Designation: D4565 – 20

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.

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, make reference to the specification for that product.

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

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1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific warning statement see 19.1.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D471 Test Method for Rubber Property—Effect 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

[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 D09.07 on Electrical Insulating Materials.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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/E171M Practice for Conditioning and Testing Flexible Barrier Packaging

3. Terminology

3.1 *Abbreviations:*

3.1.1 *DOD*—Diameter Over Dielectric. This is a short term to refer to the overall diameter over an insulated conductor.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *air core*—products in which the air spaces between cable core components (pairs, and so forth) remain in their unfilled or natural state.

3.2.2 *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.

3.2.2.1 *Discussion*—Select shielding or armoring, or both, from a variety of materials (for example, aluminum, copper, steel). The armoring is applied in a variety of ways (for example, helically wrapped, longitudinally applied, applied corrugated or smooth).

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

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

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

3.2.5.1 *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).

3.2.6 *non-gopher-resistant*—a wire or cable that is not designed to resist gopher attack (see 3.2.5).

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

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

3.2.9 *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.

3.2.10 *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, and so forth.

5. Significance and Use

5.1 Dimensional measurements, properly interpreted, provide information with regard to the conductors, insulation, or jacket. 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 test 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 test method for measuring thickness are in accordance with Test Methods D2633.

NOTE 2—For designated purposes (such as process control, and so forth), continuous uniformity thickness gauges or measuring devices are

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:

$$E_{ab} = (\text{maximum thickness}) - (\text{minimum thickness}) \quad (1)$$

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

$$E_{\%} = \frac{(\text{maximum thickness}) - (\text{minimum thickness})}{(\text{average thickness})} \times 100\,(\%) \quad (2)$$

8.4 *Precision and Bias*—The precision and bias of this test 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 test 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:

$$I = \frac{M_2 - M_1}{M_1} \times 100 \quad (3)$$

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 test 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 test 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 test 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 Preferably conduct the test in the cold chamber. 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 was conditioned to the specified temperature for 1 h, and wrapped around the mandrel in the cooling chamber 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 When the mandrel is too large for the chamber, place the sample in the low temperature chamber for the specified temperature for 1 h, and upon removal from the low temperature chamber, immediately wind around the mandrel for the specified number of 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.

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

17. Oxidative Induction Time (Polyolefin Insulation Only)

17.1 *Scope*—This test 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 test 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:

17.2.1 *Type I*—Insulation stripped from wire (no copper present), or

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

17.2.2 *Type II*—Insulation on the wire (insulation and copper conductor).

17.2.2.1 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*—Potentially, other components in the insulation material cause secondary effects. The OIT value for an insulation has the potential to 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 increases or decreases 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 have the potential to decompose and change the polyolefin oxidation mechanism and thereby the OIT value. If the oxidation mechanism is so altered, then the OIT value will not necessarily correlate to aging at normal use temperatures. Before using the OIT value to predict field performance and lifetimes, it is suggested that additional studies be undertaken 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 (DSCs) (Note 3) 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—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.

NOTE 4—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 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. 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 5—Do not use house gases that are piped throughout buildings since their purity varies significantly.

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

NOTE 6—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 act as pro- or anti-oxidants and have the potential to 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 to 145 °C.
(3) Heat at 1 °C/min from 145 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 to 220 °C.
(3) Heat at 1 °C/min from 220 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

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 Chemica Scandinavica,* Vol 21, 1967, p. 1695; Gronwold, F, *Journal of Thermal Analysis,* Vol 13, 1978, p. 419; Gronwold, F., *Pure and Applied Chemistry,* 1992.

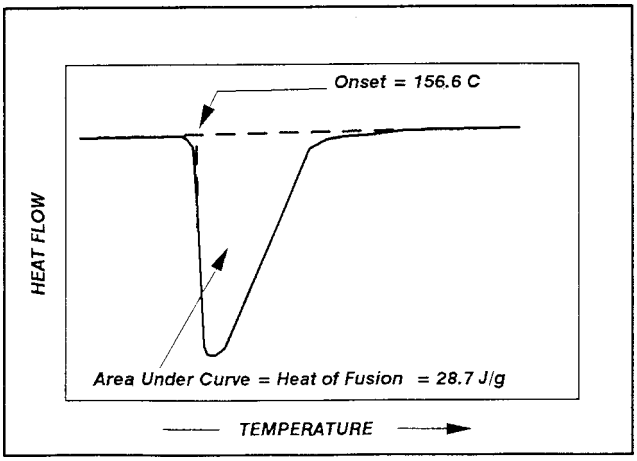

FIG. 1 Indium Calibration

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 4.)

