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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

This test method is written for use by laboratories that make use of ASTM Test Monitoring Center (TMC)[2] services (see Annex A1 – Annex A4).

The TMC provides reference oils, and engineering and statistical services to laboratories that desire to produce test results that are statistically similar to those produced by laboratories previously calibrated by the TMC.

In general, the Test Purchaser decides if a calibrated test stand is to be used. Organizations such as the American Chemistry Council require that a laboratory utilize the TMC services as part of their test registration process. In addition, the American Petroleum Institute and the Gear Lubricant Review Committee of the Lubricant Review Institute (SAE International) require that a laboratory use the TMC services in seeking qualification of oils against their specifications.

The advantage of using the TMC services to calibrate test stands is that the test laboratory (and hence the Test Purchaser) has an assurance that the test stand was operating at the proper level of test severity. It should also be borne in mind that results obtained in a non-calibrated test stand may not be the same as those obtained in a test stand participating in the ASTM TMC services process.

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 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] Until the next revision of this test method, the ASTM Test Monitoring Center will update changes in the test method by means of information letters. Information letters may be obtained from the ASTM Test Monitoring Center, ~~6555 Penn Ave., Pittsburgh, PA 15206-4489. Attention: Administrator.~~203 Armstrong Drive, Freeport, PA 16229, www.astmtmc.org. Attention: Director. This edition incorporates revisions in all information letters through No. ~~20-1.~~21-1.

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

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

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]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
D4175 Terminology Relating to Petroleum Products, Liquid Fuels, 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 –10 °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] 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.

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

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

3.1.3.1 *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 °C 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.

5.4 The results of this test method are valid when seeking qualification of oils against published specifications only when run on a test stand that has successfully met the calibration requirements specified under the TMC's ROBO test monitoring program.

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).

[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.

6.3 *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. A5.1.

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 A6. 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. A6.1 defines the locations of these ports. Mill the bottom surface of this stainless steel plate to accept a polytetrafluoroethylene (PTFE) ring seal for centered attachment of the glass vessel as described in Annex A7. 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, 300 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 A8. Reimel Machine, Inc. part number RMI-1001-DH[9,7] has been found suitable for this purpose.

6.6.1 Attach the stirrer to the reactor head by means of a packing gland constructed as described in Annex A9. Reimel Machine, Inc. part number RMI-1004-DH[9,7] has been found suitable for this purpose. 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.

6.6.2 Position the blade 6 mm from the bottom of the vessel.

6.7 *Air Supply System*—~~Capable~~ A gas source 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.

6.7.1 Ensure the subsurface feed tube opening remains below the surface of the test fluid for the duration of the test. Do not place the tube in the drain area of the reaction flask.

6.7.2 A second gas source consisting of a gas cylinder containing dilute nitrogen dioxide in air may be added along with a valve to switch between the two gas sources. The volume fraction of nitrogen dioxide in air needed is 1.13 %. See Appendix X6 for how this is derived. The volume fraction as certified by the supplier must fall in the range of 1.07 % to 1.19 %.

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*~~ *Nitrogen Dioxide Delivery System*— ~~(Ace Glass, Inc., part number D120677),~~There are two options : ~~12 mL capacity, with 0.1 mL graduations and having appropriate provisions for connection to the reaction vessel's subsurface gas delivery system—see~~ for adding nitrogen dioxide. One uses ~~Annex A10 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.~~ liquid nitrogen dioxide and the other uses dilute nitrogen dioxide in air.

6.8.1 *Graduated Tube for Liquid Nitrogen Dioxide* (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 A10 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. This graduated tube is only used for the liquid nitrogen dioxide option.

[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.

6.8.2 *Gas Cylinder Containing Dilute Nitrogen Dioxide in Air*—Second gas source using dilute nitrogen dioxide in dry air as defined in 6.7.2. This is only used for the dilute nitrogen dioxide option.

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. Two air flow meters may be used in the dilute nitrogen dioxide configuration depending on the location of the switching valve.

