Designation: D7216 – 09

Standard Test Method for Determining Automotive Engine Oil Compatibility with Typical Seal Elastomers[1]

This standard is issued under the fixed designation D7216; 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 test method described in this standard. However, the ASTM Test Monitoring Center (TMC)[2] provides a reference oil (TMC 1006-1) and an assessment of the test results obtained with this oil and the reference elastomers. By these means, the laboratory will know whether their use of the test method gives results statistically similar to those obtained by other laboratories.

The TMC also use the reference oil results on different batches of elastomers from different laboratories to update continually the total and within-laboratory standard deviation estimates. Some specifications, for example, Specification D4485, use the updated TMC standard deviation estimates, pertaining at the time test oils are evaluated, to adjust specification limits for the effects of the industry test variability.

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 TMC services. Laboratories that choose not to use those services may simply ignore those portions of the test method that refer to the TMC.

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 this test method.

1. Scope

1.1 This test method covers quantitative procedures for the evaluation of the compatibility of automotive engine oils with several reference elastomers typical of those used in the sealing materials in contact with these oils. Compatibility is evaluated by determining the changes in volume, Durometer A hardness and tensile properties when the elastomer specimens are immersed in the oil for a specified time and temperature.

1.2 Effective sealing action requires that the physical properties of elastomers used for any seal have a high level of resistance to the liquid or oil in which they are immersed. When such a high level of resistance exists, the elastomer is said to be compatible with the liquid or oil.

NOTE 1—The user of this test method should be proficient in the use of Test Methods D412 (tensile properties), D471 (effect of rubber immersion in liquids), D2240 (Durometer hardness), and D5662 (gear oil compatibility with typical oil seal elastomers), all of which are involved in the execution of the operations of this test method.

1.3 This test method provides a preliminary or first order evaluation of oil/elastomer compatibility only. Because seals may be subjected to static or dynamic loads, or both, and they may operate over a range of conditions, a complete evaluation of the potential sealing performance of any elastomer-oil combination in any service condition usually requires tests additional to those described in this test method.

1.4 The several reference elastomer formulations specified in this test method were chosen to be representative of those used in both heavy-duty diesel engines and passenger-car spark-ignition engines (the latter are covered in Annex A2). The procedures described in this test method can, however,

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products 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 Avenue, Pittsburgh, PA 15206. (www.astmtmc.cmu.edu) Attention: Administrator. This edition incorporates revisions in all information letters through No. 07-1.

also be used to evaluate the compatibility of automotive engine oils with different elastomer types/formulations or different test durations and temperatures to those employed in this test method.

NOTE 2—In such cases, the precision and bias statement in Section 12 does not apply. In addition to agreeing acceptable limits of precision, where relevant, the user and supplier should also agree: *(1)* test temperatures and immersion times to be used; *(2)* the formulations and typical properties of the elastomers; and *(3)* the sourcing and quality control of the elastomer sheets.

NOTE 3—The TMC may also issue Information Letters on this matter.

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

1.6 *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.*

1.7 This test method is arranged as follows:

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D297 Test Methods for Rubber Products—Chemical Analysis

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D471 Test Method for Rubber Property—Effect of Liquids

D1193 Specification for Reagent Water

D1566 Terminology Relating to Rubber

D2240 Test Method for Rubber Property—Durometer Hardness

D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants

D4485 Specification for Performance of Engine Oils

D5662 Test Method for Determining Automotive Gear Oil Compatibility with Typical Oil Seal Elastomers

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E178 Practice for Dealing With Outlying Observations

2.2 *SAE Standard*[4]

SAE J2643 Standard Reference Elastomers (SRE) for Characterizing the Effect of Liquids on Vulcanized Rubbers

3. Terminology

3.1 *Definitions:*

3.1.1 *automotive*, *adj*—descriptive of equipment associated with self-propelled machinery, usually vehicles driven by internal combustion engines. **D4175**

3.1.2 *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**

3.1.3 *elastomer*, *n*—a natural or synthetic polymer having the rubber-like property of substantially recovering its size and shape after removal of a deforming force. **D4175**

3.1.4 *engine oil*, *n*—a liquid that reduces friction or wear, or both, between the moving parts within an engine; removes heat particularly from the underside of pistons; and serves as combustion gas sealant for the piston rings.

3.1.4.1 *Discussion*—It may contain additives to enhance certain properties. Inhibition of engine rusting, deposit formation, valve train wear, oil oxidation and foaming are examples. **D4175**

3.1.5 *formulation*, *n*—the specific chemical composition used in manufacturing a seal elastomer or a reference oil. **D5662**

3.1.6 *hardness*, *n—of an elastomer*, the resistance to deformation or indentation.

3.1.6.1 *Discussion*—In this test method the hardness of an elastomer is measured with a Shore Durometer A (see Test Method D2240). **D4175**

3.1.7 *heavy-duty engine*, *n—in internal; combustion engine types*, one that is designed to allow operation continuously at or close to its peak output.

3.1.7.1 *Discussion*—This type of engine is typically installed in large trucks and busses as well as farm, industrial, and construction equipment. **D4485**

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

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

3.1.9.1 *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**

3.1.10 *tensile strength*, *n*—the maximum tensile stress applied in stretching a specimen to rupture. **D1566**

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

3.1.12 *ultimate elongation*, *n*—the elongation at which rupture occurs in the application of continued tensile stress. **D1566**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *compatibility*, *n—of an engine oil/elastomer combination*, a characteristic that signifies a complete or high-level of

[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 Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

resistance of the elastomer to deleterious effects imparted by contact with, or immersion in, the oil.

