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Standard Test Method for Oxidation Stability of Lubricating Greases by the Oxygen Pressure Vessel Method[1]

This standard is issued under the fixed designation D942; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method determines 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.

1.2 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 kPa with psi provided for information only.

1.2.2 *Exception*—In Fig. A1.1, A1.1, and Appendix X1, all dimensions are in millimeters, with inches 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements see Sections 6 and 7.

1.4 *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]

A240/A240M Specification for Chromium and Chromium-Nickel Stainless Steel Plate, Sheet, and Strip for Pressure Vessels and for General Applications

D525 Test Method for Oxidation Stability of Gasoline (Induction Period Method)

E1 Specification for ASTM Liquid-in-Glass Thermometers

E2877 Guide for Digital Contact Thermometers

2.2 *Other Standards:*

IP Specification for Standard IP Thermometers[3]

BS 970:1983 Part I, Section S[4]

3. Summary of Test Method

3.1 The sample of grease is oxidized in a pressure vessel heated to 99 °C (210 °F) and filled with oxygen at 758 kPa (110 psi). Pressure is observed and recorded at stated intervals. The degree of oxidation after a given period of time is determined by the corresponding decrease in oxygen pressure.

NOTE 1—The pressure vessel has been referred to as "a bomb" in previous issues of this test method.

NOTE 2—The accepted unit of pressure is the pascal (Pa) for ASTM methods and will be parenthetically included after the conventional pound-force per square inch (psi) value. The Energy Institute uses the bar as a pressure measurement. Conversion of units may be obtained as follows:

To convert from pound-force per square inch (psi) to pascal (Pa) multiply by 6.894757×10^3.

[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.

In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method has been approved by the sponsoring committee and accepted by the cooperating societies in accordance with the established procedures.

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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] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.uk.

[4] Available from British Standards Institute (BSI), 389 Chiswick High Rd., London W4 4AL, U.K., http://www.bsi-global.com.

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

To convert from pound-force per square inch (psi) to bar multiply by 0.06894757.

To convert from bar to pascal (Pa) multiply by 10^5.

4. Significance and Use

4.1 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 under dynamic service conditions, nor the stability of greases stored in containers for long periods, nor the stability of films of greases on bearings and motor-parts. It should not be used to estimate the relative oxidation resistance of different grease types.

5. Apparatus

5.1 *Oxidation Pressure Vessel, Sample Dish, Dish Holder, Pressure Gauge and Oil Bath* as described in detail in the Annex.

NOTE 3—Other constant-temperature baths may be used if they are equivalent in heat capacity and thermal gradient characteristics to the oil bath described in the Annex and can be shown to maintain the pressure vessel at the prescribed test temperature.

5.2 *Thermometer,* having a range as shown below and conforming to the requirements as prescribed in Specification E1 or in the Specifications for IP Standard Thermometers. Alternatively, digital contact thermometers such as PRTs (platinum resistance thermometers), thermistors, or thermocouples in accordance with Specification E2877 of equal or better accuracy may be used:

	Thermometer	Number
Temperature Range	ASTM	IP
95 °C to 103 °C	22C	24C
204 °F to 218 °F	22F	24F

6. Material

6.1 *Oxygen,* of not less than 99.5 % purity.

6.2 (**Warning—**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 used must be suitable and recommended for oxygen service. (*7*) Never attempt to transfer oxygen from cylinder in which it is received to any other cylinder prior to use. (*8*) Do not drop oxygen cylinders. (*9*) Keep cylinder valve closed when not in use. (*10*) Stand away from valve when opening cylinder. (*11*) Do not breathe or use technical grade oxygen for inhalation purposes. (*12*) See Compressed Gas Association Booklets G-4 and G-4-1[5] for details of safe practice in the use of oxygen.)

6.3 *n-Heptane*—(**Warning—**Flammable. Harmful if inhaled. Keep away from heat, sparks, and open flame. Keep container closed. Use with adequate ventilation. Avoid breathing vapor or spray mist. Avoid prolonged or repeated contact with skin.)

6.4 *Cleaning Solution,* capable of satisfactorily cleaning the glassware used in the test. The criterion for satisfactory cleaning shall be a matching of the quality of that obtained with chromic acid cleaning solutions (fresh chromic acid, 6 h soaking period, rinsing with distilled water and drying) or some other equivalently strong oxidizing non-chromium containing acid cleaning solutions on used sample dishes. (**Warning—**Causes severe burns. A recognized carcinogen. Strong oxidizer; contact with organic material may cause fire. Hygroscopic.) (**Warning—**Do not get in eyes, on skin, or on clothing. Avoid breathing vapor or mist. Keep container closed. Use with adequate ventilation. Do not take internally.) For this comparison, visual appearance and mass loss on heating the glassware under test conditions may be used. Detergent cleaning avoids the potential hazards and inconveniences related to handling corrosive chromic acid solutions; this procedure remains the reference cleaning practice and as such may function as an alternate to the preferred procedure, cleaning with detergent solutions.

7. Preparation of Apparatus

7.1 Clean the sample dishes from all contamination from previous runs and from dust settling from the air by washing them with n-heptane and then with a cleaning solution. Follow the final cleaning operation by a thorough tap water rinse, a distilled water rinse, and drying in an oven. Handle the clean dishes only with forceps. (**Warning—**Handle in well-ventilated area, preferably in a hood. Keep away from heat, sparks, and open flame. Keep container closed when not in use.) (**Warning—**See 6.3, and avoid skin contact, which may cause severe burns.)

