Designation: ~~D5483 – 20~~ D5483 – 21

Standard Test Method for Oxidation Induction Time of Lubricating Greases by Pressure Differential Scanning Calorimetry[1]

This standard is issued under the fixed designation D5483; 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 determination of oxidation induction time of lubricating greases subjected to oxygen at 3.5 MPa (500 psig) and temperatures between 155 °C and 210 °C.

1.2 *Warning*—The original data published in Research Report RR:D02-1314, was not analyzed in accordance the current D2PP. It also used instruments which are no longer manufactured and in a check of currently used instruments, none of the original instruments were still in use. The new precision of this test method is still to be established.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.4 *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.*

1.5 *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]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
E697 Practice for Use of Electron-Capture Detectors in Gas Chromatography
E1858 Test Methods for Determining Oxidation Induction Time of Hydrocarbons by Differential Scanning Calorimetry

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *extrapolated onset time, n*—a time determined on a thermal curve, as the intersection of the extrapolated baseline and a line tangent to the oxidation exotherm constructed at its maximum rate.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.09.0E on Oxidation of Greases.

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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.

***A Summary of Changes section appears at the end of this standard**

3.1.2 *oxidation induction time (OIT), n*—the period of time from the first exposure to an oxidizing atmosphere until the extrapolated onset time.

3.1.3 *thermal curve, n*—a graph of sample heat flow versus time.

4. Summary of Test Method

4.1 A small quantity of grease is weighed into a sample pan and placed in a test cell. The cell is heated to a specified temperature and then pressurized with oxygen. The cell is held at a regulated temperature and pressure until an exothermic reaction occurs. The extrapolated onset time is measured and reported as the oxidation induction time for the grease under the specified test temperature.

5. Significance and Use

5.1 Oxidation induction time, as determined under the conditions of this test method, can be used as an indication of oxidation stability.[3] This test method can be used for research and development, quality control and specification purposes. However, no correlation has been determined between the results of this test method and service performance.

6. Apparatus

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

6.1.1 *Pressure System,* consisting of:

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

6.1.1.2 *Temperature Sensor,* to provide an indication of the specimen/furnace temperature to ±0.4 °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 *Pressure Transducer,* or similar device to measure the pressure inside the test chamber to ±0.2 MPa, including any temperature dependence of the transducer.

6.1.1.5 A source of pressurized oxygen or air capable of sustaining a regulated gas pressure in the test chamber of up to 3.5 MPa.

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

6.1.3 *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 1—At the time that the original round robin data for this test method was generated, only DuPont Instruments (now TA instruments) manufactured equipment that met the requirements of 6.1. Subsequently, other companies have manufactured equipment meeting these requirements. Their use is permitted provided their performance is consistent with the repeatability and reproducibility described in Section 11.

NOTE 2—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.

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 *Flow Meter,* capable of reading 200 mL/min or another selected flow rate, accurate to within ±5 %. Ensure the flowmeter is calibrated.

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[3] Rhee, In-Sik, "Development of a New Oxidation Stability Test Method for Greases Using a Pressure Differential Scanning Calorimeter (PDSC)," *NLGI Spokesman*, Vol 55, No. 4, July 1991, pp. 123–132.

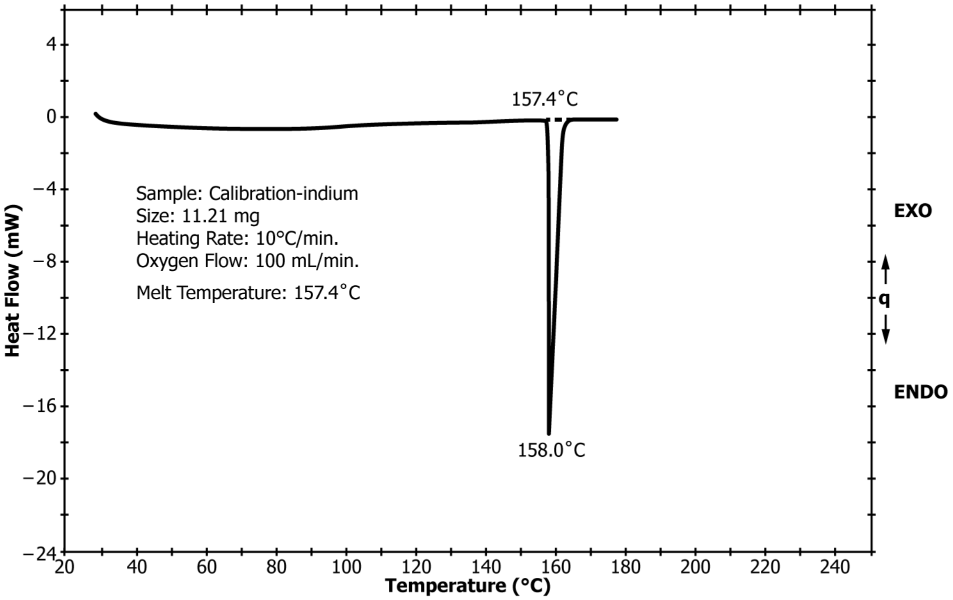


FIG. 1 Calibration

6.3 *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.

