~~Designation:D4565–99 (Reapproved 2004)ε1~~ Designation: D4565 – 10

An American National Standard

Standard Test Methods for Physical and Environmental Performance Properties of Insulations and Jackets for Telecommunications Wire and Cable[1]

This standard is issued under the fixed designation D4565; 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—Non-mandatory language was replaced throughout editorially in November 2004.~~

1. Scope*

1.1 These test methods cover procedures for the physical testing of thermoplastic insulations and jackets used on telecommunications wire and cable and the testing of physical characteristics and environmental performance properties of completed products. To determine the procedure to be used on the particular insulation or jacket or on the completed wire or cable, reference should be made to the specification for that product.

1.2 The test methods appear in the following sections of this standard:

~~1.3The values stated in inch-pound units are to be regarded as the standard except where only SI units are given.~~

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical

[1] These test methods are under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and are the direct responsibility of Subcommittee D09.18 on Solid Insulations, Non-Metallic Shieldings and Coverings for Electrical and Telecommunication Wires and Cables.

Current edition approved ~~Nov. 4, 2004.~~May 1, 2010. Published June ~~1999.~~2010. Originally approved in 1986. Last previous edition approved in ~~1994~~2004 as ~~D4565–94~~D4565–99(2004)ε1. ~~DOI: 10.1520/D4565-99R04E01.~~ DOI: 10.1520/D4565-10.

***A Summary of Changes section appears at the end of this standard.**

conversions to SI units that are provided for information only and are not considered standard, except where only SI units are given.

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.* For specific caution statement see 19.1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D471 Test Method for Rubber PropertyEffect of Liquids
D638 Test Method for Tensile Properties of Plastics
D1238 Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer
D1248 Specification for Polyethylene Plastics Extrusion Materials for Wire and Cable
D1693 Test Method for Environmental Stress-Cracking of Ethylene Plastics
D2633 Test Methods for Thermoplastic Insulations and Jackets for Wire and Cable
D3032 Test Methods for Hookup Wire Insulation
D4731 Specification for Hot-Application Filling Compounds for Telecommunications Wire and Cable
D4732 Specification for Cool-Application Filling Compounds for Telecommunications Wire and Cable
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E171 Specification for Atmospheres for Conditioning and Testing Flexible Barrier Materials

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *air core*—products in which the air spaces between cable core components (pairs, etc.) remain in their unfilled or natural state.

3.1.2 *armored wire or cable*—a wire or cable in which the shielded or jacketed or shielded and jacketed wire or cable is completely enclosed by a metallic covering designed to protect the underlying telecommunications elements from mechanical damage.

3.1.2.1 *Discussion*—Shielding or armoring, or both may be selected from a variety of materials (for example, aluminum, copper, steel) and may be applied in a variety of ways (for example, helically wrapped, longitudinally applied, applied corrugated or smooth).

3.1.3 *cable, telecommunications*—products of six or more pair.

3.1.4 *DOD*—an abbreviation for "Diameter over Dielectric." This is a short term to refer to the overall diameter over an insulated conductor.

3.1.5 *filled core*—those products in which air spaces are filled with some materials intended to exclude air or moisture, or both.

3.1.6 *gopher-resistant*—a wire or cable that resists the attack of gophers when installed directly buried.

3.1.6.1 *Discussion*—Telecommunications wire and cable products intended for direct burial in the earth are normally rated as either "gopher-resistant" or "non-gopher-resistant." User selection of products for burial will depend upon the anticipated gopher protection needed for the planned installation site. The gopher-resistant rating is assigned based upon test evaluations (evaluations are commonly performed by the Fish and Wildlife Service, US Department of the Interior, Denver, CO).

3.1.7 *non-gopher-resistant*—a wire or cable that is not designed to resist gopher attack (see 3.1.6).

3.1.8 *pair*—two insulated conductors combined with a twist.

3.1.9 *sheath*—the jacket and any underlying layers of shield, armor, or other intermediate material down to but not including the core wrap.

3.1.10 *shielded wire or cable*—a wire or cable in which the core (or inner jacket) is completely enclosed by a metallic covering designed to shield the core from electrostatic or electromagnetic interference.

3.1.11 *wire, telecommunications*—products containing less than six pair.

DIMENSIONAL MEASUREMENTS OF INSULATIONS, JACKETS, MISCELLANEOUS CABLE COMPONENTS AND COMPLETED CABLES

4. Scope

4.1 Dimensional measurements include, but are not limited to measurements of insulation and jacket thicknesses, tape and armor thicknesses, conductor diameters, DODs, core diameters, overall diameters, etc.

5. Significance and Use

5.1 Dimensional measurements, properly interpreted, provide information with regard to the conductors, insulation, or jacket.

[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.

The dimensional measurements provide data for research and development, engineering design, quality control, and acceptance or rejection under specifications.

6. Diameters

6.1 Measure diameters of essentially round items (such as insulated or uninsulated conductors) using any type of micrometer reading to at least 0.001 in. (0.025 mm) with each division of a width that facilitates estimation of each measurement to 0.0001 in. (0.0025 mm). Take a minimum of two readings, essentially at right angles to each other, and average the results.

