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Standard Test Method for Determining Automotive Gear Oil Compatibility with Typical Oil Seal Elastomers¹

This standard is issued under the fixed designation D5662; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method² covers the determination of the compatibility of automotive gear oils with specific nitrile, polyacrylate, and fluoroelastomer oil seal materials.

1.2 Users of this test method should obtain Test Methods D412, D471, and D2240 and become familiar with their use before proceeding with this test method.

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

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:³

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers Tension

D471 Test Method for Rubber Property Effect of Liquids

D2240 Test Method for Rubber Property Durometer Hardness

D5704 Test Method for Evaluation of the Thermal and Oxidative Stability of Lubricating Oils Used for Manual Transmissions and Final Drive Axles

D5760 Specification for Performance of Manual Transmission Gear Lubricants

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

2.2 SAE Standard:⁴

J2360 Lubricating Oil, Gear Multipurpose (Metric) Military Use

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *dumbbell, n*—the specific cut shape (Die C) of an elastomer as explained in the section on dumbbell specimens in Test Methods D412.

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

3.1.3 *percent ultimate elongation, n*—the stretch length at rupture of an elastomer dumbbell oil-aged by running this procedure minus the rupture stretch length of an untested dumbbell, all divided by rupture stretch length of the untested dumbbell and then multiplied by 100.

3.1.4 *percent volume change, n*—the change in volume of a test specimen as explained in the procedure for change in volume in Test Method D471.

4. Summary of Test Method

4.1 Non-reference oils are tested using a modified version of Test Method D471 on specific elastomer compounds. Measured

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.B0.03 on Automotive Gear Lubricants & Fluids.

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² Until the next revision of this test method, the ASTM Test Monitoring Center will update changes in this test method by means of Information Letters; these can be obtained from the ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, Pa 15206-4489. Attention: Administrator. This edition incorporates revisions in all Information Letters through No. 09-1-11-1.

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>.

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

quantities are percent ultimate elongation changes (further referred to as just percent elongation changes), durometer Type A hardness changes, and percent volume changes. Reference oils are run concurrently in the same oil bath to measure consistency from one test to another.

4.2 The duration of these tests is 240 h. The reference oils are available from the ASTM Test Monitoring Center (TMC).⁵ The seal materials are available through a Central Parts Distributor (CPD).⁶

5. Significance and Use

5.1 There are several major causes of automotive lubricant-related seal failures. This test method addresses only those failures caused by excessive elastomer hardening, elongation loss, and volume swell and attempts to determine the likelihood that an oil might cause premature sealing system failures in field use. This test method may be used as a requirement of a performance specification, such as Specification D5760 and J2360.

5.2 Another major cause of seal failure is the formation of carbon, varnish, and sludge-like deposits on the seal lip. The deposit-forming characteristics of automotive gear oils are evaluated in Test Method D5704. That procedure is intended in part to evaluate the potential for oils to cause premature seal failure in field service.

6. Apparatus

6.1 Specific test equipment as outlined in Test Methods D412, D471, and D2240 is required.

6.1.1 *Hardness Durometer*—See Test Method D2240.

6.1.1.1 *Calibration*—Calibrate the hardness durometer annually. Use an outside source, with standards traceable to 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.

6.1.2 *Tension Testing Machine*—See Test Method D412. Set the testing machine rate of grip separation for the percent elongation change determinations at (8.5 ± 0.8) mm/s. Calibrate the tension testing machine in accordance with Annex A2 or Annex A3, as appropriate.

6.1.2.1 *Calibration*—Calibrate the tension testing machine annually. Annual calibration shall be performed by the manufacturer, using NIST traceable standards.

6.1.3 *Glass Tubes*, having an outside diameter of 38 mm and an overall length of 300 mm. The tube is fitted loosely with an aluminum foil-covered stopper.

6.1.4 *Balance*—Use any commercially available balance capable of weighing samples to the nearest 1.0 mg.

