



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 D 4636; 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 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 these test methods are included, but the new test method is basically Method 5307.2 expanded to include Method 5308.7.

1. Scope

1.1 This test method is used to test 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 acceptable SI units are to be regarded as standard. The values 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.*

2. Referenced Documents

2.1 ASTM Standards:

D 91 Test Method for Precipitation Number of Lubricating Oils²

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)²

D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration²

D 1193 Specification for Reagent Water³

D 3339 Test Method for Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration⁴

2.2 U.S. Federal Test Method Standards:

Method 5307.2, Corrosiveness and Oxidation Stability of Aircraft Turbine Engine Lubricants⁵

Method 5308.7, Corrosiveness and Oxidation Stability of Light Oils (Metal Squares)⁵

MIL-S-13282 Refined Silver (99.95) (P07015)⁶

2.3 Other Standards:

AMS 4616 Silicon Iron Bronze (C65900)⁶

AMS 4908 Titanium Alloy—(8 % Mn) Annealed (R56080)⁶

AMS 6490 Steel (M50) (T11350)⁶

QQ-A-671 Cadmium Anod (L01900)⁶

QQ-C-576 Copper Electrolytic Tough Pitch (ETP) (C11000)⁶

QQ-M-44 Magnesium Alloy AZ31B Condition H24 or H26 (M11311)⁶

¹ 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.

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² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 05.02.

⁵ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

⁶ See ASTM DS 56, Metal and Alloys in the Unified Numbering System.

QQ-S-698 Low-Carbon Steel 1010, CR Temper No. 4 or 5 (G10100)⁶
 QQ-A-250/4 Aluminum Alloy 2024 T-3 or T-4 (A92024)⁶

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 (212°F) to 360°C (680°F). 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 test Procedure 1 are titanium, magnesium, steel (two types), bronze, silver, and aluminum. Metals used in alternative test 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 acceler-

ated 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*⁷ (Fig. 3).
- 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).

⁷ 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.

