.

1.2 *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.* See 9.1.

1.3 The values stated in SI units are standard. The values in parentheses are for information only.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1711 Terminology Relating to Electrical Insulation[3]

D 5423 Specification for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation[4]

D 6054 Practice for Conditioning Electrical Insulating Materials for Testing[4]

3. Terminology

3.1 See Terminology D 1711 for definitions of terms relating to electrical insulation.

4. Summary of Test Method

4.1 Specimens of encapsulating compound are immersed in fluids or greases for specified conditions of temperature and time. The weight and volume of each specimen is measured before and after immersion and percent change is reported.

4.2 The fluids and greases to be used as test media (8.3) are not specified by the test method and shall be agreed to by the parties to the test.

5. Significance and Use

5.1 Fluids and greases in contact with encapsulating compounds may adversely modify the encapsulant properties with resulting damage or loss of protection to components in electronic applications.

5.2 This test method provides a means for measuring the effect of fluids and greases on various encapsulants.

5.3 This test method is intended for use in research and evaluation.

6. Interferences

6.1 Test results obtained with molded or cast specimens of filled encapsulating compounds will differ from those obtained with cut or machined specimens (because of exposed fillers in the latter).

6.2 Lack of complete contact between the test medium and the surfaces of the specimen may seriously affect the results of the test.

6.3 Some encapsulants may be affected by the reagents specified for use in removing the test media from the specimens. Alternative reagents may be required, as agreed upon by the parties to the test.

7. Apparatus

7.1 *Analytical Balance*, capable of determining weight to the nearest 0.001 g.

7.2 *Oven*, forced-convection type meeting the requirements of Specification D 5423, Type I.

7.3 *Glass Dishes*, of sufficient diameter for specimens to be exposed horizontally, and of sufficient height for specimens to be completely covered by the test medium. A loose-fitting glass cover shall be provided for each dish (see 13.3).

8. Reagents and Materials

8.1 Use the following materials:

8.1.1 *Acetone*.

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[1] This test method is under the jurisdiction of ASTM Committee D-9 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] *Annual Book of ASTM Standards*, Vol 11.01.

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

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

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8.1.2 *Distilled Water*—Type III Reagent Water as defined in Specification D 1193.

8.1.3 *Isopropyl Alcohol*.

8.2 *Filter Paper*, medium retention and medium filtering speed, free of lint or foreign material.

8.3 *Test Media*—agreed-upon fluids or greases.

9. Safety Precautions

9.1 Some encapsulating compounds are known to contain toxic components, and special precautions are required in handling. Follow manufacturer's precautionary instructions and sound laboratory safety practices.

10. Sampling

10.1 Because of the diverse nature of the encapsulating compounds, and the various forms and packages of resin, hardeners, catalysts, etc. commercially available, no standard methods of sampling have been established. Select an adequate amount of material, representative of each ingredient from each lot to permit preparation of specimens as agreed upon by the parties to the test.

11. Test Specimen

11.1 Prepare three specimens of each encapsulating compound to be tested, in accordance with generally accepted practices.

11.1.1 Each specimen shall be a disk 51 ± 2.5 mm (2 ± 0.1 in.) diameter and 3 ± 0.30 mm (0.125 ± 0.01 in.) in thickness (see 6.1). Surface finish of machined specimens shall be 64 maximum.

12. Conditioning

12.1 Unless otherwise specified, condition all specimens for 40 h at Standard Laboratory Atmosphere in accordance with Procedure A of Practice D 6054.

13. Procedure

13.1 Weigh each specimen in air to the nearest 0.001 g and record value as W_1. Weigh each specimen while immersed in distilled water at room temperature and record the value as W_2. Quickly dip each specimen in isopropyl alcohol to remove water, blot dry with filter paper, and place in glass dish (7.3).

13.2 Stack all three specimens of a single composition in the same glass dish.

13.2.1 For fluid test media, insert the filter paper into the dish, cover it with fluid, then add a specimen and cover it with fluid. Repeat this procedure to form a sandwich structure of alternating filter paper and specimen layers.

13.2.2 For grease test media, omit the filter paper if the viscosity of the grease at the exposure temperature is sufficient to prevent specimens from touching. Apply grease to all exposed surfaces of the specimens and insert coated specimens into the dish to form a sandwich structure of alternating grease and specimen layers.