6.1.4.1 *Calibration*—Calibrate the balance annually. Use an outside source, with standards traceable to NIST for annual calibration. Perform checks with internal standards monthly, using NIST traceable weights. The difference between the weights and balance shall be < 0.5 mg. Calibrate internal standards annually, using an outside source, with standards traceable to NIST.

7. Reagents and Materials

7.1 Specific reference test oils are maintained and distributed by the TMC.⁵ To receive the test oils and seal materials, individual laboratories shall commit to furnishing the TMC with reference data developed using these reference materials.

7.2 The CPD is responsible for maintaining the numbering and tracking system for the seal elastomer batches used. Certain specific information concerning these reference materials is available only to the CPD. This information is used to ensure batch-to-batch consistency.

7.2.1 Information and location of the current CPD is also available from the TMC.

7.3 Specific reference seal elastomers used are a nitrile (NI), a polyacrylate (PA), and a fluoroelastomer (FL). Notation of the numbering system is established by the TMC as follows:

[Type] Y

where:

Type = NI, PA, FL, and

Y = Batch number of the particular formulation.

7.4 The shelf life for the seal elastomers is two years from the date the batch was cured. Invalidate any test with a seal cure date older than two years. Consult the TMC for any approved exceptions to this lifespan. Place a note in the comments section of the test report where exceptions have been approved.

7.4.1 Store elastomers in a refrigerator maintained at (3 to 6) °C.

7.5 The shelf life of reference oils is typically five years unless the TMC, through their analysis, specifies otherwise.

7.6 Wetting solution of Aerosol OT—0.1 % sodium dioctyl sulfosuccinate, made by a 1.0 % dilution of a 10 % solution with reagent water.

⁵ Reference oils are available from the ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15206-4489.

⁶ The Central Parts Distributor for this procedure is Test Engineering Inc., 12758 Cimarron Path, Suite 102, San Antonio, TX 78249.

8. Procedure

8.1 The testing laboratory shall conduct reference oil tests concurrently with the non-reference oil in the same oil bath. Reference oils shall perform within a specific range prescribed and evaluated by TMC for validity and updated as needed.

8.2 Prior to cutting specimens and prior to performing elongation tests for initial properties, allow 3 h for the elastomer to warm to (23 ± 2) °C, as required by Test Method D412. Referring to the procedure in Test Method D412, use Die C to cut a set of twelve dumbbell specimens out of the elastomer sheets as required for each reference and non-reference oil tested.

8.2.1 Cut the dumbbells parallel to the grain using the same unaltered dies for the entire lot. When cutting dumbbells, only cut one thickness at a time to avoid any dimensional variations.

8.2.2 Cut all elastomer specimens, including those used for measuring initial properties, from the same elastomer batch. Use these dumbbells for measuring the percent elongation changes.

8.2.3 Next, cut twelve $(25 \text{ by } 50)$ mm by (2.0 ± 0.1) mm rectangular specimens for the percent volume change and hardness testing.

8.2.4 Finally, cut twelve more NI, PA, and FL dumbbells for the purpose of determining initial elongation properties.

8.2.5 Randomly select sets of twelve dumbbells and twelve rectangular specimens for testing from the different sheets of test elastomers.

8.2.6 Use the water displacement procedure in accordance with Test Method D471 to determine the initial volume measurements. Weigh the coupon in air, M1, to the nearest 1 mg. For the weight in water, immerse the coupon in a 1.0 % wetting solution of aerosol OT, then place the coupon in distilled water, M2, at ambient temperature. Make sure no air bubbles are clinging to the coupon surface before recording the weight to the nearest 1 mg.

8.2.7 Ensure that initial elastomer properties of hardness and volume are determined prior to the start of testing. Initial elongation properties are determined just prior to running the end of test dumbbells because of instrument calibration.

