Designation: D7528 – 13

Standard Test Method for Bench Oxidation of Engine Oils by ROBO Apparatus[1]

This standard is issued under the fixed designation D7528; 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.

INTRODUCTION

Any properly equipped laboratory, without outside assistance, can use the procedure described in this test method. However, the ASTM Test Monitoring Center[2] (TMC) provides reference oils and an assessment of the test results obtained on those oils by the laboratory. By these means, the laboratory will know whether its use of the test method gives results statistically similar to those obtained by other laboratories. Furthermore, various agencies require that a laboratory utilize the TMC services in seeking qualification of oils against specifications. For example, the U.S. Army imposes such a requirement in connection with several Army engine lubricating oil specifications.

Accordingly, this test method is written for use by laboratories that utilize the portions of the test method that refer to the TMC services. Laboratories that choose not to use the TMC services may simply ignore these portions.

This test method may be modified by means of information letters issued by the TMC. In addition, the TMC may issue supplementary memoranda related to the method.

1. Scope*

1.1 This test method describes a bench procedure to simulate the oil aging encountered in Test Method D7320, the Sequence IIIG engine test method. These aged oils are then tested for kinematic viscosity and for low-temperature pumpability properties as described in the Sequence IIIGA engine test, Appendix X1 of Test Method D7320.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.2.1 *Exceptions*—There are no SI equivalents for some apparatus in Section 6, and there are some figures where inch units are to be regarded as standard.

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.* Specific warning statements are given in Sections 7 and 8.

1.4 This test method is arranged as follows:

[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.B0.07 on Development and Surveillance of Bench Tests Methods.

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[2] ASTM Test Monitoring Center, 6555 Penn Avenue, Pittsburgh, PA 15206-4489. www.astmtmc.cmu.edu.

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

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants

D4485 Specification for Performance of Active API Service Category Engine Oils

D4684 Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature

D5293 Test Method for Apparent Viscosity of Engine Oils and Base Stocks Between –5 °C and –35 °C Using Cold-Cranking Simulator

D7320 Test Method for Evaluation of Automotive Engine Oils in the Sequence IIIG, Spark-Ignition Engine

2.2 *SAE Standard:*[4]

SAE J300 Engine Oil Viscosity Classification

3. Terminology

3.1 *Definitions:*

3.1.1 *candidate oil, n*—an oil that is intended to have the performance characteristics necessary to satisfy a specification and is to be tested against that specification. **D4175**

3.1.2 *reference oil, n*—an oil of known performance characteristics, used as a basis for comparison.

3.1.2.1 *Discussion*—Reference oils are used to calibrate testing facilities, to compare the performance of other oils, or to evaluate other materials (such as seals) that interact with oils. **D4175**

3.1.3 *non-reference oil, n*—any oil other than a reference oil, such as a research formulation, commercial oil or candidate oil. **D4175**

3.1.4 *test oil, n*—any oil subjected to evaluation in an established procedure. **D4175**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aged oil, n*—a test oil after it has been subjected to the 40-h aging process in a ROBO apparatus.

3.3 *Acronyms:*

3.3.1 *ROBO, n*—Romaszewski Oil Bench Oxidation[5]

4. Summary of Test Method

4.1 The test oil is combined with a small amount of iron ferrocene catalyst and placed in a 1 L reaction vessel. That mixture is stirred and heated for 40 h at 170 ºC with air flowing across the liquid surface under negative pressure. In addition, nitrogen dioxide and air are introduced below the reaction surface. After cooling, the oxidized, concentrated test oil is subjected to pertinent viscometric tests. Evaporated oil is condensed in order to weigh it and calculate evaporative loss.

5. Significance and Use

5.1 This bench test method is intended to produce comparable oil aging characteristics to those obtained with ASTM TMC Sequence IIIGA matrix reference oils 434, 435 and 438 after aging in the Sequence IIIG engine test.

5.2 To the extent that the method generates aged oils comparable to those from the Sequence IIIG engine test, the measured increases in kinematic and MRV viscosity indicate the tendency of an oil to thicken because of volatilization and oxidation, as in the Sequence IIIG and IIIGA (see Appendix X1 in Test Method D7320) engine tests, respectively.

