



Designation: D4636 – 17

Standard Test Method for Corrosiveness and Oxidation Stability of Hydraulic Oils, Aircraft Turbine Engine Lubricants, and Other Highly Refined Oils¹

This standard is issued under the fixed designation D4636; 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.

INTRODUCTION

This test method is the result of combining Federal Test Methods 5307.2 and 5308.7. Features and details of both of these test methods are included, but the new test method is basically Method 5307.2 expanded to include Method 5308.7.²

1. Scope*

1.1 This test method covers the testing of hydraulic oils, aircraft turbine engine lubricants, and other highly refined oils to determine their resistance to oxidation and corrosion degradation and their tendency to corrode various metals. Petroleum and synthetic fluids may be evaluated using moist or dry air with or without metal test specimens.

1.2 This test method consists of a standard test procedure, an alternative Procedure 1, and an alternative Procedure 2. As there are variations possible with this test method, it will be up to the particular specification to establish the conditions required. In addition to temperature, the variables to specify if other than those of the standard procedure or alternative Procedure 1 or 2 are: test time, air flow and humidity, sample frequency, test fluid quantity, and metal specimen(s).

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3.1 *Exception*—The values in parentheses in some of the figures are provided for information only for those using old equipment based on non-SI units.

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.

1.5 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:*³

[D91 Test Method for Precipitation Number of Lubricating Oils](#)

[D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids \(and Calculation of Dynamic Viscosity\)](#)

[D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration](#)

[D1193 Specification for Reagent Water](#)

[D3339 Test Method for Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration](#)

2.2 *U.S. Federal Test Method Standards:*⁴

[FED-STD-791 Testing Method of Lubricants, Liquid Fuels, and Related Products](#)

[Method 5307.2 Corrosiveness and Oxidation Stability of Aircraft Turbine Engine Lubricants \(Withdrawn\)](#)

[Method 5308.7 Corrosiveness and Oxidation Stability of Light Oils \(Metal Squares\)](#)

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 D02.09.0D on Oxidation of Lubricants.

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² FED-STD-791D is the parent document containing both test methods. As of publication on Nov. 6, 2009, it no longer contains withdrawn Method 5307.2.

³ 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.

⁴ Available from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia PA 19111-5094 (<http://assist.daps.dla.mil>).

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

MIL-S-13282 Refined Silver (99.95) (P07015)

2.3 *Other Standards:*⁵

AMS 4616 Silicon Iron Bronze (C65900)

AMS 4908 Titanium Alloy—(8 % Mn) Annealed (R56080)

AMS 6490 Steel (M50) (T11350)

QQ-A-671 Cadmium Anod (L01900)

QQ-C-576 Copper Electrolytic Tough Pitch (ETP) (C11000)

QQ-M-44 Magnesium Alloy AZ31B Condition H24 or H26 (M11311)

QQ-S-698 Low-Carbon Steel 1010, CR Temper No. 4 or 5 (G10100)

QQ-A-250/4 Aluminum Alloy 2024 T-3 or T-4 (A92024)

3. Summary of Test Method

3.1 This test method consists of a standard test procedure (see 10.1), an alternative Procedure 1 (see 10.2), and an alternative Procedure 2 (see 10.3). The standard test procedure uses washer-shaped metal specimens stacked on the air tube, 200 mL of test oil, 10 L/h air flow rate, and periodic test oil withdrawal and evaluation. Alternative Procedure 1 uses washer-shaped metal specimens, 165 mL of test oil, 10 L/h air flow rate, and no periodic test oil sampling. Alternative Procedure 2 uses square metal specimens tied together resting vertically in the large glass tube, 100 mL of test oil, 5 L/h air flow rate, and no periodic test oil sampling.

NOTE 1—Flow rates other than those listed in this test method may be required by various specifications; if they are so used, the modification to the test method should be stated in the test report.

3.2 A large glass tube containing a sample of oil and metal specimens is placed in a constant temperature bath and heated for the specified number of hours while air is passed through the oil to provide agitation and a source of oxygen. Typically, temperatures of the bath used are from 100 °C to 360 °C. Weighed metal specimens are placed in the tube during the test. Corrosiveness of the oil is determined by loss in metal mass, and microscopic examination of the sample metal surface(s). Oil samples are withdrawn from the test oil and checked for changes in viscosity and acid number as a result of the oxidation reactions.

3.3 Metals used in the basic test and alternative Procedure 1 are titanium, magnesium, steel (two types), bronze, silver, and aluminum. Metals used in alternative Procedure 2 are copper, steel, aluminum, magnesium, and cadmium. Other metals may be specified.

3.4 Sampling of the oil for analysis is done periodically throughout the test. Alternatively, no periodic samples are taken and a final viscosity and acid number are determined for comparison with those of the original untested oil.

3.5 At the end of the test, the amount of sludge present in the oil remaining in the same tube is determined by centrifugation. Also, the quantity of oil lost during the test is determined gravimetrically.

3.6 Air is used dry in the standard test. A humidifier may be used to provide controlled moist air, if required.

4. Significance and Use

4.1 This test method simulates the environment encountered by fully formulated lubricating fluids in actual service and uses an accelerated oxidation rate to permit measurable results to be obtained in a reasonable time. The use of metals provides catalytic reactive surfaces of those materials commonly found in real systems. The high temperature and air agitation help accelerate the oxidation reactions that are expected to occur. Moisture in the air adds another realistic condition that encourages oil breakdown by facilitating acid formation.

4.2 Interpretation of results should be done by comparison with data from oils of known field performance. The accelerated conditions likely will cause one or more of the following measurable effects: mass change and corroded appearance of some metals; change of viscosity; increase in acid number; measurable reaction products in the form of sludge; and mass loss of oil due to evaporation.

