Designation: ~~D5483 – 05 (Reapproved 2015)~~ D5483 – 20

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~~ 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~~ safety, health, and ~~health~~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

~~E473~~D4175 Terminology Relating to ~~Thermal Analysis and Rheology~~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.

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 *pressure differential scanning calorimeter, (PDSC), n*—a differential scanning calorimeter, as defined in Terminology E473, that is capable of maintaining the test sample at a controlled, elevated pressure.~~

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.

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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**

~~4.2 A kinetic equation incorporated with this test method can estimate oxidation induction times at other temperatures.~~

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),* ~~equipped with the following items (see~~the essential instrumentation required ~~Fig. 1~~).to provide the minimum differential scanning calorimetric capability for these test methods include:

~~NOTE 1—At the time that the round robin data for this test method was generated, only TA Instruments[4] manufactured equipment that met the requirements of 5.1. 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 10.~~

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 ~~*Sample Enclosure,*~~ *Data Collection Device,* ~~with capability to 3.5 MPa (500 psig) at 210 °C and pressure gauge graduated at intervals of 200 kPa (28.6 psi) or less.~~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 *Thermal Analyzer.*~~

~~6.3 *Aluminum Sample Solid Fat Index (SFI),* pan (see Note 2).~~

~~6.4 *Oxidation Stability Software.*~~

~~6.5 *Calibration Software.*~~

~~6.6 *Flowmeter,* with a capacity of at least 200 mL/min.~~

~~6.7 *Sample Encapsulation Press.*~~

~~NOTE 2—It has been found that grease samples can be prepared with more consistent surface areas using SFI pans as compared to flat bottom pans, resulting in better reproducibility.~~

~~NOTE 3—See Fig. 1 for a diagram of a typical test unit.~~

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:*

[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.

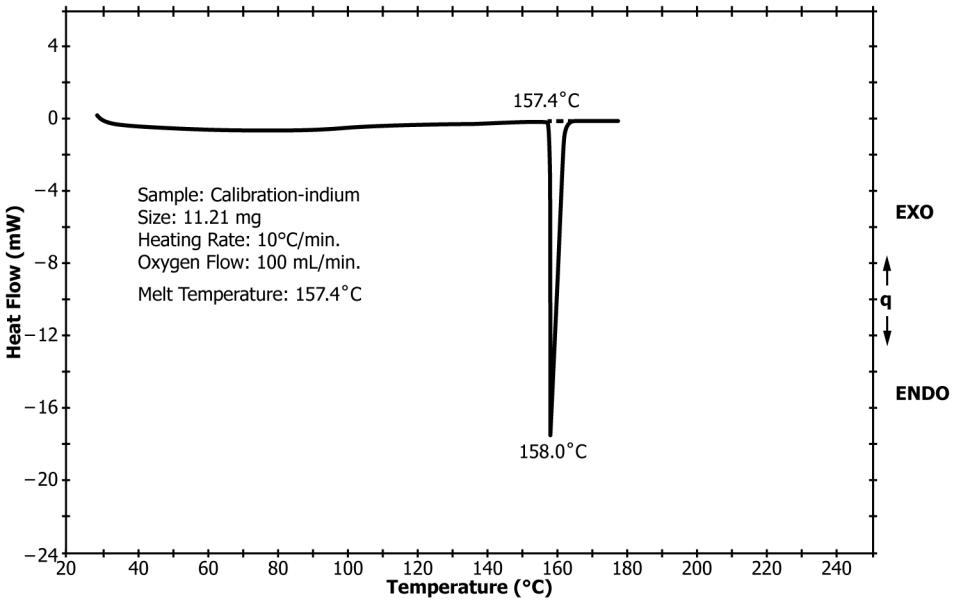

FIG. 21 Calibration

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. 21.

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 Remove both the sample pan and the reference pan from the cell, then close the cell. Slowly pressurize the cell with 3.5 MPa ± 0.2 MPa (500 psig ± 25 psig) oxygen and adjust the purge rate to 100 mL/min ± 10 mL/min using the cell outlet valve. Select the desired test temperature (either 210 °C, 180 °C, or 155 °C).~~

~~8.2.2 Program the cell to maintain the selected test temperature. If, after 10 min, the displayed cell temperature differs by more than ±0.2 °C from the selected temperature, slowly adjust the temperature controller until they agree. After making an adjustment, wait at least 5 min to make certain that the temperature is stable before continuing.~~

8.2.1 ~~Some of the newest instruments do not need this step due to their automatic calibration system. Therefore, the control thermocouple calibration~~The controller thermocouple calibration should be performed according to the ~~instrument's~~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) according to~~ 155 °C). ~~8.2.1 and 8.2.2.~~ 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.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1007.