Designation: D471 – 16a

Standard Test Method for Rubber Property—Effect of Liquids[1]

This standard is issued under the fixed designation D471; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the required procedures to evaluate the comparative ability of rubber and rubber-like compositions to withstand the effect of liquids. It is designed for testing: (*1*) specimens of vulcanized rubber cut from standard sheets (see Practice D3182), (*2*) specimens cut from fabric coated with vulcanized rubber (see Test Methods D751), or (*3*) finished articles of commerce (see Practice D3183). This test method is not applicable to the testing of cellular rubbers, porous compositions, and compressed sheet packing, except as described in 12.2.2.

1.2 Periodically, it is necessary to produce a new lot of an IRM oil to replace the dwindling supply of the current product. The Chairman of the subcommittee shall have the authority to approve the production of a replacement lot. Once produced, the technical data of the new lot shall be presented, in a comparative fashion, to that of the existing lot and balloted upon by the membership of the D11.15 subcommittee and, either subsequently or concurrently, balloted upon by the membership of the D11 main committee for approval to release the new lot for distribution.

1.3 In the event that an IRM oil becomes unavailable for distribution due to depletion, the Chairman of the subcommittee shall have the authority to approve production of a new lot and, after a meeting of the task group, regularly scheduled, or not, to release a quantity of the product for distribution sufficient enough only to address a backlog. Once the backlog is addressed, the process described in 1.2 shall be followed.

1.4 ASTM Oils No. 2 and No. 3, formerly used in this test method as standard test liquids, are no longer commercially available and in 1993 were replaced with IRM 902 and IRM 903, respectively (see Appendix X1 for details).

1.5 ASTM No. 1 Oil, previously used in this test method as a standard test liquid, is no longer commercially available and in 2005 was replaced with IRM 901; refer to Table 1 and Appendix X3 for details.

1.6 ASTM No. 5 Oil was accepted into Specification D5900 as an industry reference material in 2010 and designated as IRM 905. The composition, and properties of this immersion oil were not changed and the data in Table 1 remains current. Refer to Appendix X4 for other details.

1.7 The specifications and properties listed in Table 1 for IRM 901, IRM 902, IRM 903, and IRM 905 are also maintained in Specification D5900.

1.7.1 The subcommittee responsible for maintaining Test Method D471, presently D11.15, shall review the data in Specification D5900 to ensure that it is identical to that which appears in Test Method D471. This shall be accomplished at the time of the 5 year review or more frequently when necessary.

1.8 Historical, technical, and background information regarding the conversion from ASTM No. 1, ASTM No. 2, and ASTM No. 3 Oils to IRM 901, IRM 902, and IRM 903 immersion oils is maintained in Practice D5964.

1.8.1 The subcommittee responsible for maintaining Test Method D471, presently D11.15, shall review the data in Practice D5964 to ensure that it is identical to that which appears in Test Method D471. This shall be accomplished at the time of the 5 year review or more frequently when necessary.

1.9 This test method includes the following:

Change in Mass (after immersion)	Section 11
Change in Volume (after immersion)	Section 12
Dimensional-Change Method for Water-Insoluble Liquids and Mixed Liquids	Section 13
Change in Mass with Liquid on One Surface Only	Section 14
Determining Mass of Soluble Matter Extracted by the Liquid	Section 15
Change in Tensile Strength, Elongation and Hardness (after immersion)	Section 16
Change in Breaking Resistance, Burst Strength, Tear Strength and Adhesion for Coated Fabrics	Section 17
Calculation (of test results)	Section 18

1.10 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.11 *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.*

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[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.15 on Degradation Tests.

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TABLE 1 Specifications and Typical Properties of IRM Reference Oils

Property	IRM 901[A]	IRM 902	IRM 903	IRM 905	ASTM Method
Specified Properties:					
Aniline Point, °C (°F)	124 ± 1 (255 ± 2)	93 ± 3 (199 ± 5)	70 ± 1 (158 ± 2)	115 ± 1 (239± 2)	D611
Kinematic Viscosity (mm^2/s [cSt])					
38°C (100°F)		...	31.9–34.1	...	D445
99°C (210°F)	18.12–20.34	19.2–21.5	...	10.8–11.9	D445
Gravity, API, 16°C (60°F)	28.8 ± 1	19.0–21.0	21.0–23.0	...	D287
Viscosity-Gravity Constant	0.790–0.805	0.860–0.870	0.875–0.885	...	D2501
Flash Point COC, °C (°F)	243(469) min	232 (450) min	163 (325) min	243 (469) min	D92
Naphthenics, C_N (%)	27 (avg)	35 min	40 min	...	D2140
Paraffinics, C_P (%)	65 min	50 max	45 max	...	D2140
Typical Properties:					
Pour Point, °C (°F)	−12 (10)	−15 (5)	−42.8 (−45)	−15 (5)	D97
ASTM Color	*L* 3.5	*L* 2.0	*L* 0.5	*L* 1.0	D1500
Refractive Index	1.4848	1.5083	1.5004	1.4808	D1747
UV Absorbance, 260 nm	0.8	1.43	1.43	...	D2008
Aromatics, C_A (%)	3	10	12	4	D2140

[A] Refer to Appendix X3. Table X3.1 reflects the differences in the properties between ASTM No. 1 Oil and IRM 901 for reference purposes.

TABLE 2 IRM 901 – 2012 Batch Test Results

Property	Method	2005 Batch COA	2005 Batch Current	2012 Sample Data				2012 Batch Average
Viscosity, cSt @99°C	D445	19.58	19.5	18.8 18.8	18.7 18.8	18.8 18.9	18.7 18.7	18.8
Gravity, API @16°C	D287	28.6	28.9	28.9 28.9	28.9 28.9	28.8 28.9	28.9 28.9	28.9
Flash Point COC, °C	D92	287	313	306 299	289 303	292 294	288 294	296
Aniline Point, °C	D611	123.8	124.7	124.8 124.8	124.8 124.8	124.7 124.9	124.3 124.6	124.5
Viscosity-Gravity Constant	D2501	0.796	0.798	0.799 0.799	0.799 0.799	0.800 0.799	0.799 0.799	0.799
Naphthenics, Cn%	D2140	—	24	26 26	26 26	27 27	28 29	27
Paraffinics, Cp%	D2140	70	72	71 71	71 71	69 71	70 70	71

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

D97 Test Method for Pour Point of Petroleum Products

D287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

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

D611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents

D751 Test Methods for Coated Fabrics

D865 Test Method for Rubber—Deterioration by Heating in Air (Test Tube Enclosure)

D975 Specification for Diesel Fuel Oils

D1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer

D1415 Test Method for Rubber Property—International Hardness

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[2] 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.

D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
D1747 Test Method for Refractive Index of Viscous Materials
D2008 Test Method for Ultraviolet Absorbance and Absorptivity of Petroleum Products
D2140 Practice for Calculating Carbon-Type Composition of Insulating Oils of Petroleum Origin
D2240 Test Method for Rubber Property—Durometer Hardness
D2501 Test Method for Calculation of Viscosity-Gravity Constant (VGC) of Petroleum Oils
D2699 Test Method for Research Octane Number of Spark-Ignition Engine Fuel
D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets
D3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products
D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries
D4485 Specification for Performance of Active API Service Category Engine Oils
D4806 Specification for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel
D5900 Specification for Physical and Chemical Properties of Industry Reference Materials (IRM)
D5964 Practice for Rubber IRM 901, IRM 902, and IRM 903 Replacement Oils for ASTM No. 1, ASTM No. 2, ASTM No. 3 Oils, and IRM 905 formerly ASTM No. 5 Oil
E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

2.2 *SAE Standards:*[3]
J 300 Engine Oil Viscosity Classification

3. Summary of Test Method

3.1 This test method provides procedures for exposing test specimens to the influence of liquids under definite conditions of temperature and time. The resulting deterioration is determined by measuring the changes in physical properties, such as stress/strain properties, hardness, and changes in mass, volume, and dimension, before and after immersion in the test liquid.

3.2 The precision statement in Section 20 is based on an interlaboratory test program run in 1981, using six different rubbers with ASTM Reference Fuels B, C, D[4] and ASTM Oils No. 1 and No. 3.

3.3 The precision statement in Appendix X2 is based on an interlaboratory test program conducted in 1993 to establish replacements for ASTM Oils No. 2 and No. 3. Because of the limited number of participating laboratories, only repeatability could be evaluated, and it was necessary to use pooled values of four No. 2 type oils (No. 2 plus three candidate replacement oils) and four No. 3 type oils (No. 3 plus three candidate replacement oils). Twelve rubbers were tested in this program.

3.4 ASTM Oils No. 1, No. 2, and No. 3 have been replaced by IRM 901, IRM 902, and IRM 903, respectively.

4. Significance and Use

4.1 Certain rubber articles, for example, seals, gaskets, hoses, diaphragms, and sleeves, may be exposed to oils, greases, fuels, and other fluids during service. The exposure may be continuous or intermittent and may occur over wide temperature ranges.

