Designation: D4310 – 10 (Reapproved 2015)

Standard Test Method for Determination of Sludging and Corrosion Tendencies of Inhibited Mineral Oils[1]

This standard is issued under the fixed designation D4310; 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.

1. Scope

1.1 This test method covers and is used to evaluate the tendency of inhibited mineral oil based steam turbine lubricants and mineral oil based anti-wear hydraulic oils to corrode copper catalyst metal and to form sludge during oxidation in the presence of oxygen, water, and copper and iron metals at an elevated temperature. The test method is also used for testing circulating oils having a specific gravity less than that of water and containing rust and oxidation inhibitors.

NOTE 1—During round robin testing copper and iron in the oil, water and sludge phases were measured. However, the values for the total iron were found to be so low (that is, below 0.8 mg), that statistical analysis was inappropriate. The results of the cooperative test program are available (see Section 16).

1.2 This test method is a modification of Test Method D943 where the oxidation stability of the same kinds of oils is determined by following the acid number of oil. The number of test hours required for the oil to reach an acid number of 2.0 mg KOH/g is the *oxidation lifetime*.

1.3 Procedure A of this test method requires the determination and report of the weight of the sludge and the total amount of copper in the oil, water, and sludge phases. Procedure B requires the sludge determination only. The acid number determination is optional for both procedures.

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

1.5 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

1.6 *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.* For specific warning statements, see Section 7 and X1.1.5.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A510 Specification for General Requirements for Wire Rods and Coarse Round Wire, Carbon Steel
B1 Specification for Hard-Drawn Copper Wire
D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration
D874 Test Method for Sulfated Ash from Lubricating Oils and Additives
D943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils
D1193 Specification for Reagent Water
D3339 Test Method for Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
E1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *Energy Institute Standard:*[3]

Specification for IP Standard Thermometers

2.3 *British Standard:*[4]

BS 1829 Reference Tables for Iron v. Constantan Thermocouples

3. Terminology

3.1 *Definitions:*

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[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 D02.09.0C on Oxidation of Turbine Oils.

Current edition approved Oct. 1, 2015. Published December 2015. Originally approved in 1983. Last previous edition approved in 2010 as D4310 – 10. DOI: 10.1520/D4310-10R15.

[2] 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] Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., http://www.energyinst.org.

[4] Available from British Standards Institution (BSI), 389 Chiswick High Rd., London W4 4AL, U.K., http://www.bsigroup.com.

3.1.1 *sludge*—a precipitate or sediment from oxidized mineral oil and water.

4. Summary of Test Method

4.1 An oil sample is contacted with oxygen in the presence of water and an iron-copper catalyst at 95 °C for 1000 h. The weight of insoluble material is determined gravimetrically by filtration of the oxidation tube contents through 5 µm pore size filter disks. The total amount of copper in the oil, water, and sludge phases is also determined for Procedure A. Procedure B requires the sludge determination. The copper determination is not required. The acid number determination is optional for both procedures.

NOTE 2—Optionally, some operators may choose to: (*1*) assess the change in weight of the catalyst coil, or (*2*) determine the acid number at 1000 h, or both. The acid number may serve as a criterion to determine if measurement of insoluble material is warranted. Normally, further testing is not recommended on a highly oxidized oil (that is an oil which has attained an acid number >2.0 mg KOH/g). Instructions for these optional tests are not included in this test method.

5. Significance and Use

5.1 Insoluble material may form in oils that are subjected to oxidizing conditions.

5.2 Significant formation of oil insolubles or metal corrosion products, or both, during this test may indicate that the oil will form insolubles or corrode metals, or both, during field service. However, no correlation with field service has been established.

6. Apparatus

6.1 *Oxidation Cell,* of borosilicate glass, as shown in Fig. 1, consisting of a test tube, condenser, and oxygen delivery tube. The test tube has a calibration line at 300 mL (maximum error 1 mL). This calibration applies to the test tube without inserts at 20 °C.

6.2 *Heating Bath: Liquid Bath or Metal Block,* thermostatically controlled, capable of maintaining the oil sample in the oxidation cell at a temperature of 95 °C ± 0.2 °C, fitted with a suitable stirring device to provide a uniform temperature throughout the bath, and large enough to hold the desired number of oxidation cells immersed in the heating bath to a depth of 390 mm ± 10 mm and in the heating liquid itself to a depth of 355 mm ± 10 mm.

6.2.1 Studies have suggested that direct sunlight or artificial light may adversely influence the results of this test.[5] To minimize effects of light exposure on the lubricant being tested, light shall be excluded from the lubricant by one or more of the following ways:

6.2.1.1 Use of heated liquid baths that are designed and constructed of metal, or combinations of metals and other suitable opaque materials, that prevent light from entering the test cell from the sides is preferred. If a *viewing window* is included in the design, this *viewing window* shall be fitted with a suitable opaque cover and be kept closed when no observation is being made.

6.2.1.2 If glass heating baths are used, the bath shall be wrapped with aluminum foil or other opaque material.

6.2.1.3 Bright light entering the test cell from directly overhead can be eliminated by use of an opaque shield.

6.3 *Flowmeter,* with a flow capacity of at least 3 L of oxygen/hour, and an accuracy of ±0.1 L/h.

6.4 *Heating Bath Thermometer*—ASTM Solvents Distillation Thermometer having a range from 72 °C to 126°C and conforming to the requirements for Thermometer 40C as prescribed in Specification E1, or for Thermometer 70C as prescribed in Specifications for IP Standard Thermometers. Alternatively, temperature–measuring devices of equal or better accuracy may be used.

6.5 *Oxidation Cell Thermometer,* having a range from 80 °C to 100 °C, graduated in 0.1 °C, total length—250 mm, stem diameter—6.0 mm to 7.0 mm, calibrated for 76 mm immersion. Temperature measuring devices such as liquid-in-glass thermometers, thermocouples, or platinum resistance thermometers that provide equivalent or better accuracy and precision that cover the temperature range, may be used.

6.6 *Wire Coiling Mandrel,* as shown in Fig. 2.

6.7 *Thermometer Bracket,* for holding the oxidation cell thermometer, of 18-8 stainless steel, having the dimensions shown in Fig. 3. The thermometer is held in the bracket by two fluoro-elastomer O-rings of approximately 5 mm inside diameter. Alternatively, thin stainless steel wire may be used.

6.8 *Abrasive Cloth,* silicon carbide, 100-grit with cloth backing.

6.9 *Flexible Tubing,* poly vinyl chloride approximately 6.4 mm (¼ in.) inside diameter with a 3⁄32 in. wall for delivery of oxygen to the oxidation cell.

6.10 *Membrane Filters,* [6,7] white, plain, 47 mm or 90 mm in diameter, pore size 5 µm.