3.2.1.1 *Discussion*—The phrase "high compatibility" indicates that after contact or immersion, the elastomer properties are maintained at or near their initial level. The terms "lack of compatibility" and "low compatibility" indicate that after contact or immersion, the elastomer properties are adversely affected to an extent that could be detrimental to sealing performance.

3.2.2 *immersion test*, *n*—an operation to evaluate compatibility by determining the effect of a liquid on elastomer test specimens submerged in the liquid for a specified time and at a specified temperature.

3.2.2.1 *Discussion*—The effect of the liquid is evaluated by the difference in (typical) elastomer physical properties pre- and post-immersion.

3.2.3 *reference elastomer*, *n*—an elastomer compound prepared using a specified formulation; its immersion test properties with selected oils have been well established by the use of recognized and accepted testing and documentation procedures.

3.2.4 *tensile stress at 50 % elongation*, *n*—the stress required to stretch the uniform cross section of a test specimen to 50 % elongation.

4. Summary of Test Method

4.1 Measurements of initial volume, hardness (Durometer A) and tensile properties are made on specimens of specified dimensions cut from sheets of reference elastomers.

4.1.1 Table 1 shows the types of elastomers involved, typical of those used in heavy-duty diesel engines.

4.2 The elastomer specimens are immersed in both non-reference oil(s) and a reference oil and aged for 336 h at specified temperatures.

4.3 The effects of the test oils on the elastomers are determined by measuring the changes in volume, hardness and tensile properties resulting from the immersion in the oil.

5. Significance and Use

5.1 Some engine oil formulations have been shown to lack compatibility with certain elastomers used for seals in automotive engines. These deleterious effects on the elastomer are greatest with new engine oils (that is, oils that have not been exposed to an engine's operating environment) and when the exposure is at elevated temperatures.

5.2 This test method requires that non-reference oil(s) be tested in parallel with a reference oil, TMC 1006-1, known to be aggressive for some parameters under service conditions. This *relative* compatibility permits decisions on the anticipated or predicted performance of the non-reference oil in service.

5.3 Elastomer materials can show significant variation in physical properties, not only from batch-to-batch but also within a sheet and from sheet-to-sheet. Results obtained with the reference oil are submitted by the test laboratories to the TMC to allow it to update continually the total and within-laboratory standard deviation estimates. These estimates, therefore, incorporate effects of variations in the properties of the reference elastomers on the test variability.

5.4 This test method is suitable for specification compliance testing, quality control, referee testing, and research and development.

5.5 The reference elastomers, reference oil and the physical properties involved in this test method address the specific requirements of engine oils. Although other tests exist for compatibility of elastomers with liquids, these are considered too generalized for engine oils.

TABLE 1 Immersion Temperatures and Times for the Four Reference Elastomers[A]

Elastomer	Immersion Test Temperature, °C	Immersion Test Time, h
Nitrile (NBR)	100 ± 1	336.0 ± 0.5
Polyacrylate (ACM)	150 ± 1	336.0 ± 0.5
Fluoroelastomer (FKM)	150 ± 1	336.0 ± 0.5
Silicone (VMQ)	150 ± 1	336.0 ± 0.5

[A] Some lubricant specifications may require immersion times other than 336 h. For times < 70 h the tolerance is ± 0.25 h and for times ≥ 70 h the tolerance is ± 0.5 h (see also 1.4).

6. Apparatus

6.1 The testing equipment as specified in Test Methods D412, D471, D2240, and D5662 is required for the use of this test method.

6.2 *Balance*—Use any commercially available balance capable of weighing to the nearest 0.1 mg. Equip the balance with a suspension hook and a platform to locate a hydrostatic-weighing beaker above the balance pan.

6.2.1 *Calibration*—Calibrate the balance annually as described in Test Method D5662.

6.3 *Hardness Durometer A*—See Test Method D2240. Use a stand–mounted Durometer.

6.3.1 *Calibration*—Calibrate the hardness Durometer annually as described in Test Method D2240. Use an outside source, with standards traceable to the National Institute for Standards Technology (NIST), for annual calibration. Perform checks with internal standards weekly. Checks with internal standards shall be within ±3 points. Calibrate internal standards annually, using an outside source, with standards traceable to NIST.

6.4 *Tension Testing Machine*—See the appropriate sections of Test Method D412. The rate of grip separation for the tension testing shall be (8.5 ± 0.8) mm/s.

6.4.1 *Calibration*—Using an outside source, calibrate the tension testing machine annually, as described in Test Method D412, using standards traceable to NIST.

6.5 *Glass Tubes*—Preferably of borosilicate glass, having an outside diameter of 38 mm and an overall length of 300 mm. Fit each tube loosely with an inert sealing device (such as a cork stopper covered with aluminum foil) that will not contaminate the test oil.

6.6 *Hanger Wire*—Stainless steel, about 0.5 mm diameter, having a suitably sized eye at one end of the wire and a hook at the other end, separated by approximately 8 mm. This is used to suspend the elastomer test specimens when measuring their mass in water.

