Designation: D7098 – 21

Standard Test Method for Oxidation Stability of Lubricants by Thin-Film Oxygen Uptake (TFOUT) Catalyst B[1,2]

This standard is issued under the fixed designation D7098; 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 the oxidation stability of lubricants by thin-film oxygen uptake (TFOUT) Catalyst B. This test method evaluates the oxidation stability of petroleum products, and it was originally developed as a screening test to indicate whether a given re-refined base stock could be formulated for use as automotive engine oil[3] (see Test Method D4742). The test is run at 160 °C in a pressure vessel under oxygen pressure, and the sample contains a metal catalyst package, a fuel catalyst, and water to partially simulate oil conditions in an operating engine. In addition, the test method has since been found broadly useful as an oxidation test of petroleum products.[4]

1.2 The applicable range of the induction time is from a few minutes up to several hundred minutes or more. However, the range of induction times used for developing the precision statements in this test method was from 40 min to 280 min.

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*—Pressure units are provided in psig, and dimensions are provided in inches in Annex A1 and Annex A2, because these are the industry accepted standard and the apparatus is built according to the figures shown.

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 *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:*[5]

A314 Specification for Stainless Steel Billets and Bars for Forging

B211 Specification for Aluminum and Aluminum-Alloy Rolled or Cold-Finished Bar, Rod, and Wire (Metric) B0211_B0211M

D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration

D1193 Specification for Reagent Water

D2272 Test Method for Oxidation Stability of Steam Turbine Oils by Rotating Pressure Vessel

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

D7962 Practice for Determination of Minimum Immersion Depth and Assessment of Temperature Sensor Measurement Drift

D8164 Guide for Digital Contact Thermometers for Petroleum Products, Liquid Fuels, and Lubricant Testing

D8278 Specification for Digital Contact Thermometers for Test Methods Measuring Flow Properties of Fuels and Lubricants

E1 Specification for ASTM Liquid-in-Glass Thermometers

E144 Practice for Safe Use of Oxygen Combustion Vessels

[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 D02.09.0G on Oxidation Testing of Engine Oils.

Current edition approved Oct. 1, 2021. Published November 2021. Originally approved in 2005. Last previous edition approved in 2015 as D7098 – 08 (2015). DOI: 10.1520/D7098-21.

[2] While Catalyst B can be used for testing oxidation stability of many lubricant types, the mixture of fuel, nitro-paraffin, and catalyst components used in this test method simulates the Sequence IIIE Engine Test. Test results on several ASTM reference oils have been found to correlate with Sequence IIIE engine tests in hours for a 375 % viscosity increase. (See Ku, Chia-Soon, Pei, Patrick T., and Hsu, Stephen M., "A Modified Thin-Film Oxygen Uptake Test (TFOUT) for the Evaluation of Lubricant Stability in ASTM Sequence IIIE Test, SAE Technical Paper Series 902121, Tulsa, OK, Oct. 22-25, 1990.)

[3] Ku, C. S. and Hsu, S. M., "A Thin Film Uptake Test for the Evaluation of Automotive Lubricants," *Lubrication Engineering*, 40, 2, 1984, pp. 75–83.

[4] Selby, Theodore W., "Oxidation Studies with a Modified Thin-Film Oxygen Uptake Test", *SAE Technical Paper Series* 872127, Toronto, Ontario, Nov. 2-5, 1987.

[5] 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.

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

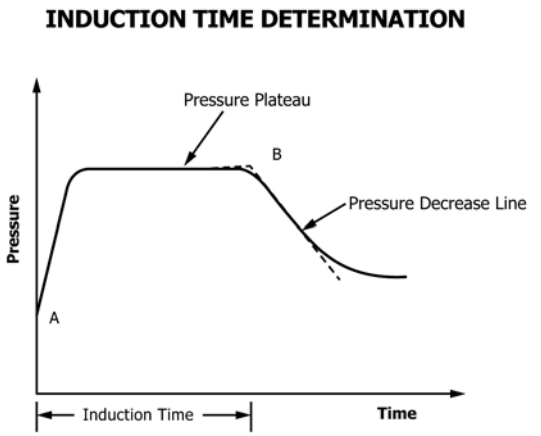

FIG. 1 Pressure versus Time Diagram of the Oxidation Test

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *break point, n*—the precise point of time at which rapid oxidation of the oil begins.

