BS 2000 :
Part 142 : 1993

Methods of test for

Petroleum and its products

Part 142. Determination of oxidation stability of lubricating grease – Oxygen bomb method

(Identical with IP 142/85(92))

Confirmed
January 2010

BSi
British Standards

Foreword

This British Standard, having been prepared under the direction of the Petroleum Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 28 February 1993.

This British Standard supersedes BS 2000 : Part 142 : 1982, which is withdrawn.

In accordance with BSI procedure this Standard was made available for public comment as 92/56099 DC.

BS 2000 comprises a series of test methods for petroleum and its products that are published by the Institute of Petroleum (IP) and have been accorded the status of a British Standard. Each method should be read in conjunction with the preliminary pages of 'IP Standard methods for analysis and testing of petroleum and related products' which gives details of the BSI/IP agreement for publication of the series, provides general information on safety precautions, sampling and other matters, and lists the methods published as Parts of BS 2000.

The numbering of the Parts of BS 2000 follows that of the corresponding methods published in 'IP Standard methods for analysis and testing of petroleum and related products'. Under the terms of the agreement between BSI and the Institute of Petroleum, the revised version of BS 2000 : Part 142 will be published by the IP (in 'Standard methods for analysis and testing of petroleum and related products' and as a separate publication). BS 2000 : Part 142 : 1993 is thus identical with IP 142/85, which was reapproved in 1992. Square brackets marked in the margin of this IP Standard indicate text that differs from the previous edition.

IP 142 was previously published as a British Standard as BS 5298 (now withdrawn) which was subsequently renumbered and issued in the BS 2000 series.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Committee reference PTC/13

IP 142/85 (1992) ASTM D942-78 (1990) BS 2000: Part 142: 1993

Determination of oxidation stability of lubricating grease – Oxygen bomb method[1]

This method was adopted as a joint ASTM–IP Standard Method in 1964.

This standard does not purport to address all of the safety problems 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.

FOREWORD This method is technically equivalent to DIN 51808

1. SCOPE

1.1. This method describes the test for determining 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.

2. SUMMARY OF METHOD

2.1. The sample of grease is oxidized in a bomb heated to 99°C and filled with oxygen at 7.5 bar. Pressure[2] 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.

3. SIGNIFICANCE

3.1. This method may be used for quality control to indicate batch-to-batch uniformity. It is not intended for the prediction of the stability of greases under dynamic service conditions nor is it intended for the prediction of the stability of greases stored in commercial containers.

4. APPARATUS

4.1. *Oxidation Bomb, Sample Dish, Dish Holder, Pressure Gauge and Oil Bath* – as described in detail in the Appendix.

NOTE 1: 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 Appendix and can be shown to maintain the bomb at the prescribed test temperature.

4.2. *Thermometer* – having a range shown below and conforming to the requirements as prescribed in ASTM specification E1 or specifications for IP Standard Thermometers:

Temperature range	Thermometer no. ASTM	IP
95 to 103°C	22C	24C

[1]This method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants.
In the IP, this method is under the jurisdiction of the Standardization Committee.

5. MATERIAL

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

6. PREPARATION OF APPARATUS

6.1. Clean the sample dishes from all contamination from previous runs and from dust settling from the air by washing them with solvent, then with hot water and soap powder, and finally with hot sulphuric acid–chromic acid 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.

6.2. 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.

7. PROCEDURE

7.1. 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 bomb, place a small ball of glass wool in the bottom of the stem.

7.2. 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[2] of 6.8 bar 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:

[2]The Institute of Petroleum uses the bar for the measurement of pressure

$1 \text{ bar} = 10^5 \text{ N/m}^2 = 10^5 \text{ Pascal}$

Room temperature °C	Pressure bar
17 to 20	5.9
20 to 23	5.9
23 to 27	6.0
27 to 30	6.1
30 to 33	6.1
33 to 37	6.2
37 to 40	6.3

Allow the bomb to stand overni[illegible] to make sure there are no leaks.

NOTE 2: It has been found that press[illegible]readings as shown above will result in a pressure reading of 7.5±0.1 bar when the bomb is placed in the bath in the following step, (6.3), 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.

7.3. Place the bomb in the oil bath maintained at a temperature of 99±0.5°C. As the pressure rises, intermittently release oxygen from the bomb until a constant pressure of 7.5±0.1 bar 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.

7.4 Start timing at the moment of immersion of the bomb in the oil bath, and continue the oxidation for the time period specified.

NOTE 3: Specifications are usually given in terms of pressure drop in bar at one or more time intervals, for instance, after 100, 200 h, etc.

8. REPORT

8.1. Report the average of duplicate determinations as pressure drop in bar for the specified test time, or times in hours, according to ASTM D942–IP 142.

9. PRECISION

9.1. The following criteria should be used for judging the acceptability of results (95% confidence):

9.1.1. *Repeatability* – Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

Mean pressure drop, bar	Repeatability
0 to 0.34	0.15
Over 0.34 to 0.68	0.25
Over 0.68 to 1.37	0.40
Over 1.37 to 3.77	0.70

9.1.2. *Reproducibility* – The results submitted by each of two laboratories should be considered suspect if they differ by more than the following amounts:

Mean pressure drop, bar	Reproducibility
0 to 0.34	0.20
Over 0.34 to 0.68	0.35
Over 0.68 to 1.37	0.60
Over 1.37 to 3.77	1.40

NOTE 4: These precision values apply only to that portion of the data for which oxygen is absorbed at a rate approximately proportional to time, and before the induction period occurs, as evidenced by a rapid acceleration in the rate of oxygen absorption in a short time interval.

NOTE 5: The following information on the precision [Note 4] of this method has been developed by the Institute of Petroleum and is being investigated by ASTM Committee D–2.

