Designation: D6594 – 20$^{\varepsilon 1}$

Standard Test Method for Evaluation of Corrosiveness of Diesel Engine Oil at 135 °C[1]

This standard is issued under the fixed designation D6594; 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.

ε[1] NOTE—Editorially updated TMC governance information in June 2022.

INTRODUCTION

Any properly equipped laboratory, without outside assistance, can use the procedure described in this test method. However, the ASTM Test Monitoring Center (TMC)[2] provides reference oils and an assessment of the test results obtained on those oils by the laboratory (see Annex A1). By these means, the laboratory will know whether their use of the test method gives results statistically similar to those obtained by other laboratories. Furthermore, various agencies require that a laboratory utilize the TMC services in seeking qualification of oils against specifications. For example, the U.S. Army imposes such a requirement in connection with several Army engine lubricating oil specifications.

Accordingly, this test method is written for use by laboratories that utilize the TMC services. Laboratories that choose not to use those services may simply ignore those portions of the test method that refer to the TMC.

This test method may be modified by means of information letters issued by the TMC. In addition, the TMC may issue supplementary memoranda related to the method (see Annex A1). For other information, refer to the research report of this test method.[3]

1. Scope*

1.1 This test method covers testing diesel engine lubricants to determine their tendency to corrode various metals, specifically alloys of lead and copper commonly used in cam followers and bearings.

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

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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:*[4]

D130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D5185 Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

D5844 Test Method for Evaluation of Automotive Engine Oils for Inhibition of Rusting (Sequence IID) (Withdrawn 2003)[5]

D6557 Test Method for Evaluation of Rust Preventive Characteristics of Automotive Engine Oils

[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.B0.02 on Heavy Duty Engine Oils.

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[2] The ASTM Test Monitoring Center will update changes in this test method by means of Information Letters. This edition incorporates revisions contained in all Information Letters through 19-2. Information Letters may be obtained from the ASTM Test Monitoring Center, 203 Armstrong Drive, Freeport, PA 16229, Attention: Director.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1443. Contact ASTM Customer Service at service@astm.org.

[4] 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.

[5] The last approved version of this historical standard is referenced on www.astm.org.

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

3. Terminology

3.1 *Definitions:*

3.1.1 *corrosion, n*—the chemical or electrochemical reaction between a material, usually a metal surface, and its environment that can produce a deterioration of the material and its properties. **D5844**

3.1.2 *developer, n—of an ASTM test method*, the assigned ASTM group, working under the supervision of its governing subcommittee and main committee, that formats the test method in accordance with the Form and Style for ASTM Standards, and continually refines the test method.

3.1.3 *developer, n—of a test procedure*, an individual or organization that selects the test apparatus and operating conditions.

3.1.4 *non-reference oil, n*—any oil other than a reference oil; such as a research formulation, commercial oil, or candidate oil. **D5844**

3.1.5 *reference oil, n*—an oil of known performance characteristics, used as a basis for comparison. **D5844**

3.1.5.1 *Discussion*—Reference oils are used to calibrate testing facilities, to compare the performance of other oils, or to evaluate other materials (such as seals) that interact with oils.

3.1.6 *specimen, n*—a piece or portion of a sample used to make a test.

3.1.7 *sponsor, n—of an ASTM test method*, an organization that is responsible for ensuring supply of the apparatus used in the test procedure portion of the test method.

3.1.7.1 *Discussion*—In some instances, such as a test method for chemical analysis, an ASTM working group can be the sponsor of a test method. In other instances, a company with a self-interest may or may not be the developer of the test procedure used within the test method, but is the sponsor of the test method.

3.1.8 *test oil, n*—any oil subjected to evaluation in an established procedure. **D6557**

4. Summary of Test Method

4.1 Four metal specimens of copper, lead, tin, and phosphor bronze are immersed in a measured amount of engine oil. The oil, at an elevated temperature, is blown with air for a period of time. When the test is completed, the copper specimen and the stressed oil are examined to detect corrosion and corrosion products, respectively.

