Designation: D2272 – 22

Standard Test Method for Oxidation Stability of Steam Turbine Oils by Rotating Pressure Vessel[1]

This standard is issued under the fixed designation D2272; 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[2] utilizes an oxygen-pressured vessel to evaluate the oxidation stability of new and in-service turbine oils having the same composition (base stock and additives) in the presence of water and a copper catalyst coil at 150 °C.

1.2 Appendix X1 describes a new optional turbine oil (unused) sample nitrogen purge pretreatment procedure for determining the percent residual ratio of RPVOT value for the pretreated sample divided by RPVOT value of the new (untreated) oil, sometimes referred to as a "% RPVOT Retention." This nitrogen purge pretreatment approach was designed to detect volatile antioxidant inhibitors that are not desirable for use in high temperature gas turbines.

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

1.3.1 *Exception*—Other units are provided in parentheses (psi, grams, and inches), because they are either the industry accepted standard or the apparatus is built according the figures in this standard, or both.

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.* For specific warning statements, see 6.2, 6.4, 6.5, 6.6, and 6.10.

1.5 *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:*[3]

B1 Specification for Hard-Drawn Copper Wire

D943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils

D1193 Specification for Reagent Water

D4742 Test Method for Oxidation Stability of Gasoline Automotive Engine Oils by Thin-Film Oxygen Uptake (TFOUT)

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

2.2 *Energy Institute Standard:*[4]

IP 229 Determination of the Relative Oxidation Stability by Rotating Bomb of Mineral Turbine Oil

2.3 *ISO Standard:*[5]

ISO 3170 Petroleum Liquids—Manual Sampling

3. Summary of Test Method

3.1 The test oil, water, and copper catalyst coil, contained in a covered glass container, are placed in a vessel equipped with a pressure gauge. The vessel is charged with oxygen to a gauge pressure of 620 kPa (90 psi, 6.2 bar) (see Eq 1), placed in a constant-temperature oil bath set at 150 °C or dry block taken to 150 °C (Fig. 1 and Fig. 2), and rotated axially at 100 rpm at an angle of 30° from the horizontal.

3.2 The number of minutes required to reach a specific drop in gauge pressure is the oxidation stability of the test sample.

$$100\ \text{kPa} = 1.00\ \text{bar} = 14.5\ \text{psi} \quad (1)$$

[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] von Fuchs, G. H., Claridge, E. L., and Zuidema, H. H., "The Rotary Bomb Oxidation Test for Inhibited Turbine Oils," *Materials Research and Standards*, MTRSA (formerly ASTM Bulletin), No. 186, December 1952, pp. 43–46; von Fuchs, G. H., "Rotary Bomb Oxidation Test," *Lubrication Engineering*, Vol 16, No.1, January 1960, pp. 22–31.

[3] 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.

