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Standard Test Method for Rubber Property—Effect of Liquids¹

This standard is issued under the fixed designation D 471; 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 test method has been approved for use by agencies of the Department of Defense to replace Methods 6001, 6111, 6121, 6211, 6231, and 6251 of Federal Test Method Standard No. 601 and for listing in the DoD Index of Specifications and Standards.

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 D 3182), (2) specimens cut from fabric coated with vulcanized rubber (see Test Method D 751), or (3) finished articles of commerce (see Practice D 3183). This test method is not applicable to the testing of cellular rubbers, porous compositions, and compressed sheet packing, except as described in 11.2.2.

1.2 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 1 for details).

1.3 This test method includes the following:

Change in Mass (after immersion)	Section 10
Change in Volume (after immersion)	Section 11
Dimensional-Change Method for Water-Insoluble Liquids and Mixed Liquids	Section 12
Change in Mass with Liquid on One Surface Only	Section 13
Determining Mass of Soluble Matter Extracted by the Liquid	Section 14
Change in Tensile Strength, Elongation and Hardness (after immersion)	Section 15
Change in Breaking Resistance, Burst Strength, Tear Strength and Adhesion for Coated Fabrics	Section 16
Calculation (of test results)	Section 17

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

1.5 *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:

- D 92 Test Method for Flash and Fire Points by Cleveland Open Cup²
- D 97 Test Method for Pour Point of Petroleum Products²
- D 287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)²

¹ This test method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.15 on Degradation Tests.

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² Annual Book of ASTM Standards, Vol 05.01.

D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers—Tension³

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)²

D 611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents²

D 751 Test Methods for Coated Fabrics⁴

D 975 Specification for Diesel Fuel Oils²

D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer²

D 1415 Test Method for Rubber Property—International Hardness³

D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)²

D 1747 Test Method for Refractive Index of Viscous Materials²

D 2008 Test Method for Ultraviolet Absorbance and Absorptivity of Petroleum Products²

D 2140 Test Method for Carbon-Type Composition of Insulating Oils of Petroleum Origin⁵

D 2240 Test Method for Rubber Property—Durometer Hardness³

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets³

D 3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products³

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries³

Motor Fuels, Section 1; Annex 2.7, Reference Materials and Blending Accessories⁶

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 Annual Book of ASTM Standards, Vol 09.01.

⁴ Annual Book of ASTM Standards, Vol 09.02.

⁵ Annual Book of ASTM Standards, Vol 10.03.

⁶ Annual Book of ASTM Standards, Vol 05.04.



3.2 The precision statement in Section 19 is based on an interlaboratory test program run in 1981, using six different rubbers with ASTM Reference Fuels B, C, D⁷ and ASTM Oils No. 1 and No. 3.

3.3 The precision statement in Appendix 2 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.

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 1, depending upon the anticipated service conditions:

5.1.1 When the temperature of the testing room is other than the standard $23 \pm 2^\circ\text{C}$ ($73 \pm 4^\circ\text{F}$) the temperature of test shall be reported.

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

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 usually 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

TABLE 1 Test Temperatures and Immersion Periods

Temperature in $^\circ\text{C}$ ($^\circ\text{F}$)		Immersion Period, h
-75 ± 2 (-103 ± 4)	85 ± 2 (185 ± 4)	22
-55 ± 2 (-67 ± 4)	100 ± 2 (212 ± 4)	46
-40 ± 2 (-40 ± 4)	125 ± 2 (257 ± 4)	70
-25 ± 2 (-13 ± 4)	150 ± 2 (302 ± 4)	166
-10 ± 2 (14 ± 4)	175 ± 2 (347 ± 4)	670
0 ± 2 (32 ± 4)	200 ± 2 (392 ± 4)	1006
23 ± 2 (73 ± 4)	225 ± 2 (437 ± 4)	2998
50 ± 2 (122 ± 4)	250 ± 2 (482 ± 4)	4990
70 ± 2 (158 ± 4)		

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 *ASTM and IRM Oils*—The test shall be conducted in one of the petroleum-base ASTM or IRM oils (Note 1) specified in Table 2 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 2 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 3, except as indicated in 6.1.3.

NOTE 2—The ASTM reference fuels in Table 3 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.

6.1.3 *Service Liquids*—Some commercial oils, fuels and other service liquids (see Table 4) 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 Tables 2 and 3. 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 are to be conducted in distilled or deionized water.

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 D 3182 and D 3183.

⁷ Reference Fuels A, B, C, and D are available from Phillips Petroleum Co., Customer Service Center, P.O. Box 968, Borger, TX 79007.

TABLE 2 Specifications and Typical Properties of ASTM and IRM Reference Oils

Property	ASTM Oil No. 1	ASTM Oil No. 5	IRM 902	IRM 903	ASTM Method
Specified Properties:					
Aniline Point, °C (°F)	124 ± 1 (255 ± 2)	115 ± 1 (239 ± 2)	93 ± 3 (199 ± 5)	70 ± 1 (158 ± 2)	D 611
Kinematic Viscosity (mm ² /s [cSt])					
38°C (100°F)	...	...	...	31.9–34.1	D 445
99°C (210°F)	18.7–21.0	10.8–11.9	19.2–21.5	...	D 445
Gravity, API, 16°C (60°F)	...	...	19.0–21.0	21.0–23.0	D 287
Viscosity-Gravity Constant	...	...	0.860–0.870	0.875–0.885	D 2140
Flash Point COC, °C (°F)	243 (469) min	243 (469) min	240 (464) min	163 (325) min	D 92
Naphthenics, C _N (%)	...	...	35 min	40 min	D 2140
Paraffinics, C _P (%)	...	...	50 max	45 max	D 2140
Typical Properties:					
Pour Point, °C (°F)	...	–15 (5)	–12 (10)	–31 (–24)	D 97
ASTM Color	...	L 1.0	L 2.5	L 0.5	D 1500
Refractive Index	...	1.4808	1.5105	1.5026	D 1747
UV Absorbance, 260 nm	...	...	4.0	2.2	D 2008
Aromatics, C _A (%)	...	4	12	14	D 2140