6.2 In case of dispute, optical methods as described in Test Methods D3032 shall be used as the referee method.

NOTE 1—For insulated conductors with dual insulation (for example, foam-skin), the DOD of the inner layer must be measured using the optical methods of Test Methods D3032.

6.3 Measure the approximate or effective diameters of non-circular cross sections (such as, irregular or oval cables or cable cores) by the use of strap gauges.

6.4 *Precision and Bias*—The precision and bias of this method for measuring diameters are in accordance with Test Methods D2633.

7. Thicknesses

7.1 Measure insulation thickness using appropriate methods specified in Test Methods D2633, except that the micrometer accuracy described in 6.1 is required. A pin gauge having the accuracy of the micrometers as specified in 6.1 is acceptable for thickness measurements made on tubular sections of insulation removed from conductors. Optical methods (as specified in 6.2) are also permitted.

7.2 Measure jacket thickness using appropriate methods specified in Test Methods D2633, except that the micrometer accuracy specified in 6.1 is required. In determining the thickness of jackets applied over corrugated shields or armors, measurements must be made in the corrugation impressions (thinnest jacket spots). Optical methods (as specified in 6.2) are also permitted.

7.3 *Precision and Bias*—The precision and bias of this method for measuring thickness are in accordance with Test Methods D2633.

NOTE 2—For designated purposes (such as, process control, etc.), continuous uniformity thickness gauges or measuring devices may be employed during processing to provide running records of jacket thicknesses. Record charts are normally maintained for a minimum of six months.

8. Eccentricity

8.1 Calculate eccentricity using measured thickness values for insulation or jacket, or both.

8.2 Calculate absolute eccentricity, E_{ab}, of insulation or jacket, or both as follows:

$$(1) \quad Eab = (\text{Maximum Thickness}) - (\text{Minimum Thickness})$$

8.3 Calculate percent eccentricity, $E_{\%}$, of insulation or jacket, or both as follows:

8.4 *Precision and Bias*—The precision and bias of this method of measuring eccentricity are in accordance with Test Methods D2633.

9. Cross-Sectional Areas

9.1 When needed, determine cross-sectional areas (usually insulations or jackets only) using the methods outlined in Test Methods D2633, except that the dimensions used in the calculations must be maintained to the accuracy specified in 6.1.

9.2 *Precision and Bias*—The precision and bias of this method for measuring cross-section areas are as specified in Test Methods D2633.

PHYSICAL AND ENVIRONMENTAL TESTS OF INSULATIONS AND JACKETS

10. Scope

10.1 Physical and environmental tests for insulations and jackets include, but are not limited to determination of some or all of the properties covered in Sections 12-25.

11. Significance and Use

11.1 Physical tests, properly interpreted, provide information with regard to the physical properties of the insulation or jacket. The physical test values give an approximation of how the insulation will physically perform in its service life. Physical tests provide data for research and development, engineering design, quality control, and acceptance or rejection under specifications.

12. Melt Flow Rate Change—Polyolefin Materials

12.1 *Raw Material Baseline*—Melt flow rate for insulation and jacket materials obtained from finished cable must be compared with the flow rates for corresponding raw materials. Determine the flow rates for the basic insulating and jacketing raw materials in accordance with the requirements of Test Method D1238. Standard conditions of test shall be as prescribed by the product

specification. If possible, obtain samples of raw materials before or during the extrusion process (but *not* after heating). Since insulating and jacketing raw materials are normally obtained and used in bulk, it is usually difficult if not impossible to relate a particular lot of raw material with a particular reel of finished wire or cable; accordingly, average raw materials values shall be established as necessary for an appropriate manufacturing time frame, unless otherwise agreed upon between the producer and the purchaser.

12.2 *Insulation Material*—Perform tests on insulation removed from finished conductors. Note that thin wall and fine gauge insulations shall be handled carefully because of entrapped air. In the case of insulation in filled cable, the preferred method is to obtain insulating material from conductors before they are exposed to the filling operation. If necessary, conductors obtained from completed filled cable shall be wiped dry and free of grease or foreign material using a dry cloth (without solvent). Chop the insulation, stripped from a conductor, as necessary to obtain specimens suitable for testing (approximately 3 g of material is required for each test). Test the chopped material as required by Test Method D1238 to determine a melt flow rate. Run three tests and average the results. Standard conditions of test shall be as indicated in 12.1.

12.3 *Jacket Material*—Jacket material used for this test must be free of filling or flooding compound. Soft filling or flooding compounds shall be removed by thoroughly wiping the jacket specimen using a clean dry cloth (without solvent); harder filling or flooding compounds shall be removed by cutting. Buffing is permitted to be used as a finishing operation to ensure clean and dry specimens. Use jacketing material removed from completed cable for performing tests. Chop the jacket material removed from the cable as is necessary to obtain specimens suitable for testing (approximately 3 g of material is required for each test). Test the chopped material as required by Test Method D1238 to determine a melt flow rate. Run three tests and average the results. Standard conditions of test shall be as indicated in 12.1.

