



# Standard Test Method for Universal Oxidation Test for Hydraulic and Turbine Oils Using the Universal Oxidation Test Apparatus<sup>1</sup>

This standard is issued under the fixed designation D 5846; 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.

## 1. Scope

1.1 This test method covers a procedure for evaluating the oxidation stability of petroleum base hydraulic oils and oils for steam and gas turbines.

1.2 This test method was developed to evaluate the oxidation stability of petroleum base hydraulic oils and oils for steam and gas turbines.

1.2.1 Rust and oxidation inhibited hydraulic, anti-wear hydraulic and turbine oils of ISO 32–68 viscosity were used to develop the precision statement. This test method has been used to evaluate the oxidation stability of fluids made with synthetic basestock and in-service oils; however, these fluids have not been used in cooperative testing to develop precision data.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 and health practices and determine the applicability of regulatory limitations prior to use.* Identified hazardous chemicals are listed in 7.3, 7.6, and 7.8. Before using this test method, refer to suppliers' safety labels, Material Safety Data Sheets, and other technical literature.

## 2. Referenced Documents

### 2.1 ASTM Standards:

A 510 Specification for General Requirements for Wire Rods and Coarse Round Wire, Carbon Steel<sup>2</sup>

B 1 Specification for Hard-Drawn Copper Wire<sup>3</sup>

D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration<sup>4</sup>

D 943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils<sup>4</sup>

D 974 Test Method for Acid and Base Number by Color-Indicator Titration<sup>4</sup>

D 3339 Test Method for Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration<sup>5</sup>

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>5</sup>

D 4740 Test Method for Stability and Compatibility of Residual Fuels by Spot Test<sup>5</sup>

D 4871 Guide for Universal Oxidation/Thermal Stability Test Apparatus<sup>5</sup>

D 5770 Test Method for Semi-Quantitative Micro Determination of Acid Number of Used Lubricating Oils<sup>6</sup>

2.2 *Institute of Petroleum Standard:*<sup>7</sup>

IP 2546 Practice for Sampling of Petroleum Products; alternate to Practice D 4057

2.3 *British Standard:*<sup>8</sup>

BS 1829 Specification for Carbon Steel Wire; alternate to Specification A 510

2.4 ASTM Adjuncts:

Reference Spot Sheet (Test Method D 4740), for visual rating of blotter spots<sup>9</sup>

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *oxidation life, n*—of an oil, the time in hours required for degradation of the oil under test.

3.1.2 *inhibited mineral oil, n*—a petroleum oil containing additives to retard oxidation.

3.1.3 *universal oxidation test, n*—the apparatus and procedures described in Guide D 4871.

## 4. Summary of Test Method

4.1 An oil sample is contacted with air at 135°C in the presence of copper and iron metals. The acid number and spot forming tendency of the oil are measured daily. The test is terminated when the oxidation life of the oil has been reached.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.09.0D on Oxidation of Lubricants.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 01.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 02.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 05.01.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 05.02.

<sup>6</sup> *Annual Book of ASTM Standards*, Vol 05.03.

<sup>7</sup> Available from the Institute of Petroleum, 61 New Cavendish St., London, W1G 7AR, U.K.

<sup>8</sup> Available from British Standards Institute, 389 Chiswick High Rd., London, W4 4AL, U.K.

<sup>9</sup> Available from ASTM International Headquarters. Order ADJD4740.

4.2 The oil is considered to be degraded when either its acid number (measured by Test Methods D 974 or D 664) has increased by 0.5 mg KOH/g over that of new oil; or when the oil begins to form insoluble solids so that when a drop of oil is placed onto a filter paper it shows a clearly defined dark spot surrounded by a ring of clear oil.

**5. Significance and Use**

5.1 Degradation of hydraulic fluids and turbine oils, because of oxidation or thermal breakdown, can result in the formation of acids or insoluble solids and render the oil unfit for further use.

5.2 This test method can be used to estimate the relative oxidation stability of petroleum-base oils. It should be recognized that correlation between results of this test and the oxidation stability in use can vary markedly with service conditions and with various oils.

**6. Apparatus**

6.1 *Heating Block*, as shown on the right in Fig. 1, and as further described in Guide D 4871, to provide a controlled constant temperature for conducting the test.

