Designation: D 3395 – 99

Standard Test Methods for Rubber Deterioration—Dynamic Ozone Cracking in a Chamber[1]

This standard is issued under the fixed designation D 3395; 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.

1. Scope

1.1 These test methods are used to estimate the cracking resistance of vulcanized rubber exposed under dynamic strain conditions to a chamber atmosphere containing ozone at a fixed partial pressure. The effect of sun or ultraviolet light is excluded.

1.2 These test methods are not applicable to materials ordinarily classed as hard rubber, but are adaptable to molded or extruded soft rubber.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazard statement, see Note 1 and 5.1.*

NOTE 1—**Warning—Ozone is a hazardous chemical.**

2. Referenced Documents

2.1 *ASTM Standards:*

D 1149 Test Method for Rubber Deterioration—Surface Ozone Cracking in a Chamber[2]

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[2]

D 4575 Test Methods for Rubber Deterioration—Reference and Alternative Method(s) for Determining Ozone Level in Laboratory Test Chambers[2]

3. Summary of Methods

3.1 The methods appear as follows:

	Sections
Method A—Tensile Elongation	5 to 8
Method B—Belt Flex	9 to 12

3.2 In Method A, rectangular test specimens are subjected to dynamic maximum amplitude tensile strain of 25 ± 3 % at a fixed frequency of 0.5 Hz (30 cpm). In Method B, flat-sheet test specimens are affixed to a fabric belt and the belt is run over a set of two pulleys that induce a surface strain (due to the bending) during passage over the pulleys.

3.3 Refer to Test Method D 4575 for the reference and optional alternative methods for the measurement of the ozone partial pressure in laboratory test chambers.

4. Significance and Use

4.1 The significance of these tests is mainly in their ability to differentiate, in a comparative sense, between different degrees of ozone resistance under the limited and specified conditions of the accelerated tests.

4.2 These test methods may not give results correlating exactly with outdoor exposure tests or service performance since the correlation of accelerated ozone tests with such performance is highly dependent upon the specific conditions of both the accelerated ozone and outdoor exposures. Conditions that influence the accelerated tests are ozone partial pressure, air flow, temperature, stress-relaxation, and the bloom of additives.

4.3 Outdoor or service performance is influenced by such weather conditions as rainfall, ambient temperature, sun light, and the strain cycles imposed.

METHOD A—TENSILE ELONGATION TEST

5. Hazards

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

6. Apparatus

6.1 *Flexing Device:*[3]

6.1.1 The device 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.

6.1.2 The action of the flexing apparatus is to impart a straight-line motion to the clamps holding the test specimens;

[1] These test methods are under the jurisdiction of ASTM Committee D-11 on Rubber and are the direct responsibility of Subcommittee D11.15 on Degradation Tests.

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[2] *Annual Book of ASTM Standards*, Vol 09.01.

[3] Model 701-1 Flexer, available from the Mast Development Co., Inc., 2212 E. 12th St., Davenport, IA 32803, has been found satisfactory for this method.

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).