6.7 *Specimen Suspension Wire*—Stainless steel, about (0.8 to 1) mm diameter to the shape and dimensions shown in Fig. 1, to hold the elastomer test specimens submerged in the immersion oil. (The specimens are attached to the suspension

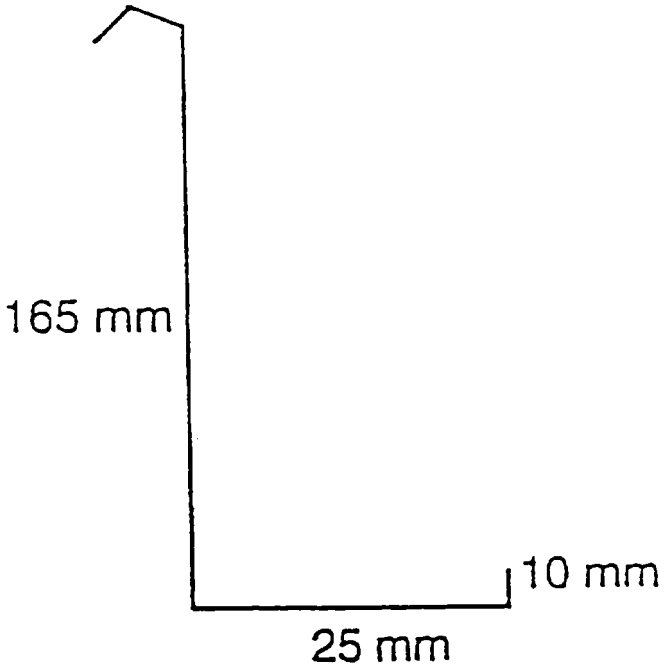

FIG. 1 Wire Hanger

wire which is hung over the edge of the glass tube and held in place by the stopper as shown in Fig. 2.)

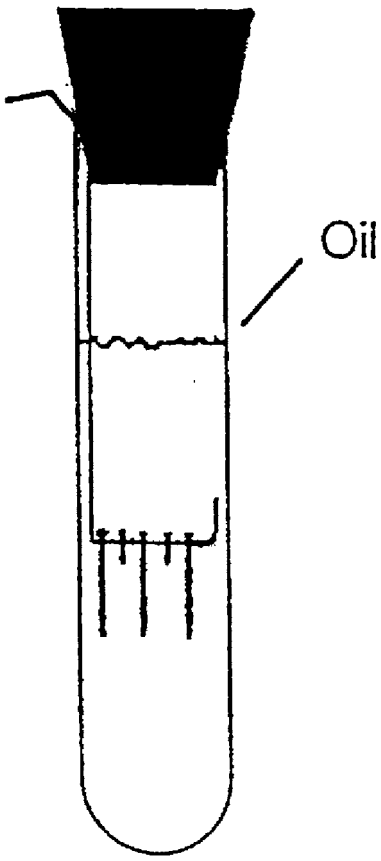

FIG. 2 Test Tube Arrangement

6.8 *Heated Immersion Test Bath or Block*—Capable of maintaining the test oil in the glass tube to within ± 1 °C of the test temperature. The immersion test bath or block shall contain a rack or holes that will accept the glass tubes specified in 6.5 and hold them in a vertical position.

6.9 *Die for Cutting Dumbbells*—Use Die C as specified in Test Method D412.

7. Reference Materials

7.1 *Reference Oil*—The reference oil is maintained and distributed by the (TMC).[2] The reference oil designation is TMC 1006-1. In order to receive this reference oil, individual laboratories shall agree to furnish the TMC with immersion test results obtained with the reference oil.

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

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean water conforming to Specification D1193 Type III deionized water or water of equivalent purity.

7.3 *Wetting Agent Solution*—0.1 % sodium dioctyl sulfosuccinate, made by a suitable dilution of concentrated solution[5] with reagent water. (**Warning—**Eye irritant.)

7.4 *Reference Seal Elastomers*—Obtain cured prepared sheets of the reference seal elastomers from the Parts Distributor (PD).[6] The sheets are at least (152 by 152) mm and have a uniform thickness of (2 ± 0.1) mm. The specific reference elastomers described in this test method are a fluoroelastomer (FKM), a polyacrylate material (ACM), a silicone rubber (VMQ), a nitrile rubber (NBR), and a VAMAC (MAC).

NOTE 4—Elastomer sheets received from the PD are numbered in the following format: [type] X. Type = the elastomer type (for example, FKM, ACM, VMQ, NBR, or MAC), and X = batch number for the particular formulation.

7.4.1 Table A1.1 shows the formulations and typical properties of some of the reference elastomers (typical of those used in heavy-duty diesel engines) listed in 7.4. These data are provided for information purposes only.

7.4.2 The PD is responsible for maintaining the numbering and tracking system for the reference elastomers and for managing the procurement of elastomers that meet the specifications of this standard. Certain specific information concerning these reference materials is available only to the PD. This information is used to ensure batch-to-batch consistency.

7.4.3 Store the reference elastomers in a location shielded from light, where the relative humidity is in the range of (40 to

[5] Aerosol OT has been found satisfactory for this purpose. (Aerosol is a trade mark of American Cyanamid Co.). Preparation of the wetting agent solution from the solid reagent is not recommended.

[6] Contact the TMC for the current company. In 2004, the company functioning in this capacity was: OH Technologies Inc., Attention Jason Bowden, PO Box 5039, 9300 Progress Parkway, Mentor OH 44060, USA.

