Designation: E2009 – 08 (Reapproved 2014)ε1

Standard Test Methods for Oxidation Onset Temperature of Hydrocarbons by Differential Scanning Calorimetry[1]

This standard is issued under the fixed designation E2009; 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.

ε1 NOTE—Warning notes were editorially updated throughout in March 2014.

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 under linear heating rate conditions and are applicable to hydrocarbons, which oxidize exothermically in their analyzed form.

1.2 *Test Method A*—A differential scanning calorimeter (DSC) is used at ambient pressure, of one atmosphere 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 *Test Method C*—A differential scanning calorimeter (DSC) is used at ambient pressure of one atmosphere of air.

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

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

D5483 Test Method for Oxidation Induction Time of Lubricating Greases by Pressure Differential Scanning Calorimetry

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

E1858 Test Method for Determining Oxidation Induction Time of Hydrocarbons by Differential Scanning Calorimetry

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology E473.

3.1.1 *oxidation (extrapolated) onset temperature (OOT)*—a relative measure of oxidative stability at the cited heating rate is determined from data recorded during a DSC scanning temperature test. The temperature at which the onset to the observed oxidation is taken as the OOT.

4. Summary of Methods

4.1 The test specimen in an aluminum container and an empty reference aluminum container or pan are heated at a specified constant heating rate in an oxygen (or air) environment. Heat flow out of the specimen is monitored as a function of temperature until the oxidative reaction is manifested by heat evolution on the thermal curve. The oxidation (extrapolated) onset temperature (OOT), a relative measure of oxidative stability at the cited heating rate, is determined from data recorded during the scanning temperature test. The OOT measurement is initiated upon reaching the exothermic reaction and measuring the extrapolated onset temperature.

4.2 For some particularly stable materials, the OOT may be quite high (>300°C) at the specified heating rate of the experiment. Under these circumstances, the OOT may be

[1] These test methods are under the jurisdiction of ASTM Committee E37 on Thermal Measurements and are 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.

reduced by increasing the pressure of oxygen purge gas. Conversely, reducing the partial pressure of oxygen (such as by the use of air) may retard reactions that proceed too rapidly, with a corresponding increase of the OOT. By admixing oxygen gas with a suitable diluent, for example, nitrogen, the OOT will be increased (see Specification D3350 and Test Methods D3895, D4565, and D5483).

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

5. Significance and Use

5.1 Oxidation onset temperature is a relative measure of the degree of oxidative stability of the material evaluated at a given heating rate and oxidative environment, for example, oxygen; the higher the OOT value the more stable the material. The OOT is described in Fig. 1. The OOT values can be used for comparative purposes and are not an absolute measurement, like the oxidation induction time (OIT) at a constant temperature (see Test Method E1858). The presence or effectiveness of antioxidants may be determined by these test methods.

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

6. Apparatus

6.1 *Differential Scanning Calorimeter (DSC) or Pressure Differential Scanning Calorimeter (PDSC)*—The essential instrumentation required to provide the minimum differential scanning calorimetric capability for these test methods includes: a DSC chamber composed of a furnace to provide uniform controlled heating of a specimen and a reference to a constant heating rate of at least 10°C/min within the applicable temperature range for these test methods; a temperature sensor to provide an indication of the specimen temperature to ±0.1°C; a differential sensor to detect heat flow (power) difference between the specimen and the reference to 0.1 mW; and the instrument should have the capability of measuring heat flow of at least 6 mW, with provision for less sensitive ranges.

NOTE 2—In certain cases when the sample under study is of high volatility (for example, low molecular weight hydrocarbons), the use of pressures in excess of 0.1 MPa (1 atmosphere) is needed. The operator is cautioned to verify (with apparatus designer) the maximum oxygen pressure at which the apparatus may be safely operated. A PDSC is used in Method B.

6.2 *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.

6.3 A high-pressure gas regulator or similar device to adjust the applied pressure in the test chamber to less than ±5 %, including any temperature dependence on the transducer, is used in Method B. (**Warning—**Use metal free of organic matter 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 3—Gas delivery tubing should be kept as short as possible to minimize *dead* volume. The link between the test chamber and pressure transducer should allow fast pressure equilibration to ensure accurate recording of the pressure above the specimen during testing.

6.4 Specimen containers are aluminum sample pans and should be inert to the specimen and reference material as well as the oxidizing gas. The specimen containers should be of suitable structural shape and integrity to contain the specimen and reference in accordance with the specific requirements of these test methods, including a pressure system consisting of a pressure vessel or similar means of sealing the test chamber at any applied pressure within the pressure limits required for these test methods. The specimen containers shall be clean, dry, and flat. A typical cylindrical specimen container has the following dimensions: height, 1.5 to 2.5 mm and outer diameter, 5.0 to 7.0 mm.

6.5 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 warning statement in 6.3).

6.6 Use an 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 or containers' masses, or both. Recommended procedure for new sample pan cleaning can be found in Annex A1.

7. Reagents and Materials

7.1 *Oxygen,* extra dry, of not less than 99.5 % by volume. (**Warning—**Oxidizer. Gas under pressure.)

7.2 *Air,* extra dry.

7.3 *Indium,* of not less than 99.9 % by mass.

7.4 *Tin,* of not less than 99.9 % by mass.

8. Sampling

8.1 If the sample is a liquid or powder, mix thoroughly prior to sampling.

8.2 In the absence of information, samples are to be analyzed as received. If some heat or mechanical treatment is applied to the sample prior to analysis, this treatment shall be in nitrogen and noted in the report. If some heat treatment is used prior to oxidative testing, then record any mass loss as a result of the treatment.

9. Precautions

9.1 **Warning—**Oxygen is a strong oxidizer and vigorously accelerates combustion. Keep surfaces clean.

9.2 If the specimen is heated to decomposition, toxic or corrosive products may be released.

9.3 For certain types of PDSC, it is recommended that the flow be set up with a *reverse flow* implementation to ensure there is no contact of decomposed hydrocarbons with incoming oxygen within the instrument. See instrument designer's recommendation on reverse flow.