13.3 Cover the assembled test specimens with a loose-fitting glass cover and expose the assembled test specimens in an oven at 71 ± 3°C (160 ± 5.4°F) for a total duration of 14 days.

13.4 Remove the specimens from the oven for measurement and observation after cumulative exposure times of 24 ± 2 h (1 day), 168 ± 2 h (7 days), and 336 ± 2 h (14 days).

13.4.1 Remove the assembled test specimens from the oven and allow them to cool to room temperature for 30 to 60 min, before conducting measurements and observations as follows:

13.4.1.1 Remove specimens one at a time from the test medium, saving the test medium in the glass dish or other convenient noncontaminating vessel. Remove (and save) high-viscosity fluids and greases from the specimen with a spatula. Wipe with a lint-free filter paper before dipping the specimen in acetone.

13.4.1.2 Dip each specimen quickly into acetone to remove the remaining test medium and blot with filter paper (see 6.3).

13.4.1.3 Within 1 min after removing the specimen from the test medium, weigh the specimen in air to the nearest 0.001 g and record the value as W_3.

13.4.1.4 Immerse each specimen in distilled water, and within 1 min after the determination in air, weigh each specimen while immersed, to the nearest 0.001 g; record the value as W_4.

13.4.1.5 Dip each specimen in isopropyl alcohol to remove the water after the weight in water (W_4) has been determined. Then blot the specimen with filter paper.

13.4.1.6 Visually examine each set of specimens and the associated test media from a single glass dish. Record the condition of the specimens and media. Note characteristics such as color change, cracking, swelling, shrinking, surface change, tackiness, etc.

13.4.1.7 When the test is to be continued, replace the specimen in the glass dish, and cover the specimen with the same test medium saved in 13.4.1.1. Reform the sandwich structure described in 13.2. If there is insufficient test medium to cover the specimens, add additional medium.

14. Calculation

14.1 Calculate the change in weight as follows:

$$\text{Change in weight, \%} = \frac{W_3 - W_1}{W_1} \times 100 \quad (1)$$

where:

W_1 = initial weight of specimen in air, mg, and
W_2 = weight of specimen in air after immersion, mg.

14.2 Calculate change in volume as follows:

$$\text{Change in volume, \%} = \frac{(W_3 - W_4) - (W_1 - W_2)}{(W_1 - W_2)} \times 100 \quad (2)$$

where:

W_3 = initial weight of specimen in water, mg, and
W_4 = weight of specimen in water after immersion, mg.

15. Report

15.1 Report the following information:

15.1.1 Identification of encapsulating compound,

15.1.2 Identification of test media used,

15.1.3 Temperature and total duration of test exposure,

15.1.4 Description of the specimen surface preparation (examples, molded, cut or machined), and

15.1.5 After each exposure period:

15.1.5.1 Change in weight, percent, for each specimen,

15.1.5.2 Average change in weight, percent, for each encapsulating compound,

15.1.5.3 Change in volume, percent, for each specimen,

15.1.5.4 Average change in volume, percent, for each encapsulating compound, and

15.1.5.5 Description of condition of specimens and test media as determined by visual examinations and recorded in 13.4.1.6.

16. Precision and Bias

16.1 *Precision*:

16.1.1 An interlaboratory study was conducted in which five laboratories tested two encapsulating compounds in two test media for each compound.

16.1.2 The data have been analyzed to express precision in terms of standard deviations. The data are given in Table 1.

16.2 *Bias*—This test method has no bias since the value for fluid and grease resistance is defined in terms of this test method.

17. Keywords

17.1 electronic; encapsulating compound; grease; microelectronics

TABLE 1 Interlaboratory Test Data

System/Medium	Average	Precision, % Absolute	
		Within Laboratory, S_w	Between Laboratory, S_b
Change in Weight			
Ia	49.3	3.69	5.11
Ib	4.35	0.70	3.47
IIa	0.27	0.17	0.39
IIb	0.09	0.17	0.65
Change in Volume			
Ia	51.32	3.67	5.44
Ib	4.73	0.75	3.73
IIa	0.48	0.22	0.33
IIb	0.45	0.36	0.87

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