55) % and the temperature in the range of (10 to 25) °C. Under these conditions the shelf life of the reference elastomers is three years from the date of cure provided by the PD. Do not use any elastomer with a seal cure date older than three years.

8. Procedure

8.1 *General Background*—Conduct the immersion tests for any non-reference oil(s) on the basis of a test series operation. A test series is a complete evaluation program, using the specified physical tests, for any selected number of non-reference oils and the reference oil using one or more of the reference elastomers. Use the same elastomer batch for both non-reference oils and the reference oil.

8.2 *Number of Test Specimens*—There are two types of test specimens: dumbbells for tensile testing and (25 by 50) mm rectangular sheets for mass measurements and hardness testing. For each elastomer/oil combination to be tested cut: *(1)* twelve dumbbell specimens (six for initial and six for final tensile testing); *(2)* six rectangular specimens (each specimen is used for both initial and final volume/hardness testing). Ensure that all reference elastomer specimens for a test series are cut from the same elastomer batch.

8.3 *Test Specimen Preparation*—Determine the number of elastomer reference sheets required for the projected testing. For each elastomer, this number will depend on the number of oils involved and the number of test specimens required (see 8.2). Condition these sheets for at least 3 h at (23 ± 2) °C as specified in Test Method D412. Ensure cutting dies are sharp; see Test Method D412 for additional information on die maintenance.

8.3.1 *Specimens for Tensile Testing*—From each reference elastomer, cut the required number of dumbbell specimens from the 2 mm thick sheets, using Die C as specified in Test Method D412, with the long axis of the dumbbell parallel to the grain. (The direction of the grain is marked on the sheets as noted in footnote A to Table A1.1.) Using a die press for the operation, cut only one sheet thickness at a time.

8.3.1.1 Depending on the number of oils being evaluated, two or more sheets will be required for each elastomer. Each sample of six dumbbells (initial and final sample sets) shall contain as close as possible an equal number of dumbbells from each of the individual sheets as required for the testing.

8.3.2 *Specimens for Mass Measurements Required for Volume Determination*—Cut the required number of (25 by 50) mm rectangular specimens from the 2 mm thick sheets. Because two or more sheets will be required (to prepare at least twelve specimens; six for at least one non-reference oil and six for the reference oil) for the total number of specimens for any elastomer, each sample of six shall contain an equal number of specimens from each of the individual sheets as required for the testing.

8.3.3 *Specimens for Hardness Testing*—Use the specimens prepared for the mass measurements (see 8.3.2).

8.4 *Measurement of the Pre-Immersion Elastomer Properties*—Measure the initial tensile properties, hardness and mass, as described in 8.4.1 to 8.4.3, of the pre-immersion specimens. Note that while the pre- and post-immersion specimens are different for the tensile measurements they are the same for the mass and hardness measurements. Ensure, therefore, that the mass and hardness are measured pre-immersion.

8.4.1 *Tensile Measurements*—Using the procedure specified in Test Method D412, test six dumbbells for each oil/elastomer combination, recording for each dumbbell the ultimate elongation and the tensile strength. To eliminate effects of variations in ambient conditions such as temperature and humidity, measure the initial tensile properties in the same time frame as the final tensile proprieties, that is, post-immersion (see 8.6.1).

8.4.2 *Hardness*—To protect the equipment, it is recommended that the hardness measurements be carried out before the mass measurements to ensure the Durometer tip is kept dry. Stack three (25 by 50) mm specimens on top of each other to obtain the requisite minimum 6 mm thickness. For ease of reference, the specimens at the top, middle and bottom of the stack will be referred to as Specimens *A*, *B*, and *C*, respectively. In accordance with Test Method D2240, determine the Durometer A hardness of the elastomer specimens, taking readings (1 ± 0.1) s after the pin makes contact with the rubber. Make repeat measurements on different parts of the specimen. The following readings are taken:

8.4.2.1 Take three readings from the top side of Specimen *A*. Turn Specimen *A* over and take three additional readings from its other side. Calculate and report the arithmetic mean of the six values.

8.4.2.2 Move Specimen *C* to the top of the stack and take another set of six readings as described in 8.4.2.1.

8.4.2.3 Move Specimen *B* to the top of the stack and take another set of six readings as described in 8.4.2.1.

8.4.2.4 Prepare a second stack of three specimens and repeat 8.4.2.1 to 8.4.2.3.

8.4.3 *Measurement of Mass*—For each elastomer/oil combination, measure and record the mass, to the nearest milligram, of each of the six (25 by 50) mm rectangular specimens in air and water using the water displacement method.

8.4.3.1 With a leather punch or cork borer, punch or cut a (2 to 5) mm diameter hole near the center of a short edge. Corners or small radiused notches can be cut for identification; do not cut V-notches.

8.4.3.2 Weigh the specimen in air to determine M_1 (if it is pre-immersion) or M_3 (if it is post-immersion).

8.4.3.3 Using the hole in its short edge, suspend the elastomer test specimen from the hanger wire. Immerse each specimen in a beaker of the wetting agent solution to remove air bubbles from the surface. Dislodge any air bubbles by agitating the specimen while in the solution.

8.4.3.4 Suspend the hanger wire and specimen from the balance hook. Inspect the submerged specimen and remove any adhering air bubbles. If the bubbles are difficult to remove, repeat 8.4.3.3. In uncommon instances, it may be necessary to devise a means of mechanically dislodging adherent air bubbles.