6.10.2.1 A digital mass flow controller may also be used to measure and control the flow rate. This type of flow controller is recommended, but not required, for the dilute nitrogen dioxide in air option.

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 SCFM ± 0.1 SCFM with 61 kPa vacuum for 40 h. Instructions for constructing the vacuum plumbing for the vessel are given in Annex A11. As explained in Annex A11, 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 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 A12 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. This valve is used with the liquid nitrogen dioxide option in 6.8.1. It is not required for the dilute nitrogen dioxide option described in 6.8.2.

6.16 *Beaker*—300 mL capacity.

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

[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.

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 (for example, geometry of fume hood, local safety considerations, use of different parts such as temperature controllers, and so forth), there is no standard ROBO apparatus assembly. As an aid to building and setting up a new ROBO apparatus, a package of information is available on the TMC website.[2] This (non-mandatory) information supplements that given in Section 6. An index to the contents of this information package is given in Appendix X5.

7. Reagents and Materials

7.1 ~~*Liquid* *Nitrogen* *Dioxide*—~~*Dioxide:* ~~Produces a reddish-brown gas with a pungent odor. (**Warning—**VERY TOXIC if inhaled or ingested. Explosive if mixed with combustible material. Irritating to eyes and respiratory system. Danger of very serious irreversible health effects.)~~

7.1.1 *Liquid Nitrogen Dioxide (Used with the Option in 6.8.1)*—Produces a reddish-brown gas with a pungent odor. (**Warning—**VERY TOXIC if inhaled or ingested. Explosive if mixed with combustible material. Irritating to eyes and respiratory system. Danger of very serious irreversible health effects.).

7.1.2 *Dilute Nitrogen Dioxide in Air (Used with the Option in 6.8.2)*—(**Warning—**Compared to liquid nitrogen dioxide, the exposure risk is greatly reduced, but not negligible.)

7.2 *Iron Ferrocene*—98 % or higher purity. (**Warning—**Do not breathe dust. Harmful if swallowed.)

7.3 *Oil*—100 Neutral, API Group II, for mixing with iron ferrocene catalyst.

7.4 *Cleaning Solvent*—Commercial heptanes, or similar solvents that evaporate without leaving a residue, are suitable. (**Warning—**flammable.)

7.5 *Acetone*—A technical grade of acetone is suitable provided it does not leave a residue upon evaporation. This is used for a final cleaning rinse. Acetone will degrade fluoroelastomer seals and can dissolve or deteriorate acrylics. (**Warning—**flammable.)

7.6 *Dry Air*—Desiccated air is suitable.

7.7 *Reference Oils*—The current TMC reference oils are required for setting up the ROBO apparatus test stand (see Section 9). The TMC[2] maintains and distributes these oils. These oils are formulated or selected to represent specific chemical types or performance levels, or both. See A2.4 for additional information regarding reference oils.

7.7.1 The TMC is responsible for managing a system that ensures the performance and formulation consistency of the reference oils. Store the reference oils in locations where the ambient temperature does not exceed 32 °C. Under these conditions, the expected shelf life of a reference oil is five years. In some circumstances, however, the TMC may specify a shelf life longer than five years. In such cases, the TMC uses documented analysis procedures to justify the longer shelf life.

7.7.2 Unless specifically authorized by the TMC, do not analyze TMC reference oils, either physically or chemically. The testing laboratory tacitly agrees to use the TMC reference oils exclusively in accordance with the TMC's published Policies for Use and Analysis of ASTM Reference Oils, and to run and report the reference oil test according to TMC guidelines.

NOTE 3—Policies for the Use and Analysis of ASTM Reference Oils are available from the TMC.[2]

8. Hazards

8.1 *Specific Hazards*—Due to nitrogen dioxide toxicity, with the exception of weighing, perform steps 10.3 – 10.8 of the procedure in the fume hood. See also 7.1.

