Designation: D2112 – 15 (Reapproved 2023)

Standard Test Method for Oxidation Stability of Inhibited Mineral Insulating Oil by Pressure Vessel[1]

This standard is issued under the fixed designation D2112; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers and is intended as a rapid method for the evaluation of the oxidation stability of new mineral insulating oils containing a synthetic oxidation inhibitor. This test is considered of value in checking the oxidation stability of new mineral insulating oils containing 2,6-ditertiary-butyl para-cresol or 2,6-ditertiary-butyl phenol, or both, in order to control the continuity of this property from shipment to shipment. The applicability of this procedure for use with inhibited mineral insulating oils of more than 12 cSt at 40 °C (approximately 65 SUS at 100 °F) has not been established.

1.2 The values stated in SI units are to be regarded as standard except where there is no direct equivalent for hardware designed on the inch-pound unit basis.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* (See warning in 6.7.)

1.4 *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]

B1 Specification for Hard-Drawn Copper Wire

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Summary of Test Method

3.1 The test specimen is agitated by rotating axially at 100 r/min at an angle of 30° from the horizontal, under an initial oxygen pressure of 620 kPa (90 psi), in a stainless steel or copper vessel (for rapid temperature equilibrium), with a glass test specimen container and copper catalyst coil, in the presence of water, at a bath temperature of 140 °C. The time for an oil to react with a given volume of oxygen is measured; completion of the test is indicated by a specific drop in pressure.

4. Significance and Use

4.1 This is a control test of oxidation stability of new, inhibited mineral insulating oils for determining the induction period of oxidation inhibitors under prescribed accelerated aging conditions. There is no proven correlation between oil performance in this test and performance in service. However, the test method may be used to check the continuity of oxidation stability of production oils.

5. Apparatus

5.1 *Oxidation Vessel*—Glass test specimen container with cover and catalyst coil, pressure gauge, thermometer, test bath, and accessories as described in Annex A1. The assembled apparatus is shown in Fig. 1, and its design shown schematically in Fig. 2.

6. Reagents and Materials

6.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3]

[1] This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.06 on Chemical Test.

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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] *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.

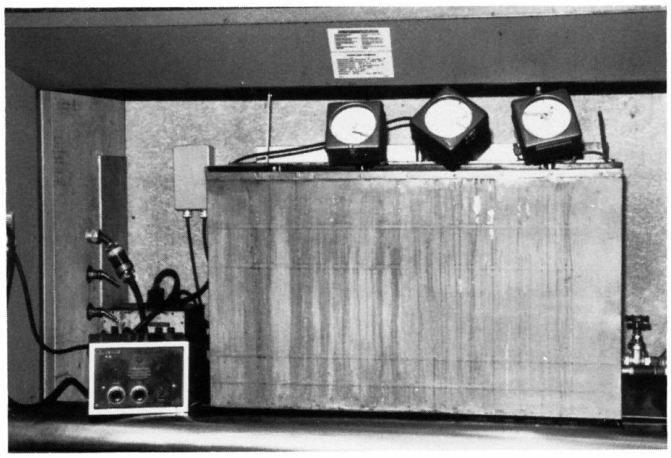

FIG. 1 Rotating Vessel Oxidation Test Apparatus

6.2 *Hydrochloric Acid,* 10 vol %.

6.3 *Silicon Carbide Abrasive Cloth,* 100-grit with cloth backing.

6.4 *Acetone,* ACS grade.

6.5 *2-Propanol,* 99 vol %, refined.

6.6 *Liquid Detergent.*

6.7 *Oxygen,* 99.5 %, with pressure regulation above 620 kPa (90 psi). (**Warning—**Oxygen vigorously accelerates combustion.)

6.8 *Potassium Hydroxide, Alcohol Solution* (1 mass %)—Dissolve 7.93 g of potassium hydroxide (KOH) pellets in 1 L of 99 % refined 2-propanol.

6.9 *Silicone Stopcock Grease.*

6.10 *Wire Catalyst*—AWG No. 14 (approximately 1.628 mm diameter) electrolytic copper wire 99.9 % purity, conforming to Specification B1. Soft-drawn copper wire of an equivalent grade may also be used.

