



Designation: D 942 – 02

An American National Standard



Designation: 142/85 (92)

Standard Test Method for Oxidation Stability of Lubricating Greases by the Oxygen Pressure Vessel Method¹

This standard is issued under the fixed designation D 942; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

This test method has been approved by the sponsoring committee and accepted by the cooperating societies in accordance with the established procedures.

This standard has been approved for use by agencies of the 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 *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.* For specific hazard statements see Sections 6 and 7.

2. Referenced Documents

2.1 ASTM Standards:

A 240 Specification for Heat-Resisting Chromium and Chromium-Nickel Stainless Steel Plate, Sheet, and Strip for Pressure Vessels²

D 525 Test Method for Oxidation Stability of Gasoline (Induction Period Method)³

E 1 Specification for ASTM Thermometers⁴

2.2 Other Standards:

IP Specification for Standard IP Thermometers⁵

BS 970:1983 Part I, Section S⁶

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 110 psi (758 kPa). 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 Institute of Petroleum 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 .

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 Gage and Oil Bath* as described in detail in the Annex.

NOTE 3—Other constant-temperature baths may be used if they are

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 on Oxidation.

In the IP, this test method is under the jurisdiction of the Standardization Committee.

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² *Annual Book of ASTM Standards*, Vol 01.03.

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ *Annual Book of ASTM Standards*, Vol 14.03.

⁵ Available from The Institute of Petroleum, 61 New Cavendish St., London W1M, 8AR, England.

⁶ Available from British Standards Institute, 2 Park St., London, England W1A2B5.

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

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 E 1 or in the Specifications for IP Standard Thermometers:

Temperature Range	Thermometer	
	ASTM	Number
95 to 103°C	22C	IP 24C
204 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, gages 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-4-1⁷ 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 D 525, 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 ± 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 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 100 psi (689 kPa) 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	psi	kPa
17 to 20	62 to 68	85	586
20 to 23	68 to 74	86	593
23 to 27	74 to 80	87	600
27 to 30	80 to 86	88	607
30 to 33	86 to 92	89	614
33 to 37	92 to 98	90	621
37 to 40	98 to 104	91	627

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 110 ± 2 psi (758 ± 14 kPa) 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 $99 \pm 0.5^\circ\text{C}$ ($210 \pm 1.0^\circ\text{F}$). As the pressure rises, if needed, intermittently release oxygen from the pressure vessel until a constant pressure of 110 ± 2 psi (758 ± 14 kPa) is obtained and maintained for at least 2 h. A gradual drop in pressure indicates a continuous leak in the pressure vessel.

⁷ Available from Compressed Gas Association, 500 Fifth Ave., New York, NY 10036.

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, 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 D 942.

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

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

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 Institute of Petroleum

(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, psig	Repeatability	Mean Pressure Drop, kPa	Repeatability
0 to 2	1	0	5
3 to 6	2	5 to 20	10
7 to 10	3	25 to 40	15
11 to 14	4	45 to 60	20
15 to 18	5	65 to 85	25
19 to 20	6	90 to 105	30
		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, psig	Reproducibility	Mean Pressure Drop, kPa	Reproducibility
0 to 3	2	0	10
4 to 6	3	5 to 20	15
7 to 9	4	25 to 35	20
10 to 12	5	40 to 50	25
13 to 15	6	55 to 65	30
16 to 17	7	70 to 80	35
18 to 20	8	85 to 95	40
		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,⁸ and were first published in 1965.

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

11. Keywords

11.1 grease; oxidation

⁸ See *IP Standards for Petroleum and Its Products*, Part 1, Appendix E.