4.2 Properties of rubber articles deteriorate during exposure to these liquids, affecting the performance of the rubber part, which can result in partial failure.

4.3 This test method attempts to simulate service conditions through controlled accelerated testing, but may not give any direct correlation with actual part performance, since service conditions vary too widely. It yields comparative data on which to base judgment as to expected service quality.

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

5. Test Conditions

5.1 *Temperature and Immersion Periods*—Unless otherwise specified the test temperature and immersion period shall be as indicated in Table 3, depending upon the anticipated service conditions, unless otherwise agreed upon between customer and supplier:

5.1.1 When the temperature of the testing room is other than the standard 23 ± 2°C (73 ± 4°F) the temperature of test shall be reported.

5.1.2 When the relative humidity (RH %) of the testing environment is known to effect the performance of a test liquid, the RH % shall be reported.

5.1.3 The choice of the immersion period will depend upon the nature of the vulcanizate, the test temperature, and the liquid to be used. To obtain information on the rate of deterioration it is necessary to make determinations after several immersion periods.

TABLE 3 A: Test Temperatures

Temperature in °C (°F)	
−75 ± 2 (−103 ± 4)	85 ± 2 (185 ± 4)
−55 ± 2 (−67 ± 4)	100 ± 2 (212 ± 4)
−40 ± 2 (−40 ± 4)	125 ± 2 (257 ± 4)
−25 ± 2 (−13 ± 4)	150 ± 2 (302 ± 4)
−10 ± 2 (14 ± 4)	175 ± 2 (347 ± 4)
0 ± 2 (32 ± 4)	200 ± 2 (392 ± 4)
23 ± 2 (73 ± 4)	225 ± 2 (437 ± 4)
50 ± 2 (122 ± 4)	250 ± 2 (482 ± 4)
70 ± 2 (158 ± 4)	

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[3] Available from Society of Automotive Engineers (SAE), 400 Commonwealth Drive, Warrendale, PA 15096.

[4] The sole source of supply of the reference fuels known to the committee at this time is Chevron Phillips Chemical Company, LP, 10001 Six Pines Drive, The Woodlands, TX 77380. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

TABLE 3 B: Immersion Periods *(continued)*

Immersion Period, h
22
46
70
166
670
1006
2998
4990

5.1.4 The tolerance for any immersion period shall be ±15 min or ±1 % of the immersion period, whichever is greater.

5.1.5 The immersion periods enumerated in Table 3 are frequently used, and are considered standard; however, they may be varied according to a material's testing requirements or anticipated exposure in service.

5.2 *Illumination*—Immersion tests shall be made in the absence of direct light.

6. Standard Test Liquids

6.1 For test purposes, it is desirable to use the liquid that will come into contact with the vulcanizate in service. For comparative tests with liquids of unknown or doubtful composition, samples of liquid from the same container or shipment shall be used. Many commercial products, particularly those of petroleum origin, are subject to sufficient variation that it is not practical to use them for test liquids. It is then advisable to use a standard test liquid, such as described in 6.1.1 and 6.1.2, covering the range of properties that may be encountered in the particular service.

6.1.1 *IRM and ASTM Oils*—The test shall be conducted in one of the petroleum-base IRM or ASTM oils (Note 1) specified in Table 1 that has its aniline point nearest that of the oil with which the vulcanizate is expected to come in contact in service except as indicated in 6.1.3.

NOTE 1—The aniline point of a petroleum oil appears to characterize the swelling action of that oil on rubber vulcanizates. In general, the lower the aniline point, the more severe the swelling action by the oil. The oils specified in Table 1 cover a range of aniline points commonly found in lubricating oils.

6.1.2 *ASTM Reference Fuels*—When gasolines or diesel fuels are to be encountered in service, the test shall be conducted in one of the ASTM reference fuels (Note 2) specified in Table 4, except as indicated in 6.1.3.

NOTE 2—The ASTM reference fuels in Table 4 have been selected to provide the maximum and minimum swelling effects produced by commercial gasolines. Reference Fuel A has a mild action on rubber vulcanizates and produces results of the same order as low swelling gasolines of the highly paraffinic, straight run type. Reference Fuels B, C, and D simulate the swelling behavior of the majority of commercial gasolines, with Reference Fuel C producing the highest swelling which is typical of highly aromatic premium grades of automotive gasoline. Reference Fuel F (diesel fuel) swells rubber vulcanizates to a lesser extent than Reference Fuel B. Reference Fuels G, H, and I are fuel-alcohol blends (gasohol), which have a stronger swelling action than the corresponding fuel alone, where blends with methanol are more severe than blends with ethanol. Reference Fuel K, a methanol-rich blend, has a substantially weaker swelling action than that of the fuel used to prepare the blend. Reference Fuels I and K are also referred to as M15 and M85, respectively.

TABLE 4 ASTM Reference Fuels

Fuel Type	Composition, Volume %
Reference Fuel A	Isooctane^{A} , 100
Reference Fuel B	Isooctane^{A} , 70; Toluene^{A} , 30
Reference Fuel C	Isooctane^{A} , 50; Toluene^{A} , 50
Reference Fuel D	Isooctane^{A} , 60; Toluene^{A} , 40
Reference Fuel E	Toluene^{A} , 100
Reference Fuel F	Diesel Fuel, Grade No. 2^{B} , 100
Reference Fuel G	Fuel D, 85; anhydrous denatured ethanol^{C} , 15
Reference Fuel H	Fuel C, 85; anhydrous denatured ethanol^{C} , 15
Reference Fuel I	Fuel C, 85; anhydrous methanol, 15
Reference Fuel K	Fuel C, 15; anhydrous methanol, 85

^{A} Motor Fuels, Section 1, Test Method D2699

^{B} Specification D975.

^{C} Anhydrous ethanol denatured with unleaded gasoline, Section 4, Performance Requirements, Specification D4806.

6.1.3 *Service Liquids*—Some commercial oils, fuels and other service liquids (see Table 5) are either non-petroleum or are compounded from special petroleum hydrocarbon fractions or mixtures of petroleum hydrocarbon fractions and other ingredients resulting in materials having properties beyond the range of the reference fluids listed in Table 1 and Table 4. Immersion tests of rubber vulcanizates that are to come into contact with such fluids should be made in the actual service liquid.

6.1.4 *Water*—Since the purity of water varies throughout the world, all water immersion tests shall be conducted in distilled or deionized water.

TABLE 5 ASTM Service Liquids

Liquid	Composition
Service Liquid 101^{A}	di-2 ethyl hexyl sebacate, 99.5 mass %; phenothiazine, 0.5 mass %
Service Liquid 102^{B}	IRM 901, 95 mass %; hydrocarbon compound additive^{C} , 5 mass % (29.5–33.5 mass % sulfur, 1.5–2.0 mass% phosphorus, 0.7 mass % nitrogen)
Service Liquid 103^{D}	tri-*n*-butyl phosphate, 100 mass %
Service Liquid 104^{E}	ethylene glycol^{F} , 50 volume %; distilled water, 50 volume %
Service Liquid 105^{G,H}	ASTM Reference Oil TMC 1006
Service Liquid 106^{I,H}	ARM 200 (Aerospace Reference Material 200)

^{A} Service liquid 101 is intended to simulate the swelling action of synthetic diester-type lubricating oils.

^{B} Service liquid 102 approximates the swelling behavior of hydraulic oils.

^{C} This hydrocarbon oil additive properties are as follows: Kinematic viscosity (mm^2/s [cSt]) at 99°C (210°F) – 9.70 ± 0.52 (Test Method D445); Flash point COC °C (°F) – 45 (113) min (Test Method D92); Density at 16°C (61°F) – 1.065 ± 0.015 (Test Method D1217).

^{D} Service liquid 103 simulates phosphate ester-type aircraft hydraulic oils.

^{E} Service liquid 104 approximates the swelling behavior of automotive engine coolant.

^{F} Ethylene glycol, reagent grade, shall be used.

^{G} Service Liquid 105 is an API SJ/ILSAC GF-2 SAE 5W-30 reference engine oil meeting the requirements of Specification D4485 and SAE J 300. The sole source of supply known to the committee at this time is ASTM Test Monitoring Center, 6555 Penn Avenue, Pittsburgh, PA 15206–4489. Fax: (412) 365–1047.

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

^{I} Service Liquid 106 is intended to eventually replace Service Liquid 101 which is no longer readily available for purchase as a mixture. The sole source of supply of ARM 200 known to the committee at this time is SAE, 400 Commonwealth Drive, Warrendale, PA 15096.

