



Designation: ~~D4636~~—14 ~~D4636~~ – 17

# Standard Test Method for Corrosiveness and Oxidation Stability of Hydraulic Oils, Aircraft Turbine Engine Lubricants, and Other Highly Refined Oils<sup>1</sup>

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

*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.<sup>2</sup>

### 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:*<sup>3</sup>

[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](#)

<sup>1</sup> 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.

Current edition approved July 1, 2014; June 1, 2017. Published July 1, 2014; June 2017. Originally approved in 1986. Last previous edition approved in 2009/2014 as ~~D4636-09~~ D4636 – 14, DOI: ~~10.1520/D4636-14~~ 10.1520/D4636-17.

<sup>2</sup> 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.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

## 2.2 U.S. Federal Test Method Standards:<sup>4</sup>

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)

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

## 2.3 Other Standards:<sup>5</sup>

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~~ 200 mL of test oil, ~~10 L~~ 10 L air flow rate, and periodic test oil withdrawal and evaluation. Alternative Procedure 1 uses washer-shaped metal specimens, ~~165 mL~~ 165 mL of test oil, ~~10 L~~ 10 L 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~~ 100 mL of test oil, ~~5 L~~ 5 L 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~~ 100 °C to ~~360°C~~ 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.

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

<sup>5</sup> See ASTM DS 56, Metal and Alloys in the Unified Numbering System.

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).