8.4.3.5 Weigh the specimen in water to determine M_2 (if it is pre-immersion) or M_4 (if it is post-immersion).

8.5 *Immersion Testing*—Conduct all immersion tests for a test series concurrently in the same heated immersion test bath or block.

8.5.1 Pour (150 ± 5) mL of test oil into the immersion test tubes. Four test tubes are required for each elastomer/oil combination. In each tube, suspend three rectangular specimens or three dumbbell specimens from the stainless steel wire hanger, as shown in Fig. 1. Locate inert (to oil or rubber) spacers, such as stainless steel washers (1 to 2) mm thick, between each test specimen to prevent specimens from touching each other or the test tube wall (see Fig. 2). Cover each test tube with a stopper as specified in 6.5.

8.5.2 Set the heating block/bath temperature to the appropriate value for the elastomer under test. (Table 1 shows the immersion test temperatures and immersion times to be used for the reference elastomers.) When the test temperature has been attained, insert the test tubes into the heating block/bath.

8.5.2.1 To minimize effects of temperature variations within the heating block/bath, spread the tubes randomly, avoiding placing all the tubes in the same portion or putting tubes containing the same samples together.

8.5.2.2 Ensure that no specimen touches another specimen or the test tube wall. Such an occurrence invalidates the test.

8.5.2.3 To ensure that aging conditions are equivalent for both non-reference and reference oils, insert all tubes in the heating block/bath within 8 h of each other.

8.5.2.4 Each tube shall be in the block/bath for the period specified in Table 1. The time starts when a tube is inserted in the heating block/bath, provided the latter is already at the correct temperature. If desired, as a check, insert a dummy tube containing an appropriate amount of oil and measure its temperature.

8.5.3 At the end of the aging period, remove the specimens from the test tubes and place them (while on the suspension wire) on a clean, absorbent towel or surface to cool to ambient temperature. The cooling period shall not exceed 30 min. If necessary, increase the cooling rate by placing the specimens on a surface that allows cooling from both sides.

8.5.4 At the end of the cooling period, remove the specimens from the suspension wire and place them on a new, clean, absorbent towel or surface. Remove the excess oil with a clean absorbent towel. Begin the final testing (30 to 60) min after removal of the specimen from the test tube. Take no more than 2 h to complete this final testing.

8.6 *Measurement of Post-Immersion Physical Properties*:

8.6.1 Using the procedure described in 8.4.1, measure the tensile strength and ultimate elongation of both the pre- and post-immersion specimens.

8.6.2 Measure the hardness and the mass in air and water of the post-immersion specimens using the procedures described in 8.4.2 and 8.4.3, respectively.

9. Calculations

9.1 For each combination of reference elastomer/test oil calculate the change in properties as follows:

NOTE 5—A negative change indicates a reduction in performance after immersion in the oil. For the hardness change, negative and positive changes indicate that the specimen softened or hardened, respectively.

9.1.1 *Ultimate Elongation Change*:

$$\Delta E = 100[(E_f - E_i)/E_i] \quad (1)$$

where:
ΔE = ultimate elongation change, %,
E_i = initial ultimate elongation, %, and
E_f = final ultimate elongation, %.

9.1.2 *Tensile Strength Change*:

$$\Delta TS = 100[(TS_f - TS_i)/TS_i] \quad (2)$$

where:
ΔTS = tensile strength change, %,
TS_i = initial tensile strength, MPa, and
TS_f = final tensile strength, MPa.

9.1.3 *Volume Change*:

$$\Delta V = 100[\{(M3 - M4) - (M1 - M2)\}/(M1 - M2)] \quad (3)$$

where:
ΔV = volume change, %,
$M1$ = initial mass in air, g,
$M2$ = initial mass in water, g,
$M3$ = final mass in air, g, and
$M4$ = final mass in water, g.

9.1.4 *Durometer A Hardness Change*:

$$\Delta H = (H_f - H_i) \quad (4)$$

where:
ΔH = hardness change, Durometer A units,
H_i = initial hardness, Durometer A units, and
H_f = final hardness Durometer A units.

10. TMC 1006-1 Reference Oil Testing

10.1 As specified in 8.1, the reference oil TMC 1006-1 is evaluated simultaneously with each set of non-reference oil tests.

10.2 Prior to conducting a reference oil test, procure a supply of TMC 1006-1 directly from the TMC. Each reference oil sample is identified using a unique set of identification codes on the container labels.

10.3 Report the results of the TMC 1006-1 reference oil tests to the TMC (see Section 11).

10.3.1 Transmit reference oil test data to the TMC by electronic means or by telephone facsimile immediately upon completion of the test analysis. Include all of the reporting forms in the transmission.

NOTE 6—Specific protocols for the electronic transmission of test data to the TMC are available from the TMC.

10.4 *Evaluation of Reference Oil Test Results*—Upon receipt of the transmitted test results for TMC 1006-1, the TMC will review the test for operational adherence to the published test method.

10.4.1 If the test is found to be operationally valid, the reference oil results will be evaluated by the TMC using acceptance criteria established by the Elastomer Test Surveillance Panel. The reference oil acceptance criteria are subject to change at the discretion of this Panel.

