~~Designation:D6186–98 (Reapproved 2003)ε1~~ Designation: D 6186 – 08

An American National Standard

Standard Test Method for Oxidation Induction Time of Lubricating Oils by Pressure Differential Scanning Calorimetry (PDSC)[1]

This standard is issued under the fixed designation D 6186; 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.

~~ε[1] NOTE—Warning notes were editorially moved into the standard text in August 2003.~~

1. Scope*

1.1 This test method covers the determination of oxidation induction time of lubricating oils subjected to oxygen at 3.5 MPa (500 psig) and temperatures between 130 and 210°C.

~~1.2The values stated in SI units are to be regarded as the standard.~~

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.2.1 *Exception*—Pressure measurement appears in MPa with psig provided in parentheses 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. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *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.

2.1.2 *oxidation induction time, (OIT)*, *n*— a period of time during which the oxidation rate accelerates from zero to a maximum and which corresponds to the extrapolated onset time.

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

3. Summary of Test Method

3.1 A small quantity of oil 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 lubricating oil at the specified test temperature.

4. Significance and Use

4.1 Oxidation induction time, as determined under the conditions of this test method, may be used as an indication of oxidation stability.[2] This test method is faster than other oil oxidation tests and requires a very small amount of sample. It may be used for research and development, quality control, and specification purposes. However, no correlation has been established between the results of this test method and service performance.

5. Apparatus

5.1 *Pressure Differential Scanning Calorimeter (PDSC)*, equipped with the following items:

5.1.1 *Sample Enclosure*, with capability to 3.5 ± 0.2 MPa (500 ± 25 psig) at 210°C and pressure gauge graduated at intervals of 200 KPa (28.6 psig) or less.

5.1.2 *Thermal Analyzer.*

5.1.3 *Aluminum Solid Fat Index (SFI) Sample Pan*—See Note 1.

5.1.4 *Oxidation Stability Software.*

---

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

Current edition approved ~~May 10, 2003.~~Dec. 1, 2008. Published ~~August 2003.~~January 2009. Originally approved in 1997. Last previous edition approved in ~~1998~~2003 as D 6186–98(2003)ε1.

[2] Rhee, In-Sik, "Development of New Oxidation Stability Test Method for Lubricating Oils Using a Pressure Differential Scanning Calorimeter (PDSC)," *NLGI Spokesman*, Vol 65, No. 3, June 2001, pp. 16–23.

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

5.1.5 *Calibration Software.*

5.1.6 *Calibrated Flowmeter*, with a capacity of at least 200 mL/min and graduated in intervals of 5 mL or less.

5.1.7 *Sample Encapsulation Press* .

NOTE 1—It has been found that when oil samples are prepared with SFI pans which have more consistent surface areas than standard flat bottom pans, reproducibility is improved.

NOTE 2—Stainless steel or copper tubing is compatible with oxygen.

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

6. Reagents and Materials

6.1 *Oxygen*, a minimum purity of 99.5 % oxygen by volume. (**Warning—**Oxidizer. Gas under pressure.)

6.2 *Indium*, of not less than 99.9 % indium by mass.

7. Calibration

7.1 *Sample Temperature Calibration* :

7.1.1 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.

7.1.2 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 requires approximately 2 min. Using the outlet valve, adjust and maintain the oxygen purge rate through the flowmeter at 100 ± 10 mL/min.

7.1.3 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, perform the temperature correction 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.

7.2 *Temperature Controller Calibration* :

7.2.1 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, 155, or 130°C).

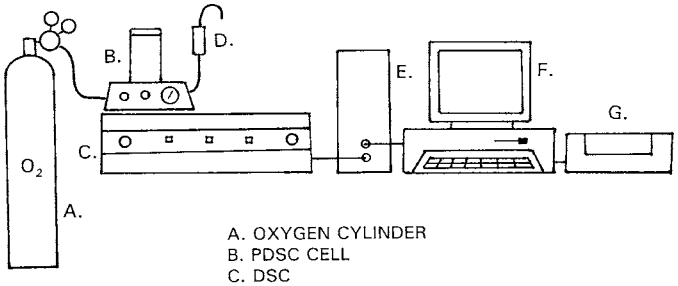
7.2.2 Program the cell to maintain the selected test temperature. If, after 10 min, the displayed cell temperature differs by more than ± 0.2°C from the selected temperature, slowly adjust the temperature controller until they agree. After making an adjustment, wait at least 5 min to make certain that the temperature is stable before continuing. If the PDSC equipment does not have this function, the control calibration shall be followed according to the equipment manufacturer's recommendations.

