Standard Test Method for Rubber Deterioration—Surface Ozone Cracking in a Chamber[1]

This standard is issued under the fixed designation D 1149; 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 This test method covers the estimation of the resistance of vulcanized rubber to cracking when exposed to an atmosphere containing ozone. The rubber specimens are kept under a surface tensile strain, and the ozone content or partial pressure in the test chamber is maintained at a fixed value.

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

1.3 *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.* For a specific hazard statement, see Note 1 and 6.1.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 518 Test Method for Rubber Deterioration—Surface Cracking[2]

D 1171 Test Method for Rubber Deterioration—Surface Ozone Cracking Outdoors or Chamber (Triangular Specimens)[2]

D 1349 Practice for Rubber—Standard Temperatures for Testing[2]

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

3. Summary of Test Method

3.1 Rubber specimens are prepared from tensile sheets vulcanized between protective aluminum foil or polyester film. Specimens are conditioned at the temperature of test for a controlled time.

3.2 These specimens, under a surface of tensile strain, are exposed in a chamber containing a known, controlled concentration of ozone in air, at a controlled prescribed temperature. The partial pressure or concentration of the ozone can be varied. The specimens are examined at intervals and their condition is recorded.

NOTE 2—Refer to Test Methods 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 In service, rubbers must resist the deterioration that ozone cracking produces. This test method provides methods for estimating this performance feature of rubbers. Such test methods can be used for producer-consumer acceptance, referee purposes, and product development.

4.2 The test method may not give results correlating exactly with outdoor exposure tests, since the results are highly dependent on specific conditions. However this test method has been developed to improve this correlation. Conditions that influence accelerated tests are ozone content, relaxation of stress, temperature, and degree of bloom of additives. Additional conditions that influence outdoor tests include the amount of sunshine and rainfall.

4.3 In rubber compounds where ozone resistance has been imparted by the use of waxes or chemical agents, it is important that the specified conditioning of the test specimens before exposure and the temperature of that exposure approximate that of the projected use. The composition and hence the performance of the protective film formed by the diffusion of these materials to the surface of the specimen is a sensitive function of time and temperature.

5. Ozone Test Apparatus

5.1 *Test Chamber*—Requirements for an acceptable ozone test chamber are sufficient air-ozone throughput rate, sufficient internal circulation, and sufficient internal volume. A secondary requirement is that of controlling the temperature within acceptable limits. An acceptable ozone test chamber can be custom-made in a particular laboratory or one of the commercial manufactured chambers which are available may be used. The ozone test chambers shall conform to the following requirements:

5.1.1 The test chamber shall be constructed of a material

---

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

Current edition approved Nov. 10, 1999. Published December 1999. Originally published as D 1149 – 51. Last previous edition D 1149 –91 (1997).

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

with minimal reaction to ozone.

5.1.2 The volume of the chamber shall be at least 0.11 to 0.14 m^3 (4 to 5 ft^3).

5.1.3 A means for generating and controlling an air-ozone stream shall be provided. The generating source shall be located outside of the chamber. The source of air can be either drawn directly from the laboratory or from a compressed air supply. In either case adequate filtration of foreign matter from the stream must be provided. The air-ozone stream shall be introduced into the chamber in such a manner that stratification of ozone is prevented.

5.1.4 Irradiation of an air stream with a mercury vapor lamp is the most common source of the necessary ozonized air stream.[3] The rate of ozone generation in this system is easily controlled by adjusting the power input to the lamp. This adjustment may be manually or, preferably, automatically controlled.

5.1.5 The air-ozone replacement rate or throughput rate must be of a magnitude such that no appreciable reduction in ozone concentration results from the introduction of test specimens. This minimum replacement rate will vary with the ozone level, temperature, number of test specimens introduced, and their reaction with ozone. For many chambers operating under normal conditions (at an ozone partial pressure of approximately 50 mPa), an air-ozone replacement rate of a three-fourth change per minute is an acceptable and adequate value. For thorough and accurate work, especially under unusual conditions, the minimum or safe replacement rate should be determined.

5.1.6 A means of providing adequate internal circulation shall be provided. The air-ozone velocity in the chamber shall be at least 0.6 m/s (2 ft/s). Where it is doubtful that such velocities exist, the installation of an ordinary 1700 r/min electric motor and fan blade of approximately 150-mm (6-in.) diameter and 20 to 30° pitch will produce such air velocities. The motor itself shall not be in the chamber. An extension shaft shall be used with an appropriate seal.

5.1.7 A means of controlling the temperature of the chamber shall be provided. The temperature regulation should be capable of maintaining the test temperature within ± 1°C (± 1.8°F).

5.1.7.1 Ozone testing at optional subambient test temperatures requires the use of refrigerated test chambers meeting the other requirements of this test method.

5.1.8 Chambers must be equipped such that the operator is never exposed to harmful concentrations of ozone. This is done most commonly by exhausting the ozone from the test chamber before opening; however, other venting and protective measures may be used.

6. Hazards

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

7. Sampling of Test Compounds or Products

7.1 A sampling process should be used to ensure that the tests on specimens form either compounded and cured rubber sheets or specimens prepared from products are representative of the lot or other units to be compared.

8. Test Specimens

8.1 The following types of specimens are standard (refer to Test Method D 518):

8.1.1 *Specimen A*—a rectangular strip, 25 by 150 mm (1 by 6 in.), having a thickness between 1.9 and 2.5 mm (0.075 and 0.10 in.). This specimen is tested at 20 % elongation unless otherwise specified.

8.1.2 *Specimen B*—a rectangular strip, 25 by 95 mm (1 by 3.75 in.), having a thickness between 1.9 and 2.5 mm (0.075 and 0.10 in.). This specimen is tested in the form of a bent loop shown in Fig. 1.

