Designation: E1858 – 23

Standard Test Methods for Determining Oxidation Induction Time of Hydrocarbons by Differential Scanning Calorimetry[1]

This standard is issued under the fixed designation E1858; 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 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 *Units*—The values stated in SI units are to be regarded as standard. Imperial units are provided for user convenience and are not the 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 7.4 and 12.10.

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.*

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 Polyolefins 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 and Liquid Fuels (Mini Method—Atmospheric)

D5885/D5885M 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

E3142 Test Method for Thermal Lag of Thermal Analysis Apparatus

2.2 *Other Standards:*

ISO 11357–6 Plastice-Differential Scanning Calorimetry (DSC) — Part 6: Determination of Oxidation Induction

[1] These test methods are under the direct jurisdiction of Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Calorimetry and Mass Loss.

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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.

Time (Isothermal OIT) and Oxidation Induction Temperature (Dynamic OIT)[3]
CEC L-85–T-99 Oxidative Stability of Lubricants Measured by PDSC[4]

3. Terminology

3.1 *Definitions:*

3.1.1 Specific technical terms used in these test methods are given in Terminology E473, including differential scanning calorimetry, extrapolated onset value, and exotherm.

3.2 *Oxidation Induction Time (OIT), n*—the time interval required to isothermally initiate oxidation.

3.2.1 *Discussion*—The oxidation induction time is considered a quantitative measure of oxidative stability and often as a relative indicator of anti-oxidant content.

4. Summary of Test Method

4.1 In thermal analysis, a physical property of a material is measured either as a function of time at a specified constant temperature, or more frequently, as a function of temperature under conditions of a fixed rate of temperature change. The measured property is the dependent variable and the measured temperature and time are the independent variables.

4.2 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 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.

4.3 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 D5885/D5885M, 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. Interferences

6.1 This test method involves the continuous monitoring of the specimen temperature within the test chamber's enclosed environment of a flowing, static, or self-generated gaseous atmosphere during execution of the stipulated procedure. In *DSC* apparatus, the temperature sensor utilized to measure the specimen temperature is not in direct contact with the specimen. The measured temperature is that of the temperature sensor itself. To obtain the correct specimen temperature, the *DSC* apparatus must be temperature calibrated at equivalent experimental conditions so that the recorded temperature correctly indicates the specimen temperature (see Test Methods E967 and E3142).

6.2 Temperature sensors are subject to degraded performance with age and exposure to the *DSC* test chamber atmosphere. It is therefore imperative that the apparatus is temperature calibrated regularly. Committee E37 recommends at a minimum annual calibration of all signals or more frequently.

7. Apparatus

7.1 *Differential Scanning Calorimeter (DSC) or Pressure Differential Scanning Calorimeter (PDSC)*—Multiple generations of differential scanning calorimeters from numerous commercial suppliers, as well as in-house custom apparatus, utilizing a variety of temperature and heat flow sensors in various configurations may be available to the user. While all such apparatus capabilities may not be equivalent, for purposes of this test method, any *DSC* instrumentation that meets the following criteria should be able to generate acceptable results

7.1.1 *DSC Test Chamber,* composed of:

7.1.1.1 *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.

7.1.1.2 *A Temperature Sensor,* to provide an indication of the specimen/furnace temperature to ±0.4 °C.

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

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

7.1.2 *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 °C/min constant to 1 % and an isothermal temperature constant to ±0.4 °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

[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 the Coordinating European Council website, http://www.cectests.org.