10.4.2 In the event the TMC find the reference oil test results are unacceptable, an explanation of the problem relating to the failure will be provided to the testing laboratory. If there is an obvious operational reason for the failed test, correct the problem before carrying out another test series. If the reason

for failure is not obvious, re-check all test-related equipment for compliance to the test method and good laboratory practice.

10.5 *Status of Non-reference Oil Tests Relative to TMC Reference Oil Test*—The batch of non-reference oil tests is considered valid only if the TMC inform the testing laboratory that results reported for the reference oil are acceptable.

11. Report

11.1 Use the standardized report form set and data dictionary for reporting the test oil results.

NOTE 7—Either download the actual report forms and data dictionary from the TMC website at ftp://ftp.astmtmc.cmu.edu or obtain them in hardcopy format from the TMC.

11.2 Report the following information:

11.2.1 Test completion date.

11.2.2 Elastomer type and batch number.

11.2.3 Test oil identity.

11.2.4 Test temperature, °C.

11.2.5 Test duration, h.

11.2.6 Aging block or bath identification.

11.2.7 For each elastomer/oil combination, report:

11.2.7.1 Percent volume change, including the six individual values, the arithmetic mean and the standard deviation. Report volume change to two decimal places.

11.2.7.2 Durometer A hardness change, including the individual means for each of the six specimens, the arithmetic mean of these six values and the standard deviation. Report hardness change to the nearest whole Durometer A points, including the plus or minus sign.

11.2.7.3 Percent tensile strength change, including the six individual values, the arithmetic mean and the standard deviation. Report percent tensile strength change to one decimal place.

11.2.7.4 Percent ultimate elongation change, including the six individual values, the arithmetic mean and the standard deviation. Report percent ultimate elongation change to one decimal place.

11.3 *Precision of Reported Units*—Use Practice E29 for rounding-off data. Use the rounding-off method to report data to the required precision.

11.4 *Outliers*—Report all data generated that was successfully measured. Do not apply any statistical measure for outlier screening. If any of the six individual values for any parameter were not measured successfully because of *(1)* grip slippage, *(2)* specimen breakage outside the test area, *(3)* a nick to the test specimen, or *(4)* an obvious material flaw (such as an air bubble), then record an asterisk (*) for that particular measurement and provide a description of the problem in the comment section of Form 7 of the test report. Compute the arithmetic mean and standard deviation using the remaining individual values. If more than two individual values for any of the four parameters need to be reported as an asterisk (*), the test is invalid.

TABLE 2 FLUOROELASTOMER Reference Oil Precision Data

NOTE—These statistics are based on results obtained on Test Monitoring Center reference oils between August 23, 2001 and August 9, 2007.

Variable	$s_{i.p.}$	i.p.	s_R	R
Volume Change, %	0.15	0.42	0.18	0.50
Hardness Change, Points	1.39	3.89	2.00	5.60
Tensile Strength Change, %	4.10	11.48	4.61	12.91
Elongation Change, %	6.27	17.56	8.85	24.78

TABLE 3 NITRILE Reference Oil Precision Data

NOTE—These statistics are based on results obtained on Test Monitoring Center reference oils between August 23, 2001 and August 10, 2007.

Variable	$s_{i.p.}$	i.p.	s_R	R
Volume Change, %	0.68	1.90	0.71	1.99
Hardness Change, Points	1.48	4.14	1.71	4.79
Tensile Strength Change, %	7.48	20.94	7.79	21.81
Elongation Change, %	6.34	17.75	6.41	17.95

TABLE 4 POLYACRYLATE Reference Oil Precision Data

NOTE—These statistics are based on results obtained on Test Monitoring Center reference oils between August 23, 2001 and August 8, 2007.

Variable	$s_{i.p.}$	i.p.	s_R	R
Volume Change, %	0.70	1.96	0.73	2.04
Hardness Change, Points	1.64	4.59	1.65	4.62
Tensile Strength Change, %	7.22	20.22	7.26	20.33
Elongation Change, %	8.99	25.17	9.12	25.54

TABLE 5 SILICONE Reference Oil Precision Data

NOTE—These statistics are based on results obtained on Test Monitoring Center reference oils between August 23, 2001 and August 7, 2007.

Variable	$s_{i.p.}$	i.p.	s_R	R
Volume Change, %	1.86	5.21	2.07	5.80
Hardness Change, Points	1.17	3.28	2.22	6.22
Tensile Strength Change, %	4.80	13.44	4.84	13.55
Elongation Change, %	7.28	20.38	7.50	21.00

TABLE 6 VAMAC Reference Oil Precision Data

NOTE—These statistics are based on results obtained on Test Monitoring Center reference oils between November 17, 2003 and August 10, 2007.

Variable	$s_{i.p.}$	i.p.	s_R	R
Volume Change, %	1.99	5.57	2.29	6.41
Hardness Change, Points	1.05	2.94	1.05	2.94
Tensile Strength Change, %	6.86	19.21	7.22	20.22
Elongation Change, %	9.22	25.82	9.71	27.19

Legend:
$s_{i.p.}$ = intermediate precision standard deviation.
i.p. = intermediate precision.
s_R = reproducibility standard deviation.
R = reproducibility.

12. Precision and Bias

12.1 Test precision is established on the basis of reference oil test results (for operationally valid tests) monitored by the ASTM Test Monitoring Center. The data are reviewed semi-annually by the Engine Oil Elastomer Compatibility Surveillance Panel. Contact the ASTM TMC for current industry data.