9. New and Existing Test Stand Calibration

9.1 *New Test Stand at New Test Laboratory Calibration*—For new ROBO apparatus at existing laboratory, proceed to 9.2. For existing ROBO apparatus test stands, proceed to 9.3.

9.1.1 Obtain the required, current reference oils from the TMC for the purpose of setting up a new ROBO apparatus stand.[2] (See 7.7.2 and Annex A2 for conditions of use for the TMC reference oils.)

9.1.2 Test the assigned reference oils according to the procedure described in Section 10.

9.1.2.1 It is imperative that the vacuum control valve (VCV) set position be set on the first set-up test and not changed again for subsequent set-up qualifying runs.

9.1.2.2 If the VCV set position is changed by more than ±0.125 revolutions after the start of the first qualifying set-up test run, all previous tests in the set-up test sequence are void; repeat the test stand setup runs from 9.1.1 – 9.1.4.

9.1.3 Determine the viscometric properties of the aged reference oils as described in Section 12 and report according to Section 13.

9.1.4 Report test results to the TMC using the standardized reporting protocols (see 9.3.3 and Section 13). Be sure to include all required operational parameters as defined in the reporting protocol data dictionary.

9.1.5 Review all initial set-up results on new instruments and receive approval from the TMC.

9.1.5.1 Test results will be posted to the TMC website. Lab identification will be coded by the TMC for confidentiality of the testing laboratory.

9.1.6 If all the required test stand set-up runs meet the current, approved ROBO TMC calibration requirements[11] (both operationally and statistically), the TMC will notify the laboratory that it can proceed with calibrating the test stand per 9.3.

9.1.7 If the TMC's review determines that the required test stand set-up runs do not collectively meet the approved requirements (both operationally and statistically), the TMC will notify the laboratory that additional adjustments need to be made to the test stand and one or more of the set-up runs will have to be repeated.

9.2 *New Test Stand at Existing Laboratory Calibration:*

9.2.1 Laboratory can proceed with calibrating the test stand per 9.3.

9.3 *Existing Test Stand Calibration:*

9.3.1 *Reference Oil Test Frequency*—The TMC requires test stands to pass periodic calibration verification with reference oils supplied by the TMC. These calibration verification runs are typically run on blind-coded reference oil samples.

9.3.1.1 Prior to conducting a TMC reference oil test for the purpose of stand calibration, procure a supply of reference oil directly from the TMC. (See 7.7.2 and Annex A2 for conditions of use for the TMC reference oils.) The reference oils are usually supplied directly to a testing laboratory with blind-coded identification numbers to ensure that the laboratory is not influenced by prior knowledge of a reference oil's acceptable performance results in assessing the test results. The TMC will determine which specific reference oil or oils the laboratory shall test in accordance with the calibration requirements.

9.3.1.2 Initial calibration verification of a new test stand or repeated consecutive unacceptable calibration verifications on a test stand requires passing two consecutive TMC reference oil tests.

9.3.1.3 The same nitrogen dioxide delivery configuration must be used to re-verify the calibration status and then continued to be used for subsequent certified runs.

[11] The ROBO LTMS Calibration Requirements document is available at: http://www.astmtmc.org/ftp/docs/ltms/ltms.pdf.

9.3.1.4 Certain operational changes to the test stand, as specified in the TMC calibration requirements,[11] voids the TMC test stand calibration status and requires passing two consecutive TMC reference oil tests to re-verify the calibration status of the modified test stand.

9.3.1.5 During the time of conducting a reference oil test on one test stand, non-reference oil tests may be conducted on other previously calibrated stands.

9.3.2 *Test Numbering:*

9.3.2.1 The test number shall follow the format *AAA-BB-CCCC.AAA* represents the test stand identification. *BB* represents the number of tests since last reference. *CCCC* represents the total number of tests on the stand. As an example, 6-10-175 represents the 175 test on Stand 6 and the tenth test since the last reference. Consecutively number all tests on a given stand.