7.2 If lacquer is found after a run, clean the inside of the oxidation pressure vessel and the metal supports for the pressure vessel dishes by immersing in an appropriate solvent capable of removing the lacquer, such as gum solvent as specified in Test Method D525, and scrubbing with a bristle brush followed by drying. Scrub further with water and a fine scouring powder until all the lacquer deposits are removed. Follow the scouring operation by a thorough tap water rinse, a distilled water rinse, and drying in an oven. Handle the clean metal supports only with forceps.

8. Procedure

8.1 Fill each of the five dishes with 4.00 g ± 0.01 g of grease. Distribute the samples in the dishes in a uniform layer with a smooth level upper surface. Place the filled dishes on the five bottom shelves of the holder, leaving the top shelf to act as

[5] Available from Compressed Gas Association (CGA), 4221 Walney Rd., 5th Floor, Chantilly, VA 20151-2923, http://www.cganet.com.

a cover to prevent condensing volatile products from dropping into the grease samples. When assembling the pressure vessel, place a small ball of glass wool in the bottom of the stem.

8.2 Place the dish holder in the pressure vessel with a sealing gasket in place, and close the pressure vessel by tightening the bolts slowly and uniformly. Clear the air from the pressure vessel by introducing oxygen slowly until a pressure of 689 kPa (100 psi) is attained, then allow the oxygen to escape slowly; repeat four times. Bring the oxygen pressure to a value as shown in the following table:

Room Temperature		Pressure	
°C	°F	kPa	psi
17 to 20	62 to 68	586	85
20 to 23	68 to 74	593	86
23 to 27	74 to 80	600	87
27 to 30	80 to 86	607	88
30 to 33	86 to 92	614	89
33 to 37	92 to 98	621	90
37 to 40	98 to 104	627	91

Allow the pressure vessel to stand overnight to make sure there are no leaks.

NOTE 4—It has been found that pressure readings as shown above will result in a pressure reading of 758 kPa ± 14 kPa (110 psi ± 2 psi) when the pressure vessel is placed in the bath in the following step, 8.3, and consequently no release of oxygen will be required in most cases. This minimizes the chance of a leak developing at the valve after the overnight check for leaks has shown the pressure vessel to be satisfactory.

8.3 Place the pressure vessel in the oil bath maintained at a temperature of 99 °C ± 0.5 °C (210 °F ± 1.0 °F). As the pressure rises, if needed, intermittently release oxygen from the pressure vessel until a constant pressure of 758 kPa ± 14 kPa (110 psi ± 2 psi) is obtained and maintained for at least 2 h. A gradual drop in pressure indicates a continuous leak in the pressure vessel. Observe and record the pressure at least every 24 h. In case a leak develops, do not report the results but repeat the test.

8.4 Start timing at the moment of immersion of the pressure vessel in the oil bath, and continue the oxidation for the time period specified.

NOTE 5—Specifications are usually given in terms of pressure drop in pounds per square inch, or kilopascals at one or more time intervals, for instance, after 100 h, 200 h, and so forth.

9. Report

9.1 Report the average of duplicate determinations as pressure drop in pounds per square inch, or kilopascals for the specified test time, or times in hours, according to Test Method D942.

10. Precision and Bias

10.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Mean Pressure Drop, kPa (psi)	Repeatability
0 to 34.5 (0 to 5)	13.8 (2)
Over 34.5 to 68.9 (5 to 10)	20.7 (3)
Over 68.9 to 138 (10 to 20)	41.4 (6)
Over 138 to 379 (20 to 55)	68.9 (10)

10.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Mean Pressure Drop, kPa (psi)	Reproducibility
0 to 34.5 (0 to 5)	20.7 (3)
Over 34.5 to 68.9 (5 to 10)	34.5 (5)
Over 68.9 to 138 (10 to 20)	55.2 (8)
Over 138 to 379 (20 to 55)	138 (20)

NOTE 6—These precision values apply only to that portion of the data for which oxygen is absorbed at a rate approximately proportional to time (for example, the induction period). The end of the induction period is evidenced by a rapid acceleration in the rate of oxygen absorption in a short time interval.

10.2 The following information on the precision of this test method has been developed by the Energy Institute (London). The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.2.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Mean Pressure Drop, kPa	Repeatability	Mean Pressure Drop, psig	Repeatability
0	5	0 to 2	1
5 to 20	10	3 to 6	2
25 to 40	15	7 to 10	3
45 to 60	20	11 to 14	4
65 to 85	25	15 to 18	5
90 to 105	30	19 to 20	6
110 to 125	35		
130 to 140	40		

10.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Mean Pressure Drop, kPa	Reproducibility	Mean Pressure Drop, psig	Reproducibility
0	10	0 to 3	2
5 to 20	15	4 to 6	3
25 to 35	20	7 to 9	4
40 to 50	25	10 to 12	5
55 to 65	30	13 to 15	6
70 to 80	35	16 to 17	7
85 to 95	40	18 to 20	8
100 to 110	45		
115 to 125	50		
130 to 135	55		
140	60		

These precision values have been obtained by statistical examination of interlaboratory test results,[6] and were first published in 1965.

[6] See *IP Standards for Petroleum and Its Products,* Part 1, Appendix E.