4.2 A reference oil is tested with each group of tests to verify test acceptability.

5. Significance and Use

5.1 This test method is intended to simulate the corrosion process of non-ferrous metals in diesel lubricants. The corrosion process under investigation is that believed to be induced primarily by inappropriate lubricant chemistry rather than lubricant degradation or contamination. This test method has been found to correlate with an extensive fleet database containing corrosion-induced cam and bearing failures.[3]

6. Apparatus

6.1 The main apparatus consists of the following items of standard wall borosilicate glassware as shown in Figs. 1-6.

6.1.1 *Main Sample Tube,* Fig. 1.

6.1.2 *Sample Tube Head,* Fig. 2.

6.1.3 *Air Tube,* Fig. 3.

6.1.4 *Thermocouple Tube,* Fig. 4.

6.1.5 *Condenser,* Allihn Type, Fig. 5.

6.1.6 *Assembled Apparatus,* Fig. 6.

6.2 Additional glassware items and assembly accessories needed are:

6.2.1 *Hanger* (for metal specimens), of stainless steel, having the dimensions listed in Fig. 7.

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

6.3 Other items and equipment are:

6.3.1 Heating bath, with constant temperature control within ±0.5 °C of test temperature with an immersion depth of 23 cm to 35 cm. Oil baths are recommended. (**Warning—**There are exposed hot surfaces on apparatus. Avoid skin contact by use of protective equipment.)

6.3.2 *Ventilation,* to adequately remove fumes during heating.

6.3.3 *Air Supply,* use air from a clean, dry source.

6.3.3.1 An air drier is required when air needs to be conditioned. The method used is optional provided the air characteristics of 6.3.3 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.

6.3.4 *Flowmeter,* capable of measuring 10 L/h ± 1 L/h.