The following criteria should be used for judging the acceptability of results (95% confidence):

(*a*) *Repeatability* – Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

Mean pressure drop, bar	Repeatability
0	0.05
0.05 to 0.20	0.10
0.25 to 0.40	0.15
0.45 to 0.60	0.20
0.65 to 0.85	0.25
0.90 to 1.05	0.30
1.10 to 1.25	0.35
1.30 to 1.40	0.40

(*b*) *Reproducibility* – The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amounts:

Mean pressure drop, bar	Reproducibility
0	0.10
0.05 to 0.20	0.15
0.25 to 0.35	0.20
0.40 to 0.50	0.25
0.55 to 0.65	0.30
0.70 to 0.80	0.35
0.85 to 0.95	0.40
1.00 to 1.10	0.45
1.15 to 1.25	0.50
1.30 to 1.35	0.55
1.40	0.60

NOTE: These precision values have been obtained by statistical examination of inter-laboratory test results* and were first published in 1965.

APPENDIX

APPARATUS

A1. *Oxidation Bomb* – of the type and conforming to the dimensions shown in Fig. 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 ASTM Specification A240, for Heat-Resisting Chromium and Chromium-Nickel Stainless Steel Plate, Sheet, and Strip for Pressure Vessels[3] or to BS 970:1983, Part 1 (Steel 302 S31 or 321 S31). The bomb shall be capable of withstanding with safety a working pressure of 12.4 bar at 99°C and provided with a lead gasket for sealing. To facilitate cleaning, give a high polish to the interior surfaces of the bomb, the lid, and the inside of the pipe carrying the pressure gauge. The bomb shall be so constructed that its volume without the dish holder and dishes is 185±6 ml, measured to the level of the gauge. This

*See Appendix E.

[3]*Annual Book of ASTM Standards*, Vols. 01.03 and 01.04.

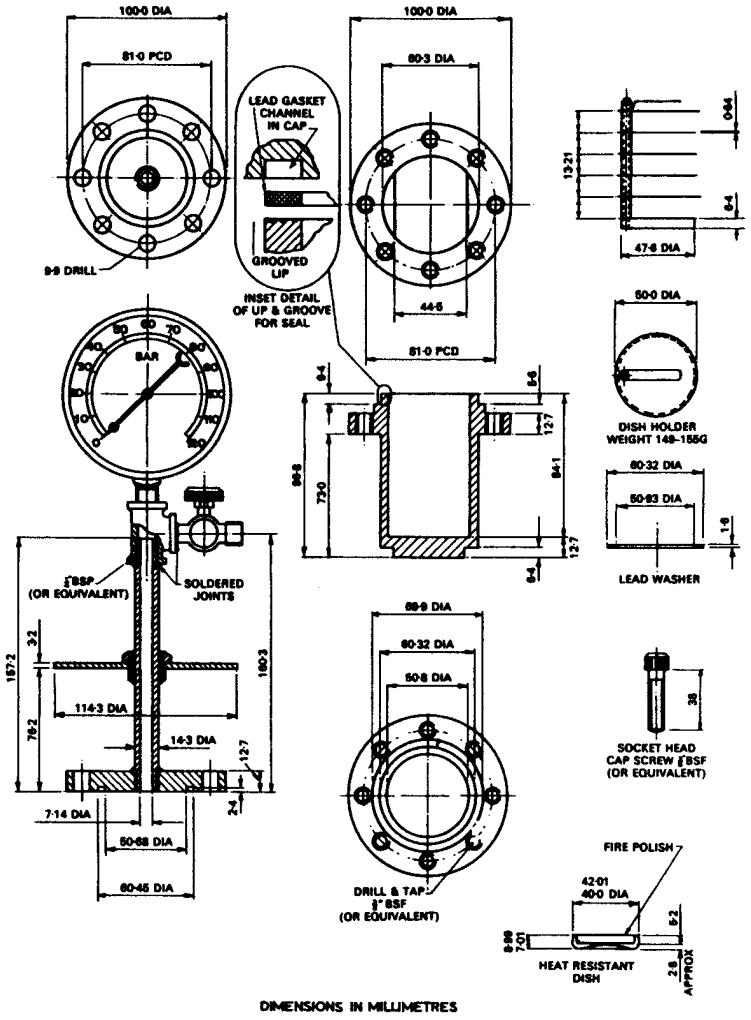

Fig. 1. Apparatus for oxidation stability test of lubricating greases.

may be checked by assembling the bomb, removing the gauge, and measuring the amount of liquid required to fill the bomb to the level of the gauge connexion.

A2. *Pressure Gauge* – suitable for use with oxygen and mineral oil, shall be of the indicating type, graduated in intervals of 0.1 bar per scale division, with a maximum reading of at least 8.2 bar and capable of an accuracy of at least 0.05 bar within the interval 6.2 to 8.2 bar. Attach the gauge to the stem as shown in Fig. 1. A recording pressure gauge may be used in place of the prescribed indicating pressure gauge if it satisfies the volume and accuracy requirements previously stated.

A3. *Oil Bath* – capable of being controlled at 99 ± 0.5°C, with a temperature gradient less than 0.5°C in the body of the oil used, and of sufficient depth to allow submersion of the bomb 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 bomb. Provide the bath with a thermometer well such that the 96.8°C 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 bomb is submerged approximately 50 mm below its surface. Arrange the bath so that there are no draughts or wide fluctuations in temperature around the gauges.

A4. *Dish Holder* – constructed of 18% chromium, 8% nickel alloy steel as described for the oxidation bomb, shall conform to the dimensions prescribed in Fig. 1.

A5. *Sample Dishes* – of glass conforming to the dimensions prescribed in Fig. 1.