6.1.1 Test cells are maintained at a constant elevated temperature by means of a heated aluminum block which surrounds each test cell.

6.1.2 The test cells shall fit into the block to a depth of  $225 \pm 5$  mm. When centered, the side clearance of the 38 mm outside diameter glass tube to the holes in the aluminum block shall not exceed 1 mm in any direction.

6.2 *Temperature Control System*, as shown at lower left in Fig. 1, and as further described in Guide D 4871, to maintain the test oils in the heating block at  $135 \pm 0.5^\circ\text{C}$  for the duration of the test.

6.3 *Gas Flow Control System*, as shown in the upper left in Fig. 1, and as further described in Guide D 4871, to provide dry air at a flow rate of  $3.0 \pm 0.5$  L/h to each test cell.

6.3.1 A gas flow controller is required for each test cell.

6.3.2 Flowmeters shall have a scale length sufficiently long to permit accurate reading and control to within 5 % of full scale.

6.3.3 The total system accuracy shall meet or exceed the following tolerances: Inlet pressure regulator within 0.34 kPa (0.05 psig) of setpoint; total flow control system reproducibility within 7 % of full scale; repeatability of measurement



FIG. 1 Apparatus, Showing Gas Flow Control System, Temperature Control System, and Heating Block

within 0.5 % of full scale.

6.4 *Oxidation Cell*, borosilicate glass, as shown in Fig. 2, and as further described in Guide D 4871. This consists of a test cell of borosilicate glass, standard wall; 38 mm outside diameter,  $300 \pm 5$ -mm length, with open end fitted with a 34/45 standard-taper, ground-glass outer joint.

6.5 *Gas Inlet Tube*, as shown in Fig. 2, and as further described in Guide D 4871. This consists of an 8-mm outside diameter glass tube, at least 455 long, lower end with fused capillary 1.5 to 3.5 mm inside diameter. The capillary bore shall be  $15 \pm 1$  mm long. The lower tip is cut at a  $45^\circ$  angle.

6.6 *Basic Head*, as shown in Fig. 2, and as further described in Guide D 4871. This is an air condenser, with 34/45 standard-taper, ground-glass inner joint, opening for gas inlet tube, septum port for sample withdrawal, and exit tube to conduct off-gases and entrained vapors. Overall length shall be  $125 \pm 5$  mm.

6.7 Test precision was developed using the universal oxidation/thermal stability test apparatus described in Guide

D 4871.<sup>10</sup> Alternate apparatus designs for sample heating and for temperature and flow control shall be acceptable provided they are shown to maintain temperature and gas flow within the specified limits.

## 7. Reagents and Materials

7.1 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.<sup>11</sup> 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.

7.2 *Abrasive Cloth*, silicon carbide, 100-grit with cloth backing.

7.3 *Acetone*, reagent grade. (**Warning**—Acetone is flammable and a health hazard.)

7.4 *Air*, dry with dew point  $-60^\circ$ .

7.5 *Electrolytic Copper Wire*, 1.63 mm in diameter (No. 14 American Wire Gage or No. 16 Imperial Standard Wire Gage), 99.9 % purity, conforming to Specification B 1, is preferred.

7.6 *Heptane*, knock-test grade, conforming to the following requirements: (**Warning**—*n*—Heptane is flammable and a health hazard.)

Density at 20°C	0.6826 to 0.6839
Refractive index at 20°C	1.3876 to 1.3879
Solidification temperature, min	$-90.72^\circ$
Distillation	50 % shall distill between $98.38^\circ$ and $98.48^\circ$ . Temperature rise between 20 and 80 % recovered shall be $0.20^\circ$ max