12.4 *Calculation*—Calculate the percent increase in flow rate as follows:

where:

I = increase, %,
M_1 = melt index of raw material, and
M_2 = melt index of material from the finished cable.

12.5 *Precision and Bias*—The precision and bias of this method for measuring melt-flow rate changes are basically in accordance with Test Method D1238.

13. Tensile and Elongation Tests

13.1 *Insulation Material*—Provide test specimens by removing insulation from finished conductors. (See Test Specimen section of Test Methods D2633 for methods of removing the conductor.) Perform tests in accordance with Test Method D638 to determine such properties as tensile strength (nominal), yield strength, and percentage elongation at break. The speed of testing shall be as prescribed by the product specifications.

13.2 *Jacket Material*—Provide test specimens by die cutting jacket segments removed (cut) from finished cable. Perform testing in accordance with Test Method D638 to determine such properties as tensile strength (nominal), yield strength and percentage elongation at break. The speed of testing shall be as prescribed in the product specifications.

13.3 *Precision and Bias*—The precision and bias of these methods for measuring tensile and elongation properties of insulations and jackets are in accordance with Test Method D638.

14. Insulation and Jacket Shrinkback (Oven Test)

14.1 *Insulation Material*—Perform tests on insulated conductors. Unless otherwise specified, test a minimum of one sample of each color of insulation from a cable. Immediately prior to testing, cut specimens 8 in. (200 mm) long from the center of a 5-ft (1.5-m) length; then reduce them to 6 in. (150 mm) by trimming each end of the specimen. Place these specimens in a forced air type circulating oven or in a forced convection type circulating air oven for 4 h at the temperature prescribed. The specimens shall be placed on a layer of preheated talc or felt. At the end of the conditioning period, cool the wire to room temperature and measure the shrinkback of the insulation. Shrinkback is defined as the total shrinkage of the insulation from both ends of the specimen in inches (or millimetres).

14.2 *Jacket Material*—Perform tests on slabs cut from the cable jacket. Unless otherwise specified, cut a minimum of four test specimens, each 2 in. (51 mm) long, 0.25 in. (6.3 mm) wide, and the same thickness as the jacket. Make the lengthwise cuts parallel to the longitudinal axis of the cable with each specimen spaced circumferentially in 90° increments around the cable periphery. For cables that are longitudinally shielded or armored, one of the specimens shall be cut from a portion of the jacket lying directly over the outer shield or armor overlap. Place these specimens on a layer of preheated talc or felt in a forced-air type circulating oven or in a forced-convection type circulating air oven for 4 h at the temperature prescribed. At the end of the conditioning period, cool the specimens to room temperature and measure the shrinkback of the jacket material. Shrinkback is defined as the total lengthwise shrinkage in inches (or millimetres).

14.3 *Precision and Bias*—No statement is made about either the precision or bias of these methods for measuring shrinkback since the result merely states whether there is conformance to the criteria for success specified in the product specification.

15. Insulation Shrinkback (Solder Test)

15.1 Test specimens of finished insulated conductor for solder shrinkback. Unless otherwise specified, test a minimum of one

specimen of each insulation color. Immediately prior to testing, cut 8-in. (200-mm) specimens from the center of a 5-ft (1.5-m) length and then reduce each specimen to 6 in. (150 mm) by trimming each end of the specimen. Using any convenient method, strip 0.5 in. (13 mm) of insulation from one end of the specimen. Using a solder pot maintained at a temperature of approximately 320 °C, immerse the bared conductor to a depth of 0.25 in. (6 mm) into the molten solder and hold for a period of 20 s. Remove the specimen and measure the amount of insulation shrinkback occurring as a result of the heat exposure. Shrinkback in inches (or millimetres) is the total measured length of the bared conductor minus the original length of the bared conductor (0.5 in. (13 mm)).

15.2 *Precision and Bias*—No statement is made about either the precision or bias of this method for measuring shrinkback since the result merely states whether there is conformance to the criteria for success specified in the product specification.

16. Cold Bend (Insulation Only)

16.1 Tests shall be performed on insulated conductors. The insulation shall not show any cracks visible by normal or corrected-to-normal vision, when a specimen of insulated conductor that has been subjected to the specified temperature for 1 h, upon removal from the cooling chamber, is immediately wound around a mandrel at least six adjacent turns. Test temperature and mandrel diameter shall be as prescribed by the product specification. Bending shall be at an approximately uniform rate so that the time consumed is not more than 1 min.

16.2 *Precision and Bias*—The precision of these tests has not been determined. No statement can be made about the bias of this method for insulation cold bend since a standard material is not available.

17. Oxidative Induction Time (Polyolefin Insulation Only)

17.1 *Scope*—This method covers the determination of an Oxidative Induction Time (OIT) value for polyolefin insulation materials removed from completed wire or cable products. This OIT value is determined by a thermoanalytical measurement of the onset time for the exothermic oxidation of insulation in pure oxygen, at a specified temperature. For commentary and additional information on the background, development, and significant details of this test procedure, see Appendix X1.

