Designation: D 5483 – 02

An American National Standard

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.

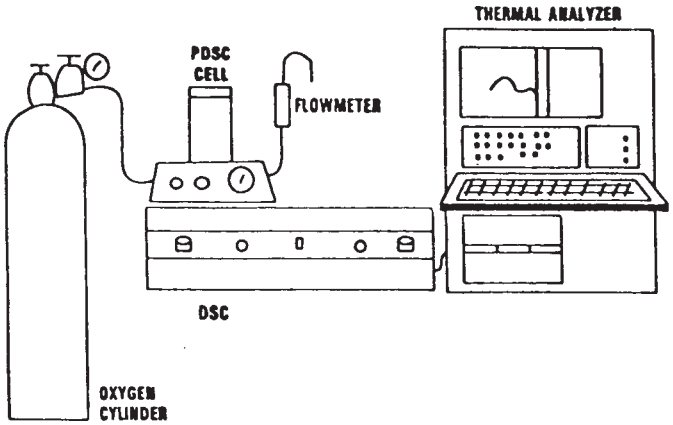

FIG. 1 PDSC Test Unit

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]

[1] This test method is under the jurisdiction of ASTM Committee D02 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.

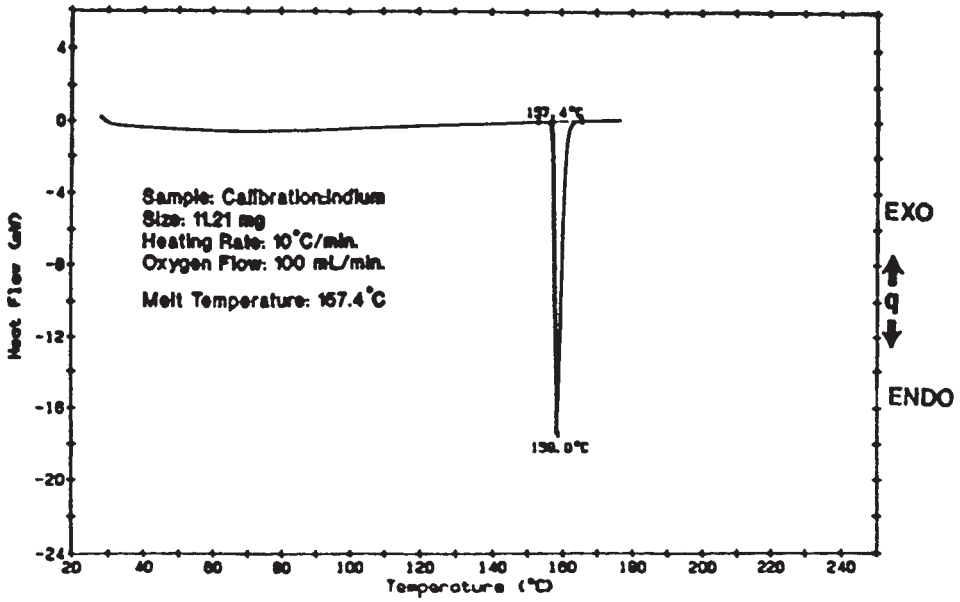

FIG. 2 Calibration

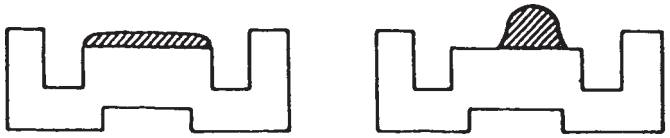

FIG. 3 Sample Preparation on SFI Pan

6.1.1 *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.

6.2 *Thermal Analyzer*.

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

6.4 *Oxidation Stability Software*. (**Warning**—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.)

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—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 3—**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 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).

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

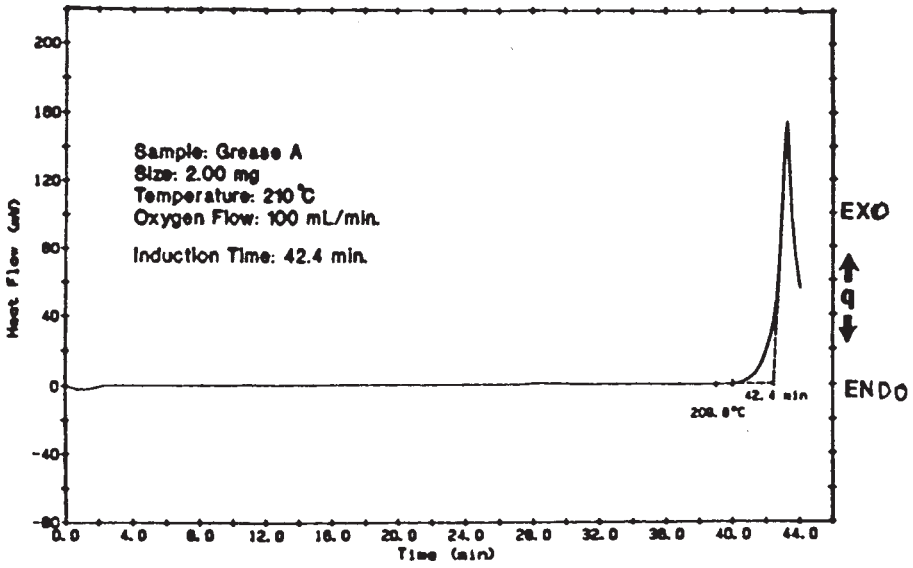

FIG. 4 Typical PDSC Thermal Curve

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.