8.1.3 *Specimen C*—a tapered strip, shown in Fig. 2, having a thickness between 1.9 and 2.5 mm (0.075 and 0.10 in.). This specimen is tested in extension at 10, 15, or 20 % elongation as specified. The actual elongation increases from the wide to the narrow end of the specimen.

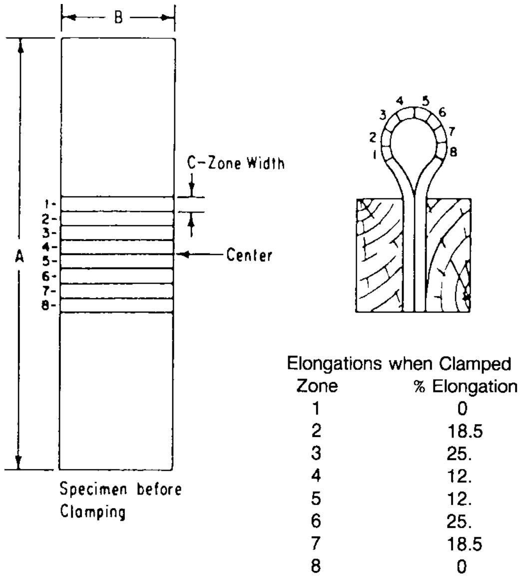
8.2 Other types of specimen may be tested, but the results may not agree with tests made with standard specimens. (See Test Method D 1171 for use of a specimen having a triangular cross section.)

8.3 Unless otherwise specified, the specimen shall be prepared with the grain in the length direction and duplicate specimens shall be tested. The type of specimen to be used shall be prescribed in specifications.

9. Procedure

9.1 Adjust ozone chamber to conditions for exposure.

9.1.1 Standard ozone pressures shall be 25 mPa, 50 mPa, 100 mPa, and 200 mPa with a ± 10 % tolerance on each



Elongations when Clamped

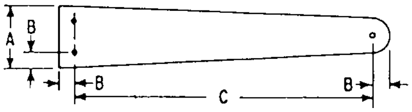
Zone	% Elongation
1	0
2	18.5
3	25.
4	12.
5	12.
6	25.
7	18.5
8	0

	A	B	C
mm	95	25	3.2
in.	3¾	1	0.125

FIG. 1 Cross-Sectional View of Mounted Looped Specimens Showing Elongation at Different Parts

---

[3] BHK Company part no. 87–0062–04 may be used.



	A	B	C
mm	25	6	125
in.	1	¼	5

FIG. 2 Die-Cut Tapered Specimen

pressure (see Note 3). Other partial pressures may, of course, 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 specimen. If specimens are loaded into a chamber that has been exhausted of ozone, the ozone concentration should reach ± 10 % of the selected ozone concentration within 15 min after the start of exposure.

NOTE 3—Test Methods D 4575 discusses methods and problems of measurement of ozone concentration. At 760 mm mercury atmospheric pressure (sea level) 1 mPa equals 0.99 parts per hundred million (pphm) ozone.

9.1.2 Since new specimens tend to absorb ozone rapidly, a drop in ozone concentration may be noted in chambers that are not exhausted when they are first loaded. Generation and control of ozone to the chamber also shall recover to ± 10 % of the selected partial pressure within 15 min. These criteria may limit the number of new samples loaded at one time for a given test chamber.

9.1.3 The standard test temperature shall be 40°C (104°F). Any other temperature may be chosen for the test and the associated conditioning. For certain types of protective films such as petroleum waxes, subambient temperature testing may be of significant importance.

9.2 Mount and condition the specimens.

9.2.1 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.[4] 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.

9.2.2 The foil or film covered sheets shall be stored for a minimum of 3 h at the agreed test temperature.

9.2.3 Strip off protective foil or film, then extend specimens *A* and *C* to the prescribed elongation and fasten them to holders inert to ozone (for example, metal or lacquered wood). Bend specimen *B* in the form of a loop and fasten it in a holder inert to ozone as shown in Fig. 1. (See Test Method D 518 for suitable wooden holders.) Initiate conditioning immediately after mounting specimens.

9.2.4 For normal testing, condition the mounted specimens for 24 h in an ozone-free atmosphere at the temperature at which they are to be exposed to ozone.

9.2.4.1 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.

9.2.5 For better correlation with some types of exposure, condition the specimens for 1 h in an ozone-free atmosphere at the temperature at which they are to be exposed.

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

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

9.2.6.1 This frequency will depend on the resistance to ozone attack of the rubbers being tested. Recommended observation magnification is 7×, except in the case of the triangular specimen of Test Method D 1171, where the magnification shall be 2×.

9.2.7 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.

10. Report

10.1 Report the following information:

10.1.1 Identification of materials tested,

10.1.2 Ozone partial pressure or concentration, in mPa, both nominal and that actually measured on a daily basis,

10.1.3 Temperature of test,

10.1.4 Condition period and temperature, in particular whether 24 h (see 9.2.4) or 1 h (see 9.2.5) was used,

10.1.5 Type of specimen used and elongation or mounting during a test, and

10.1.6 Time to the first observed cracking, or a description of the character of the ozone cracks at various periods of exposure.

11. Precision and Bias

11.1 Both the exposure rating and description of cracking are essentially rank order or qualitative comparison methods. Precision as normally expressed for quantitative measurement test methods is not directly applicable.

12. Keywords

12.1 chamber; deterioration; ozone; rubber; surface; test

---

[4] A-200 Mylar polyester film has been found suitable for this purpose.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).