



# Standard Test Methods for Rubber Deterioration—Cracking in an Ozone Controlled Environment<sup>1</sup>

This standard is issued under the fixed designation D1149; 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 ( $\epsilon$ ) 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 These test methods are used to estimate the effect of exposure, under surface tensile strain conditions, either dynamic or static, in an atmosphere containing specified levels of ozone concentration, expressed as partial pressure (refer to [Note 1](#)), on vulcanized rubber, rubber compounds, molded or extruded soft rubber, and other specified materials, or as may be determined empirically. The effect of naturally occurring sunlight or light from artificial sources is excluded.

1.2 Previously published ASTM documents Test Method [D518](#) and Test Methods [D3395](#) have been included in these test methods, D1149, in 2007. Please refer to [Note 2](#).

1.2.1 Test Method [D518](#) and Test Methods [D3395](#) have henceforth been withdrawn and superseded by Test Methods D1149. When Test Methods D1149 is cited, or otherwise referenced, a notation shall be included to this effect. Please refer to section [3.2](#) for the appropriate references.

1.3 The specified conditions of exposure to ozone in the controlled environments are accelerated in comparison to outdoor exposure. These accelerated ozone test methods may not give results which correlate with outdoor exposure tests or service performance.

1.4 All materials, instruments, or equipment used for the determination of mass, force, dimension, ozone concentration, partial pressure, temperature, velocity, and gas exchange rate shall have direct traceability to the National Institute for Standards and Technology, or other internationally recognized organization parallel in nature.

1.5 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only. Many of the stated SI units are direct conversions from the U.S. Customary System to accommodate the

instrumentation, practices, and procedures that existed prior to the Metric Conversion Act of 1975.

1.6 *This standard involves hazardous materials, specifically ozone. It may also involve hazardous operations and equipment. 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.*

NOTE 1—A discussion and explanation regarding the measurement of ozone concentrations based upon parts of ozone per unit of air versus partial pressure is provided in Test Methods [D4575](#), specifically Appendices X1 and X2. Test Methods [D4575](#) is also recommended as a source of background information regarding standards involving materials exposed to ozone.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

[D518 Test Method for Rubber Deterioration—Surface Cracking \(Withdrawn 2007\)](#)<sup>3</sup>

[D1171 Test Method for Rubber Deterioration—Surface Ozone Cracking Outdoors \(Triangular Specimens\)](#)

[D1349 Practice for Rubber—Standard Conditions for Testing](#)

[D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets](#)

[D3395 Test Methods for Rubber Deterioration—Dynamic Ozone Cracking in a Chamber \(Withdrawn 2007\)](#)<sup>3</sup>

[D4482 Test Method for Rubber Property—Extension Cycling Fatigue](#)

[D4575 Test Methods for Rubber Deterioration—Reference and Alternative Method\(s\) for Determining Ozone Level in Laboratory Test Chambers](#)

NOTE 2—Test Method [D518](#) and Test Methods [D3395](#) have been

<sup>1</sup> These test methods are 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

incorporated into these test methods. They remain technically unchanged and will be superseded by Test Methods D 1149. Please refer to Test Method **D1171** for tests which are to be performed outdoors.

### 3. Summary of Test Methods

3.1 There are two methods described:

3.1.1 *Method A—Dynamic Strain (formerly Test Methods D3395):*

3.1.1.1 *Method A, Procedure A1 Dynamic Tensile Elongation (formerly Test Methods D3395 Method A)*—rectangular test specimens are subjected to dynamic maximum amplitude tensile strain of  $25 \pm 3\%$  at a fixed frequency of 0.5 Hz (30 cpm).

3.1.1.2 *Method A, Procedure A2 Dynamic Belt Flex Test (formerly Test Methods D3395 Method B)*—rectangular test specimens are affixed to a fabric belt which is continuously rotated over two vertically opposed pulleys that induce a cyclical surface strain (due to flexing) during the moments of passage over the pulleys.

3.1.2 *Method B—Static Strain (formerly Test Method D518):*

3.1.2.1 *Method B, Procedure B1 Static Strain (formerly Test Method D518, Method A)*—rectangular specimens are exposed to a continuous elongation of 20 %.

3.1.2.2 *Method B, Procedure B2 Static Strain Looped Specimen Test (formerly Test Method D518, Method B)*—rectangular specimens are mounted, in a looped fashion, causing a continuous strain to be applied on the looped portion.

3.1.2.3 *Method B, Procedure B3 Static Percent Elongation Test (formerly Test Method D518, Method C)*—tapered specimens are exposed to specified percentages of elongation (10, 15, and 20 %) during the course of exposure.

3.1.2.4 *Method B, Procedure B4 Static Strain Triangular Specimen (formerly Test Method D1171)*—triangular specimens are mounted around a wooden mandrel causing a continuous strain to be applied to the looped portion.

(1) *Method B, Procedure B4-A Exposure Rating (formerly Test Method D1171 Method A).*

(2) *Method B, Procedure B4-B Quality Retention Rating (formerly Test Method D1171 Method B).*

3.2 The Procedures appear as follows:

Procedure—Description	Sections
A1—Dynamic Tensile Elongation	7 – 10 (formerly D3395 Method A)
A2—Dynamic Belt Flex	11 – 14 (formerly D3395 Method B)
B1—Straight Specimens (Static Elongation)	15 – 17/24 (formerly D518/D 1149 Method A)
B2—Bent Loop Specimen	18 – 20/24 (formerly D518/D 1149 Method B)
B3—Tapered Specimens (Static Elongation)	21 – 23/24 (formerly D518/D 1149 Method C)
B4-A—Triangular Specimens	24 (formerly D1171 Method A Exposure Rating)
B4-B—Triangular Specimens	24 (formerly D1171 Method B Quality Retention Rating)

### 4. Significance and Use

4.1 The significance of these test methods lies in the ability to differentiate between the degrees of ozone resistance under the limited and specified conditions of the accelerated tests.

The degree of resistance being judged by the appearance and magnitude of the formation of cracks in the surface of the subject material.

4.2 In service, rubber materials deteriorate when exposed to ozone. It is imperative to have test methods in which simple, accelerated time/exposure, comparisons of the material's ability to resist cracking caused by ozone exposure can be empirically evaluated. Such tests can be used for producer/consumer acceptance, referee purposes, research, and development.

4.3 These methods are not necessarily suited for use in purchase specifications as there may be no correlation with service performance as actual service conditions (outdoor exposure) vary widely due to geographic location and, therefore, may not yield repeatable or reproducible results.

### 5. Apparatus

5.1 The apparatus employed to provide a controlled environment containing specified concentrations of ozone and partial pressures, at specified temperatures, is described in **5.2**, being common to all test methods enumerated herein. The ancillary equipment required to perform individual procedures is described in their respective sections.

#### 5.2 Ozone Test Apparatus:

5.2.1 *Test Chamber*—General requirements for an acceptable ozone test chamber are adequate control and measurement of ozone concentration levels based upon partial pressure (refer to **Note 1**); volume exchange rate; temperature; internal chamber circulation of the gas (air–ozone mixture, refer to **Note 3**); and internal chamber volume. An acceptable ozone test chamber may be individually manufactured for a particular application, or a commercially manufactured chamber. The ozone test chambers shall conform to the following requirements:

**NOTE 3**—Dalton's Law and the gas equation is used to express ozone content as partial pressure. The partial pressure of ozone in a mixture with air,  $P(O_3)$ , is given in millipascals (mPa).

