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STANDARDS

Interface Practices Subcommittee

SCTE STANDARD

SCTE 251 2018

Test Procedure for Determining the Thermal Oxidative Stability of Foamed Polyethylene

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1. Introduction

1.1. Executive Summary

When attempting to place standardized performance values on a product, it is necessary to also provide standardized test methods to ensure repeatability of measurements. This document is intended to provide test method details and implementation of ASTM D4565 for testing of foamed cable dielectric materials.

1.2. Scope

This method covers the determination of an Oxidative Induction Time (OIT) value for coaxial cable, foamed polyethylene, and insulation materials removed from completed cable products. This test procedure is based on the ASTM D4565. The OIT value is determined by a thermo-analytical measurement of the onset time for the exothermic oxidation of insulation in pure oxygen, at a specified temperature.

1.3. Benefits

This document is designed to benefit manufacturers and end users of product tested to this procedure by supplying a standardized method for determining an Oxidative Induction Time (OIT) value for coaxial cable, foamed polyethylene, and insulation materials removed from completed cable products.

1.4. Intended Audience

This document is intended for anyone desiring to make industry standard OIT measurements of coaxial cable, or for anyone acquiring product purported to have been tested using this method.

1.5. Areas for Further Investigation or to be Added in Future Versions

None.

2. Normative References

The following documents contain provisions, which, through reference in this text, constitute provisions of this document. At the time of Subcommittee approval, the editions indicated were valid. All documents are subject to revision; and while parties to any agreement based on this document are encouraged to investigate the possibility of applying the most recent editions of the documents listed below, they are reminded that newer editions of those documents might not be compatible with the referenced version.

2.1. SCTE References

- No normative references are applicable.

2.2. Standards from Other Organizations

- No normative references are applicable.

2.3. Published Materials

- No normative references are applicable.

3. Informative References

The following documents might provide valuable information to the reader but are not required when complying with this document.

3.1. SCTE References

- ANSI/SCTE 74 2011 Specification for Braided 75 Ohm Flexible RF Coaxial Drop Cables

3.2. Standards from Other Organizations

- ASTM D4565

3.3. Published Materials

- No informative references are applicable.

4. Compliance Notation

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shall not	This phrase means that the item is an absolute prohibition of this document.
forbidden	This word means the value specified shall never be used.
should	This word or the adjective "*recommended*" means that there may exist valid reasons in particular circumstances to ignore this item, but the full implications should be understood and the case carefully weighted before choosing a different course.
should not	This phrase means that there may exist valid reasons in particular circumstances when the listed behavior is acceptable or even useful, but the full implications should be understood and the case carefully weighed before implementing any behavior described with this label.
may	This word or the adjective "*optional*" means that this item is truly optional. One vendor may choose to include the item because a particular marketplace requires it or because it enhances the product, for example; another vendor may omit the same item.
deprecated	Use is permissible for legacy purposes only. Deprecated features may be removed from future versions of this document. Implementations should avoid use of deprecated features.

5. Abbreviations and Definitions

5.1. Abbreviations

OIT	oxidative induction time
DSC	digital scanning calorimeter

ANSI	American National Standards Institute
ISBE	International Society of Broadband Experts
SCTE	Society of Cable Telecommunications Engineers

5.2. Definitions

Trishield	Cable with three outer conductor shielding layers.
Quadshield	Cable with four outer conductor shielding layers.

6. General

Two types of insulation test samples are described. Type II sample description is presented for information only. Type I samples are to be used:

- *Type I*—Insulation removed from cable (no copper present) (Recommended method).
  - Use Type I samples to measure the intrinsic stability of the material and the efficacy of thermal stabilizers such as antioxidants.
- *Type II*—Insulation on the wire (insulation and copper conductor).
  - Use Type II samples to evaluate not only the thermal stability, but also the metal deactivation efficacy of the additives.

Significance and Use:

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

- the intrinsic thermal stability of the material,
- the type and concentration of antioxidants and other thermal stabilizers present,
- the type and concentration of metal deactivators present, and the test temperature.

