Designation: D 5968 – 00a

An American National Standard

Standard Test Method for Evaluation of Corrosiveness of Diesel Engine Oil[1]

This standard is issued under the fixed designation D 5968; 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

The method described in this test method is based on the gas turbine lubricant corrosion and oxidation test described in Federal Test Method Standard 791, Method 5308. Because this test method relates to corrosion in diesel engines rather than in gas turbines, temperatures, metal coupons, and certain parts of the test procedure were modified to be more appropriate for heavy duty diesel engines.

The method described in this test method can be used by any properly equipped laboratory, without outside assistance. 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 on the Cummins Bench Corrosion Test.[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. Correlation with field experience has been established.[4]

1.2 The values stated in acceptable 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.* Specific hazard statements are given in 5.3.1, 6.6, 6.7, 6.8, 6.9, 6.10, 6.11, 7.1.1, 7.1.2, 7.1.5, and 7.4.1.

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[5]

D 4636 Test Method for Corrosiveness and Oxidation Stability of Hydraulic Oils, Aircraft Turbine Engine Lubricants, and Other Highly Refined Oils[6]

D 5185 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)[7]

E 691 Practice for Conducting an Inter-Laboratory Study to Determine the Precision of a Test Method[8]

[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] ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15206-4489, Telephone: (412) 365-1000, Fax: (412) 365-1045 (reference oil test telephone reports), Fax: (412) 365-1047 (other messages), Telephone Oil Assignments: (412) 365-1004.

[3] Available from ASTM Headquarters. Request RR:D02-1322. The research report and this test method are supplemented by Information Letters and Memoranda issued by the ASTM Test Monitoring Center. This edition incorporates revisions contained in all information letters through No. 00–1. Users of this test method shall contact the ASTM Test Monitoring Center to obtain the most recent of these.

[4] Wang, J. C., and Cusano, C. M., "Development of A Bench Test to Detect Oils Corrosive to Engine Components," SAE Technical Paper No. 940790, 1994.

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

[6] Discontinued, Test Method D 4636 served as the basis for this test method. See 1994 *Annual Book of ASTM Standards*, Vol 05.03.

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

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

2.2 *U.S. Federal Test Method Standards:*[9]

Federal Test Method Standard 791, Method 5308.7 Corrosiveness and Oxidation Stability of Light Oils (Metal Squares)

3. Summary of Test Method

3.1 Four metal coupons 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 coupons and the stressed oil are examined to detect corrosion.

3.2 An industrial reference oil is tested with each group of tests to verify test acceptability.

4. Significance and Use

4.1 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.

5. Apparatus

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

5.1.1 *Main Sample Tube*, Fig. 1.

5.1.2 *Sample Tube Head*, Fig. 2.

5.1.3 *Air Tube*, Fig. 3.