5.2.1.1 The internal chamber shall be constructed of a material with minimal reaction to ozone, preferably stainless steel, contained within an integral external structure which provide unencumbered access to the mechanical, electrical, display, and control devices.

(1) The internal chamber shall be accessible through a door having an adequate closure mechanism and seal to prevent loss of partial pressure or adversely affect the ozone concentration levels throughout the duration of a test. It is recommended that the door have a means by which to prevent inadvertent opening during the intended duration of a test.

(2) The access door may be equipped with an observation window. This shall be of tempered glass and shall be sealed to prevent loss of partial pressure or adversely affect the ozone concentration level throughout the duration of a test.

(3) The internal chamber may be equipped with a source of illumination. It shall be situated in a manner so that neither the temperature of the internal chamber, the ozone concentration level, or partial pressure is adversely affected. Illumination is to be used in a temporary fashion, intended for intermediate

viewing of the specimens. Long term use, in excess of a consecutive 5-min period, or more than a total of 15 min, during any given 24-h period, invalidates determinations and therefore requires monitoring and reporting.

(4) The internal chamber may be equipped with shelves, or racks, on which to place specimens. They shall be constructed of a material with minimal reaction to ozone, preferably stainless steel. They shall be of a design that minimizes the effect on the introduction, circulation, exchange, or exhaust of the gas (air–ozone mixture).

5.2.1.2 The internal volume of the chamber shall be no less than 0.11 m (4 ft<sup>3</sup>). The internal chamber volume may be greater, provided the prescribed levels of ozone concentration and partial pressure are maintained (refer to 5.2.1.3(1) and Note 1).

5.2.1.3 A means for generating, measuring, and controlling ozone concentrations levels and partial pressure shall be provided. The ozone generating source shall be isolated from the internal chamber and within the integral external structure.

(1) The generation and introduction of ozone shall be by a means whereby ozone concentration levels in the internal test chamber of between 25 and 200 pphm (parts per hundred million)  $\pm 10\%$  at 100 kPa (atmospheric pressure at mean sea level, MSL) and the equivalent pressures of between 25 and 200 mPa  $\pm 10\%$  are maintained throughout the duration of the test (refer to Notes 1 and 2). The preferred method of ozone generation being either quartz UV, corona discharge (dielectric), or a combination of both.

(2) The test chamber ozone concentration levels and pressures shall be either infinitely variable between those prescribed or, at minimum, selectable at 25, 50, 100, 200 pphm and mPa, respectively. Broader ranges of ozone concentration and partial pressure are acceptable, provided the specified concentrations of ozone and partial pressure are maintained as specified.

(3) The means of measurement of the ozone concentration shall be by any of the methods described in Test Methods D4575, with ultraviolet (UV) absorption measurement being preferred as it has been shown to be more accurate and precise.

5.2.1.4 The source of air may be ambient, or from a compressed air supply. In either case, adequate filtration of particulate matter from the source shall be provided. The moisture content of the air source shall be minimized, either through filtration, a desiccant, or by other means.

5.2.1.5 The flow of the gases (air and ozone) shall be introduced into the chamber in a manner that prevents stratification or stagnation.

5.2.1.6 The gas (air–ozone mixture) exchange rate shall be of a magnitude such that no appreciable reduction in ozone concentration or partial pressure results from the introduction of test specimens.

(1) The exchange rate will vary with the gas (air–ozone mixture) level, temperature, number of test specimens introduced, and their reaction with ozone.

(2) A gas (air–ozone mixture) exchange rate of 75 % of the total volume of the internal chamber per minute has been found to be an acceptable and adequate rate, and shall be considered to be the minimum acceptable rate of gas exchange.

(3) As indicated, this exchange rate will vary, dependent upon the enumerated variables, and the minimum exchange rate shall be established locally, as agreed upon between customer and supplier, or between laboratories, but shall not be less than that described in 5.2.1.6(2).

5.2.1.7 A means of providing internal chamber circulation of the gas (air–ozone mixture) shall be provided by an electric fan capable of maintaining a constant velocity throughout the duration of a test. The velocity shall be no less than 0.6 m/s (2 ft/s) as measured at  $50 \pm 1$  mm ( $1.97 \pm 0.04$  in.) from the forward edge of the fan blades on the internal side of the chamber.

(1) The fan motor shall not be located within the internal chamber. The fan motor shall employ an extension shaft, or drive mechanism, that isolates the motor from the internal chamber with an ozone resistant seal that assures the integrity of separation between the motor and the internal chamber.

(2) The fan blades shall be recessed from the chamber and separated, or enclosed, by a protective mesh or grid-like cage.

5.2.1.8 A means of controlling the internal chamber temperature to within  $\pm 1^\circ\text{C}$  ( $\pm 1.8^\circ\text{F}$ ) of that established, or desired, throughout the duration of a test, shall be provided.

(1) The standard test temperature shall be  $40 \pm 1^\circ\text{C}$  ( $104 \pm 1.8^\circ\text{F}$ ). Other temperatures may be established locally, agreed upon between customer and supplier, or between laboratories. It is recommended that the standard temperatures given in Practice D1349 be followed.

(2) Testing at subnormal or elevated temperatures requires the use of refrigerated chambers or chambers having additional heating capability. These shall meet all other requirements as described heretofore.

5.2.1.9 Chambers shall be equipped so that concentrations of ozone are safely exhausted from the internal test chamber so as not to introduce them to the proximal ambient atmosphere (refer to 1.4). This shall be accomplished by properly exhausting the gas (air–ozone mixture) from the internal test chamber before the opening of the access door. It is recommended that the gas be exposed to a suitable ozone destruction device, such as a catalytic unit, before it is vented to the atmosphere (refer to 1.4).

## 6. Hazards

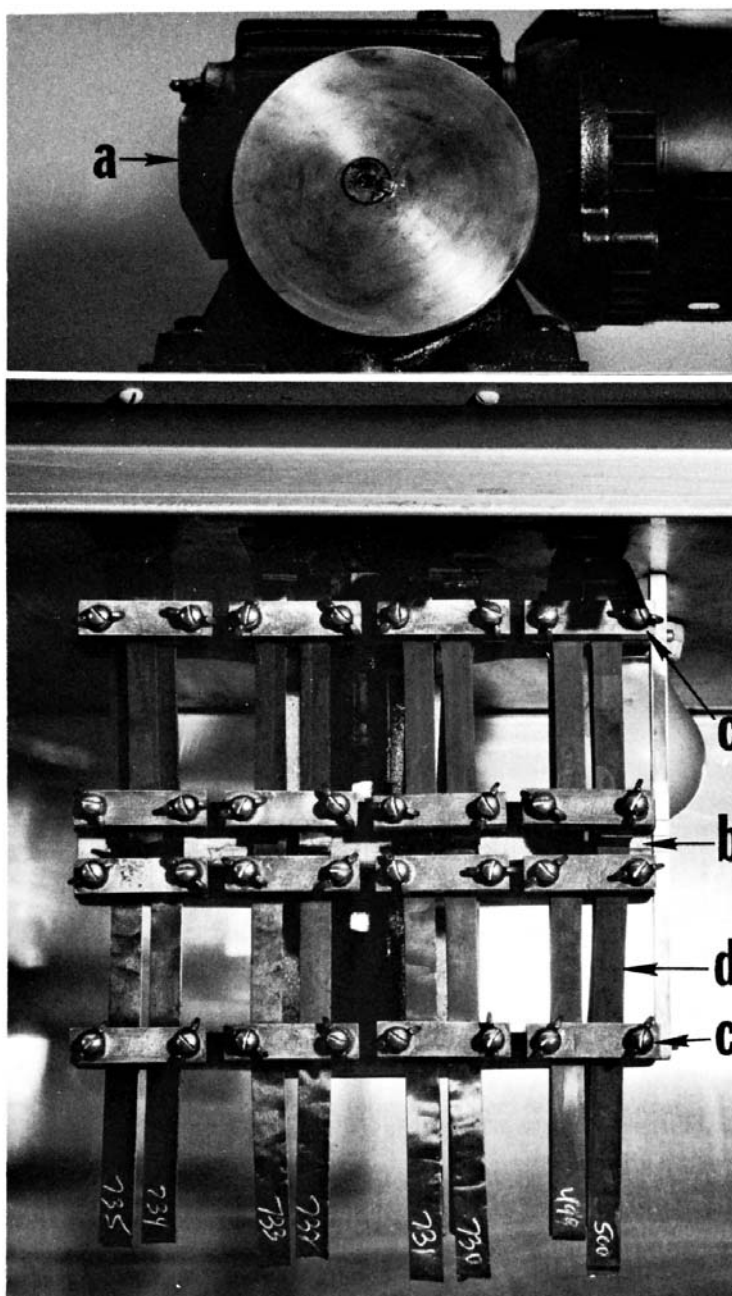
6.1 **Warning**—Ozone is a hazardous substance. Consult and follow all applicable laws, rules, and regulations regarding exposure to ozone.