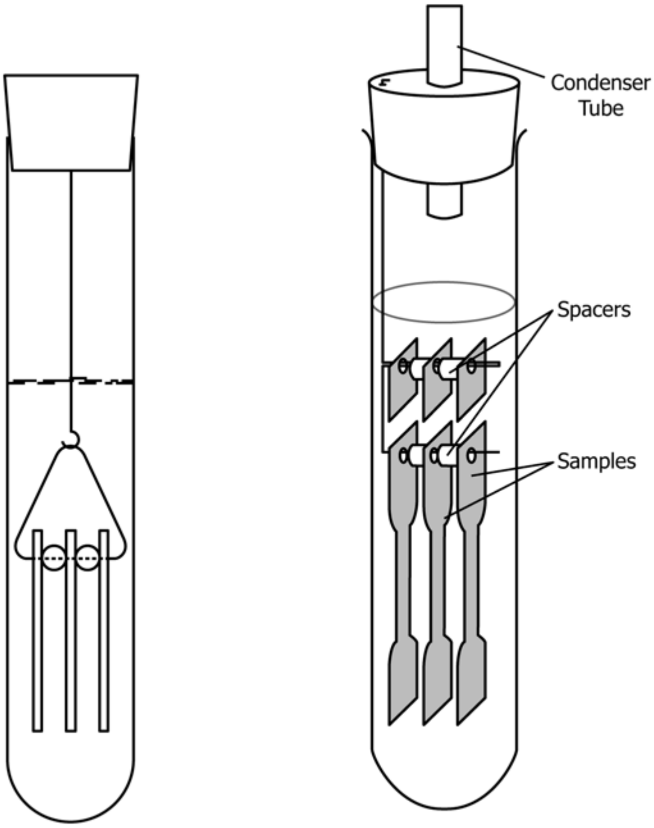

FIG. 1 Methods of Separation Showing Single Level Specimen Hangar (Left) and Dual Level Specimen Hangar (Right)

7. Preparation of Specimen

7.1 Except as otherwise specified in the applicable specifications, specimens shall be prepared in accordance with the requirements of Practices D3182 and D3183.

8. Apparatus

8.1 *For non-volatile liquids, a glass test tube,* having an outside diameter of 38 mm (1.5 in.) and an overall length of 300 mm (12 in.) fitted loosely with a stopper (see 8.2.1) shall be used. Glass beads shall be used in the liquid as a bumper and to separate the specimens; refer to 8.3.1 and Fig. 1.

8.2 *For volatile liquids,* the test tube described in 8.1 shall be tightly fitted with a stopper (see 8.2.1) and a reflux condenser. An air-cooled reflux condenser shall be used for ASTM Oils No. 1[5,6] and No. 5 and for IRM 902[5,6] and IRM 903[5,6] at test temperatures of 125°C (257°F) or more. This condenser shall be a glass tube, approximately 500 mm (20 in.) or longer, in length and 15 mm (0.6 in.) in outside diameter, tightly fitted to the immersion tube by means of a stopper (see 8.2.1). The bottom of the condenser tube shall project about 12 mm (0.47 in.) below the stopper. A suitable (water-cooled) reflux condenser shall be used for water or other low-boiling liquids. Glass beads shall be used as in 8.1.

8.2.1 The stopper shall not contaminate the test liquid. When in doubt, cover the stopper with aluminum foil.

8.3 *Maintaining Test Temperatures*—The apparatus and method chosen to maintain the specified temperature during immersion testing varies with test conditions, requirements, and circumstances. Water baths, appropriate temperature transfer oil baths, hot air ovens Note 3, or aging blocks may be used.

NOTE 3—When hot air ovens are used, it should be noted that contamination with volatile components of the immersion liquid may occur. This can affect test results, when the same ovens are subsequently used for hot air aging.

8.3.1 The preferred method for elevated-temperature testing is the use of aging block ovens (test tube type), as they have the widest temperature capability and are designed specifically for fluid immersion tests as described in Test Method D865.

8.3.2 *Ovens*—Type IIB ovens specified in Specification E145 are satisfactory for use through 70°C. For higher temperatures, Type IIA ovens are necessary.

8.3.2.1 The interior size shall be as follows or of an equivalent volume:

Interior size of air oven:

min	300 by 300 by 300 mm (12 by 12 by 12 in.)
max	900 by 900 by 1200 mm (36 by 36 by 48 in.)

8.3.2.2 Provision shall be made for placing immersion test tubes in the oven without touching each other or the sides of the aging chamber and allowing proper air circulation around them.

8.3.2.3 The heating medium for the aging chamber shall be air circulated within it at atmospheric pressure.

8.3.2.4 The source of heat is optional but shall be located in the air supply outside of the aging chamber proper.

8.3.2.5 A recording thermometer located in the upper central portion of the chamber near the center of the aging specimens shall be provided to record the actual aging temperature.

8.3.2.6 Automatic temperature control by means of thermostatic regulation shall be used.

8.3.2.7 The following special precautions shall be taken in order that accurate, uniform heating is obtained in all parts of the aging chamber:

8.3.2.8 The heated air shall be thoroughly circulated in the oven by means of mechanical agitation. When a motor-driven fan is used, the air must not come in contact with the fan motor brush discharge because of danger of ozone formation.

8.3.2.9 Baffles shall be used as required to prevent local overheating and dead spots.

8.3.2.10 The thermostatic control device shall be so located as to give accurate temperature control of the heating medium. The preferred location is adjacent to the recording thermometer.

8.3.2.11 An actual check shall be made by means of maximum reading thermometers placed in various parts of the oven to verify the uniformity of the heating.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:D11-1004 (for ASTM Oil No. 1) and RR:D11-1069 (for IRM 902 and 903). Detailed information on IRM 901 will be available in a forthcoming Research Report.

[6] The sole source of supply of ASTM Oil No. 5, IRM 901, IRM 902, and IRM 903 known to the committee at this time is R. E. Carroll, Inc., P.O. Box 5806, Trenton, NJ 08638-0806. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

9. Alternative Test Specimens and Dual Level Specimen Hangars

9.1 Alternative specimens to those enumerated in Sections 10, 11, 12, and 14 shall be 25 by 25 by 2.0 ± 0.1 mm or 1.0 by 1.0 by 0.079 ± 0.004 in. and prepared as described in the test specimen sections of each.

9.1.1 The employment of alternative specimens shall be noted in the report, refer to 19.1.12, or when the specimen differs in size or configuration from that described in the procedure.

9.2 When molded specifically for immersion tests, or as non-immersed controls, the specimens shall be obtained from the same (single source) sample.

9.3 The specimens may be used in performing different post-immersion tests.

9.4 When employing a dual level specimen hangar (Fig. 1), immersion specimens shall be from a single source (sample) and identical composition. Immersion and specimen suspension shall follow the procedure described in Section 8.

9.5 When combining specimens of differing configurations, such as those shown in Fig. 1, on a dual specimen hangar, the procedure for immersion of each shall be followed. However, caution must be exercised to assure that the specimens are completely immersed in the immersion media and it may be necessary to use more than 100 cm^3 (100 mL) of the immersion media, that is, 150 cm^3 (150 mL) as described in Section 15, and potentially as much as 240 cm^3 (240 mL). This shall be noted in the report, refer to 19.1.12.

10. Test Specimens—Change in Mass or Volume

10.1 The standard specimen shall be rectangular, having dimensions of 25 by 50 by 2.0 ± 0.1 mm or 1.0 by 2.0 by 0.079 ± 0.004 in. Specimens from commercial articles shall be the thickness of the material as received when they are less than 2.0 mm or 0.079 in.; otherwise they should be reduced to a thickness of 2.0 mm or 0.079 in. Data obtained on test specimens having different original thicknesses are not comparable.

11. Procedure for Change in Mass

11.1 Test three specimens of a single composition. Calculate the test results of the three specimens (see 18.1, Eq 1) and average the results. Weigh each specimen in air to the nearest 1 mg, record the mass as M_1 , and immerse in the test tube (see 8.1) containing 100 cm^3 of the test liquid (see 11.1.1). Separate each test specimen from any adjacent test specimen and the walls of the test tube by approximately 6 mm (0.25 in.), for example, by perforated glass beads[7] as shown in Fig. 1. The materials used to suspend and separate the specimens shall not affect the test liquid or the rubber.

11.1.1 Test liquids shall not be reused.

11.1.1.1 For non-volatile liquids, condition the test tube assembly at the specified temperature within the limits given in 5.1 for the specified length of time. A check of the actual temperature of the test liquid contained in the test tube should be made to ensure that the liquid is within the test temperature specified.

11.1.1.2 For volatile liquids, fit the test tube assembly with a reflux condenser (see 8.2) and condition at the specified temperature within the limits given in 5.1 for the specified length of time. A check of the actual temperature of the test liquid contained in the test tube should be made to ensure that the test liquid is within the test temperature specified.

11.2 After the immersion test has proceeded for the required length of time, remove the test specimens. If the immersion has been at elevated temperature, cool the test specimens to room temperature by transferring them to a cool clean portion of the test liquid for 30 to 60 min. Then dip the specimens quickly in acetone (see Note 4) at room temperature, blot lightly with filter paper free of lint and foreign material, place them immediately in a tared, stoppered weighing bottle, and determine the mass after test, M_2, of each specimen to the nearest 1 mg. After weighing, again immerse the test specimen in the same test liquid (see 12.2.1) if data are desired on the progressive changes that occur with increasing time of immersion. It is important that each manipulation take place promptly with the least possible lapse of time. When utilizing liquids that tend to volatilize at room temperature, no more than 30 s should elapse between removal from the test liquid and stoppering the weighing bottle.