TABLE 3 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 ASTM Motor Fuels, Section 1, Annex 2.7, Table 32 (Isooctane), and Table 33 (Toluene).

^B Specification D 975.

^C Anhydrous ethanol denatured with unleaded gasoline (CDA formula No. 20), available from Archer-Daniels-Midland, P.O. Box 1470, Decatur, IL 62525.

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.⁸ Glass beads shall be used in the liquid as a bumper and to separate the specimens.

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^{9,10} and No. 5¹⁰ and for IRM 902^{9,10} and IRM 903^{9,10} at test temperatures of 125°C (257°F) or more. This condenser shall be a glass tube, approximately 500 mm (20 in.) 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

⁸ Test tubes available under Part No. LGB24 and LGB24C (case of 30) from Edwin H. Benz Co. Inc., 73T Maplehurst Ave., Providence, RI 02908 or under Part No. LG226 from Precision Scientific, 2777 Washington Blvd., Bellwood, IL 60104, have been found suitable for this purpose.

⁹ Information about ASTM Oil No. 1 is included in Research Report RR: D11-1004 and information about IRM 902 and IRM 903 in Research Report RR: D11-1069. Both reports are available from ASTM Headquarters.

¹⁰ ASTM Oil No. 1, IRM 902 and IRM 903 may be ordered from Penreco, 4426 East Washington Blvd., Los Angeles, CA 90023-4476. ASTM Oils No. 1, No. 5, IRM 902, and IRM 903 are distributed by R. E. Carroll, Inc., P.O. Box 5806, Trenton, NJ 08638-0806.

TABLE 4 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	ASTM Oil No. 1, 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- <i>n</i> -butyl phosphate, 100 mass %
Service Liquid 104 ^E	ethylene glycol ^F , 50 volume %; distilled water, 50 volume %

^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 is available as Angiamol 99 from Lubrizol Corp., 29400 Lakeland Blvd., Wickliffe, OH 44092. Properties are as follows: Kinematic viscosity (mm²/s [cSt]) at 99°C (210°F) – 9.70 ± 0.52 (Test Method D 445); Flash point COC °C (°F) – 45 (113) min (Test Method D 92); Density at 16°C (61°F) – 1.065 ± 0.015 (Test Method D 1217).

^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.

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, or aging blocks¹¹ may be used.

8.3.1 The preferred method for above room temperatures is the use of aging blocks, since they have the widest temperature capability.

8.3.2 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.

9. Test Specimens—Change in Mass or Volume

9.1 The standard specimen shall be rectangular, having

¹¹ Aging blocks, available under Model No. LGB9 (9 cavities) and LGB28 (28 cavities) from Edwin H. Benz Co., Inc., 73T Maplehurst Ave., Providence, RI 02908, have been found suitable for this purpose.

dimensions of 25 by 50 by 2.0 ± 0.1 mm (1 by 2 by 0.08 ± 0.004 in.). Specimens from commercial articles shall be the thickness of the material as received when they are less than 2 mm (0.08 in.); otherwise they should be reduced to a thickness of 2.0 ± 0.1 mm (0.08 ± 0.004 in.). Data obtained on test specimens having different original thicknesses are not comparable.

10. Procedure for Change in Mass

10.1 Test three specimens of a single composition. Calculate the test results of the three specimens (see 17.1, 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 cm³ of the test liquid (see 10.1.1). 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 beads¹² as shown in Fig. 1. The materials used to suspend and separate the specimens shall not affect the test liquid or the rubber.

10.1.1 Test liquids shall not be reused.

10.1.1.1 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.

10.1.1.2 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.

10.2 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 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 11.2.1) 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.

10.3 Relatively viscous oils may be difficult to remove from the specimens with a quick acetone 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



FIG. 1 Method of Separation

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 dip and blotting as described in 10.2. Report when this alternate method of specimen cooling is used.

11. Procedure for Change in Volume

11.1 *Water Displacement Method for Water-Insoluble Liquids and Mixed Liquids*—Test three specimens, calculate the test results for each specimen (see 17.2, Eq 2), and average the results. Obtain the mass M_1 of each specimen (see 11.2.2) 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) to remove water (see 11.2.3), 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 cm³ of liquid (see 10.1.1) to the test tube and complete the immersion test as described in 10.1.1.1 or 10.1.1.2. At the end of the required immersion period, remove each specimen from the 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 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 11.2.2 and Note 3). When data on progressive changes with increasing time of immersion are desired, dip each specimen after weighing in alcohol (methanol or ethanol) to remove water (see 11.2.3), blot dry with filter paper free of lint and foreign material, and

¹² Hollow perforated glass beads, available under Catalog No. 11-311C from Fisher Scientific Co., 711T Forbes Ave., Pittsburgh, PA 15219, have been found suitable for this purpose.