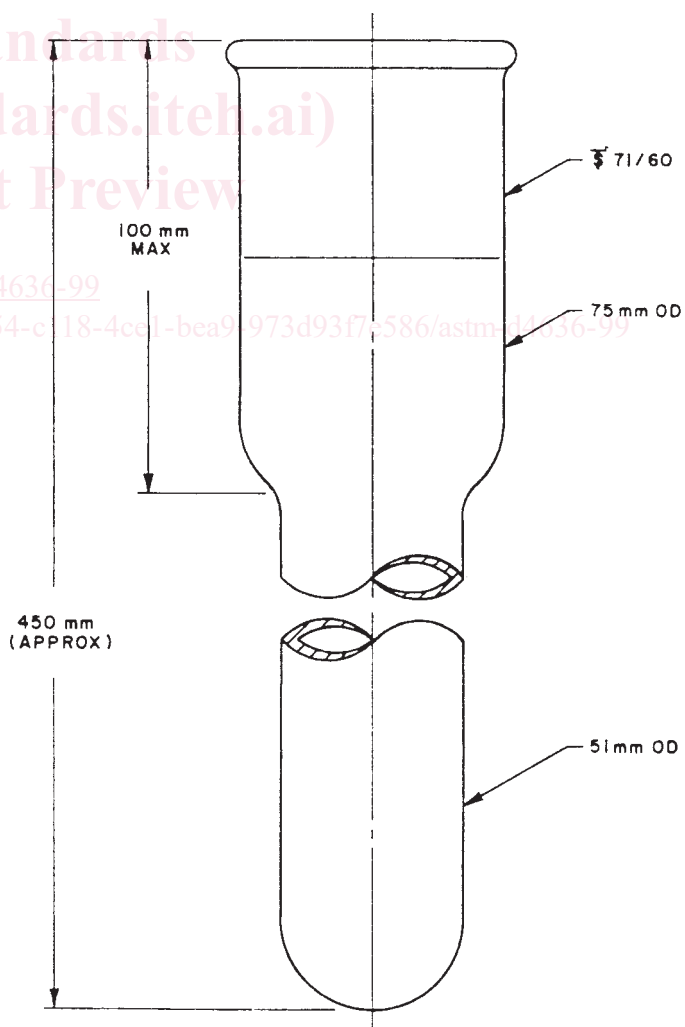


FIG. 1 Sample Tube

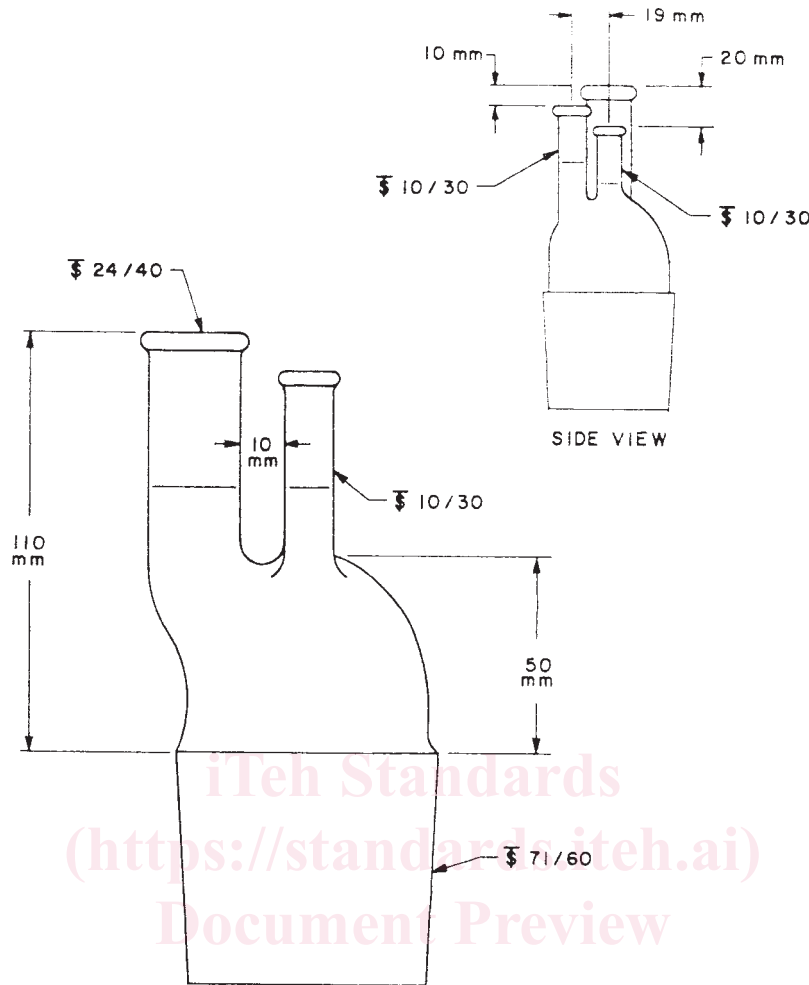


FIG. 2 Sample Tube Head

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 $\pm 0.5^\circ\text{C}$ ($\pm 1^\circ\text{F}$) of test temperature with an immersion depth of 250 \pm 20 mm. Oil baths or aluminum block baths are recommended, but above 220°C (428°F) use aluminum block bath or other similar non-oil-type heating medium.

NOTE 2—**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°C (-90°F) maximum; for moist air, moisture

is 10 ± 1 mg water/L air, standard conditions of 21°C (70°F)/105 kPa (1 atm).