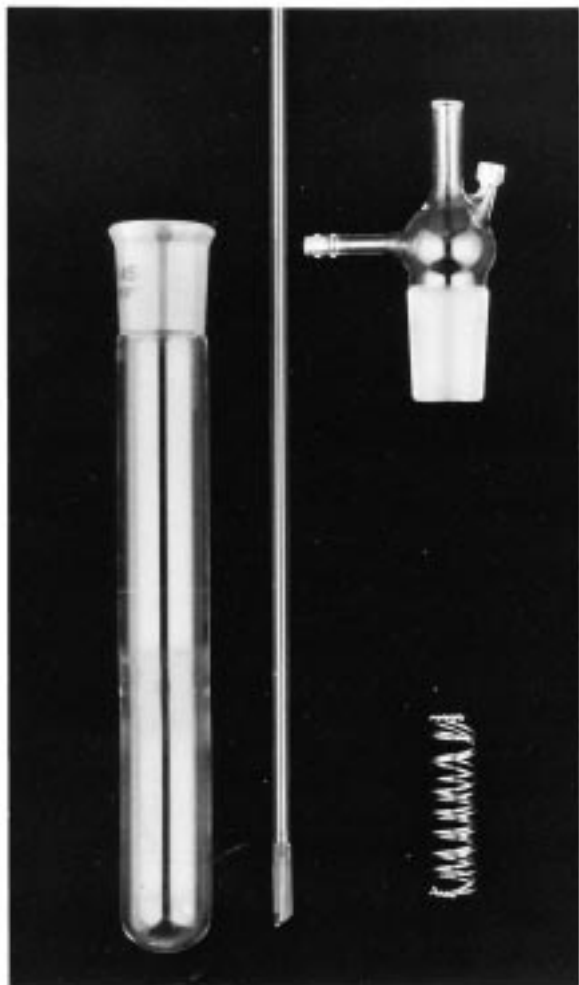
7.7 *Low-Metalloid Steel Wire*, 1.59 mm in diameter (No. 16 Washburn and Moen Gage). Carbon steel wire, soft bright annealed and free from rust, of Grade 1008 as described in Specification A 510, is preferred. Similar wire conforming to British Standard 1829 is also satisfactory.

7.8 *Propanol-2 (iso-Propyl Alcohol)*, reagent grade. (**Warning**—*iso*-Propyl alcohol is flammable and a health hazard.)

7.9 *Test Paper*, chromatographic or filter paper, cellulose, medium porosity, qualitative or quantitative grade. Cut the paper into 50 mm squares or use as larger sheets, ruled with hard pencil into 50 mm squares without cutting.<sup>12</sup>

## 8. Sampling

8.1 Samples for this test can come from tanks, drums, small



**FIG. 2 Test Cell, Including Oxidation Cell, Gas Inlet Tube, Basic Head, and Finished Catalyst Coil**

<sup>10</sup> A standard commercial apparatus has been found satisfactory for the purpose of this test method. This apparatus, including heating block, temperature control system, flow control system, and glassware, is available from Falex Corp., 1020 Airpark Dr., Sugar Grove, IL 60554. Glassware for the universal oxidation test apparatus is also available from W. A. Sales, Ltd., 419 Harvester Court, Wheeling, IL 60090.

<sup>11</sup> *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.

<sup>12</sup> Whatman 1 grade, available in 57 by 46 cm sheets, have been found satisfactory for this test method. Paper sheets should be stored without folding, rolling, or bending, in a tightly closed container.

containers, or operating equipment. Therefore, use the applicable apparatus and techniques described in Practice D 4057 or IP 2546 to obtain suitable samples.

8.2 Special precautions to preserve the integrity of a sample will not normally be required. Follow good laboratory practice. Avoid undue exposure of samples to sunlight or strong direct light. Use only samples that are homogeneous on visual inspection.

## 9. Preparation of Apparatus

### 9.1 *Cleaning Glassware:*

9.1.1 Clean new glassware by washing with a hot detergent solution, using a bristle brush; rinse thoroughly with tap water. When any visible deposits remain, soak with hot detergent solution and repeat rinses. After final cleaning by soaking with a suitable cleaning solution<sup>13</sup> rinse thoroughly with tap water then distilled water and dry at room temperature or in an oven. A final rinse with *iso*-propyl alcohol or acetone will hasten drying at room temperature.

9.1.2 Clean used glassware immediately following the end of a test. Drain the used oil completely. Rinse all glassware with heptane to remove traces of oil. Then clean the glassware by the procedure described in 9.1.1 before re-use.

