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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 and 210°C.

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

1.3 *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]

E473 Terminology Relating to Thermal Analysis and Rheology

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.

3.1.4 *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.

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 Fig. 1).[4]

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 *Sample Enclosure,* 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.

[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.

[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.

[4] The sole source of supply of the apparatus known to the committee at this time is TA Instruments, Inc., 109 Lukens Drive, New Castle, DE 19720. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

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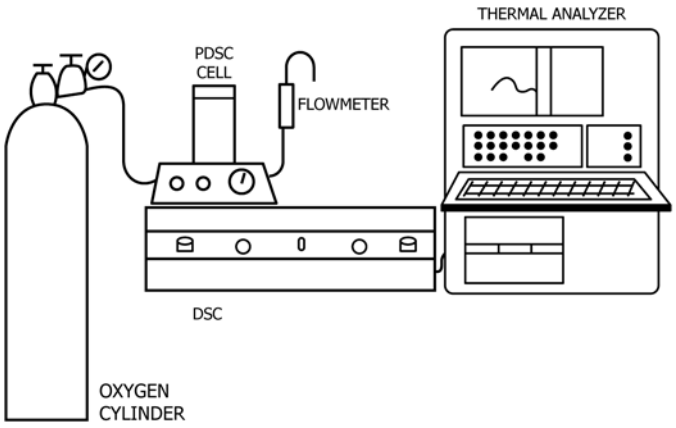

FIG. 1 PDSC Test Unit

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:*

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 ± 0.2 MPa (500 ± 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 ± 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 ± 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. 2.

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.[5]

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 ± 0.2 MPa (500 ± 25 psig) oxygen and adjust the purge rate to 100 ± 10 mL/min using the cell outlet valve. Select the desired test temperature (either 210, 180, 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.

8.2.3 Some of the newest instruments do not need this step due to their automatic calibration system. Therefore, the control thermocouple calibaration 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, 180, or 155°C) according to 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.

9.2 Weigh 2.0 ± 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. 3).

NOTE 5—Examples of suitable and poor sample on pan patterns are shown in Fig. 3.

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.

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

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