11.3 Relatively viscous oils may be difficult to remove from the specimens with a quick acetone (see Note 4) dip and blotting or even more vigorous wiping when the specimens are cooled in the test liquid after immersion; this can yield erroneous test results. Since these oils do not readily volatilize, specimens may be cooled by suspending them for about 30 min in air at room temperature shielded from draft, after removal from the immersion liquid at the test temperature. This will allow the majority of the oil to drip off the surface of the specimen. Then proceed with the acetone (see Note 4) dip and blotting as described in 11.2. Report when this alternate method of specimen cooling is used.

12. Procedure for Change in Volume

12.1 *Water Displacement Method for Water-Insoluble Liquids and Mixed Liquids*—Test three specimens, calculate the test results for each specimen (see 18.2, Eq 2), and average the results. Obtain the mass M_1 of each specimen (see 12.2.2) in air, to the nearest 1 mg, and then obtain the mass M_2 of each specimen immersed in distilled water at room temperature. Quickly dip each specimen in alcohol (methanol or ethanol (see Note 4)) to remove water (see 12.2.3), blot dry with filter paper free of lint and foreign material, and place in the immersion apparatus described in 8.1 or 8.2. Add 100 cm^3 of liquid (see 11.1.1) to the test tube and complete the immersion test as described in 11.1.1.1 or 11.1.1.2. At the end of the required immersion period, remove each specimen from the

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[7] The sole source of supply of hollow perforated glass beads (Catalog No. 11-311C) known to the committee at this time is Fisher Scientific Co., 711T Forbes Ave., Pittsburgh, PA 15219. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

test tube. Cool the specimens to room temperature by transferring them to a cool, clean portion of the test liquid for 30 to 60 min, then dip quickly in acetone (see Note 4) at room temperature, blot lightly with filter paper free of lint or foreign material, place in a tared, stoppered weighing bottle, weigh, and record the mass as M_3. Remove each specimen from the bottle, weigh in distilled water, and record the mass as M_4 in immediate consecutive order to determine the water displacement after immersion (see 12.2.2 and Note 5). When data on progressive changes with increasing time of immersion are desired, dip each specimen after weighing in alcohol (methanol or ethanol (see Note 4)) to remove water (see 12.2.3), blot dry with filter paper free of lint and foreign material, and again immerse in the same test liquid (see 12.2.1). It is important that each manipulation following removal of the test specimen from the test medium take place promptly with the least possible lapse in time. When utilizing liquids that tend to volatilize at room temperature, no more than 30 s should elapse between removal from the test liquid and stoppering the weighing bottle, and no more than an additional 30 s between removal from the weighing bottle and immersion in water.

12.2 Relatively viscous oils may be difficult to remove from the specimens with a quick acetone (see Note 4) dip and blotting or even more vigorous wiping when specimens are cooled in the test liquid after immersion; this can yield erroneous test results. Refer to 11.3 for an alternate method of specimen cooling.

NOTE 4—Acetone, methanol, or ethanol may have an adverse effect on some materials, it is acceptable to use isopropyl alcohol (IPA) or other highly evaporative evaporate substance which will not have a deleterious effect on the material or produce a lasting residue. The use of an alternative substance shall be reported as described in 19.1.12.

12.2.1 Report if fresh test liquid is used, which can be a requirement for certain test methods or specifications.

12.2.2 It is important that all air bubbles clinging to the test specimen be removed before weighing in distilled water. If, in the course of weighing, air bubbles appear on the surface of the specimen, or the computed volume changes 0.5 % in 5 min, the specimen is too porous to permit volume determination in this manner. In that case, the initial volume of the specimen, if the latter is a simple geometrical solid, can be determined from the overall dimensions by employing an appropriate mensuration formula, and the same procedure followed in determining the volume after the immersion test; or, if volume increase occurs principally in the thickness dimension, a simple change in thickness may be substituted for change in volume. If, in the course of weighing the test specimen floats, AISI No. 316 stainless steel can be used as ballast to immerse the test specimen in the test liquid. In those cases where ballast is needed to submerge the specimen, the following procedure may be used.

12.2.2.1 Weigh the test specimen with ballast in water,

12.2.2.2 Weigh the ballast alone in water, and

12.2.2.3 Determine the difference between weighings, and proceed with the calculations.

12.2.3 The acetone (see Note 4) dip before and after immersion shall be omitted when water is employed as the test medium.

NOTE 5—A Jolly balance, adequately shielded from air currents, may be used for making these determinations. When utilizing liquids that tend to volatilize at room temperature, no more than 30 s should elapse between removal from the test liquid and completion of the weighing operation.

12.3 *Displacement Method for Water-Soluble Liquids and Mixed Liquids (Other Than Water)*—For immersion liquids that are readily miscible with water or react with it, the water displacement method as described in 12.1 may not be suitable. For such liquids that are not too viscous or volatile at room temperature, weighings for M_2 and M_4 may be made in the immersion liquid instead of water with weighing for M_4 made in a fresh portion of the immersion liquid. These values are then used in calculating volume change using Eq 2 in 18.2. If this is not practicable, the water displacement method shall be used, except that the final weighing for M_4 is omitted and change in volume is calculated according to Eq 3 in 18.2. This formula may be only approximate if the immersion liquid is a mixture, because the density of the absorbed liquid may differ from that of the bulk. Also the density of any matter extracted from the rubber may differ from that of the immersion liquid.

13. Dimensional-Change Method for Water-Insoluble Liquids and Mixed Liquids

13.1 *Dimensional Change Method*—Measure the original length and width of three test specimens used in 12.1 to the nearest 0.5 mm (0.02 in.) using an average of three measurements in each direction recording the dimensions as L_0 and W_0, respectively. Measure the thickness of each test piece using a dial micrometer as described in 6.3 of Test Methods D412, and record as T_0. Place specimens in the immersion apparatus described in 8.1 and 8.2. Add 100 cm^3 of liquid (see 11.1.1) to the test tube and complete the immersion test as described in 11.1.1.1 or 11.1.1.2. At the end of the required immersion period, remove each specimen from the test tube and bring to room temperature by transferring them to a cool clean portion of the test liquid for 30 to 60 min. Dip the specimens quickly in acetone (see Note 4) at room temperature, blot lightly with filter paper free of lint and foreign material, and promptly remeasure as described above, recording the immersed length, width, and thickness as L, W, and T, respectively. If the immersion liquid tends to volatilize at room temperature, the measurements should be completed within 30 s after removing the test specimens from the liquid at room temperature. Calculate test results according to Eq 4 in 18.3 and report the average of three specimens.

14. Procedure for Change in Mass with Liquid on One Surface Only

14.1 *Scope*—This test method provides a procedure for exposing thin sheet materials (rubber-coated fabrics, diaphragms, etc.) that are in contact with the immersion liquid on only one surface under definite conditions of time and temperature.

14.2 *Test Specimen*—A disk 60 mm (2.4 in.) in diameter and thickness of thin sheet or rubber-coated fabric being tested.

14.3 *Apparatus*—The essential features are illustrated in Fig. 2 and consist of a base plate, *A*, an open-ended cylindrical chamber, *B*, which is held tightly against the test specimen, *C*,

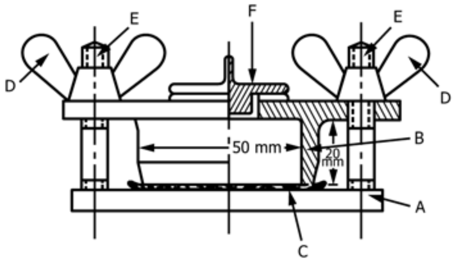

FIG. 2 Typical Surface Immersion Apparatus

by wing nuts, *D*, mounted on bolts, *E*. During the test, the opening in the top of the chamber is tightly closed by a suitable plug, *F*.

14.4 *Procedure*—Weigh the test specimen in air to the nearest 1 mg, record as M_1, and place in the apparatus as indicated in Fig. 2. Fill the chamber of the apparatus with the test liquid to a depth of 15 mm (0.6 in.), replace plug *F* and complete the test at the specified conditions as described in 5.1 and 5.2. At the end of the required immersion period, bring the apparatus to standard room temperature, remove the test liquid, and release the test specimen. Remove any excess liquid from the surface by wiping or blotting with filter paper free of lint and foreign material and place the specimen immediately in a tared, stoppered weighing bottle. Determine the mass of the specimen to the nearest 1 mg and record as M_2. When the immersion liquid tends to volatilize at room temperature, no more than 30 s should elapse between removal of the test specimen from the liquid and stoppering the weighing bottle. Use Eq 5 in 18.4 to calculate test results. If more than one specimen of the same composition is tested, report the result as the average.