4.3 This test method is most suitable for oils containing oxidation and corrosion inhibitors. Without such ingredient(s), the severe test conditions will yield rather drastic changes to the oil.

5. Apparatus

5.1 The main apparatus consists of the following items of standard wall borosilicate glassware as shown in Figs. 1-5:

5.1.1 *Main Sample Tube* (Fig. 1).

5.1.2 *Sample Tube Head* (Fig. 2).

5.1.3 *Air Tube* (Note 2, Fig. 3).

NOTE 2—An 800 mm air tube may be used for alternate Procedure 1 or 2 when using the condenser as opposed to the sample tube head in the standard procedure.

5.1.4 *Condenser, Allihn Type* (Fig. 4).

5.1.5 *Condenser, Allihn Type*, Fig. 4 with 71/60 joint.

5.1.6 *Assembled Apparatus* (Fig. 5).

5.2 Additional glassware items and assembly accessories needed are:

5.2.1 *Spacers (for Metal Specimen)*, of borosilicate glass, standard wall, 9 mm outside diameter, 6 mm length.

5.2.2 *Oil Sampling Tube, Borosilicate Glass*, 4 mm outside diameter, with sampling end approximately 600 mm to reach into the main sample tube. The tube is bent U-shape with exit end fitted by a one-hole stopper to a 25 mL filtering flask. The exit end may be any convenient length.

5.2.3 *Adapter*, polytetrafluoroethylene for 10/18 joint for sealing of air tube to sample tube head.

5.3 Other items and equipment are:

5.3.1 *Heating Bath*, constant temperature within ± 0.5 °C of test temperature with an immersion depth of 250 mm \pm 20 mm. Oil baths or aluminum block baths are recommended, but above 220 °C, use aluminum block bath or other similar non-oil-type heating medium. (**Warning**—There are exposed hot surfaces on apparatus. Avoid contact with exposed skin by use of protective equipment as required.)

5.3.2 *Hood*, ventilation to adequately remove fumes during heating.

⁵ See ASTM DS 56, Metal and Alloys in the Unified Numbering System.

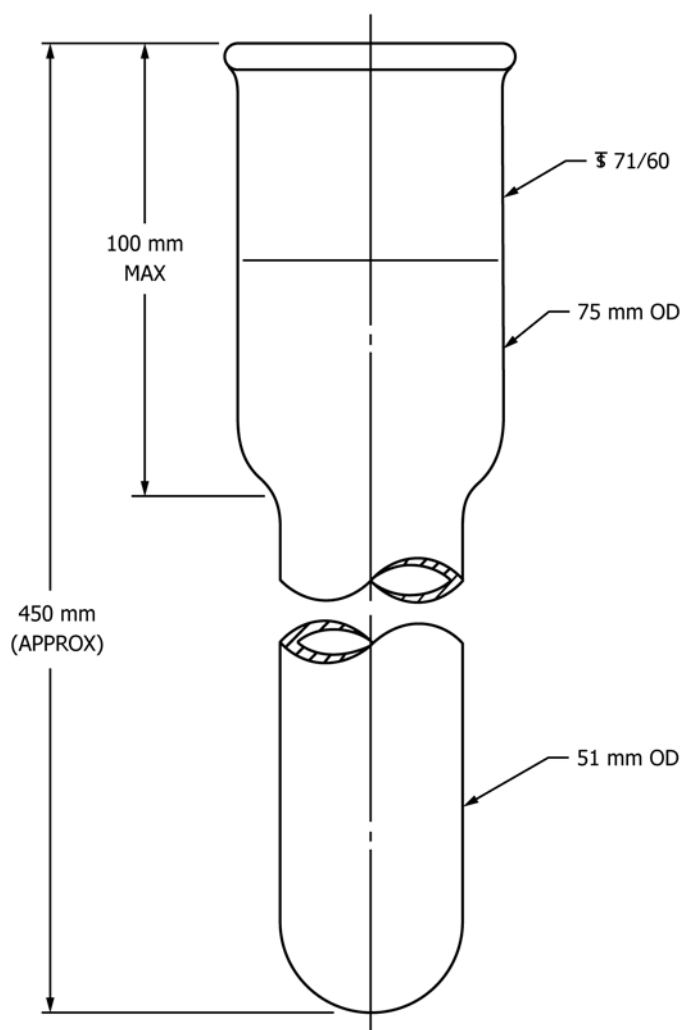


FIG. 1 Sample Tube

5.3.3 *Air Supply*, free of reactive contaminants. For dry air, dew point is $-68\text{ }^{\circ}\text{C}$ maximum; for moist air, moisture is $10\text{ mg} \pm 1\text{ mg water/L air}$, standard conditions of $21\text{ }^{\circ}\text{C}/105\text{ kPa}$.

5.3.4 *Flowmeter*, capable of measuring $10\text{ L/h} \pm 1\text{ L/h}$ at same standard conditions as in 5.3.3.

5.3.5 *Balance*, analytical, sensitivity 0.1 mg .

5.3.6 *Balance, Laboratory*, 2500 g capacity, 0.1 g sensitivity.

5.3.7 *Centrifuge*, capable of relative force of 840 ± 40 relative centrifugal force at the tip of the tubes.

5.3.8 *Centrifuge Tubes*, Test Method D91, cone-shaped, 100 mL.

5.3.9 *Microscope*, with 20-diameter magnification.

5.3.10 *Assembly Fixture*, wood (slotted to hold metal squares for tying with cord) as shown in Fig. 6.

5.3.11 *Cord or Wire*, for tying metal squares together. Suitable cord should be lightweight of cleaned linen, cotton, nylon, or ceramic fiber; suitable wire is nichrome or tantalum.

