



Designation: D5885/D5885M – 17

# Standard Test Method for Oxidative Induction Time of Polyolefin Geosynthetics by High-Pressure Differential Scanning Calorimetry<sup>1</sup>

This standard is issued under the fixed designation D5885/D5885M; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers a procedure for the determination of the oxidative induction time (OIT) of polyolefin geosynthetics using high-pressure differential scanning calorimetry.

1.2 The focus of the test is on geomembranes, but geogrids, geonets, geotextiles, and other polyolefin-related geosynthetics are also suitable for such evaluation.

1.3 This test method measures the oxidative induction time associated with a given test specimen at a specified temperature and pressure.

1.4 This is an accelerated test for highly stabilized materials. It is applicable only to material whose OIT values under 3.4 MPa of oxygen is greater than 30 min at 150 °C.

1.5 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.6 *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.* Specific precautionary statements are given in Section 8.

1.7 *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.*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D35 on Geosynthetics and is the direct responsibility of Subcommittee D35.02 on Endurance Properties.

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## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

D3895 Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry

D4439 Terminology for Geosynthetics

D4491/D4491M Test Methods for Water Permeability of Geotextiles by Permittivity

D4565 Test Methods for Physical and Environmental Performance Properties of Insulations and Jackets for Telecommunications Wire and Cable

D4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets

E473 Terminology Relating to Thermal Analysis and Rheology

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E967 Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers

G88 Guide for Designing Systems for Oxygen Service

## 3. Terminology

### 3.1 Definitions:

3.1.1 For definitions of terms related to geosynthetics, refer to Terminology D4439.

3.1.2 Definitions of terms applying to thermal analysis appear in Terminology E473.

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *differential scanning calorimetry (DSC), n*—a technique in which the difference in heat flow inputs into a substance and a reference material is measured as a function of temperature or time, while the substance and reference material are subjected to a controlled-temperature program. (See Terminology E473.)

3.2.2 *geomembrane, n*—an essentially impermeable geosynthetic composed of one or more synthetic sheets. (See Terminology D4439.)

<sup>2</sup> 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.

3.2.2.1 *Discussion*—In this test method, essentially impermeable means that no measurable liquid flows through a geosynthetic when tested in accordance with Test Methods D4491/D4491M.

3.2.3 *geosynthetic, n*—a planar product manufactured from polymeric material used with soil, rock, earth, or other geotechnical engineering-related material as an integral part of a man-made project, structure, or system. (See Terminology D4439.)

3.2.4 *high-pressure differential scanning calorimetry (HPDSC), n*—differential scanning calorimetry in which the substance and reference material are exposed to a controlled superambient atmosphere.

3.2.5 *index test, n*—a test procedure that may be used to establish an order for a set of specimens with respect to the property of interest.

3.2.6 *oxidative induction time (OIT), n*—the elapsed time between first exposure to an oxidizing gas and the onset to oxidation of a material under isothermal conditions.

3.2.6.1 *Discussion*—Oxidative induction time is an index test parameter dependent upon a wide range of experimental conditions including temperature, pressure of oxygen, purge gas flow rate, and the presence or absence of catalysts.

## 4. Summary of Test Method

4.1 The specimen to be tested is heated from room temperature at a constant rate in a non-purging, high-pressure oxygen environment at a defined pressure. When the specified temperature has been reached, the specimen is then held at that temperature until the oxidative reaction is displayed on the thermal curve. The OIT is the time interval from the start of the temperature program test to the onset of the oxidative reaction.

4.2 In this procedure, an elevated pressure of oxygen is used to accelerate the reaction and to reduce analysis time.

4.3 Unless otherwise specified, the temperature used in this test method shall be 150 °C, and the chamber pressure is to be maintained at 3.4 MPa [500 psi] using a constant volume test condition.

## 5. Significance and Use

5.1 The oxidative induction time is a characteristic of a compounded polyolefin product that is dependent not only on the type and amount of additives present, but also on the type of resin. In well-behaved systems, this test method can be used as a quality control measure to monitor the stabilization in geosynthetics as received from a supplier.

5.2 When this test method is used to compare different geomembrane formulations containing different antioxidant packages, then those results shall be considered valid only at the temperature of test.