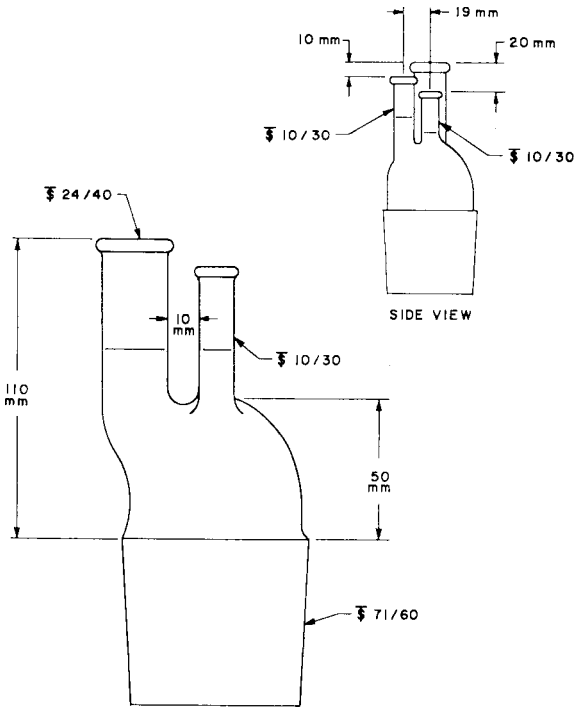

FIG. 2 Sample Tube Head

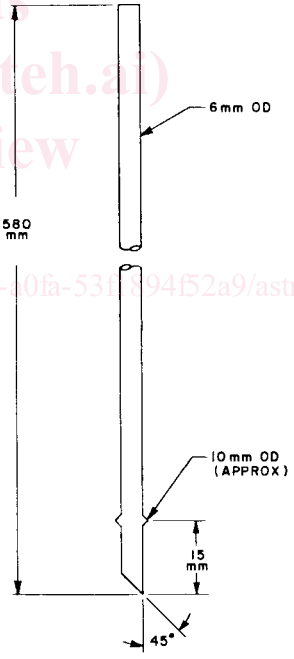

FIG. 3 Air Tube

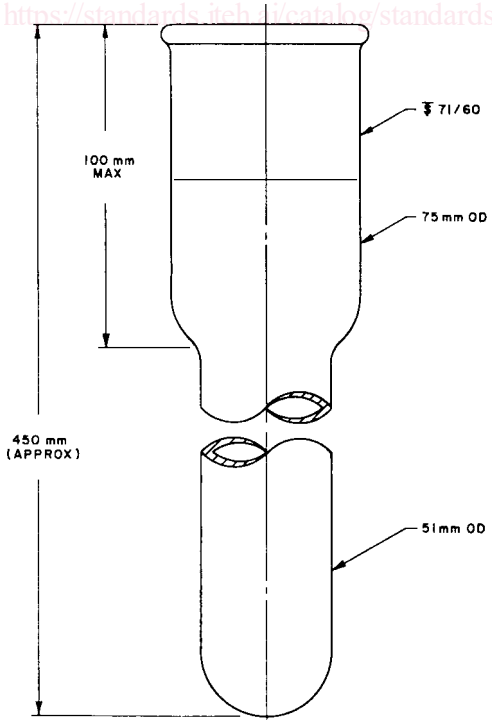

FIG. 1 Sample Tube

5.1.4 *Thermocouple Tube*, Fig. 4.

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

5.1.6 *Assembled Apparatus*, Fig. 6.

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 main sample tube. Tube is bent U-shape with exit end

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

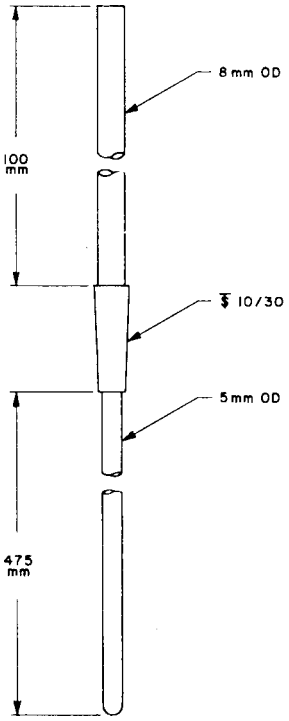

FIG. 4 Thermocouple Tube

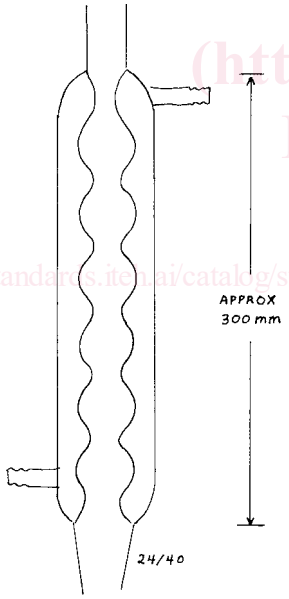

FIG. 5 Condenser, Allihn Type

fitted by a one-hole stopper to a 25-mL filtering flask. Exit end may be any convenient length.

5.2.3 *Adapter*, [10,11] Polytetrafluoroethylene for 10/18 joint for sealing of air tube to sample tube head.

[10] A satisfactory source for this item is Kontes Glass Co., Vineland, NJ 08360.

[11] 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,[1] which you may attend.

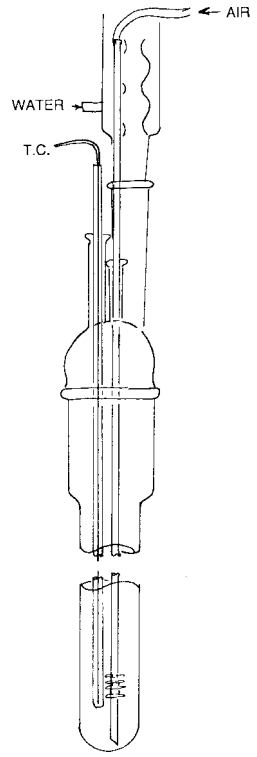

FIG. 6 Assembled Apparatus

5.3 Other items and equipment are:

5.3.1 *Heating Bath*, constant temperature control within ± 0.5°C (±1°F) of test temperature with an immersion depth of 250 ± 20 mm. Oil or aluminum baths are recommended. (**Warning**—There are exposed hot surfaces on apparatus. Avoid skin contact by use of protective equipment.)