7.3 *Cell Base Pressure Gauge Calibration* —Conduct the calibration using a calibrated pressure transducer or a previously calibrated gauge according to the pressure cell manufacturer's instructions.

8. Procedure

8.1 Before starting a test, the control thermocouple calibration shall be conducted at the test temperature (either 210, 180, 155, or 130°C) according to 7.2.1 and 7.2.2. When the test temperature is not known, conduct the calibration at 210°C.

8.2 Weigh 3.0 ± 0.2 mg of oil into a new sample pan. Spread the sample evenly upon the flat portion. Do not spill any of the sample into the trough portion of the pan. A flat bottom pan can be used if the sample is placed upon a 0.5 cm diameter circle in the center of the pan.



A. OXYGEN CYLINDER
B. PDSC CELL
C. DSC
D. FLOWMETER
E. MODULE INTERFACE
F. MICROPROCESSOR
G. PLOTTER

FIG. 1 PDSC Test Unit

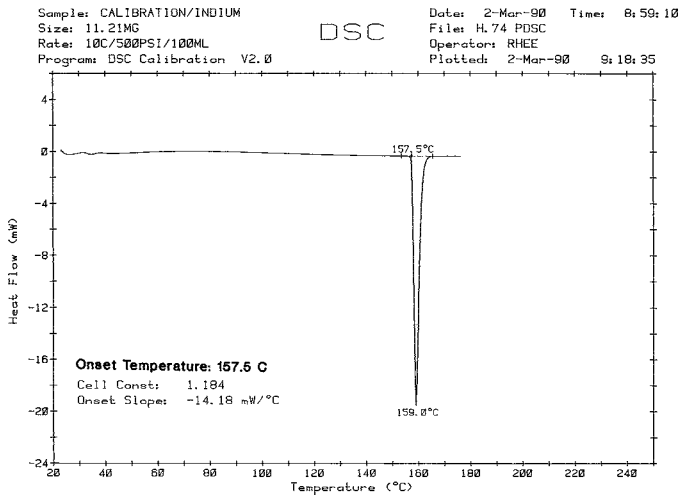


FIG. 2 Calibration Curve for PDSC

8.3 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 a new 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.

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

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

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

8.7 As soon as the pressure has equilibrated, check the cell purge rate and adjust and maintain at 100 ± 10 mL/min with the outlet valve.

8.8 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.

8.9 Plot the thermal curve and measure the extrapolated onset time for the oxidation exotherm. Report this time, to the nearest 1 min, as the oxidation induction time for the sample. If more than one oxidation exotherm is observed, report the oxidation induction time for the largest exotherm.

NOTE 5—A typical thermal curve is shown in Fig. 3.

8.10 If the induction time is less than 10 min, rerun the test at the next lower temperature, starting at 8.2. Allow the cell to cool to ambient temperature before running the test at the next lower temperature.

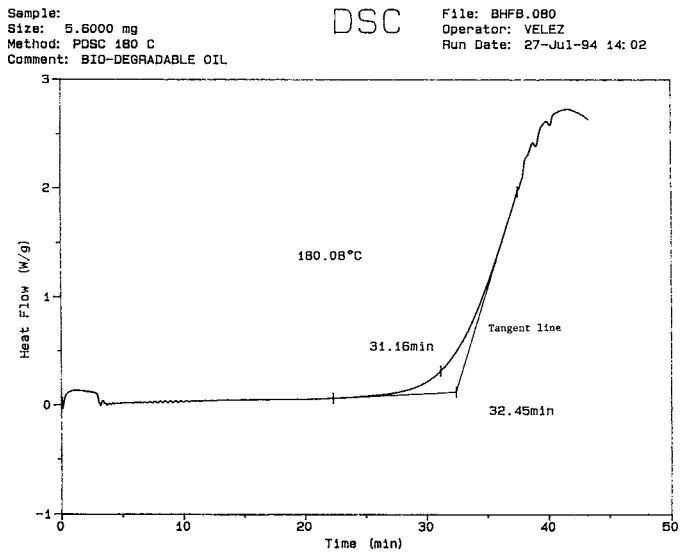


FIG. 3 A Typical Thermal Curve From PDSC Test