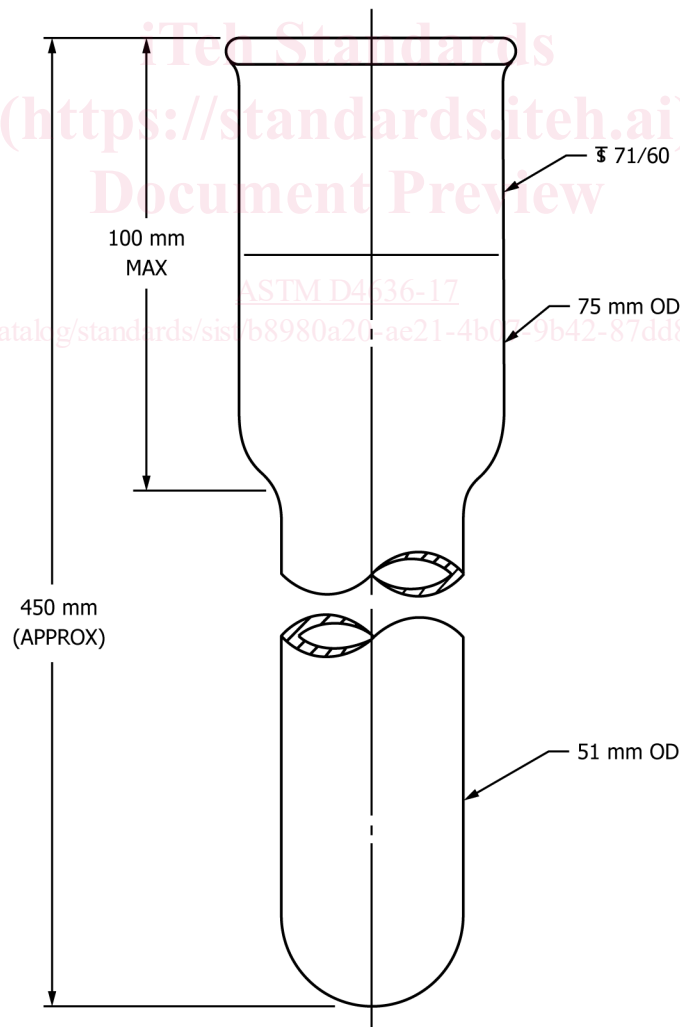


FIG. 1 Sample Tube

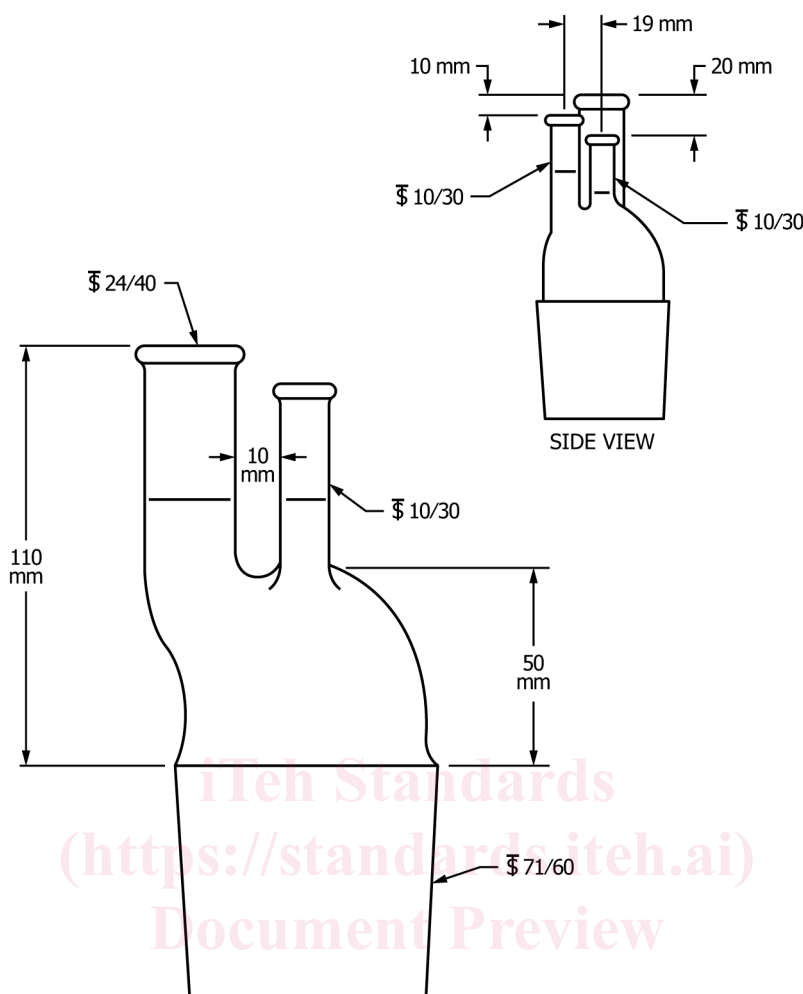


FIG. 2 Sample Tube Head

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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/9 mm outside diameter, 6-mm/6 mm length.

5.2.2 Oil Sampling Tube, Borosilicate Glass, 4-mm/4 mm outside diameter, with sampling end approximately 600-mm/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/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  $\pm 0.5^\circ\text{C} \pm 0.5^\circ\text{C}$  of test temperature with an immersion depth of 250/250 mm  $\pm 20$ -mm/20 mm. Oil baths or aluminum block baths are recommended, but above 220°C/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.