3.1.2 *oxidation induction time, n*—the time until the oil begins to oxidize at a relatively rapid rate as indicated by the decrease of oxygen pressure.

3.1.3 *oxygen uptake, n*—oxygen absorbed by oil as a result of oil oxidation.

4. Summary of Test Method

4.1 The test oil is mixed in a glass container with four other liquids used to simulate engine conditions: (*1*) an oxidized/nitrated fuel component (Annex A3), (*2*) a mixture of soluble metal naphthenates (lead, iron, manganese, and tin naphthenates (Annex A4), (*3*) a nitro-paraffinic compound, and (*4*) Type I reagent water.

4.2 The glass container holding the oil mixture is placed in a pressure vessel equipped with a pressure sensor. The pressure vessel is sealed, charged with oxygen to a pressure of 620 kPa (90 psig), and placed in an oil or dry bath at 160 °C at an angle of 30° from the horizontal. The pressure vessel is rotated axially at a speed of 100 r⁄min forming a thin film of oil within the glass container resulting in a relatively large oil-oxygen contact area.

4.3 The pressure of the pressure vessel is recorded continuously from the beginning of the test and the test is terminated when a rapid decrease of the pressure vessel pressure is observed (Point B, Fig. 1). The period of time that elapses between the time when the pressure vessel is placed in the oil or dry bath and the time at which the pressure begins to decrease rapidly is called the oxidation induction time and is used as a measure of the relative oil oxidation stability.

5. Significance and Use

5.1 This test method was originally developed to evaluate oxidation stability of lubricating base oils combined with additives chemistries similar to those found in gasoline engine oils and service.[2]

5.2 This test method is useful for screening formulated oils before engine tests. Within similar additive chemistries and base oil types, the ranking of oils in this test appears to be predictive of ranking in certain engine tests. When oils having different additive chemistries or base oil type are compared, results may or may not reflect results in engine tests. Only gasoline engine oils were used in generating the precision statements in this test method.

6. Apparatus

6.1 *Oxidation Bath and Pressure Vessel*—See appropriate Annex (Annex A1[6] or Annex A2[7]) for detailed description of apparatus and accessories for equipment described in this test method.

NOTE 1—To reduce vapor odors when opening pressure vessel after use, a hood may be desirable.

6.2 *Precision Pressure Gauge*—Use a certified precision pressure gauge to accurately control the oxygen feed to the pressure vessel. The gauge shall have a sufficient range to encompass 0 kPa to 650 kPa (~90 psig) required by the test method with division 2.0 kPa (~0.5 psig) or better to enable readings to be made to 2.0 kPa (~0.25 psig).

6.3 *Thermometer,* digital (DCT) or liquid-in-glass styles shall be used to check the bath temperature monthly at a minimum. The thermometer shall be able to be read with an accuracy of ±0.1 °C at the level of 160 °C, such as D02-DCT-06 in Specification D8278. Thermometers, digital (DCT) or analog, need to be checked for accuracy at least one time per year. See Specification E1 and Practice D7962 for guidance.

7. Reagents

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.[8]

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

7.3 *Acetone,* CH_3COCH_3.

7.4 *Air,* containing 2000 ppm nitrogen dioxide, NO_2 (commercially available compressed gas mixture, certified within ±5 %).

[6] The sole source of supply of the apparatus known to the committee at this time is Koehler Instrument Co., Inc., 1595 Sycamore Ave., Bohemia, NY11716 and Stanhope-Seta, London St., Chertsey, Surrey, KT16 8AP, U.K. 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 apparatus known to the committee at this time is Tannas Co., 4800 James Savage Rd., Midland, MI 48642. 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.

[8] *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.