6.11 *Filter Holder,* [6,8] 47 mm or 90 mm, consisting of a borosilicate glass funnel and a funnel base with a coarse grade (40 µm to 60 µm) fritted-glass filter support or stainless steel screen support such that the filter can be clamped between the ground-glass sealing surfaces of the funnel and its base by means of a metal clamp.

6.12 *Weighing Bottle,* [6,9] cylindrical body with ground-glass stopper; approximate inside diameter 45 mm, height of body 65 mm, capacity 60 mL.

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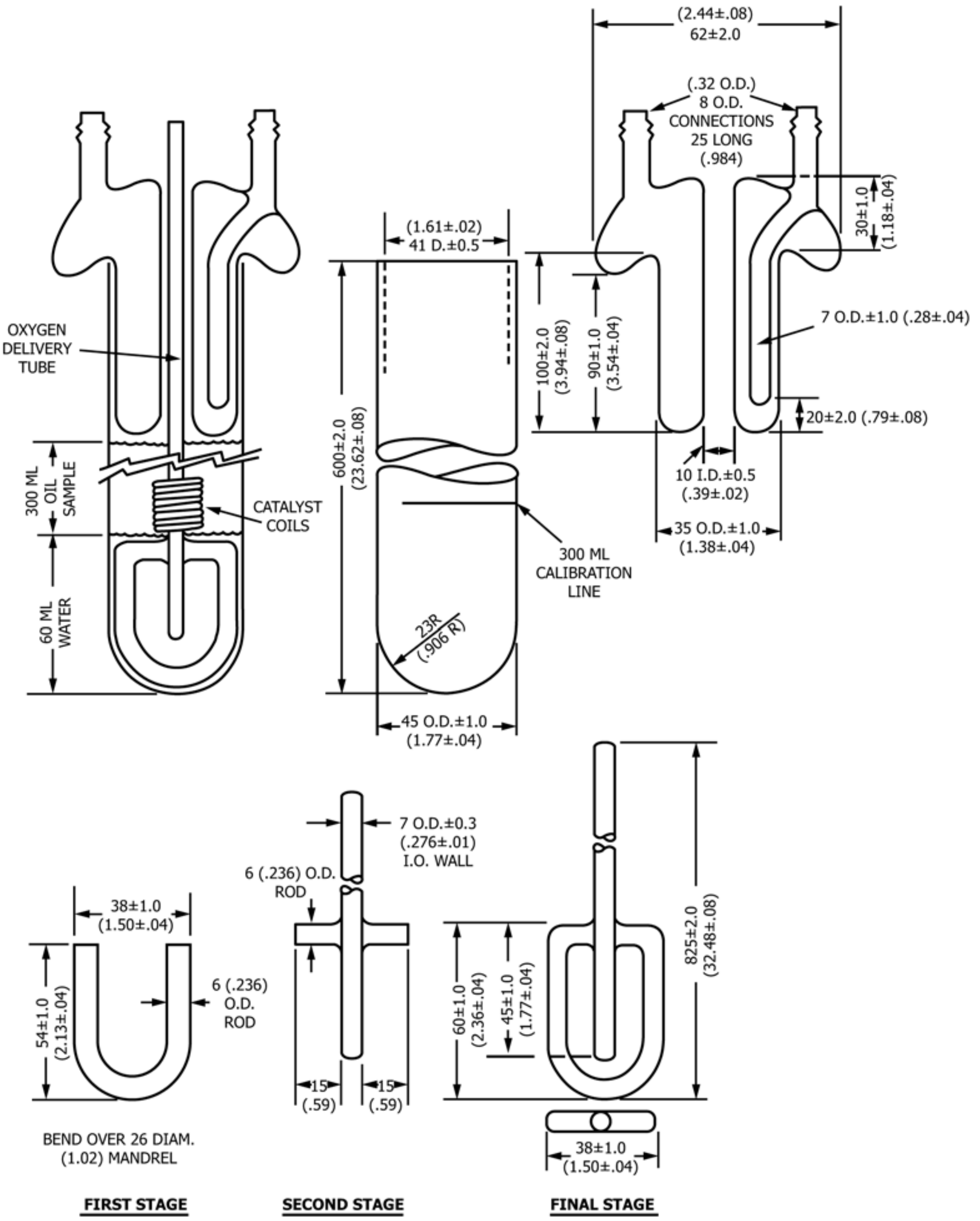
[5] Supporting data (summary of the results of these studies) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1365.

[6] 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.

[7] The sole source of supply of the Millipore SM membrane filters (MF-type, cellulose esters) known to the committee at this time is Millipore Filter Corp., Bedford, MA.

[8] The sole source of supply of the Millipore Pyrex XX-10-047-00 or XX-10-047-30 filter holder known to the committee at this time is Millipore Filter Corp., Bedford, MA.

[9] The sole source of supply of the Fisher 3-415 weighing bottle, size G, known to the committee at this time is Fisher Scientific Co., Pittsburgh, PA.



All dimensions are in millimetres (inches)

NOTE 1—The oxidation test tube has a calibration line at 300 mL. This calibration applies to the test tube alone at 20 °C.

NOTE 2—Open tube ends to be ground and fire-polished.

FIG. 1 Oxidation Cell

6.13 *Vacuum Source,* to provide pressure reduction to 13.3 kPa ± 0.7 kPa (100 mm ± 5 mm Hg) absolute pressure.

6.14 *Cooling Vessel*—A desiccator or other type of tightly covered vessel for cooling the weighing vessels before weighing. The use of a drying agent is not recommended.

6.15 *Drying Oven,* capable of maintaining a temperature of 105 °C ± 2 °C.

6.16 *Forceps,* having unserrated tips.

6.17 *Syringe,* 50 mL Luer-Lok with 12 in. needle.