5.3 This test method is intended as a geosynthetic test. Use of the OIT value to estimate the lifetime of the geomembrane from which the test specimen is taken is not addressed, nor shall it be used for this purpose.

5.3.1 The OIT measurement is an accelerated thermal aging test and, as such, interpretation of resulting data may be

misleading if done by an inexperienced operator. Caution should be exercised in data interpretation since oxidation reaction kinetics are a function of temperature and the properties of the additives contained in the geosynthetic sample. For example, OIT values are often used to select optimum resin formulations. Certain antioxidants, however, may generate poor OIT results even though they may be adequate at their intended use temperature and vice versa.

5.4 This test method can be used for other purposes such as manufacturing control and research and development.

5.5 Oxidation induction time is strongly dependent upon test temperature and the partial pressure of oxygen. The higher the test temperature or the oxygen partial pressure, or both, the shorter the oxidation induction time.

5.5.1 The use of high test temperature, however, may have deleterious effects. The first of these is the potential volatilization of additive packages used to stabilize the test materials. The second is the potential for the influence of chemical mechanisms which are not significant at end-use operation conditions.

5.5.2 This test method uses high oxygen pressure to accelerate the test period while making use of lower test temperatures to protect additive packages.

5.6 The results from this test method may or may not correlate with those obtained by other OIT measurements such as Test Method D3895 or Test Methods D4565.

## 6. Apparatus

6.1 *Differential Scanning Calorimeter*—Thermal analysis equipment capable of heating rates up to  $20 \pm 1$  °C/min and of automatically recording the differential heat flow between the test sample and a reference sample is necessary. The equipment must be capable of measuring sample temperature to  $\pm 1$  °C while maintaining a set temperature to  $\pm 0.5$  °C.

NOTE 1—Modern computer-based instrumentation equipped with “iso-track” modes provides adequate specimen temperature control.

6.2 *Data-Presentation Device*—A printer, plotter, recorder, or other recording output device capable of displaying heat flow on the y-axis versus time on the x-axis as output signals from differential scanning calorimeters in 6.1.

6.3 *High-Pressure DSC Cell*—A unit capable of maintaining pressure up to 3.4 MPa [500 psig]. The system shall be equipped with a pressure gage to monitor the internal pressure of the cell to permit manual release of pressure to maintain desired level.

NOTE 2—The gage shall be accurate to 2 % at 3.4 MPa [500 psig].

NOTE 3—All pressures in this test method are indicated relative to atmosphere pressure—that is, they are “gage” pressures.

6.4 *High-Pressure Oxygen Cylinder Regulator*—A pressure regulator capable of regulating a pressure up to 5.5 MPa [800 psi]. The outlet of the cylinder is to be linked to the high-pressure cell using a *clean* stainless steel tube.

6.5 *Analytical Balance*, 0.1-mg sensitivity.

6.6 *Specimen Holders*, degreased aluminum pans, 6.0 to 7.0-mm diameter.

6.7 *Core Hole Borer*, cork borer or arch punch producing 6.3-mm [0.25-in.] disks.

## 7. Reagents and Materials

7.1 All chemical reagents used in this test method shall be analytical grade unless otherwise specified.

7.2 *Hexane or Acetone*, for cleaning specimen pans and stainless steel tubing, see 8.2.

7.3 *Indium (99.999 % Purity)*, for calibration purposes, see 9.1.

7.4 *Oxygen*, purity >99.5 % for the test atmosphere.

## 8. Precautions

8.1 Oxygen is a strong oxidizer that vigorously accelerates combustion. Keep oil and grease away from equipment using or containing oxygen.

8.2 The stainless steel tube connecting the high-pressure cell to the oxygen cylinder must be thoroughly cleaned by hexane (or acetone) and then dried before being connected to the cell.

8.3 The use of pressurized oxygen requires appropriate and careful handling procedures. The user of this test method shall be familiar with the precautions described in Guide G88.

## 9. Sampling

9.1 The test sample is compression molded into sheet format (thickness of  $250 \pm 15 \mu\text{m}$ ) prior to analysis. Specimen disks (6.4-mm diameter) cut from the sheet will have a weight of approximately 5 to 10 mg, depending on sample density.

