



Designation: **D2112–01a (Reapproved 2007) D2112 – 15**

## Standard Test Method for Oxidation Stability of Inhibited Mineral Insulating Oil by Pressure Vessel<sup>1</sup>

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

NOTE 1—A modification of this test method, which uses the same procedure and apparatus but a higher (150°C) bath temperature, has been published as Test Method [D2272](#).

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

[B1 Specification for Hard-Drawn Copper Wire](#)

~~[D2272 Test Method for Oxidation Stability of Steam Turbine Oils by Rotating Pressure Vessel](#)~~

[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 ~~rpm/min~~ at an angle of 30° from the horizontal, under an initial oxygen pressure of ~~90 psi (620 kPa)~~, 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~~, ~~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](#).

<sup>1</sup> 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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<sup>2</sup> 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.



FIG. 1 Rotating Vessel Oxidation Test Apparatus

## 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.<sup>3</sup>

6.2 *Hydrochloric Acid,  $\pm 0$  vol %*—10 vol %.

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

6.4 *Acetone*, *sp*—ACS grade.

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

6.6 *Liquid Detergent*.

6.7 *Oxygen*, 99.5 %, with pressure regulation above 90 psi (620 kPa) 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 (0.0641 in. (1.628 mm)) (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 ~~Material~~ 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 to 48 mm and stretched to a height of 40 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

<sup>3</sup> *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 Analytical 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.

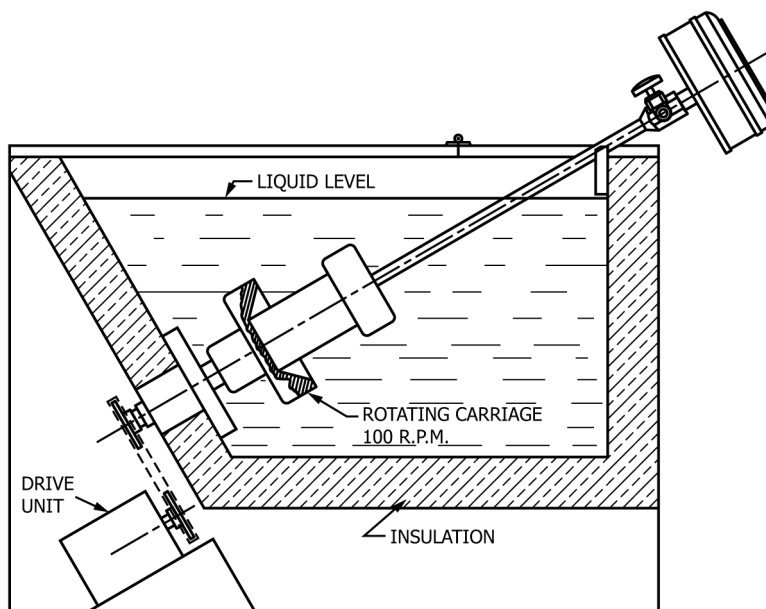


FIG. 2 Schematic Drawing of Rotary Vessel

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 21](#)).

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

## 9. Procedure

9.1 *Charging*—Weigh  $50 \pm 0.5$  g of oil sample into the container, add 5 mL of distilled water, and cover with a 2-in. (51-mm) 51-mm (2-in.) watch glass or a 257.2-mm 1/4-in. (57.2-mm) (2 1/4-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 [8.38.2](#). Add 5 mL of distilled water to the vessel and slide the test specimen container and cover lid into the vessel body (see [Note 32](#)). 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 gage-nipple gauge-nipple with a thin coating of stopcock grease or TFE-fluorocarbon, or both, and screw the gage 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 90 psi (620 kPa) 620 kPa (90 psi) and release to the atmosphere. Adjust the regulating valve on the oxygen supply tank to 90 psi 620 kPa (90 psi) at a room temperature of 25°C (77°F) 25°C. For each 2.8°C (5.1°F) above or below this temperature, add or subtract 1 psi (7 kPa) 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 43](#)). 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 auxiliary heater is used, keep it on for the first 5 min of the run and then turn it off (see [Note 54](#)). 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  $\pm 0.1^\circ\text{C}$  (see [Note 65](#)).

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  $\pm 0.1^\circ\text{C}$  during the entire test run is the most important single factor ensuring good repeatability and reproducibility of test results.

