Designation: D7873 − 20

Standard Test Method for Determination of Oxidation Stability and Insolubles Formation of Inhibited Turbine Oils at 120 °C Without the Inclusion of Water (Dry TOST Method)[1]

This standard is issued under the fixed designation D7873; 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 is used to evaluate the sludging tendencies of steam and gas turbine lubricants during the oxidation process in the presence of oxygen and metal catalyst (copper and iron) at an elevated temperature. This test method may be used to evaluate industrial oils (for example, circulating oils and so forth).

1.2 This test method is a modification of Test Method D4310 where the sludging and corrosion tendencies of the same kinds of oils are determined after 1000 h at 95 °C in the presence of water. Water is omitted in this modification.

1.3 The values stated in SI units are to be regarded as standard.

1.3.1 *Exception*—The values in parentheses in some of the figures are provided for information only for those using old equipment based on non-SI units.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous substance that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Use Caution when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A510M Specification for General Requirements for Wire Rods and Coarse Round Wire, Carbon Steel (Metric) (Withdrawn 2011)[3]
B1 Specification for Hard-Drawn Copper Wire
D943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils
D1193 Specification for Reagent Water
D2272 Test Method for Oxidation Stability of Steam Turbine Oils by Rotating Pressure Vessel
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4310 Test Method for Determination of Sludging and Corrosion Tendencies of Inhibited Mineral Oils
E1 Specification for ASTM Liquid-in-Glass Thermometers
E230/E230M Specification for Temperature-Electromotive Force (emf) Tables for Standardized Thermocouples

2.2 *Other Standards:*

Specification for IP Standard Thermometers[4]
ISO 3696 Water for Analytical Laboratory Use—Specification and Test Methods[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *sludge, n*—a precipitate or sediment from oxidized mineral oil that is insoluble in n-heptane.

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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.

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

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

[5] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

4. Summary of Test Method

4.1 A total of six to eight tubes containing 360 mL of sample each are heated at 120 °C with oxygen in the presence of an iron-copper catalyst. Each tube is removed over time and the sample is analyzed by Test Method D2272 and the insolubles are measured until the RPVOT residual ratio reaches below 25 % or an agreed-upon percentage or specified time. Test run for a specified time(s) may be run using a single tube or as many as specified by the requestor. The mass of insoluble material of each oil sample is determined gravimetrically by filtration of a 100 g oil sample through a membrane filter with pore size 1 µm. The insoluble mass (mg/kg oil) is plotted against RPVOT residual ratio. The insoluble mass in milligrams per kilogram oil at 25 % or an agreed-upon RPVOT residual ratio or specified time is reported.

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, resulting in varnish formation during field service. The level of varnish formation in service will be dependent on many factors (turbine design, reservoir temperature, duty-cycle, for example. peaking, cycling, or base-load duty, maintenance, and so forth) and a direct correlation between results in this test and field varnish formation are yet to be established.

5.3 Oxidation condition at 120 °C under accelerated oxidation environment of Test Method D4310 and measurement of sludge and RPVOT value could reflect a practical oil quality in actual turbine operations. Results from this test should be used together with other key lubricant performance indicators (including other established oxidation and corrosion tests) to indicate suitability for service.

6. Apparatus

6.1 *Oxidation Cell,* of borosilicate glass, as shown in Fig. 1, consisting of a test tube, condenser, and oxygen delivery tube. It is recommended to have a test tube with a calibration line at 360 mL (maximum error 1 mL). This calibration applies to the test tube without inserts at 20 °C.

6.2 *Heating Bath,* thermostatically controlled, capable of maintaining the oil sample in the oxidation cell at a temperature of 120 °C ± 0.5 °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 355 mm ± 10 mm. Heated metal block baths meeting the test method requirements may also be used.

6.2.1 Studies have suggested that direct sunlight or artificial light may adversely influence the results of this test. 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 98 °C to 152 °C and conforming to the requirements for Thermometer 41C as prescribed in Specification E1, or for Thermometer 81C as prescribed in Specifications for IP Standard Thermometers. Alternatively, temperature-measuring devices of equal or better accuracy and precision may be used.