9.3.3 *Reporting of Reference Oil Test Results*—Report the results of all reference oil tests to the TMC according to the following instructions:

9.3.3.1 Transmit results according to the ROBO Standardized Report Forms and Data Dictionary[12] to the TMC within five days of test completion via electronic data transfer protocol as outlined in the Data Communication Committee, Electronic Test Report Transmission Model (ETRTM).[13]

NOTE 4—Be sure to collect data on all the required parameters defined in the ROBO Standardized Data Dictionary[12] (see Section 13). Validity evaluation of test results cannot be made if critical evaluation parameters are missing.

9.3.4 *Evaluation of Reference Oil Test Results*—The TMC evaluates the reference oil test results for both operational validity and statistical acceptability. The TMC may consult with the test laboratory in case of difficulty, as follows:

9.3.4.1 Upon receipt of the reference oil test results from the test laboratory, the TMC evaluates the laboratory's reported operational parameters for compliance with the current test method. For operationally valid tests, the TMC then evaluates the pass/fail parameters for statistical validity. The TMC sends a test confirmation report to the test laboratory indicating the overall validity of the calibration test results, and disclosing the non-blind industry reference oil code.

9.3.4.2 In the event the reference oil test is unacceptable, the test laboratory shall provide an explanation of the problem relating to the failure. If the problem is not obvious, carry out operational re-checks (instrumentations, settings, and procedures). Following the re-checks, the TMC assigns another reference oil for testing by the laboratory. If this reference oil test is unacceptable, a reassessment of the stand setup as described in 9.1 or 9.2 may be necessary.

9.3.4.3 It is recognized that a certain percentage of calibration tests will fall outside the acceptance limits because of the application of statistics in the development of the acceptance limits. The TMC decides, with consultation as needed with industry experts (testing laboratories, members of the ASTM Technical Guidance Committee, the surveillance panel, and so forth), whether the reason for any failure of a reference oil test is a false alarm, testing apparatus, testing laboratory, or industry-related problem. The ROBO surveillance panel adjudicates all industry problems.

9.3.5 *Reference Oil Accountability:*

9.3.5.1 Laboratories conducting calibration tests are required to provide a full accounting of the identification and quantities of all reference oils used.

9.3.5.2 With the exception of analysis required in this test method, no additional physical or chemical analysis of new or used reference oils is permitted without the express permission of the TMC. (See 7.7.2 and Annex A2 for conditions of use for the TMC reference oils.)

10. Procedure

10.1 *Vacuum Control Valve Setting*—For a new ROBO apparatus test stand, set the vacuum control valve as described in Annex

[12] The ROBO Standardized Report Forms and Data Dictionary specification is available at: ftp://ftp.astmtmc.org/datadict/robo/current/.

[13] The Data Communication Committee, Electronic Test Report Transmission Model (ETRTM) document is available at: ftp://ftp.astmtmc.org/docs/datacommunicationscommittee/electronic_test_report_transmission_specification/.

A13. The control valve setting is critical as it affects the severity of the test. For all subsequent runs involving test oils, use exactly the same control valve setting to that used during the last successful TMC calibration verification run.

10.2 *Catalyst Preparation:*

10.2.1 Weigh 0.1 g ± 0.001 g of iron ferrocene (see warning in 7.2) into an appropriate container such as a 250 mL glass jar.

10.2.2 Add 99.9 g ± 0.1 g of API Group II 100 Neutral oil to obtain 0.100 % ± 0.001 % (mass) iron ferrocene.

10.2.3 Mix thoroughly, until the catalyst is completely in solution as determined by a lack of visible particles.

NOTE 5—This may take 1 h or more.

10.3 *Vessel Seal Check:*

10.3.1 Start subsurface dry-air flow at a rate of 185 mL/min.

10.3.2 On an assembled vessel, install the acrylic block flow meter between the top connection of the vacuum control valve and the vacuum source. Apply vacuum to the vessel and block the vacuum relief orifice long enough to assure the system will attain 85 kPa with a subsurface airflow of 185 mL/min.