## METHOD A, PROCEDURE A1—TENSILE ELONGATION TEST

### 7. Apparatus

7.1 *Flexing Device:*





a—gear head and eccentric drive  
 b—traveling bar with specimen clamps, driven through roof of ozone chamber  
 c—stationary bars with specimen clamps  
 d—specimens

**FIG. 1 Method A, Procedure A1, Dynamic Strain Ozone Tester**

7.1.1 The device<sup>4</sup> used for this test consists of a flexing framework, a motor with gear head reducer, and an eccentrically driven vertical shaft with a reciprocating stroke of 25 mm (1 in.). To the end of this shaft, a parallel bar is attached and to

<sup>4</sup> The sole source of supply of the apparatus (Model DynaStretch™) known to the committee at this time is Ozone Research Equipment Co., a Division of CCSi, Inc., 1145 Highbrook Avenue, #500, Akron, OH 44301. 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,<sup>1</sup> which you may attend.

this a series of four dual clamps. Above and below the traveling bar are two stationary bars that contain four clamps each. These constitute the flexing framework

7.1.2 The action of the flexing apparatus is to impart a straight-line motion to the clamps holding the test specimens; four specimens on the top bar, four on the bottom bar. This motion is in the plane of the common centerline of each set of opposing grips (see Fig. 1).

7.2 *Ozone Chamber*—The ozone chamber shall conform to the requirements specified in Section 5. It shall be equipped to

permit flexing of the specimens from 0 to  $25 \pm 3$  % extensions at a rate of 0.5 Hz (30 cpm), refer to [Fig. 1](#).

## 8. Test Specimens

8.1 The test specimens shall be rectangular strips  $10.00 \pm 0.03$  mm ( $0.40 \pm 0.01$  in.) wide by  $100 \pm 25$  mm ( $4 \pm 1$  in.) in length, cut from test sheets that have been prepared in accordance with Practice [D3182](#). The grain or direction of milling of uncured stock shall be in the lengthwise direction of the specimen. Duplicate test specimens shall be tested whenever possible.

NOTE 4—It is strongly suggested that all rubber sheets for ozone testing be cured between aluminum foil or polyester film<sup>5</sup> 0.1 mm in thickness. The foil will adhere mildly to most commercial rubbers. At the time specimens are cut for ozone testing the foil can be removed easily. This furnishes a “fresh” surface and the 24 h or other pre-test conditioning period, can be reckoned from the formation of this fresh “bloom-free” surface.

## 9. Procedure

### 9.1 Pre-Test Conditioning—Nominal Period:

9.1.1 Prior to testing, condition all free-surface (that is, no foil specimens) for  $24.0 \pm 0.5$  h at  $23 \pm 2^\circ\text{C}$  in a sealed opaque container that has an ozone-free atmosphere. Different compounds are to be contained separately in the sealed opaque container.

9.1.2 Select other pre-test conditioning periods consistent with certain technical requirements or purchaser and seller agreements.

### 9.2 Operation of Test Apparatus:

9.2.1 By use of the slow-speed switch, accurately position the reciprocating shaft at the mid-point of its stroke. Mount the test specimens and clamp them in place so that no discernible bend or bow is evident. Close the door to the ozone chamber and allow a short period (5 min) for the ozone partial pressure to stabilize. Start the test, noting the time and date of starting ( $\pm 1$  min).

9.2.1.1 There exists a range of fixturing devices to accomplish the dynamic strain of 0 to 25 % elongations at 30 cpm (refer to [7.2](#)). The objective is to mount the specimens in the fixture at the point of 0 % elongation (zero strain). This may require that one set be mounted at a time, for example, the uppermost specimens are mounted at 0 % elongation, while the lowermost specimens are at 25 % elongation and vice versa. This may be accomplished by jogging the fixture’s reciprocating shaft into a 0 % elongation position for each of the fixture’s specimen holders.

9.2.1.2 If permanent set % measurement is a consideration in the dynamic strain test, the procedure outlined in Test Method [D4482](#), 10.1.6 through 10.1.10 may be adapted for this purpose. If the specimens are adjusted, it shall be noted in the report (10.1.7) following the reporting procedure in Test Method [D4482](#).

9.2.2 The standard ozone partial pressure shall be  $50 \pm 5$  mPa (equivalent to 50 pphm at 100 kPa atmospheric pressure). Other partial pressures may be selected according to the

particular objectives of any testing program. Refer to these as optional partial pressures. Measure the ozone partial pressure once a day for routine work and more often for special test conditions. Conduct this analysis with the chamber containing test specimens. If the number of specimens is kept at a minimum, “empty” and “loaded” ozone partial pressures will closely agree. Determine the magnitude of the reduction of ozone partial pressure for “empty” versus “loaded” for each set of operating variables employed. This will vary for each laboratory. Refer to Test Methods [D4575](#) for ozone analysis procedures.

9.2.3 The standard temperature shall be  $40 \pm 1^\circ\text{C}$  ( $104 \pm 2^\circ\text{F}$ ). Higher temperatures cause acceleration in the rate of ozone attack. These higher temperatures shall be optional.

9.2.4 Make observations for detecting the appearance of cracking at maximum extension and with sufficient frequency to be able to detect the first appearance of ozone cracking. This frequency will depend on the resistance to ozone attack of the rubbers being tested. Recommended observation magnification is 7 $\times$ . When comparisons are being made with a standard reference material, exposures may be made for a fixed time and the degree of cracking evaluated. Make observations in such a way as to minimize ozone partial pressure changes (that is, remove specimens quickly and keep the door closed as much as possible).

## 10. Report

10.1 The report shall include the following:

10.1.1 Description of the specimens, identification of the rubber compounds, time and temperature of vulcanization.

10.1.2 Conditioning time and temperature.

10.1.3 Date and time of the start of exposure.

10.1.4 Test chamber temperature, both nominal and that measured on a daily basis.

10.1.5 Test chamber ozone partial pressure, in mPa, both nominal and that measured on a daily basis.

10.1.6 Type of specimen and elongation or mounting type used during the test.

10.1.7 Elapsed time or number of test cycles to the first observed cracking and/or a description of the character of the ozone cracking at the various observation. If testing to pass/fail, report the total exposure time.

