



Designation: D1149 – 07

Standard Test Methods for Rubber Deterioration—Cracking in an Ozone Controlled Environment¹

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 (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the 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1.2.1 Test Method **D518** and Test Methods **D3395** have henceforth been withdrawn and superseded by Test Methods D1149.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:²

D518 Test Method for Rubber Deterioration—Surface Cracking³

D1171 Test Method for Rubber Deterioration—Surface Ozone Cracking Outdoors or Chamber (Triangular Specimens)

D1349 Practice for Rubber—Standard Temperatures 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 Chamber³

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 incorporated into these test methods. They remain technically unchanged and will be superseded by Test Methods D 1149. Please refer to Test

¹ 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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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.

³ Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

Method **D1171** for Test Method **D518** 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.

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—Triangular Specimens	24 (formerly D1171)

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 ft³). The internal chamber shall 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 ± 1 mm (1.97 ± 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

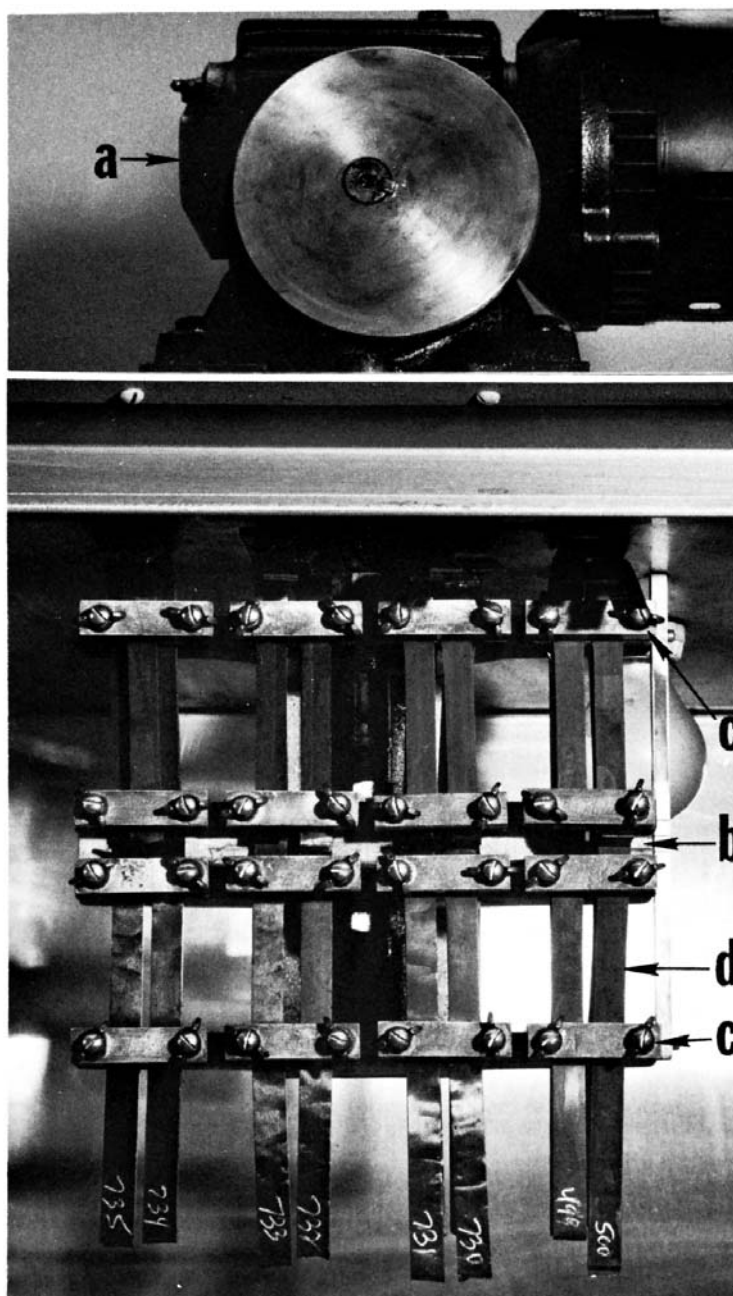
7. Apparatus

7.1 Flexing Device:

7.1.1 The device⁴ 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 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).

⁴ 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,¹ which you may attend.



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

7.2 *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 ± 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 ± 0.03 mm (0.40 ± 0.01 in.) wide by 100 ± 25 mm (4 ± 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 film⁵ 0.1 mm in thickness. The foil will adhere mildly to most commercial rubbers. At the time

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