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Designation: D 6594 – 00

An American National Standard

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

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 is used to test 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 the 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:*

D 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test[4]

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)[5]

D 5844 Test Method for Evaluation of Automotive Engine Oils for Inhibition of Rusting (Sequence IID)[5]

D 6557 Test Method forEvaluation of the Rust Preventive Characteristics of Automotive Engine Oils[6]

E 691 Practice for Conducting an Inter-Laboratory Study to Determine the Precision of a Test Method[7]

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

[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 on Automotive Lubricants.

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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 00-1. Information Letters may be obtained from the ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15206-4489, Attention: Administrator.

[3] Available from ASTM Headquarters. Request the High Temperature Corrosion Bench Test Research Report, RR:D02-1443.

[4] *Annual Book of ASTM Standards*, Vol 05.01.

[5] *Annual Book of ASTM Standards*, Vol 05.03.

[6] *Annual Book of ASTM Standards*, Vol 05.04.

[7] *Annual Book of ASTM Standards*, Vol 14.02.

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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 ± 0.5°C (± 1°F) of test temperature with an immersion depth of 250 ± 20 mm. 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 *Dry Air Supply*, with a dew point of −68°C.

6.3.3.1 When air needs to be conditioned an air drier is

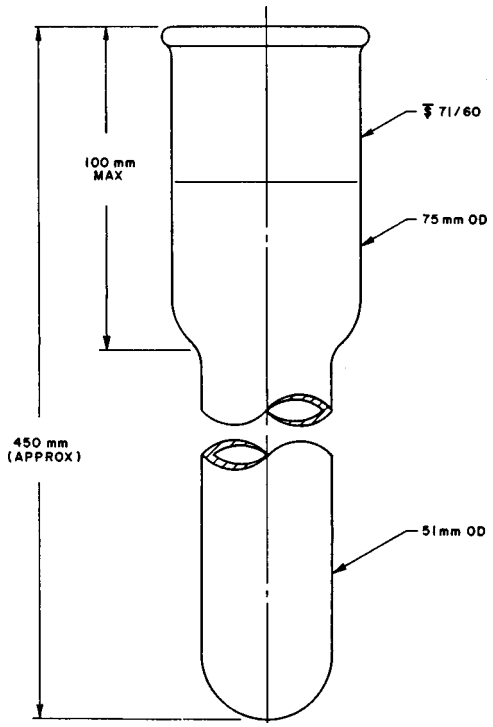

FIG. 1 Sample Tube

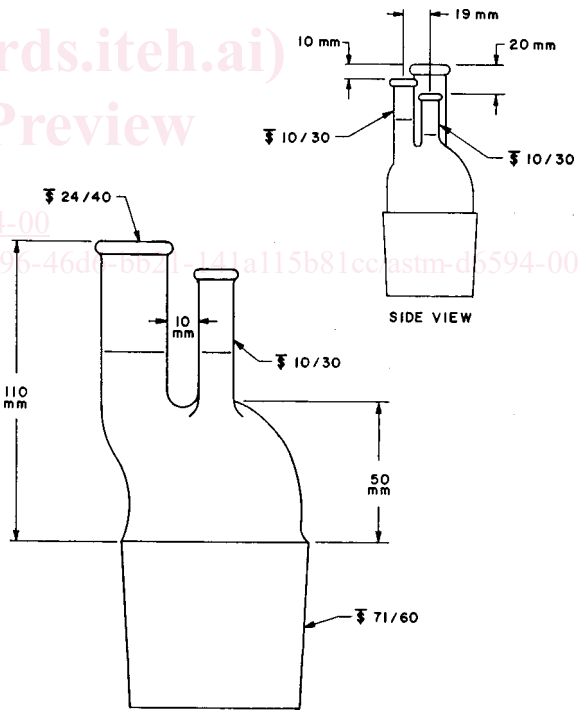

FIG. 2 Sample Tube Head

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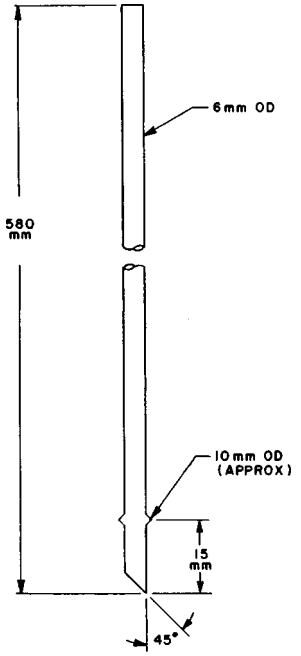