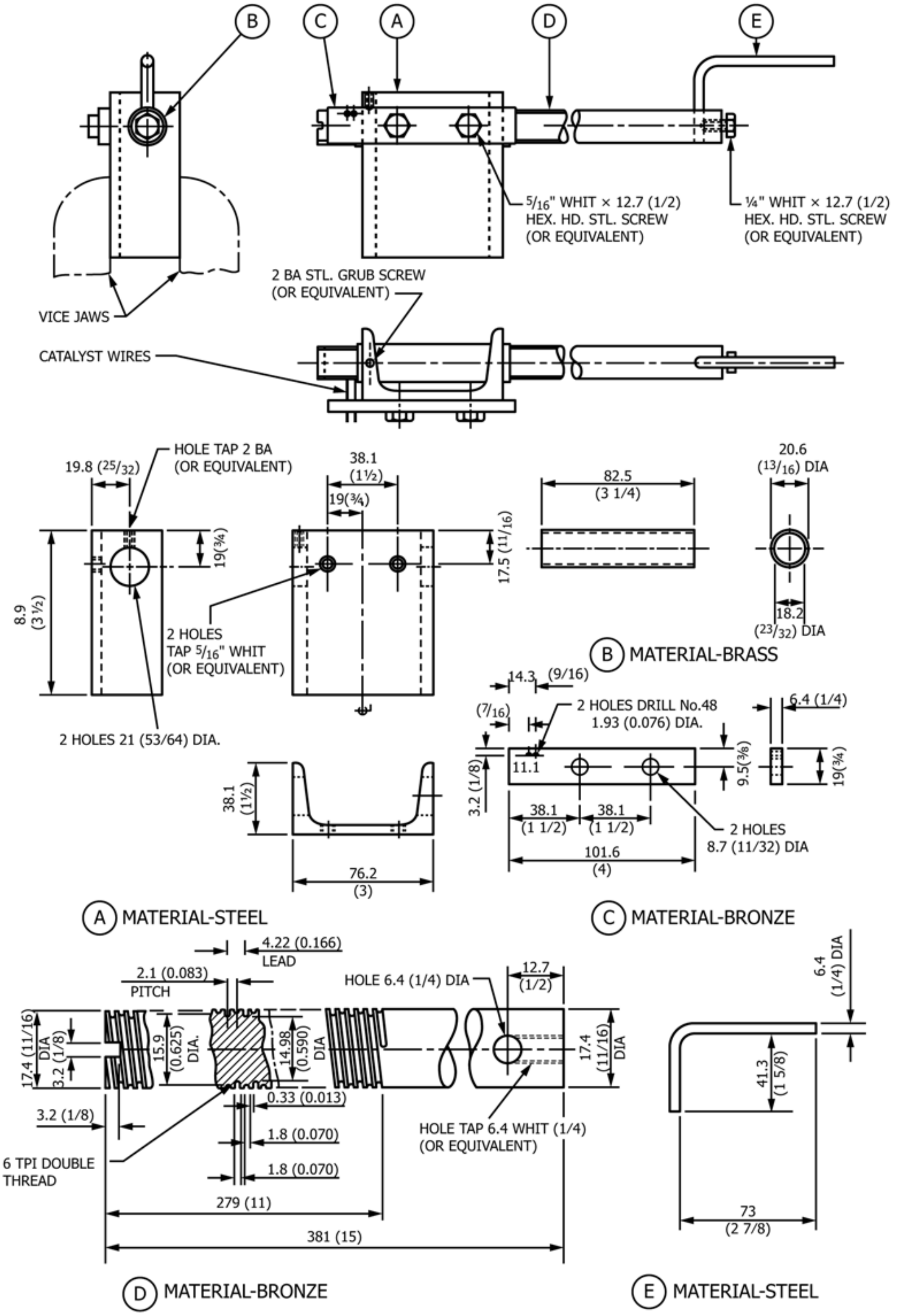
6.18 *Separatory Funnels,* with a capacity of 1000 mL.

6.19 *Rubber Policeman.*

6.20 *Pipette Bulb.*

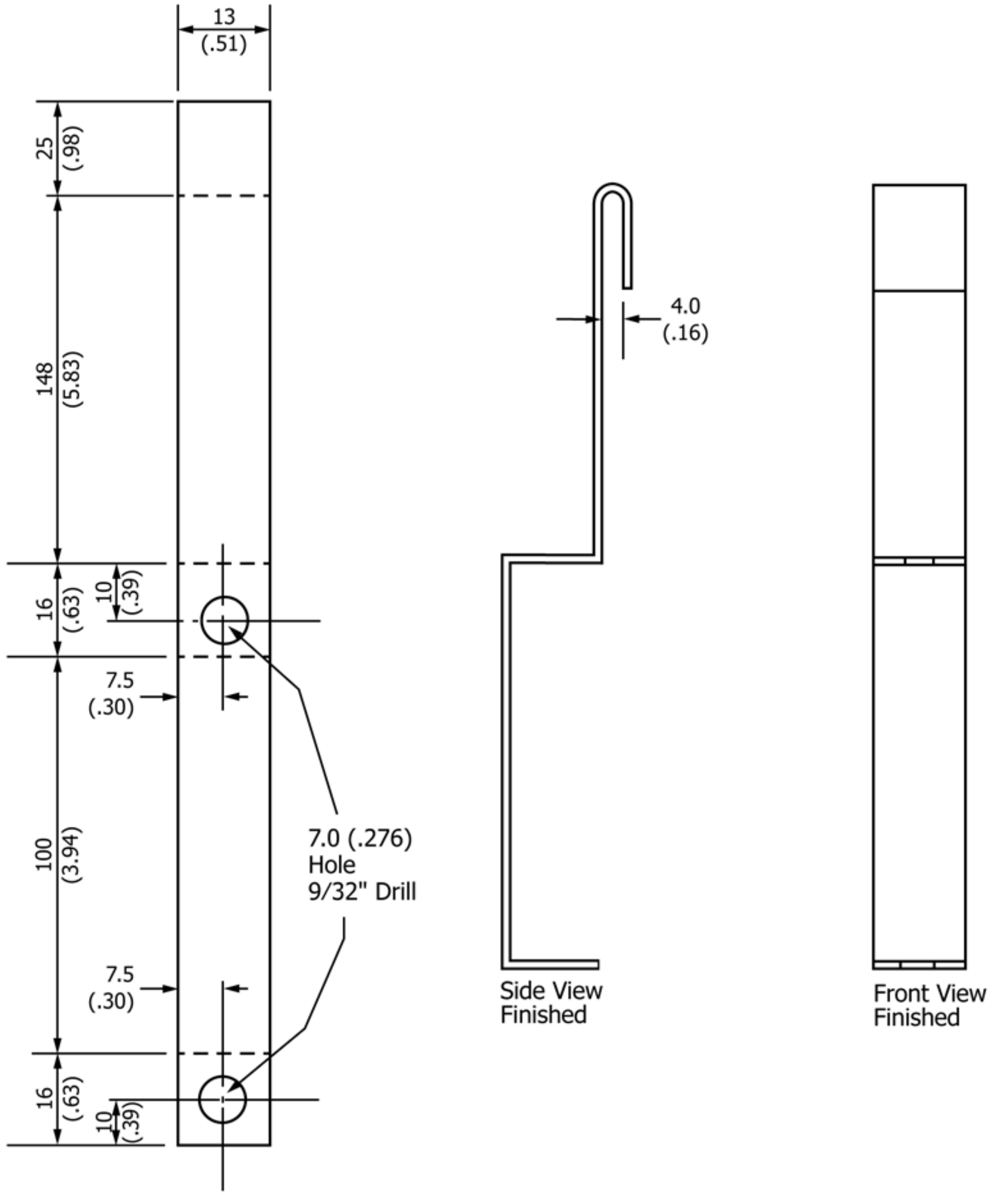
6.21 *Syringe,* glass or plastic, with Luer-Lok locking connector, 10 mL capacity for sampling.

