



Designation: D8206 – 18

Standard Test Method for Oxidation Stability of Lubricating Greases—Rapid Small-Scale Oxidation Test (RSSOT)¹

This standard is issued under the fixed designation D8206; 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 quantitative determination of the oxidation stability of lubricating greases with a dropping point above the test temperature.

1.2 This test method determines the resistance of lubricating greases to oxidation when stored statically in an oxygen atmosphere in a sealed system at an elevated temperature under conditions of test.

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

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, health, and 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

[D942 Test Method for Oxidation Stability of Lubricating Greases by the Oxygen Pressure Vessel Method](#)

[D4057 Practice for Manual Sampling of Petroleum and Petroleum Products](#)

[D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants](#)

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.

2.2 ISO Standard:³

[EN 60751:2008 Industrial platinum resistance thermometers and platinum temperature](#)

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *break point, n*—pressure in the test instrument, which is 10 % below the maximum pressure of the actual test run.

3.1.2 *induction period, n*—time elapsed between starting the heating procedure of the pressure vessel and the break point, measured in minutes.

4. Summary of Test Method

4.1 For this test, 4.00 g \pm 0.01 g of sample is weighed in a glass dish at a temperature of 20 °C \pm 5 °C and then introduced into a pressure vessel, which is subsequently charged with oxygen to 700 kPa \pm 5 kPa.

4.1.1 The test is initiated by heating the pressure vessel to a temperature of 140 °C \pm 0.5 °C or 160 °C \pm 0.5 °C, depending on the selected test.

4.2 The pressure is recorded continuously until the break point is reached.

5. Significance and Use

5.1 This test method measures the net change in pressure resulting from consumption of oxygen by oxidation and gain in pressure due to formation of volatile oxidation by-products. This test method may be used for quality control to indicate batch-to-batch uniformity. It predicts neither the stability of greases stored in containers for long periods, nor the stability of films of greases on bearings and motor parts.

5.2 Induction period as determined under the conditions of this test method can be used as an indication of oxidation stability. 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.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

6. Apparatus

6.1 *General*—This test method uses an automatically controlled oxidation tester⁴ (see Fig. 1), comprising a pressure vessel capable of being heated quickly and fitted with a pressure sensor capable of measuring pressures up to 1800 kPa and a temperature sensor capable of reading to 0.1 °C accuracy. Pressure and temperature in the pressure vessel are recorded continuously during the test. The pressure vessel is fitted with oxygen inlet and outlet valves and a means of automatically releasing the pressure at the end of the test. The integrated cooling fan cools the pressure vessel from the test temperature to ambient temperature by a flow of air. See Annex A1 for detailed information.

6.2 *Analytical Balance*, with a resolution of ± 0.001 g.

6.3 *Glass Dish* (see Fig. 3)—A borosilicate glass dish with a diameter of $41 \text{ mm} \pm 1 \text{ mm}$ and a height of $8 \text{ mm} \pm 0.9 \text{ mm}$ (according to Test Method D942; see Fig. A1.1).

6.4 *Temperature Calibration Equipment* (see Fig. 4), comprising a cover and a calibrated temperature sensor. The calibrated temperature sensor is fixed to a depth of $15 \text{ mm} \pm$

0.5 mm. The temperature sensor is calibrated to the nearest 0.1 °C by an approved calibration service, such as one that is traceable to the National Institute of Standards and Technology (NIST) or to a national authority in the country in which the equipment is used or manufactured.

6.5 *Pressure Calibration Equipment* (see Fig. 5), comprising a calibrated pressure sensor. The pressure sensor is calibrated to the nearest 10 kPa, by an approved calibration service, such as one that is traceable to the National Institute of Standards and Technology (NIST) or to a national authority in the country in which the equipment is used or manufactured.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

⁴ The sole source of supply of the instrument known to the committee at this time is Anton Paar's RapidOxy instrument, available from Anton Paar ProveTec GmbH, Ludwig-Erhard-Ring 13, 15827 Blankenfelde-Mahlow, Germany, www.anton-paar.com. 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.

⁵ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the United States *Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



FIG. 1 Example of RSSOT Instrument



FIG. 2 Example of Rear View of Instrument with Oxygen Inlet and Outlet Valve

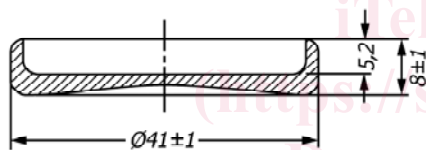


FIG. 3 Glass Dish (sample container)

NOTE 1—All dimensions are in millimetres.

7.2 *Ethanol*—Minimum purity 94 %. A solvent for the removal of oxidation residues from the pressure vessel has to be used. Commercially available ethanol of 94 % minimum purity has been found to be suitable to remove residues from the test vessel.

7.3 *Cleaning Solution*—A laboratory liquid, alkaline high-performance concentrated cleaner for immersion and ultrasonic baths, diluted in water to the manufacturer’s recommendation and capable of satisfactorily cleaning the glassware used in the test. (**Warning**—Danger! Wear protective gloves/eye protection. May cause skin irritation or serious eye damage.)

7.4 *Oxygen*, of not less than 99.5 % purity. (**Warning**—Danger! Since oxygen vigorously accelerates combustion, observe the following procedures: (1) Keep oil and grease away from oxygen at high pressure. Keep oil and grease away from all regulators, gauges, and control equipment. (2) Use oxygen only with equipment conditioned for oxygen service by careful cleaning to remove oil and grease from area in contact with high-pressure oxygen. (3) Keep combustibles away from oxygen and eliminate ignition sources. (4) Keep surfaces clean to prevent ignition or explosion, or both, upon contact with high pressure oxygen. (5) Always use a pressure regulator to deliver oxygen. Release regulator tension before opening

oxygen cylinder. (6) All equipment must be suitable and recommended for oxygen service.)