Discussion—Other components in the foam material may cause secondary effects. The OIT value for insulation may be significantly altered by contamination or 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, ANSI/SCTE 74 requires 180 °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.

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

7. Apparatus, Reagents and Materials

Calorimeter—This OIT Test is performed using commercial analyzers known as Differential Scanning Calorimeters (DSC) similar to the Perkin Elmer DSC8000 or equivalent which measures 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. See **Note 1**

Nitrogen—Use cylinder nitrogen (99.9% purity or better) for purging of cells. See **Note 2**

Oxygen—Use cylinder oxygen (99.9% purity or better) during the oxidation stage. See **Note 2**

Pans—Standard aluminum DSC pans (6 mm in diameter) are required to hold specimens during testing. The pans should be open and not sealed. See **Note 3**

Degreasing—To degrease pans, wash in reagent grade acetone for 1 minute and dry in a stream of dry nitrogen. Use sufficient acetone to thoroughly wash the pans, that is, 200mL/100 pans. Ultrasonic cleaning of the pans in acetone is acceptable. In addition, it is also acceptable to use a muffle oven at 120 °C for 12 hours.

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

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

Table 1 - Literature Values for Calibration Standards

Calibration Standard	Melting Temperature, °C T_m	Heat of Fusion (J/g) ΔH_m
Indium (In)	156.61	28.7
Tin (Sn)	232.00	60.7

8. Instrument Calibration

Instrument Preparation—Clean instrument cells between testing of different material formulations. Follow the instrument manual procedure for cleaning cells or hold the cells at 700 °C for 10 minutes in oxygen. Care must be taken to remove the aluminum pan from both the sample and reference position.

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

Indium—The experimental sequence for the indium calibration is:

1. Equalize at 50 °C (in nitrogen).
2. Heat at 10 °C/minute from 50 °C to 145 °C.
3. Heat at 1 °C/minute 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.

Tin—The experimental sequence for the tin calibration is:

1. Equalize at 50 °C (in nitrogen).
2. Heat at 10 °C/minute from 50 °C to 220 °C.
3. Heat at 1 °C/minute 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.

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

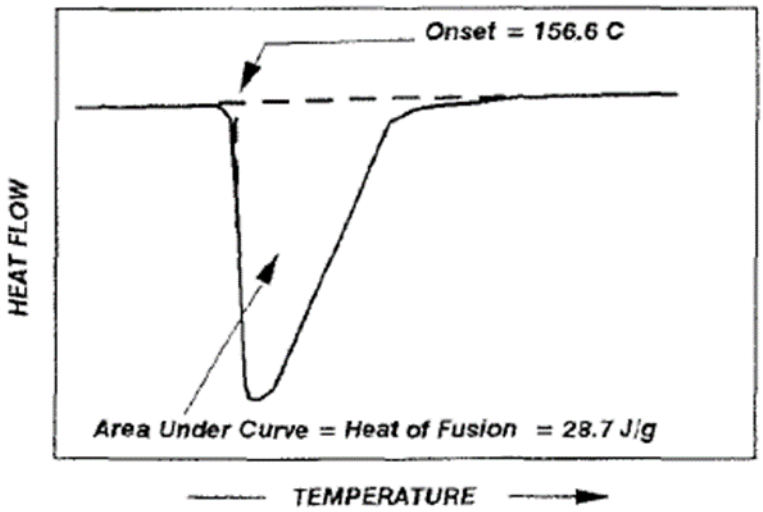

Figure 1 - Indium Calibration

Calibration Criteria—An instrument in calibration will validate the melting temperatures of pure indium and pure tin at 156.6 °C ± 90 °C ± 2 °C and 232.0 °C ± 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. The instrument calibration shall be conducted within two months, or more frequently, of any test using this procedure since this test requires accurate temperature control. (**See Note 1**)

Gas Flow Rate—Use an oxygen flow rate of 50 ± 5 mL/min as measured with a mass flow meter electronically, with a bubble meter or calibrated rotameter. Other flow rates between 50 and 200 mL/min are permitted, but must be reported. (**See Note 4 and 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 180 ° C and monitor the specimen temperature for 10 minutes. If necessary, refer to **Note 8** for procedural strategies to make the measured specimen temperature equal to the desired test temperature.