6.22 *Syringe Sampling Tube,* Grade 304 stainless steel tubing, 2.11 mm (0.083 in.) outside diameter, 1.60 mm (0.063 in.) inside diameter, 559 mm ± 2 mm (24.0 in. ± 0.08 in.) long, with one end finished at 90° and the other end fitted with a Luer-Lok female connector.



Dimensions are in millimetres (inches).

FIG. 2 Mandrel for Winding Catalyst Coils



All dimensions are in millimetres (inches).
Material: 18-8 Stainless Steel. 22 Gauge (0.792 mm).

FIG. 3 Thermometer Bracket

7. Reagents and Materials

7.1 *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.[10] 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D1193.

7.3 *Acetone*—Reagent grade. (**Warning—**Health hazard, flammable.)

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[10] *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.

7.4 *Cleaning Reagent,* cleaning by a 24 h soak at room temperature in either Nochromix[6,11] (**Warning—**Corrosive, health hazard) or in Micro[6,12] solution.

7.5 *n-heptane,* Reagent grade. (**Warning—**Flammable. Harmful if inhaled.)

7.6 *Hydrochloric Acid* (**Warning—**Toxic and corrosive.), concentrated [(36 mass % (relative density 1.19)].

7.7 *Isopropyl Alcohol*—Reagent grade. (**Warning—**Flammable.)

7.8 *Catalyst Wires:*

7.8.1 *Low-Metalloid Steel Wire,* 1.59 mm (0.0625 in.) in diameter (No. 16 Washburn and Moen Gauge).

NOTE 3—Carbon steel wire, soft bright annealed and free from rust of Grade 1008 as described in Specification A510 is satisfactory. Similar wire conforming to BS 1829, is also satisfactory. If these steels are not available, other equivalent steels may be used, provided they are found to be satisfactory in comparative tests using this Test Method D4310.

7.8.2 *Electrolytic Copper Wire,* 1.63 mm (0.064 in.) in diameter (No. 16 Imperial Standard Wire Gauge or No. 14 American Wire Gauge), 99.9 % purity, conforming to Specification B1. Soft copper wire of an equivalent grade may also be used.

NOTE 4—Alternatively, suitably prepared catalyst coils may be purchased from a supplier.

7.9 *Detergent,* water-soluble.[6,13]

7.10 *Oxygen*—(**Warning—**Oxygen vigorously accelerates combustion) 99.5 % minimum purity, with pressure regulation adequate to maintain a constant flow of gas through the apparatus. The use of a two-stage pressure regulator on tank oxygen is recommended.

8. Sampling

8.1 Samples for this test can come from tanks, drums, small containers, or even operating equipment. Therefore, use the applicable apparatus and techniques described in Practice D4057.

8.2 For one single determination the minimum required sample size is 300 mL.

9. Preparation of Apparatus

9.1 *Cleaning Catalyst*—Immediately prior to winding a catalyst coil, clean a 3.00 m ± 0.01 m length of iron wire and an equal length of copper wire with wads of absorbent cotton wet with *n*-heptane and follow by abrasion with abrasive cloth until a fresh metal surface is exposed. Then wipe with dry absorbent cotton until all loose particles of metal and abrasive have been removed. In subsequent operations handle the catalyst wires with clean gloves (cotton, rubber, or plastic) to prevent contact with the skin.

9.2 *Preparation of Catalyst Coil*—Twist the iron and copper wires tightly together at one end for three turns and then wind them simultaneously alongside each other on a threaded mandrel (see Fig. 2), inserting the iron wire in the deeper thread. Remove the coil from the mandrel, twist the free ends of the iron and copper wires together for three turns, and bend the twisted ends to conform to the shape of the spiral coil. The overall length of the finished coil should be 225 mm ± 5 mm (8.9 in. ± 0.2 in.). If necessary, the coil may be stretched to give the required length (Note 4 and Note 5).

NOTE 5—The finished catalyst coil is a double spiral of copper and iron wire, 225 mm ± 5 mm (8.9 in. ± 0.2 in.) overall length and 15.9 mm to 16.5 mm (0.625 in. to 0.650 in.) inside diameter. The turns of wire are evenly spaced, and two consecutive turns of the same wire are 3.96 mm to 4.22 mm (0.156 in. to 0.166 in.) apart, center to center. The mandrel shown in Fig. 2 is designed to produce such a coil. Using this mandrel, the iron wire is wound on a thread of 14.98 mm (0.590 in.) diameter, while the copper wire is wound on a thread of 15.9 mm (0.625 in.) diameter. The smaller diameter is to allow for "springback" of the steel wire after winding, so as to give 15.9 mm consistent inside diameter. Use of a very soft annealed steel wire may allow use of identical thread diameters for the two wires. Any arrangement that leads to the coil configuration described above is satisfactory.

9.3 *Catalyst Storage*—The catalyst coil may be stored in a dry, inert atmosphere prior to use. A suitable procedure for catalyst storage is given in Appendix X1. Before use it should be inspected to assure that no corrosion products or contaminating materials are present. For overnight storage (less than 24 h) the coil may be stored in *n*-heptane.

9.3.1 *n*-heptane used for catalyst storage must be free of traces of water and corrosive materials. Redistilled *n*-heptane conforming to 7.5 and stored in a tightly sealed bottle is suitable.

9.4 *Cleaning New Glassware*—Wash new oxygen delivery tubes, condensers, and test tubes with a hot detergent solution and rinse thoroughly with tap water. Clean the interiors of the test tubes, exteriors of the condensers, and both interiors and exteriors of the oxygen delivery tubes with a cleaning reagent. Rinse thoroughly with tap water until all cleaning solution is removed. Rinse all parts with reagent water and allow to dry at room temperature or in an oven. The final reagent water rinse may be followed by an isopropyl alcohol rinse, or acetone rinse optionally followed by dry air blowing to hasten drying at room temperature.

