



Standard Test Methods for Evaluating Compatibility Between Cable Filling and Flooding Compounds And Polyolefin Wire and Cable Materials¹

This standard is issued under the fixed designation D 4568; 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 evaluate the compatibility between cable filling or cable flooding compounds, or both, and polyolefin materials used in the manufacture of wire and cable that are usually in intimate contact with the filler or floodant, or both.

1.2 These test methods are useful to ensure compatibility and to verify that new formulations of filling or flooding compounds will have no deleterious effect upon the other polyolefin materials being used or, conversely, use these methods to ensure that other polyolefin wire and cable materials are evaluated for possible use not degraded by contact with fillers or floodants already in use.

1.3 Whenever two sets of values are presented, in different units, the values in the first set are the standard, while those 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.*

2. Referenced Documents

2.1 ASTM Standards:

D 1711 Terminology Relating to Electrical Insulation²

D 1928 Practice for Preparation of Compression-Molded Polyethylene Test Sheets and Test Specimens³

D 2633 Test Methods for Thermoplastic Insulations and Jackets for Wire and Cable⁴

D 4730 Specification for Flooding Compounds for Telecommunications Wire and Cable⁴

D 4731 Specification for Hot-Application Filling Compounds for Telecommunications Wire and Cable⁴

D 4732 Specification for Cool-Application Filling Compounds for Telecommunications Wire and Cable⁴

D 5423 Specification for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation⁴

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in these test methods, refer to Terminology D 1711

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *aging (act of), n*—exposure of materials to filling or flooding compound at a specified temperature for a specified time.

3.2.2 *filling compound*—any of several materials (see Specifications D 4731 and D 4732) used to fill the air spaces in the cores of multi-conductor insulated wires and cables for the purpose of excluding air or moisture or both; especially with regard to telecommunications wire and cable intended for direct burial.

3.2.3 *flooding compound*—any of several materials (see Specification D 4730) used to flood wire or cable sheath interfaces (for example, the region between core wrap and a shield, between a shield and a jacket, etc.) to eliminate or to minimize normal voids or air spaces in these areas; especially with regard to telecommunications wire and cable intended for direct burial.

4. Significance and Use

4.1 Cable filling and flooding compounds are normally semi-solid at room temperature and fluid in varying degrees at elevated temperatures. They are normally applied in a liquid state and at an elevated temperature during wire and cable manufacturing. The completed finished wire or cable is exposed to various ambient conditions during its useful life. If not carefully selected, components of filling or flooding compounds may act to degrade the materials they contact, short term or long term. The following methods are intended to minimize the chances of such problems occurring.

¹ These test methods are under the jurisdiction of ASTM Committee D-9 on Electrical and Electronic Insulating Materials and are the direct responsibility of Subcommittee D09.18 on Solid Insulations, Nonmetallic Shieldings, and Coverings for Electrical Wires and Cables. Members of ASTM Committee D-2 on Petroleum Products and Lubricants, and ASTM Committee B-7 on Light Metals and Alloys, have contributed to these test methods.

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

³ *Annual Book of ASTM Standards*, Vol 08.01.

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

4.2 Some of the effects that might occur include, but are not limited to:

4.2.1 Delamination of coated metal shields or screens in completed wire and cable. Delamination is primarily a function of the test temperature and the type of laminant used, so test results are unlikely to vary significantly between filling or flooding compounds of a common family (for example, petroleum based filling or flooding compounds).

4.2.2 Degradation of physical properties of insulation, jackets, core coverings, etc. Likely manifestations of degradation of plastic material include embrittlement of some materials and excessive softening of other materials.

4.3 Since the magnitude of any given effect will vary, some test procedures will be more critical than others. It is not, therefore, intended that every listed procedure be performed with every compatibility study. Perform procedures to the extent required by product specifications or as agreed upon between the producer and the purchaser.

5. Apparatus

5.1 *Oven*—Forced-convection oven, conforming to Specification D 5423, Type II.

5.2 *Containers*—Glass or other containers of suitable size, shape, and make-up as required to hold the designated specimens.

5.3 *Clamps*, or other devices as suitable for holding specimens in the oven.

5.4 *Tensile Strength Testing Apparatus*, as specified by the test requirement.

5.5 *Mill, Press, and Molding Equipment*, as needed for preparation of compression molded specimens as required.