9. Sample Preparation

Foamed polyethylene Insulation Sample—

Samples from prototype run including only the center conductor, precoat and insulation- Remove the insulation from a 3-foot (*91.4* cm) sample and split the dielectric lengthwise, and carefully remove the dielectric from the center conductor. Consideration should be given to carefully removing the precoat from the dielectric with a razor blade capable of highly accurate cutting. ***Every effort should be made to prevent contamination of the insulation with oils from fingers, oils on the cutting blade or cutting surface.***

Samples- Cut 6 inches (15.2 cm) from the 3-foot sample and age for 14 days at 90 °C ± 90 °C ± 2 °C in a circulating air oven. Take three samples from the remaining dielectric material, one from each end and one from the middle.

For the aged sample, the test specimen shall be taken from within 1/4 inch of the end of the aged sample.

Samples from a finished production cable- Remove the insulation from a 3-foot (91 cm) sample of completed cable by removing the jacket, braid, Trishield and Quadshield tapes (if any). Split the dielectric and shielding tape lengthwise, and carefully remove it from the center conductor. Carefully remove the shielding tape from the dielectric with a razor blade capable of highly accurate cutting. Consideration should be given to carefully removing the precoat from the dielectric with a razor blade capable of highly accurate cutting. Every effort should be made to prevent contamination of the insulation with oils from fingers, oils on the cutting blade or cutting surface.

Samples- Cut 6 inches (15.2 cm) from the 3-foot sample and age for 14 days at 90 °C ± 90 °C ± 2 °C in a circulating air oven. Take three samples from the remaining dielectric material, one from each end and one from the middle.

For the aged sample, the test specimen shall be taken from within 1/4 inch of the end of the aged sample.

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

Sample Type—Determine the OIT value for an insulation using a: Type I sample—Insulation stripped from the copper or copper clad steel wire.

Specimen/Pan Arrangements—Use a single 5 to 6-mm long specimen of insulation. The length is such that the specimen fits neatly into the pan.

Specimen Weight—Record the specimen weight to ±0.1mg. Each specimen weight should be within 1 mg of the other specimens in the series with a target weight of 5 mg. (**See Note 6**)

10. Test Procedure

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

Initial Temperature—equalize the specimen at 40 °C ± 0.3 °C.

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

Heat to Test Temperature—Heat at 20 °C/minute to the test temperature of 180 °C ± 0.3 °C with nitrogen gas purging the DSC cell. (**See Note 7**)

Gas Switch—Hold at test temperature for 5 minutes to establish thermal equilibrium after which, switch from the nitrogen purge to pure oxygen at a flow rate of 50 ± 5mL/min. Define this switch time as T0. Measure the Oxidative Induction Time (OIT) from this time (T0).

Specimen Test Temperature—If possible, record the specimen temperature 5 minutes after T0 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. (**See Note 8**)

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

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 8.0**).

11. OIT Calculation

Use either of the following two procedures to determine the Oxidative Induction Time (OIT) values for the specimens. (**See Note 9**)

Procedure 1 (Recommended Method)—OIT1 (Tangent Method):

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.

Draw an extension to the baseline extrapolating any signal drift. For an example see dashed line (c) in Figure 2.

Draw a tangent (dashed line (d) in Figure 2) at the inflection point of the exothermic peak and extend this tangent to intersect the baseline (c).

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

The Oxidative Induction Time by the tangent procedure is defined as the time from oxygen introduction (T0) to this onset time.