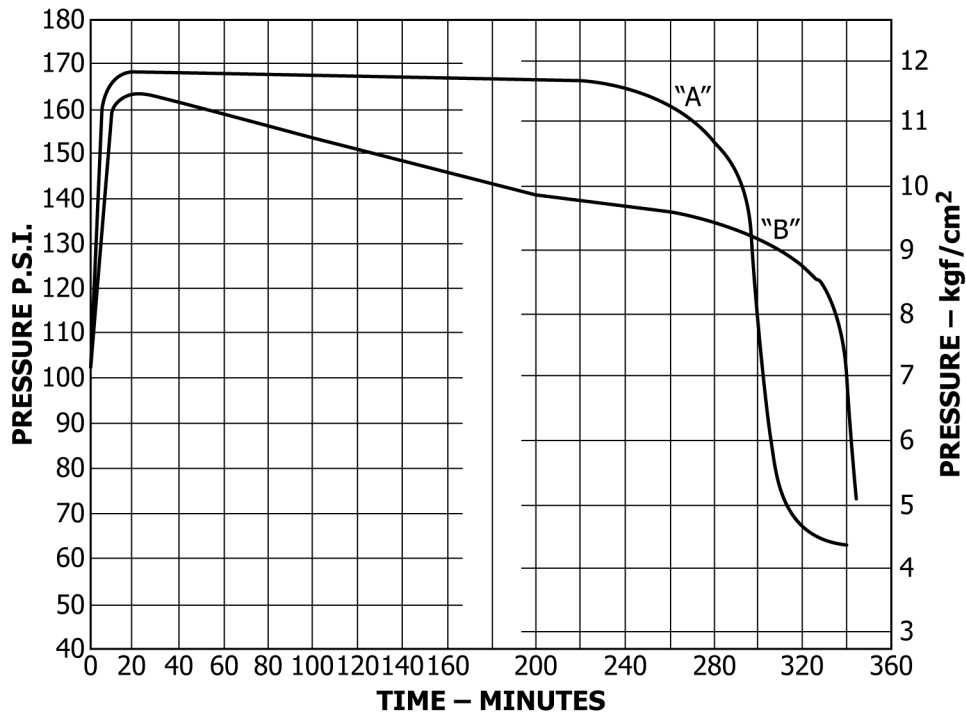


FIG. 3 Pressure Versus Time Plot of Two Rotary Vessel Oxidation Test Runs

9.3 Keep the vessels completely submerged and maintain rotation continuously and uniformly throughout the test. A standard rotational speed of  $100 \pm 5$  rpm/min is required; any appreciable variations in this speed could cause erratic results. If a dial gage/gauge is used, take readings every 5 min.

9.4 The test is complete after the pressure drops more than  $25 \text{ psi} (172 \text{ kPa})$  below the maximum pressure. The  $25 \text{ psi} (172 \text{ kPa})$  pressure drop usually, but not always, coincides with an induction-type “period of rapid pressure drop.” When it does not, the operator should question whether a valid experiment has been produced/produced (Note 6).

NOTE 6—A typical experiment is shown in Fig. 3 as Curve A. The maximum pressure expected to be reached within 30 min; a pressure plateau is established and an induction-type pressure drop is observed. Curve B, in which there is a gradual decrease in pressure before the induction break is recorded, is more difficult to evaluate. The gradual decrease in pressure could be due to a vessel leak; however, some synthetic fluids will generate this type of curve. If a leak is suspected, repeat the test in a different vessel. If the same type of curve is derived when the test is repeated, the experiment is likely valid.

9.5 After termination of the test, remove the vessels from the oil bath, dip briefly into and swirl around in a bath of light mineral oil or detergent and water to wash off the adhering bath oil. Rinse off the vessels with hot water, then immerse in cold water to bring them quickly to room temperature. Allow the vessel to fully cool before bleeding off excess oxygen pressure and opening the vessel/vessel (Note 7).

NOTE 7—A hazardous situation can arise when excess oxygen is bled off immediately upon removal of the vessel from the bath since it may be accompanied by hot oil and steam. (See 6.7).

## 10. Interpretation of Results

10.1 Observe a plot of the recorded pressure versus time and establish the plateau pressure (see Note 76). Also record the time at the point on the falling part of the curve where the pressure is  $25 \text{ psi} (172 \text{ kPa})$  less than the established plateau pressure. Plateau pressures in duplicate tests should not differ by more than  $5 \text{ psi} (35 \text{ kPa})$ .

10.2 The vessel life of the test specimen is the time in minutes from the start of the test to a  $25 \text{ psi} (172 \text{ kPa})$  pressure drop from the level of the established plateau.

## 11. Report

11.1 Report test method used.

11.2 Report the time as the average of two duplicate determinations and the difference of the individual determinations. The recipient of the report can then be reassured that the determination is not suspect, as specified in 12.1.

## 12. Precision and Bias

12.1 The following criteria should be used for judging the acceptability of results (95 % probability):