



Standard Test Method for Evaluation of Corrosiveness of Diesel Engine Oil at 135°C¹

This standard is issued under the fixed designation D 6594; 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.

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

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 and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:⁴

D 130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test ~~D2355 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)~~

D 5185 Test Method for Determination of Additive Elements, Wear Metals, and Contaminants in Used Lubricating Oils and Determination of Selected Elements in Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

D 5844 Test Method for Evaluation of Automotive Engine Oils for Inhibition of Rusting (Sequence IID)

D 6557 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 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 ~~06-2-07-1~~. Information Letters may be obtained from the ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15206-4489, Attention: Administrator.

³ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1443.

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.

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. **D 5844**

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. **D 5844**

3.1.5 *reference oil, n*—an oil of known performance characteristics, used as a basis for comparison. **D 5844**

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. **D 6557**

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 $\pm 0.5^\circ\text{C}$ ($\pm 1^\circ\text{F}$) of test temperature with an immersion depth of 23 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 ± 1 L/h.

6.3.5 *Ruler*, 30-cm minimum length, with 1-mm graduations.

6.3.6 *Balance*, with a capacity of 2500 g and sensitivity of 0.1 g.

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

6.3.8

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

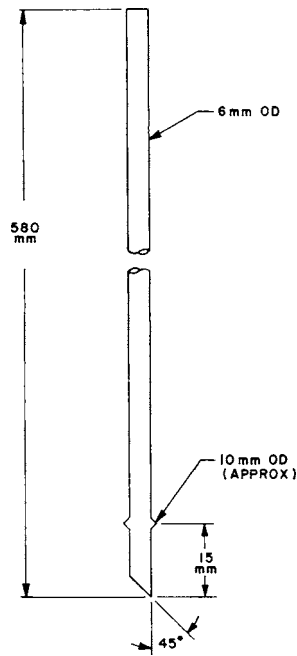


FIG. 3 Air Tube

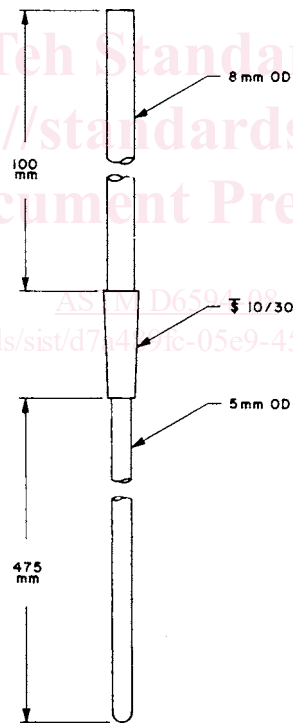


FIG. 4 Thermocouple Tube

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.⁵

⁵ Withdrawn.

⁵ *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 *Annual 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.