17.2 *Summary of Test Method*—This method describes the instrument calibration procedures, sample preparation, experimental procedure, and calculation methods for determining OIT values for polyolefin insulation materials. An insulated wire sample is removed from a completed cable/wire product and wiped to remove filling compounds that are present in the completed cable/wire. Two types of insulation test samples are described: *Type I*—Insulation stripped from wire (no copper present), or *Type II*—Insulation on the wire (insulation and copper conductor).

17.2.1 Use Type I samples to measure the intrinsic stability of the material and the efficacy of thermal stabilizers such as antioxidants.

17.2.2 Use Type II samples to evaluate not only the thermal stability, but also the metal deactivation efficacy of the additives.

17.3 *Significance and Use*:

17.3.1 The OIT value measures the oxidative thermal stability of a material and is primarily dependent on:

17.3.1.1 the intrinsic thermal stability of the material,

17.3.1.2 the type and concentration of antioxidants and other thermal stabilizers present,

17.3.1.3 the type and concentration of metal deactivators present, and

17.3.1.4 the test temperature.

17.3.1.5 *Discussion*—Other components in the insulation material may cause secondary effects. The OIT value for an insulation may be significantly altered by additives such as pigments, fillers, and processing aids as well as catalyst residues from the cable, wire, insulation, or resin manufacture. The OIT value may increase or decrease depending on whether these additives and residues act as oxidation inhibitors or promoters at the test temperature. At typical test temperatures (for example, 170 to 220 °C), compounds present in the polyolefin material may decompose and change the polyolefin oxidation mechanism and thereby the OIT value. If the oxidation mechanism is so altered, then the OIT value may not correlate to aging at normal use temperatures. Before using the OIT value to predict field performance and lifetimes, additional studies may be required to establish a correlation between the OIT value measured at high temperature and the performance of the polyolefin under typical field conditions.

17.3.2 The OIT value is useful as a product performance test, quality control parameter, or a research and development tool for polyolefin materials.

17.4 *Apparatus, Reagents and Materials*:

17.4.1 *Calorimeter*—This OIT Test is performed using commercial analyzers known as Differential Scanning Calorimeters (DSC) which measure heat flow as a function of time and temperature. A DSC with isothermal control and specimen temperature precision of at least ± 0.1 °C is required.

NOTE 3—This test requires accurate temperature and atmosphere control in the DSC specimen compartment. DSC manufacturers offer choices in cell configuration and temperature control parameters that may affect this required control. For example, in some power compensation DSCs, use of the two-hole platinum specimen holder lids with a special "flow-through" swing-away block cover is required. Therefore, the user may wish to consult equipment-specific literature and with the equipment manufacturer to optimize the operation of individual DSCs for this test.

17.4.2 *Nitrogen*—Use cylinder nitrogen (99.9 % purity or better) for purging of cells.

17.4.3 *Oxygen*—Use cylinder oxygen (99.9 % purity or better) during the oxidation stage.

NOTE 4—Do not use house gases that are piped throughout buildings since their purity may vary significantly.

17.4.4 *Pans*—Standard aluminum DSC pans (6 mm in diameter) are required to hold specimens during testing.

NOTE 5—Do not use copper pans because the variable oxidation state of the copper leads to imprecision in determination of the OIT value. Do not use metal screens (for example stainless steel mesh) since they can be pro- or anti-oxidants and may reduce precision and accuracy of the OIT measurement.

17.4.4.1 *Degreasing*—To degrease pans, wash in Reagent Grade acetone for 1 min and dry in a stream of dry nitrogen. Use sufficient acetone to thoroughly wash the pans, that is, ~200 mL/100 pans. Ultrasonic cleaning of the pans in acetone is acceptable.

17.4.5 *Temperature Standards*—Use pure (>99.9 %) indium and tin as temperature calibration standards. See Table 1.

17.4.6 *Balance*—An analytical balance to weigh specimens with a sensitivity of ±0.1 mg or better.

17.5 *Instrument Calibration*

17.5.1 *Instrument Preparation*—Clean instrument cells between testing of different material formulations. Follow the instrument manual procedure for cleaning cells or hold the cells at 530 °C for 10 min in oxygen.

17.5.2 *Temperature Calibration*—Follow the instrument manual procedures for temperature calibration of the instrument using the following heating programs and calibration criteria.

17.5.2.1 *Indium*—The experimental sequence for the indium calibration is

(1) Equilibrate at 50 °C (in nitrogen).
(2) Heat at 10 °C/min from 50 °C to 145 °C.
(3) Heat at 1 °C/min from 145 °C to 165 °C.
(4) Cool specimen to below 50 °C.
(5) Repeat steps (*1*) through (*4*).
(6) Use melting temperatures and heat of fusion from second scan for calibration purposes.

17.5.2.2 *Tin*—The experimental sequence for the tin calibration is

(1) Equilibrate at 50 °C (in nitrogen).
(2) Heat at 10 °C/min from 50 °C to 220 °C.
(3) Heat at 1 °C/min from 220 °C to 240 °C.
(4) Cool specimen to below 50 °C.
(5) Repeat steps (*1*) through (*4*).
(6) Use melting temperatures and heat of fusion from second scan for calibration purposes.