6.5 *Oxidation Cell Thermometer,* A 76 mm immersion LIG having a range of 110 °C to 130 °C, graduated in 0.1 °C, total length of 300 mm ± 5 mm, and stem diameter of 6.0 mm to 7.0 mm. Alternatively, temperature-measuring devices or DCT, of equal or better accuracy and precision may be used. Temperature of the sample shall be measured at 76 mm from the top of the sample. See Fig. 2 and Fig. 3.

NOTE 1—Temperature gradient within the sample may exist from the heating system and temperature control design.

6.6 *Wire Coiling Mandrel,* as shown in Fig. 4.

6.7 *Thermometer Bracket,* for holding the oxidation cell thermometer, of 18-8 stainless steel, having the dimensions shown in Fig. 5. 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 2.4 mm (3⁄32 in.) wall for delivery of oxygen to the oxidation cell.

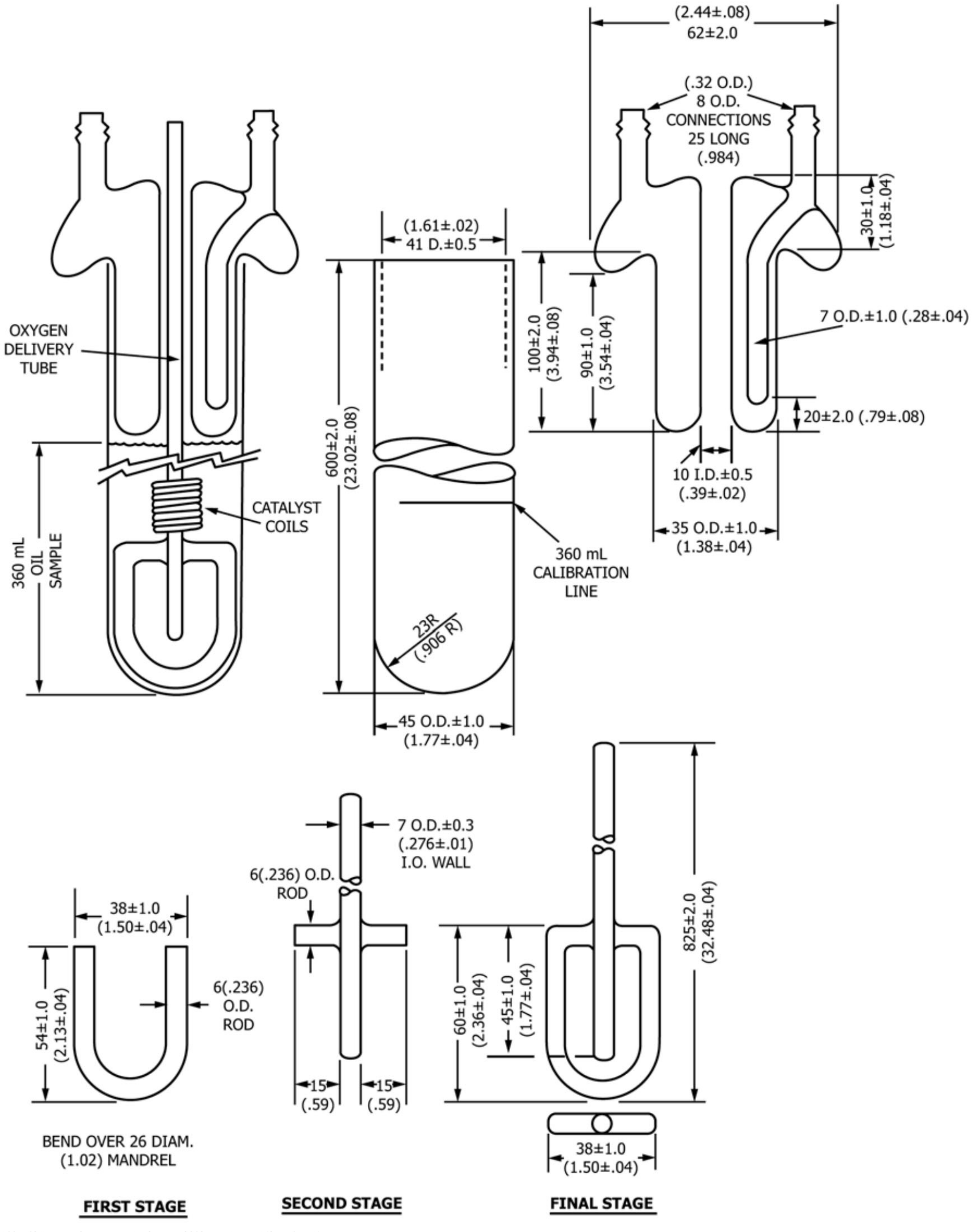
6.10 *Membrane Filters,* white, plain, 47 mm in diameter, pore size 1 µm. The recommended membrane filters are PTFE and cellulose acetate plus nitrocellulose material.