### 9.2 *Cleaning Catalyst:*

9.2.1 Clean equal lengths of iron and copper wire with wads of absorbent cotton wet with heptane or other saturated paraffinic solvent of comparable boiling point. Follow by abrasion with 100-grit silicon carbide abrasive cloth until fresh metal surfaces are exposed. Wipe with dry absorbent cotton to remove loose particles of metal and abrasives. Repeat with fresh cotton until no particles are visible. In the following operations, handle the catalyst with clean gloves (cotton, rubber, or plastic) to prevent contamination from oils on the skin.

9.2.2 One procedure for preparing clean catalyst wire is to cut  $0.50 \pm 0.01$  m lengths of wire. Hold one end of the wire tightly with a pair of clean pliers or in a vise while cleaning with the abrasive cloth. Reverse ends of the wire and repeat.

9.2.3 Alternately, clean a longer length of wire (3 to 5 m) and then cut  $0.50 \pm 0.01$  m lengths from the clean wire.

### 9.3 *Preparation of Catalyst Coil:*

9.3.1 Twist the iron and copper wires tightly together at one end for three twists. With the two wires parallel, wind the wires around a cylindrical mandrel to produce a single coil 15.9 to 16.5 mm in inside diameter. The mandrel described in Test Method D 943 is satisfactory, but other cylindrical metal or wood stock can be used. Remove the coil from the mandrel and secure the free ends with three twists. Bend the twisted ends to conform to the shape of the spiral coil. Stretch the coil to produce a finished coil with an overall length of  $80 \pm 8$  mm as shown in Fig. 2.<sup>14</sup>

9.3.2 Store the catalyst coil in a dry, inert atmosphere until use. For storage up to 24 h, the coil can be stored in heptane.

<sup>13</sup> Nochromix Reagent, available from Godax Laboratories, New York, NY, has been found satisfactory for this purpose. Because of extreme hazards, chromic acid cleaning solution is not recommended.

<sup>14</sup> Prepared catalyst coils for use with this test method are available from C and P Catalyst, P.O. Box 520984, Tulsa, OK 74152.

Before use, inspect stored coils to ensure that no corrosion products or contaminating materials are present.

9.4 Use a fresh catalyst coil for each test. Do not reuse coils.

## 10. Procedure

10.1 Adjust the heating block to maintain the oil in the oxidation test cell at  $135 \pm 0.5^\circ\text{C}$ .

10.1.1 Other test temperatures can be used but should be specified.

10.2 Weigh  $100 \pm 1$  g of test fluid into the oxidation test cell.

10.3 Place a cleaned catalyst coil in the oil and fit the basic head and gas delivery tube into the test cell so that the tip of the gas delivery tube rests on the bottom of the test cell inside the catalyst coil, as shown in Fig. 3.

10.4 Insert the test cell into the preheated constant temperature block. Wait 0.5 to 1.0 h for the oil to warm to  $135 \pm 0.5^\circ\text{C}$ .

10.5 Connect an air delivery tube from the flow control system to the gas inlet tube and adjust the flowmeter control to deliver dry air at  $3 \pm 0.5$  L/h. Record this time as the start of the test.

10.6 Recheck the air flow after the test is underway and at



**FIG. 3 Assembled Test Cell Ready to Insert Into Heating Block**

least once daily during the test. Adjust to maintain the air flow as needed.<sup>15</sup>

10.7 Withdraw a  $2.0 \pm 0.2$  g oil sample for determination of acid number at one-day intervals after the start of the test.

10.7.1 Additional samples are taken for acid number determination if desired. More frequent sampling at a time when the acid number is increasing rapidly is helpful to more precisely determine the time to a 0.5 acid number increase.

10.8 Determine the acid number by Test Method D 3339 or D 5770; alternate test methods such as Test Methods D 664 or D 974 are used when necessary for oils with short test life. Plot the acid number versus time and terminate the test when an increase in acid number of 0.5 mg KOH/g oil compared to the value for new oil is exceeded. Determine the time to an 0.5 acid number increase from the plot. Alternatively, calculate the time using Eq 1.

10.9 For sludge determination, place a sheet or circle of test paper in a horizontal position on a flat surface in an area shielded from direct drafts. Remove a small sample of oil from the test cell using a micro pipette or glass stirring rod. Allow one drop (approximately 0.05 mL) of liquid to fall onto the test paper, approximately in the center of the sheet.