8.3 Fill the test tubes with (150 ± 5) mL of non-reference or reference oil as appropriate.

8.4 Use four test tubes for each elastomer/oil combination. In each tube, suspend from a stainless steel wire hanger bent at a 90° angle (dimensions shown in Fig. 1) three rectangular specimens and three dumbbells in each of the four tubes. Place spacers in between the specimens to aid in the separation. The width of the spacer(s) shall be (1.7 ± 0.3) mm. The spacer material shall not affect the liquid or the rubber.

8.4.1 Fig. 2 shows the arrangement of spacers and test specimens.

8.4.2 Top the test tube with a stopper wrapped in aluminum foil.

8.4.3 Test the non-reference oil using one or more of the three different seal elastomers with the same batch of elastomers as being used for the reference oil.

8.4.4 Place the tubes randomly in an oil bath capable of maintaining a test oil temperature within ± 1 °C for a period of (240 ± 0.5) h (see Table 1).

8.4.4.1 Measure the test oil temperature with a thermocouple or resistance temperature detector (RTD) inside a dummy test tube containing bath oil within the oil bath.

8.4.4.2 Record test oil temperature at a minimum of once every minute.

8.4.4.3 Calculate the percent deviation for the elastomer material test oil temperature shown in Table 1 using the following equation. Record the results in the test report.

$$\text{percent deviation} = \sum_{i=1}^n \left(\frac{M_i}{0.5R} \times \frac{T_i}{D} \right) \times 100 \quad (1)$$

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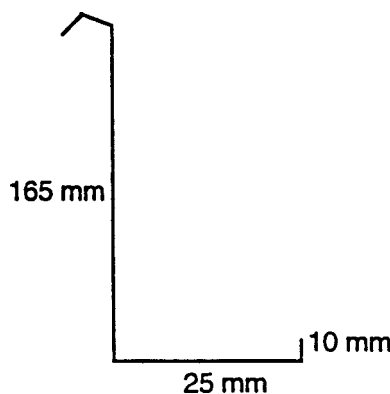


FIG. 1 Wire Hanger

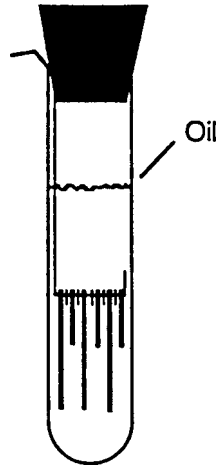


FIG. 2 Test Tube Arrangement

TABLE 1 Test Oil Temperatures

Material	Test Oil Temperature, °C	Percent Deviation Limits
Nitrile	100 ± 1	1%
Polyacrylate	150 ± 1	1%
Fluoroelastomer	150 ± 1	1%

M_i = absolute value of the magnitude of the test parameter out of specification limit at occurrence i ,

R = test parameter specification range,

T_i = length of time the test parameter was outside of specification range at occurrence i . (T_i is assumed to be no less than the recorded data-acquisition frequency, unless supplemental readings are documented), and

D = test duration in same units as T_i .

8.4.4.4 Invalidate any test that exceeds the percent deviation limits in Table 1.

8.4.5 Conduct all reference and non-reference oil testing on each seal elastomer in the same oil bath. Complete reference oil and non-reference oil tests for each seal elastomer within 8 h of each other to be considered the same test.

8.5 At the end of the test period, remove the specimens from the hot oil using the wire hanger and place them on a clean absorbent towel. Allow the specimens to cool for no longer than 30 min.

8.5.1 Remove the specimens from the wire hanger, and place them on a clean absorbent towel. Remove the excess oil with a clean absorbent towel, and begin testing.

8.6 Determine type A hardness testing, percent volume in air and water, and percent elongation, as done in 8.2.6 and 8.2.7. Testing shall be completed within 2 h of removal from the test oil.

8.7 Observe the following notes/modifications to Test Method D471.

8.7.1 Report percent change in elongation (see Test Method D412) and percent volume change (see Test Method D471) from the original using the same water displacement procedure described in 8.2.6.