5.3.12 When air must be conditioned, there is need for an air drier or humidifier. The method used is optional provided the air characteristics of 5.3.3 are attained. For drying, a satisfactory method is the use of a glass column containing 8-mesh

anhydrous calcium sulfate with a column diameter such that velocity of air does not exceed 1.2 m/min . For humidifying, a satisfactory device is included in Appendix X1 to provide the required moist air.

5.3.13 *Oven*, optional, to dry glassware at elevated temperature.

5.3.14 *Forceps*, stainless steel.

5.3.15 *Thermocouple*, 70 cm sheathed. (See Note 3.)

NOTE 3—A resistance temperature device (RTD) is also acceptable.

5.3.16 *Brush*, short-bristled, stiff (typewriter-cleaning brush or equivalent).

6. Reagents and Materials

6.1 *Purity of Reagents*—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.⁶ 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Specification D1193. Referee situations require Type II distilled water defined by Specification D1193.

6.3 *Metal Specimens*:^{7, 8}

6.3.1 *Washer-Shaped Metal Specimens*, 6.35 mm inside diameter by 19.05 mm ($\frac{3}{4}$ in.) outside diameter by 0.81 mm thick in the following metals:

R56080	Titanium 8 % Mn
M11311	Magnesium AZ31B
T11350	Steel M50
G10100	Steel grade 1010
C65900	Silicon-iron-bronze
P07015	Silver 99.95
A92024	Aluminum Alloy 2024 T-3 or T-4

6.3.2 *Square-Shaped Metal Specimens* (as shown in Fig. 6), 0.81 mm by 25.4 mm square in the following metals:

C11000	Copper (ETP)
G10100	Steel Grade 1010
A92024	Aluminum Alloy 2024 T-3 or T-4
M11311	Magnesium AZ31B
L01900	Cadmium Anod

6.4 *Abrasive Paper*, silicon carbide of 240 and 400-grit. “Wet or dry,” “waterproof,” or iron-containing abrasives, such as natural emery, are not satisfactory.

6.4.1 *Silicon-Carbide Grains*, 150 mesh.

6.5 *Cotton*, absorbent.

⁶ *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.

⁷ The sole source of supply of the metal specimens known to the committee at this time is Metaspec Co., P.O. Box 27702, San Antonio, TX 78227-0707.

⁸ If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.

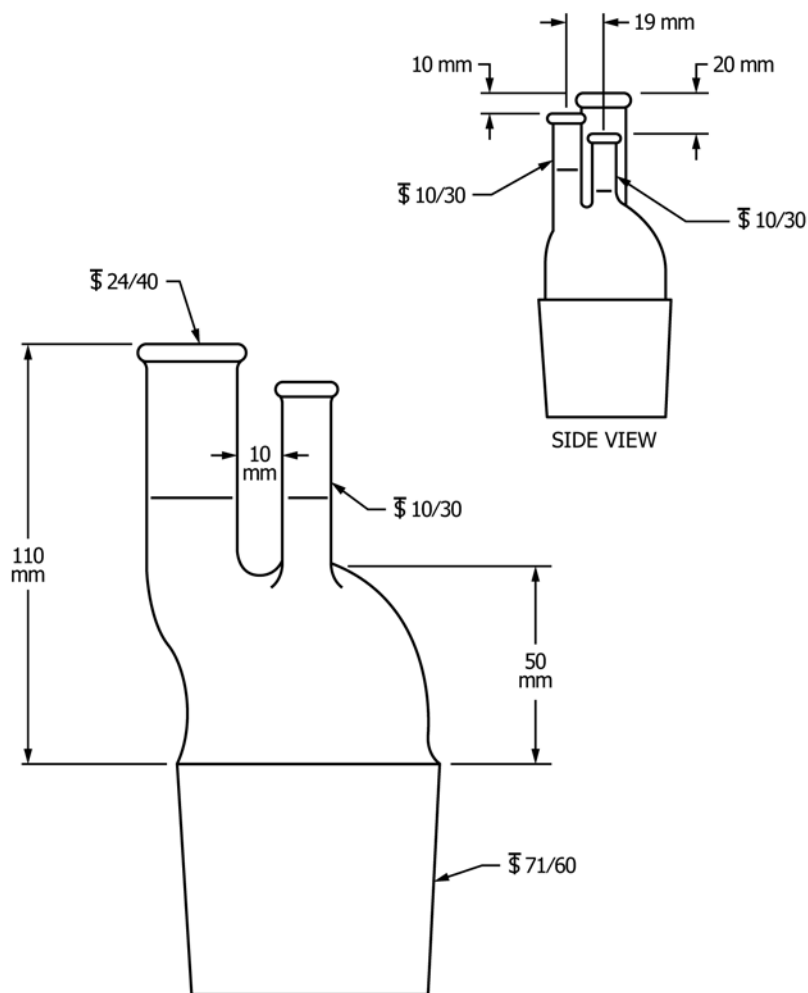


FIG. 2 Sample Tube Head

6.6 *n*-Heptane. (**Warning**—Flammable. Harmful if inhaled.)

6.7 Acetone. (**Warning**—Extremely flammable. Vapors may cause flash-fire.)

6.8 Nitric Acid, concentrated. (**Warning**—Poison. Corrosive. Strong oxidizer.)

6.9 Degreasing Solvents, *n*-Heptane or toluene. See **Note 4**.

NOTE 4—*n*-Heptane or toluene have been used in some laboratories; however, it is not known how this may affect the precision statement, which was determined using chlorinated solvents.