10.3.2.1 The acrylic block air flow meter shall read less than 0.6 SCFM.

10.4 *Preset Vacuum Flow*—With the vacuum still applied to the vessel, set the air flow through the reactor to 2.0 SCFM ± 0.1 SCFM by bleeding air, if needed, into the vacuum line between the vacuum source and the condenser. Maintain the vacuum pressure at 61 kPa ± 1.7 kPa by adjusting the vacuum relief valve. Once these parameters are set, shut off the vacuum and remove the acrylic block flow meter from the system.

10.5 *Sample Preparation and Charging Nitrogen Dioxide:*

NOTE 6—Steps 10.5.1 – 10.5.3 may be carried out in any order or simultaneously.

10.5.1 *Sample Preparation*—Introduce 3.0 g ± 0.1 g of prepared iron ferrocene catalyst solution and 197.0 g ± 1.0 g test oil to the reaction vessel. See Appendix X1 for suggested mixing procedures. If the direct weighing procedure (X1.1.2) is used, do the vessel seal check (10.3) and the preset vacuum flow (10.4) procedure after the apparatus is reassembled.

NOTE 7—The total mass of oil in the reactor is 200 g ± 1.0 g (197.0 g ± 1.0 g from the test oil and 3.0 g from the catalyst solution).

10.5.1.1 Start the stirrer motor and agitate at 200 r⁄min ± 5 r⁄min.

10.5.2 Make the electrical connections to the heater. (**Warning—**To avoid electric shock and possible ignition spark, check that the power is de-energized before making electrical connections.)

10.5.3 *Charging Nitrogen ~~Dioxide—~~Dioxide:* ~~Transfer 2.0 mL ± 0.1 mL of liquid nitrogen dioxide (see Section 8 and warning in 7.1) into the graduated tube. See Appendix X2 for examples of how the transfer may be made.~~

10.5.3.1 *Liquid Nitrogen Dioxide Option Only*—Transfer 2.0 mL ± 0.1 mL of liquid nitrogen dioxide (see Section 8 and warning in 7.1) into the graduated tube. See Appendix X2 for examples of how the transfer may be made.

10.5.3.2 *Dilute Nitrogen Dioxide Option Only*—The amount of nitrogen dioxide introduced can be calculated. An amount equivalent to 2.0 mL ± 0.1 mL of liquid nitrogen dioxide is required. See Appendix X6 for example calculation.

10.6 *Oil Aging:*

10.6.1 *General*—Begin the oil aging by setting the time and temperature and turning on the vacuum.

10.6.1.1 Complete steps 10.6.2 – 10.6.5 within 1 min; the order in which they are carried out is not important.

10.6.2 Set the time controller to 40 h to initiate the oil aging.

10.6.3 Set the temperature controller to 170 °C and commence heating.

10.6.4 Adjust the temperature controller voltage output to 25 V to 40 V.

10.6.5 Turn the vacuum system on.

10.6.6 ~~Immediately after the previous steps, adjust the nitrogen dioxide precision needle valve to allow introduction of nitrogen dioxide in a controlled and gradual manner into the inlet flow stream. Ensure that the nitrogen dioxide is completely depleted from the tube and introduced into the reactor within 12 h ± 1 h.~~Start the nitrogen dioxide flow.

10.6.6.1 For the liquid nitrogen dioxide option, immediately after the previous steps, adjust the nitrogen dioxide precision needle valve to allow introduction of nitrogen dioxide in a controlled and gradual manner into the inlet flow stream. Ensure that the nitrogen dioxide is completely depleted from the tube and introduced into the reactor within 12 h ± 1 h.

