

Test Monitoring Center

@ Carnegie Mellon University 6555 Penn Avenue, Pittsburgh, PA 15206, USA http://astmtmc.cmu.edu 412-365-1000

ROBO Information Letter 16-1 Sequence No. 1 March 11, 2016

ASTM consensus has not been obtained on this information letter. An appropriate ASTM ballot will be issued in order to achieve such consensus.

TO: ROBO Mailing List

SUBJECT: Numerous Revisions to ROBO Test Method D7528

The ROBO Surveillance Panel has approved numerous revisions to the ROBO Test Method D7528. The revisions are attached. Of special note, major revisions have been made to sections 9 and 10, and Annexes A1 through A4 have been added. The test method now requires test labs to use TMC services in order to conduct the test method in a valid manner when seeking qualification of oils against published specifications (refer to attached Introduction and Section 5.4). These changes are effective with the date of this information letter.

Also, at a June 23, 2014 meeting, ASTM Section D02.B0.10 on Standards Acceleration approved standardized wording describing the role of the Test Monitoring Center. Subcommittee B has requested that the TMC incorporate this wording into all test methods through the information letter system. D7528 has been revised to incorporate this wording.

Alar Hanley

Alan Flamberg Chair ROBO Surveillance Panel

Frank m Failer

Frank M. Farber Director ASTM Test Monitoring Center

Attachments

c: ftp://ftp.astmtmc.cmu.edu/docs/bench/robo/procedure_and_ils/IL16-1.pdf

Distribution: Electronic Mail

INTRODUCTION

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

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.

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.

6.6 *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-DH⁹.⁷ 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-DH⁹,⁷ 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.

6.6.2 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.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.

6.12 *Vacuum Control Valve*—A stainless steel needle valve with 1/4-in. outside diameter tube connections and a flow coefficient (Cv) of 0.37 has been found suitable for this application.

7.7 *Reference Oils*—The current TMC reference oils are required for setting up the ROBO apparatus test stand (see Section 9). The TMC² 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.

Replace current Section 9 in its entirety with the following. Rename Table 2 to Table 1 and change all table references accordingly.

9. New and Existing Test Stand Calibration

9.1 Set-up qualifying test runs for a New ROBO Apparatus Test Stand. For existing ROBO Apparatus Test Stands, proceed to Section 9.2.

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 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.

9.1.2.1.1 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 set-up runs from 9.1.1 through 9.1.4.

9.1.3 Determine the viscometric properties of the aged reference oils as described in Sections 12 and report according to Section13.

9.1.4 Report test results to the TMC using the standardized reporting protocols (See Sections 9.2.2 and 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

¹²(both operationally and statistically), the TMC will notify the laboratory that it can proceed with calibrating the test stand per section 9.2.

Add footnote 12 and renumber existing footnote 12 to 15.

¹² The ROBO TMC Calibration Requirements document is available at internet address: <u>ftp://ftp.astmtmc.cmu.edu/docs/bench/robo/procedure_and_ils/ROBO_TMC_Calibration_Requirements.pdf</u>

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 Existing Test Stand Calibration

9.2.1 *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.

9.2.1.1 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 A2. for conditions of use for the TMC reference oils.) The reference oils are usually supplied directly to a testing laboratory 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.

9.2.1.2 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.

9.2.1.3 Certain operational changes to the test stand, as specified in the TMC Calibration Requirements¹², 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.

9.2.1.4 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.

9.2.2 Test Numbering

9.2.2.1 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 10 test since the last reference. Consecutively number all tests on a given stand.

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

9.2.3.1 Transmit results according to the ROBO Standardized Report Forms and Data Dictionary¹³ to the TMC within 5 days of test completion via electronic data transfer protocol as outlined in the Data Communication Committee, Electronic Test Report Transmission Model (ETRTM)¹⁴.

Add footnote 13 and 14.

¹³ The ROBO Standardized Report Forms and Data Dictionary specification is available at internet address: ftp://ftp.astmtmc.cmu.edu/datadict/robo/current/

¹⁴ The Data Communication Committee, Electronic Test Report Transmission Model (ETRTM) document is available at internet address: ftp://ftp.astmtmc.cmu.edu/docs/datacommunicationscommittee/electronic_test_report_transmission_specification/

Insert new Note 4.

Note 4--Be sure to collect data on all the required parameters defined in the ROBO Standardized data dictionary² (see Section 13.) Validity evaluation of test results cannot be made if critical evaluation parameters are missing.

Renumber existing footnotes accordingly

9.2.4 *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:

9.2.4.1 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.

9.2.4.2 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 set-up, as described in 9.1 may be necessary.

9.2.4.3 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.

9.2.5 Reference Oil Accountability:

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

9.2.5.2 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 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 A9. 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.