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

[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**

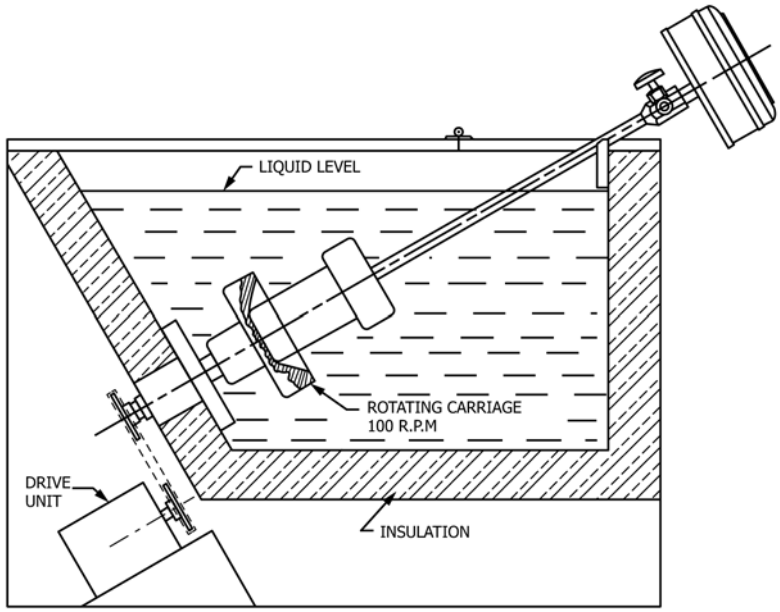

FIG. 1 Schematic Drawing of the Rotary Vessel Test Apparatus

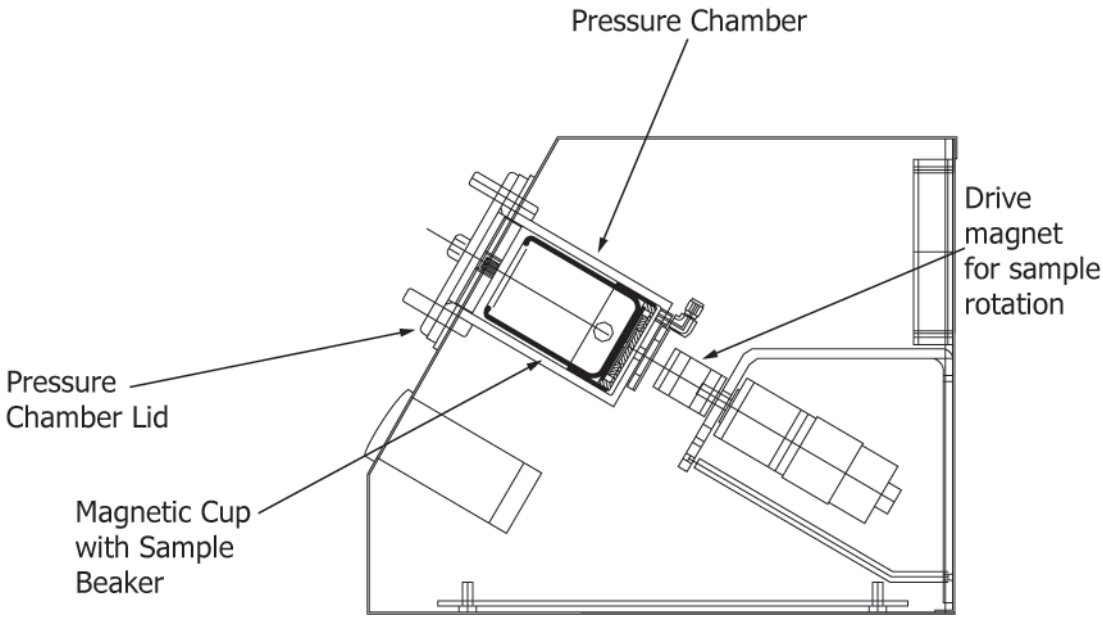

FIG. 2 RPVOT Metal Block Bath Instrument

4. Significance and Use

4.1 The estimate of oxidation stability is useful in controlling the continuity of this property for batch acceptance of production lots having the same operation. It is not intended that this test method be a substitute for Test Method D943 or be used to compare the service lives of new oils of different compositions.

4.2 This test method is also used to assess the remaining oxidation test life of in-service oils.

Method A

5. Apparatus

5.1 *Method A, Liquid Bath RPVOT*—Oxidation Vessel, Glass Sample Container with Four-Hole PTFE Disk, Hold-Down Spring, Catalyst-Coil, Pressure Gauge, Thermometer, and Test Bath as described in Annex A1. The assembled apparatus is shown schematically in Fig. 1 and Fig. A1.6.

5.2 *Method B, Dry Block Bath RPVOT*—See Section 13 for this additional option.

5.3 *Temperature Display*—The temperature shall have a displayed resolution to 0.1 °C or better and be calibrated as described in Annex A1 on an annual basis.

5.4 *Pressure Display*—The pressure readout, whether analog or digital, shall be calibrated as described in Annex A1.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests in the final cleaning stages. 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.[6] 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.

6.2 *Isopropyl Alcohol,* reagent grade. (**Warning**—Flammable. Health hazard.)

6.3 *Liquid Detergent.*

6.4 *n-Heptane,* 99.0 minimum mol % (pure grade). (**Warning**—Flammable. Health hazard.)