10.6.6.2 Because changes to the nitrogen dioxide flow rate can affect precision, it is imperative that nitrogen dioxide be introduced to the reactor in a controlled and gradual manner. Using a flow rate target of 0.167 mL/h, monitor nitrogen dioxide depletion closely in the first 2 h to 4 h, the aim being to introduce 0.5 mL during that time period. Introduce the remaining 1.5 mL at a similar flow rate, ensuring that the total of 2.0 mL is delivered between 11 h and 13 h. A run is invalid if the flow of nitrogen dioxide exceeds 0.5 mL during any 1 h period.

10.6.6.3 For the dilute nitrogen dioxide option, switch to dilute nitrogen dioxide for 12.0 h. A run is invalid if the flow of dilute nitrogen dioxide in air deviates from the required 185 mL/min by more than 6 % during at any of the observations. At least 6 observations during the first 6 h of the air flow must be made and recorded with the last observation being made at about 6 h. The air flow may be adjusted at these times. If all of the readings before adjustments are within 5 % of 185 mL/min, then no more observations are required. If the air flow deviates by more than 4 % during the first 6 h, then six more observations are required from hours 6 to 12. After 12.0 h, switch back to the dry-air supply for the remainder of the test.

10.6.6.4 If any deviations from 185 mL/min of more than 2 mL/min were observed (or calculated at the 12 h switching time), then calculate and report the time-averaged flow rate. See Appendix X7 for examples.

10.7 *Shutdown:*

10.7.1 At the end of the 40 h cycle, allow the system to cool to room temperature while maintaining the airflow and agitation.

10.7.2 Turn off the vacuum. (The vacuum flow can be turned off at any time after completion of the 40 h cycle.) Bleed the pressure by opening a port, for example, the sample addition port. Drain the aged oil into a suitable container.

10.8 *Mass Percent Volatiles Collected:*

10.8.1 Drain the condensed liquid from the vacuum trap system into a tared vessel. Determine and record the mass of the condensed liquid to the nearest 0.1 g.

10.8.2 Calculate as follows:

$$\text{Mass \% volatiles, \% m/m} = 100\,\frac{\text{M(volatiles)}}{\text{M(fresh)}} \tag{1}$$

where:

M(fresh) = 200 g = the mass of fresh oil added to the reactor in 10.5.1, and
M(volatiles) = mass, g, of condensate collected in 10.8.1.

NOTE 8—The significance of the % volatiles parameter is under investigation.

11. Cleaning

11.1 Clean the reaction vessel with cleaning solvent (see warning in 7.4).

11.1.1 Scrub any residual material off the glass surface while taking care not to scratch the inside of the vessel. Perform a final rinse with acetone (see warning in 7.5).

11.2 Clean the vacuum control valve.

11.2.1 Flush the valve with cleaning solvent or carburetor cleaner, followed with an acetone rinse to remove and avoid any carbon deposits that could reduce or plug the valve orifice.

11.2.2 Additional optional cleaning may be needed in cases where there is insufficient vacuum flow (see 10.4). If vacuum flow is sufficient, skip to step 11.3.

11.2.2.1 Disassemble the valve and remove any carbon deposits from the plug and inside seat of the valve body.

11.2.2.2 Flush as in 11.2.1.

11.2.2.3 Reassemble the vacuum control valve, ensuring that the valve setting is at exactly the same position to that used during the last successful TMC calibration verification run.

11.3 Clean the underside of the reactor cap and all shafts or probes protruding downward into the vessel with cleaning solvent and a lightweight, lint-free towel. Rinse with acetone.

11.4 Ensure that subsurface air supply lines are clear, then clean them with cleaning solvent and reassemble when dry.

11.5 Clean the acrylic block flow meter with cleaning solvent. Do not use acetone which can dissolve or deteriorate acrylics.

12. Calculations and Determination of Test Results

12.1 *Increase in Kinematic Viscosity at 40 °C:*

12.1.1 Calculate as follows:

$$\text{Percent viscosity increase (PVIS)} = 100\,\frac{[\text{KV(aged)} - \text{KV(fresh)}]}{\text{KV(fresh)}} \tag{2}$$

where:

KV(aged) = kinematic viscosity, mm^2/s, at 40 °C of the aged oil as determined by Test Method D445, and
KV(fresh) = kinematic viscosity, mm^2/s, at 40 °C of the fresh oil as determined by Test Method D445.