6.11 *Filter Holder,* 47 mm, consisting of a borosilicate glass funnel and a funnel base with a coarse grade fritted-glass filter support with a length of 40 µm to 60 µm, 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,* cylindrical body with ground-glass stopper; approximate inside diameter 65 mm, height of body 45 mm, capacity 60 mL.

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.



NOTE 1—All dimensions are in millimetres (inches).

NOTE 2—The oxidation test tube has a calibration line at 360 mL. This calibration applies to the test tube alone at 20 °C.

NOTE 3—Open tube ends to be ground and fire-polished.

FIG. 1 Oxidation Cell

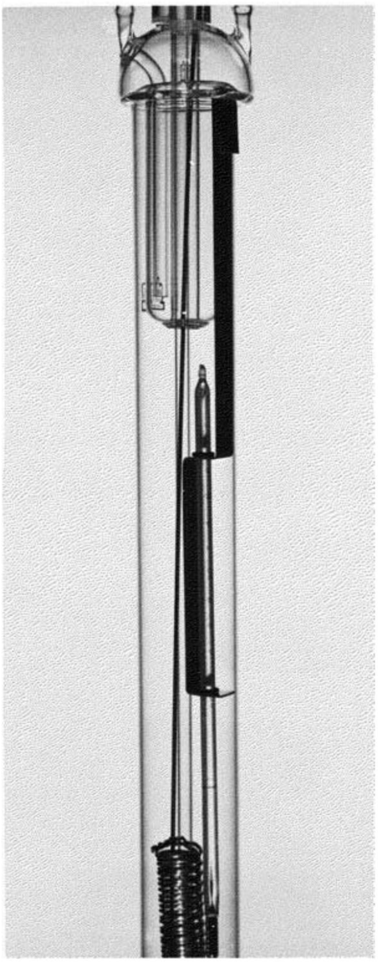


FIG. 2 Oxidation Cell with Thermometer

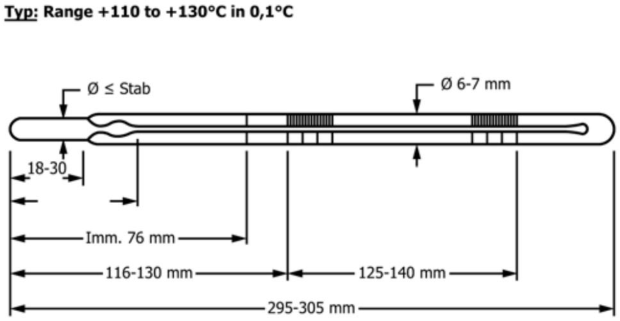


FIG. 3 76 mm Immersion LIG Thermometer

6.15 *Drying Oven,* capable of maintaining a temperature of 70 °C ± 5 °C.

6.16 *Forceps,* having unserrated tips.

6.17 *Rubber Policeman.*

6.18 *Pipette Bulb.*

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 shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6]

7.2 *Reagent Water,* Unless otherwise indicated, reference to water shall be understood to mean distilled, deionized water as defined by Type I or Type II in Specification D1193 or Grade 3 in ISO 3696.