7. Hazards

7.1 Consult Safety Data Sheets for all materials used in this test method.

8. Preparation of Apparatus

8.1 *Catalyst Preparation*—Immediately before use, polish the copper wire with silicon carbide abrasive cloth and wipe free from abrasives with a clean dry cloth. Wind approximately 3 m of the wire into a coil having an outside diameter of 44 mm to 48 mm and stretched to a height of 40 mm to 42 mm. Clean the coil thoroughly with acetone and allow it to air-dry. Immediately after air drying, insert the coil with a twisting motion into the glass test specimen container. Handle the coil only with clean tongs to avoid contamination. Weigh the coil and the container to the nearest 0.1 g and record the weight. Prepare a new coil for each test specimen.

8.2 *Alternative Method of Catalyst Preparation*—Wind approximately 3 m of copper wire into a coil of the dimensions specified in 8.1, and add to the glass container. Weigh the coil and container to the nearest 0.1 g and record the weight. Wash the coil by filling the container above the level of the coil with 10 % hydrochloric acid by volume for 30 s. Discard the acid and rinse the coils three times with tap water followed by three times with distilled water. Reweigh the coil and container and determine by difference the water retained in the system. The coils are now ready for use. This procedure has been found to be acceptable for treatment of commercially available, prepackaged, preformed coils that meet the requirement described in this test method. Use a new coil for each test specimen.

8.3 *Cleaning of Vessel*—Wash the vessel body, lid, and inside of vessel stem with hot detergent solution and with water. Rinse inside of stem with 2–propanol and blow dry with clean dry air. An alternative cleaning solution is the use of a 50/50 volumetric blend of methanol and acetone; it has been found to be effective in cleaning sludge from the vessel. If the vessel body, lid, or inside of stem smells sour after simple cleaning, wash with alcoholic KOH solution and repeat as before (see Note 1).

NOTE 1—Insufficient cleaning of the vessel may adversely affect test results.

9. Procedure

9.1 *Charging*—Weigh 50 g ± 0.5 g of oil sample into the container, add 5 mL of distilled water, and cover with a 51 mm (2-in.) watch glass or a 57.2 mm (2¼-in.) PTFE disk with one or four holes and retaining spring. If rinse water is present in the container, compensate for it by using less added water based on the water retention determined in 8.2. Add 5 mL of distilled water to the vessel and slide the test specimen container and cover lid into the vessel body (see Note 2). Apply a thin coating of silicone stopcock grease to the O-ring vessel seal located in the gasket groove of the vessel lid to provide lubrication, and insert the lid into the vessel body. Place the vessel cap over the vessel stem, and tighten by hand. Cover the threads of the gauge-nipple with a thin coating of stopcock grease or TFE-fluorocarbon, or both, and screw the gauge into the top-center tap of the vessel stem. A pressure transducer can also be used. Flush the vessel twice with oxygen supplied to the vessel at 620 kPa (90 psi) and release to the atmosphere. Adjust the regulating valve on the oxygen supply tank to 620 kPa (90 psi) at a room temperature of 25 °C. For each 2.8 °C above or below this temperature, add or subtract 7 kPa (1 psi) unit to attain the required initial pressure. Fill the vessel to this required pressure and close the inlet valve securely by hand. If desired, test the vessel for leaks by immersion in water (see Note 3). Prepare a duplicate test specimen in exactly the same way.

NOTE 2—The water between the vessel well and the test specimen container aids heat transfer.

NOTE 3—If the vessel was immersed in water to check for leaks, dry the outside of the wet vessel by any convenient means such as an air blast or a towel. Such drying is advisable to prevent subsequent introduction of free water into the hot oil bath, which would cause sputtering.

9.2 *Oxidation*—Bring the heating bath to the test temperature of 140 °C while the stirrer is in operation. Insert the vessels into the rotating carriages and note the time. If an