5.6 *Miscellaneous Lab Equipment*—Towels, knives, etc. as required for cleaning and cutting specimens.

6. Sampling

6.1 Ensure that samples of filling and flooding compounds and the other affected wire and cable materials to be evaluated are representative of the materials to be used or to be found in the finished products.

COMPATIBILITY EVALUATION BETWEEN COATED METALS AND FILLING OR FLOODING COMPOUNDS

7. Scope

7.1 This test method is specifically for polyolefin coatings on aluminum and steel shielding and armoring material used in telecommunications wire and cables.

8. Specimen Preparation

8.1 Cut a specimen of coated metal tape (shielding tape, armor tape, screen tape) 6 in. (150 mm) long for evaluation. If a choice of tape widths is possible, select a width narrow enough to fit easily into a container (beaker, graduated cylinder, etc.).

8.2 Heat a quantity of the filling or flooding compound to a temperature such that all components of the compound are in solution and the compound is a homogenous pourable liquid (98°C minimum). Maintain at a temperature higher than 102°C

only if necessary for pouring. A standardized pouring temperature of $100 \pm 2^\circ\text{C}$ is preferred in order to produce uniform testing. Record melting and pouring temperatures used, together with other relevant laboratory data.

8.3 Pour the melted compound into the container holding the coated metal tape. Pour sufficient material to ensure that the tape is completely covered.

9. Specimen Aging

9.1 Place the immersed metal tape into an oven preheated to the temperature mutually agreed upon between the wire and cable manufacturer and the purchaser. Unless otherwise specified, maintain this temperature for a period of 168 h (7 days) minimum. See Note 1.

NOTE 1—It may be desirable to age specimens at more than one temperature ($60 \pm 1^\circ\text{C}$ and $80 \pm 1^\circ\text{C}$ are commonly used) or to age all specimens to a failure time, or both. If specimens are being aged to failure, it is suggested that sets of specimens be removed from the oven at the end of interim periods (that is, after 7 days, after 14 days, etc).

10. Specimen Examination

10.1 After the aging period is concluded and before cooling, extract the metal tape from the container of filling or flooding material. Allow as much as possible of the compound to drain from the specimen. Avoid wiping the specimen and do not expose it to solvents to remove the filling or flooding material. (See Note 2.)

NOTE 2—High melt-temperature filling and flooding materials may have to be removed from the tape by mechanical or other means. If no other choice is possible, it may be necessary to reheat the container of immersed tape to permit extracting it, but recognize that such reheating may adversely bias test results by causing the coated metal(s) to behave in a manner not encountered during a normal cable life. Recorded laboratory data should include descriptions of any special techniques used to extract tape specimens.

10.2 Allow the specimen to cool to room ambient conditions.

10.3 Visually examine (normal vision or corrected-tonormal vision, without magnification) the specimen of coated metal tape for evidence of delamination.

11. Interpretation of Results

11.1 Unless otherwise specified, consider any visual evidence of delamination of coated metal to be a failure.

11.2 *Report*—The report shall be in accordance with Section 23.

COMPATIBILITY EVALUATIONS FOR POLYOLEFIN INSULATIONS AND FILLING OR FLOODING COMPOUNDS

12. Scope

12.1 This test method is specifically for polyolefin insulating materials used in telecommunications wires and cables.

13. Specimen Preparation and Initial Testing

13.1 Obtain samples of typical insulated conductors for evaluation.

13.1.1 Where various insulated conductor sizes are available for evaluation, test the smallest conductor size.

13.1.2 Unless otherwise permitted by the product specification, evaluate white insulation. (White insulation is normally heavily loaded and is common to most telecommunications wire and cable products.) Evaluate the other basic telecommunications wire and cable colors when required by the product specification or when otherwise deemed necessary.

13.1.3 Use a sample length long enough to permit testing before and after exposure to the filling or flooding materials in question.

13.2 Unless otherwise specified, cut the samples to obtain a minimum of 10 specimens for each color and material being evaluated and for each environmental condition described (see Note 1). Unless otherwise specified, a set of specimens consists of a minimum of one specimen of each color and material in each test configuration (such as straight and pigtail) being evaluated.

13.3 Select at least one specimen of each color and material under study and measure tensile strength (nominal) and percentage elongation at the break of the insulation with the conductor removed (see Test Specimen section of Test Methods D 2633 for methods of removing the conductor). Use test methods, gage marks, and initial jaw separation in accordance with Test Methods D 2633. Record results for each specimen “before aging.”