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

7.4 *Cleaning Reagent,* cleaning by a 24 h soak at room temperature in a free rinsing liquid acid cleaner with a pH of 2 to 4.5.

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

7.6 *Isopropyl Alcohol,* Reagent grade. (**Warning**—Flammable.)

7.7 *Catalyst Wires,*

7.7.1 *Low-Metalloid Steel Wire*—1.59 mm (0.0625 in.) in diameter (No. 16 Washburn and Moen Gage).

NOTE 2—Carbon steel wire, soft bright annealed and free from rust of Grade 1008 as described in Specification A510M is satisfactory. Similar wire conforming to Specification E230/E230M is also satisfactory

7.8 *Electrolytic Copper Wire,* 1.63 mm (0.064 in.) in diameter (No. 16 Imperial Standard Wire Gage or No. 14 American Wire Gage), 99.9 % purity, conforming to Specification B1.

NOTE 3—Alternatively, suitably prepared steel and copper catalyst coils may be purchased from a supplier.

7.9 *Detergent,* free rinsing, water-soluble, anionic detergent with a pH of 9.5 to 11.

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 at a specified time the minimum required sample size is 360 mL. However, 6 to 8 tubes will be required to develop the data points to obtain the sludge mass at 25 % or agreed-upon residual RPVOT ratio by logarithmic interpolation. Therefore, approximately 2200 mL to 2900 mL will be required for this test.

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

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

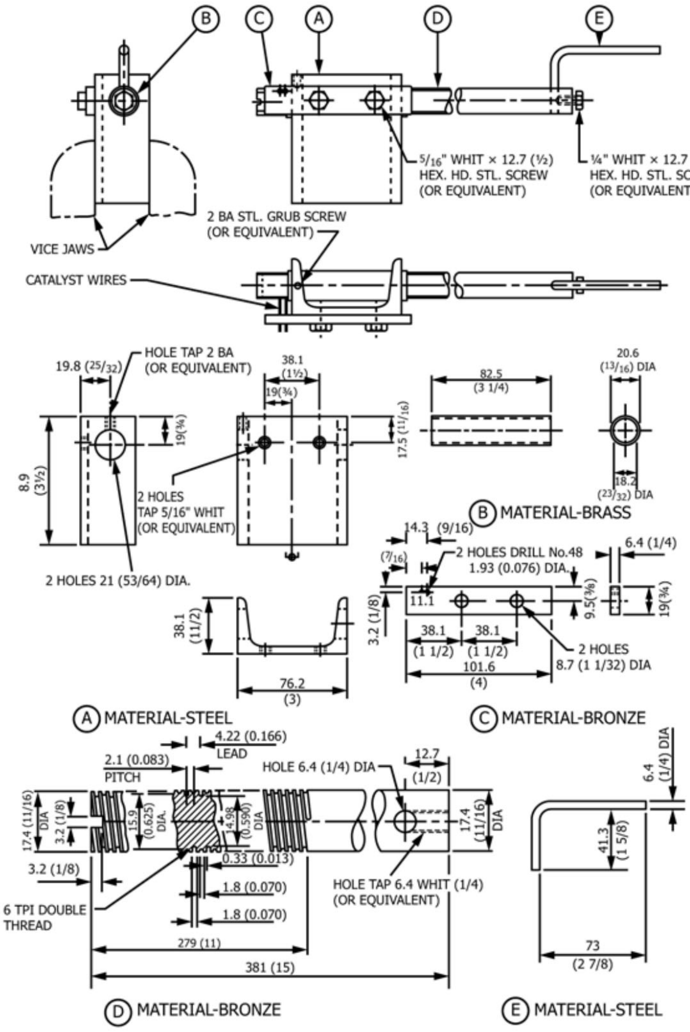


FIG. 4 Mandrel for Winding Catalyst Coils

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.