5.3.2 *Hood Ventilation*, to adequately remove fumes during heating.

5.3.3 *Air Supply*, dry air, with a dew point of −68°C (−90°F) maximum and free of reactive contaminants.

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

5.3.5 *Balance*, analytical sensitivity 0.1 mg.

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

5.3.7 *Microscope*, with 20-diameter magnification.

5.3.8 *Assembly Fixture*, wood slotted to hold coupons squares (assembly as shown in Fig. 7) for tying with wire.

5.3.9 When air needs to be conditioned there is a need for an air drier. 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.

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

5.3.11 *Forceps*, stainless steel.

5.3.12 *Thermocouple*.

5.3.13 *Brush*, short-bristled, stiff (old-style typewriter cleaning brush or equivalent).

6. Reagents and Materials

6.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated it is intended that all reagents

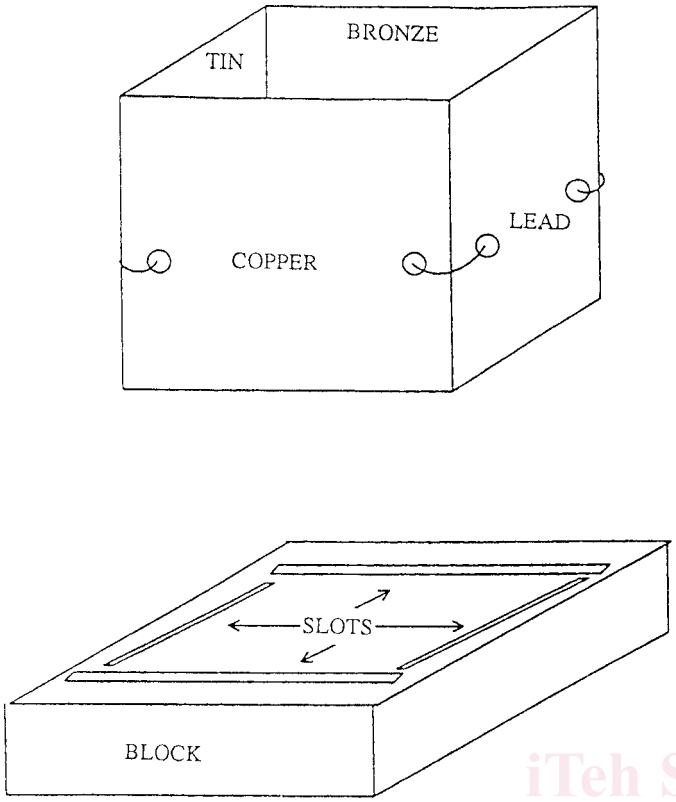

FIG. 7 Arrangement of Metal Coupons

conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[12]

6.2 *Metal Specimens:* [11,13]

6.2.1 Coupons, 0.081 cm thick by 2.5 cm square, one each, with two drilled holes (as shown in Fig. 7), as follows:

6.2.1.1 Copper (R401-A),

6.2.1.2 Lead (R401-lead),

6.2.1.3 Tin (R401-tin), and

6.2.1.4 Phosphor Bronze (R401-LEADz).

6.3 *Nichrome Wire*, clean (for tying coupons together).

6.4 *Abrasive Paper*, 240 grit aluminum oxide and 400 grit silicon-carbide.[14] Do not use iron-containing abrasives such as natural emery.

6.5 *Cotton*, absorbent.

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

6.7 *Degreasing Solvents*, Trichlorotrifluorethane or 1,1,1-trichloroethane. (**Warning**—Harmful if inhaled.)

6.8 *Glassware Cleaning Solution*, mix 35 mL of saturated sodium dichromate (aqueous) solution and 1000 mL of concentrated sulfuric acid. (**Warning**—Causes severe burns.)

6.9 *Carbon Remover for Glassware*, [11,15] Oakite Stripper R-8. (**Warning**—Corrosive, causes severe burns.)

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

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

6.12 *Filter Paper*.

6.13 *Kimwipe Tissues*, or similar.

6.14 *Industrial Reference Oil*.[2]

7. Preparation of Apparatus

7.1 *Cleaning of Glassware from Previous Run*:

7.1.1 Rinse all glassware items and the air tube adapter with degreasing solvent to remove residual oil, and air dry. (**Warning**—Harmful if inhaled.)

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

7.1.3 Wash all glassware items and the air tube adapter with detergent.[11,16] Rinse with water to remove detergent, and dry.

7.1.4 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.