10.1.8 If desired, a photograph may be taken to provide a permanent record for evaluation.

## METHOD A, PROCEDURE A2—BELT FLEX TEST

### 11. Apparatus

#### 11.1 Belt Flex Device:

11.1.1 The test apparatus shall consist of a suitable metal framework to accommodate the belt and pulleys as specified below. The framework shall be equipped to accept either of two sets of pulleys; one set, 63.5 mm (2½ in.) in diameter and one set, 102 mm (4 in.) in diameter. The upper pulley of each set of two shall be powered to rotate the test belt at a cyclic frequency of 0.67 Hz (40 r/min) with the 63.5-mm diameter pulleys, and at 1.04 Hz (62.5 r/min) when using the 102-mm diameter

<sup>5</sup> An uncoated, biaxially oriented polyethylene terephthalate film (boPET) has been found satisfactory for this purpose.

pulleys. Alternatively, the drive shaft may be rotated at 3.75 Hz (225 r/min) to obtain the specified belt rotation rate. The lower pulley shaft shall be allowed to move up and down, having an 18-kg (40-lb) mass suspended from it to maintain sufficient tension on the specimen belt to achieve maximum conformation of the test specimens and belt around each pulley.

11.1.2 The size of this test apparatus requires that a large rectangular box be used to house the device. Fig. 2 shows a typical ozone chamber used for this test device. The dimensions of this box are 1370 by 310 by 380 mm (54 by 12¼ by 15 in.) with a volume of 0.16 m<sup>3</sup> (104 in.<sup>3</sup>). Fig. 2 is a schematic diagram of the test apparatus.

11.2 *Ozone Chamber*—The ozone chamber shall conform to the requirements specified in Section 5.

11.3 The standard ozone partial pressure shall be 50 ± 5 mPa. Other partial pressures may be selected for special research test programs.

## 12. Test Specimens

12.1 The specimen belt, to which the samples are either cured or cemented, shall be a one-ply belt of 1.1-kg/m<sup>2</sup> (32-oz/yd<sup>2</sup>) square woven cotton duck, 100 mm (4 in.) wide and 2300 ± 25 mm (90 ± 1 in.) in circumference. The belt should be coated with a typical rubber friction or coat compound, 0.2 mm (0.08 in.) thick. If the belt contains cut edges, these should be coated with a flexible curing type cement or other material inert to ozone. This will reduce ozone decomposition by contact with these surfaces. The rubber coat so applied should be cured. The test specimens shall be prepared by either cutting a specimen 100 by 25 mm (4 by 1 in.) from a standard tension sheet and cementing to the belt with the milled grain being parallel to the 100-mm (4-in.) specimen length; or a specimen shall be cured on the belt using a special mold. The latter is the preferred method, since there is less difficulty in preventing

separation of the test specimen from the belt during flexing. The test is carried out with duplicate specimens.

12.2 Fig. 3 shows a drawing of a multicavity mold used to prepare molded specimens.

12.3 The cured specimen shall be 100 by 50 by 3 ± 0.20 mm (4 by 2 by 0.13 in.), protected during cure by the use of aluminum foil or polyester film on the surface to be exposed, refer to Note 4.

## 13. Procedure

13.1 Prior to test, condition all free-surface test specimens separately for 24.0 ± 0.5 h at 23 ± 2°C in a sealed opaque container that has an ozone-free atmosphere.

13.2 Install the belt, with the adhered specimens, on the pulleys in the test chamber. Start the belt when the door is closed. For a chamber of the internal volume as mentioned in 11.1.2, a period of 30 min is required for the ozone partial pressure to stabilize. Also for a chamber of this size the “empty” (no belt or test specimens) ozone partial pressure should be set approximately 20 % higher than the intended “test” ozone level (that is, set chamber at 60 mPa for a test pressure of 50 mPa). Make minor adjustments of pressure as the test proceeds. Additionally, a more stable ozone level will be obtained if voltage regulation is maintained on the ozone generation equipment. Expose the specimens for a period of 48 h as described in Test Methods D1149 at 40 ± 1°C (104 ± 2°F).

13.3 During the 48-h period, observe the exposed specimens at maximum test strain at intervals of 1 h for the first 6 h, and thereafter at 24, 30, and 48-h periods. Examine with a 7× magnifier and record the time of initial cracking. During this examination take precautions to minimize ozone partial pressure changes (that is, quick observation period, with the door open for a minimum time).

13.4 If desired, the exposure may be continued for the purpose of observing the rate of growth of the cracks or the development of any characteristics or unusual surface effects.

## 14. Report

14.1 The report shall include the following:

14.1.1 Description of the specimens, identification of the rubber compounds, time and temperature of vulcanization.

14.1.2 Conditioning time and temperature.

14.1.3 Date and time of the start of exposure.

14.1.4 Test chamber temperature, both nominal and that measured on a daily basis.

14.1.5 Test chamber ozone partial pressure, in mPa, both nominal and that measured on a daily basis.

14.1.6 Type of specimen and elongation or mounting type (cured or cemented) used during the test.

14.1.7 Size of pulleys used.

14.1.8 Elapsed time or number of test cycles to the first observed cracking and/or a description of the character of the ozone cracking at the various observation. If testing to pass/fail, report the total exposure time.