9.5 *Cleaning Used Glassware*—Immediately following termination of a test, drain the oil completely from the test tube. Rinse all the glassware with *n*-heptane to remove traces of oil, wash with a hot detergent solution using a long-handled brush, and rinse thoroughly with tap water. If deposits still adhere to the glassware, a method that has been found useful is to fill the test tubes with detergent solution, insert the oxygen delivery tubes and condensers, and place the tubes in the bath at test temperature. Several hours soaking in this manner often serves to loosen all adhering deposits except iron oxide. Subsequent rinsing with hot (50 °C) hydrochloric acid will serve to remove iron oxide. After all deposits are removed, rinse all glassware with a cleaning reagent. Rinse thoroughly with tap water until all cleaning reagent is removed. Rinse all parts with reagent water and allow to dry at room temperature or in an oven. The

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[11] The sole source of supply of Nochromix known to the committee at this time is Godax Laboratories, Inc., 720-B Erie Avenue, Takoma Park, MD 20912.

[12] The sole source of supply of Micro known to the committee at this time is International Products Corp., P.O. Box 70, Burlington, NJ 08016.

[13] Alconox has been found satisfactory for this purpose.

final reagent water rinse may be followed by an isopropyl alcohol rinse, or acetone rinse optionally followed by dry air blowing, to hasten drying at room temperature. Store glassware in a dry dust-free condition until ready to use.

10. Procedure for Oxidizing the Oil

10.1 Adjust the heating bath to a temperature high enough to maintain the oil in the oxidation test cell at the required temperature of 95 °C ± 0.2 °C.

10.2 Fill the empty oxidation test tube with 300 mL of the oil sample to the graduation mark. Slide the catalyst coil over the inlet of the oxygen delivery tube. If the wires are uneven at one end of the coil, position the coil so that this end is down. Place the oxygen delivery tube with the coil into the test tube. Place the condenser over the oxygen delivery tube and test tube. Immerse the test tube in the heating bath. Adjust the heating bath liquid level so that the tube is immersed in the liquid to a depth of 355 mm ± 10 mm. Connect the condenser to the cooling water. The temperature of the outlet water should not exceed 32 °C at any time during the test.

10.3 Connect the oxygen delivery tube to the oxygen supply (see 7.10) through the flowmeter using new poly vinyl chloride flexible tubing no more than 600 mm in length. Before using, the interior of the new tubing should be rinsed with *n*-heptane and blown dry with air. Adjust the rate of flow to 3 L ± 0.1 L and continue flow for 30 min.

10.4 Raise the condenser unit from the oxidation cell and add 60 mL of reagent water through the opening thus provided. The test is considered to start at this point.

10.5 Throughout the duration of the test, maintain the temperature of the oil-water mixture (sample temperature) at 95 °C ± 0.2 °C in each test cell with oxygen flowing. Accomplish this by maintaining the bath at the temperature that is found necessary to give the required 95 °C sample temperature. The temperature of the bath is always higher than the sample temperature due to the cooling effect of the oxygen gas flow, and depends on heating bath medium, capacity, circulation, and on the number of tests cells in the bath. Measure the sample temperature by a temperature measuring device positioned in the oxidation cell by a temperature measuring device bracket, as in Fig. 4 (see Note 6). Make temperature measurements only with new oil samples, and preferably with dummy cells used specifically for temperature measurement. When an actual test sample is used, remove the temperature measuring device immediately after temperature measurement is complete. Check the temperature in this way in various parts of a multiple-cell bath to verify uniformity of temperature control. Once the required bath temperature is found, maintain at that temperature ±0.2 °C.

NOTE 6—With the arrangement shown in Fig. 4, the 76 mm immersion point of the temperature measuring device is positioned at the oil surface. To allow for heating of the stem portion of the temperature measuring device above the immersion point in the upper portion of the test cell, subtract 0.10 °C from the temperature measuring device reading to obtain the true test temperature.

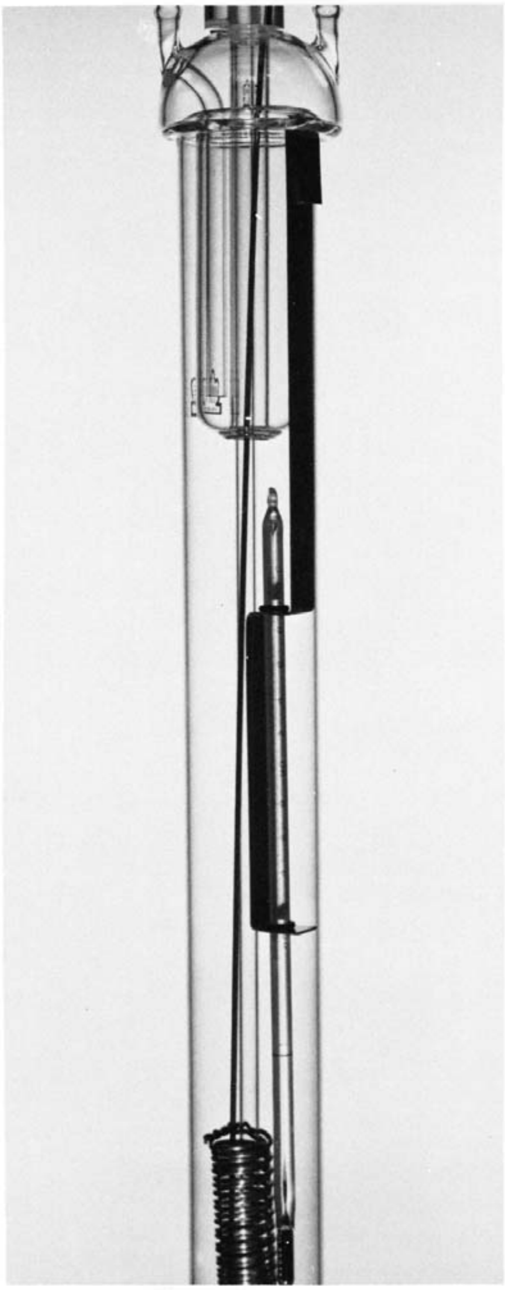
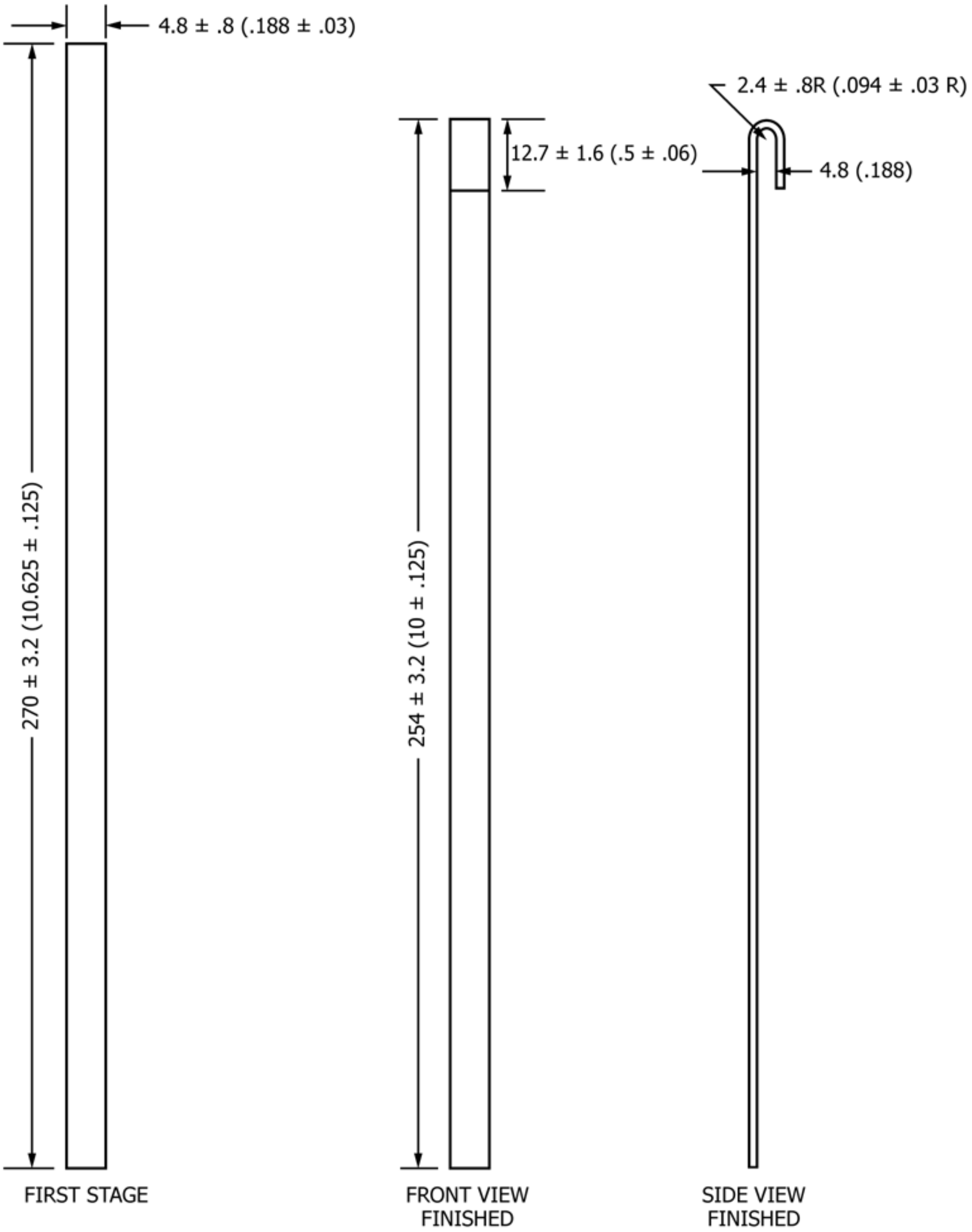