7.1.5 A more elaborate glass cleaning procedure can be used, if it is for a given situation. This cleaning procedure is necessary in a referee situation unless a cleaning solution can be used which is satisfactory to all parties involved. Fill and immerse all glassware items with glassware cleaning solution and soak for 3 to 16 h. (**Warning**—Corrosive, causes severe burns.)

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

7.2 *Cleaning of Glassware (New)*:

7.2.1 Proceed as in 7.1.3 and 7.1.6 in that order (omit 7.1.1, 7.1.2, 7.1.4, and 7.1.5).

7.3 *Assembly*—Assemble as shown in Fig. 6 using only the test oil to lubricate glass joints during assembly.

7.4 *Preparation of Metal Specimens*:

7.4.1 Wash a length of the metal tying wire with tetrahydrofuran and acetone and allow to dry. (**Warning**—This and the following preparation processes should be performed under a fume hood.)

7.4.2 The metal squares are prepared as follows:

7.4.2.1 Using the 240 grit abrasive paper, remove all surface blemishes from both sides and all four edges of each square, and any burrs from the drilled holes. Finish polishing with 400 grit paper wetted by acetone to remove marks from previous polishing. A good technique is to place abrasive paper on a flat surface, then rub the specimen with longitudinal strokes in a

[12] *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.

[13] Satisfactory metal specimens may be obtained from: Test Engineering, Inc. (TEI), 12718 Cimarron Path, San Antonio, TX 78249-3423. This is the only coupon source to be used for obtaining a valid reference run and data for certification.

[14] Suitable abrasive paper meeting these specifications is included with the metal coupons from the source indicated in 6.2.

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

[16] A detergent found satisfactory is Alconox made by Alconox, Inc., 215 Park Ave. S., New York, NY 10003.

direction perpendicular to that used with 240 grit paper. Use a different sheet of paper for each metal type.

7.4.2.2 In all succeeding steps, handle the squares only with tongs or filter paper until the final weighing. If large defects or particles are present on the metal coupons, course sand paper should be used first to remove them; this is followed by polishing with the 240 and 400 grit abrasive paper.

(1) Store the polished metal coupon in tetrahydrofuran and proceed until all coupons are polished.

(2) Remove each square from the tetrahydrofuran, clean all metal dust from the square by rubbing vigorously with clean pads of absorbent cotton until a fresh pad remains unsoiled.

(3) Wash squares in tetrahydrofuran and allow them to dry in a dessicator.

(4) Immediately weigh each square to within 0.1 mg.

(5) Arrange all squares in the wooden assembly fixture in the pattern shown in Fig. 7. The sequence should be: lead, copper, tin, phosphor bronze.

7.4.2.3 Using only forceps to handle the clean wire, tie the squares together as shown in Fig. 7.

8. Procedure

8.1 *Preparation for New Test Set-Up for Reference Oil and Test Oils*:

8.1.1 Insert the tied coupons in the test tube, positioning the squares vertically (so that the air tube can be inserted to touch the bottom of the test tube). Weigh the air tube, test tube, and contents together to within 0.1 g.

8.1.2 Add 100 ± 1 mL of oil to the test tube, reweigh the air tube, test tubes, and contents together to within 0.1 g, and determine the weight of oil added.

8.1.3 Assemble the test tube and condenser and mount the assembly so that 30 ± 5 cm of test tube is submerged in the bath with the sample operating at a temperature of 121 ± 0.5°C.

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

8.1.5 Insert the air tube (orifice-end down) through the condenser and into the oil sample and support it so that its orifice is within 0.3 cm of the bottom of the tubes.

8.2 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.

8.3 *End of Test*—After 168 h at 121°C, shut off the air-flow, disassemble, and check test setup as follows:

8.3.1 Remove air supply and disconnect condenser.

8.3.2 Remove test tube from the constant-temperature bath, allow it to cool, and wipe off the outside of the tube with a naphtha-dampened cloth.

8.3.3 Re-weigh the air tube, test tube, and contents to within 0.1 g, determine weight of oil sample remaining, and compute the percentage of weight loss resulting from evaporation of oil (see 10.1). If the evaporation loss is greater than 8 %, leakage is present. Correct the leak, and repeat the determination, using fresh oil sample and new coupons.

8.3.4 Using forceps, withdraw the coupons from the test tube, and remove the wire holding them together. (Retain the test tube and sample for further examination).

