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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 D 5483; 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 the standard.

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:*

E 473 Terminology Relating to Thermal Analysis[2]

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 E 473, 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.

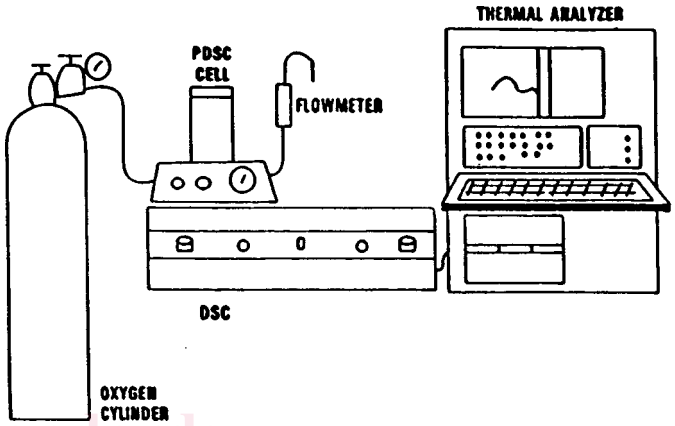

FIG. 1 PDSC Test Unit

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]

6.1.1 *Sample Enclosure*, with capability to 3.5 MPa (500 psig) at 210°C and pressure gage graduated at intervals of 200 kPa (28.6 psi) or less.

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.O9.0E on Greases.

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

[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] Available from TA Instruments, Inc., 109 Lukens Drive, New Castle, DE 19720. At the time that the round robin data for this test method was generated, only this company 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.

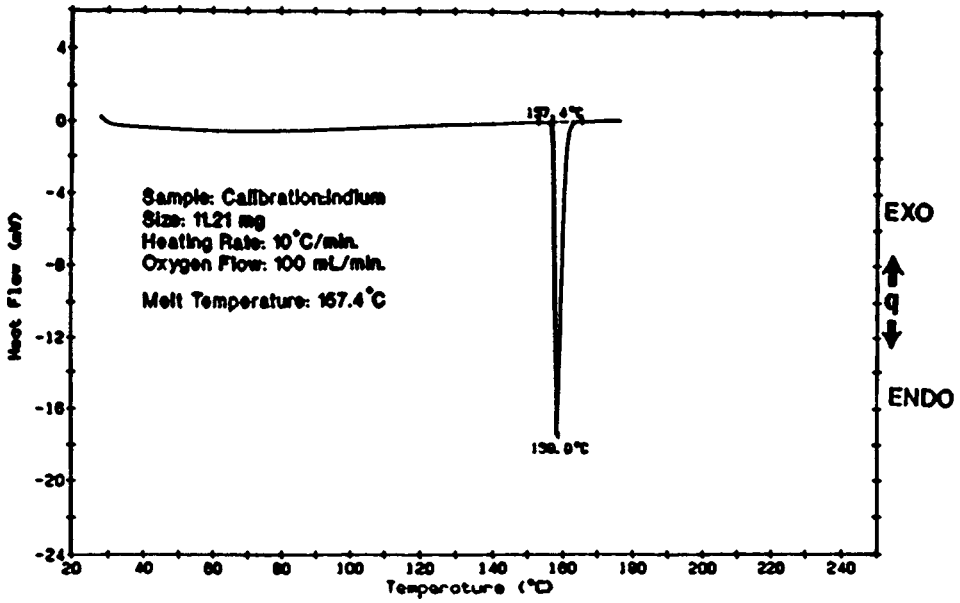

FIG. 2 Calibration

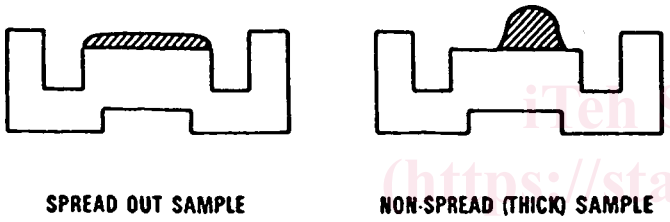

FIG. 3 Sample Preparation on SFI Pan

6.2 *Thermal Analyzer*.

6.3 *Aluminum Sample Solid Fat Index (SFI)*, pan (see Note 1).

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 1—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 2—**Caution:** In addition to other precautions, use stainless steel or copper tubing which is compatible with oxygen, and pressure gages which are designated for use with oxygen.

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 % by volume.

NOTE 4—**Warning:** Oxidizer. Gas under pressure.

7.2 *Indium*, of not less than 99.9 % 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 5), 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 5—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

[5] Supporting data are available from ASTM Headquarters. Request RR:D02-1007.