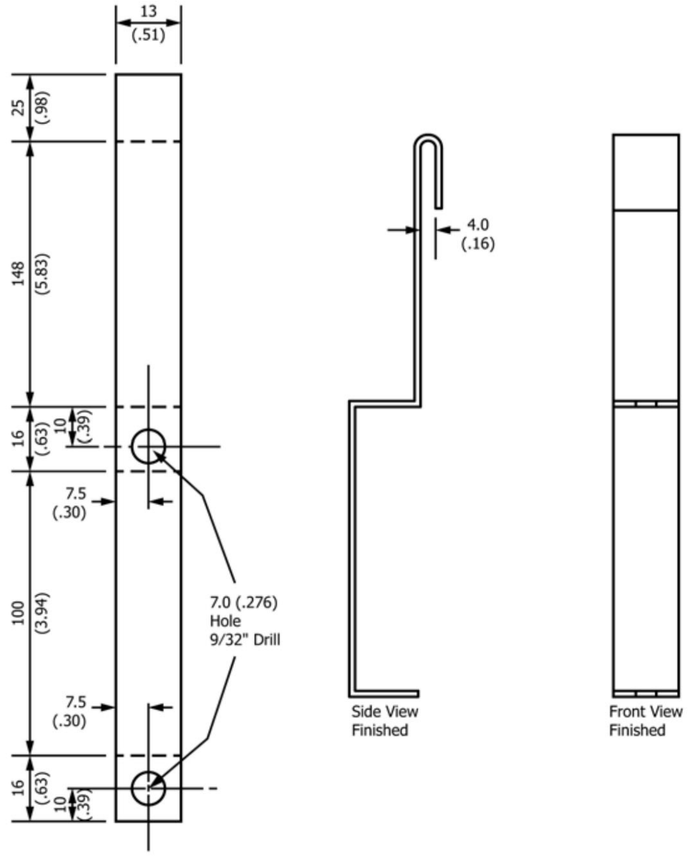
9.2 *Preparation of Catalyst Coil*—Twist the iron and copper wires tightly together at one end for three turns and then wind them simultaneously alongside each other on a threaded mandrel (see Fig. 4), inserting the iron wire in the deeper thread. Remove the coil from the mandrel, twist the free ends 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).

NOTE 4—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. 4 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 ensure 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 (7.9) and rinse thoroughly with tap water. Clean the interiors of the test tubes, exteriors of the condensers, and both interiors



NOTE 1—All dimensions are in millimetres (inches).

NOTE 2—Material—18-8 stainless steel, 22 gage (0.792 mm).

FIG. 5 Thermometer Bracket

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 (7.9) 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 95 °C. (Note that the tube with hot detergent should be put into a Test Method D943 bath at 95 °C, not the dry TOST bath at 120 °C.) Several hours soaking in this manner often serves to loosen all adhering deposits except iron oxide. Subsequent rinsing with cleaning reagent (7.4) 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 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 Before aging the fresh oil, set aside or measure the RPVOT of the un-aged sample.

10.2 Adjust the heating bath to a temperature high enough to maintain the oil sample temperature in the oxidation test cell, with oxygen flowing, at the required temperature of 120 °C ± 0.5 °C.

10.3 Fill a clean empty oxidation test tube with 360 mL of the oil sample. 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. Inspect the condenser for any contaminants. If there any signs of contamination, clean the condenser. 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.

10.4 Inspect the oxygen delivery tube for any condensation or contaminants. If there any signs of contamination or condensation, replace the tube. 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/h ± 0.1 L/h.

10.5 Prepare tube(s) using the procedures in 10.2 and 10.3.

10.6 Throughout the duration of the test, maintain the temperature of the heating bath according to 10.1.

10.7 It is highly recommended to use one heating bath for tubes when testing several tubes of same oil to obtain the sludge mass at 25 % or agreed-upon residual RPVOT ratio.

11. Procedure for Handling Test Oil Samples

11.1 Upon completion of test time interval, disconnect the oxygen delivery tube from the oxygen supply and remove one oxidation test cell apparatus from the heating bath. The test cell should be placed in a secure tube holder. Immediately remove the condenser and catalysts before the oil cools, allowing the test oil to drain thoroughly back into the test cell tube for 2 min.