12.1.1 Tables 2-6 summarize reference oil intermediate precision and reproducibility of the test. The tabulated values

are current as of August 2007. The Surveillance Panel updates these values as necessary.

12.1.2 *Intermediate Precision Conditions*—Conditions where test results are obtained with the same test method using the same test oil, with changing conditions such as operators, measuring equipment, test stands, test engines, and time.

NOTE 8—Intermediate precision is the appropriate term for this test method rather than repeatability which defines more rigorous within-laboratory conditions.

12.1.2.1 *Intermediate Precision Limit (i.p.)*—The difference between two results obtained under intermediate precision conditions that in the long run, in the normal and correct conduct of the test method, exceed the values shown in Tables 2-6 in only one case in twenty. When only a single test result is available, the Intermediate Precision Limit can be used to calculate a range (test result ± Intermediate Precision Limit) outside of which a second test result would be expected to fall about one time in twenty.

12.1.3 *Reproducibility Conditions*—Conditions where test results are obtained with the same test method using the same test oil in different laboratories with different operators using different equipment.

12.1.3.1 *Reproducibility Limit (R)*—The difference between two results obtained under reproducibility conditions that would, in the long run, in the normal and correct conduct of the test method, exceed the values in Tables 2-6 in only one case in twenty. When only a single test result is available, the Reproducibility Limit can be used to calculate a range (test result ± Reproducibility Limit) outside of which a second test result would be expected to fall about one time in twenty.

12.1.4 *Bias*—No estimate of the bias for the procedure is possible because the performance results for an oil are determined only under the specific conditions of the test and no absolute standards exist.

13. Keywords

13.1 automotive engine oil; compatibility; elastomer; engine oil; immersion; seal(s)

ANNEXES

(Mandatory Information)

A1. FORMULATIONS AND PHYSICAL PROPERTIES FOR THE REFERENCE ELASTOMERS TYPICALLY USED IN HEAVY-DUTY DIESEL ENGINES

A1.1 Table A1.1 lists the formulation data and typical physical properties for cured elastomer sheets provided by the PD (see 7.4). It is the responsibility of the PD to ensure that the elastomer sheets conform to all requirements, including curing, formulation, and properties.

TABLE A1.1 Formulation Data and Typical Physical Properties for Four of the Reference Elastomer Materials in 7.4[A]

Elastomer	Ingredients	Parts, by Mass	Durometer A Hardness,[B] Points	Tensile Strength,[C] MPa	Ultimate Elongation,[B] %	Specific Gravity[D] mg/m^3
Fluoroelastomer (FKM)	Rubber[E]	100.00	71	≥13	270	1.84
	Maglite D	3.00				
	N-990 Carbon Black	30.00				
	Calcium Hydroxide – Reagent Grade	6.00				

	Press Cure: 10 min @ 177 °C					
	Post Cure: 16 h @ 232 °C					
Polyacrylate (ACM)	Rubber[F]	100.00	66	11.9	175	1.31
	N-550 Carbon Black	65.00				
	Stearic Acid	1.00				
	Substituted diphenylamine[G]	2.00				
	Processing aid[H]	2.00				
	Sodium Stearate	4.00				
	Quaternary ammonium compound[I]	2.00				

	Press cure: 12 min @ 170 °C					
	Post cure: None					
Silicone (VMQ)	Rubber[J]		30 to 80	50 to 400	400 to 1200	1.10 to 2.20

	Press cure: 5 min @ 188 °C					
	Post cure: 4 h @ 200 °C					
Nitrile (NBR)	Rubber[K]	100.00	68	19.6	290	1.25
	Zinc Oxide	5.00				
	Stearic Acid	2.00				
	Antidegradent[L]	2.00				
	N-774 Carbon Black	70.00				
	Polysulfide[M]	5.00				
	Dicumyl peroxide[N]	3.00				

	Press cure: 12 min @ 170 °C					
	Post cure: None					

[A] Manufacturer shall mark each elastomer sheet to designate the direction of the grain of the material.
[B] Test Method D2240.
[C] Test Method D412.
[D] Test Method D297.
[E] Viton A-275C or Fluorel FC-2123 have been found satisfactory for this purpose. (Viton is a trademark of Dupont Dow Elastomers; Fluorel is the trademark of 3M.)
[F] HyTemp 4051 EP has been found satisfactory for this purpose. (HyTemp is a trademark of Zeon Chemicals.)
[G] Naugard 445 has been found satisfactory for this purpose. (Naugard is a trademark of Uniroyal Chemical.)
[H] TE 80 has been found satisfactory for this purpose. (TE is a trademark of Technical Processing.)
[I] HyTemp NPC-50 has been found satisfactory for this purpose.
[J] Dow Corning Product ID.24122V-BLK has been found satisfactory for this purpose.
[K] Nipol DN3350 has been found to be satisfactory for this purpose. (Nipol is a trademark of Zeon Chemicals.)
[L] Stangard has been found satisfactory for this purpose. (Stangard is a trademark of Harwick Chemical.)
[M] Thiokol TP-95 has been found satisfactory for this purpose. (Thiokol is a trademark of Morton International.)
[N] Varox DCP40KE has been found satisfactory for this purpose. (Varox is a trademark of R.T. Vanderbilt.)