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

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

5.3.5 Balance, analytical, sensitivity 0.1 mg/0.1 mg.

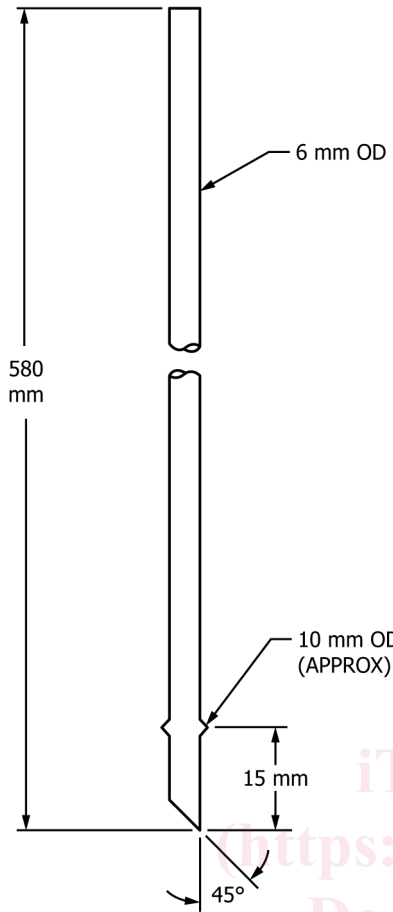


FIG. 3 Air Tube

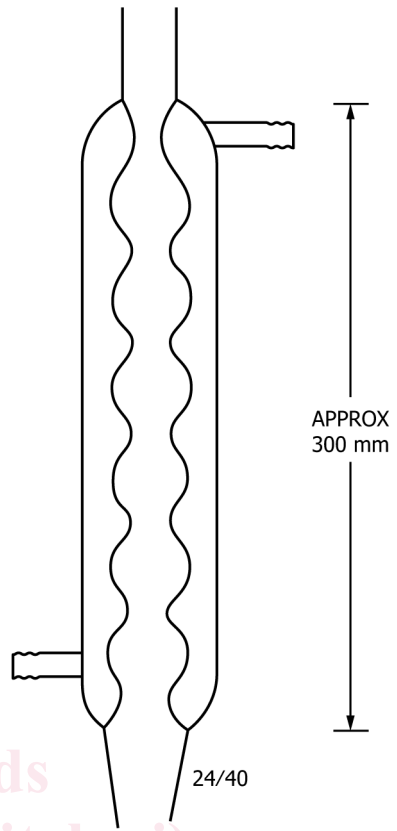


FIG. 4 Condenser, Allihn Type

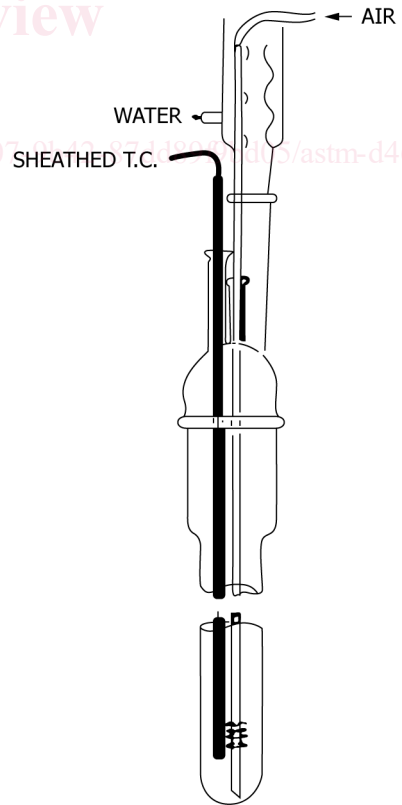


FIG. 5 Assembled Apparatus

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

5.3.7 *Centrifuge*, capable of relative force of  $840 \pm 40$  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~~; 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.<sup>6</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.

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*:<sup>7, 8</sup>

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:

<sup>6</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>7</sup> 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.

<sup>8</sup> 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 committee<sup>1</sup>, which you may attend.

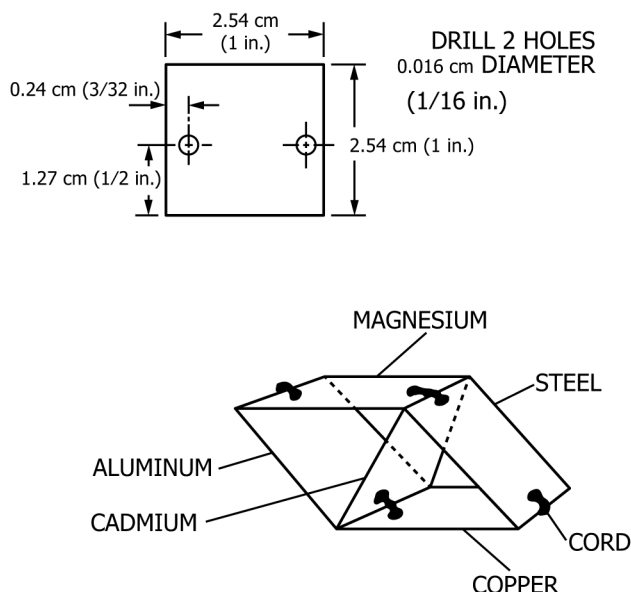


FIG. 6 Metal Square Dimensions and Arrangement