17.5.2.3 *Melting Temperature*—For calibration purposes, define the melting temperature as the extrapolated onset of the melting peak, not the peak maximum (see Fig. 1).

17.5.3 *Calibration Criteria*—An instrument in calibration will validate the melting temperatures of pure indium and pure tin at 156.6 ± 0.2 °C and 232.0 ± 0.5 °C, respectively. In addition, the heat of fusion for indium and tin will be 28.7 ± 0.8 J/g and 60.7 ± 2.0 J/g, respectively. Check the instrument calibration every one to two months or more frequently since this test requires accurate temperature control. (See Note 3.)

17.5.4 *Gas Flow Rate*—Use an oxygen flow rate of 50 ± 5 mL/min as measured with a bubble meter or calibrated rotameter. Other flow rates between 50 and 200 mL/min are permitted, but must be reported.

NOTE 6—It is desirable that the tubing connecting the gas switching point and the calorimeter cell have an inside volume less than 20 mL.

NOTE 7—The average OIT value at 100 mL/min was ~3 % lower than the OIT measured at 50 mL/min. OIT values determined at 100 mL/min had ~5 % improved precision over OIT values obtained at 50 mL/min.

17.5.5 *Test Temperature*—If possible, run a blank specimen to ensure that the instrument can maintain the test temperature within ± 0.3 °C. Heat the cell to the desired test temperature (typically 200 °C) and monitor the specimen temperature for 10 min. If necessary, refer to 17.7.6 for procedural strategies to make the measured specimen temperature equal to the desired test temperature.

17.6 *Sample Preparation*

17.6.1 *Insulated Wire Sample*—Remove the insulated wires from completed wire or cable products by removing the outer cable sheath, inner metallic shields, and any core wraps. Split the outer cable sheath lengthwise, and peel open the sheath and any metallic shields to reveal the inner core with the insulated wire pairs.

17.6.2 *Sample Cleaning*—Wipe the insulated wire sample with a clean cotton cloth or paper towel to remove any filling compound. Do not use solvents to clean the insulated wire.

17.6.3 *Sample Type*—Determine the OIT value for an insulation using either a: *Type I sample*—Insulation stripped from the copper wire (see 13.1), or *Type II sample*—Insulated wire (insulation and copper wire).

TABLE 1 Literature Values for Calibration Standards[A]

Calibration Standard	Melting Temperature, °C T_m	Heat of Fusion (J/g) ΔH_m
Indium (In)	156.61	28.7
Tin (Sn)	232.0	60.7

[A] Rossini, F. D., *Applied Chemistry, Vol 22 1970, p. 557; Gronwold, F., Acta Chem. Scand.* Vol 21, 1967, p. 1695; Gronwold, F, *J. Therm. Analysis*, Vol 13, 1978, p. 419; Gronwold, F., *Pure and Applied Chemistry*, 1992.

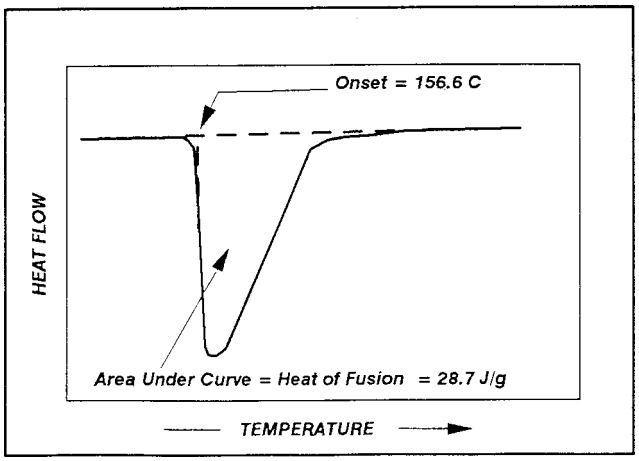

FIG. 1 Indium Calibration

17.6.4 *Specimen/Pan Arrangements*—Use a single 5 to 6-mm long specimen of insulation (or insulated wire). The length is such that the specimen fits neatly into the pan. (See Fig. 2).

17.6.5 *Specimen Weight*—Record the specimen weight to ±0.1 mg.

NOTE 8—To determine the insulation sample weight, strip a 100-mm section of the insulated wire and weigh the stripped insulation. Divide the insulation weight by the sample length to determine the insulation weight per mm (W_i). Multiplying the specimen length (5 to 6 mm) by this factor (W_i) will give the weight for the insulation specimen.

17.7 *Procedure*

17.7.1 *Load Specimens*—Place the specimen (specimen and pan) in the specimen position and an empty aluminum pan in the reference position of the instrument.

17.7.2 *Initial Temperature*—Equilibrate the specimen at or below 60 °C.

17.7.3 *Flush Cell*—Hold at this initial temperature for 5 min while the nitrogen purge flushes the cell at a flow rate of ~50 to 60 mL/min.

17.7.4 *Heat to Test Temperature*—Heat at 20 °C/min to the test temperature (typically 200 °C) with nitrogen gas purging the DSC cell.