8.4 *Preparing Squares for Examination*:

8.4.1 Using forceps, wash each square individually in tetrahydrofuran.

8.4.2 Repeat the washing, using fresh tetrahydrofuran, scrubbing the squares with the short-bristled brush until the tetrahydrofuran shows no additional discoloration. Use a piece of Kimwipe, dampened with acetone, to rub and wipe the coupons repeatedly until the tissue remains clean after wiping. Allow the squares to air-dry. The reaction products that are to be removed by this cleaning process may tend to have a stronger affinity to the bronze material, and therefore may be more difficult to remove from the bronze coupon. Improper removal of the reaction products from the coupons may result in inaccurate weight change measurements. With the bronze coupon, brushing may need to be more vigorous to remove the reaction products, but care must be taken not to scrub to the point where any of the coupon surfaces are being polished or abraded.

8.5 *Examining Each Square*:

8.5.1 Re-weigh each square to the nearest 0.1 mg and compute (in mg/cm^2 of surface) the change in weight of each square (see 10.2).

8.5.2 Rate both sides of the copper coupon according to Test Method D 130, and note the color of any stains present on the copper and bronze squares.

8.6 *Examining the New Oil and the Oil Sample in the Test Tube*:

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

9. Reference Oil Testing

9.1 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.

9.1.1 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.

9.1.1.1 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.

9.1.2 Request a reference oil assignment from the TMC for the CBT Test. The TMC shall determine the specific reference oil to be tested by the laboratory. Assignments shall be made by the TMC using the unique identifying codes on the reference oil container labels. Provide the TMC with the bath identification number for the test.

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

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

9.1.4.1 Use the data reporting formats detailed in Annex A2 (see Figs. A2.1 through 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.

9.1.4.2 Transmit reference test data by electronic means, or by telephone facsimile, to the TMC 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.

9.1.4.3 In addition to the transmitted data, send by mail or other courier one copy of the completed standard final reference test report to the TMC. The signatory line on the mailed Final Report Cover Sheet (Fig. A2.1) requires an original signature by an authorized representative of the testing laboratory. This signature is not to be a copy or mechanically reproduced. The signature affirms the statements made in the affidavit on the Final Report Cover Sheet. Mail the final test report so that it is received at the TMC within 30 days from the test completion date.

9.1.5 *Evaluation of Reference Test Oil Results*—Upon receipt of the transmitted TMC reference oil test results, the TMC shall review the test for operational adherence to the published procedure. If the test is found to be operationally valid, the reference oil results shall 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.

9.1.5.1 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. The validity findings are considered preliminary until the formally signed final report of the data is received and reviewed by the TMC. Discrepancies between the initial transmitted data and the mailed final report may result in the suspension or reversal of the preliminary validity decision.

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

9.1.6 *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.

10. Calculations

10.1 *Evaporation Loss*:

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

where:

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

10.2 *Change in Metal Square Weight*:

$$M = \frac{M_2 - M_1}{2 \times (W \times Le)} \quad (2)$$

where:

M = change in metal weight per surface area, mg/cm^2,
M_1 = final weight, mg,
M_2 = original weight, mg,
W = width of metal square, cm, and
Le = length of metal square, cm.

10.3 *Change in Metal Concentration in the Used Oil*:

$$C = C_2 - C_1 \quad (3)$$

where:

C = change in metal concentration before and after test,
C_1 = average of the duplicate measurement of metal concentration in new oil, and
C_2 = average of the duplicate measurement of metal concentration in used oil.

10.3.1 *Correction Factor*—Apply a correction factor of 0.276 to the lead coupon batches designated by the Central Parts Distributor (CPD) with a serial number ending in "-A" or –Cx" (where x denotes a number designating the coupon batch cut). Multiply non-reference test results for change in lead (C in 10.3) by this correction factor as follows:

$$C_{\text{Lead Corrected}} = C_{\text{Lead}} \times 0.276 \quad (4)$$

where:

$C_{\text{Lead Corrected}}$ = corrected change in lead concentration, ppm, and
C_{Lead} = change in lead concentration, before and after test, ppm, as determined in 10.3.

11. Report (See Annex A2 for Report Format)

11.1 Report the raw data of the calibration and the analysis of the NIST reference oil SRM1085a.

11.2 Report concentrations of copper, lead, and tin in oil before and after adjustment based on the internal standard, and the difference (C in 10.3).

11.2.1 Report the corrected change in lead concentration ($C_{\text{Lead Corrected}}$ in 10.3.1), and the applied correction factor (0.276), for non-reference oils.

11.3 Report the tarnish rating of the copper coupon based on the highest rating (most corrosion) if the rating is different for either side.

11.4 Report the change in weight of each of the coupons in mg/cm^2.