FIG. 3 Air Tube

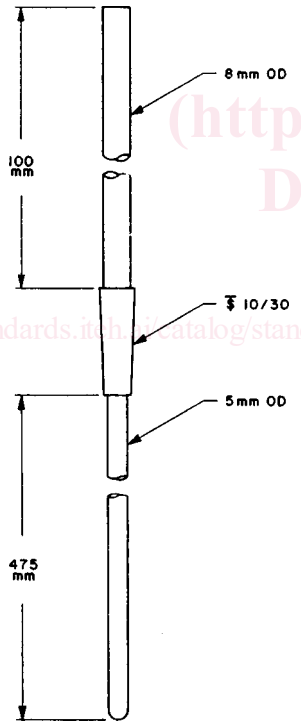

FIG. 4 Thermocouple Tube

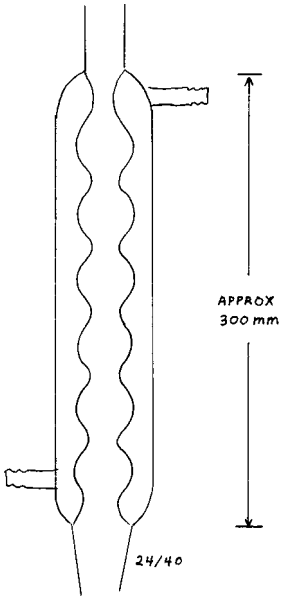

FIG. 5 Condenser, Allihn Type

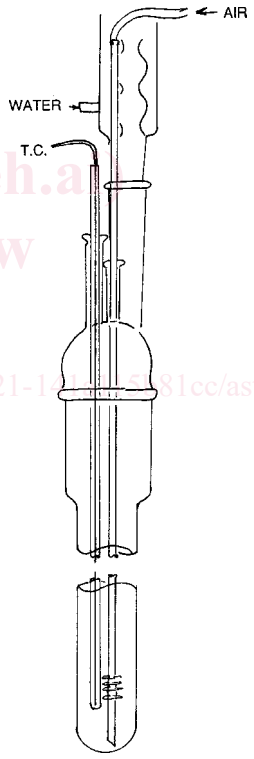

FIG. 6 Assembled Apparatus

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

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

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

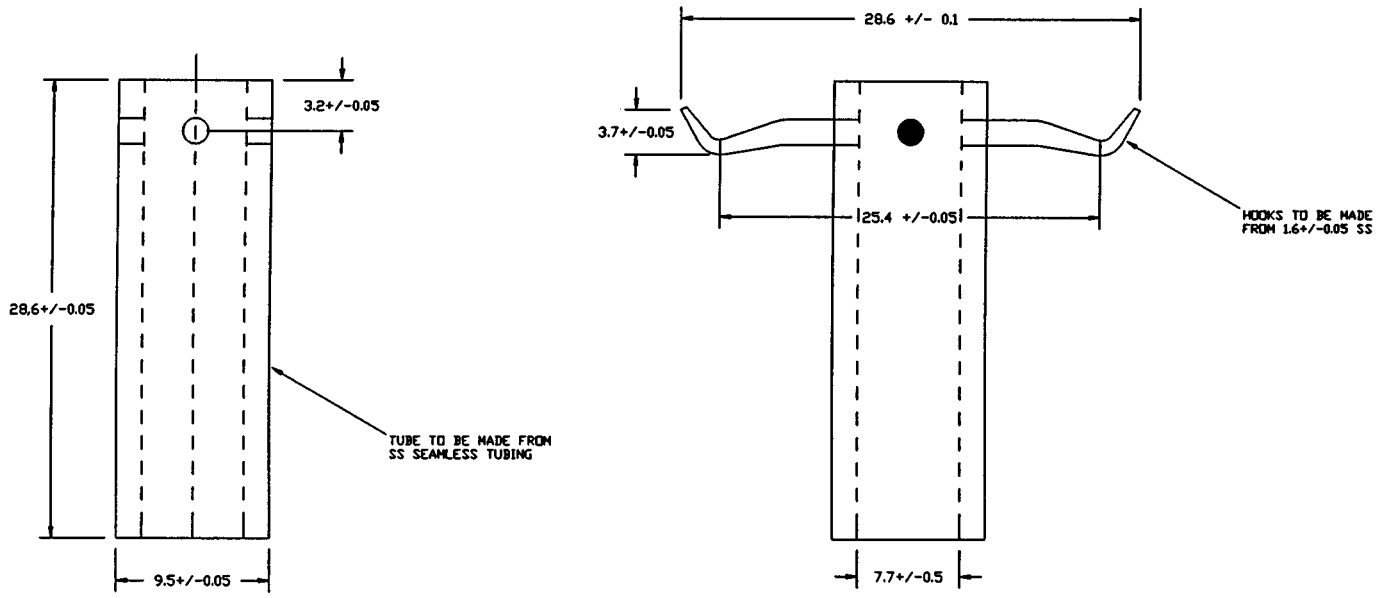
6.3.8 *Forceps*, stainless steel; or gloves (powder free).