13.4 In addition to straightened specimens of insulated conductor, prepare pigtail samples of insulated conductor by wrapping individual specimens for 10 complete turns around a mandrel equal to the outside diameter of the insulated conductor. As a minimum, prepare enough pigtail samples to permit examination as specified in 15.2.

13.5 Heat a quantity of the filling or flooding compound to a temperature such that all components of the compound are in solution and the compound is a homogenous pourable liquid (98°C minimum). Maintain at a temperature higher than 102°C only if necessary for pouring. A standardized pouring temperature of $100 \pm 2^\circ\text{C}$ is preferred in order to produce uniform testing. Record melting and pouring temperatures used together with other relevant laboratory data.

13.6 Coat some (normally one half) of the specimens (both straight and pigtail) with the filling or flooding material. Dip the specimens or paint the heated and liquified material on with a brush, etc., so the outer surface is completely covered 0.001 to 0.002 in. (0.025 to 0.050 mm). Do not soak the specimens in the compound in a dipping or similar process (expose to excess quantities of compound for a matter of seconds rather than hours; see Note 3).

NOTE 3—The intent of this procedure is to provide heat aging of insulating and jacketing material in an oven and not in hot grease; hence, the prohibition against soaking as described in 13.6 and 18.5 of this test method.

14. Specimen Aging

14.1 Place the insulated conductor specimens, coated and uncoated, into an oven preheated to the temperature mutually agreed upon between the cable manufacturer and the purchaser. Unless otherwise specified, maintain this temperature for a period of 672 h (28 days) minimum. See Note 1 and Note 4.

NOTE 4—Testing of insulations and jackets may be continued beyond the 28 day minimum period to obtain more data. For long term testing (up to a year of aging), interim examinations at monthly intervals are recommended. Interim intervals beyond 1 year of aging may be lengthened appropriately.

15. Specimen Retest and Examination

15.1 At the conclusion of the test period, remove the sets of specimens (both straight and pigtail) from the ovens. Using a paper towel or a clean dry cloth (no solvents), gently wipe the specimens to remove as much of the filling or flooding material as possible without unduly stressing the specimens. Do not straighten the pigtail specimens during this cleaning process. Allow them to cool to room ambient conditions.

15.2 Visually examine (normal or corrected-to-normal vision, without magnification) the pigtail specimen(s) of insulated conductor for evidence of cracking.

15.3 Test the heat aged specimens of straight insulated conductors (coated and uncoated) as in 13.3. Record the results for each specimen after aging, and calculate the percentage difference between “before” and “after” test results.

16. Interpretation of Results

16.1 Unless otherwise specified, consider visual evidence of cracking in pigtail specimens to be a failure.

16.2 Except as noted in 16.1, Pass or Fail interpretations of insulation compatibility evaluations are as agreed upon between the parties involved, and based upon the determination of percentage retention of properties at the end of heat aging at the agreed-upon temperature.

16.3 *Report*—The report shall be in accordance with Section 23.

COMPATIBILITY EVALUATIONS FOR POLYOLEFIN JACKETING COMPOUNDS AND FILLING AND FLOODING COMPOUNDS

17. Scope

17.1 This test method is specifically for polyolefin jacketing materials used in telecommunications wires and cables.

18. Specimen Preparation and Initial Testing

18.1 Obtain typical samples of the particular jacketing material to be evaluated. Perform tests on slab samples of jacketing material prepared from the granular or pelletized raw materials, or on samples of actual extruded jackets removed from wires or cables (provided that such jackets were not exposed to filling or flooding materials). Unless otherwise specified, prepare slab samples in accordance with Procedure C of Practice D 1928. Use slabs or jacket samples large enough to permit all required testing both before and after aging to the filling or flooding materials in question. Use molded plaques 0.045 in. (1.14 mm) minimum to 0.080 in. (2.03 mm) maximum in thickness.

18.2 Cut the samples to obtain the necessary number of specimens for each material evaluated and for each environmental condition described. Unless otherwise specified, prepare a minimum of three specimens per material per condition.

18.3 Select specimens of each material under study and measure tensile strength (nominal) and percentage elongation at break of jacket material. Use test methods, gage marks, and initial jaw separation in accordance with Practice D 2633. Record results for each specimen before exposure.

