Designation: D 5885 – 97

Standard Test Method for Oxidative Induction Time of Polyolefin Geosynthetics by High-Pressure Differential Scanning Calorimetry[1]

This standard is issued under the fixed designation D 5885; 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 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 SI units are to be regarded as the standard. The values stated in parentheses are provided for information only.

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.

2. Referenced Documents

2.1 *ASTM Standards:*

D 3417 Test Method for Heats of Fusion and Crystallization of Polymers by Thermal Analysis[2]

D 3895 Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry[2]

D 4439 Terminology for Geosynthetics[3]

D 4491 Test Methods for Water Permeability of Geotextiles by Permittivity[3]

D 4565 Test Methods for Physical and Environmental Performance Properties of Insulations and Jackets for Telecommunications Wire and Cable[4]

D 4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets[5]

E 473 Terminology Relating to Thermal Analysis[6]

E 967 Practice for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers[6]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

G 88 Guide for Designing Systems for Oxygen Service[6]

3. Terminology

3.1 *Definitions:*

3.1.1 *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 E 473.)

3.1.2 *geomembrane*, *n*—an essentially impermeable geosynthetic composed of one or more synthetic sheets. (See Terminology D 4439.)

3.1.2.1 *Discussion*—In this test method, essentially impermeable means that no measurable liquid flows through a geosynthetic when tested in accordance with Test Method D 4491.

3.1.3 *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 D 4439.)

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

[1] This test method is under the jurisdiction of ASTM Committee D-35 on Geosynthetics and is the direct responsibility of Subcommittee D35.02 on Endurance Properties.

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[2] *Annual Book of ASTM Standards*, Vol 08.02.

[3] *Annual Book of ASTM Standards*, Vol 04.13.

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

[5] *Annual Book of ASTM Standards*, Vol 08.03.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

3.1.5 *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.

3.1.6 *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.

3.1.6.1 *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 and the corresponding reference material are 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 an 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 D 3895 or Test Methods D 4565.

6. Apparatus

6.1 *Differential Scanning Calorimeter*—Thermal analysis equipment capable of heating rates up to 20 ± 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 ±1°C while maintaining a set temperature to ±0.5°C.

NOTE 1—Modern computer-based instrumentation equipped with "isotrack" modes provide 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 and 8.3.

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.