6.3.9 *Thermocouple*, or equivalent. Use sheathed thermocouple when used directly in contact with oil. Use unsheathed thermocouple when thermocouple well is used; fill thermocouple well with a heat transfer medium.

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

7. Reagents and Materials

7.1 *Purity of Reagents*—Use reagent grade chemicals in all



NOTE 1—All dimensions in mm.

FIG. 7 Specimen Hanger

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[8].

7.2 *Metal Specimens*[9,10]

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:

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*[11], 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[12,10]. (**Warning—**Causes severe burns.)

7.7 *Tetrahydrofuran (THF)*. (**Warning—**Toxic and flammable. Health hazard.)

7.8 *Degreasing Solvent*, (THF recommended).

7.9 *Naphtha*, Aromatic. (**Warning—**Flammable. Health hazard.)

7.10 *Reference Oil*[2].

8. Preparation of Apparatus

8.1 *Cleaning of Glassware*:

8.1.1 Rinse all glassware items and the air tube adapter with degreasing 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 7.6) and soak for 3 to 16 h. (**Warning—**Corrosive, causes severe burns.)

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

8.1.4.3 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.

8.2 *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 Using a sanding block with a specimen holder, remove all surface blemishes from both sides and all four edges of each

[8] *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).

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

[10] 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 Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

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

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

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specimen with 240-grit abrasive paper. Remove any burrs from the drilled holes with a 1/16 in. drill bit. 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 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 tetrahydrofuran.

8.3.4 Just prior to a test start, remove each specimen from the tetrahydrofuran, 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 tetrahydrofuran and allow them to dry in a desiccator.

9. Procedure

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

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

9.2 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.

9.3 Place the sample tube top on the sample tube.

9.4 Weigh the air tube, sample tube, and contents together within 0.1 g.

9.5 Add 100 ± 1 mL of test oil volumetrically to the sample tube by syringe. Reweigh the air tube, sample tube, and contents together to within 0.1 g, and determine the weight of the oil added.

9.6 Assemble the sample tube and condenser and mount the assembly so that 30 ± 5 cm of the sample tube is submerged in the bath with the test oil temperature set at 135 ± 0.5°C.

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

9.8 To begin testing, connect the source of clean, dry air (5 ± 0.5 L/h) to the air tube and allow the air to flow for 168 h. House air with pre-drying or bottle reagent grade air should be used. Use a calibrated flow meter in setting airflow rates.

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

9.9.1 Remove air supply and disconnect condenser.

9.9.2 Remove sample tube from the bath, allow it to cool at least for 15 min, and wipe off the outside of the tube with a cloth dampened with naphtha.

9.9.3 Make the following measurements: Re-weigh the air tube, sample tube, and contents to within 0.1 g, determine weight of the oil sample remaining, and compute the percentage of weight loss resulting from evaporation of oil (see 10.5.1). If the evaporation loss is greater than 8 % leakage is present. Correct the leak, and repeat the determination, using a fresh oil sample and new specimens.

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 or gloves, wash the copper specimen in tetrahydrofuran, 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 D 130.

10.4 Immediately after calibration of the ICP-AES instrument (as specified in Test Method D 5185), use Test Method D 5185 to determine the concentration of copper, lead, and tin in both the new and used oil.

10.5 *Calculations*:

10.5.1 *Evaporation Loss:*

$$L = [(W_1 - W_2)/W_3] \times 100 \quad (1)$$

where:

L = percent evaporation loss,
W_1 = initial weight of air tube, sample tube and contents, including test oil,
W_2 = final weight of air tube, sample tube and contents, including test oil, and
W_3 = initial weight of test oil.

10.5.2 Change in metal concentration in the used test oil:

$$\Delta C = C_2 - C_1 \quad (2)$$

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 respect to the reference test-monitoring program.

11.1.1 Prior to requiring a reference oil test, procure a supply of reference oils directly from the TMC. These oils have been formulated or selected to represent specific chemistry types, or performance levels, or both. Each reference oil sample is identified using a unique set of identification codes on the container labels. The coded reference samples provide for a blind reference-testing program to protect against the possibility of bias in the results.