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

Insert new Note 5, renumber existing notes 4 through 8 as notes 6 through 10.

Note 5: This may take one hour or more.

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 \pm 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 \pm 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:

10.5.1 Sample Preparation—Introduce (3.0 ± 0.1) g of prepared iron ferrocene catalyst solution and (197.0 ± 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.

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 ± 1) h. 10.6.6.1 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 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 and 13) h. A run is invalid if the flow of nitrogen dioxide exceeds 0.5 mL during any 1 h period.

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.

13. Report

13.1.1 Report reference oil test results to the TMC according to the ETRTM protocols described in 9.2.3.1.

Insert the following Annex A1 through A4 and renumber subsequent annexes.

ANNEXES

(Mandatory Information)

A1. ASTM TEST MONITORING CENTER ORGANIZATION

A1.1 *Nature and Functions of the ASTM Test Monitoring Center (TMC)*—The TMC is a non profit organization located in Pittsburgh, Pennsylvania and is staffed to: administer engineering studies; conduct laboratory inspections; perform statistical analyses of reference oil test data; blend, store, and ship reference oils; and provide the associated administrative functions to maintain the referencing calibration program for various lubricant tests as directed by ASTM Subcommittee D02.B0 and the ASTM Executive Committee. The TMC coordinates its activities with the test sponsors, the test developers, the surveillance panels, and the testing laboratories. Contact TMC through the TMC Director at:

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

A1.2 *Rules of Operation of the ASTM TMC*—The TMC operates in accordance with the ASTM Charter, the ASTM Bylaws, the Regulations Governing ASTM Technical Committees, the Bylaws Governing ASTM Committee D02, and the Rules and Regulations Governing the ASTM Test Monitoring System. A1.3 *Management of the ASTM TMC*—The management of the Test Monitoring System is vested in the Executive Committee elected by Subcommittee D02.B0. The Executive Committee selects the TMC Director who is responsible for directing the activities of the TMC.

A1.4 *Operating Income of the ASTM TMC*—The TMC operating income is obtained from fees levied on the reference oils supplied and on the calibration tests conducted. Fee schedules are established by the Executive Committee and reviewed by Subcommittee D02.B0.

A2. ASTM TEST MONITORING CENTER: CALIBRATION PROCEDURES

A2.1 *Reference Oils*—These oils are formulated or selected to represent specific chemical, or performance levels, or both. They are usually supplied directly to a testing laboratory under code numbers to ensure that the laboratory is not influenced by prior knowledge of acceptable results in assessing test

results. The TMC determines the specific reference oil the laboratory shall test.

A2.1.1 *Reference Oil Data Reporting*—Test laboratories that receive reference oils for stand calibration shall submit data to the TMC on every sample of reference oil they receive. If a shipment contains any missing or damaged samples, the laboratory shall notify the TMC immediately.

A2.2 Calibration Testing:

A2.2.1 Full scale calibration testing shall be conducted at regular intervals. These full scale tests are conducted using coded reference oils supplied by the TMC. It is a laboratory's responsibility to keep the onsite reference oil inventory at or above the minimum level specified by the TMC test engineers.

A2.2.2 *Test Stands Used for Non Standard Tests*—If a non standard test is conducted on a previously calibrated test stand, the laboratory shall conduct a reference oil test on that stand to demonstrate that it continues to be calibrated, prior to running standard tests.

A2.3 *Reference Oil Storage*—Store reference oils under cover in locations where the ambient temperature is between -10 °C and +50 °C.

A2.4 *Analysis of Reference Oil*—Unless specifically authorized by the TMC, do not analyze TMC reference oils, either physically or chemically. Do not resell ASTM reference oils or supply them to other laboratories without the approval of the TMC. The reference oils are supplied only for the intended purpose of obtaining calibration under the ASTM Test Monitoring System. Any unauthorized use is strictly forbidden. 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 results according to TMC guidelines. Additional policies for the use and analysis of ASTM Reference Oils are available from the TMC.

A2.5 *Conducting a Reference Oil Test*—When laboratory personnel are ready to run a reference calibration test, they shall request an oil code via the TMC website.

A2.6 *Reporting Reference Oil Test Results*—Upon completion of the reference oil test, the test laboratory transmits the data electronically to the TMC, as described in Section 13. The TMC reviews the data and contacts the laboratory engineer to report the laboratory's calibration status. All reference oil test results, whether aborted, invalidated, or successfully completed, shall be reported to the TMC.

A2.6.1 All deviations from the specified test method shall be reported.

