



Designation: D471 – 15a

Standard Test Method for Rubber Property—Effect of Liquids¹

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.*

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
<i>Specified Properties:</i>					
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
<i>Typical Properties:</i>					
Pour Point, °C (°F)	–12 (10)	–12 (10)	–42.8 (–45)	–15 (5)	D97
ASTM Color	L 3.5	L 2.5	L 0.5	L 1.0	D1500
Refractive Index	1.4848	1.5105	1.5004	1.4808	D1747
UV Absorbance, 260 nm	0.8	4.0	1.43	...	D2008
Aromatics, C _A (%)	3	12	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, C _N %	D2140	—	24	26 26	26 26	27 27	28 29	27
Paraffinics, C _P %	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

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, D⁴ and ASTM Oils No. 1 and No. 3.

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.

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 $23 \pm 2^\circ\text{C}$ ($73 \pm 4^\circ\text{F}$) 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)	



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

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**.

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 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- <i>n</i> -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 (mm²/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.

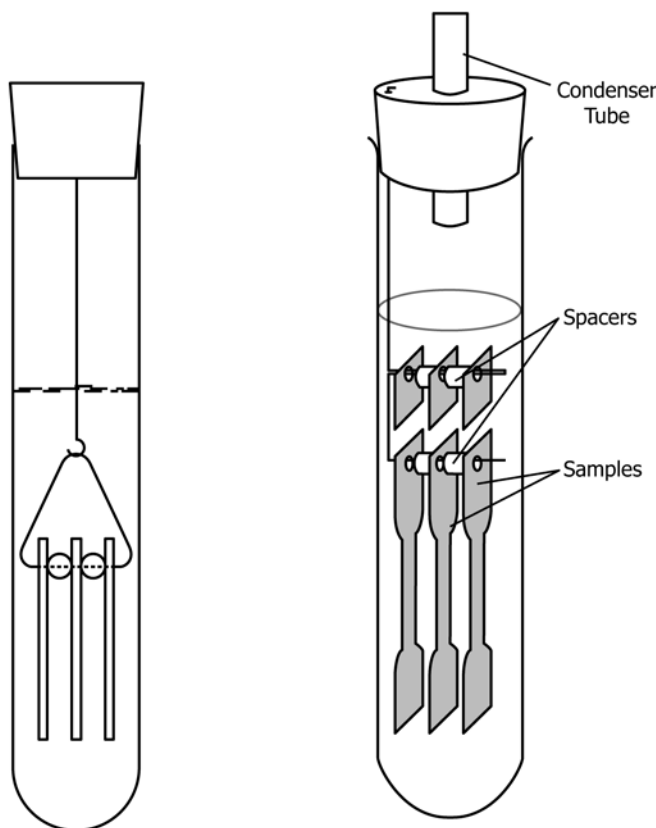


FIG. 1 Methods of Separation Showing Single Level Specimen Hanger (Left) and Dual Level Specimen Hanger (Right)

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,

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.

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.