14.1.9 If desired, a photograph may be taken to provide a permanent record for evaluation.

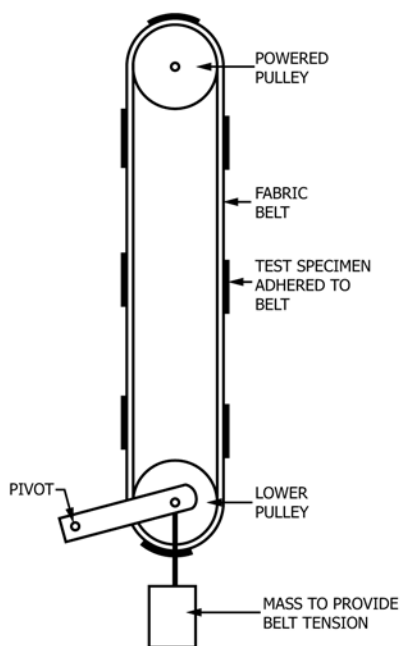
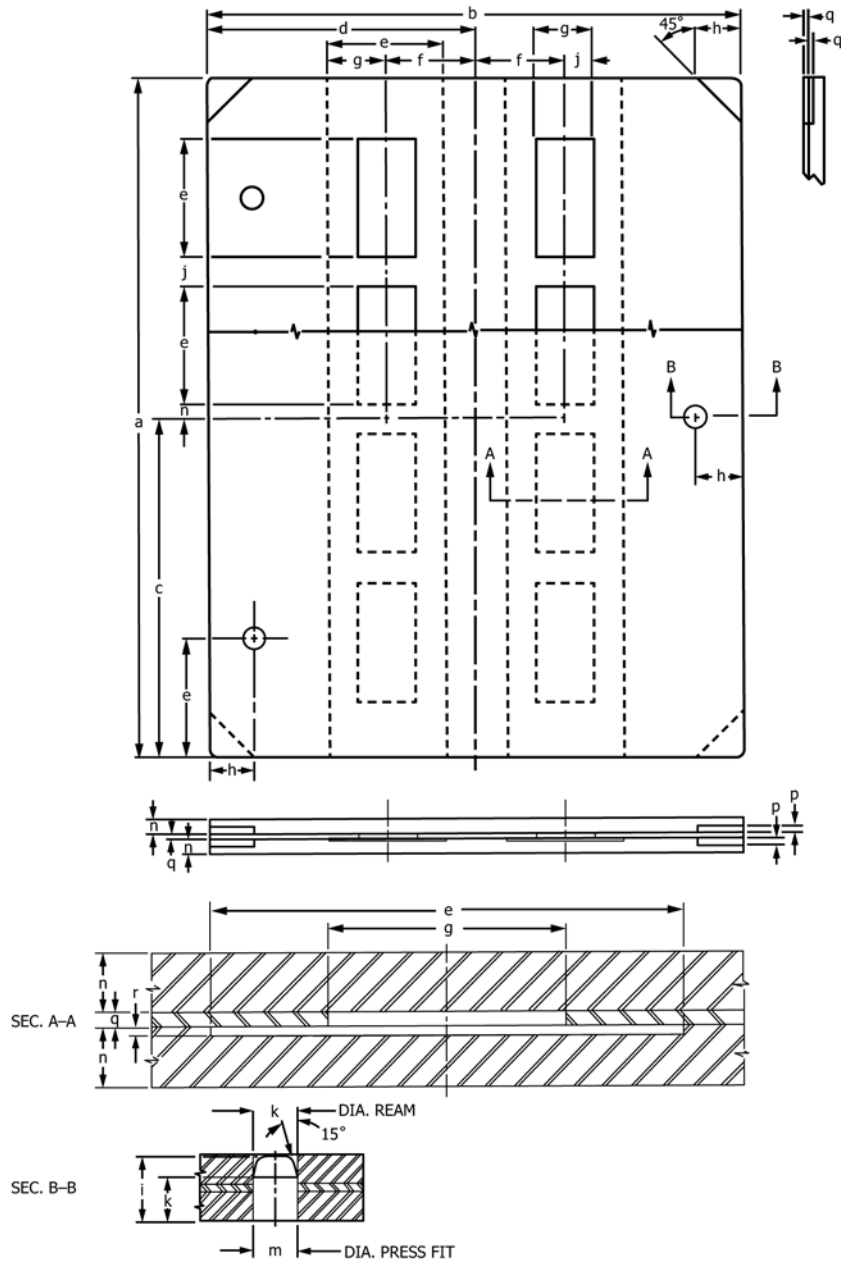


FIG. 2 Schematic Diagram of Method A, Procedure A2 Tester



mm	in.	mm	in.
(a) 584	23.0	(i) 27.78	1.094
(b) 457	18.0	(j) 25	1.0
(c) 292	11.5	(k) 19.05	0.750
(d) 228	09.0	(m) 18.92	0.745
(e) 102	04.0	(n) 12.7	0.50
(f) 76	03.0	(p) 6.3	0.25
(g) 51	02.0	(q) 3.17	0.125
(h) 38	01.5	(r) 1.65	0.065

FIG. 3 Method A, Procedure A2 Mold Schematic

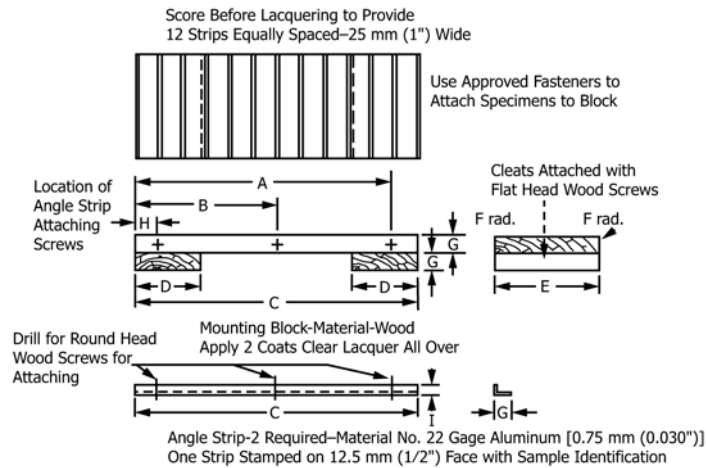
**METHOD B, PROCEDURE B1—EXPOSURE OF STRAIGHT SPECIMENS**

**15. Apparatus**

15.1 *Mounting Block*—A rectangular wooden block shall be used for supporting the extended specimens. The block shall be

140 mm (5.5 in.) wide and approximately 380 mm (15 in.) long and shall have a thickness of not less than 22 mm (0.875 in.). The block shall have the grain running lengthwise and shall be suitably reinforced on the back to prevent warping. Both of the 380-mm edges of the face carrying the specimens shall be rounded with a 3-mm (0.125-in.) radius. (Fig. 4 shows the





	A	B	C	D	E	F	G	H	I
mm	350	190	380	90	140	3	22	32	12
in.	15.75	7.5	15.0	3.5	5.5	0.125	.0875	1.25	0.5

FIG. 4 Apparatus for Mounting Straight Rubber Test Specimens, Method B, Procedure B1

details of construction.) The block shall have a smooth finish and shall be painted with two coats of clear lacquer or spar varnish.

15.2 *Fasteners*—Aluminum tacks, or other suitable material fasteners inert to ozone shall be used for fastening the specimens to the edges of the wooden block.

15.3 *Angle Strips*—Right angled aluminum molding strips 13 by 22 mm (0.5 by .0875 in.) and of approximately No. 22 gauge thickness, for shielding the specimens where tacked and bent over the edges of the block. Strips made from commercial aluminum sheet of Alloy No. 2-S are suitable.

15.4 An alternative device (refer to Fig. 5), constructed of a material with minimal reaction to ozone, such as stainless steel or aluminum, with dimensions suitable to stretch the test specimen 20 % (refer to Section 16) and allowing for a minimum space between specimens of 3.175 mm (0.125 in.). The specimen grip area shall be grooved or crosshatched to prevent slippage.

15.5 *Ozone Chamber*—The ozone chamber shall conform to the requirements specified in Section 5.

**16. Test Specimens**

16.1 The test specimens shall be rectangular strips 25 mm (1 in.) in width by 150 mm (6 in.) in length cut from standard laboratory test sheets (refer to Practice D3182) having a thickness of 1.9 mm (0.075 in.) minimum and 2.5 mm (0.100 in.) maximum. The “milling grain” shall be in the lengthwise direction of the specimen.

16.2 Duplicate test specimens shall be tested whenever possible.

**17. Procedure**

17.1 Firmly fasten the test specimens at one end to one long side of the test block, spacing them 6 mm (0.55 in.) apart and using three aluminum tacks per specimen. Then draw the strips



FIG. 5 Alternative Apparatus for Mounting Straight Rubber Test Specimens, Method B, Procedure B1

across the face of the block in such a manner as to cause an extension of 20 %, measured between gage marks that shall be 100 mm (4 in.) apart and centered at the middle of each strip. Fasten the other end of each specimen in the same way to the opposite long side of the block. Mount the aluminum shields by means of screws on each of the long sides so that the 22-mm



(0.875-in.) leg covers the tacked ends of the specimens and the 13-mm (0.15-in.) leg covers the specimens at the bend on to the face of the block.

17.2 Follow the exposure and other procedures described in Section 24.

17.3 Alternate methods of mounting and stretching the specimens are permitted providing they do not adversely effect the quality of the test.