15. Procedure for Determining Mass of Soluble Matter Extracted by the Liquid

15.1 This test method is applicable for immersions in volatile-type immersion liquids, such as ASTM reference fuels (see 6.1.2). Conduct the immersions as described in 11.1, 11.1.1.1, and 11.1.1.2. Test three specimens of a single composition, and report the result as the average. The extracted matter can be determined either by the difference in mass of the specimen before and after immersion or by evaporating the test liquid and weighing the non-volatile residue (Note 6). The mass is determined in air to the nearest 1 mg.

NOTE 6—Both methods are subject to error. Results obtained by the mass difference of specimens before and after immersion can be affected by possible oxidation of the material during immersion. Results obtained by evaporating the test liquid may be affected by partial loss of volatile extracted material during drying. The method of choice will depend on the material under test and the test conditions. For example, the evaporation method would be preferred if further testing is to be done on the extracted matter.

15.2 *Mass-Difference Method*—At the end of the required immersion period, remove the test specimens from the test tube and dry to a constant mass at a temperature of approximately 40°C (104°F) and an absolute pressure of 20 kPa (150 mm Hg). Record the original mass of the test specimen before immersion as M_1, the mass after immersion as M_2, and express the change in mass as a percentage of the original mass, using Eq 6 in 18.5. Report the results as the average if more than one specimen of the same composition is tested.

15.3 *Evaporation Method*—Transfer the liquid from the test tube in which the three test specimens have been immersed to a suitable vessel, wash the specimens in the test tube three times with 25 cm^3 of fresh liquid and transfer the liquid to the same vessel. Evaporate the liquid and dry the residue to a constant mass under an absolute pressure of 20 kPa (150 mm Hg) and a temperature of approximately 40°C (104°F). Record the sum of the original mass of three specimens before immersion as $\sum M_1$, the mass of the dried residue as M_3, and express M_3 as a percentage of $\sum M_1$, using Eq 7 in 18.5.

16. Changes in Tensile Strength, Elongation, and Hardness

16.1 *Original Properties*—The original tensile strength, ultimate elongation (Test Methods D412, Die C) and hardness (Test Methods D1415 or D2240) shall be determined, using three specimens cut from the sheet or article adjacent to those that are to be immersed in the liquid. Report results in accordance with the appropriate test method.

16.2 *Immediate Deteriorated Properties*—For determining the tensile strength, ultimate elongation, and hardness, prepare three test specimens from flat vulcanized sheets 2.0± 0.1 mm (0.08 ± 0.004 in.) in thickness (see 10.1) using Die C of Test Methods D412. Measure the thickness of each test specimen, place the specimens in the test tube described in 8.1, and add 150 cm^3 of liquid (see 11.1.1) to the test tube. Complete the immersion period in accordance with 11.1.1.1 or 11.1.1.2. At the end of the required immersion period, remove the specimens from the test tubes and cool immediately to room temperature in a fresh sample of the same liquid for 30 to 60 min. Then quickly dip each sample in acetone (see Note 4) and blot lightly with filter paper that is free of lint and foreign material and immediately apply bench marks.

16.2.1 Determine the tensile strength and ultimate elongation in accordance with Test Methods D412, using the original unimmersed thickness or cross-sectional area (see 18.6, Eq 8). Determine the hardness of each immersed specimen in accordance with Test Methods D1415 or D2240. The time interval between removal from the cool liquid and testing shall be not less than 2 or more than 3 min.

16.2.2 It may be desirable to calculate the tensile strength based on the swollen cross-sectional area. In that case Eq 9 in 18.6 shall be used. Report when this method of calculation is employed.

16.3 Relatively viscous oils may be difficult to remove from the specimens with a quick acetone (see Note 4) dip and blotting or even more vigorous wiping when specimens are cooled in the test liquid after immersion; this can yield erroneous test results. Refer to 11.3 for an alternate method of specimen cooling.

16.4 *Properties After Evaporation of Test Liquid*—This test method is applicable for immersions in volatile-type immersion liquids, such as ASTM reference fuels (see 6.1.2). For determining the tensile strength, ultimate elongation, and hardness of specimens after evaporation of the immersion liquid, immerse the specimens in the test liquid in accordance with 16.2. At the end of the required immersion time, remove the specimens and, if necessary, cool them to room temperature in a fresh sample of the same liquid for 30 to 60 min.

16.4.1 For room temperature drying, suspend the specimens for 4 ± 0.25 h in air at ambient conditions shielded from drafts. After the drying period apply bench marks and determine tensile strength and ultimate elongation in accordance with Test Methods D412 using the original thickness or cross-sectional area of the untreated specimens (see 18.6, Eq 8) and hardness according to Test Methods D1415 or D2240.

16.4.2 For determining properties after complete evaporation of the test liquid, suspend specimens after the 4-h air-drying cycle at room temperature for 20 h ± 15 min at 70 ± 2°C (158 ± 4°F) in a circulating-air oven. After the heat drying cycle, remove the specimens from the oven, allow them to cool to room temperature and measure the required physical properties as described in 16.4.1. Report when the heat drying procedure is used.

16.5 The tensile strength and ultimate elongation after immersion and evaporation also can be expressed as percent change from the original values, using Eq 10 in 18.6, and hardness in unit change from the original value, according to Eq 11 in 18.6.

17. Changes in Breaking Resistance, Burst Strength, Tear Strength, and Adhesion for Coated Fabrics

17.1 *Original Properties*—The original properties for breaking resistance, burst strength, tear strength, and adhesion shall be determined in accordance with Test Methods D751, using the specified number of specimens cut from the coated fabric adjacent to those that are to be immersed in the liquid, or, if necessary, from other pieces of coated fabric that were produced from the same lot(s) of materials under the same conditions.

17.2 *Immediate Deteriorated Properties*—For determining breaking resistance, burst strength, tear strength, and adhesion, prepare the number of specimens specified for each parameter in Test Methods D751. Place the specimens in the test tube described in 8.1 and add 150 cm^3 of liquid (see 11.1.1) to the test tube. Complete the immersion test in accordance with 11.1.1.1 or 11.1.1.2. At the end of the required immersion period, remove the specimens from the test tubes, and cool immediately to room temperature in a fresh sample of the same liquid for 30 to 60 min. Then quickly dip each specimen in acetone (see Note 4) and blot lightly with filter paper that is free of lint or foreign material. Measure the required physical properties in accordance with Test Methods D751 (see 18.7). The time interval between removal from the cool liquid and testing shall be not less than 2 min or more than 3 min.

17.3 Relatively viscous oils may be difficult to remove from the specimens with a quick acetone (see Note 4) dip and blotting or even more vigorous wiping when specimens are cooled in the test liquid after immersion; this can yield erroneous test results. Refer to 11.3 for an alternate method of specimen cooling.

17.4 *Properties After Evaporation of Volatile Test Liquid*—For determining breaking resistance, burst strength, tear strength, and adhesion of specimens after evaporation of a volatile immersion liquid, such as ASTM reference fuels (see 6.1.2), immerse the specimens in the test liquid in accordance with 16.2. At the end of the required immersion time, remove the specimens, suspend them for 2 h ± 5 min at ambient conditions shielded from draft and then place them in a circulating-air oven at a temperature of 70 ± 2°C (158 ± 4°F) for a period of 2 h ± 5 min. At the end of the drying period, remove the specimens from the oven, allow them to cool to room temperature and measure the required physical properties in accordance with Test Methods D751 (see 18.7). The time interval between removal from the oven and testing shall be not less than 1 h or more than 2 h.

17.5 If desired, report results after immersion and evaporation in percent change from the original property values, as described in 16.5.

18. Calculation

18.1 Calculate the percent change in mass as follows (Section 11):

$$\Delta M, \% = \frac{(M_2 - M_1)}{M_1} \cdot 100 \quad (1)$$

where:

ΔM = change in mass, %,
M_1 = initial mass of specimen in air, g, and
M_2 = mass of specimen in air after immersion, g.

18.2 Calculate the percent change in volume as follows (Section 12):

$$\Delta V, \% = \frac{(M_3 - M_4) - (M_1 - M_2)}{(M_1 - M_2)} \cdot 100 \quad (2)$$

$$\Delta V, \% = \frac{(M_3 - M_1)}{d(M_1 - M_2)} \cdot 100 \quad (3)$$

where:

ΔV = change in volume, %,
M_1 = initial mass of specimen in air, g,
M_2 = initial mass of specimen in water, g,
M_3 = mass of specimen in air after immersion, g,
M_4 = mass of specimen in water after immersion, g, and
d = density of immersion liquid at standard laboratory room temperature, Mg/m^3.

18.3 Calculate the percent change in dimensions as follows (Section 13):

$$\Delta L, \% = \frac{(L - L_0)}{L_0} \cdot 100 \quad (4)$$

where:

ΔL = change in length, %,
L_0 = initial length of specimen, mm, and
L = length of specimen after immersion, mm.