8.3 *Cell Base Pressure Gage Calibration*:

8.3.1 The calibration should be conducted using a calibrated pressure transducer or a previously calibrated gage 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.

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.

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 8.5 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 ± 0.2 MPa (500 ± 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 ± 10 mL/min with the outlet valve.

9.8 After a duration of 120 min from the time when the oxygen valve was opened, close the oxygen valve and slowly release the cell pressure by opening the cell pressure release valve. In the case of a sample for which the approximate oxidation induction time is known, the test can be stopped after the oxidation exotherm has occurred.

9.9 Plot the thermal curve and measure the extrapolated onset time for the oxidation exotherm. Report this time, to the nearest tenth of one minute, as the oxidation induction time for the sample. If more than one oxidation exotherm is observed, record the oxidation induction time for the largest exotherm (See Fig. 4).

NOTE 7—A typical thermal curve is shown in Fig. 4.

9.10 If the induction time is less than 10 min, rerun the test at the next lower temperature, starting at 9.2. Allow the cell to cool to ambient temperature before running the test at the next lower temperature.

9.11 After the oxidation induction time requirement specified in 8.10 is satisfied, perform a duplicate test.

9.12 If the difference between the two results is greater than the 95% determinability limit stated in the Precision and Bias section of this test method (Section 12), then reject the results and determine two more oxidation induction times for the grease by returning to 9.2. Otherwise, average the oxidation induction times of both runs.

10. Calculation of Induction Times for Other Temperatures

10.1 After an oxidation induction time has been determined for a particular grease, a value can be estimated for other temperatures using the following equation[2]:

$$t = A \exp (17\,500/T) \quad (1)$$

where:
t = estimated oxidation induction time, min,
A = oxidation coefficient of the grease, and
T = temperature, K (for desired temperature).

The oxidation coefficient (A) is constant for a given grease and is calculated by (Eq 1) using the oxidation induction time reported in 9.1.2, thus,

$$A = \text{oxidation induction time}/\exp(17\,500/\text{test temperature, K}). \quad (2)$$

The estimated oxidation induction time can be used as a guide for choosing appropriate alternative test temperatures for a grease. The estimated oxidation induction time is not a part of the report for this test method.

11. Report

11.1 Report the following information:

11.1.1 Report, to the nearest tenth of one minute, the average value calculated in 9.12 as the oxidation induction time (OIT) for the sample.

11.1.2 Report the test temperature.

12. Precision and Bias [6]

12.1 The precision of this test method as determined by the statistical examination of interlaboratory test results involving eleven samples, five laboratories, three test temperatures (155, 180, and 210°C), and oxidation induction times of from 9.0 to 45.3 minutes is as follows.

12.1.1 *Determinability*— The difference between the pair of determinations averaged to obtain a test result would, in the long run, in the normal and correct operation of the test method, exceed the following value in only one case in twenty:

$$\text{Determinability} = 0.59\sqrt{m} \quad (3)$$

where:
m = the mean of the two determinations

12.1.2 *Repeatability*— The difference between successive results (each the mean of a pair of determinations) obtained by the same operator with the same apparatus under constant operating conditions on identical material, would in the long run, in the normal and correct operation of the test method, exceed the following value in only one case in twenty:

$$\text{Repeatability} = 0.42\sqrt{m} \quad (4)$$

where:
m = is the mean of the two results

12.1.3 *Reproducibility*—The difference between two independent results (each the mean of a pair of determinations) obtained by different operators working in different laboratories would, in the long run, exceed the following value only one case in twenty:

$$\text{Reproducibility} = 0.71\sqrt{m} \quad (5)$$

where:
m = the mean of the two test results.

12.2 *Bias*—The procedure in this test method has no bias because the value of oxidation induction time can be defined only in terms of a test method.

13. Keywords

13.1 differential scanning calorimetry; lubricating grease; OIT; onset temperature; oxidation; oxidation coefficient; oxidation induction time; PDSC; thermal analysis

[6] Supporting data may be obtained from ASTM Headquarters. Request RR:D02-1314.

APPENDIX

(Nonmandatory Information)

X1. PDSC ROUND ROBIN DATA

X1.1 PDSC Round Robin Data

X1.1.1 See Table X1.1.

NOTE 1—The numbers in this table are the oxidation induction times in minutes. The pairs of oxidation times for each grease represent two single determinations.

Lab	Grease Sample										
	A	B	C	D	E	F	G	H	I	J	K
1	40.0	13.2	51.3	11.6	15.4	13.7	44.8	16.8	36.2	11.6	30.6
	45.3	13.6	48.1	11.3	14.8	16.0	46.8	17.3	31.6	10.3	31.3
2	41.3	15.1	48.8	10.4	15.9	14.0	39.5	17.2	30.5	11.2	31.4
	43.4	15.6	48.8	10.1	14.7	13.7	42.2	15.7	29.0	11.6	31.0
3	44.9	15.5	48.6	9.6	15.9	11.4	44.0	14.6	32.8	10.6	30.8
	41.9	17.2	47.4	9.2	13.9	13.5	43.0	14.9	24.0	9.0	31.2
4	42.4	15.5	48.8	10.2	14.9	13.6	42.7	16.5	28.8	10.7	33.9
	43.8	15.5	48.7	10.5	15.2	13.3	43.0	16.9	28.9	11.1	34.5
5	39.8	13.6	50.1	10.9	17.5	13.0	47.4	16.1	12.9	10.3	28.6
	42.5	13.3	49.7	11.3	15.5	13.9	45.0	14.6	10.5	10.1	28.7
Test temperature, °C	210	210	210	210	210	210	180	180	210	210	155