OIT_1= (tangent) T_2 -T_0

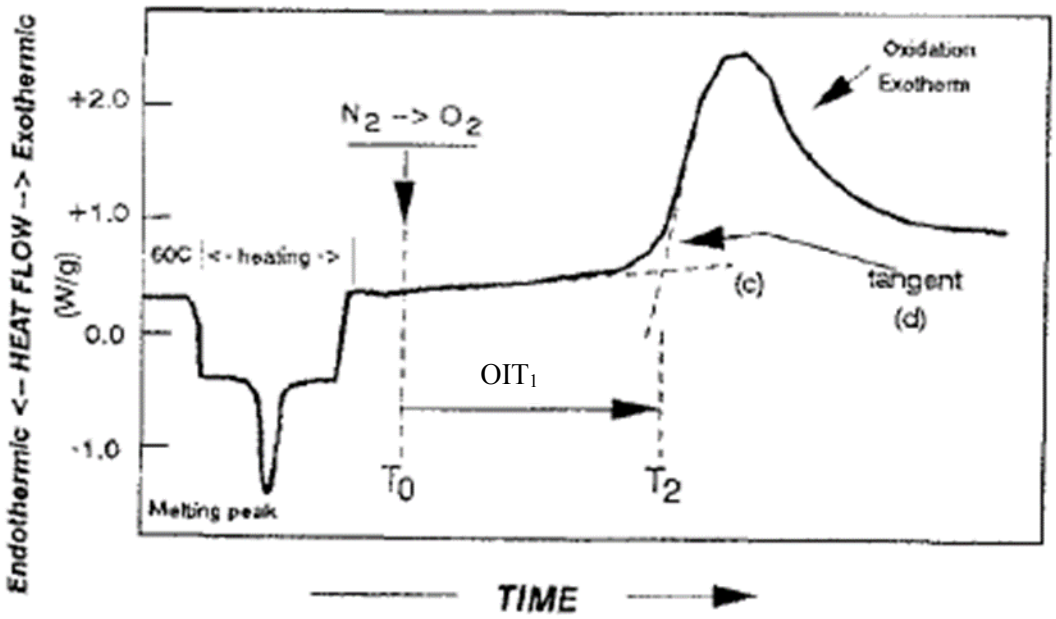

Figure 2 - OIT_1, Tangent Method

Procedure 2—OIT2 (Offset Method):