Calculate the percent change in width, ΔW, and thickness, ΔT, accordingly.

18.4 Calculate the change in mass for single surface exposure as follows (Section 14):

$$\Delta M_A, \text{kg/m}^2 = \frac{(M_2 - M_1)}{A} \cdot 1000 \quad (5)$$

where:

ΔM_A = change in mass per unit surface area, kg/m^2,
M_1 = initial mass of specimen in air, g,
M_2 = mass of specimen in air after exposure to the test liquid, g, and
A = area of the specimen in actual contact with the test liquid, mm^2.

18.5 Calculate percent extracted soluble matter as follows (Section 15):

$$M_E, \% = \frac{(M_1 - M_2)}{M_1} \cdot 100 \quad (6)$$

$$M_E, \% = \frac{M_3}{\sum M_1} \cdot 100 \quad (7)$$

where:

M_E = extracted soluble matter, %,
M_1 = initial mass of specimen in air, g,
M_2 = mass of specimen in air after immersion and drying, g,
$\sum M_1$ = sum of the initial mass of three specimens in air, g, and
M_3 = mass of the residue in air after evaporating and drying the immersion liquid of three specimens, g.

18.6 Calculate tensile strength of the specimens both on immediate deteriorated properties and properties after evaporation of the test liquid as described in the Calculation section of Test Methods D412.

18.6.1 Based on the original unstretched cross-sectional area:

$$TS_o = \frac{F}{A} \quad (8)$$

18.6.2 Based on the swollen unstretched cross-sectional area:

$$TS_s = \frac{F}{A\left(1 + \frac{\Delta V}{100}\right)^{2/3}} \quad (9)$$

18.6.3 To express tensile strength and ultimate elongation after immersion as a percentage change from the original properties, use the following formula:

$$\Delta P, \% = \frac{P_i - P_o}{P_o} \cdot 100 \quad (10)$$

18.6.4 Calculate hardness change after immersion in hardness units:

$$\Delta H = H_i - H_o \quad (11)$$

where:

TS_o = tensile stress based on original unstretched cross-sectional area,
TS_s = tensile stress based on swollen unstretched cross-sectional area,
F = observed force,
A = original unstretched cross-sectional area of the test specimen before immersion,
ΔV = volume swell after immersion, %,
ΔP = change in property (tensile strength and ultimate elongation) after immersion, %,
P_o = original property before immersion,
P_i = property after immersion,
ΔH = hardness change after immersion, units,
H_o = original hardness before immersion, units, and
H_i = hardness after immersion, units.

18.7 Calculate breaking resistance, burst strength, tear strength, and adhesion both on the immediate deteriorated properties and properties after evaporation of volatile test liquid as described in Test Methods D751. If desired, results may also be expressed in percentage change after immersion as detailed in 18.6.3.

19. Report

19.1 State that the test was conducted in accordance with Test Method D471 and report the following:

19.1.1 Description of the sample and its origin,

19.1.2 Date and temperature of testing room (see 5.1),

19.1.3 Duration, temperature, and date of vulcanization of test specimens,

19.1.4 Dates of the various periods of exposure,

19.1.5 Immersion liquid used,

19.1.6 Temperature of exposure,

19.1.7 Exposure period,

19.1.8 All observed and recorded data, to include the type of properties being reported,

19.1.9 Results calculated in accordance with Section 18,

19.1.10 Statement of condition of exposed specimens from visual and manual examination,

19.1.11 Report which test method was used for determination of hardness, and

19.1.12 Any deviations from standard test methods.

20. Precision and Bias[8]

20.1 This precision and bias section deals with property changes in tensile strength, ultimate elongation, hardness, and volume after immersion in test liquids and has been prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical calculation details.

20.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials used in the particular interlaboratory test program as described in the following paragraphs. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that the parameters are applicable to the particular group of materials and the specific testing protocols of the test method.

20.3 A Type 1 interlaboratory test program was evaluated in 1981 using six different vulcanized rubber compounds (materials). These compounds were prepared in one laboratory and test specimens were distributed to nine participating laboratories. Each laboratory prepared and purchased from approved sources, the liquids and fuels, or both, used for testing. A test

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[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1069.

result (as used for these calculations) is the average of three individual test specimen values. Each laboratory conducted tests (that is, obtained one test result) on each of two days. Both repeatability and reproducibility are therefore short term; a period of a few days separates test results.

20.4 The results of the precision evaluation are given in Table 6 and Table 7 for each of the six rubbers or materials for immersion tests in (*1*) Reference Fuels B, C and D, for 70 h at 23 ± 2°C (73 ± 4°F), and (*2*) ASTM Oil No. 2 and ASTM Oil No. 3 for 70 h at 150 ± 2°C (302 ± 4°F).

20.5 The precision is given in terms of *Sr, r, SR*, and *R* for four measured physical properties: (*1*) percent change in tensile strength, (*2*) percent change in ultimate elongation (that is, percent change in percent elongation), (*3*) hardness change in hardness units, and (*4*) percent change in volume of the test specimen. No values are given for (*r*) and (*R*), the normal precision parameters used to express relative precision because numerous average values for properties (*1*), (*2*), and (*4*) are near zero, thus resulting in extremely large (*r*) and (*R*) values. These large values are essentially meaningless for precision comparisons when average values are near zero.

20.6 The precision of the test method may be expressed in the following statements that use an appropriate value of *r* and *R*. The appropriate value is that value of *r* or *R*, associated with a mean level in the tables closest to the mean level under consideration for any immersion liquid or rubber, for any test result for a similar material in routine testing operations.

20.7 *Repeatability*—The repeatability, *r*, of this test method has been established as the appropriate value tabulated in Table 6 and Table 7. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from nonidentical sample populations.

20.8 *Reproducibility*—The reproducibility, *R*, of this test method has been established as the appropriate value tabulated in Table 5 through Table 7. Two single test results obtained in two different laboratories, under normal test method

TABLE 6 Type 1 Precision Data[A]