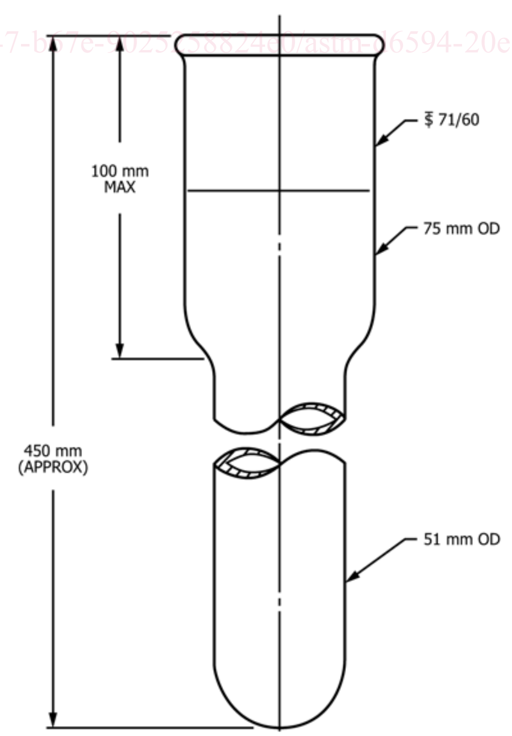


FIG. 1 Sample Tube

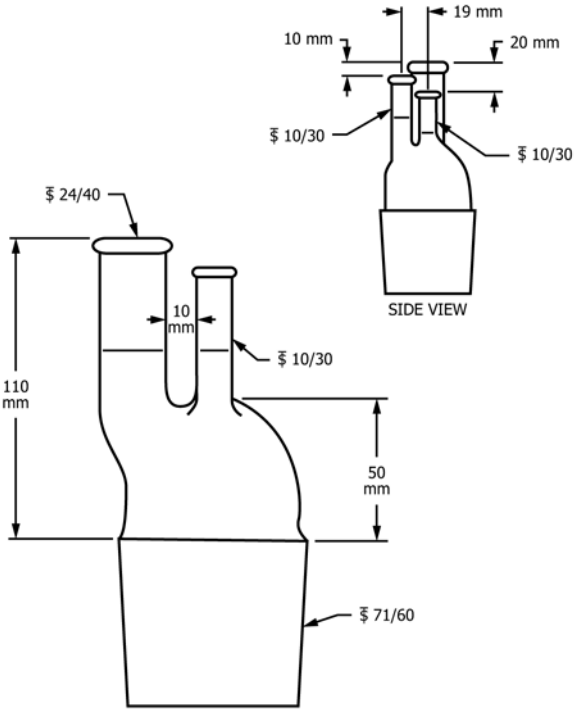


FIG. 2 Sample Tube Head

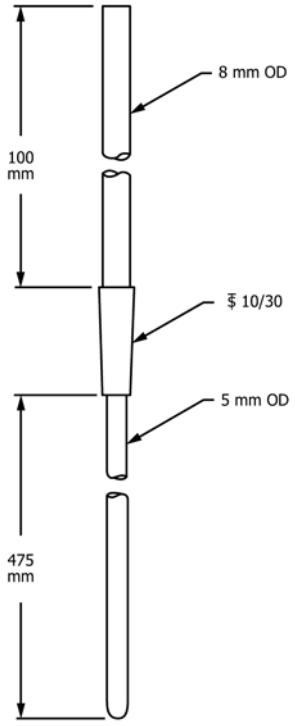


FIG. 4 Thermocouple Tube

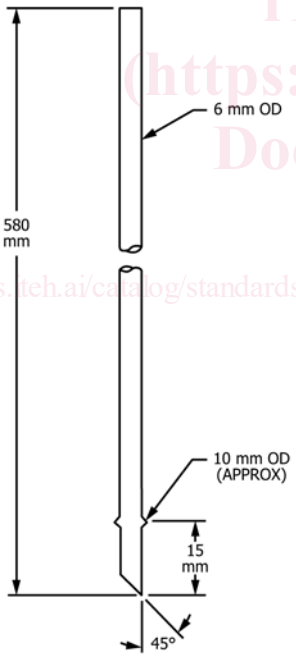


FIG. 3 Air Tube

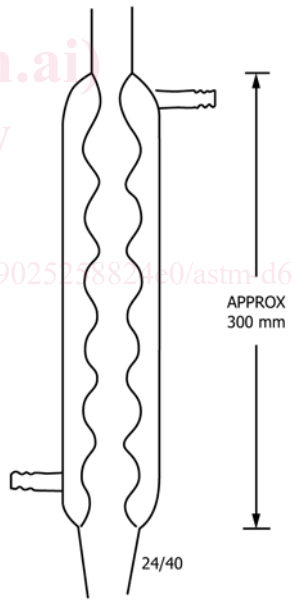


FIG. 5 Condenser, Allihn Type

6.3.5 *Syringe,* capable of accurately measuring out 100 mL of liquid.

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

6.3.7 *Forceps,* stainless steel; or gloves (powder free).

6.3.8 *Thermocouple,* or equivalent. Use sheathed thermocouple when the thermocouple is in direct contact with oil. When a thermocouple well is used, use a sheathed or unsheathed thermocouple in the well to control sample temperature; fill thermocouple well with a heat transfer medium.

6.3.9 *Sanding Block and Holder,* for specimen preparation.

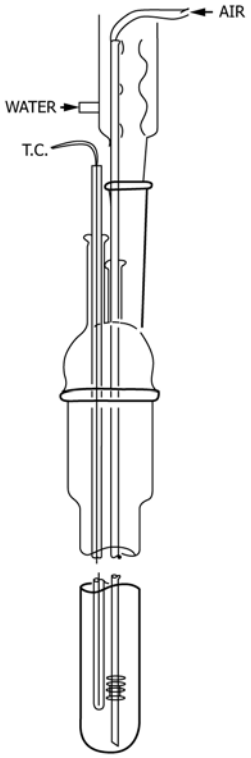


FIG. 6 Assembled Apparatus

7. Reagents and Materials

7.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications maintained by the Committee on Analytical Reagents of the American Chemical Society.[6]

7.2 *Metal Specimens*[7,8]

7.2.1 Specimens are 0.081 cm thick by 2.5 cm square, except the lead specimen, which is 0.157 cm thick. One specimen from each of the following metal types, each with two drilled holes (as shown in Fig. 7), is required:

7.2.1.1 *Copper* (R401-A),

7.2.1.2 *Lead* (R401-lead),

7.2.1.3 *Tin* (R401-tin), and

7.2.1.4 *Phosphor Bronze* (R401-PBz).

7.3 *Abrasive Paper,*[9] 240-grit aluminum oxide and 400-grit silicon carbide. Do not use iron-containing abrasives such as natural emery.

7.4 *Cotton,* 100 %.