NOTE 9—The endothermic peak observed during this heating stage is the melting transition of the polyolefin and can be used for identification (for example, to distinguish between high-density polyethylene, low-density polyethylene, and polypropylenes).

17.7.5 *Gas Switch*—Hold at test temperature for 5 min to establish thermal equilibrium after which switch from the nitrogen purge to pure oxygen at a flow rate of 50 ± 5 mL/min. Define this switch time as T_0. Measure the Oxidative Induction Time (OIT) from this time (T_0).

17.7.6 *Specimen Test Temperature*—If possible, record the specimen temperature 5 min after T_0 with a precision of ± 0.1 °C or better. The specimen temperature must be within ± 0.3 °C of the desired test temperature. If this temperature is more than ± 0.3 °C from the required test temperature, prepare a new specimen and modify the temperature program to ensure OIT measurement is made at the required temperature.

NOTE 10—If 200.0 °C was the desired test temperature and the temperature at $T_0 + 5$ min was 200.7 °C, then set the upper limit of the temperature program to 199.3 °C to correct for the overshoot of the instrument. Alternatively, monitor and adjust the specimen temperature continuously during the experiment to maintain the desired temperature within ± 0.3 °C.

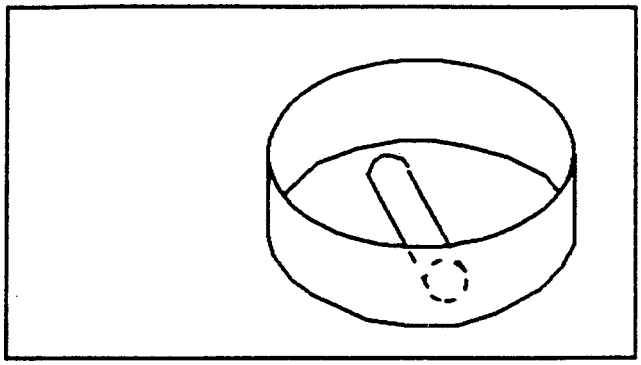

FIG. 2 Specimen/Pan Arrangement

17.7.7 *Specimen Scan*—Continue the test in pure oxygen until the exothermic peak is observed (on the chart recorder or computer screen).

17.7.8 *Data Collection*—Plot the data normalized as heat flow (W/g) versus time. Expand the *x*-axis as much as possible to facilitate analysis. Vary the *y*-axis depending on the procedure used to determine the OIT (see 17.8).

17.8 *OIT Calculation*—Use either of the following two procedures to determine the Oxidative Induction Time (OIT) values for the specimens.

NOTE 11—The OIT_1 calculation uses a threshold measure to define the incipient point for the polyolefin oxidation. The OIT_2 calculation defines the onset of the major exothermic reaction (that is, the autocatalytic oxidation reaction).

17.8.1 *Procedure 1—OIT_1 (Offset Method):*

17.8.1.1 Plot data with a full scale *y*-axis of 1.0 W/g (or smaller). (See Fig. 3.)

17.8.1.2 Expand the *x*-axis so that full scale on the *x*-axis ranges from $T_0 - 2$ min to 5 to 10 min past the onset of the oxidation exotherm. This expansion helps to assist in analysis by the offset procedure.

17.8.1.3 Draw an extension to the baseline extrapolating any instrument drift. For an example see dashed line (a) in Fig. 3.

17.8.1.4 Draw a second line parallel to baseline (a) at a distance of 0.05 W/g above the baseline. See dashed line (b) in Fig. 3.

17.8.1.5 The intersection of the dashed line (b) with the signal trace is defined as the onset of oxidative degradation and is denoted as T_1.

17.8.1.6 The Oxidative Induction Time by the offset procedure is defined as the time from oxygen introduction (T_0) to this onset:

17.8.2 *Procedure 2*—OIT_2 (*Tangent Method):*

17.8.2.1 Plot data with a *y*-axis sufficient to show full melting endotherm of the polyolefin and the oxidation endotherm. For a 5 mg polyolefin specimen, a *y*-axis of 4 to 5 W/g is adequate.

17.8.2.2 Draw an extension to the baseline extrapolating any signal drift. For an example see dashed line (c) in Fig. 4.

17.8.2.3 Draw a tangent (dashed line (d) in Fig. 4) at the inflection point of the exothermic peak and extend this tangent to intersect the baseline (c).

17.8.2.4 The point of intersection is the onset of oxidative degradation by the tangent method. This onset time is denoted as T_2.

17.8.2.5 The Oxidative Induction Time by the tangent procedure is defined as the time from oxygen introduction (T_0) to this onset time.

17.9 *Report*

17.9.1 Report the following information:

17.9.1.1 Melting temperatures (°C) for indium and tin together with the date of the last determination,

17.9.1.2 Heats of fusion (J/g) for indium and tin together with the date of the last determination,

17.9.1.3 Gas flow rate (mL/min),

17.9.1.4 Parameters for each specimen (stripped insulation, insulated wire, specimen mass, etc.),

17.9.1.5 Specimen temperature 5 min after gas switch to oxygen ($T_0 + 5$ min), and

17.9.1.6 OIT_1 (offset) or OIT_2 (tangent). (Unless otherwise specified by the user, the reported OIT shall be OIT_2, tangent method.)