11.2 Allow the apparatus and oil in the test tube to cool for 24 h ± 1 h out of direct sunlight.

11.3 Place a rubber stopper on top of the tube and shake it vigorously for 30 s to make it homogeneous (see Note 5).

11.4 Remove a 100 g oil sample from the test tube and use 50 g to measure RPVOT (Test Method D2272) of the used oil. Store the other 50 g of used oil in case a repeat RPVOT is required. Save the remainder of the used oil to ensure that 100 g of the used oil is available for the filtering.

11.5 To minimize effects of interpolation, it is recommended that the additional tubes be removed at approximately 168 hour intervals and processed as indicated in 11.1 through 11.4 for determinations of 25 % or an agreed upon RPVOT residual testing is required.

NOTE 5—An alternative procedure to ensure homogeneity is to vigorously shake the tube for 30 s and then pour the entire contents into an appropriately sized container such as a 500 mL glass jar. 50 g of sample is then taken for RPVOT and 100 g sample for filtration as specified in 12.1. Shake the jar each time a quantity of sample is removed and store the remaining sample in case repeat testing is required.

NOTE 6—If it is anticipated that reaching 25 % or an agreed-upon RPVOT residual will require testing longer than 1008 hours, it is suggested that additional tubes be tested or 11.1 through 11.4, for the first tube, be delayed by an approximate number of hours so that sufficient tubes are available to provide at least one data point with RPVOT less than 25 % (or an agreed-upon percentage) or the interval time between tube removal could be increased as appropriate.

12. Procedure for Determination of Sludge Mass

12.1 Remove a 100 g (± 0.1 g) sample into a clean beaker. It has been noted that the filtration process can be very slow. Therefore, splitting the oil into two different samples may be helpful. Although some oils may require more than one filter membranes, turbine oils with low sludging tendency will typically only use one filter membrane. Prior to filtering the oil, weigh two filter membranes to the nearest milligram in weighing vessels (E_1 mg and F_1 mg) (see Note 7 and Note 8). 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 sample into the two filter funnels (Note 9). After the oil is filtered through, rinse the filter funnels with n-heptane, and allow air to pass through the filter briefly. 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 n-heptane. In cases where large amounts of insolubles are generated, a rubber policeman may be used to scrape the walls of the beaker. Do not use less than 50 mL of n-heptane for each filter in this first rinsing procedure. Then, in a second rinsing operation, rinse each filter with an additional 25 mL of n-heptane. Up to 300 mL of solvent may be needed to complete rinsing of the sludge. The final rinses of n-heptane from this second operation should be completely colorless after passing through the filters.

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 1 h in the oven at 70 °C ± 5 °C. Allow the weighing vessels to cool in the cooling vessel 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 mass 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 mass (E_2 mg and F_2 mg).

NOTE 7—Weighing bottles, watch glasses (one as receptacle, one as a cover), glass petri dishes or aluminum foil dishes have been used for this purpose.

NOTE 8—More than two filter membranes may be used if a large amount of sludge is present.

NOTE 9—Occasionally, despite steps taken to improve filtration times, (that is, simultaneous filtration) the filtration process proceeds at a very slow rate. 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. The reason for stopping the filtration is to avoid introduction of additional dust from the air which would result in a higher than the actual insoluble value.

12.3 RPVOT residual ratio is defined as the percentage (%) obtained when by dividing the aged RPVOT by the fresh RPVOT value. For example, if the aged and fresh sample RPVOT is 500 min and 750 min, respectively, then the RPVOT residual ratio is 66.7 %.

12.4 A diagram may be drawn (see Fig. 6, for example) of the residual RPVOT versus milligrams insoluble per kilogram oil. The test is over when the residual RPVOT is less than 25 % or an agreed-upon percentage or specified time is reached. It may be helpful to estimate the amount of insolubles at 25 % or an agreed-upon residual RPVOT using the equation in 13.3.