6.5 *Oxygen,* 99.5 %, with pressure regulation to 620 kPa (90 psi, 6.2 bar). (**Warning**—Vigorously accelerates combustion.)

6.6 *Potassium Hydroxide, Alcohol Solution (1 %)*—Dissolve 12 g of potassium hydroxide (KOH) pellets in 1 L of the isopropyl alcohol. (**Warning**—Flammable. Health hazard.)

6.7 *Silicone Carbide Abrasive Cloth,* 100-grit with cloth backing.

6.8 *Silicone Stopcock Grease.*

6.9 *Wire Catalyst, Electrolytic Copper Wire,* 1.63 mm ± 1 % (0.064 in. ± 1 %) 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.

6.10 *Acetone,* reagent grade. (**Warning**—Flammable. Health hazard.)

6.11 *Reagent Water,* conforming to Specification D1193, Type II.

7. Sampling

7.1 Samples for this test method can come from tanks, drums, small containers, or even operating equipment. As the results obtained by this method are readily affected by traces of impurities, avoid contamination during sampling and subsequent handling; especially for used fluids. Samples shall be prepared and decanted in accordance with the procedures given in ISO 3170 and stored away from light in dark colored bottles.

8. Preparation of Apparatus

8.1 *Catalyst Preparation*—Before use, polish approximately 3 m of the copper wire with a silicon carbide abrasive cloth and wipe free from abrasives with a clean, dry cloth. Wind the wire into a coil having an outside diameter 44 mm to 48 mm and weight of 55.6 g ± 0.3 g and stretched to a height of 40 mm to 42 mm. Clean the coil thoroughly with isopropyl alcohol, air-dry, and insert inside the glass sample container by a turning motion, if necessary. A new coil is used for each sample. For extended storage, the prepared coil may be packaged in a dry, inert atmosphere. For overnight storage (less than 24 h), the coils may be stored in *n*-Heptane.

NOTE 1—Commercially available and prepackaged coils prepared as described in 8.1 can also be used for the test.[7]

8.2 *Cleaning of Vessel*—Wash the vessel body, cap, and inside of vessel stem with a suitable solvent (for example, petroleum spirit, heptane, or acetone.) Wash with hot detergent solution and rinse thoroughly with water. Rinse the inside of the stem with isopropyl alcohol and blow dry with clean compressed air. Keep the plastic valve out of the hot detergent to prevent its deterioration. Failure to remove oxidation residue can adversely affect test results.

8.3 *Cleaning of Glass Container*—Drain and rinse with a suitable solvent (for example, non-reagent petroleum spirit, heptane, or acetone). Soak or scrub in an aqueous detergent solution. Brush thoroughly and flush thoroughly with tap water. Rinse with isopropyl alcohol, followed by distilled water and air dry. If any insolubles remain, soak overnight in an acid-type cleaning solution and repeat the above procedure starting from the tap water flush. Do not use chipped or cracked glassware.

8.4 *Cleaning of Polytetrafluoroethylene (PTFE) Disk*—Remove any residual oil with a suitable solvent and clean by brushing with detergent solution. Rinse thoroughly with tap water, followed by distilled water rinse and air dry.

9. Procedure

9.1 *Charging*—Weigh the glass sample container with a freshly cleaned catalyst coil. Weigh 50 g ± 0.5 g of oil sample into the container; also add 5 mL of reagent water. Add another 5 mL of reagent water to the vessel body and slide the sample container into the vessel body (see Note 2). Cover the glass container with a 57.2 mm (2 ¼ in.) PTFE disk and place a hold-down spring[8] on top of the PTFE disk. Apply a thin coating of silicone stopcock grease to the O-ring vessel seal located in the gasket groove of the vessel cap to provide lubrication, and insert the cap into the vessel body.

NOTE 2—The water between the vessel wall and the sample container aids heat transfer.

9.1.1 Tighten the closure ring by hand. Cover the threads of the gauge-nipple with a thin coating of stopcock grease (PTFE

[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.

[7] Prepackaged coils were provided for RR:D02-1409.

[8] PTFE disk with 4-holes and hold down spring were provided for RR:D02-1409.