18.4 Heat a quantity of the filling or flooding compound to a temperature such that all components of the compound are in solution and the compound is a homogenous pourable liquid (98°C minimum). Maintain at a temperature higher than 102°C only if necessary for pouring. A standardized pouring temperature of $100 \pm 2^\circ\text{C}$ is preferred in order to produce uniform testing. Record melting and pouring temperatures used together with other relevant laboratory data.

18.5 Coat some (normally one half) of the jacket specimens with the filling or flooding material. Coat only one side of the jacket material. Apply the filling or flooding material using a spatula, or paint the heated and liquified material on with a brush, etc., so the outer surface is completely covered 0.001 to 0.002 in. (0.025 to 0.050 mm). Do not soak the specimens in the compound. Expose to excess quantities of compound for a matter of seconds rather than hours (see Note 3). Since the specimen is not immersed, the problem here lies in obtaining coating that will stay on the specimens during the subsequent heat aging (see Note 5).

NOTE 5—Flow characteristics of filling and flooding materials will differ. The bulk of some filling or flooding materials may tend to flow off the specimens with specimens suspended at elevated temperatures. In such instances, continue the test, recognizing that only a thin surface coating may remain and this affects the specimens only minimally. Alternatively, lay the coated specimens in a shallow dish rather than suspending them, recognizing that such a configuration represents an excessively severe test. Record all such conditions in the test report.

19. Specimen Heat Aging

19.1 Place the material specimens, coated and uncoated, into an oven preheated to the temperature mutually agreed upon between the cable manufacturer and the purchaser. Hang the specimens near the center of the oven. Unless otherwise specified, maintain this temperature for a period of 672 h (28 days) minimum. See Note 1 and Note 4.

20. Specimen Retest and Examination

20.1 At the conclusion of the test period, remove the sets of specimens from the oven. Using a paper towel or a clean dry cloth (no solvents), gently wipe to remove as much of the filling or flooding material as possible from each specimen. Allow the specimen(s) to cool to room ambient conditions.

20.2 Visually examine (normal vision or corrected-to-normal vision, without magnification) the cooled specimen(s) of jacketing material for evidence of swelling or distortion.

20.3 Test each specimen as in 18.3. Record the results for each specimen after aging, and calculate the percentage difference between before and after test results.

21. Interpretation of Results

21.1 Unless otherwise specified, consider visual evidence of obvious and excessive swelling or distortion of jacket material to be a failure.

21.2 Except as noted as 21.1, pass or fail interpretations of jacket compatibility evaluations are as agreed upon between the parties involved, and are based upon the determination of percentage retention of properties at the end of heat aging at the agreed upon temperature.

21.3 *Report*—The report shall be in accordance with Section 23.

22. Projection of Data

22.1 As mentioned in Note 1 and Note 4, plotting of interim data at a given temperature is useful to establish trends at that temperature for insulations or jackets and filling or flooding materials under study.

22.2 As suggested in Note 1, plotting data at various test temperatures on a common base may make it possible to make life prediction estimates for other temperatures for given insulations or jackets exposed to particular filling or flooding materials.

23. Report

23.1 Report the following information:

23.1.1 Identification of all materials undergoing test,

23.1.2 Date of preparation of test specimens,

23.1.3 Total number of specimens of each type prepared,

23.1.4 Procedures used to prepare test specimens, as appropriate,

23.1.5 Melting temperatures used to liquify filling or flooding materials, or both,

23.1.6 Special conditions used for heat aging of jacket specimens,

23.1.7 Oven temperatures used,

23.1.8 Oven conditioning periods, if other than specified herein,

23.1.9 A record of visual observations made and of tensile and elongation data recorded,

23.1.10 Acceptance criteria used for evaluation, and

23.1.11 Evaluation finding (pass, fail, or other).

24. Precision and Bias

24.1 The precision and bias of tensile and elongation measurements specified in these test methods are as specified in Test Methods D 2633.

24.2 No statement is made about either the precision or the bias of these test methods for making visual observations or for the comparison of before and after test results since the results merely state whether there is conformance to the criteria for success specified in the procedure.

25. Keywords

25.1 compatibility; filling compound; flooding compound; polyolefin coated metals; polyolefin insulation; polyolefin jacket

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