Rubber	Change in Tensile Strength, %					Change in Elongation,%					Change in Hardness, Units				
	Within-Lab			Between-Lab		Within-Lab			Between-Lab		Within-Lab			Between-Lab	
	Mean	*Sr*	*r*	*SR*	*R*	Mean	*Sr*	*r*	*SR*	*R*	Mean	*Sr*	*r*	*SR*	*R*
ASTM Reference Fuel B:															
CR	–48.0	3.12	8.8	3.26	9.2	–43.7	4.55	12.9	5.36	15.2	–16.0	0.76	2.1	1.87	5.3
FKM	–7.9	1.94	5.5	3.55	10.0	–0.1	3.25	9.2	3.58	10.1	–2.3	0.90	2.6	1.68	4.8
FVMQ	–30.6	4.17	11.8	6.08	17.2	–27.1	2.47	7.0	7.05	19.9	–9.6	1.12	3.2	2.16	8.1
NBR-1	–63.6	2.90	8.2	4.27	12.1	–50.3	3.44	9.7	7.24	20.5	–9.8	0.87	2.5	1.32	3.7
NBR-2	–11.8	2.59	7.3	3.75	10.6	–16.3	2.41	6.8	4.55	12.9	–12.7	1.15	3.3	1.73	4.9
CO	–8.6	3.35	9.5	5.23	14.8	–26.0	4.19	11.9	3.04	8.6	–12.4	1.00	2.8	1.68	4.8
Pooled Values	...	3.07	8.7	4.47	12.6	...	3.52	10.0	5.38	15.2	...	0.98	2.8	1.76	5.0
ASTM Reference Fuel C:															
CR	–57.4	2.70	7.6	3.49	9.9	–54.2	3.51	9.9	5.47	15.5	–17.7	1.58	4.5	3.36	9.5
FKM	–11.1	3.32	9.4	6.34	17.9	–0.6	2.29	6.5	6.55	18.5	–2.8	0.80	2.3	1.64	4.3
FVMQ	–30.9	2.74	7.8	7.20	20.4	–28.4	4.01	11.3	6.83	19.4	–9.7	1.76	5.0	2.87	8.1
NBR-1	–68.9	2.77	7.8	4.17	11.8	–58.1	5.27	14.9	5.53	15.7	–10.7	1.33	3.8	1.22	3.5
NBR-2	–14.8	3.78	10.7	5.15	14.6	–21.0	2.87	8.1	6.30	17.8	–18.3	1.12	3.2	2.95	8.3
CO	–18.0	3.24	9.2	3.38	9.6	–33.9	4.52	12.8	2.64	7.5	–17.3	1.18	3.3	1.85	5.2
Pooled Values	...	3.10	8.8	5.16	14.6	...	3.89	11.0	5.73	16.2	...	1.35	3.8	2.43	6.9
ASTM Reference Fuel D:															
CR	–54.8	2.69	7.6	2.36	6.6	–50.9	1.83	5.2	3.62	10.2	–16.9	0.60	1.7	2.57	7.3
FKM	–9.2	2.26	6.4	6.00	17.0	–0.2	2.83	8.0	6.83	19.3	–1.9	0.85	2.4	2.46	6.9
FVMQ	–33.2	3.45	9.8	5.90	16.7	–28.5	3.44	9.7	7.13	20.2	–9.6	1.39	3.9	2.48	7.0
NBR-1	–67.4	2.01	5.7	4.00	11.3	–55.8	2.35	6.7	5.05	14.3	–10.9	1.06	3.0	0.98	2.8
NBR-2	–12.4	3.05	8.6	2.56	7.2	–18.3	1.78	5.0	6.21	17.6	–15.1	0.79	2.2	3.39	9.6
CO	–14.6	3.02	8.5	3.36	9.5	–30.0	3.08	8.7	3.31	9.4	–15.3	1.17	3.3	3.41	9.7
Pooled Values	...	2.81	7.9	4.29	12.1	...	2.63	7.4	5.63	15.9	...	1.02	2.9	2.67	7.6
ASTM Oil No. 1: (Reference Table 1 and Table X3.1.)															
CR	–15.4	2.78	7.9	5.01	14.2	–31.3	1.94	5.5	5.20	14.7	–8.6	1.39	3.9	2.80	7.9
FKM	–6.1	5.25	14.8	6.11	17.3	–9.3	4.38	12.4	5.74	16.2	–2.8	1.84	5.2	3.34	9.4
FVMQ	–4.3	5.93	16.8	9.48	26.8	–11.9	4.89	13.8	4.34	12.3	–2.4	1.54	4.4	3.20	9.0
NBR-1	–18.9	3.55	10.0	6.38	18.1	–12.5	5.07	14.3	10.49	29.7	–4.0	1.09	3.1	2.74	7.8
NBR-2	–7.7	3.38	9.6	9.67	27.4	–70.7	3.67	10.4	7.17	20.3	17.2	1.32	3.7	2.08	5.9
CO	20.2	4.81	13.6	7.25	20.5	–48.5	3.72	10.5	4.17	11.8	8.6	0.67	1.9	3.24	9.2
Pooled Values	...	4.43	12.5	7.52	21.3	...	4.06	11.5	6.55	18.5	...	1.36	3.8	3.11	8.8
ASTM Oil No. 3: (Reference Table 1.)															
CR	–42.5	3.43	9.7	4.19	11.9	–42.0	2.64	7.5	4.41	12.5	–19.1	1.27	3.6	4.46	12.6
FKM	–11.3	4.27	12.1	17.20	48.7	–2.4	4.58	12.9	7.95	22.5	–2.9	1.15	3.3	3.67	10.4
FVMQ	–18.2	2.84	8.0	10.65	30.1	–17.9	5.28	14.9	6.83	19.3	–1.9	1.25	3.5	1.74	4.9
NBR-1	–37.2	5.32	15.1	12.82	36.6	–19.5	6.12	17.3	12.19	34.5	–12.7	1.44	4.1	4.67	13.2
NBR-2	–27.0	5.67	16.0	14.64	41.4	–69.7	3.80	17.3	5.72	16.2	10.6	1.39	3.9	2.22	8.3
CO	21.7	6.50	18.4	10.29	29.1	–39.7	5.42	10.7	4.40	12.4	3.3	0.97	2.8	2.73	7.7
Pooled Values	...	4.85	13.7	12.33	34.9	...	4.76	13.5	7.55	21.4	...	1.25	3.5	3.61	10.2

[A] *Sr* = repeatability standard deviation, in measurement units; *r* = repeatability = 2.83 × *Sr*, *SR* = reproducibility standard deviation, in measurement units; and *R* = reproducibility = 2.83 × *SR*.

TABLE 7 Precision Data[A]

Rubber	Change in Volume, %				
	Within-Laboratory			Between-Laboratories	
	Mean	*Sr*	*r*	*SR*	*R*
ASTM Reference Fuel B:					
CR	66.2	0.94	2.7	5.40	15.3
FKM	1.5	0.36	1.0	0.34	1.0
FVMQ	5.1	0.48	1.4	0.92	2.8
NBR-1	46.9	0.38	1.1	3.96	11.2
NBR-2	12.0	0.73	2.1	0.63	1.8
CO	15.6	0.39	1.0	1.27	3.6
Pooled Values	...	0.60	1.7	2.95	8.4
ASTM Reference Fuel C:					
CR	108.9	1.04	2.9	4.99	14.1
FKM	2.6	0.46	1.3	0.32	0.9
FVMQ	17.8	0.53	1.5	1.61	4.5
NBR-1	76.8	0.59	1.7	3.47	9.8
NBR-2	22.6	0.69	2.0	0.77	2.2
CO	25.3	0.51	1.4	0.59	1.7
Pooled Values	...	0.64	1.8	2.60	7.4
ASTM Reference Fuel D:					
CR	90.6	1.52	4.3	3.15	8.9
FKM	2.3	0.33	0.9	0.56	1.6
FVMQ	15.9	0.92	2.6	3.05	8.6
NBR-1	62.2	1.23	3.5	2.10	5.9
NBR-2	18.1	1.38	3.9	1.55	4.4
CO	20.4	0.89	2.5	1.25	3.5
Pooled Values	...	1.11	3.1	2.26	6.4
ASTM Oil No. 1: (Reference Table 1 and Table X3.1.)					
CR	15.8	0.50	1.4	0.61	1.7
FKM	1.2	0.29	0.8	1.12	3.2
FVMQ	0.6	0.57	1.6	1.05	3.0
NBR-1	3.4	0.69	1.9	1.88	5.3
NBR-2	–11.2	0.45	1.3	0.47	1.3
CO	–5.2	0.42	1.2	0.64	1.8
Pooled Values	...	0.50	1.4	1.67	4.7
ASTM Oil No. 3:					
CR	75.6	1.03	2.9	1.84	5.2
FKM	3.1	1.47	4.2	1.97	5.6
FVMQ	2.8	0.61	1.7	3.75	2.4
NBR-1	31.4	0.18	1.5	4.21	11.9
NBR-2	–2.1	0.14	2.5	0.67	1.9
CO	4.2	0.24	0.7	0.77	2.2
Pooled Values	...	0.88	2.5	2.70	7.6

[A] Sr = repeatability standard deviation, in measurement units; r = repeatability = 2.83 × Sr; SR = reproducibility standard deviation, in measurement units; and R = reproducibility = 2.83 × SR.

procedures, that differ by more than the tabulated *R* (for any given value) must be considered to have come from different or nonidentical sample populations.

20.9 *Bias*—In test terminology, bias is the difference between an average test value and the reference (or true) value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias, therefore, cannot be determined.

21. Keywords

21.1 elevated temperature; fluid immersion; immersion oil; IRM reference oil; liquid immersion; reference fuel; reference oil; rubber articles; rubber products; service liquid

APPENDIXES

(Nonmandatory Information)

X1. REPLACEMENT OF ASTM OILS NO. 2 AND NO. 3 WITH IRM 902 AND IRM 903

X1.1 ASTM Oils No. 2 and No. 3 became subject to cancer hazard warning label requirements under the OSHA Hazard Communication Standard, published November 25, 1983. This contributed to the discontinuation of commercial production in 1990.

X1.2 ASTM Subcommittee D11.15 on Degradation Testing began a program in 1985–1986 to establish replacements for these oils, with the objective to match the action on vulcanized rubbers as closely as possible, while eliminating the need for cancer hazard labeling (negative modified Ames Test). An interlaboratory test program (ITP) was organized in 1993 with six candidate oils as No. 2 and No. 3 replacements from three different suppliers, using twelve different types of vulcanized rubbers. As a result of this ITP, two oils from Calumet Lubricants Company were selected as best (but not equivalent) matches for the ASTM oils, and confirmed independently by ASTM Subcommittee D11.15 and the SAE Committee on Automotive Rubber Specifications (SAE-CARS). Calumet No. 2, the replacement for No. 2 oil, provided a closer match than Calumet No. 3, the replacement for No. 3 oil. The two oils were established as industry reference materials, IRM 902 (Calumet No. 2, replacement for ASTM Oil No. 2) and IRM 903 (Calumet No. 3, replacement for ASTM Oil No. 3).

X1.2.1 Details of the ITP are described in Research Report RR:D11-1069, which can be obtained from ASTM headquarters. A precision statement on volume swells generated in this test program is given in Appendix X2.

X1.3 Since the inventory of ASTM Oils No. 2 and No. 3 was depleted by the end of 1993, Committee D-11 published Emergency Standard ES 27 in 1994 to address the replacement with IRM 902 and IRM 903.

X2. PRECISION AND BIAS—TEST PROGRAM FOR THE REPLACEMENT OF ASTM OILS NO. 2 & NO. 3

X2.1 This precision statement was prepared using data from the comprehensive ITP evaluation program conducted in 1993 to establish replacement oils for ASTM Oils No. 2 and No. 3. Because of the limited number of laboratories (three), only within-laboratory variation or repeatability could be evaluated. Some terminology used in this appendix is contained in Practice D4483. Refer to Practice D4483 for background details.