7.5 *Acetone* (ACS), sulfur free. (**Warning—**Flammable. Health hazard.)

7.6 *Glassware Cleaning Solution,* Contrad (trademark) 70.[10,8] (**Warning—**Health hazard.)

7.7 *Heptane.* (**Warning—**Flammable. Health hazard.)

7.8 *Cleaning Solvent*—Cyclohexane or heptane, industrial grade. (**Warning—**Both are flammable and health hazards.)

7.9 *Reference Oil.*[2]

8. Preparation of Apparatus

8.1 *Cleaning:*

8.1.1 Rinse all items and the air tube adapter with cleaning solvent to remove residual oil, and air-dry.

8.1.2 Wash all glassware items and the air tube adapter with detergent. Rinse with tap water, distilled water, and dry.

8.1.3 The following more thorough glassware cleaning procedure can be used, if it is required for a given situation:

8.1.3.1 Fill and immerse all glassware items with glassware cleaning solution (see 7.6) and soak for 3 h to 16 h. (**Warning—**Corrosive, causes severe burns.)

8.1.3.2 Remove glassware from cleaning solution; rinse several times with tap water, followed by distilled water, and oven-dry.

8.1.3.3 This more thorough glass cleaning procedure is necessary in a referee situation, unless an alternative glassware cleaning solution is available that is satisfactory to all parties involved.

8.2 *Assembled Apparatus,* shown in Fig. 6.

8.3 *Preparation of Metal Specimens:*

8.3.1 In all succeeding steps, handle the specimens only with stainless steel forceps or powder-free gloves until the final weighing. If large defects or particles are present on the metal specimens, remove them first using coarse sandpaper, followed by polishing with the 240 and 400-grit abrasive papers, as described in 8.3.2.

8.3.2 Remove any burrs from the drilled holes with a 1.5113 mm, #53 (drill wire gauge) drill bit. Using a sanding block with a specimen holder, remove all surface blemishes from both sides and all four edges of each specimen with 240-grit abrasive paper. Finish polishing with 400-grit paper wetted by acetone to remove marks from previous polishing.

8.3.2.1 A good technique is to 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.

8.3.2.2 Make sure that the specimen edges are polished in the same manner as the surfaces. Carry out this procedure using normal room lighting and without magnification of the specimen surface. Do not scribe or otherwise mark the surfaces.

8.3.3 Store the polished metal specimens in acetone.

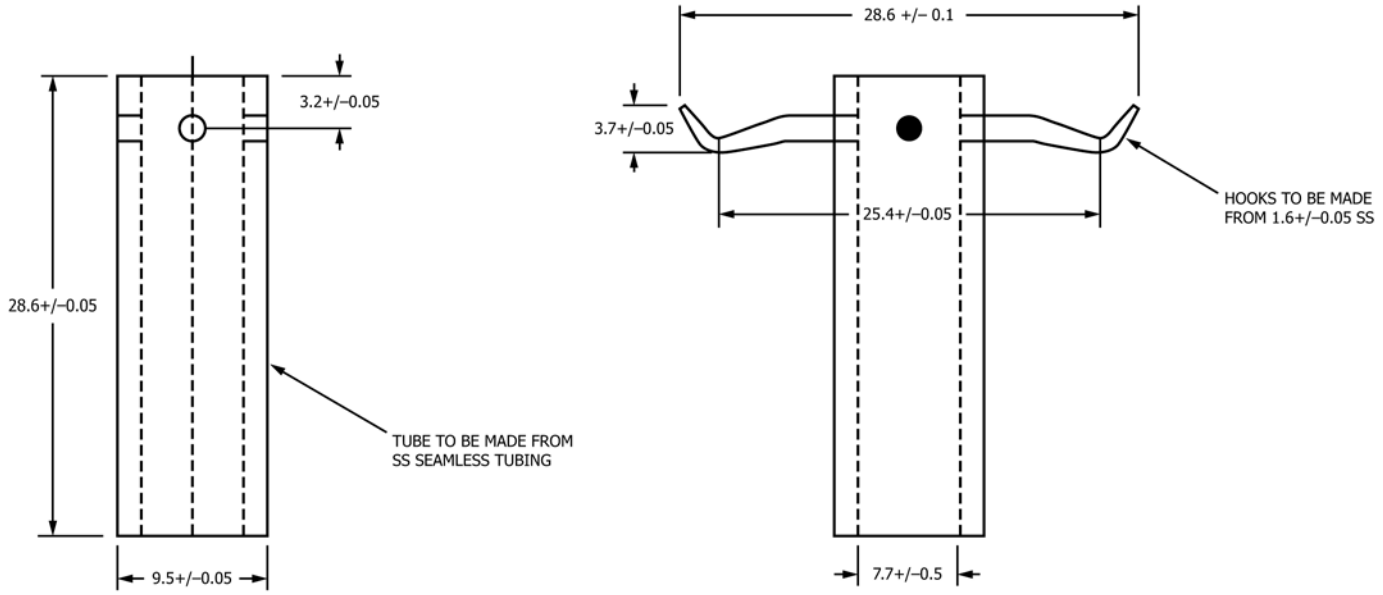
[6] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[7] Obtain metal specimens from Test Engineering, Inc. (TEI), 12718 Cimarron Path, San Antonio, TX 78249-3423.