11.1.1.1 The testing laboratory tacitly agrees to use the TMC reference oils exclusively in accordance with the TMC's published Policies for Use and Analysis of ASTM Reference Oils, and to run and report the reference oil test according to TMC guidelines.

NOTE 2—Policies for the Use and Analysis of ASTM Reference Oils is available from the TMC.

11.1.2 Request a reference oil assignment from the TMC for this test method. The TMC will determine the specific reference oil to be tested by the laboratory. Assignments will be

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made by the unique identifying codes on the reference oil container labels. Provide the TMC with the bath identification number for the test.

11.1.3 Run the TMC reference oil test according to the test method and in the same manner as the non-reference oil test(s).

11.1.4 *Reporting of Reference Oil Test Results*—Report the results of all reference oil tests to the TMC according to the following directives:

11.1.4.1 Use the data reporting formats detailed in Annex A2 (see Figs. A2.1-A2.4) for reporting all TMC reference oil test data to the TMC. Report only the reference oil results to the TMC. Do not include any non-reference test data. Complete all of the required blank fields on the forms.

11.1.4.2 Transmit reference test data to the TMC by electronic means or by telephone facsimile immediately upon completion of the test analysis. Include all of the reporting forms in the transmission.

NOTE 3—Specific protocols for the electronic transmission of test data to the TMC are available from the TMC.

11.1.5 *Evaluation of Reference Test Oil Results*—Upon receipt of the transmitted TMC reference oil test results, the TMC will review the test for operational adherence to the published test method. If the test is found to be operationally valid, the reference oil results will be evaluated using acceptance criteria established by the governing surveillance panel. The reference oil acceptance criteria are subject to change at the discretion of the surveillance panel.

11.1.5.1 If the transmitted test is found to be both operationally valid and statistically acceptable, the testing laboratory will be notified of the acceptable status of the reference test. The uncoded TMC reference oil identification will also be disclosed to the testing laboratory.

11.1.5.2 In the event that a TMC reference oil test is found to be unacceptable, an explanation of the problem relating to the failure will be provided to the testing laboratory. If there is an obvious operational reason for the failed test, the problem shall be corrected before requesting another TMC reference oil assignment. If the reason for the failure is not obvious, all test-related equipment shall be re-checked for compliance to the test method and good laboratory practice. Following this re-check the TMC will assign another TMC reference oil for testing.

11.1.6 *Status of Non-reference Oil Tests Relative to TMC Reference Oil Tests*—The batch of non-reference tests is considered valid only if the results of the TMC reference oil test meet the predetermined acceptance specifications for the particular reference oil tested.

12. Report

12.1 Report (see Annex A2 for Report Format) the tarnish rating of the copper specimen (as determined in 10.3) based on the highest rating (most corrosion) if the rating is different for either side.

12.2 Report the concentrations of copper, lead, and tin in the new oil (C_1 in 10.5.2) and stressed oil (C_2 in 10.5.2), and the respective changes in metal concentrations ($\Delta\ C$ in 10.5.2).

13. Precision and Bias

13.1 *Precision*—The precision of the test method was determined by performing round robin tests in seven laboratories, in accordance with guidelines in Practice E 691.

13.1.1 *Intermediate Precision (I.P.)*, (formerly called repeatability) is defined as the difference between two results obtained by the same operator in the same laboratory, using the same test method on the same oil. Based on round robin tests, Intermediate Precision, in the long run, in the normal and correct conduct of the test method, would exceed the following *I.P.* values in only one case in twenty.

13.1.2 *Reproducibility (R)*, is defined as the difference between two single and independent results obtained by different operators working in different laboratories, using the same test method on the same oil. Based on round robin tests, Reproducibility, in the long run, in the normal and correct conduct of the test method, would exceed the following *R* values in only one case in twenty.

13.1.3 Δ *Copper, ppm* (with natural log transformation):

$$S_{I.P.} = 0.272 \qquad I.P. = 0.754 \tag{3}$$

$$S_R = 0.400 \qquad R = 1.109$$

13.1.4 Δ *Lead*, ppm.

$$S_{I.P.} = 6.14 \qquad I.P. = 43.02 \tag{4}$$

13.1.5 Δ *Tin*, content precision to be determined when an appropriate reference oil is located.

13.2 Bias is unknown at this time, but will be determined as more data are collected.

14. Keywords

14.1 corrosion; diesel engine; lubricant

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