5.3 This bench test procedure has potential use in specifications and classifications of engine lubricating oils, such as Specification D4485.

6. Apparatus

6.1 *Balances:*

6.1.1 *Analytical Balance*—Capable of weighing 200 g with a minimum indication resolution of 0.1 g.

6.1.2 *Analytical Balance*—Capable of weighing 0.1 g with a minimum indication resolution of 0.001 g.

6.2 *Fume Hood,* that vents to the outside atmosphere (see Section 8).

6.3 *Reaction Vessel* (ACE Glass, Inc. part number D120676),[6,7] a 1 L, thick-walled glass vessel having a nominal 100 mm inner diameter and with a bottom, sample/drain valve. The lower half has an Instatherm[8,7] coating, rated at approximately 400 W, for heating the test mixture. A diagram is shown in Fig. A1.1.

6.4 *Vessel Head*—The vessel head is a stainless steel plate of sufficient diameter to completely cover the lower glass vessel and provide ample material for a sturdy mounting system. Reimel Machine, Inc. part number RMI-1002-DH[9,7] has been shown to be suitable for this application. The vessel head may also be constructed as described in Annex A2. Users may also source some parts from Reimel Machine, Inc. and some in-house. Ensure the plate has a center hole for an agitator shaft and threaded ports to allow filling and for the attachment of air/nitrogen dioxide lines, vacuum control and relief valves, and a temperature probe. Fig. A2.1 defines the locations of these ports. Mill the bottom surface of this stainless steel plate

[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 SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, http://www.sae.org.

[5] Kinker, B. G., Romaszewski, R. A., and Palmer, P. A., "ROBO–A Bench Procedure to Replace Sequence IIIGA Engine Test," *Journal of ASTM International (JAI)*, Vol 4, No. 10, 2007, Paper ID JAI 100916. Available online from www.astm.org.

[6] The sole source of supply of the apparatus known to the committee at this time is Ace Glass, Inc., P.O. Box 688, 1430 NW Blvd., Vineland, NJ 08362-0688.

[7] If you are aware of alternative suppliers, please provide this information to ASTM. Your comments will receive careful consideration at a meeting of the responsible technical committee[1] which you may attend.

[8] Instatherm is a registered trademark of Ace Glass, Inc., P.O. Box 688, 1430 NW Blvd., Vineland, NJ 08362-0688.

[9] The sole source of supply of the apparatus known to the committee at this time is Reimel Machine, Inc., 2575 Wyandotte Rd., Willow Grove, PA 19090.

to accept a polytetrafluoroethylene (PTFE) ring seal for centered attachment of the glass vessel as described in Annex A3. Reimel Machine, Inc. part number RMI-1007-DH[9,7] has been found suitable for this purpose.

6.5 *Stirrer Motor*—An electric motor with drill chuck collet capable of sustained operation at 200 r/min ± 5 r/min.

6.6 *Stirrer*—An 8 mm diameter stainless steel rod, 30 mm long with a means of attaching a blade assembly at the bottom. The turbine blade assembly diameter is 2.58 in. (65.5 mm) with 1.4 mm thick blades attached at a 45° pitch with an overall blade height of 0.985 in. (25.0 mm). Construct the stirrer as described in Annex A4. Reimel Machine, Inc. part number RMI-1001-DH[9,7] has been found suitable for this purpose. Attach the stirrer to the reactor head by means of a packing gland constructed as described in Annex A5. Reimel Machine, Inc. part number RMI-1004-DH[9,7] has been found suitable for this application. Attach the stirrer to the stirrer motor by inserting the 8 mm steel rod through the opening in the reactor head and the packing gland, and insert PTFE rope packing to create a seal. Position the blade 6 mm from the bottom of the vessel.

6.7 *Air Supply System*—Capable of delivering an uninterrupted flow of dry air into the test oil via a subsurface feed throughout the reaction time period. An in-line, desiccant-charged, drying system has been found suitable. Ensure the subsurface feed tube opening remains below the surface of the test fluid for the duration of the test.

NOTE 1—As the amount of test oil remaining at the end of the test is not always known at the beginning of the test, it is advisable to configure the dry-air tube location such that the opening of the tube is as close to the agitator and as close to the bottom of the reactor as practical (without contacting the agitator or blocking the tube opening).