6.10 Carbon Remover for Glassware^{9, 8}. (**Warning**—Causes severe burns.)—Mix 35 mL of saturated sodium dichromate (aqueous) solution and 1000 mL of concentrated sulfuric acid. (**Warning**—Corrosive.)

6.11 Glassware Cleaning Solution (**Warning**—Causes severe burns.)—Mix 35 mL of MICRO¹⁰ in water or 35 mL of NOCHROMIX¹¹ and 1000 mL of concentrated sulfuric acid.

6.12 Metal Cleaning Solution—Mix equal parts of 15 g NaOH per litre of water and 15 g of Na₃PO₄ per litre of water. (**Warning**—Corrosive.)

7. Hazards

7.1 As this test method investigates the corrosive effect of oil on metal in an artificial environment, precautions must be taken to prevent premature oxidation of metal specimens by stray chemicals during test preparations. The cleaned specimens shall be touched only by surfaces known to be free of interfering contaminants. Do not touch cleaned specimen(s) with hands. Maintain clean glassware (see **Appendix X2**) in a

⁹ The sole source of supply of the carbon remover for glassware known to the committee at this time is Oakite Products, Inc., 50 Valley Rd., Berkeley Heights, NJ 07922.

¹⁰ International Products Corp., P.O. Box 70, Burlington, NJ 08016-0070.

¹¹ Godax Laboratories, Inc., 720-B Erie Avenue, Takoma Park, MD, 20912.

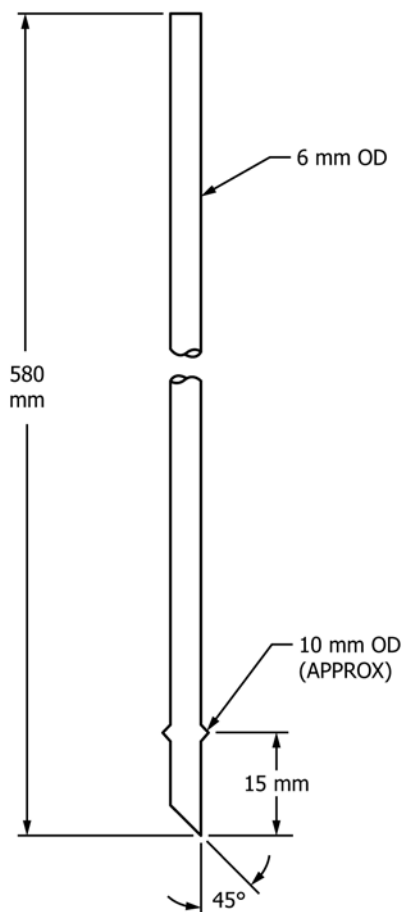


FIG. 3 Air Tube

dust-free cabinet; rinse again and dry if clean glass has been stored unused for more than a week.

7.2 *Heating Bath*, *n*-heptane, toluene, acetone, carbon remover, nitric and sulfuric acids, and sodium hydroxide all require caution in handling as detailed in 5.3.1 and 6.6 – 6.12.

8. Sampling

8.1 As this test method is performed on uniform, finished oils, no special sampling procedure is needed for this oil.

8.2 Preparation of Metal Specimens:

8.2.1 Collect the required number and types of specimens to be used in the test method. Measure the diameter of each specimen and specimen hole to the nearest 0.1 cm.

8.2.2 Clean and polish the metal specimen face surfaces and inner and outer edges using 240-grit abrasive paper. Examine surfaces to ensure that no pitting, etching, or other signs of corrosion are present when viewed at 20× under the microscope.

8.2.3 From this point, handle specimens only with forceps, ashless filter paper, or clean polyethylene gloves. Store specimen immersed in separate containers of acetone until ready for final polishing.

8.2.4 Finish polishing metal washer specimens or square metal specimens. Use either technique described in 8.2.4.1 or 8.2.4.3.

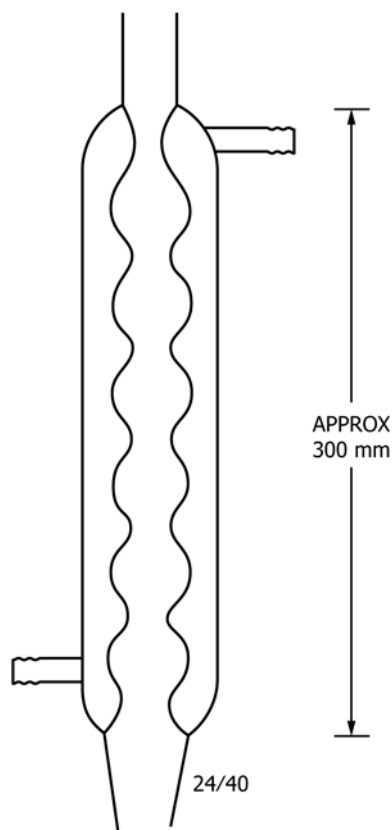


FIG. 4 Condenser, Allihn Type

NOTE 5—Square metal specimens are required by alternative Procedure 2 in 10.3.

8.2.4.1 Finish polishing with 400-grit paper to remove marks from previous polishing. A good technique is to place abrasive paper on flat surface, then rub the specimen with longitudinal strokes in a direction perpendicular to that used with 240-grit paper. Use a different sheet of paper for each metal type.

8.2.4.2 Swab polished specimens with *n*-heptane followed by acetone until a fresh cotton pad remains clean when wiped on the specimen.

8.2.4.3 Finish polishing both sides of each specimen to a mirror finish using 150-mesh silicon-carbide grit (held on an acetone-moistened cotton pad).

8.2.4.4 Clean all metal dust from each square by rubbing vigorously with clean pads of absorbent cotton until a fresh pad remains unsoiled.