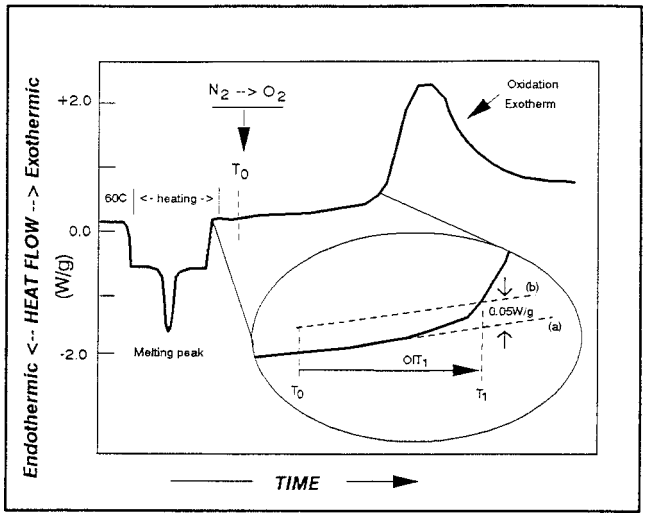

FIG. 3 OIT_1 Offset Method

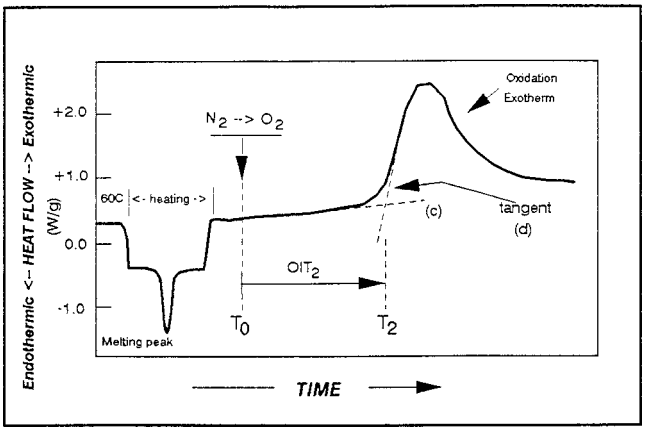

FIG. 4 OIT_2 Tangent Method

TABLE 2 Summary of Precision Data

Sample (Five specimens were run for each sample)	Mean OIT Value (min)	Repeatability Within Laboratory (σ_I)		Reproducibility Laboratory-to-Laboratory (σ_R)	
		(min)	(%)	(min)	(%)
HDPE Insulation (stripped from wire)					
OIT_1	121.6	4.5	3.7	7.3	6.0
OIT_2	126.4	2.8	2.2	7.2	5.7
HDPE Insulation (with copper wire)					
OIT_1	62.6	6.2	10.0	10.0	16.0
OIT_2	69.5	3.6	5.2	8.6	12.3

17.9.1.7 If multiple specimens are tested, report average OIT values and standard deviations.

17.10 *Precision and Bias*

17.10.1 *Precision*—The precision of this test method for measuring Oxidative Induction Time, using Type I and Type II samples, is illustrated in Table 2. These statistics were determined from round robin studies between thirteen laboratories using both heat-flux and power-compensated thermal analyzers. All data and reports of the task force that developed this method are on file at ASTM.[3]

NOTE 12—The task force summarized its finding in a paper by V. J. Kuck published in the *Proceedings of the 6th International Conference on Plastics in Telecommunications* (Pub. Plastics & Rubber Institute, London, England), September, 1992.

17.10.2 *Bias*—The test for oxidative induction time has no bias since it is defined in terms of this method.

NOTE 13—This test method employs indium and tin as internal standards for calibration of temperature and caloric sensing, and requires strict control of the test conditions to increase precision and hopefully to reduce the bias in the OIT measurement. However, as is mentioned in 17.3 and in Appendix X1, materials which are in the polyolefin may decompose at the high temperatures used, causing a shift of the OIT from the value for the polyolefin. Such a shift is important in the use of this method for quality control of the polyolefin compound. It is important to recognize that the same shift is a bias when the test is used to measure the OIT of the polyolefin.

18. Oxygen Induction Time (Cable Filling Compound Only)

18.1 *Scope*—This method covers a procedure for determining, by thermal analysis, the oxidative induction time of filling compound removed from completed wire or cable.

NOTE 14—For additional information on wire and cable filling compounds, refer to Specifications D4731 and D4732.

18.2 *Apparatus, Reagents and Materials*:

18.2.1 This test is normally performed using commercial devices commonly referred to as Differential Scanning Calorimeters (DSC) or as Differential Thermal Analyzers (DTA). Use of another apparatus is permitted if it is demonstrated to yield comparable results. The following reagents and materials are also required to perform this test:

[3] ~~Perkin Elmer's Differential Scanning Calorimeters and TA Instruments Differential Thermal Analyzer with a DSC cell have been found to produce acceptable results. Equivalent equipment producing comparable results may be used.~~

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D09-1034.