12.2 *Low-Temperature Viscometric Properties:*

12.2.1 Using Test Method D5293, measure the Cold Cranking Simulator (CCS) viscosity of the ROBO-aged oil at the temperature specified for the SAE W grade of the fresh oil. This temperature can be found in the SAE J300 Viscosity Classification System (hereafter referred to as SAE J300).

12.2.1.1 If the measured CCS viscosity is less than or equal to the maximum CCS viscosity specified in SAE J300 for the SAE W grade of the fresh oil, measure the MRV viscosity by Test Method D4684 at the MRV temperature specified in SAE J300 for the SAE W grade of the fresh oil.

12.2.1.2 If the measured CCS viscosity is higher than the maximum CCS viscosity specified in SAE J300 for the SAE W viscosity grade of the fresh oil, measure the MRV viscosity by Test Method D4684 at 5 °C higher than the MRV temperature specified in SAE J300 for the original SAE W viscosity grade of the fresh oil (that is, at the MRV temperature specified in SAE J300 for the next higher SAE W viscosity grade).

TABLE 1 Test Precision[A]

Variable	Intermediate Precision		Reproducibility	
	$S_{i.p.}$[B]	i.p.[C]	S_R[B]	R[C]
PVIS[D]	0.191	0.535	0.267	0.748
MRV viscosity[D]	0.25	0.70	0.40	1.12

[A] These statistics are based on results obtained from an interlaboratory program in which seven samples were tested in seven laboratories on ten test rigs (see 14). The samples consisted of SAE 5W-XX and 10W-30 multigrade engine oils including ASTM Test Monitoring Center Reference Oils 434, 435, and 438.
[B] S = Standard deviation.
[C] This value is obtained by multiplying the standard deviation by 2.8.
[D] The original units for PVIS are percent viscosity increase. The original units for MRV viscosity are mPa·s. These parameters are transformed using ln(result). When comparing two test results on these parameters, first apply this transformation to each test result. Compare the absolute difference between the transformed results with the appropriate (intermediate precision or reproducibility) precision limit.

13. Report

13.1 *Report Forms*—For TMC reference oil tests, use the standardized report form set and data dictionary.

NOTE 9—Report the non-reference oil test results on these same forms if the results are intended to be submitted as candidate oil results against a specification.

13.1.1 Report reference oil test results to the TMC according to the ETRTM protocols described in 9.3.3.1.

13.2 *Reporting Units*—Report results in SI units.

13.3 Report the following:

13.3.1 Kinematic viscosity at 40 °C, by Test Method D445, of the test oil before and after aging.

13.3.1.1 Report to two decimal places for viscosities between 10 mm^2/s and 100 mm^2/s and to one decimal place for viscosities >100 mm^2/s.

13.3.2 Percent increase in kinematic viscosity at 40 °C after aging (PVIS)—see 12.1.

13.3.2.1 Report to nearest 0.1 %.

13.3.3 SAE W grade of the fresh oil.

13.3.4 The CCS viscosity and temperature of measurement of the ROBO-aged oil by Test Method D5293.

13.3.5 The MRV viscosity, yield stress and temperature of measurement of the aged oil by Test Method D4684—see 12.2.1.1 and 12.2.1.2.

13.3.6 The option used to add nitrogen dioxide. Liquid nitrogen dioxide or dilute nitrogen dioxide.

13.3.6.1 If the dilute nitrogen dioxide option was used, calculate and report the total amount of nitrogen dioxide delivered to the reactor to the nearest one-tenth of a milliliter.

14. Precision and Bias[14]

14.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory tests results is given in Table 1.

[14] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1660. Contact ASTM Customer Service at service@astm.org.