8.2.4.5 Wash specimens in *n*-heptane, rinse with fresh solvent, and allow to air dry.

8.2.5 Store specimens in a dust-free place if to be used immediately or under *n*-heptane if there is to be delay before use.

8.2.6 Immediately prior to use, dry and weigh each metal specimen to the nearest 0.1 mg.

8.3 Oil Sample Size (Note 6 and Note 7):

8.3.1 The total sample requirement is 250 mL, 200 mL ± 2 mL for test and 50 mL for pretest determination of viscosity and acid number.

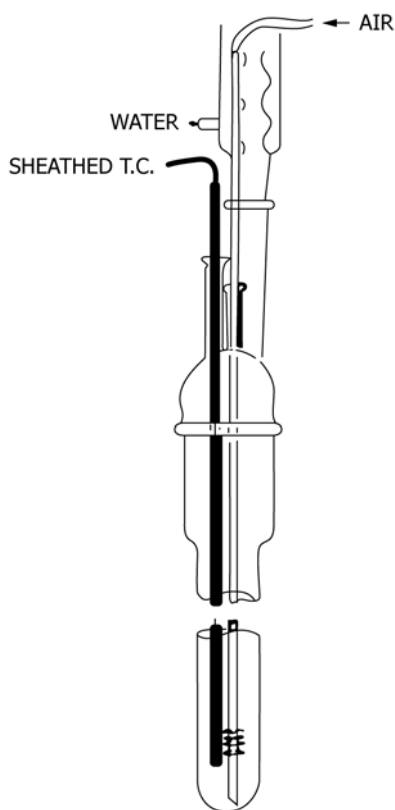


FIG. 5 Assembled Apparatus

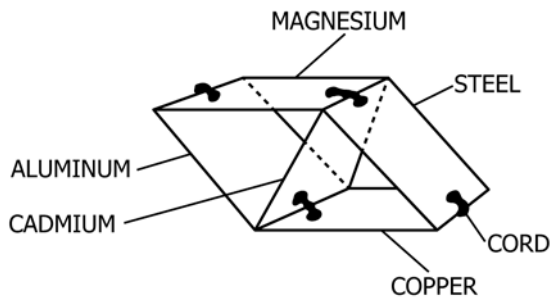
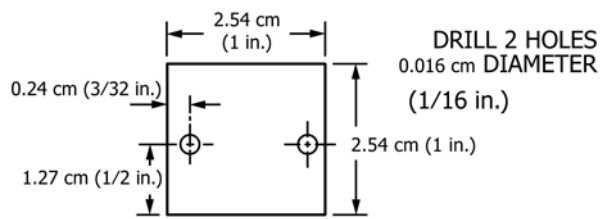


FIG. 6 Metal Square Dimensions and Arrangement

NOTE 6—An alternative Procedure 1, which deletes the requirement for periodic oil sampling throughout the test period may be specified. Alternative Procedure 1 requires an initial oil charge of 165 mL ± 2 mL (see 10.2).

NOTE 7—An alternative Procedure 2, which deletes the requirement for periodic oil sampling and requires square metal specimens instead of washer-shaped metal specimens may be specified. Alternative Procedure 2 requires an initial oil charge of 100 mL ± 1 mL (see 10.3).

9. Preparation of Apparatus

9.1 Cleaning of Glassware (from Previous Run):

9.1.1 Rinse all glassware items and the air tube adapter with degreasing solvent to remove residual oil, and air dry.

9.1.2 Fill or immerse the sample tube, air tube, and the 9 mm glass spacers in carbon remover at room temperature until carbonaceous deposits are removed. Water rinse after removal.

9.1.3 Wash all glassware items and the air tube adapter with detergent.^{12, 8} Rinse with water to remove detergent, and dry.

9.1.4 Fill and immerse all glassware items with glassware cleaning solution and soak for 3 h to 16 h (see Note 8).

NOTE 8—This cleaning procedure is only necessary in a referee situation unless a cleaning solution, which is satisfactory to all parties involved, can be used.

9.1.5 Remove glassware from cleaning solution, rinse several times with tap water followed by distilled water, and oven dry (see Note 8).

9.1.6 Store all items in a dust-free cabinet until needed for test. If stored longer than 1 week, rinse again before use with distilled water and dry.

9.2 Cleaning of Glassware (New)—Proceed as in 9.1.3 and 9.1.6 in that order (omit 9.1.1, 9.1.2, 9.1.4, and 9.1.5).

9.3 Assembly—Assemble as shown in Fig. 5, using only the test oil to lubricate glass joints during assembly.

10. Procedure

10.1 Standard Test Procedure:

10.1.1 Determine acid number of the test oil in accordance with Test Method D664.

10.1.2 Determine the kinematic viscosity of the test oil at 40 °C and 100 °C in accordance with Test Method D445.

10.1.3 Turn on heating control to bring temperature of heating medium to a temperature that will maintain the oil sample within ±0.5 °C of the specified temperature (Warning—There are exposed hot surfaces on apparatus. Avoid contact with exposed skin by use of protective equipment as required.).

10.1.4 When assembling the clean sample tubes and accessories, use only the actual test oil to lubricate joints of each assembly.

10.1.5 Slide the prepared metal washer specimens onto the air tube in the following order: aluminum (bottom), silver, bronze, low carbon steel, M50 steel, magnesium, and titanium (top). Use one glass spacer only between each metal specimen.

10.1.6 Place the air tube with specimens into the sample tube.

10.1.7 Position the sample tube head on the sample tube with the air tube protruding through the center glass joint.

10.1.8 Seat the TFE-fluorocarbon adapter on the air tube with the air tube resting on the bottom of the sample tube and tighten the gland.