NOTE 4—If the sample requires homogenization prior to analysis, one of the procedures given in Appendix X1, Appendix X2, or Appendix X3 is recommended. Poor sample uniformity will adversely affect test precision.

NOTE 5—If the sample contains a layer or layers of polymers other than polyolefins, the polyolefin may be tested separately from the entire cross section. A recommended procedure is given in Appendix X4.

9.2 Compression mold these assembled parts into a uniform plaque to a thickness of 0.25 mm [10 mil] (see Practice D4703).

NOTE 6—The temperature at which molding takes place may be at or above the test temperature of this test method. Prolonged exposure to air at these temperatures may induce a negative bias into OIT measurement. Molding should be performed at as low a temperature and as quickly as possible to minimize this bias.

9.3 Cut test specimens from the plaque using a 6.3-mm [0.25-in.] bore hole cutter or punch.

## 10. Calibration

10.1 Using Test Method E967, temperature calibrate the differential scanning calorimeter using indium metal and a heating rate of  $1 \text{ }^\circ\text{C}/\text{min}$  from 145 to  $165 \text{ }^\circ\text{C}$ .

10.1.1 Perform the calibration step at least once a month or whenever changes have occurred in the experimental setup.

## 11. Procedure

NOTE 7—Procedures for preparing the test specimen may be different for different polyolefin geosynthetic products, for example, geomembranes, geonets, geogrids, or geotextiles.

11.1 Prepare a specimen with a mass of  $5 \pm 1 \text{ mg}$ .

11.2 Place the weighed specimen into the cleaned specimen pan.

11.3 Place the specimen and reference pans into the cell.

NOTE 8—Open pans are used in this test method.

11.4 Secure the top plate of the test chamber and tighten the cell system.

11.5 Close the pressure release valve and the outlet valve. Open the inlet and pressurize the cell to 3.4 MPa [500 psi] oxygen. Close the inlet valve.

11.6 Commence programmed heating of the specimen from ambient temperature to  $150 \text{ }^\circ\text{C}$  at a rate of  $20 \text{ }^\circ\text{C}/\text{min}$ . Zero time is taken at the initiation of the temperature program. Hold the temperature isothermally at  $150 \text{ }^\circ\text{C}$  until the oxidative exothermic peak is detected. At the same time, the thermal curve of the entire test is being recorded. Other test temperature may be used, with the agreement of all parties concerned, but must be reported.

11.7 A slight gain in pressure at the beginning of the test is anticipated as the temperature of the cell increases. Decrease the pressure back to 3.4 MPa [500 psi] by slightly opening the pressure release valve until the pressure comes down to 3.4 MPa. Alternatively, if the pressure increase with an instrument is known, pressurize the test cell to a slightly lower value so that the pressure in the cell is 3.4 MPa [500 psi] at the conclusion of the heating ramp cycle to test temperature.

NOTE 9— $150 \text{ }^\circ\text{C}$  is the temperature typically used for polyethylene-based material, and  $170 \text{ }^\circ\text{C}$  for polypropylene-based materials.

NOTE 10—The time to onset of oxidation varies with stabilizer package and may be more than 900 min. For a first-time specimen, the isothermal time period of 1000 min is suggested.

NOTE 11—The DSC cell should be cleaned by holding the cell at a temperature of  $400 \text{ }^\circ\text{C}$  for 3 min under air or oxygen atmosphere.

## 12. Analysis Response

12.1 Plot the data with the heat flow signal on the y-axis, versus time on the x-axis.

12.2 Determine the value for OIT in the following manner:

12.2.1 Plot data with a y-axis sensitivity sufficient to show the full oxidative exotherm. A full-scale sensitivity of 5 W/g is usually adequate.

12.2.2 Extend the horizontal baseline generated prior to the onset to oxidation.

NOTE 12—For the oxidation exotherm containing a small shoulder peak at the beginning of oxidation, a sigmoidal baseline may be more appropriate than the straight baseline.

12.2.3 Draw a tangent at the inflection point of the exothermic peak and extend this tangent to intersect with the baseline.