FIG. 4 Oxidation Cell with Temperature Measuring Device

10.6 Add additional reagent water to the oxidation cell as required (see Note 7), at least every 2 weeks during the test, to restore the water level to the shoulder of the oxygen delivery tube. Add the water using the sampling tube and the 50 mL capacity syringe.

NOTE 7—Under some circumstances, the level of water cannot be observed because of deposits or emulsion formation. Marking the upper oil level of the filled oxidation tube by some suitable means and maintaining this level by periodic water additions will keep the proper amount of water in the cell. The correct level for water additions may, if desired, be indicated by a movable metal strip (see Fig. 5) that is clamped to the outside of the oxidation test tube by, for example, an adjustable ring-type hose clamp. To use this indicator, the lower end of the strip is set at the upper oil level when the test is started. As the test proceeds and water evaporates to cause the oil level to fall, sufficient makeup water is added, particularly just before oil samples are taken, to return the oil level to the level marked by the indicator strip.



NOTE 1—All dimensions are in millimetres (inches). Material: Type 304 Stainless Steel. 22 Gauge (0.792 mm).

FIG. 5 Oil Level Indicator Strip

11. Procedure for Handling End of Test Oil

11.1 Upon completion of 1000 h of test time, remove the oxidation apparatus from the heating bath and remove the condenser.

11.2 If an end of test acid number measurement is required, remove the sample as follows:

11.2.1 While the oxidation apparatus and the oil are still hot, raise and support the oxygen delivery tube together with the catalyst coil just clear of the oil in the oxidation tube and allow to drain for 5 min to 10 min. Lower the oxygen delivery tube together with the catalyst coil so that the end of the oxygen delivery tube is about in the middle of the oil layer. Raise and support the oxygen delivery tube together with the catalyst coil just clear of the oil in the oxidation tube and allow to drain for about 5 min. Carefully lift the oxygen delivery tube out of the oxidation tube and using a pipette bulb, remove a 3 mL aliquot of sample into an appropriate vial. Quickly replace the delivery tube over the oil to continue draining for an additional 25 min to 30 min.

11.2.2 If using a syringe sampling tube to remove a specimen for acid number measurement, insert the sampling tube down through the center hole in the condenser and submerge to approximately the middle of the oil layer. Withdraw 6 mL of oil into the syringe then let the sampling tube rest on the bottom of the oxidation tube for 5 min to allow water to settle to the bottom of the syringe. At the end of 5 min, adjust the sample volume to 3 mL and remove the sampling tube from the oxidation cell. This method allows most of the water withdrawn with the test oil to be returned to the test cell. The 3 mL sample is dispensed into a sample vial for acid number analysis by Test Methods D664 or D3339. Shake the test oil sample thoroughly before taking a sample from the vial for titration.

11.3 If an end of test acid number measurement is not required, raise and support the oxygen delivery tube together with the catalyst coil just clear of the oil in the oxidation tube and allow to drain about 30 min. Suspend the oxygen delivery tube/catalyst coil assembly over a 1000 mL beaker. Pour the contents of the oxidation tube into the beaker. Place a temperature measuring device in the beaker and wait for the sample temperature to drop to 50 °C (122 °F) before proceeding further. Use 250 mL of *n*-heptane from a wash bottle in portions to rinse the catalyst coil and walls of the oxidation tube into the beaker. Care should be taken to remove all traces of oil from the coil. Continue rinsing the oxidation tube with 100 mL of water from a wash bottle. Add the water washing to the same beaker containing the heptane washings (see Note 8). Briefly stir the oil-heptane-water mixture, cover with a watch glass, and allow to stand away from light for a period of 16 h to 20 h (see Note 9).

NOTE 8—Occasionally solid material may adhere to the walls of the oxidation tube, the catalyst coil, the oxygen tube, or condenser and resist displacement by heptane or water. This material is recovered by manual scraping using a rubber policeman and heptane washes. The additional material and heptane washes are added to the oil-heptane water mixture.