¹² The sole source of supply of the detergent known to the committee at this time is Alconox, Inc., 215 Park Avenue South, New York City, NY 10003.

10.1.9 Insert the 70 cm long-sheathed thermocouple making sure the closed end of the sheath is at least 10 mm from the sample tube wall.

10.1.10 Weigh the entire apparatus to the nearest 0.1 g.

10.1.11 Add 200 mL \pm 2 mL of test oil in accordance with 8.3 to the sample tube, reweigh entire assembly to the nearest 0.1 g, and determine the mass of the sample added.

10.1.12 Position the sample tube in the heat medium at test temperature to an immersion depth of 250 mm \pm 20 mm.

10.1.13 Insert the condenser and start water flow. Maintain water temperature at 18 °C \pm 3 °C.

10.1.14 Connect the thermocouple.

10.1.15 After the sample tube has been in a liquid heating medium for 15 min, connect the dry air supply, adjust the air flow rate to 10 L/h \pm 1 L/h, and begin the test time. For an aluminum block heating medium without liquid heat transfer medium, allow 1 h between insertion of sample tube into heater and start of air flow and timing.

10.1.16 Adjust heat control to ensure oil sample temperature is held within \pm 0.5 °C of required value. Verify sample temperature and air flow occasionally during test specifically just prior to each sampling time.

10.1.17 Sample the test oil (10 mL sample) at the following times: 16 h, 24 h, 40 h, 48 h, 64 h, 72 h, 88 h, and 96 h.

10.1.18 Preweigh the sample flask, stopper, and tube to the nearest 0.1 g so that an accurate mass of sample withdrawn can be determined.

10.1.19 Perform sampling by withdrawing the thermocouple sheath and inserting the 4 mm tube attached to the sampling flask. Using a rubber bulb or similar source of slight suction on the flask tube, draw the required sample to a premarked level in the sampling flask. Do not interrupt air flow nor remove the same tube from the heat during sampling.

10.1.20 Determine and record mass of each sample removed during test.

10.1.21 Determine acid number in accordance with Test Method D664 of all samples.

NOTE 9—If acid number is not determined on the same day of sampling, samples should be refrigerated up to a maximum of 1 week prior to determination.

10.1.22 Determine viscosity in accordance with Test Method D445 at 40 °C on all samples and viscosity at 100 °C on original and final sample only.

NOTE 10—Due to the reduced sample volume availability, viscosity measurement is made using the semimicro viscometers listed in Test Method D445. In addition, it may be necessary to determine acid number using a titration sample size less than that required by Test Method D664. The semimicro Test Method D3339 may be used to determine acid number.

10.1.23 *Shutdown:*

10.1.23.1 After the final sample is taken, shut off the air flow and condenser water, remove the condenser, and disconnect the thermocouple. Note the presence of liquid or solid material on the inside of the condenser.

10.1.23.2 Remove the sample tube from the heat medium, wipe tube exterior, allow to cool, and, if necessary, clean the tube exterior by rinsing with *n*-heptane or toluene. Weigh the assembly to the nearest 0.1 g.

10.1.23.3 Remove the air tube, polytetrafluoroethylene adapter, and sample tube head.

10.1.23.4 Remove the air tube with metal specimens, rinse with *n*-heptane or toluene, and slide the specimens off the air tube carefully into a clean absorptive surface. Keep the specimens in order for identification, but if processing of specimens is to be delayed, store them under *n*-heptane.

10.1.23.5 Drain the test oil completely from the sample tube into a clean glass container and hold for sludge determination. Note the appearance of any deposits on the inside of the sample tube.

10.1.24 *Sludge Determination*—Take a representative portion, after well stirring the final test oil, and centrifuge a 25 mL aliquot for 1 h at a relative centrifugal force of 840 \pm 40 in accordance with Test Method D91. Record the volume of solid or semisolid sludge obtained to the nearest 0.1 mL.

10.1.25 *Metal Specimens, Post-Test Processing:*

10.1.25.1 Rinse the metal specimens individually with *n*-heptane or toluene. Wipe each metal specimen with *n*-heptane or toluene-wetted cotton pads until a fresh pad remains clean.

10.1.25.2 Rinse each specimen with clean *n*-heptane, then acetone, then air dry and weigh to the nearest 0.1 mg.

10.1.25.3 If there are visible carbon deposits remaining on the specimens, clean as indicated in Appendix X2.

10.2 *Alternative Procedure 1:*

10.2.1 Perform the standard test as in 10.1 except as follows:

10.2.2 Use 165 mL \pm 2 mL of the sample at the start.

10.2.3 Do no periodic sampling. Sample the oils only at completion of the test time. No sample tube head is needed. Use the condenser described in 5.1.5.

10.3 *Alternative Procedure 2:*

10.3.1 Perform the standard test as in 10.1 except as follows:

10.3.2 Use 100 mL \pm 1 mL of the sample at the start.

10.3.3 Use 5 L/h \pm 0.5 L/h air flow rate.

10.3.4 Do no periodic sampling. Sample the oil only at completion of the test time. No sample tube head is needed. Use the condenser described in 5.1.4 or 5.1.5.

10.3.5 Instead of washer-shaped metal specimens, use square metal specimens as defined in 6.3.2.

10.3.5.1 Arrange metal squares in a wooden assembly fixture in the pattern shown in Fig. 6, making certain that the magnesium and copper squares do not touch each other.

10.3.5.2 Clean the cord or wire by boiling in distilled water for 10 min and allow to air dry.

10.3.5.3 Using forceps or cotton (linen) gloves, or both, to handle the cord or wire, tie the squares together as shown in Fig. 6. Due to potential contamination, use of latex gloves is not recommended.