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

NOTE 8—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:

17.6.3.1 *Type I Sample*—Insulation stripped from the copper wire (see 13.1), or

17.6.3.2 *Type II Sample*—Insulated wire (insulation and copper wire).

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).

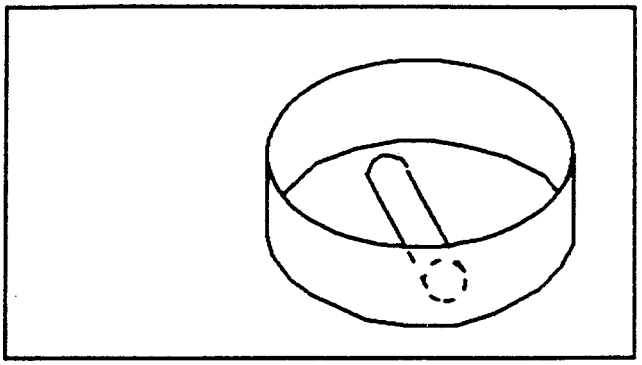

FIG. 2 Specimen/Pan Arrangement

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

NOTE 9—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 10—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 11—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.

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 12—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:

$$OIT_1(\text{offset}) = T_1 - T_0 \quad (4)$$

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.

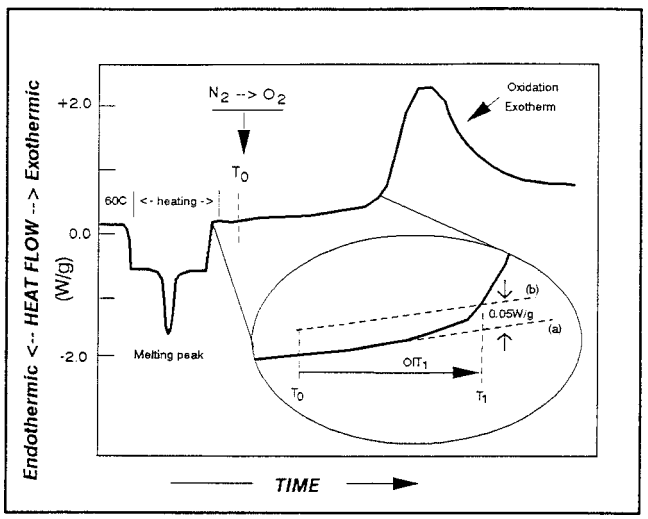

FIG. 3 OIT_1 Offset Method

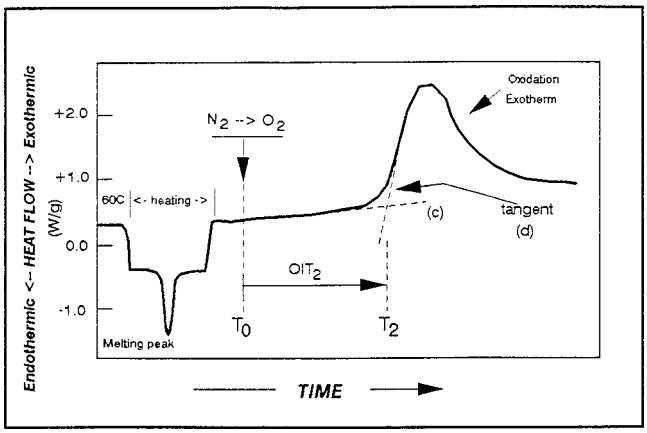

FIG. 4 OIT_2 Tangent Method

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).

TABLE 2 Summary of Precision Data

Sample[A]	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

[A] Five specimens were run for each sample

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.

$$OIT_2\ (\text{tangent}) = T_2 - T_0 \quad (5)$$

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, and so forth),

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.)

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