A2. TEST PROCEDURE FOR REFERENCE ELASTOMERS TYPICALLY USED IN SPARK-IGNITION ENGINES

A2.1 Overview

A2.1.1 The test procedure described in this annex uses five elastomer seals typical of those used in passenger-car, spark-ignition engines. Table A2.1 shows the types of elastomers involved. They differ from those described in Table 1 and 7.4 which are more commonly used in heavy-duty diesel engines. The apparatus and the TMC 1006-1 reference oil testing are identical to those described in Sections 6 and 10, respectively. The procedure described in this annex differs, however, from that in Section 8 in that the tensile stress change at 50 %

TABLE A2.1 Immersion Temperatures and Times for the Reference Elastomers[A]

Elastomer	Immersion Test Temperature, °C	Immersion Test Time, h
Hydrogenated Nitrile (HNBR-1)	100 ± 1	336.0 ± 0.5
Polyacrylate (ACM-1)	150 ± 1	336.0 ± 0.5
Fluoroelastomer (FKM-1)	150 ± 1	336.0 ± 0.5
Silicone (VMQ-1)	150 ± 1	336.0 ± 0.5
Ethylene acrylate (AEM-1)	150 ± 1	336.0 ± 0.5

[A] Some lubricant specifications may require immersion times other than 336 h. For times < 70 h, the tolerance is ± 0.25 h and for times ≥ 70 h, the tolerance is ± 0.5 h (see also 1.4).

elongation is also determined. In all other respects, the procedure is identical to that described in Section 8.

A2.2 Reference Materials

A2.2.1 The reference materials are identical to those described in Section 7 with the exception of 7.4 and 7.4.1 which are replaced by A2.2.2 and A2.2.2.1, respectively:

A2.2.2 *Reference Seal Elastomers*—The specific reference elastomers described in this annex are a hydrogenated nitrile rubber (HNBR-1), a polyacrylate rubber (ACM-1), a fluoroelastomer rubber (FKM-1), a silicone rubber (VMQ-1) and an ethylene acrylate rubber (AEM-1). Obtain cured prepared sheets of the reference seal elastomers from the Parts Distributor (PD)5. The sheets are at least (152 by 152) mm and have a uniform thickness of (2 ± 0.1) mm. The PD shall mark each elastomer sheet to designate the direction of grain.

NOTE A2.1—Elastomer sheets received from the PD are numbered in the following format: [TYPE][Batch]. TYPE = the elastomer type (for example, FKM-1, ACM-1, VMQ-1, HNBR-1, or AEM-1); X = batch number for the particular formulation. For instance, HNBR-1BC-1.

A2.2.2.1 Information on the formulations for the reference elastomers listed in A2.2.2 is given in SAE J2643 (Appendix B for ACM-1, Appendix D for AEM-1, Appendix E for HNBR-1, Appendix F for VMQ-1, and Appendix H for FKM-1).

A2.3 Procedure

A2.3.1 Follow the procedure described in Section 8 with the exception of 8.4.1, 8.5.2, 8.5.2.4, and 8.6.1, which are replaced by A2.3.1, A2.3.2, and A2.3.3, respectively.

A2.3.2 *Tensile Measurements*

Using the procedure specified in Test Method D412, test six dumbbells for each oil/elastomer combination, recording for each dumbbell the ultimate elongation, the tensile strength and the tensile stress at 50 % elongation. To eliminate effects of variations in ambient conditions such as temperature and humidity, measure the initial tensile properties in the same time frame as the final tensile proprieties, that is, post-immersion (see A2.3.3).

A2.3.3 Set the heating block/bath temperature to the appropriate value for the elastomer under test. (Table A2.1 shows the immersion test temperatures and immersion times to be used for the reference elastomers described in A2.2.2.) When the test temperature has been attained, insert the test tubes into the heating block/bath.

A2.3.3.1 Each tube shall be in the block/bath for the period specified in Table A2.1. The time starts when a tube is inserted in the heating block/bath, provided the latter is already at the correct temperature. If desired, as a check, insert a dummy tube containing an appropriate amount of oil and measure its temperature.

A2.3.3.2 Using the procedure described in A2.3.1, measure the tensile strength, the tensile stress at 50 % elongation and ultimate elongation of both the pre- and post-immersion specimens.

A2.4 Calculations

A2.4.1 In addition to the calculations described in Section 9, carry out the following Tensile Stress Change at 50 % Elongation::

$$\Delta TS_{50} = 100[(TSf_{50} TSf_{50} - TSi_{50})/TSi_{50} \quad \text{(A2.1)}$$

where:

ΔTS_{50} = tensile stress change at 50 % elongation, %
TSi_{50} = initial tensile stress at 50 % elongation, MPa, and
TSf_{50} = final tensile stress at 50 % elongation, MPa.

A2.5 Report

A2.5.1 Report as described in Section 11 with the following addition: percent tensile stress change at 50 % elongation including the six individual values, the arithmetic mean and the standard deviation. Report percent tensile stress change at 50 % elongation to one decimal place.

A2.6 Precision and Bias

A2.6.1 Section 12 applies with the exception of 12.1.1 which is replaced by A2.6.1. Test data from which intermediate precision and reproducibility for the elastomers described in A2.2.2can be determined are currently being accumulated by the TMC. The Surveillance Panel will establish the test precision for these elastomers as soon as possible.