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

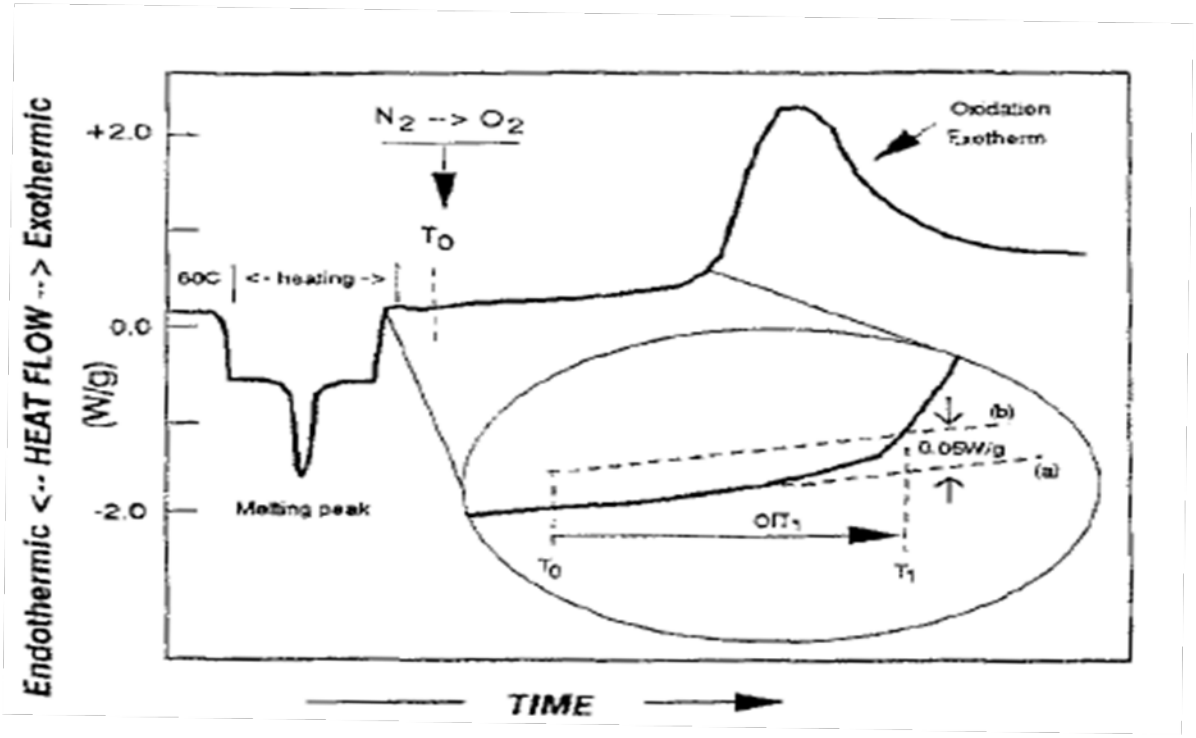

Figure 3 - OIT_2, Offset Method

Expand the x-axis so that full scale on the x-axis ranges from T0 minus 2 minutes, to 5 to 10 minutes past the onset of the oxidation exotherm. This expansion helps to assist in analysis by the offset procedure.

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

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

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.

The Oxidative Induction Time by the offset procedure is defined as the time from oxygen introduction (T0) to this onset:

OIT_2= (offset) $T_1 - T_0$

12. Report

Report the following information:

- Date of test
- Name of technician conducting test
- Part number of cable which the insulation was removed from (if available),

- HDPE material part number and percentage used in foam mixture (if available),
- LDPE material part number and percentage used in foam mixture (if available),
- Nucleating package part number and percentage used in foam mixture (if available),
- Melting temperatures (°C) for indium and tin together with the date of the last calibration,
- Heats of fusion (J/g) for indium and tin together with the date of the last calibration,
- Gas flow rate (mL/min), Note that sample was taken from a specimen of stripped insulation (unless otherwise tested),
- Specimen temperature 5 min after gas switch to oxygen (T_0 + 5 min), and OIT1 (tangent) or OIT2 (offset). (Unless otherwise specified by the requestor, the reported OIT shall be OIT1, tangent method.)
- If multiple specimens are tested, report average OIT values and standard deviation.
- Report final results each test – [(OITi – OITf) / OITi] * 100, where OITi = value before aging, OITf = value after aging. Pass criterion is ≥ 70% of initial value per ANSI/SCTE 74 2011.

13. Notes

NOTE 1: 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.

NOTE 2: Do not use house gases that are piped throughout buildings since their purity may vary significantly.

NOTE 3: 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 poor anti-oxidants and may reduce precision and accuracy of the OIT measurement.

NOTE 4: It is desirable that the tubing connecting the gas switching point and the calorimeter cell have an inside volume less than 20 mL.

NOTE 5: The average OIT value when measured at 100mL/min flow rate was about 3% lower than the OIT measured at 50mL/min. OIT values determined at 100mL/min had about 5% improved precision over OIT values obtained at 50mL/min.

NOTE 6: 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 (Wi). Multiplying the specimen length (5 to 6 mm) by this factor (Wi) will give the weight for the insulation specimen.

NOTE 7: 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).

NOTE 8: Assuming that 180.0°C was the desired test temperature and the temperature at T_0 + 5 min was 180.7°C, then set the upper limit of the temperature program to 179.3°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.

NOTE 9: The OIT_1 calculation uses a threshold measure to define the incipient point for the foamed polyethylene oxidation. The OIT_2 calculation defines the onset of the major exothermic reaction (that is, the autocatalytic oxidation reaction).

NOTE 10: 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, 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.