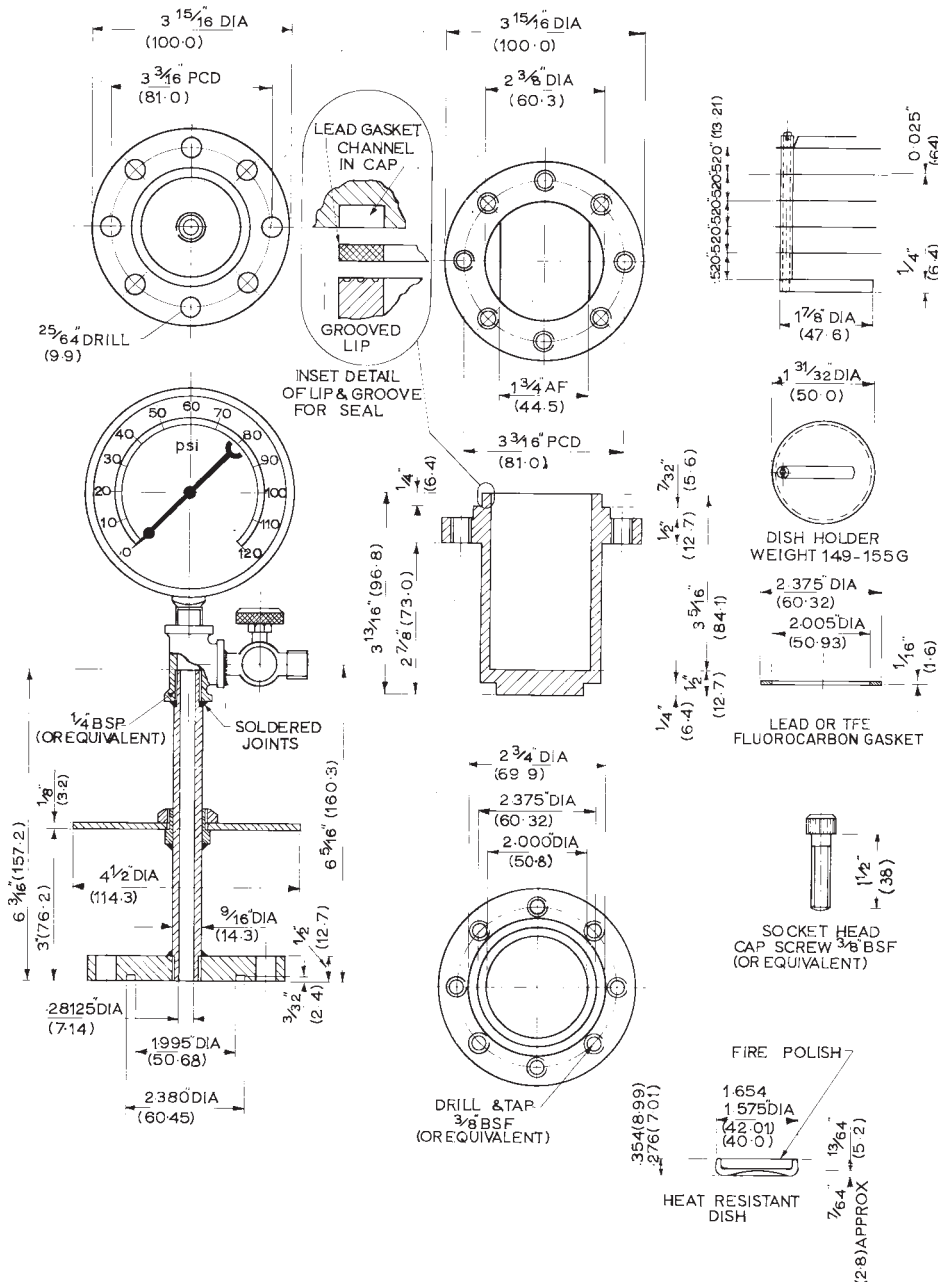
ANNEX

(Mandatory Information)

A1. APPARATUS

A1.1 *Oxidation Pressure Vessel*, of the type and conforming to the dimensions shown in Fig. A1.1, shall be made of 18 % chromium, 8 % nickel alloy steel. A suitable material is an alloy steel conforming to Grade S, Type 304, of Specifications A 240 or BS 970: 1983 Part 1, Section S (steel 302S31 or 321S31). The pressure vessel shall be capable of withstanding with safety a working pressure of 180 psi (1241 kPa) at 99°C

(210°F) and provided with a lead or TFE-fluorocarbon gasket for sealing. To facilitate cleaning, give a high polish to the interior surfaces of the pressure vessel, the lid, and the inside of the pipe carrying the pressure gage. The pressure vessel shall be so constructed that its volume without the dish holder and dishes is 185 ± 6 mL, measured to the level of the gage. This may be checked by assembling the pressure vessel, removing



NOTE—All dimensions are in inches (millimetres).

FIG. A1.1 Apparatus for Oxidation Stability Test of Lubricating Greases

the gage, and measuring the amount of liquid required to fill the pressure vessel to the level of the gage connection. A screw-on lid may be used instead of the bolt-on lid shown in Fig. A1.1.

A1.2 *Pressure Gage*, suitable for use with oxygen and mineral oil, shall be of the indicating type, graduated in intervals of 1 psi (6.89 kPa) per scale division, with a maximum reading of at least 120 psi (827 kPa) and capable of an accuracy of at least 0.5 psi (3.45 kPa), within the interval 90 to 120 psi (621 to 827 kPa). Attach the gage to the stem as shown in Fig. A1.1. A recording pressure gage may be used in place of the prescribed indicating pressure gage if it satisfies the volume and accuracy requirements previously stated.

A1.3 *Oil Bath*, capable of being controlled at $99 \pm 0.5^{\circ}\text{C}$ ($210 \pm 1^{\circ}\text{F}$), with a temperature gradient less than 1°F (0.5°C) in the body of the oil used, and of sufficient depth to allow

submersion of the pressure vessel to the proper depth. Circulation of the oil heating medium by a pump or stirring is recommended. Sufficient heat capacity shall be provided to return the bath to the required temperature within 60 min after immersion of the pressure vessel. Provide the bath with a thermometer well such that the 96.8°C (206°F) point of the thermometer is at the same level as the upper surface of the bath cover. Adjust the level of the bath so that the top of the pressure vessel is submerged approximately 50 mm below its surface. Arrange the bath so that there are no drafts or wide fluctuations in temperature around the gages.

A1.4 *Dish Holder*, constructed of 18 % chromium, 8 % nickel alloy steel as described for the oxidation pressure vessel, shall conform to the dimensions prescribed in Fig. A1.1.

A1.5 *Sample Dishes*, of glass conforming to the dimensions prescribed in Fig. A1.1.

SUMMARY OF CHANGES

Subcommittee D02.09 has identified the location of selected changes to this standard since the last issue (D 942–02) that may impact the use of this standard.

- (1) Changed the document list and cleaning procedure.
- (2) Changed the instructions in Paragraph 8.2.
- (3) Replaced the word “bomb” with “pressure vessel” throughout.

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