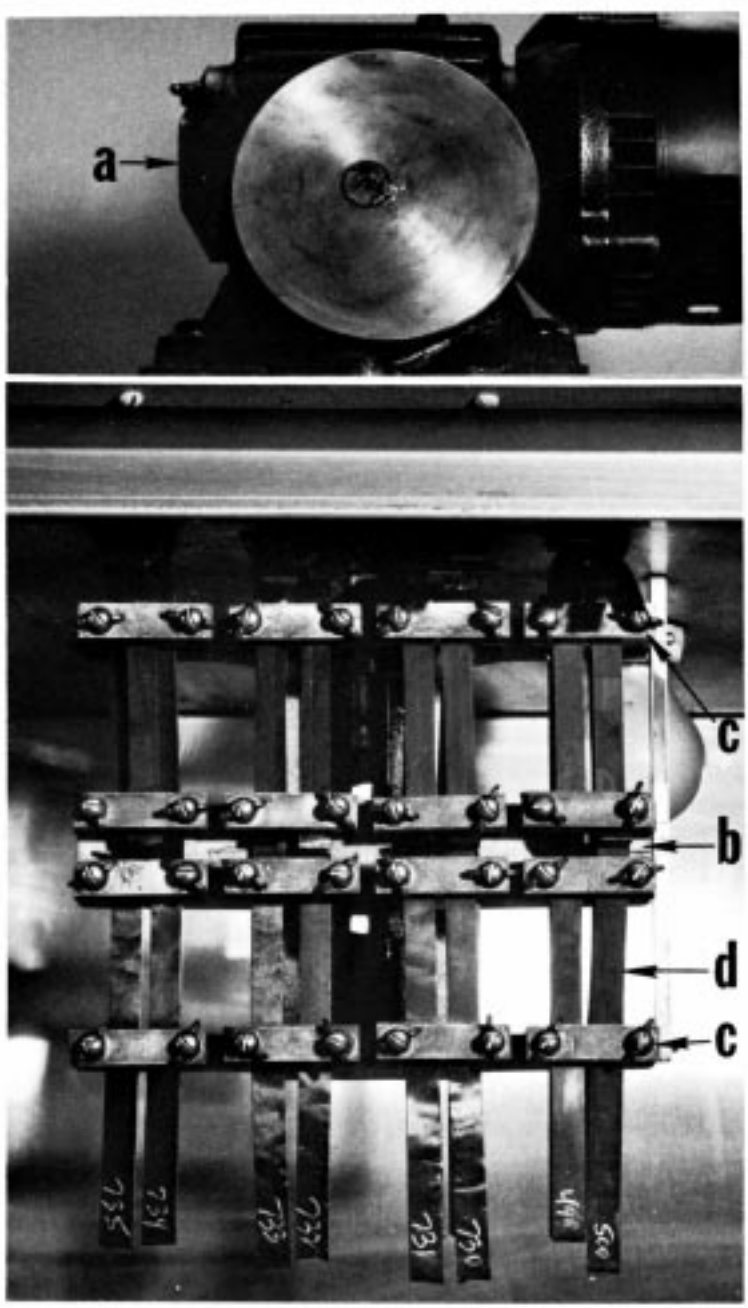
6.2 *Ozone Chamber*— The ozone chamber shall conform to the requirements specified in Test Method D 1149. It shall be equipped to permit flexing of the specimens from 0 to 25 ± 3 % extensions at a rate of 0.5 Hz (30 cpm).

6.3 *Apparatus for Measuring Ozone Partial Pressure*—The apparatus for measuring ozone partial pressure shall conform to the requirements specified in Test Method D 4575.

7. Test Specimens

7.1 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 D 3182. 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 2—It is strongly suggested that all rubber sheets for ozone testing



Method A
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 Dynamic Strain Ozone Tester

be cured between aluminum foil or polyester film[4] 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.

8. Procedure

8.1 *Pre-Test Conditioning—Nominal Period*:

8.1.1 Prior to testing, condition all free-surface (that is, no foil specimens) for 24.0 ± 0.5 h at 23 ± 2°C in a sealed opaque container that has an ozone-free atmosphere. Different compounds are to be contained separately in the sealed opaque container.

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

8.2 *Operation of Test Apparatus*:

8.2.1 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 (±1 min).

8.2.2 The standard ozone partial pressure shall be 50 ± 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 Method D 4575 for ozone analysis procedures.

8.2.3 The standard temperature shall be 40 ± 1°C (104 ± 2°F). Higher temperatures cause an acceleration in the rate of ozone attack. These higher temperatures shall be optional.

8.2.4 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×. 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 door closed as much as possible).

9. Report

9.1 The report shall include the following:

9.1.1 Identification of the materials tested and dates of testing,

9.1.2 Ozone partial pressure, both nominal and that actually measured on a daily basis,

9.1.3 Temperature of the test,

9.1.4 Time or number of extension cycles to the first observed cracking or a description of the character of the ozone cracks at various periods of exposure, and

9.1.5 Pre-conditioning information (that is, number of hours of ozone-free conditioning) at 23 ± 2°C.

METHOD B—BELT FLEX TEST

10. Apparatus

10.1 *Belt Flex Device*:

10.1.1 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 rpm) with the 63.5-mm diameter pulleys, and at 1.04 Hz (62.5 rpm) when using the 102-mm diameter pulleys. Alternatively, the drive shaft may be rotated at 3.75 Hz (225 rpm) 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.

10.1.2 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 12¼by 15 in.) with a volume of 0.16 m^3 (10^4 $in.^3$). Fig. 3 is a schematic diagram of the test apparatus.