NOTE 9—The purpose of the 16 h to 20 h waiting period is to allow sufficient time for equilibration of insoluble material with the oil-heptane and water phases. This procedure also improves the ease of filtration of the sludge by allowing time for coalescence of the material into a more filterable form.

12. Procedure for Determination of Sludge Weight

12.1 Prior to filtering the oil-water mixture, weigh two filter membranes to the nearest mg in weighing vessels (A_1 mg and B_1 mg) (see Note 10, Note 11). Mount two filter holders on 1000 mL filter flasks. Assemble the two filter holders with the two membranes. Handle the membranes only with forceps having unserrated tips. Apply vacuum 13.3 kPa ± 0.7 kPa (100 mm ± 5 mm Hg) absolute pressure and carefully decant approximately equal portions of the oil-heptane layer into the two filter funnels without adding any of the water layer (Note 12). After the oil layer is filtered through, rinse the filter funnels with *n*-heptane, allow air to pass through the filter briefly, and begin addition of the water layer to the two filters. After the contents of the beaker have been divided approximately equally between the two filter funnels, thoroughly rinse the walls of the beaker and of the funnel with portions of water and then with portions of *n*-heptane. A rubber policeman may be used to scrape the walls of the beaker. Do not use less than 50 mL of water and 250 mL of *n*-heptane for each filter in this first rinsing procedure. Then, in a second rinsing operation, rinse each filter with an additional 100 mL of *n*-heptane. The final rinses of *n*-heptane from this second operation should be completely colorless after passing through the filters.

NOTE 10—Weighing bottles, watchglasses (one as receptacle, one as a cover), glass petri dishes or aluminum foil dishes have been used for this purpose.

NOTE 11—More than two filter membranes may be used if a large amount of sludge is present.

NOTE 12—Filtration of the oil-heptane layer will proceed at an optimum rate if no water is introduced into the filter during the filtration. A suitable technique employs the use of a separatory funnel to separate the water from the oil layer before beginning the filtration.

Occasionally, because of the nature of the deposits, filtration proceeds at a very slow rate despite precautions in avoiding simultaneous filtration of oil and water layers. In such cases prolonged (overnight) filtration times may be considered. However, unless the filtration is being directly attended, filtration should be stopped, that is, filter equipment brought to atmospheric pressure. Then leave solvent on the filter and cover the filter holder with a tight cover until the filtration at specified vacuum conditions is resumed the next day.

NOTE 13—The use of microwave-digestion method is an acceptable alternative to ashing.

12.2 With the vacuum applied, remove the clamp and funnel from the filter membrane and funnel base. Rinse the surface of the membrane with a gentle stream of *n*-heptane, directing the stream from the edge towards the center so as to remove final traces of oil from the membrane. Maintain the vacuum for a short time to remove final traces of *n*-heptane. Transfer the membranes to the identical weighing vessels used in the initial weighing and dry for at least 1 h in the oven at 105 °C. Allow the weighing vessels to cool in the cooling vessel in the vicinity of the balance for at least 2 h. Weigh the filters (in the weighing vessels) to the nearest mg. Return the weighing vessels with the filter membranes to the oven and dry, cool, and reweigh. When the difference in the weight of the insoluble material before and after successive drying/weighing operations is less than either 2 mg or 5 %, report the last weighing as the final weight (A_2 mg and B_2 mg).

13. Procedure for Determination of Copper in Oil, Water, and Sludge for Procedure A

13.1 *Preparation of Oil and Water Layers for Copper Determination*—After completion of the filtration proceedings (see 12.2), transfer the oil-heptane and the water filtrates from the two 1000 mL filter flasks to 1000 mL separatory funnels. Separate the oil-heptane and water layers. Weigh two beakers of the proper size to the nearest gram. Weigh into one of the beakers the total amount of oil-heptane mixture from the separatory funnel to the nearest gram ($W_{O\text{-}H}$ g) and weigh into the second beaker the total amount of water from the separatory funnel to the nearest gram (W_w g).

13.2 *Analysis for Copper:*

13.2.1 *Direct Method (homogeneous sample)*— Determine the copper content on the oil-heptane mixture and on the water solution according to any suitable method, such as atomic absorption (AA), direct current plasma (DCP), inductively coupled plasma (ICP) or X-ray fluorescence (XRF) ($P_{1O\text{-}H}$ % or $P_{2O\text{-}H}$ ppm; P_{1W} % or P_{2W} ppm). If the results are below 1000 ppm, report in ppm; if the results are 0.100 % or more, report in percent.

13.2.2 *Ash Method (non-homogeneous sample)*—For the insoluble material (sludge) and in case the oil-heptane mixture and the water solution are not homogeneous, use the ash method. After the last weighing of the filters (see 12.2), ash the two filters with the insoluble material using the sulfated ash procedure of Test Method D874.

13.2.3 If the ash method is used for the oil-heptane mixture or the water solution, evaporate the solvent or the water and ash the residue using the sulfated ash procedure of Test Method D874. Weighing and calculating the ash content are not necessary. Dissolve the ash by washing down the walls of the vessel with 5 mL concentrated hydrochloric acid. Digest on a steam bath for 15 min to effect the solution of all copper present. Cool the samples to room temperature and transfer the acid solution to a 50 mL volumetric flask and dilute to volume with distilled water. Determine the copper content on the water solution according to any suitable method, such as AA, DCP, ICP or XRF. (P_{3I} %, P_{4I} ppm; $P_{3O\text{-}H}$ %, $P_{4O\text{-}H}$ ppm; P_{3W} %, P_{4W} ppm).

14. Calculations

14.1 Weight of insoluble material, in milligrams:

$$I = \left(A_2 - A_1\right) + \left(B_2 - B_1\right) \text{ mg} \tag{1}$$

where:

I = insoluble material, mg,

A_1, B_1 = initial weight of filter membrane plus weighing bottle or watch glass, mg, and

A_2, B_2 = final weight of filter membrane plus weighing bottle or watch glass, mg.

14.2 *For Procedure A*—Weight of copper in oil, water, and insoluble material in milligrams.