**METHOD B, PROCEDURE B2—EXPOSURE OF BENT LOOP SPECIMENS**

**18. Apparatus**

18.1 *Clamping Strips*—Made of medium-soft wood for clamping the specimens. Each strip shall be 13 mm (0.5 in.) in thickness, 25 mm (1 in.) in width, and 575 mm (23 in.) in length. Holes shall be drilled through the 13-mm thickness of each strip at intervals of 40 mm (1.6 in.) starting 14 mm (0.6 in.) from one end. The holes shall be made approximately 4 mm (0.160 in.) in diameter, and shall match in paired strips. The strips shall be fastened together using appropriate round-head chromium-plated or galvanized iron machine screws fitted with nuts.

18.2 *Clamping Devices*—Made from aluminum also may be used in place of the wood clamping strips. Several types of metal angles and channels have been successfully used, the following is an example:

18.2.1 The use of aluminum channels 3.5 mm (0.138 in.) thick by 298 mm (11.75 in.) in length by 19 mm (0.75 in.) wide and 19 mm high, joined together to hold the specimens. The channel sides have three holes drilled 8.0 mm (0.31 in.) in diameter 32 mm (1.25 in.) from each end and 150 mm (5.875 in.) in the center of the channel. The channels shall be fastened together using appropriate hexagon head chromium-plated or galvanized iron screws fitted with washers and wing nuts.

18.3 *Base Panel*—Made of medium-soft wood upon which the clamped specimens are mounted. It shall be 530 mm (24 in.) long, 205 mm (8 in.) wide, and at least 13 mm (0.5 in.) thick. Three crosspieces each 205 mm (8 in.) long, 25 mm (1 in.) wide, and 6 mm (0.25 in.) thick, shall be fastened to the base. The crosspieces shall be mounted 19 mm (0.75 in.) from each end of the panel and at the center. Each crosspiece shall be held in place by three 50 mm (2 in.) round head chromium plated or galvanized iron machine screws. These screws shall be fitted with washers on the underside of the base panel and shall fit into a countersink. The screws shall pass through the panel and through the crosspieces, 32 mm (1.25 in.) from each end of the crosspieces and through the center. These screws shall protrude above the surface of the panel to a height of about 35 mm (1.38 in.) and shall be used to fasten the wooden specimen strips securely to the base as described in 12.2.

18.4 All wooden panels, strips, and crosspieces, shall be painted with two coats of clear lacquer or spar varnish.

18.5 *Ozone Chamber*—The ozone chamber shall conform to the requirements specified in Section 5.

**19. Test Specimens**

19.1 The test specimens shall be rectangular strips 25 mm (1 in.) wide by 95 mm (3.75 in.) in length, cut from standard laboratory test sheets (refer to Practice D3182) having a thickness of 1.9 mm (0.075 in.) minimum and 2.5 mm (0.100 in.) maximum. The “milling grain” shall be in the lengthwise direction of the specimen.

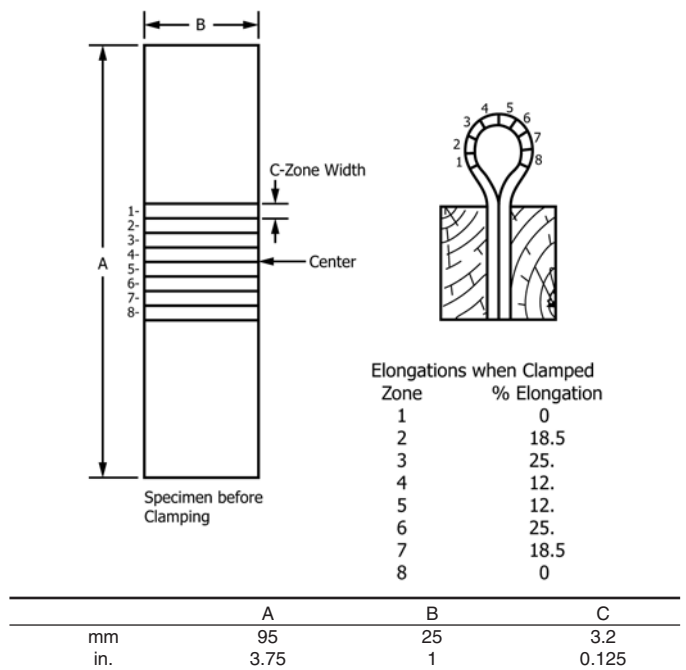
19.2 Duplicate test specimens shall be tested whenever possible.

**20. Procedure**

20.1 Loop the test specimens until their ends meet and then insert these ends between the paired wooden or aluminum strips until they are flush with the underside of the strips. The minimum distance between the specimens shall be 6 mm (0.25 in.). Clamp the wooden strips together by means of machine screws so that the specimens are firmly held in place. As a result of this procedure, 25 mm (1 in.) of each end of the specimen will be covered by the wooden strips, which will act as a protective shield. The remaining 43 mm (1.75 in.) of the specimen shall form a loop having a varying elongation along its length, as shown in Fig. 6.

20.2 Mount the rack of clamped specimens on the cross-pieces attached to the base panel by passing the protruding machine screws of the panel between the paired wooden or aluminum strips and fastening them with nuts and washers, as shown in Fig. 7.

20.3 Follow the exposure and other procedures described in Section 24.



**FIG. 6 Cross-Sectional View of Mounted Looped Specimen Showing Elongation at Different Parts, Method B, Procedure B2**

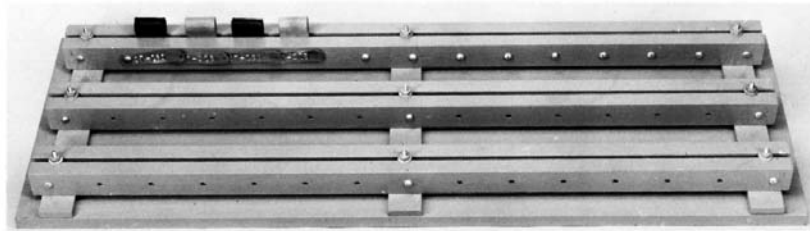


FIG. 7 Assembled Panel, Showing Method of Mounting Looped Specimens

**METHOD B, PROCEDURE B3—EXPOSURE OF TAPERED SPECIMENS**

**21. Apparatus**

21.1 *Mounting Frames*—Wooden frames for mounting the test specimens shall have the following dimensions: inside width, 100 mm (4 in.); overall width, 175 mm (7 in.); inside length, 300 mm (12 in.); overall length, 380 mm (15 in.). For constructing the frames, 25 mm (1 in.) thick medium softwood shall be used, and the members shall be joined with dowels using waterproof glue for the bond. The frame shall be planed smooth and painted with two coats of clear lacquer or spar varnish.

21.2 *Fasteners*—Aluminum tacks, size No. 6, made from Alloy No. 51-S, or stainless steel staples for fastening the specimens to the wooden frame.

21.3 *Angle Strips*—Aluminum strips 38 mm (1.5 in.) wide and of approximately No. 22 gage, for shielding the specimens. Strips made from commercial Alloy No. 2-S are suitable.

21.4 An alternative device (refer to Fig. 5), constructed of a material with minimal reaction to ozone, such as stainless steel or aluminum, with dimensions suitable to stretch the test specimen 10 %, 15 %, and 20 % (refer to 23.3) and allowing for a minimum space between specimens of 3.175 mm (0.125 in.). The specimen grip area shall be grooved or crosshatched to prevent slippage.

21.5 *Ozone Chamber*—The ozone chamber shall conform to the requirements specified in Section 5.