12.2.4 The time at the intersection, measured from the initiation of the temperature program from ambient temperature is the onset of oxidative degradation and is taken as the OIT value.

12.2.5 Measure the OIT as shown in Fig. 1.

12.2.6 The tangent method used to measure the oxidation time is the preferred method, but the selection of the appropriate tangent to the exotherm sloped line is, at times, difficult if

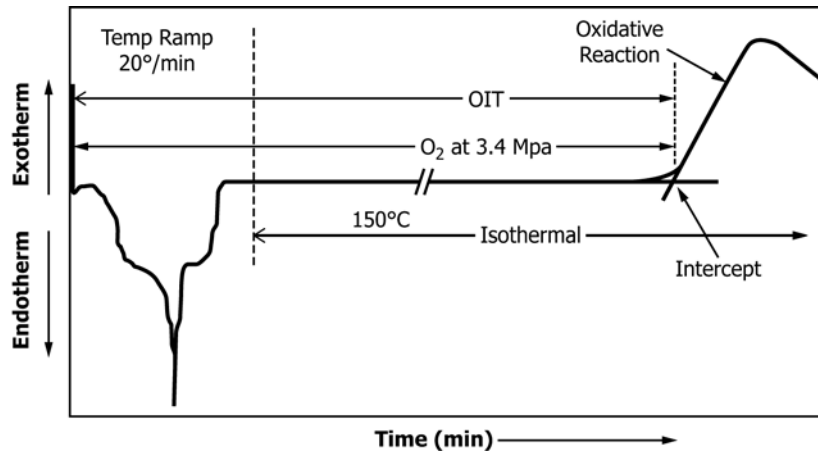


FIG. 1 Testing Temperature Curve

the exothermic peak has a leading edge. It is possible that exothermic peaks with leading edges occur if the oxidation reaction is slow.

NOTE 13—The oxidation may not occur as a smooth transition and multiple steps or slopes may be produced. When this occurs, resampling and retesting is recommended. Use of one of the homogenization methods mentioned in the appendixes of this standard may be used to obtain a homogeneous specimen and a single exothermic peak.

NOTE 14—If multiple slopes result from the oxidation process, OIT needs to be defined to accurately reflect the oxidation of the polymer. It is up to the user to determine which slope best represents the material property for an application. It must be noted in the report if the tangent line is not drawn from the first steep slope.

NOTE 15—If the selection of the appropriate baseline is not obvious using the tangent method, try the offset method. Draw a second baseline parallel to the first baseline at a distance of 0.05 W/g above the first baseline. The intersection of this second line with the exotherm signal is defined as the onset of oxidation. The time from this intersection to zero time is the OIT.

NOTE 16—Other gas-flow rates or specimen weights, or both, may be used if shown to be equivalent.

12.3 Report the OIT for a single specimen.

NOTE 17—If replicate tests are required by the parties involved, a mean value shall be calculated as being representative of the material being evaluated.

13. Report

13.1 Report the following information:

- 13.1.1 Identification of the specimen,
- 13.1.2 Mass and configuration of the test specimen,
- 13.1.3 Method of conditioning the test specimen if different from that specified herein,
- 13.1.4 The single OIT value from the test, and
- 13.1.5 The recorded pressure of oxygen during the isothermal portion of the thermal curve.

14. Precision and Bias<sup>3</sup>

14.1 An interlaboratory study using this test method was conducted in 1995–1996 using two polyethylene samples that

<sup>3</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D35-1003. Contact ASTM Customer Service at service@astm.org.

were not geosynthetic materials. Following temperature calibration with indium, each of six laboratories tested the two test specimens with four replications. Instruments from a single manufacturer were employed. The results were treated by Practice E691.

NOTE 18—Instruments supplied by TA Instruments<sup>4</sup> were used in this interlaboratory test. While attempts were made to include apparatus from other suppliers, only laboratories with equipment supplied by this vendor were willing to participate in the interlaboratory study. Other vendors may supply apparatus suitable for use in this test method. Their use is permitted provided performance is shown to be consistent with the repeatability and reproducibility described in this section.