A3. ASTM TEST MONITORING CENTER: MAINTENANCE ACTIVITIES

A3.1 Special Reference Oil Tests-To ensure continuous severity and precision monitoring, calibration

tests are conducted periodically throughout the year. Occasionally, the majority or even all of the industry's test stands will conduct calibration tests at roughly the same time. This could result in an unacceptably large time frame when very few calibration tests are conducted. The TMC can shorten or extend calibration periods as needed to provide a consistent flow of reference oil test data. Adjustments to calibration periods are made such that laboratories incur no net loss or gain in calibration status.

A3.2 Special Use of the Reference Oil Calibration System—The surveillance panel has the option to use the reference oil system to evaluate changes that have potential impact on test severity and precision. This option is only taken when a program of donated tests is not feasible. The surveillance panel and the TMC shall develop a detailed plan for the test program. This plan requires all reference oil tests in the program to be completed as close to the same time as possible, so that no laboratory/stand calibration status is left pending for an excessive length of time. In order to maintain the integrity of the reference oil monitoring system, each reference oil test is conducted so as to be interpretable for stand calibration. To facilitate the required test scheduling, the surveillance panel may direct the TMC to lengthen and shorten reference oil calibration status. To ensure accurate stand, or laboratory, or both severity assessments, conduct non reference oil tests the same as reference oil tests.

A3.3 *Donated Reference Oil Test Programs*—The surveillance panel is charged with maintaining effective reference oil test severity and precision monitoring. During times of new parts introductions, new or re blended reference oil additions, and procedural revisions, it may be necessary to evaluate the possible effects on severity and precision levels. The surveillance panel may choose to conduct a program of donated reference oil tests in those laboratories participating in the monitoring system, in order to quantify the effect of a particular change on severity and precision. Typically, the surveillance panel requests its panel members to volunteer enough reference oil test results to create a robust data set. Broad laboratory participation is needed to provide a representative sampling of the industry. To ensure the quality of the data obtained, donated tests are conducted on calibrated test stands. The surveillance panel shall arrange an appropriate number of donated tests and ensure completion of the test program in a timely manner.

A3.4 *Intervals Between Reference Oil Tests*—Under special circumstances, such as extended downtime caused by industry wide parts or fuel shortages, the TMC may extend the intervals between reference oil tests. Such extensions shall not exceed one regular calibration period.

A3.5 *Introducing New Reference Oils*—Reference oils produce various results. When new reference oils are selected, participating laboratories will be requested to conduct their share of tests to enable the TMC to recommend industry test targets. ASTM surveillance panels require a minimum number of tests to establish the industry test targets for new reference oils.

A3.6 *TMC Information Letters*—Occasionally it is necessary to revise the test method, and notify the test laboratories of the change, prior to consideration of the revision by Subcommittee D02.B0. In such a case, the TMC issues an Information Letter. Information Letters are balloted semi annually by Subcommittee D02.B0, and subsequently by D02. By this means, the Society due process procedures are applied to these Information Letters.

A3.6.1 *Issuing Authority*—The authority to issue an Information Letter differs according to its nature. In the case of an Information Letter concerning a part number change which does not affect test results, the TMC is authorized to issue such a letter. Long term studies by the surveillance panel to improve the test procedure through improved operation and hardware control may result in the issuance of an Information Letter. If obvious procedural items affecting test results need immediate attention, the test sponsor and the TMC issue an Information Letter and present the background and data supporting that action to the surveillance panel for approval prior to the semiannual Subcommittee D02.B0 meeting.

A3.7 *TMC Memoranda*—In addition to the Information Letters, supplementary memoranda are issued. These are developed by the TMC and distributed to the appropriate surveillance panel and participating laboratories. They convey such information as batch approvals for test parts or materials, clarification of the test procedure, notes and suggestions of the collection and analysis of special data that the TMC may request, or for any other pertinent matters having no direct effect on the test performance, results, or precision and bias.

A4. ASTM TEST MONITORING CENTER: RELATED INFORMATION

A4.1 *New Laboratories*—Laboratories wishing to become part of the ASTM Test Monitoring System will be requested to conduct reference oil tests to ensure that the laboratory is using the proper testing techniques. Information concerning fees, laboratory inspection, reagents, testing practices, appropriate committee membership, and rater training can be obtained by contacting the TMC Director.

A4.2 *Information Letters: COTCO Approval*—Authority for the issuance of Information Letters was given by the committee on Technical Committee Operations in 1984, as follows: "COTCO recognizes

that D02 has a unique and complex situation. The use of Information Letters is approved providing each letter contains a disclaimer to the affect that such has not obtained ASTM consensus. These Information Letters should be moved to such consensus as rapidly as possible."

A4.3 *Precision Data*—The TMC determines the precision of test methods by analyzing results of calibration tests conducted on reference oils. Precision data are updated regularly. Current precision data can be obtained from the TMC.