X2.2 In the evaluation program twelve compounds were tested in groups of four; each group of four was tested by three laboratories. There are only three duplicates (one duplicate set of tests in each laboratory) for an estimate of within-laboratory variation for any combination of rubber and oil. This is an inadequate number of degrees of freedom (DF) for such an estimate. However, a reasonable assumption may be made that for any given rubber, the true test variation with each of the four oils is equivalent. On this basis for any candidate set of the four oils, the three DF estimates of test standard deviation for each of the four oils may be pooled to obtain a twelve DF estimate of test standard deviation. The within-laboratory precision of this appendix is based on such pooled values.

X2.3 The precision results are given in Table X2.1 for percent volume swell. This is the only property that was evaluated for precision, since it shows the highest sensitivity to variation.

TABLE X2.1 Precision Data for Within-Laboratory Variation

NOTE 1—*Sr* = repeatability standard deviation; *r* = repeatability = 2.83 × *Sr*; (*r*) = repeatability on relative basis (percent of percent); and *CV* = coefficient of variation, % = (*Sr*/Mean) × 100.

Rubber	Change in Volume, % (Pooled Values)				
	Mean	*Sr*	*r*	(*r*)	*CV*
ASTM Oil No. 2 Types:[A]					
ACM	1.3	0.57	1.60	119.0	42.2
AEM	27.8	0.82	2.33	8.4	3.0
CR	36.0	1.63	4.61	12.8	4.5
ECO	3.9	0.58	1.65	42.7	15.1
EPDM	120.2	2.17	6.14	5.1	1.8
EVM	34.1	0.60	1.68	4.9	1.8
FKM	0.7	0.20	0.57	76.5	27.0
FVMQ	0.4	0.31	0.87	198.0	70.0
HNBR	9.4	0.30	0.85	9.1	0.3
NBR	7.6	0.35	0.99	13.1	4.6
TPV	48.4	1.98	5.62	11.6	4.1
VMQ	10.4	1.36	3.84	37.0	13.1
ASTM Oil No. 3 Types:[B]					
ACM	8.3	1.31	3.71	44.6	15.8
AEM	50.3	0.71	2.01	4.0	1.4
CR	68.1	1.15	3.26	4.8	1.7
ECO	7.1	1.22	3.48	48.5	17.1
EPDM	161.5	5.19	14.70	9.1	3.2
EVM	58.7	1.04	2.94	5.0	1.8
FKM	1.6	0.34	0.95	59.1	20.9
FVMQ	2.0	0.43	1.21	59.5	21.0
HNBR	15.9	0.46	1.29	8.1	2.9
NBR	14.8	0.72	2.05	13.8	4.9
TPV	79.5	1.92	5.43	6.8	2.4
VMQ	43.4	2.30	6.52	15.0	5.3

[A] Pooled results for No. 2 oil plus three candidate replacements.

[B] Pooled results for No. 3 oil plus three candidate replacements.

X2.4 Table X2.1 lists the repeatability standard deviation, *Sr*; the repeatability, *r* , in units of % volume swell, the relative repeatability, (*r*), which is a percent of a percent for volume swell measurements, and for completeness, the coefficient of variation, CV, in %. The mean volume swell values range from near zero to about 160 %. The precision parameters also display a wide range. Regression and graphical analysis reveals that (*1*) there is a direct (positive slope) log-log relationship for *Sr* versus mean volume swell, and (*2*) an inverse (negative slope) relationship between the relative repeatability (*r*) and mean volume swell. These relationships apply to both sets of data and to the combined data sets.

X2.5 The results of Table X2.1 may be applied to within-laboratory data comparisons using either IRM 902 or IRM 903 for any commercial or proprietary compound by selecting the Table X2.1 rubber closest to the commercial compound and selecting the level of volume swell value in Table X2.1 closest to the measured volume swell of the commercial rubber.

X2.5.1 Two test results of the commercial rubber that differ by more than the tabulated *r* or (*r*) values as selected by the above process, must be considered to have come from different sample populations (that is, be significantly different).

X2.5.2 Alternatively normal testing operations should produce values for duplicate test results that are within the *r* and (*r*) intervals as selected by the above process.

X2.6 Bias reference values do not exist for this type of testing, since the value of the test property is defined exclusively by the test method. Bias, therefore, cannot be determined.

X3. REPLACEMENT OF ASTM OIL NO. 1 WITH IRM 901

X3.1 A precision statement has not been prepared due to the sudden unavailability of ASTM Oil No. 1. The Precision and Bias statement will be prepared in accordance with Practice D4483 at the conclusion of the forthcoming interlaboratory test program.

X3.2 Table X3.1 reflects the data that was acquired during a study at the time that ASTM No. 1 Oil was being replaced by IRM 901 and a supply of ASTM No. 1 remained in existence. It is provided for reference only. The data was obtained in 2005 in a study conducted by Smithers Scientific Services, Akron, Ohio.

TABLE X3.1 ASTM Oil No. 1 Replacement Program

Test	#1 CR	#2 EVM	#3 EPDM	#4 NBR	#5 HNBR	#6 ECO	#7 ACM	#9 AEM	#10 VMQ	#11 TPV
ORIGINAL PROPERTIES										
Hardness, Shore A	73	79	74	61	69	72	72	74	64	75
Tensile strength, psi	2877	2863	2153	3208	3818	1977	1990	2267	1295	745
Stress @ 300 % strain	...	...	...	1200	...	...	...	...	1110	...
Elongation at break, %	259 %	155 %	268 %	625 %	262 %	284 %	171 %	284 %	339 %	120 %
IMMERSION – ASTM #1										
Hardness, Shore A	66	72	47	59	67	71	68	72	59	63
Tensile strength, psi	2660	2741	1061	3504	4124	2007	2060	2310	1456	666
Stress @ 300 % strain	...	...	...	1580	4070	...	...	...	1190	...
Elongation at break, %	235 %	156 %	133 %	576 %	307 %	233 %	169 %	269 %	350 %	78 %
Change in hardness, points	−7	−7	−27	−2	−2	−1	−4	−2	−5	−12
Change in tensile strength, %	−7.54 %	−4.26 %	−50.72 %	9.23 %	8.01 %	1.52 %	3.52 %	1.90 %	12.43 %	10.60 %
Change in stress @ 300 %, %	...	...	...	...	...	...	...	...	...	...
Change in elongation @ break, %	−9.27 %	0.65 %	−50.37 %	−7.84 %	17.18 %	−17.96 %	−1.17 %	−5.28 %	3.24 %	−35.00 %
Change in weight, %	6.17 %	5.90 %	36.63 %	−1.01 %	−0.02 %	−0.31 %	−1.32 %	2.49 %	1.85 %	32.89 %
Change in volume, %	9.89 %	8.10 %	48.18 %	−0.64 %	0.29 %	0.26 %	−1.69 %	4.34 %	2.69 %	35.96 %
IMMERSION – IRM 901 (REPLACEMENT)										
Hardness, Shore A	67	73	47	60	68	71	70	72	58	60
Tensile strength, psi	2512	2608	1600	3390	4174	2107	2084	2355	1400	649
Stress @ 300 % strain	...	...	...	1550	4160	...	...	...	1250	...
Elongation at break, %	213 %	131 %	168 %	564 %	302 %	249 %	180 %	274 %	334 %	77 %
Change in hardness, points	−6	−6	−27	−1	−1	−1	−2	−2	−6	−15
Change in tensile strength, %	12.69 %	−8.91 %	−25.69 %	5.67 %	9.32 %	6.58 %	4.72 %	3.88 %	8.11 %	−12.89 %
Change in stress @ 300 %, %	...	...	...	...	...	...	...	...	...	...
Change in elongation @ break, %	17.76 %	−15.48 %	−37.31 %	−9.76 %	15.27 %	−12.32 %	5.26 %	−3.52 %	−1.47 %	−35.83 %
Change in weight, %	5.63 %	5.59 %	36.02 %	−1.20 %	−0.26 %	−0.46 %	−1.45 %	1.87 %	2.40 %	30.92 %
Change in volume, %	9.11 %	7.95 %	47.89 %	−0.82 %	0.40 %	0.01 %	−1.29 %	3.65 %	2.27 %	34.32 %
AGING CONDITIONS	TIME	TEMP								
#2-EVM	70 h	125°C								
#3-EPDM	70 h	125°C								
#4-NBR	70 h	100°C								
#5-HNBR	70 h	150°C								
#6-ECO	70 h	100°C								
#7-ACM	70 h	125°C								
#9-AEM	70 h	125°C								
#10-VQM	70 h	150°C								
#11-TPV	70 h	100°C								

X4. REPLACEMENT OF ASTM OIL NO. 5 WITH IRM 905

X4.1 A Precision and Bias statement will be prepared in accordance with Practice D4483 at the conclusion of the forthcoming interlaboratory test program.

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