8.7.1.1 When using a wire hanger to aid in the weighing of the test coupon, deduct the weight of the wire hanger from the gross weight to determine the actual weight of the test coupon. Record the actual weight of the test coupons in the test report and use the actual weights for the percent volume change calculations.

8.7.1.2 Conduct all reference and non-reference oil testing on each seal elastomer in an oil bath capable of maintaining a test oil temperature within $\pm 1^\circ\text{C}$ for a period of 240 ± 0.5 h (see Table 1).

8.7.2 Report durometer Type A hardness change points from original (see Test Method D2240).

8.7.2.1 On a hard horizontal surface, stack the three rectangular specimens on top of each other to obtain the 6 mm thickness required by Test Method D2240. Hardness readings are to be taken 1 s after the pin makes contact with the elastomer. Take three readings on each side of the rectangular specimen and report the average of all six readings.

8.7.2.2 After taking the first set of measurements, rotate the bottom specimen to the top of the stack and take a second set of measurements.

8.7.2.3 Rotate the bottom specimen to the top one more time to obtain the third set of measurements.

8.7.3 For each data set, calculate the average value and the sample standard deviation using the equation:

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where:

σ = sample standard deviation,
 n = number of data points in the set,
 X_i = individual data set value,
 \bar{x} = mean of the data set.

Change in volume, % = $[(M3 - M4) - (M1 - M2)] / (M1 - M2) \times d \times 100$

where:

$M1$ = the original weight in air,
 $M2$ = the original weight in water,
 $M3$ = the end of test weight in air,
 $M4$ = the end of test weight in water, and
 d = the density of the medium in which the specimen was weighed. In this case water is used, so the multiplication is by 1.

9. Reference Oil Testing

9.1 Test the TMC reference oils along with each batch of non-reference oil tests. Run the reference oils simultaneously with, and in the same bath as, the non-reference oils.

NOTE 1—Annex A1 discusses the involvement of the ASTM TMC with respect to the reference test-monitoring program.

9.1.1 Prior to requiring a reference oil test, procure a supply of reference oils directly from the TMC. These oils have been formulated or selected to represent specific chemistry types, or performance levels, or both. Each reference oil sample is identified using a unique set of identification codes on the container labels.

9.1.1.1 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 2—Policy for the Use and Analysis of ASTM Reference Oils is available from the TMC.

9.1.2 Request reference oil assignments from the TMC for this test method. The TMC will determine the specific reference oils to be tested by the laboratory. Assignments will be made by the unique identifying codes on the reference oil container labels. Provide the TMC with the bath identification number for each test.

9.1.3 Run the TMC reference oil tests according to the test method and in the same manner as the non-reference oil test(s).

9.1.4 *Reporting of Reference Oil Test Results*—For reference oil tests, the standardized report form set and data dictionary for reporting the test results and for summarizing the operational data are required. The report forms and data dictionary are available on the ASTM Test Monitoring Center Web Page at <http://www.astmtmc.cmu.edu/> or can be obtained in hardcopy format from the TMC.

9.1.4.1 Report only the reference oil results to the TMC. Do not include any non-reference test data. Complete all of the required blank fields on the forms. Round test results according to Practice

9.1.4.1 Report the reference oil test results to the TMC within five days of completion.

9.1.4.2 Report only the reference oil results to the TMC. Do not include any non-reference test data. Complete all of the required blank fields on the forms. Round test results according to Practice E29.

9.1.4.2 When reporting reference oil test results to the TMC, transmit via the ASTM Data Communications Committee Test Report Transmission Model (see Section 2—Flat File Transmission Format) available from the ASTM TMC. Transmit the complete report form package and any other supporting information to the ASTM TMC within five days of test completion. A copy of the final test report shall be mailed within 30 days of test completion to the ASTM Test Monitoring Center, 6555 Penn Avenue Pittsburgh, PA 15206-4489.