7. Reagents and Materials

7.1 *Oxygen,* extra dry, of not less than 99.5 % purity by volume. (**Warning—**Oxidizer. Gas under pressure. In addition to other precautions, use stainless steel or copper tubing which is compatible with oxygen, and pressure gauges which are designated for use with oxygen.)

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

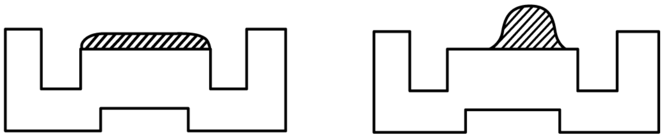
8. Calibration

8.1 *Sample Temperature Calibration:*

8.1.1 Weigh approximately 10 mg of indium into an aluminum sample pan, insert a lid and crimp the lid to the pan using the encapsulation press. Place the crimped pan onto the sample platform in the pressure cell. Seal an empty pan in the same manner and place it on the reference platform. Set the cell cover in place and close the cell.

8.1.2 Open the oxygen cylinder valve slightly and set a pressure of 3.5 MPa ± 0.2 MPa (500 psig ± 25 psig) on the cell inlet line with the pressure regulator. Partially open the inlet valve on the cell and allow the pressure to slowly build up in the cell. This should require approximately 2 min. Using the outlet valve, adjust the oxygen purge rate through the flowmeter to 100 mL ⁄min ± 10 mL ⁄min. The open position of these valves should remain fixed during the test.

8.1.3 Set the thermal analyzer to heat from ambient temperature (approximately 22 °C) to 180 °C) at a programmed rate of 10 °C ⁄min. After completion of the run, measure the melting temperature of the indium. If the melting temperature differs from 157.4 °C ± 0.2 °C (see Note 4), correct the difference by using either the hardware or software calibration procedure described in the manufacturer's instruction manual. If the hardware calibration procedure is used, the temperature correction should be performed under 3.5 MPa (500 psig) oxygen pressure with a 100 mL ⁄min purge rate. A typical melting calibration curve is shown in Fig. 1.



SPREAD OUT SAMPLE NON-SPREAD (THICK) SAMPLE

FIG. 2 Sample Preparation on SFI Pan

NOTE 4—The melting temperature of indium is 156.6 °C at atmospheric pressure, but has been found to be elevated to 157.4 °C under the conditions of this test method, 3.5 MPa (500 psig) of oxygen.[4]

8.2 *Temperature Controller Calibration:*

8.2.1 The controller thermocouple calibration should be performed according to the instrument's manual.

8.3 *Cell Base Pressure Gauge Calibration*—The calibration should be conducted using a calibrated pressure transducer or a previously calibrated gauge according to the pressure cell manufacturer's instructions.

9. Procedure

9.1 Before starting a test, the control thermocouple calibration shall be conducted at the test temperature (either 210 °C, 180 °C, or 155 °C). When the test temperature is not known, the calibration should be conducted at 210 °C. Ignore this step if the instrument already has an automatic temperature controller calibration system.

9.2 Weigh 2.0 mg ± 0.1 mg of grease into a sample pan. Spread the sample evenly upon the flat portion. Do not spill any of the sample into the trough portion of the pan (See Fig. 2).

NOTE 5—Examples of suitable and poor sample on pan patterns are shown in Fig. 2.

9.3 Place the uncovered pan containing the sample onto the platform of the cell according to the PDSC manufacturer's instructions for placing the sample pan. Place an empty pan of the same configuration onto the cell platform according to the PDSC manufacturer's instructions for placing the reference pan. Close the cell and the pressure release valve.

9.4 Beginning at ambient temperature (approximately 22 °C), program the sample temperature to increase at a rate of 100 °C⁄min to the test temperature.

9.5 Allow the sample to equilibrate at the test temperature for 2 min.

NOTE 6—This step did not appear in the test method which was used in the round robin to generate the precision statement. The round robin test method used the software of a PDSC manufacturer to determine when equilibration at test temperatures occurred. Step 9.5 removes this dependence on one PDSC manufacturer and is not expected to significantly affect the measured OIT since this step precedes the pressurization of the test cell with oxygen.

9.6 Open the oxygen valve and slowly pressurize the cell to 3.5 MPa ± 0.2 MPa (500 psig ± 25 psig). This should require approximately 2 min to reach maximum pressure. The oxidation induction time is measured from the time when the oxygen valve is opened.

9.7 As soon as the pressure has equilibrated, check the cell purge rate and adjust to 100 mL⁄min ± 10 mL⁄min with the outlet valve.

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1007. Contact ASTM Customer Service at service@astm.org.