14.2 Precision:

14.2.1 For polyethylene, two values, each the mean of duplicate determinations, should be considered suspect if they differ by more than:

$$r = 95 \% \text{ repeatability limit (within laboratory) } = 6.5 \%$$

$$R = 95 \% \text{ reproducibility limit (between laboratories) } = 25 \%$$

14.2.2 The respective standard deviations among test results, related to the above values by the factor of 2.8 are:

$$S_r = \text{repeatability standard deviation} = 2.3 \%$$

$$S_R = \text{reproducibility standard deviation} = 9.1 \%$$

14.3 Bias—The OIT measurement is an index test for which no standard reference materials are available.

14.3.1 A polyethylene sample, characterized with this standard performed at 150 °C and 3.4 MPa [500 psig] oxygen pressure, yielded a mean OIT value of 231 min with a repeatability standard deviation of 5.7 min (2.5 %) and a reproducibility standard deviation of 18 min (7.6 %).

14.3.2 The same polyethylene sample was also tested in 1993 by Section 17 of Test Methods D4565 performed at 200 °C and 10 kPa [15 psia] oxygen pressure, conditions different than those used in this test method. In that test, the

<sup>4</sup> TA Instruments, 109 Lukens Drive, New Castle, DE 19720.





mean was 31.4 min, with a repeatability standard deviation of 1.6 min (5.1 %) and a reproducibility standard deviation of 3.1 min (9.8 %).<sup>5</sup>

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D09-1034. Contact ASTM Customer Service at service@astm.org.

## 15. Keywords

15.1 differential scanning calorimetry; geogrid; geomembrane; geonets; geopipes; geosynthetics; geotextiles; oxidation; polyethylene; polyolefins; polypropylene

## APPENDIXES

### (Nonmandatory Information)

#### X1. SAMPLE HOMOGENIZATION USING A TORQUE RHEOMETER

X1.1 If desired, the test sample may be homogenized prior to OIT analysis to minimize local variations of stabilizer concentration. A torque rheometer equipped with a mixing head is suitable for such purposes.

X1.2 Each sample should be blended at 60 r/min for 10 min, using a chamber temperature of 150 to 160 °C for polyethylene resins and 180 to 190 °C for polypropylene resins. The mixing

chamber should be purged with nitrogen to suppress polymer and additive degradation.

X1.3 After a mixing time of 10 min, quench-cool the samples in liquid nitrogen or ice water. After sufficient cooling, compression mold the required amount of material into sheet format.

#### X2. SAMPLE HOMOGENIZATION USING A CRYOGENIC GRINDER

X2.1 If desired, the test sample may be homogenized prior to OIT analysis to minimize local variations of stabilizer concentration. A cryogenic grinder may be used to prepare a more homogeneous sample.

X2.2 Follow the instructions and safety precautions recommended by the manufacturer of the grinder.

X2.3 Be sure to cut the sample into small enough pieces, precool long enough, and grind long enough to create a mixture of fine particles with no residual larger chunks.

X2.4 Let the sample warm to room temperature prior to exposure to the air to prevent condensation on the sample prior to compression molding the material into a sheet.

#### X3. SAMPLE HOMOGENIZATION USING A TWO-ROLL MILL

X3.1 If desired, the test sample may be homogenized prior to OIT analysis to minimize local variations of stabilizer concentration. A small, lab two-roll mill may be used to prepare a homogeneous sample.

X3.2 Small pieces of the sample are placed on a hot, two-roll mill that is turning. Conditions are dependent on the specific material and equipment being used, and so are outside the scope of this appendix.

X3.3 Conditions should be selected that can melt and mix the sample within 90 s. A nitrogen blanket (flow of nitrogen gas onto the sample) can help reduce oxidation but is not required.

X3.4 After the sample is well mixed, quench-cool the samples in liquid nitrogen or ice water. After sufficient cooling, compression mold the required amount of material into sheet format.



#### X4. SAMPLING INDIVIDUAL LAYERS FOR ANALYSIS

X4.1 If desired, an individual layer may be separated from the sample as a whole and analyzed for HPOIT separately. One method to do that is to cut multiple thin sections or thick microtomes followed by using a sharp instrument to remove or

separate a particular layer. After an adequate amount of material is separated, it may be made into a plaque directly or be homogenized using one of the procedures in this appendix.

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