10.3.5.4 Insert the tied metal squares into the sample tube, positioning the squares vertically (so that the air tube can be inserted to touch the bottom of the tube).

10.3.5.5 When the oil specification requires only two metal specimen squares, place the squares in the sample tube in such a manner as to form a “V” in the bottom of the tube. No tying is necessary.

10.3.6 Upon completion of 10.1.23.3, prepare square metal specimens for examination as follows:

10.3.6.1 Use forceps to withdraw the metal squares from the test tube and remove the cords holding them together.

10.3.6.2 Using forceps, wash each square individually in *n*-heptane or toluene.

10.3.6.3 Repeat the washing, using fresh *n*-heptane or toluene and scrubbing the squares with the short-bristled brush until the solvent shows no additional discoloration, and allow the squares to dry. Alternatively, scrubbing with *n*-heptane-moistened 100 % cotton balls until they look clean has also been found acceptable.

10.3.6.4 Weigh each square to the nearest 0.1 mg. Measure the face dimensions of each panel to the nearest 0.1 cm.

11. Calculation

11.1 Calculate the oil mass loss as follows:

$$\% \text{ Loss} = \frac{W_2 - (W_3 + W_4)}{W_2 - W_1} \times 100 \quad (1)$$

where:

W_1 = mass of tube assembly,

W_2 = mass of tube assembly plus sample at start,

W_3 = mass of tube assembly plus sample at end of test, and

W_4 = accumulated mass of samples removed during test.

11.2 Calculate the viscosity change as follows:

$$\% \text{ Viscosity change} = \left(\frac{V_2 - V_1}{V_1} \right) \times 100 \quad (2)$$

where:

V_1 = viscosity before test, cSt (mm²/s), and

V_2 = viscosity after test, cSt (mm²/s).

11.3 Calculate the neutralization number change as follows:

$$\text{Acid number change, mg KOH/g} = T_2 - T_1 \quad (3)$$

where:

T_1 = acid number before test, mg KOH/g, and

T_2 = acid number after test, mg KOH/g.

11.4 Calculate the metal specimen mass change as follows:

$$\text{Metal mass change, mg/cm}^2 = (M_2 - M_1)/A \quad (4)$$

where:

M_1 = mass of specimen before test, mg,

M_2 = mass of specimen after test, mg, and

A = area of metal specimen based on the face surface without consideration for the edge area, cm².

11.4.1 For washer specimens:

$$A = \frac{\pi}{2} (D_2^2 - D_1^2) \quad (5)$$

where:

D_1 = initial diameter of hole, nearest 0.1 cm, and

D_2 = initial diameter of specimen, nearest 0.1 cm.

11.4.2 For square specimens:

$$A = 2 LW \quad (6)$$

where L and W are the two face dimensions, nearest 0.1 cm.

12. Report

12.1 Report kinematic viscosity, expressed in centistokes (millimetres squared per second) for initial and final samples at 40 °C and 100 °C. Report percentage change to the nearest 0.1 % from original viscosity for all periodic and final samples at 40 °C and for final sample at 100 °C.

12.2 Report the acid number for original and final samples, expressed in mg KOH/g. Report the change in neutralization number in milligrams KOH/g for all periodic and final samples.

12.3 Report the mass change of each metal specimen from the initial. When Appendix X2 cleaning is used, report mass change before and after electrocleaning or nitric acid soak. Express the change in milligrams per squared centimetres calculated to the nearest 0.1 mg/cm².

12.4 Report the appearance of the metal specimens after cleaning with respect to any pitting, etching, or other corrosion observed without magnification and with 20× magnification.

12.5 Report the volume % of sludge in oil to the nearest 0.1 mL.

12.6 Report the weight percent of oil loss during test to the nearest 0.1 %.

12.7 Report the test conditions and any irregularities or deviations from required test procedures and conditions.

12.8 Report the presence of liquid or solid material on the inside of the condenser at the end of the test.

12.9 Report the appearance of the deposit on the sample tube at the end of the test.

13. Precision and Bias

13.1 Precision was determined under test conditions of 175 °C for a 96 h duration using the standard test procedure and alternate Procedure 1. Precision for alternate Procedure 2 was not determined. The precision of this test method as determined by statistical examination of interlaboratory test results in the ranges indicated is stated in Tables 1-3.

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

13.1.2 *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, and in the normal and correct operation of the test method, exceed the following value only in one case in twenty.

TABLE 1 Precision Table for Percent Change in Viscosity at 40 °C and at 100 °C in the Range 0 % to 12 %

	Repeatability, % Change	Reproducibility, % Change
Standard Test Procedure	1.6	3.2
Alternate Procedure 1	2.0	2.7

TABLE 2 Precision Table for Acid Number Change

Range of Change in Acid Number mg KOH/g	Repeatability		Reproducibility	
	Standard Test Procedure	Alternate Procedure 1	Standard Test Procedure	Alternate Procedure 1
0.0 to 1.0	0.32	0.23	0.69	0.62
1.0 to 2.0	0.49	0.36	1.53	1.43

TABLE 3 Precision Table for Corrosion Up to 0.3 mg/cm

	Repeatability	Reproducibility
Standard and Alternate Procedure 1	0.2	0.3

13.2 *Bias*—Since there is no acceptable reference material suitable for determining the bias procedure for measuring corrosiveness and oxidation stability of hydraulic oils, aircraft turbine engine lubricants, and other highly refined oils in Test Method D4636, bias cannot be determined.

14. Keywords

14.1 hydraulic fluid; lubricant; oxidation stability

APPENDIXES

(Nonmandatory Information)

X1. TEST GAS HUMIDITY CONTROL

X1.1 *Air Humidifier*—The precise method of humidifying the test air is optional. A satisfactory device is shown in Fig.

