



Standard Test Methods for Determining Oxidation Induction Time of Hydrocarbons by Differential Scanning Calorimetry¹

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^{ε1} NOTE—Warning notes were editorially updated throughout in May 2015.

1. Scope

1.1 These test methods describe the determination of the oxidative properties of hydrocarbons by differential scanning calorimetry or pressure differential scanning calorimetry and is applicable to hydrocarbons that oxidize exothermically in their analyzed form.

1.2 *Test Method A*—A differential scanning calorimeter (DSC) is used at ambient pressure, for example, about 100 kPa of oxygen.

1.3 *Test Method B*—A pressure DSC (PDSC) is used at high pressure, for example, 3.5 MPa (500 psig) oxygen.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 These test methods are related to ISO 11357–6 but is different in technical content. These test methods are related to CEC L-85–T but includes additional experimental conditions.

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 6.4 and 11.10.

2. Referenced Documents

2.1 ASTM Standards:²

D3350 Specification for Polyethylene Plastics Pipe and Fittings Materials

D3895 Test Method for Oxidative-Induction Time of Poly-

olefins by Differential Scanning Calorimetry

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

D5482 Test Method for Vapor Pressure of Petroleum Products (Mini Method—Atmospheric)

D5885 Test Method for Oxidative Induction Time of Polyolefin Geosynthetics by High-Pressure Differential Scanning Calorimetry

D6186 Test Method for Oxidation Induction Time of Lubricating Oils by Pressure Differential Scanning Calorimetry (PDSC)

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

E1860 Test Method for Elapsed Time Calibration of Thermal Analyzers

2.2 Other Standards:

ISO 11357–6 Plastics-Differential Scanning Calorimetry (DSC) — Part 6: Oxidation Induction Time³

CEC L-85–T Hot Surface Oxidation⁴

3. Terminology

3.1 Definitions:

3.1.1 Specific technical terms used in these test methods are given in Terminology E473.

4. Summary of Test Method

4.1 The test specimen in an aluminum pan and the reference aluminum pan are heated to a specified constant test temperature in an oxygen environment. Heat flow out of the specimen is monitored at an isothermal temperature until the oxidative

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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.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

⁴ Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, <http://www.sae.org>.

reaction is manifested by heat evolution on the thermal curve. The oxidative induction time (OIT), a relative measure of oxidative stability at the test temperature, is determined from data recorded during the isothermal test. The OIT measurement is initiated upon reaching the isothermal test temperature.

4.2 For some particularly stable materials, the OIT may be quite long (>120 min) at the specified elevated temperatures of the experiment. Under these circumstances, the OIT may be reduced by increasing the isothermal temperature or increasing the pressure of oxygen purge gas, or both. Conversely, reactions that proceed too rapidly, with a short OIT, may be extended by decreasing the test temperature or reducing the partial pressure of oxygen, or both. By admixing oxygen gas with a suitable diluent, for example, nitrogen, the OIT will be increased (see Test Methods [D3895](#), [D4565](#), [D5482](#), [D6186](#), and Specification [D3350](#)).

NOTE 1—For some systems, the use of copper pans to catalyze oxidation will reduce the oxidation induction time for a particular temperature. The results, however, will not correlate with non-catalyzed tests.

5. Significance and Use

5.1 Oxidative induction time is a relative measure of the degree of oxidative stability of the material evaluated at the isothermal temperature of the test. The presence, quantity or effectiveness of antioxidants may be determined by this method. The OIT values thus obtained may be compared from one hydrocarbon to another or to a reference material to obtain relative oxidative stability information.

5.2 Typical uses include the oxidative stability of edible oils and fats (oxidative rancidity), lubricants, greases, and polyolefins.

6. Apparatus

6.1 *Differential Scanning Calorimeter or Pressure Differential Scanning Calorimeter*, the essential instrumentation required to provide the minimum differential scanning calorimetric capability for these test methods include:

6.1.1 *DSC Test Chamber*, composed of:

6.1.1.1 *A Furnace(s)*, to provide uniform controlled heating of a specimen and reference to a constant temperature or at a constant rate within the applicable temperature range of these test methods.

6.1.1.2 *A Temperature Sensor*, to provide an indication of the specimen/furnace temperature to $\pm 0.4^\circ\text{C}$.

6.1.1.3 *Differential Sensors*, to detect a heat flow difference between specimen and reference with a sensitivity of 5 μW .

6.1.1.4 A means of sustaining a *Test Chamber Environment* of a purge gas of 50 mL/min within 5 %.

6.1.2 *Temperature Controller*, capable of executing a specific temperature program by operating the furnace(s) between selected temperature limits at a rate of temperature change of $40^\circ\text{C}/\text{min}$ constant to 1 % and an isothermal temperature constant to $\pm 0.4^\circ\text{C}$.

NOTE 2—In certain cases when the sample under study is of high volatility (for example, low molecular weight hydrocarbons), either the use of pressures in excess of one atmosphere or lower temperatures may be required. The operator is cautioned to verify (with apparatus manufac-

turer) the maximum oxygen pressure at which the apparatus may be safely operated.

6.1.3 *A Data Collection Device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for DSC are heat flow, temperature and time.

NOTE 3—The capability to record the first derivative of the heat flow curve will be helpful in cases where the baseline is not constant.

6.2 *Containers (pans, crucibles, etc.)*, that are inert to the specimen and reference materials and that are of a suitable structural shape and integrity to contain the specimen and reference materials.

6.3 For use in Test Method B.

6.3.1 *Pressure System*, consisting of:

6.3.1.1 *A Pressure Vessel*, or similar means of sealing the test chamber at any applied oxygen pressure within the pressure limits of these test methods.

6.3.1.2 A source of pressurized oxygen or air capable of sustaining a regulated gas pressure in the test chamber of up to 3.2 MPa.

6.3.1.3 *A Pressure Transducer*, or similar device to measure the pressure inside the test chamber to ± 0.2 MPa, including any temperature dependence of the transducer.

NOTE 4—The link between the test chamber and the pressure transducer should allow for fast pressure equilibrium to ensure accurate recording of the pressure above the specimen during testing.

6.3.1.4 *A Pressure Regulator*, or similar device to adjust the applied pressure in the test chamber to ± 0.2 MPa of the desired value.

6.3.1.5 *A Ballast*, or similar means to maintain the applied pressure in the test chamber constant to ± 0.2 MPa.

6.3.1.6 *Valves*, to control the gas in the test chamber or to isolate components of the pressure system.

6.4 *Flow meter*, capable of reading 50 mL/min or another selected flow rate, accurate to within ± 5 %. Ensure the flowmeter is calibrated for oxygen. Contact a supplier of flow meters for specific details on calibration, see [Note 6](#), following Section [11.4](#). (**Warning**—Use metal or fluoropolymer tubing with oxygen rather than the commonly used rubber or polyvinyl chloride plastic tubing. There have been hazardous situations with prolonged use of certain polymer tubing in oxygen service.)

NOTE 5—Gas delivery tubing should be kept as short as possible to minimize “dead” volume.

6.5 *Analytical Balance* with a capacity of at least 100 mg and capable of weighing to the nearest 0.01 mg or less than 1 % of the specimen mass.

6.6 *Specimen Containers*, and sample holders are the aluminum sample pans and should be inert to the sample and the oxidizing gas. The pans shall be clean, dry, and flat. A typical cylindrical pan has the following dimensions: height, 1.5 to 2.5 mm and outer diameter, 5.0 to 6.0 mm.

6.6.1 New sample pans shall be cleaned by the procedure found in [Annex A1](#).