18.2.1.1 Use commercial cylinder nitrogen for purging instrument cells.

18.2.1.2 Use oxygen in this method equal to or better than 99.6 % extra dry grade.

18.2.1.3 Small specimen pans are required to hold the specimens while in the instrument cells. Pans shall be aluminum.

18.2.1.4 Use No. 316 stainless steel screen (40 mesh) to cover specimens in the pans.

18.2.1.5 Use pure metal standards such as indium, tin, lead, or zinc for instrument calibrations as recommended by the instrument manufacturer.

18.3 *Sample Preparation*—Select at random a short-length section, approximately 1 ft (300 mm) long, of completed wire or cable. Remove the core from the wire or cable section; accomplish this by pulling or pushing the core out without cutting the jacket. Remove the filling compound from the core and isolate it from the cable or wires so as not to contaminate the compound. Obtain all samples of filling compound by taking them from manufactured cable or wire rather than by obtaining them in an unprocessed condition.

18.4 *Instrument Preparation*—Clean the instrument cells after they have been standing overnight and between the testing of different material formulations. To clean the cells, bring them up to temperature and hold them at approximately 400 °C for a period of 10 min in nitrogen.

18.5 *Instrument Calibration*—Adjust temperature scales according to instrument manual instructions until the determined melting point of pure indium metal is indicated as 156.6 °C at a heating rate of 5 °C/min.

NOTE 15—This note on calibration is written specifically for the instruments described in footnote 10. Other equipment may be equally suitable but may yield slightly different results when testing identical specimens. For the Perkin-Elmer DSC, run several pure metal standards (such as, indium, tin, lead, zinc) through their melting point at a heating rate of 5 °C/min. Plot melting temperatures and interpolate to find the required set point. Repeat calibrations require only adjustments for the indium melt temperature.

For the DuPont DTA with a DSC Cell, set the instrument in the isothermal mode and calibrate the starting-temperature dial according to the instrument manual. Alternately, the dial may be set to a reading that results in a corrected thermocouple read-out of the required temperature.

18.6 *Preparation*—Place 3 to 5 mg of the filling material to be tested into an aluminum pan and cover this with a clean stainless steel screen. Crimp the pan to hold the screen in place.

18.7 Place the prepared specimen pan in the instrument cell. Flush the cell for 5 min using cylinder nitrogen at a flow rate of 200± 25 cm^3/min. Following the nitrogen purge, increase the cell-specimen temperature (at a heating rate of 10 °C/min) from the initial temperature to 190 ± 2 °C. Once temperature equilibrium of 190 °C has been reached (steady recorder signal), switch to oxygen flow at the same flow rate and simultaneously start the time base recording.

18.8 Record the start of the oxygen injection as time" zero." Maintain the isothermal temperature of 190 °C in the pure dry oxygen atmosphere until the oxidative reaction exotherm appears on the thermogram (see Fig. 5). When the test is completed, turn off the recorder, switch the gas back to nitrogen, and allow the cell temperature of the instrument to cool to ambient temperature. Remove and discard the pan and specimen.

18.9 On the recorder chart, draw an extension to the recorded base line beyond the oxidative reactive exotherm. Extrapolate the slope of the oxidative reactive exotherm to intercept the extended base line. The oxidative induction time (OIT) is measured to within ±1 min from zero time to the intercept point.

18.10 *Precision and Bias*—This test method is based on the exotherm obtained when the filling compound degrades. As such, precision of the test method is strongly dependent on the extent to which the filling compound under test is degraded. Since no calculation of OIT is possible, a comparison between the measured and the true values cannot be achieved.

19. Insulation Adhesion

19.1 Test specimens of finished insulated conductor for insulation adhesion. Prepare specimens by first trimming insulated wire specimens to 5 in. (130 mm) in length. Remove the insulation (by progressively removing short sections) from one end of the wire until only a 1-in. (25-mm) length of undisturbed insulation remains at the other end of the specimen. **Warning**—Exercise great

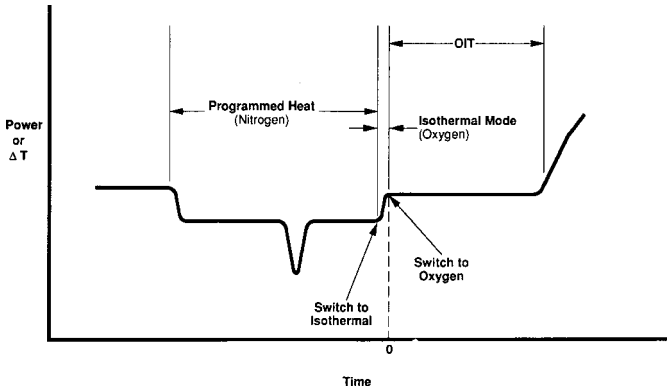

FIG. 5 Evaluation of Oxidative Induction Time (OIT) from Recorded–Time–Base Thermogram