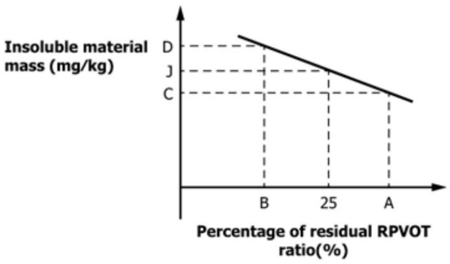


FIG. 6 Oxidation Stability versus Insolubles Generation

13. Calculations

13.1 Mass of insoluble material, in milligrams:

$$I = (E_2 - E_1) + (F_2 - F_1) \text{ mg} \tag{1}$$

where:

I = insoluble material, mg,
E_1, F_1 = initial mass of each filter membrane plus weighing bottle or watch glass, mg, and
E_2, F_2 = final mass of each filter membrane plus weighing bottle or watch glass, mg.

NOTE 10—If only one filter is used, F_1 and F_2 are both zero.

13.2 Mass of insoluble material, in milligrams per kilogram oil:

$$J = (I / 100 \text{ g})(1000 \text{ g/kg}) \tag{2}$$

where:

J = insoluble material per kilogram oil, and
I = insoluble material, mg.

13.3 The amount of insolubles (*J*) at 25 % or agreed-upon RPVOT residual can be estimated by logarithmic interpolation using the following equation. For the purpose of this example, 25 % RPVOT residual ratio is used.

$$LOG(J) = LOG(C) + [LOG(D) - LOG(C)](A - 25)/(A - B)$$

$$J = 10^{[LOG(C) + [LOG(D) - LOG(C)](A - 25)/(A - B)]} \tag{3}$$

where:

J = insoluble material, mg/kg,
A = percentage of residual RPVOT ratio when the insoluble mass was last measured above 25 %,
B = percentage of residual RPVOT ratio when the insoluble mass was last measured below 25 %,
C = insoluble material mass at A %, mg/kg, and
D = insoluble material mass at B %, mg/kg.

14. Report

14.1 Report the following information:

14.1.1 Report the mass of insoluble material in milligrams per kilogram and times for each tube.

14.1.2 Mass of insoluble material in milligrams per kilogram oil at 25 % or agreed-upon RPVOT residual ratio, or specified time.

14.1.3 Report the time it takes to reach 25 % or agreed-upon RPVOT residual ratio or specified time test was terminated as agreed with requestor.

14.1.4 Report the method (A or B) used to measure RPVOT. The method used should be consistent throughout the specific testing period.

15. Precision and Bias[7]

15.1 *Precision*—The precision of the test method for the mass of insoluble material (sludge) as obtained by the technical examination of interlaboratory test results is as follows:

15.1.1 *Repeatability Limit (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

15.1.1.1 *Repeatability*—2.0877 $(X + 0.295)^{0.75}$ mg/kg; X is the average of two values.

15.1.1.2 *Example Repeatabilities:*

Where X = 10; Repeatability = 12 mg/kg
Where X = 100; Repeatability = 66 mg/kg
Where X = 1000; Repeatability = 371 mg/kg

15.1.2 *Reproducibility Limit (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

15.1.2.1 *Reproducibility*—4.2818 $(X + 0.295)^{0.75}$; X is the average of two values mg/kg

15.1.2.2 *Example Reproducibilities:*

Where X = 10; Reproducibility = 25 mg/kg
Where X = 100; Reproducibility = 136 mg/kg
Where X = 1000; Reproducibility = 762 mg/kg

15.2 This precision statement was prepared with four turbine oils. These oils gave the following range of results:

15.2.1 Insoluble Material (sludge)—0 mg to 1800 mg.

15.3 *Bias*—The procedure in Test Method D7873 has no bias, because the values of mass of insoluble material are defined only in terms of this test method.

16. Keywords

16.1 antiwear hydraulic oils; circulating oils; copper; corrosion; dry TOST; gas turbine lubricants; hydraulic oils; inhibited mineral oils; insoluble material; metal catalysts; oxidation; RPVOT; sludge; steam turbine lubricants; turbine oils; varnish

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1769. Contact ASTM Customer Service at service@astm.org.

APPENDIX

(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.* (**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.

SUMMARY OF CHANGES

Subcommittee D02.09 has identified the location of selected changes to this standard since the last issue (D7873 – 13 (2017)) that may impact the use of this standard. (Approved July 1, 2020.)

(1) Revised Note 10.

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