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AMERICAN SOCIETY FOR TESTING AND MATERIALS  
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## Standard Test Method for Oxidation Stability of Lubricating Greases by the Oxygen Bomb Method<sup>1</sup>

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 reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*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.*

<sup>ε1</sup> NOTE—Section 11 was added editorially in October 1995.

### 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 Vessels<sup>2</sup>

E 1 Specification for ASTM Thermometers<sup>3</sup>

#### 2.2 Other Standards:

IP Specification for Standard IP Thermometers<sup>4</sup>

BS970:1983 Part I, Section S<sup>5</sup>

Compressed Gas Association Booklets G-4 and G-4-1<sup>6</sup>

### 3. Summary of Test Method

3.1 The sample of grease is oxidized in a bomb 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 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  $6.894757 \times 10^3$ .

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 Bomb, Sample Dish, Dish Holder, Pressure Gage and Oil Bath* as described in detail in the Annex.

NOTE 2—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 bomb at the

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-2 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 01.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.03.

<sup>4</sup> Available from The Institute of Petroleum, 61 New Cavendish St., London W1M, 8AR, England.

<sup>5</sup> Available from British Standards Institute, 2 Park St., London, England W1A2B5.

<sup>6</sup> Available from Compressed Gas Assn., 500 Fifth Ave., New York, NY 10036.

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

## 6. Material

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

NOTE 3—**Precaution:** 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 for details of safe practice in the use of oxygen.

6.2 *n-Heptane*. (**Warning**—See Note 4.)

NOTE 4—**Warning:** Flammable. Harmful if inhaled. **Precaution**—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.

6.3 *Sulfuric Acid-Chromic Acid*. (**Warning**—See Note 5.)

NOTE 5—**Warning:** Causes severe burns. A recognized carcinogen. Strong oxidizer; contact with organic material may cause fire. Hygroscopic. **Precaution**—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.

## 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 (**Precaution**—See Note 6.), then with hot water and soap powder, and finally with hot sulfuric acid-chromic acid solution (**Precaution**—See Note 7.). 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.

NOTE 6—**Precaution:** Handle in well-ventilated area, preferably in a hood. Keep away from heat, sparks, and open flame. Keep container closed when not in use.

NOTE 7—**Precaution:** Avoid skin contact, which may cause severe burns.

7.2 If lacquer is found after a run, clean the inside of the

oxidation bomb and the metal supports for the bomb dishes by immersing in hot solvent 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  $4.00 \pm 0.01$  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 bomb, place a small ball of glass wool in the bottom of the stem.

8.2 Place the dish holder in the bomb and close the bomb by tightening the bolts slowly and uniformly. Clear the air from the bomb 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 bomb to stand overnight to make sure there are no leaks.

NOTE 8—It has been found that pressure readings as shown above will result in a pressure reading of  $110 \pm 2$  psi ( $758 \pm 14$  kPa) when the bomb 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 bomb to be satisfactory.

8.3 Place the bomb 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 bomb until a constant pressure of  $110 \pm 2$  psi ( $758 \pm 14$  kPa) is obtained and maintained for at least 2 h. A gradual drop in pressure indicates a continuous leak in the bomb. 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 bomb in the oil bath, and continue the oxidation for the time period specified.

NOTE 9—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, etc.

## 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.