**22. Test Specimens**

22.1 The test specimens shall be die-cut tapered strips having outside dimensions as shown in Fig. 8, cut from standard laboratory test sheets having a thickness of 1.9 mm (0.075 in.) minimum and 2.5 mm (0.100 in.) maximum. The grain shall be in the lengthwise direction of the specimen.

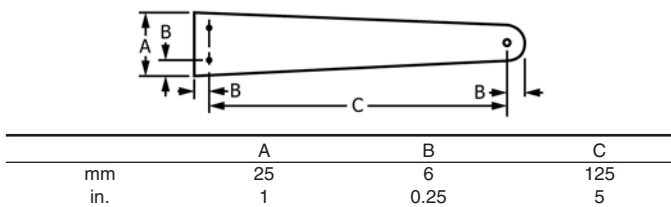


FIG. 8 Die-Cut Tapered Specimen

22.2 Duplicate test specimens shall be tested whenever possible.

**23. Procedure**

23.1 Place an identification mark on the broad end of each test specimen, using materials that will not have a deleterious effect on the specimens during aging and that shall not extend beyond the area covered by the aluminum angle strips.

23.2 Superimpose upon the specimen a template conforming to the dimensions shown in Fig. 8, and mark the specimen with a sharp pointed pencil to conform with the holes in the template.

23.3 Draw parallel lines on the frame at the desired distance to give the required overall elongation in accordance with the following table:

Overall Elongation, %	Distance Between Parallel Lines, mm (in.)
10	140 (5.50)
15	146 (5.75)
20	152 (6.00)

23.4 Fasten the stretched specimen to the frame in such a manner that the fasteners or staples driven through the penciled dots on the specimen will coincide with the awl points on the two lines. These awl points are spaced according to the holes in a template for the required elongation, as shown in Fig. 9. Because of the taper, the elongation for any one area will vary with the width of the strip. By placing bench marks at regular intervals along the center line of the tapered strip prior to stretching, it is possible to determine the percentage elongation for any given area by measuring the distance between the bench marks after the strip has been elongated, and applying the following formula:

$$\text{Elongation, \%} = [(L_a - L_o)/L_o] \times 100 \quad (1)$$

where:

$L_a$  = length after stretching, and

$L_o$  = length before stretching.

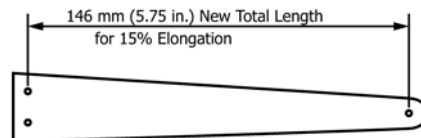


FIG. 9 Template for Spacing Holes in Frame for Mounting Elongated Specimens (Template Shown is for 15% Elongation)

23.5 After the specimens have been mounted, mount the aluminum shield on each of the long sides so that the tacks holding the specimens and the markings are covered.

23.6 Follow the exposure and other procedures described in Section 24.

#### **24. Procedures Common to Method B, Procedures B1, B2, and B3 (Procedure B4 has different parameters)**

24.1 Standard ozone pressures shall be 25 mPa, 50 mPa, 100 mPa, and 200 mPa with a  $\pm 10\%$  tolerance on each pressure (see **Note 1** and **Note 3**) and refer to Section 5.

24.1.1 Other partial pressures may be selected according to the particular goals of the test program, and they shall be referred to as optional partial pressures. The ozone concentration or partial pressure shall be measured once a day for routine work and more often for special test conditions. This measurement shall be made with the ozone chamber “loaded” with specimens. If specimens are loaded into a chamber that has been exhausted of ozone, the ozone concentration should reach  $\pm 10\%$  of the selected ozone concentration within 15 min.

24.1.2 Conduct an analysis with the chamber containing test specimens. When the number of specimens is kept at a minimum, “empty” and “loaded” ozone partial pressures will closely agree. Determine the magnitude of the reduction of ozone partial pressure for “empty” versus “loaded” for each set of operating variables employed. This will vary for each laboratory. Refer to Test Methods **D4575** for ozone analysis procedures.

24.2 The standard temperature shall be  $40 \pm 1^\circ\text{C}$  ( $104 \pm 2^\circ\text{F}$ ). Temperature affects the rate of ozone degradation. Other test and conditioning temperatures, elevated or subnormal may be selected and may be of significant importance.

24.3 Adjust the ozone chamber to conditions for exposure prior to the introduction of the specimens.

24.4 All rubber sheets for ozone testing shall be vulcanized between aluminum foil 0.05 mm (0.002 in.) in thickness or polyester film of similar thickness.<sup>5</sup> These films will mildly adhere to most commercial rubbers. At the time specimens are cut for ozone testing, the foil or film can be easily removed providing a fresh surface.

24.5 The foil or film covered sheets shall be stored in an ozone-free atmosphere for a minimum of 3 h at the agreed test temperature.

24.6 Strip off protective foil or film, then extend specimens for Procedures B1 and B3 to the prescribed elongation and fasten them to their respective holders. Bend the specimens for Procedure B2 in the form of a loop and fasten in the holder heretofore described.

**NOTE 5**—Other types of specimens may be tested, but the results may not agree with tests made with standard specimens. (See Test Method **D1171** for use of a specimen having a triangular cross section.)

24.7 Initiate conditioning immediately after mounting specimens.

24.8 Condition the mounted specimens for 24 h in an ozone-free atmosphere at the temperature at which they are to be exposed to ozone.

24.8.1 Minimize the time of exposure to ambient conditions during transfer to the ozone test chamber, which shall have been preset to the agreed test temperature.

24.8.2 Other conditioning times and procedures may be used, but shall be clearly indicated in the report.

24.9 Begin exposure at the proper temperature and ozone partial pressure.

24.10 Make observations for detecting the appearance of cracking with sufficient frequency to be able to detect the first appearance of ozone cracking.

24.10.1 This frequency will depend on the resistance to ozone attack of the rubbers being tested. Recommended observation magnification is 7 $\times$ , except in the case of the triangular specimen where the magnification shall be 2 $\times$  (refer to **Note 5**).

24.11 When comparisons are being made with a standard reference material, exposures may be made for a fixed time. Evaluate the degree of cracking using an agreed upon scale.

#### **25. Report**

25.1 The report shall include the following:

25.1.1 Description of the specimens, identification of the rubber compounds, time and temperature of vulcanization.

25.1.2 Conditioning time and temperature.

25.1.3 Date and time of the start of exposure.

25.1.4 Test chamber temperature, both nominal and that measured on a daily basis.

25.1.5 Test chamber ozone partial pressure, in mPa, both nominal and that measured on a daily basis.

25.1.6 Type of specimen and elongation or mounting type used during the test.

25.1.7 Elapsed time or number of test cycles to the first observed cracking or a description of the character of the ozone cracking at the various observation, or both. If testing to pass/fail, report the total exposure time.

25.1.8 If desired, a photograph may be taken to provide a permanent record for evaluation.

#### **26. Precision and Bias**

26.1 Since all methods and procedures yield essentially qualitative comparison data, candidate test compound versus a control, precision as normally expressed for quantitative measurement test methods is not directly applicable.

#### **27. Method B, Procedure B4**

##### **EXPOSURE OF TRIANGULAR SPECIMENS**

27.1 *Significance and Use*—This test procedure is not applicable to materials ordinarily classed as hard rubber, but is adaptable to molded or extruded soft rubber materials and sponge rubber for use in window weatherstripping as well as similar automotive applications.

27.2 *Summary of Test Procedures:*

27.2.1 Procedures are given for preparing triangular cross-section specimens, for mounting them in strained condition around specified circular mandrels, and for rating the effect of exposure as evidenced by the appearance of surface cracks.