6.8 *Graduated Tube* (Ace Glass, Inc., part number D120677),[6,7] 12 mL capacity, with 0.1 mL graduations and having appropriate provisions for connection to the reaction vessel's subsurface gas delivery system—see Annex A6 for more details. By receiving liquid phase nitrogen dioxide from a gas bottle, this tube allows measurement of nitrogen dioxide depletion from the tube over the course of the reaction.

6.9 *Temperature Control System*—A controller and probe capable of being programmed to control reaction temperature via low output wattage at or below 40 V ac and with an operational hysteresis of 0.1 °C using an on/off algorithm. Alternatively, a proportional-integral-derivative (PID) algorithm may also be used. Position the temperature probe tip so that it is level with the bottom of the turbine blade with a distance of 8 mm between the probe center and the blade edge.

6.9.1 As the temperature may not be uniform throughout the reactor, it is important from the point of view of precision that the temperature is always monitored and controlled at the specified position inside the reactor. When reassembling the reactor for a new run, reposition the probe, if necessary, as it is easily bent.

6.10 *Flow Meters:*

6.10.1 *Acrylic Block Airflow Meter* (King Instrument Co., 7520 Series, Order number 2C-17),[10,7] having a scale of 0.4 to 4 Standard Cubic Feet per Minute (SCFM), with ¼-in. NPT threaded female pipe end. It is used for measuring air flow in 10.3.2. The machined fitting for the top of the flow meter shall accommodate the vacuum line from the condenser to the reactor with a ⅜-in. inside diameter or larger. The machined fitting for the bottom of the flow meter shall accommodate the ¼-in. vacuum control valve.

NOTE 2—SCFM is the volumetric flow rate of a gas corrected to *standardized* conditions of temperature, pressure, and relative humidity, thus representing a precise mass flow rate. However, the definitions of *standard* conditions vary. In this method, the flow meter is calibrated with air at *standard* conditions defined as a temperature of 70°F, a pressure of 14.6 psia and 0 % relative humidity.

6.10.2 *Airflow Meter,* with a scale calibrated in mL/min for measuring subsurface airflow of 185 mL/min in 10.3.1 and 10.3.2.

6.11 *Vacuum System*—A pump with a free air capability of at least 160 L/min is required to ensure a constant air flow across the reaction surface in the vessel of 2.0 ± 0.1 SCFM with 61 kPa vacuum for 40 h. Instructions for constructing the vacuum plumbing for the vessel are given in Annex A7. As explained in Annex A7, it is critical to follow these instructions precisely.

6.12 *Vacuum Control Valve*—A stainless steel needle valve with ¼-in. outside diameter tube connections and a flow coefficient (Cv) of 0.37. A McMaster-Carr Supply Company needle valve part number 45585K86[11, 7] has been found suitable for this application.

6.13 *Vacuum Trap System*—Supplies coolant at an inlet temperature < 20 °C to the vacuum trap condensers in order to remove vapors from the effluent prior to entering (and possibly damaging) the vacuum system and has a means of recovering the distillate for weighing. Redundant (serial) condensers are beneficial as long as the required airflow across the reaction surface is maintained. Annex A8 provides information on two systems that have been found to be satisfactory.

6.14 *Time Controller*—A timing device accurate to 1 min is used to deactivate the heat source.

6.15 *Precision Needle Valve*—Having a low Cv for precise control of the flow of nitrogen dioxide. Examples of valves that have been found satisfactory are given in Appendix X3.

6.16 *Beaker*—300 mL capacity.

6.17 *Glass Jar*—250 mL capacity which can be sealed.

6.18 *Shaker*—Use either a reciprocal or an elliptical shaker.

6.19 *Assembled ROBO Apparatus*—Fig. X4.1 shows an example of an assembled ROBO apparatus. However, because it is assembled from different components, some of which are site specific (e.g., geometry of fume hood, local safety considerations, use of different parts such as temperature

[10] The sole source of supply of the apparatus known to the committee at this time is King Instrument Co., 12700 Pala Drive Garden Grove, CA 92841.

[11] The sole source of supply of the apparatus known to the committee at this time is McMaster-Carr Supply Company, P.O. Box 740100, Atlanta, GA 30374-0100.