5.3.4 *Flowmeter*, capable of measuring 10 ± 1 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 D 91, 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

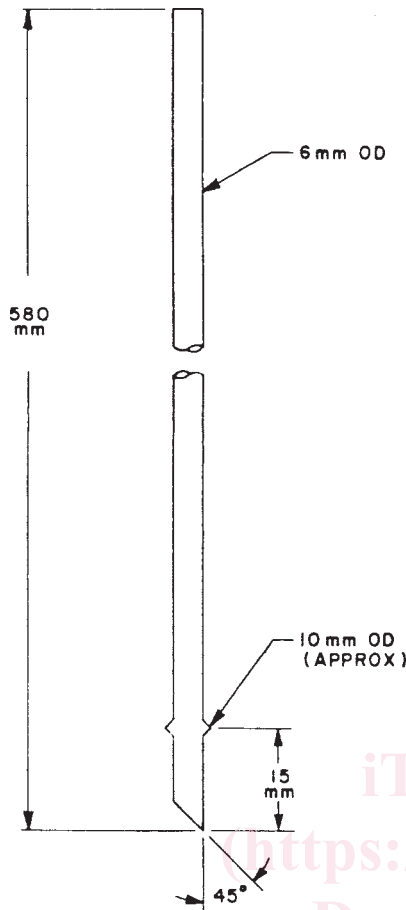


FIG. 3 Air Tube

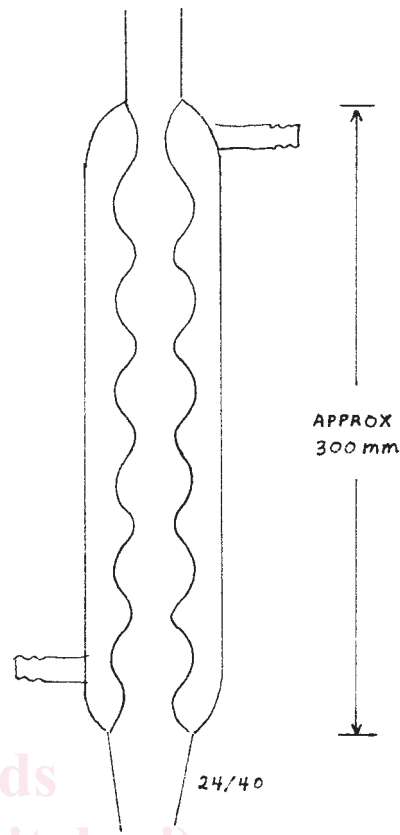


FIG. 4 Condenser, Allihn Type

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*⁸, 70 cm (27.6 in.) sheathed.

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.⁹ 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.

⁸ A resistance temperature device (RTD) is also acceptable.

⁹ *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.

6.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Specification D 1193. Referee situations require Type II distilled water defined by Specification D 1193.

6.3 *Metal Specimens*:¹⁰

6.3.1 *Washer-Shaped Metal Specimens*, 6.35-mm (1/4-in.) inside diameter by 19.05-mm (3/4-in.) outside diameter by 0.81 mm (0.032 in.) 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 (0.032-in.) by 25.4-mm (1-in.) 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, various grades, including 240 and 400 grit. Many papers, including “wet or dry,” “waterproof,” or iron-containing abrasives, such as natural emery are not satisfactory.

¹⁰ Satisfactory metal specimens may be obtained from: Metaspac Co., P.O. Box 27702, San Antonio, TX 78227-0707.