The type, time, temperature, and other conditions of exposure are not given but shall be agreed upon by the purchaser and the seller and shall be completely described in the test reports.

27.2.2 Two techniques are described, the exposure rating and the quality-retention rating.

27.2.3 Molded or extruded rubber products must withstand the effects of ozone cracking for satisfactory use. This test enables a simple comparison of performance to be made under specified test conditions in a laboratory test chamber. No direct correlation between test performance and service performance can be claimed due to the wide range of service conditions.

27.2.3.1 This test is may also be performed outdoors, following the procedures outlined in Test Method D1171.

27.3 Apparatus:

27.3.1 *Mounting Mandrel*—A wood mandrel 50 mm (2 in.) in outside diameter, suitably coated with spar varnish or a clear lacquer. The mandrel may be supported by any convenient method.

27.3.2 *Fastening Wire*—Enameled copper wire.

27.4 Test Specimen:

27.4.1 The triangular cross-section test specimen shall be a molded, extruded, or sponge section with a continuous skin or surface layer, 250 mm (10 in.) long, and cured in a straight position. The cross-sectional dimensions shall be as shown in Fig. 10. Duplicate specimens shall be tested.

27.4.2 It is recommended that the manufacturer observe a minimum rest period of 24 h between compound mixing and compound preparation, and a minimum rest period of 1/2 h between compound preparation and curing when preparing specimens. The finish of the specimens shall be as smooth as possible.

PROCEDURE B4 – A EXPOSURE RATING

27.5 Procedure:

27.5.1 *Mounting of Specimens*—Place bench marks on each specimen with a crayon, 190 mm (7.5 in.) apart and 32 mm (1.25 in.) from the ends. Form the specimens into a loop by placing the 19-mm (0.75-in.) sides together and tying securely with wire at the bench marks. Slip the complete loop over the mandrel. Place as many specimens on the mandrels as length of mandrel will permit.

27.5.2 Conditioning and Positioning:

27.5.2.1 *Conditioning Period*—After the specimens are mounted allow them to condition for 70 to 72 h at 40 ± 1°C (104 ± 2°F) either in a temperature controlled cabinet or preferably in the ozone cabinet itself. After this conditioning period transfer to the ozone chamber or, if already conditioning in this apparatus, switch on the ozone so that it rises to a level of 50 ± 5 mPa partial ozone pressure.

27.5.2.2 *Positioning*—The position of mounted specimens exposed in the ozone test chamber shall be such that there is complete circulation of air around the specimens and the specimens do not touch anything during the necessary handling.

27.5.3 *Test Conditions*—Expose specimens for 72 h at 40 ± 1°C (104 ± 2°F) at an ozone level of 50 ± 5 mPa partial ozone pressure, unless otherwise specified by agreement between the purchaser and the seller. The specimens may be removed from the test chamber after 24, 48 and 72-h exposure for rating.

27.5.4 *Rating of Exposed Specimens*—When the specified exposure period is completed, examine the specimens before removal from the mandrel, and evaluate the degree of cracking by comparison with the reference standards shown in Fig. 11 using a 2× power headgear binocular magnifier or equivalent. If the size of the cracks at 2× magnification is less than those shown on one photograph but greater than those shown on the next lower rating picture, give the lower rating.

27.5.5 Hazards:

27.5.5.1 **Warning**—Ozone is a hazardous substance. Consult and follow all applicable laws, rules, and regulations regarding exposure to ozone.

27.5.6 Report:

27.5.6.1 Report the following information:

27.5.6.2 Description of the specimen, giving the identification number of compound and vulcanization data, if known, including date,

27.5.6.3 Date of start of exposure, and complete information describing conditions of exposure,

27.5.6.4 Rating number of exposed specimens, and

27.5.6.5 If desired, a photograph at 2× magnification.

PROCEDURE B4 – QUALITY-RETENTION RATING

27.5.7 Procedure:

27.5.7.1 **In ozone chamber exposure Procedure B4, no rating is intended, rather, there shall be no cracks permitted under 2× magnification.** The report is pass/fail (no cracks/cracks).

27.5.7.2 *Exposure Procedure B-4*—Expose specimens for 70 h at 40 ± 1°C (104 ± 2°F) at an ozone level of 50 ± 5 mPa partial ozone pressure, unless otherwise specified by agreement between the purchaser and seller. The specimens may be removed from the test chamber after 22, 46, and 70-h exposure, for examination.

27.5.7.3 On materials known to be substantially resistant to ozone, test specimens and fixtures as described under Method B, Procedure B2 in Test Method D1149 (formerly Procedure B in Test Method D518) may be used.

27.5.8 Report:

27.5.8.1 Report the following information:

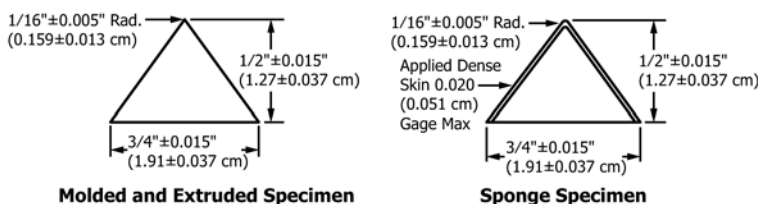


FIG. 10 Cross-Sectional Dimension of Test Specimens



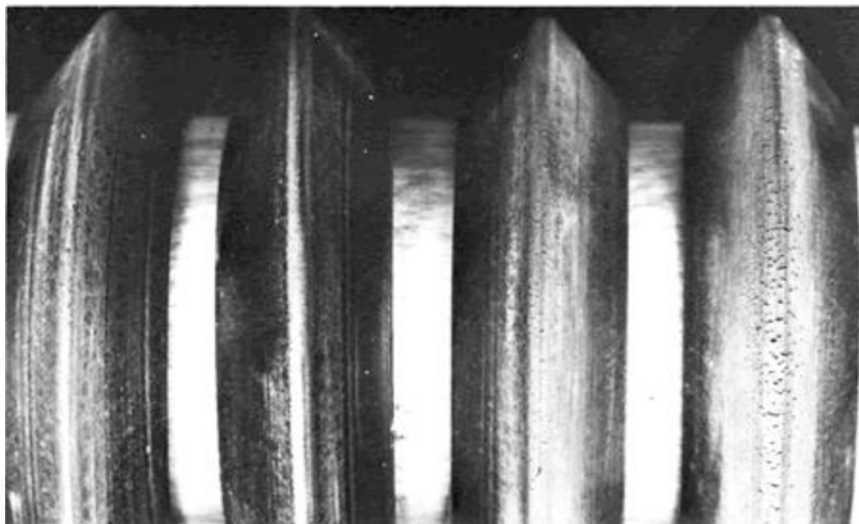


FIG. 11 Ratings for Weathering Tests of Rubber Compounds (Specimens Mounted on Mandrel) (2×)

27.5.8.2 Description of the specimen, giving the identification number of the compound and date of vulcanization,

27.5.8.3 Date of start of exposure and complete information describing condition of exposure,

27.5.8.4 Crack rating number composed of three digits representing three readings taken each two weeks for six weeks or taken at three equal intervals, and

27.5.8.5 Quality-retention value pass/fail (no cracks/cracks).

27.5.9 *Precision and Bias*—Both the exposure rating and the quality-retention rating are essentially rank order or qualitative comparison methods. Precision as normally expressed for quantitative-measurement test methods is not directly applicable.

## 28. Keywords

28.1 chamber; cracking; deterioration; ozone; rubber; surface; test

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