10.2 *Test Chamber*:

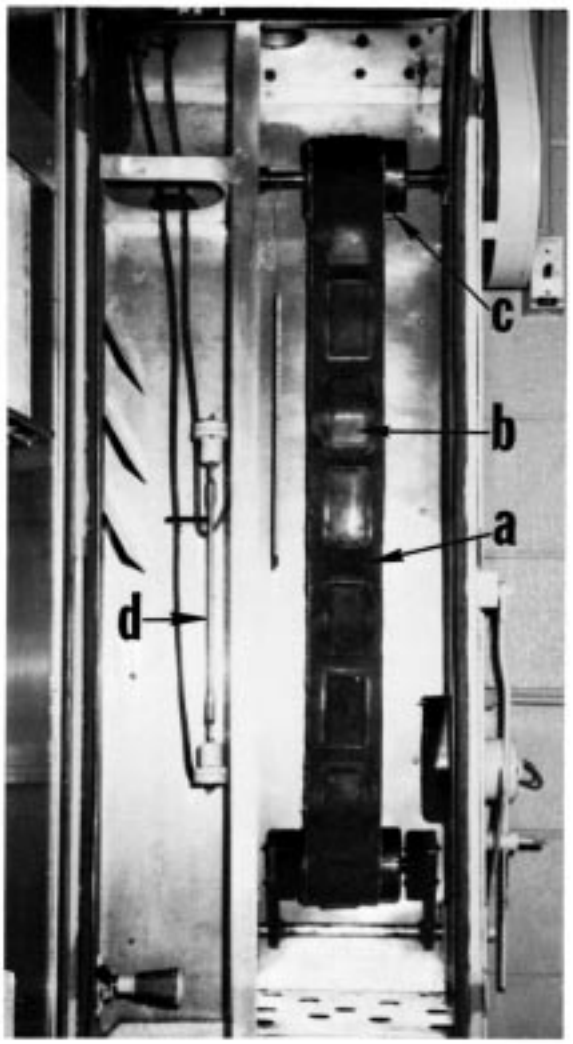
10.2.1 The material for constructing the test chamber and equipment for measuring ozone partial pressure shall conform to those described in Test Method D 1149.

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

11. Test Specimens

11.1 The specimen belt, to which the samples are either cured or cemented, shall be a one-ply belt of 1.1-kg/m^2 (32-oz/yd^2) 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.

[4] A 200 Mylar, or its equivalent, has been found satisfactory for this method.



Method B (Door Open)
a—belt
b—attached test specimen
c—upper pulley
d—UV ozone generator tube

FIG. 2 Dynamic Strain Ozone Tester

11.2 Fig. 4 shows a drawing of a multicavity mold used to prepare molded specimens.

11.3 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.

12. Procedure

12.1 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.

12.2 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 10.1.2, 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

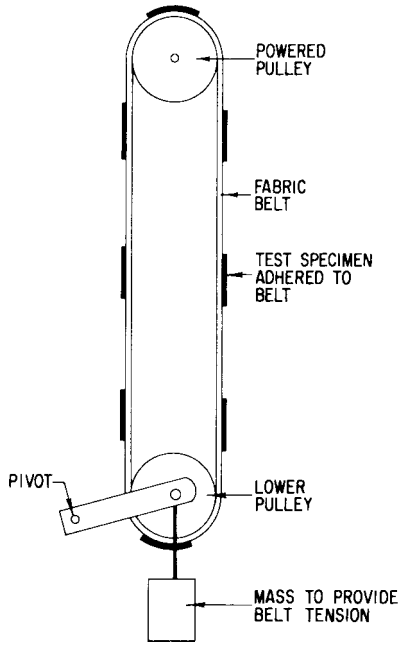


FIG. 3 Schematic Diagram of Method B Tester

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 Method D 1149 at 40 ± 1°C (104 ± 2°F).

12.3 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, door open for a minimum time).

12.4 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.