14.2.1 *Direct Method:*

$$W_{cu \text{ in oil}} = 10\, W_{O-H} P_{1O-H} \text{ mg or} \tag{2}$$

$$W_{cu \text{ in oil}} = W_{O-H} P_{2O-H}/1000 \text{ mg} \tag{3}$$

$$W_{cu \text{ in water}} = 10\, W_W P_{1W} \text{ mg or} \tag{4}$$

$$W_{cu \text{ in water}} = W_W P_{2W}/1000 \text{ mg} \tag{5}$$

where:

$W_{cu \text{ in oil}}$ = weight of copper in oil, mg,

$W_{\text{O-H}}$ = weight of oil-heptane mixture, g,

$P_{1O\text{-}H}$ = copper content of oil-heptane mixture, % mass,

$P_{2O\text{-}H}$ = copper content of oil-heptane mixture, ppm,

$W_{cu \text{ in water}}$ = weight of copper in water, mg,

W_W = weight of water solution, g,

P_{1W} = copper content of water solution, % mass, and

P_{2W} = copper content of water solution, ppm.

14.2.2 *Ash Method:*

$$W_{cu \text{ in Ins}} = 500\, P_{3I} \text{ mg or} \tag{6}$$

$$W_{cu \text{ in Ins}} = 0.05\, P_{4I} \text{ mg} \tag{7}$$

$$W_{cu \text{ in oil}} = 500\, P_{3O-H} \text{ mg or} \tag{8}$$

$$W_{cu \text{ in oil}} = 0.05\, P_{4O-H} \text{ mg} \tag{9}$$

$$W_{cu \text{ in water}} = 500\, P_{3W} \text{ mg or} \tag{10}$$

$$W_{cu \text{ in water}} = 0.05\, P_{4W} \text{ mg} \tag{11}$$

where:

$W_{cu \text{ in Ins}}$ = weight of copper in insoluble material, mg

14.2.2.1 P is the copper content of water in the 50 mL flask derived from:

$$P_{3I} = \text{insoluble material, \% mass,} \tag{12}$$

$$P_{4I} = \text{insoluble material, ppm,} \tag{13}$$

$$P_{3O-H} = \text{oil} - \text{heptane mixture, \% mass,} \tag{14}$$

$$P_{4O-H} = \text{oil} - \text{heptane mixture, ppm,} \tag{15}$$

$$P_{3W} = \text{water solution, \% mass, and} \tag{16}$$

$$P_{4W} = \text{water solution, ppm.} \tag{17}$$

14.2.3 *Total Copper:*

$$W_{cu \text{ total}} = W_{cu \text{ in oil}} + W_{cu \text{ in water}} + W_{cu \text{ in Ins}} \text{ mg} \tag{18}$$

15. Report

15.1 Report the test method number and the procedure used (Procedure A or B).

15.2 *For Procedure A:*

15.2.1 Weight of insoluble material in milligrams, and

15.2.2 Weight of total copper in oil, water, and insolubles in milligrams.

15.2.3 When a rating of the corrosion of the catalyst metals is desired, the system as described in Appendix X2 can be used.

15.2.4 Acid number at end of test in mg KOH/g and test method used for the determination (optional).

15.3 *For Procedure B:*

15.3.1 Weight of insoluble material in milligrams.

15.3.2 Acid number at end of test in mg KOH/g and test method used for the determination (optional).

16. Precision and Bias[14]

16.1 *Precision*—The precision of the test method for the weight of insoluble material (sludge) and for the weight of total copper in oil, water, and insoluble material (sludge) as obtained by the technical examination of interlaboratory test results is as follows:

16.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials, would, in the long run and in the normal and correct operation of the test method, exceed the following values only in 1 case in 20:

weight of insoluble material (sludge): $4.6X^{2/3}$

weight of total copper: $1.2X^{4/5}$

where: X denotes the mean value.

16.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would, in the long run and in the normal and current operation of the test method, exceed the following values only 1 case in 20:

weight of insoluble material (sludge): $6.3\,X^{2/3}$

weight of total copper: $3.3\,X^{4/5}$

where: X denotes the mean value.

16.2 This precision statement was prepared with data on four new (unused) mineral oil-based steam turbine lubricants

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[14] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1528.

and four new (unused) mineral oil based anti-wear hydraulic oils. These oils gave the following range of results:

16.2.1 *Insoluble Material (Sludge)*—4.6 mg to 250 mg.

16.2.2 *Total Copper*—0.9 mg to 390 mg.

16.3 *Bias*—The procedure in Test Method D4310 has no bias, because the values of weight of insoluble material and weight of total copper in oil, water, and insolubles are defined only in terms of this test method.

17. Keywords

17.1 antiwear hydraulic oils; circulating oils; copper; corrosion; hydraulic oils; inhibited mineral oils; insoluble material; metal catalysts; oxidation; sludge; steam turbine lubricants; turbine oils

APPENDIXES

(Nonmandatory Information)

X1. PROCEDURE FOR PACKAGING CATALYST COILS

X1.1 Materials

X1.1.1 *Test Tubes,* borosilicate glass, 250 mm length, 25 mm outside diameter, approximately 22 mm inside diameter.

X1.1.2 *Caps,* for test tubes, polyethylene cylindrical shape designed to closely grip outside surface of test tube.

X1.1.3 *Desiccant Bags,* silica gel granules.

X1.1.4 *Flushing Tube,* stainless steel or glass, approximately 5 mm (3⁄16 in.) outside diameter, 305 mm (12 in.) long, to deliver nitrogen to bottom of test tube.

X1.1.5 *Nitrogen Gas,* 99.7 % minimum purity. (**Warning—**Compressed gas under high pressure. Gas reduces oxygen available for breathing.)

X1.2 Procedure

X1.2.1 Flush a new test tube with nitrogen gas, using the flushing tube, to blow out any loose particles. The tube must be visibly clean and dry. Hold the tube on an angle and gently slide the catalyst coil into the tube. Add a desiccant bag that has been folded lengthwise to fit in the tube. Insert the nitrogen flushing tube down the middle of the test tube, to the bottom, and blow nitrogen through the tube for several seconds. Immediately after withdrawing the flushing tube, seal the test tube with a polyethylene cap.

X2. OPTIONAL CORROSION INDICATOR

X2.1 Some investigators may desire to rate the catalyst coils as a qualitative indication of the corrosion properties of the oil. Iron and copper wire appearance tables (Table X2.1, Table X2.2) are appended here for information only.

X2.1.1 Only the outside surface of the coil shall be rated.

TABLE X2.1 Appearance of Iron Wire

Rating	Description
Bright	Freshly polished appearance
Tarnished	No red-brown rust specks of corrosion, but overall discoloration
Light rust	Rusting confined to not more than six spots
Moderate rust	Rusting confined to 7 to 12 spots
Severe rust	Rusting with more than 13 spots
Coated	Deposits on the iron wire prevent an estimate of the surface condition

TABLE X2.2 Appearance of Copper Wire

Rating	Description
Bright	Freshly polished appearance
Tarnished	Moderate discoloration
Green	Distinct green color
Black	Very dark color
Coated	Deposits on the copper wire prevent an estimate of the surface condition

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