X1.1. Air enters through a length of 0.95-cm tubing and discharges through a 2.54-cm diameter diffuser stone. The

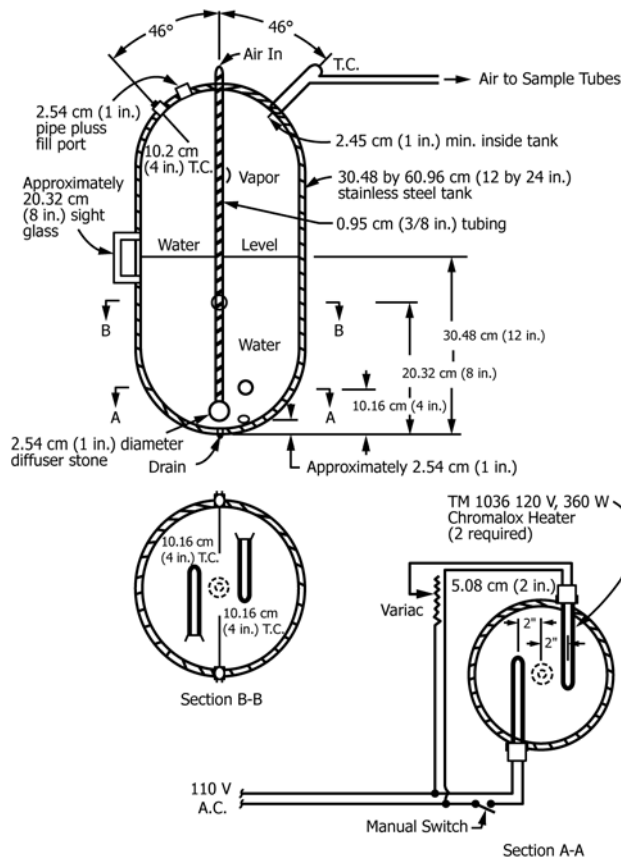


FIG. X1.1 Air Moisturizer

controlled temperature is that indicated by the thermocouple near the air exit fitting. One heater is operated by an on-off switch and used only for initial preheating. A second heater is in circuit with a variable voltage transformer. The transformer is adjusted to control the air exit temperature required to give the proper moisture content. The stainless steel tank^{13, 8} is insulated over the entire exterior surface and placed within a refrigerator. The air exit fitting and line are well insulated to avoid moisture condensation. The exit line length within the refrigerator is held to a minimum and the downstream portion of the line between the refrigerator and sample tube must not encounter a temperature region lower than the control temperature, or condensation will occur.

X1.2 The preceding apparatus will satisfactorily serve as an air humidifier for several sample tubes, up to a known total of 16. At this flow rate (160 L/h), the following control parameters are typical for achieving a moisture of 10 mg of water/L of air:

Exit air temperature	15 °C
Water temperature	15.6 °C
Refrigerator temperature	12.2 °C
Control heater power	>10 W

¹³ The sole source of supply of the stainless steel tank known to the committee at this time is A. C. Tank Co., POB 389, Burlington, WI 53015.

X2. SPECIMEN CLEANING

X2.1 If there are visible deposits on the specimens, they must be electrocleaned. The individual specimens, except aluminum, are cathodically cleaned in hot 75 °C to 85 °C electrocleaning solution (see 6.12) for a period of 15 s to 30 s to a current density of 0.08 amp/cm². Remove from the bath,^{8, 14} rinse in tap water, and swab with cotton to remove

¹⁴ A satisfactory bath for electrocleaning consists of a 1 L glass beaker, hot plate, and variable dc voltage source capable of supplying current of 1 A. The sole source of supply of the voltage source known to the committee at this time is Electro Products Laboratories, Inc., 6125 W. Howard Street, Chicago, IL 60648.

X1.2.1 The air temperature is monitored daily; however, with satisfactory insulation, the transformer setting should not require adjustment during a 96 h test.

X1.3 The exit air temperature is selected to obtain the desired moisture content. Moisture content may be determined gravimetrically using a U-shaped Schwartz tube with standard-taper glass stoppers. The tube is filled with anhydrous calcium sulfate and weighed to the nearest 0.1 mg. The air moisturizer is allowed to temperature equilibrate at the required total airflow. While maintaining the total flow through the moisturizer, the weighing tube is connected to any one of the sample tube air lines. After a 1 h flow period, the tube mass gain should be 100 mg ± 10 mg. If necessary, adjust air exit temperature control to obtain proper moisture content. Any change in the total air flow necessitates recalibration of moisture content since the air velocity through the moisturizer could affect the relative humidity of the air. In the event that a test is performed with a lesser number of sample tubes than that for which originally calibrated, unused flowmeters should be left on to maintain the normal total airflow.

loose deposits. (Repeat the electrocleaning step, as necessary, to remove all deposits.) Rinse the specimens in acetone, air dry, and reweigh. Soak the aluminum specimen in concentrated nitric acid for 15 min, then water rinse, and swab with cotton to remove loose deposits. (Repeat the cleaning as necessary to remove all deposits.) Rinse the specimens in acetone, air dry, and reweigh.

NOTE X2.1—If metal types other than those cited in this test method are used, the compatibility of the electrocleaning procedure with metal composition should be determined and, if necessary, other appropriate procedures used.

SUMMARY OF CHANGES

Subcommittee D02.09.0D has identified the location of selected changes to this standard since the last issue (D4636 – 14) that may impact the use of this standard. (Approved June 1, 2017.)

(1) Revised Eq 4 in subsection 11.4.

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