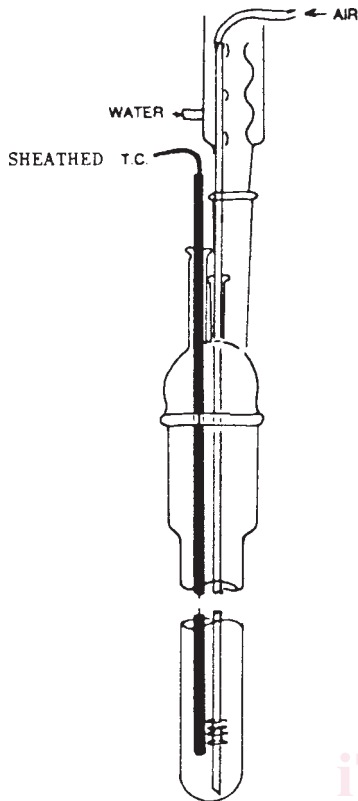


FIG. 5 Assembled Apparatus

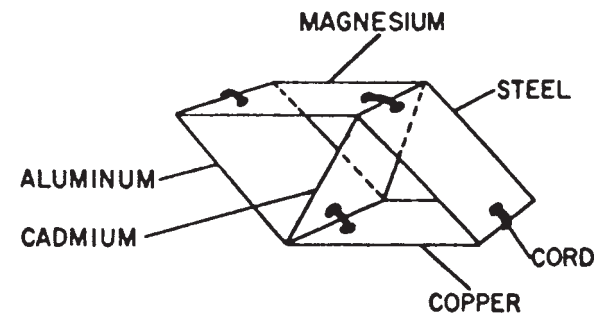
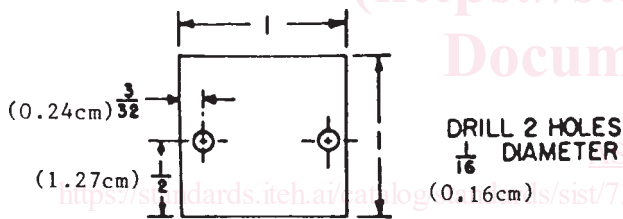


FIG. 6 Metal Square Dimensions and Arrangement

6.4.1 Silicon-Carbide Grains, 150 mesh.

6.5 Cotton, absorbent.

6.6 *n*-Heptane.

NOTE 3—**Warning:** Flammable. Harmful if inhaled.

6.7 Acetone.

NOTE 4—**Warning:** Extremely flammable. Vapors may cause flash-fire.

6.8 Nitric Acid, concentrated.

NOTE 5—**Warning:** Poison. Corrosive. Strong oxidizer.

6.9 Degreasing Solvents:¹¹ *n*-Heptane or toluene.

6.10 Carbon Remover for Glassware¹² (**Warning**—See Note 7)—Mix 35 mL of saturated sodium dichromate (aqueous) solution and 1000 mL of concentrated sulfuric acid.

NOTE 6—**Warning:** Corrosive.

6.11 Glassware Cleaning Solution (**Warning**—See Note 7)—Mix 35 mL of MICRO¹³ in water or 35 mL of NOCHROMIX¹⁴ and 1000 mL of concentrated sulfuric acid.

NOTE 7—**Warning:** Causes severe burns.

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.

NOTE 8—**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 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 Notes 2-8.

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.

¹¹ *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.

¹² Oakite Stripper R-8, available from Oakite Products, Inc., 50 Valley Rd., Berkeley Heights, NJ 07922, has been found satisfactory for this purpose.

¹³ International Products Corp., P.O. Box 118, Trenton, NJ 08601-0118.

¹⁴ Godax Laboratories, Inc., 6 Varick St., New York, NY 10013.

NOTE 9—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 10 and Note 11):

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

NOTE 10—An alternative test Procedure 1, which deletes the requirement for periodic oil sampling throughout the test period may be specified. Alternative test Procedure 1 requires an initial oil charge of 165 ± 2 mL (see 10.2).

NOTE 11—An alternative test 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 ± 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.¹⁵ 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 to 16 h (see Note 12).

NOTE 12—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 12).

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 10.1.6 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 by Test Method D 664.

10.1.2 Determine the kinematic viscosity of the test oil at 40°C and 100°C by Test Method D 445.

10.1.3 Turn on heating control to bring temperature of heating medium to a temperature that will maintain the oil sample within $\pm 0.5^\circ\text{C}$ ($\pm 1^\circ\text{F}$) of the specified temperature (**Warning**—See Note 2).

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.

10.1.9 Insert the 70-cm (27.6-in.) 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 ± 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 ± 20 mm.

10.1.13 Insert the condenser and start water flow. Maintain water temperature at $18 \pm 3^\circ\text{C}$ ($65 \pm 5^\circ\text{F}$).

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 ± 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 $\pm 0.5^\circ\text{C}$ ($\pm 1^\circ\text{F}$) 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.

¹⁵ A detergent found satisfactory is Alconox made by Alconox, Inc., 215 Park Avenue South, New York City, NY 10003.