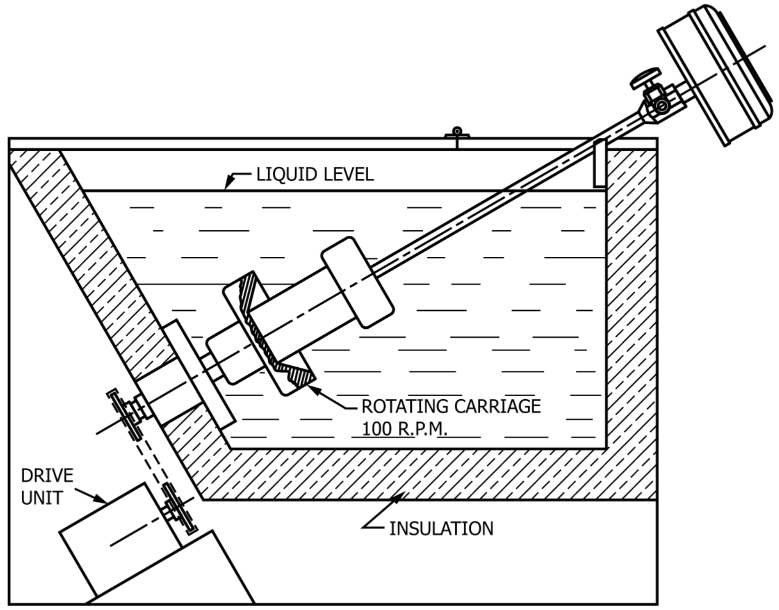

FIG. 2 Schematic Drawing of Rotary Vessel

auxiliary heater is used, keep it on for the first 5 min of the run and then turn it off (see Note 4). Allow the bath temperature to level out at the test temperature; this must occur within 10 min after the vessels are inserted. Maintain the test temperature within ±0.1 °C (see Note 5).

NOTE 4—The time for the bath to reach the operating temperature after insertion of the vessels may differ for different apparatus assemblies and should be observed for each unit. The objective is to find a set of conditions that does not permit a drop of more than 2 °C after insertion of the vessels and allows the vessel pressure to reach a plateau within 15 min as shown in Curve A of Fig. 3.

NOTE 5—Maintaining the *correct* temperature within the specification limits of ±0.1 °C during the entire test run is the most important single factor ensuring good repeatability and reproducibility of test results.

9.3 Keep the vessels completely submerged and maintain rotation continuously and uniformly throughout the test. A standard rotational speed of 100 r/min ± 5 r/min is required;

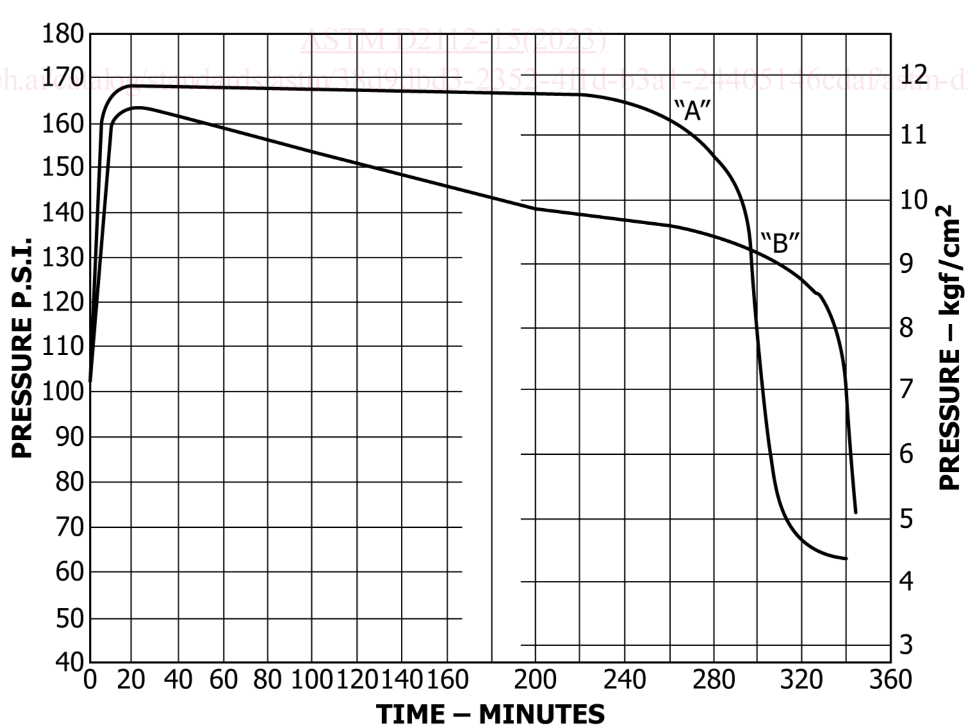

FIG. 3 Pressure Versus Time Plot of Two Rotary Vessel Oxidation Test Runs