9.1.4.3 For electronic transfer of test results, use the ASTM Data Communications Committee Test Report Transmission Model (see Section 2—Flat File Transmission Format) available from the ASTM TMC.

9.1.5 *Evaluation of Reference Test Oil Results*—Upon receipt of the transmitted TMC reference oil test results, the TMC will review the tests for operational adherence to the published test method. If the tests are found to be operationally valid, the reference oil results will be evaluated using acceptance criteria established by the governing surveillance panel. The reference oil acceptance criteria are subject to change at the discretion of the surveillance panel.

9.1.5.1 If the transmitted tests are found to be both operationally valid and statistically acceptable, the testing laboratory will be notified of the acceptable status of the reference test.

9.1.5.2 In the event that a TMC reference oil test is found to be 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, the problem shall be corrected before requesting additional TMC reference oil assignments. If the reason for the failure is not obvious, all test-related equipment shall be re-checked for compliance to the test method and good laboratory practice. Following this re-check, the TMC will assign the necessary TMC reference oils for testing.

9.1.5.3 Should a reference test be determined to be statistically or operationally invalid, repeat all testing on that particular oil/elastomer pair.

9.1.6 *Status of Non-reference Oil Tests Relative to TMC Reference Oil Tests*—The batch of non-reference oil tests, which

accompany the reference oil tests, are considered valid only if the results of the reference oil tests are determined to be statistically and operationally valid.

9.2 *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 baths. The surveillance panel shall arrange an appropriate number of donated tests and ensure completion of the test program in a timely manner.

10. Precision and Bias

10.1 Test precision is established on the basis of reference oil test results (for operationally valid tests) monitored by the ASTM TMC. The data are reviewed annually by the OSCT Surveillance Panel. Contact the ASTM TMC for the current industry data. Table 2 summarizes reference oil precision of the test as of Aug. 2, 2004.

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

NOTE 3—Intermediate precision is the appropriate term for this test method, rather than repeatability, which defines more rigorous within-laboratory conditions.

10.1.1.1 *Intermediate Precision Limit (i.p.)*—The difference between two results obtained under intermediate precision conditions that would in the long run, in the normal and correct conduct of the test method, exceed the value shown in Table 2, 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 \pm Intermediate Precision Limit) outside of which a second test result would be expected to fall about one time in twenty.

10.1.2 *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.

10.1.2.1 *Reproducibility Limit (R)*—The difference obtained under reproducibility conditions that would in the long run, in the normal and correct conduct of the test method, exceed the value shown in Table 2, 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 \pm Reproducibility Limit) outside of which a second test result would be expected to fall about one time in twenty.

10.2 No estimate of the bias for this 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.

11. Keywords

11.1 compatibility; elastomer; elongation change; gear oil; hardness change points; oil seal; volume change

ANNEXES

(Mandatory Information)

A1. THE ROLE OF THE TEST MONITORING CENTER

A1.1 The ASTM Test Monitoring Center (TMC) is a nonprofit organization located at 6555 Penn Ave., Pittsburgh, PA

TABLE 2 OSCT Reference Oil Precision^A

$S_{i.p.}$ = intermediate precision standard deviation,
i.p. = intermediate precision,
 S_R = reproducibility standard deviation, and
R = reproducibility.

Variable	Intermediate Precision		Reproducibility	
	$S_{i.p.}$	i.p. ^B	S_R	R ^B
Percent Elongation	12.27	34.36	12.44	34.83
Durometer Type A Hardness	5.11	14.31	5.12	14.34
Percent Volume Change	3.19	8.93	3.19	8.93

^A These statistics are based on results obtained on Test Monitoring Center Reference Oils 160, 161, and 162 over the period from Aug. 1, 2002 through Aug. 2, 2004.

^B This value is obtained by multiplying the standard deviation by 2.8.