13. Report

13.1 The report shall include the following:

13.1.1 Description of the specimens, identifying the rubber compounds, time and temperature of vulcanization, cured or cemented type of specimen mounting, size of specimen, and conditioning information,

13.1.2 Date and time of start of exposure,

13.1.3 Ozone partial pressure with belt running (daily or more frequently),

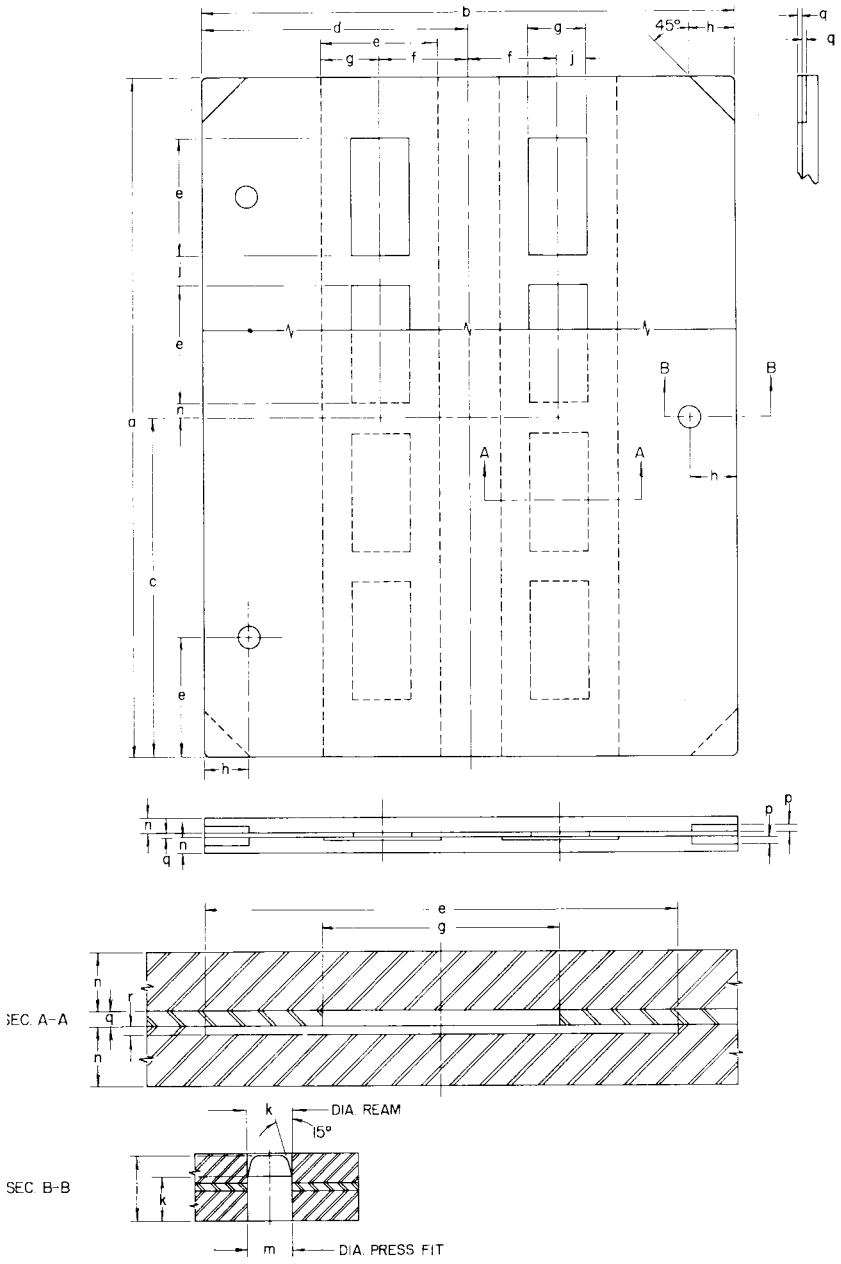
13.1.4 Temperature of chamber,

13.1.5 Time or cycles to initial cracking,

13.1.6 Total exposure time, or number of test cycles, and

13.1.7 Size of pulleys used.

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



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	(9.0)	(*m*) 18.92	(0.745)
(*e*) 102	(4.0)	(*n*) 12.7	(0.50)
(*f*) 76	(3.0)	(*p*) 6.3	(0.25)
(*g*) 51	(2.0)	(*q*) 3.17	(0.125)
(*h*) 38	(1.5)	(*r*) 1.65	(0.065)

FIG. 4 Mold Drawing Legend

14. Precision and Bias

14.1 Precision and bias statements are in the process of being prepared. They will be added to this method when they are completed.