7.5 *Lint-Free Cleaning Tissue*—Suitable for sensitive surfaces, that will not scratch the surface.

7.6 *O-Ring Seal* (see A1.2)—Made of material being resistant to oxygen and heat, typically fluoro-elastomer FPM/FKM coated with polytetrafluoroethylene (PTFE).

7.7 *Temperature Calibration Fluid*—Stable middle distillate fuel liquid with a flash point above 60 °C and boiling point above 170 °C. (**Warning**—Flammable. Keep away from heat and open flame. Keep container closed. Use with adequate ventilation. Avoid prolonged or repeated contact with skin.)

NOTE 1—Any hydrocarbon fuel with sufficient stability and a known induction period may be used as a verification fluid. In general, verification fluid with a certified induction period is available from the manufacturer of the instrument.

8. Hazards

8.1 To provide protection against the possible explosive rupture of the pressure vessel, the instrument shall be operated behind an appropriate safety shield or hood sash.

9. Sampling

9.1 Sample in accordance with Practice D4057. The sample presented for analysis should be large enough to make possible the selection of a representative portion for testing. Examine for any indication of non-homogeneity such as oil separation, phase changes, or gross contamination. If any abnormal conditions are found, obtain a new sample.

10. Preparation of Instrument

10.1 From the previous run, remove the glass dish from the pressure vessel using tongs or forceps.

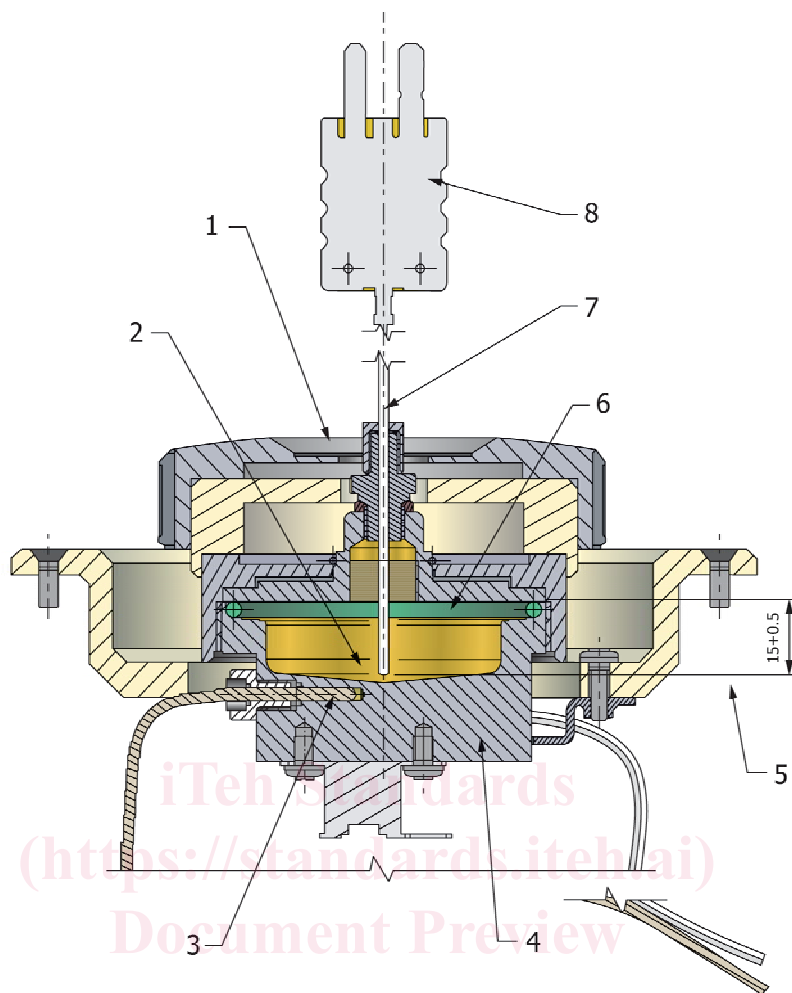


FIG. 4 Temperature Calibration Equipment

1. Calibration cover with duct for temperature calibration sensor
2. Calibration fluid
3. Temperature sensor for block temperature
4. Heating block
5. Immersion depth of temperature calibration sensor and bottom of calibration cover
6. Seal
7. Temperature calibration sensor
8. Connector plug to measuring device

NOTE 1—All dimensions are in millimetres.

10.2 Wipe the pressure vessel, the seal groove, and the screw cap of the pressure vessel with lint-free cleaning tissue (see 7.5) soaked with ethanol until free of gum or other oxidation residues.

10.3 Thoroughly clean the glass dish by removing the grease first mechanically and then by using the cleaning solution (see 7.3). Follow the final cleaning by a tap water rinse, a distilled water rinse, and drying in an oven. Handle the clean dishes only with forceps.

10.4 Allow the test sample cup and cover to dry in air and visually inspect for cleanliness.

NOTE 2—Compressed air is generally unsuitable to speed up evaporation of solvent because it can contain traces of oil that could contaminate the next test.

10.5 A new O-ring seal must be used for each test to prevent contamination from the previous test.

11. Calibration

11.1 Check the instrument regularly and according to your laboratory's specifications with suitable reference material. Pressure and temperature are critical parameters. Proper calibration of the respective sensors is therefore important. Recommended interval for temperature and pressure calibration is 12 months.

11.2 *Calibration of Temperature Sensor*—Calibrate the temperature sensor to the nearest 0.1 °C using the temperature calibration equipment according to A2.1.