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AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Test Method for Determining Oxidation Induction Time of Hydrocarbons by Differential Scanning Calorimetry[1]

This standard is issued under the fixed designation E 1858; 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 the procedure for determining 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 Computer or electronic-based instruments, techniques or data treatment equivalent to this test method may also be used.

NOTE 1—Users of this test method are expressly advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user of this test method to determine the necessary equivalency prior to use.

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

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

1.5 The values stated in SI units are regarded as 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 Note 6 and Note 12.

2. Referenced Documents

2.1 *ASTM Standards:*

D 3350 Specification for Polyethylene Plastic Pipe and Fitting Materials[2]

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

D 4565 Test Method for Physical Environmental Performance Properties of Insulations and Jackets for Telecommunications Wire and Cable[3]

D 5482 Test Method for Oxidation Induction Time of Lubricating Greases by Pressure Differential Scanning Calorimetry[4]

D 5885 Test Method for Oxidative Induction Time of Polyolefin Geosynthics By High Pressure Differential Scanning Calorimetry[5]

E 473 Terminology Relating to Thermal Analysis[6]

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

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

3. Terminology

3.1 *Definitions*:

3.1.1 Specific technical terms used in this test method are given in Terminology E 473.

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 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 D 3895, D 5482, Specification D 3350, and Test Method D 4565).

NOTE 2—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.

[1] This test method is under the direct jurisdiction of Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods and Recommended Practices.

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

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

[4] *Annual Book of ASTM Standards*, Vol 05.03.

[5] *Annual Book of ASTM Standards*,, Vol 04.09.

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

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*, capable of providing a heating rate of at least 40°C/min and automatically recording the differential heat flow between the sample and reference material to the required sensitivity and precision. The instrument should have the capability of measuring heat flow of at least 5 mW, with provision for less sensitive ranges. Isothermal temperatures must be held within ± 0.4°C of a set temperature.

NOTE 3—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 manufacturer) the maximum oxygen pressure at which the apparatus may be safely operated.

6.2 *Recorder or Printer/Plotter*, or similar device, is used capable of displaying heat flow on the *Y*-axis and time on the *X*-axis (see Fig. 1). Time base shall be accurate to ± 0.1 min and be readable to 0.1 min.

NOTE 4—The capability to record the first derivative of the heat flow curve will be helpful in cases where the baseline is not constant.

6.3 *A High Pressure Gas Regulator*, suitable for oxygen service, is used in Test Method B.

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

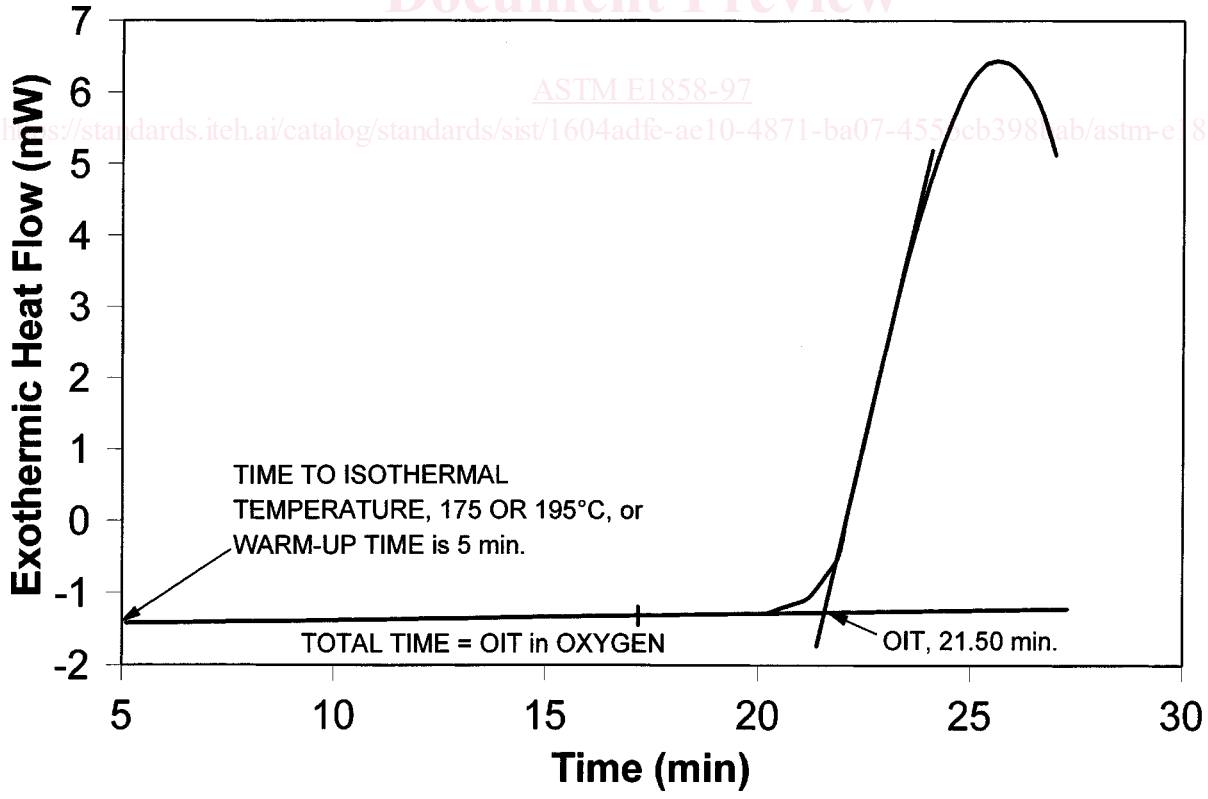
NOTE 6—**Caution:** Use metal or fluoropolymer tubing with oxygen rather than the commonly used rubber or polyvinyl chloride plastic tubing. There have been hazardous situations with prolonged use of certain polymer tubing in oxygen service.

6.4 *Flow meter*, capable of reading 25 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 9, following Section 11.4.

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 Capsules*, 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 Recommended procedure for new sample pan cleaning can be found in Annex A1.



NOTE 1—A curve obtained by performing the OIT test method for hydrocarbon oxidation

FIG. 1 Hydrocarbon Oxidation by DSC