[8] The sole source of supply of the apparatus known to the committee at this time is noted in the adjoining footnote. 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,[1] which you may attend.

[9] Suitable abrasive paper meeting these specifications is included with the metal specimens when the specimens are ordered.

[10] Contrad 70 is available from Decon Laboratories, Inc., 460 Glennie Circle, King of Prussia, PA 19406, (800) 332–6647.



NOTE 1—All dimensions in mm.

FIG. 7 Specimen Hanger

8.3.4 Just prior to a test start, remove each specimen from the acetone, and clean all metal dust from the specimen using 100 % cotton. Rub with a light-to-medium touch to remove particles but do not polish the specimen further.

8.3.5 Wash specimens in acetone, and allow them to dry in a desiccator.

9. Procedure

9.1 Add 100 mL ± 1 mL of oil to the sample tube by syringe.

9.2 Place the specimen hanger onto the air tube, and hang test specimens on their respective hooks.

9.2.1 Arrange the specimens on the hanger in the sequence: lead, copper, tin, and phosphor bronze.

9.3 Insert the air tube with the attached specimens into the sample tube so that the air tube rests on the bottom of the sample tube.

9.4 Place the sample tube head on the sample tube.

9.5 Place the assembled sample tube and condenser into the bath so that the sample tube is submerged 23 cm to 35 cm in the bath with the test oil temperature set at 135 °C ± 0.5 °C.

9.6 Start the flow of the cooling water through the condenser jacket.

9.7 To begin testing, connect the source of clean, dry air 5 L/h ± 0.5 L/h to the air tube and allow the air to flow for 168 h. Use a calibrated flow meter in setting airflow rates.

9.8 *End of Test*—After 168 h at 135 °C, shut off the airflow and disassemble.

9.8.1 Remove air supply and disconnect condenser.

9.8.2 Remove sample tube from the bath, and allow it to cool to room temperature.

10. Test Results

10.1 Remove the air tube with the attached specimens from the sample tube. Do not touch the specimens with hands. Retain the sample tube and test oil for further examination.

10.2 Using forceps, wash the copper specimen in heptane, and discard the other specimens.

10.3 Rate the copper specimen for tarnish according to the Strip Examination, Interpretation, and Report sections of Test Method D130.

10.4 Immediately after calibration of the ICP-AES instrument (as specified in Test Method D5185), use Test Method D5185 to determine the concentration of copper, lead, and tin in both the new and used oil. No decimal results shall be recorded.

10.4.1 Any measured concentration results that round to zero shall be reported as zero.

10.5 *Calculations*—Change in metal concentration in the used test oil:

$$\Delta C = C_2 - C_1 \quad (1)$$

where:

ΔC = change in metal concentration before and after test,
C_1 = measurement of metal concentration in new test oil (as determined in 10.4), and
C_2 = measurement of metal concentration in used test oil (as determined in 10.4).

11. Reference Oil Testing

11.1 Test a TMC-coded reference oil along with each batch of non-reference oil tests. Run the reference oil simultaneously with, and in the same bath as, the non-reference oils.

NOTE 1—Annex A1 discusses the involvement of the ASTM TMC with