10.10 Allow the liquid to spread on the paper at room temperature for at least 1 h, but no longer than 3 h (Note 1). Observe the test spot and note any inhomogeneity in the form of a dark ring or spot of insoluble solids. Rate the spot according to the following scale, and terminate the test if the spot rating reaches five.

NOTE 1—Some oil additives will change color when exposed to air or light on the test paper. Such a change gives the appearance of a sludge spot, but generally such reactions take longer than 3 h to develop.

#### 10.11 Rating Scale for Sludge Spot.<sup>16</sup>

Reference Spot No.	Characterizing Features
1	Homogeneous spot with no inner ring
2	Faint or poorly defined inner ring
3	Well defined, thin inner ring, only slightly darker than the background
4	Well defined inner ring, thicker than ring in No. 3, and somewhat darker than the background
5	Very dark solid or nearly solid center area, much darker than the background

10.12 If a test sample is consumed, through vaporization or sample removal for acid number determination, to the extent that the catalyst coil becomes exposed above the surface of the oil before the condemning limit for acid number or sludge is reached, the test should be terminated.

## 11. Calculation

11.1 Calculate the oxidation life of the oil,  $L$ , as the number of hours to a 0.5 mg KOH/g oil acid number increase as follows:

$$L = A + ((I + 0.5 - C) \times (B - A)) / (D - C) \quad (1)$$

where:

- $I$  = the acid number of the oil before the start of the test,
- $A$  = the number of test hours when acid number was last measured at less than a 0.5 mg KOH/g increase over  $I$ ,
- $B$  = the number of test hours when acid number was determined to be greater than 0.5 mg KOH/g above  $I$ ,
- $C$  = acid number at  $A$  hours, and
- $D$  = acid number at  $B$  hours.

## 12. Report

12.1 If the test is terminated because of acid number increase, report the oxidation life of the oil as the number of hours to a 0.5 mg KOH/g acid number increase.

12.2 If the test is terminated because of a spot rating of five, report the oxidation life of the oil as the number of hours until the spot was first observed, with (five spot) following the value.

12.3 If the test sample is terminated because of oil depletion and exposure of the catalyst coil above the surface of the oil, report the oxidation life of the oil as greater than the number of hours at which the test was terminated.

12.4 Other qualitative observations, such as color change, formation of sublimate or varnish on the glass, changes in appearance of the catalyst, and so forth, are reported as desired.

## 13. Precision and Bias<sup>17</sup>

### 13.1 Precision:

13.1.1 The precision of the test method for oxidation life as obtained by statistical examination of interlaboratory test results is as follows:

13.1.2 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, and in the normal and correct operation of the test method, exceed the following values only in one case in 20:

0.0614  $X$  for antiwear hydraulic oils

0.0486  $X$  for steam and gas turbine oils

where  $X$  denotes mean value of the oxidation test life, measured in hours to a 0.5 increase in acid number.

13.1.3 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test materials would, in the long run, and in the normal and correct operation of the test method, exceed the following values only in one case in 20:

0.0918  $X$  for antiwear hydraulic oils

0.1400  $X$  for steam and gas turbine oils

where  $X$  denotes mean value of the oxidation test life, measured in hours to a 0.5 increase in acid number.

13.1.4 The precision statement for this test method was determined in two separate round robins. The round robin for antiwear hydraulic oils containing zinc dialkyldithiophosphate used eight new (unused) mineral oil-based lubricants tested by nine cooperators. Oil test life was from 49 to 486 h (raw scores.) The round robin for steam and gas turbine oils used six new (unused) mineral oil-based lubricants tested by five

<sup>15</sup> Exhaust gases need not be collected. Vent exhaust gases properly.

<sup>16</sup> The spot descriptions are taken from Test Method D 4740. A reference spot sheet for that test is available from ASTM International at a nominal charge. Request Adjunct ADJD4740. The oil circle shown in the adjunct is much darker than commonly observed in this test method, but the appearance of the spots are fairly typical.

<sup>17</sup> The research report is being developed.

cooperators. The oils covered oxidation lives from 62 to 718 h (raw scores.)

13.2 *Bias*:

13.2.1 The procedure in this test method has no bias because the value of the oxidation life is defined only in terms of this test method.

#### **14. Keywords**

14.1 hydraulic fluids; oxidation stability; turbine oils; universal oxidation test

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