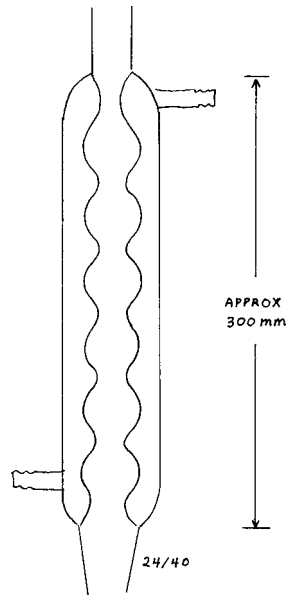


FIG. 5 Condenser, Allihn Type

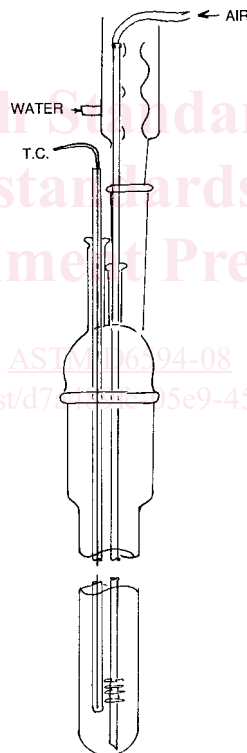


FIG. 6 Assembled Apparatus

7.2 Metal Specimens^{6,7}

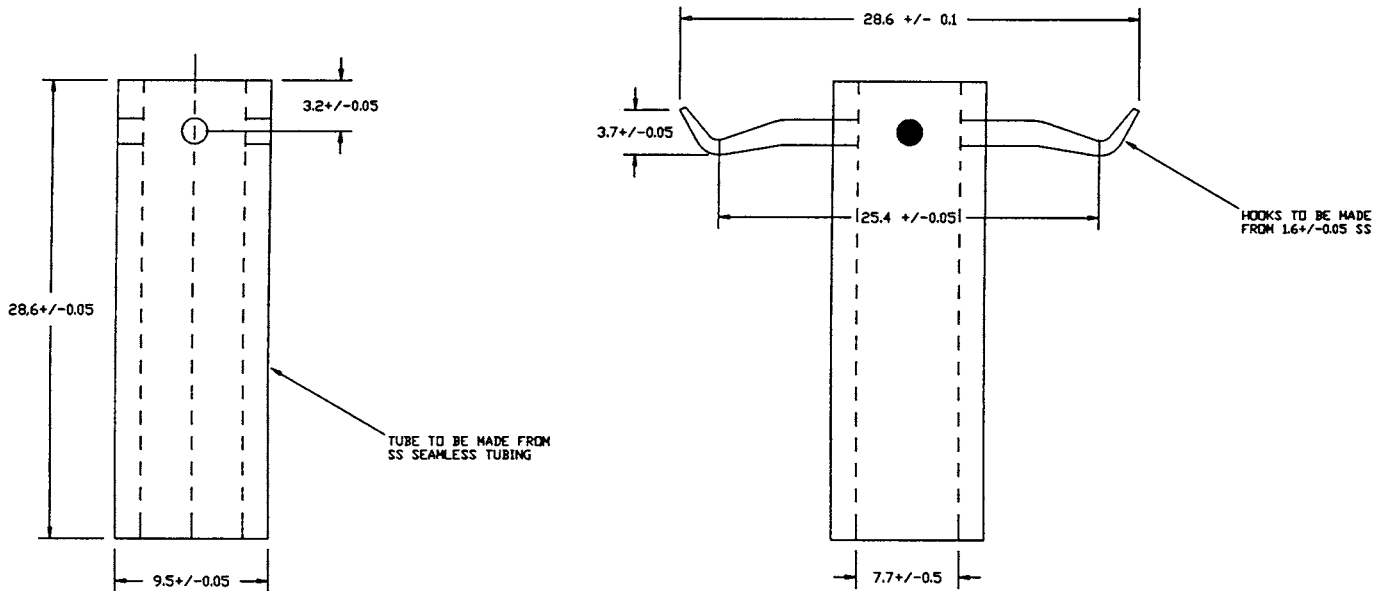
7.2.1 Specimens are 0.081 cm thick by 2.5 cm square, except that lead specimen that is 0.178 cm thick. One specimen from each of the following metal types, each with two drilled holes, is required:

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

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⁷ 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, which you may attend.



NOTE—All dimensions in mm.

FIG. 7 Specimen Hanger

- 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,⁸ 240-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, NOCHROMIX.^{7,9} (**Warning**—Causes severe burns.)

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

7.8 Solvent—Use only mineral spirits meeting the requirements of Specification D 235, Type II, Class C for Aromatic Content (0-2% vol), Flash Point (142°F/61°C, min) and Color (not darker than +25 on Saybolt Scale or 25 on Pt-Co Scale). (**Cleaning Solvent**—Cyclohexane or heptane, industrial grade. (**Warning**—Combustible. Health hazard.) Obtain a Certificate of Analysis for each batch of solvent from the supplier. Both are flammable and health hazards.)

7.9 Reference Oil.²

8. Preparation of Apparatus

8.1 Cleaning of Glassware

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

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

8.1.4.1 Fill and immerse all glassware items with glassware cleaning solution (see

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 to 16 h.

(**Warning**—Corrosive, causes severe burns.)

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

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

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, which you may attend.

⁸ Suitable abrasive paper meeting these specifications is included with the metal specimens when the specimens are ordered.

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⁹ Obtain NOCHROMIX from Godax Laboratories, Inc., 720-B Erie Avenue, Takoma Park, MD 20912.