Designation: D572 – 04 (Reapproved 2010)

Standard Test Method for Rubber—Deterioration by Heat and Oxygen[1]

This standard is issued under the fixed designation D572; 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 a procedure to determine the relative deterioration resistance of vulcanized rubber in a high temperature and high pressure oxygen environment. There may be no exact correlation between this accelerated test and natural aging of rubber because of the varied conditions of natural aging. This accelerated test is suitable for laboratory compound or product comparisons.

NOTE 1—For evaluating rubber vulcanizates under less severe conditions more nearly approaching natural aging, the use of Test Methods D573 and D865 is recommended.

1.2 The values stated in SI units are to be regarded as the standard. The values given 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 specific precautionary statement, see Note 2.)

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D15 Method of Compound and Sample Preparation for Physical Testing of Rubber Products (Withdrawn 1975)[3]

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D454 Test Method for Rubber Deterioration by Heat and Air Pressure

D573 Test Method for Rubber—Deterioration in an Air Oven

D865 Test Method for Rubber—Deterioration by Heating in Air (Test Tube Enclosure)

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Summary of Test Method

3.1 Specimens of vulcanized rubber are exposed to a deteriorating influence at a specified elevated temperature and oxygen pressure for known periods of time, after which their physical properties are determined. These are compared with the properties determined on the original specimens and the changes noted.

3.2 Unless otherwise specified, the determination of the physical properties shall be carried out in accordance with Test Methods D412.

3.3 Except as may be otherwise specified in this test method, the requirements of Practices D3182 and D3183 shall be complied with and are made part of this test method.

3.4 In case of conflict between the provisions of this test method and those of detailed specifications or test methods for a particular material, the latter shall take precedence.

4. Significance and Use

4.1 Rubber and rubber products must resist the deterioration of physical properties with time caused by oxidative and thermal aging. This test method provides a way to assess these performance characteristics of rubber, under certain accelerated conditions as specified.

4.2 Please refer to the Annex in Test Method D573 for important information on standard compounds used for precision testing for accelerated test aging evaluation.

5. Oxygen-Pressure Vessel

NOTE 2—**Caution:** Adequate safety provisions are important when heating oxidizable organic materials in oxygen since the rate of reaction may become very rapid in some cases, particularly if large surface area is

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

Current edition approved May 1, 2010. Published November 2010. Originally approved in 1940. Last previous edition approved in 2009 as D572 – 04 (2009). DOI: 10.1520/D0572-04R10.

[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] The last approved version of this historical standard is referenced on www.astm.org.

exposed, and very high pressures may be developed. If the same equipment is used for the oxygen-pressure test and the air-pressure heat test, Test Method D454, care must be exercised to see that the thermostatic controls are properly set, since the specimens may react very rapidly with oxygen at the temperature of the air-pressure heat test. Liquids acceptable as heating media for one test may be hazardous when used for the other test.

5.1 The oxygen-pressure chamber shall consist of a metal vessel designed to retain an internal atmosphere of oxygen gas under pressure, with provisions for placing rubber specimens within it and subjecting the whole to controlled uniform temperature. Because of the superior temperature control and heat transfer, metal vessels completely immersed in a liquid medium are recommended for purposes of referee tests. The apparatus shall conform to the following requirements:

5.1.1 The size of the chamber shall be optional but shall be such that the specimens may be suspended therein vertically without undue crowding and without touching each other or the sides of the chamber.

5.1.2 The source of heat is optional but shall be located outside of the aging chamber proper.

5.1.3 The heating medium is optional. Water, air, or other fluids known to be safe in the presence of oxygen may be used. Water has an advantage because of its rapid heat transfer and noncombustible nature. If air is used, the heated air shall be thoroughly circulated by means of mechanical agitation, and baffles shall be used as required to prevent local overheating and dead spots. Oils or other combustible fluids are extremely hazardous in the presence of oxygen and should not be used as heating media for this test.

5.1.4 Automatic temperature control of the heating medium by means of thermostatic regulation shall be used. The regulation system shall be provided with power failure protection and over-shoot protection to prevent accidental runaway temperature increase.

5.1.5 The temperature should be automatically recorded over the entire test period using a temperature-measuring device capable of measuring at the specific temperature to within ±1°C. For apparatus not equipped with automatic recording capabilities, temperature shall be measured with sufficient frequency to ascertain that the temperature limits specified in 10.2 are adhered to. If the pressure chamber is completely immersed, the temperature may be taken as that of the heating medium. The sensitive element of the temperature-measuring device shall be close to the pressure chamber but not touching it. If the pressure chamber is not completely immersed in the heating medium, the sensing element may be placed in a thermometer well extending into the pressure chamber. The thermometer well should be filled with water, or oil, to a depth sufficient to cover the element, in order to facilitate heat transfer. If it is confirmed by actual check that the temperature of the oxygen within the chamber is the same as that of the heating medium, the temperature may be taken in the heating medium instead of in the thermometer well. If air is used as the heating medium, a check of the oven temperature shall be made by means of temperature-indicating devices placed in various parts of the oven to verify the uniformity of heating. In any case, it is desirable to verify the recorded temperature by checking with a temperature-indicating device having its sensitive element directly exposed to the oxygen within the pressure chamber.

5.1.6 Positive, rapid, and complete circulation of the heating medium shall be maintained so as to assure accurate, uniform heating.

5.1.7 The pressure chamber shall be equipped with a reliable safety valve or rupture diaphragm set for release at 3450 kPa (500 psi) of pressure.

5.1.8 No copper or brass parts shall be exposed to the atmosphere, nor used in the pressure chamber and tubing or valves leading to it.

6. Sampling

6.1 The sample size shall be sufficient to allow for the determination of the original properties on three specimens and also on three or more specimens for each exposure period of the test. At least 24 h must elapse between completion of the vulcanization of the samples and the start of the aging test.

6.2 When minimum requirements are specified, one test on three dumbbells shall be considered sufficient. But if the results are below the specified requirements, two additional specimens shall be prepared from the original sample and tested. Should the results of either of these tests be below the specified requirements, the sample shall be considered to have failed to meet the specifications.

7. Test Specimens

7.1 Dumbbell-shaped specimens prepared as described in Test Methods D412 shall be considered standard. Their form shall be such that no mechanical, chemical, or heat treatment will be required after exposure in the pressure chamber. If any adjustments (for example, to thickness) are necessary, they should be performed prior to exposure.

7.2 The cross-sectional dimensions of test specimens for calculating the physical properties shall be measured prior to exposure in the aging chamber. Gage lines used for measuring elongations shall be applied after the specimens have been aged. Only specimens of similar dimensions having approximately the same exposed areas may be compared with each other.

8. Number of Test Specimens

8.1 At least three test specimens shall be used to determine the original physical properties of each sample and also three or more specimens of the same material for each exposure period of the test.

8.2 When minimum requirements are specified, one test shall be made for tensile strength and elongation. If the results are below the specified requirements, two additional specimens shall be prepared from the original sample and tested. Should the results of either of these tests be below the specified requirements, the samples shall be considered to have failed to meet the specifications.

9. Tests of Unaged Specimens

9.1 Determine the stress-strain properties or tensile strength and ultimate elongation and any other required properties of

the original unaged specimens within 96 h of the start of the aging period. Discard results on specimens that are found to be imperfect and retest.

9.2 When rubber compounds are to be tested for the purpose of determining compliance with specifications, it shall be permissible to determine the original properties required in 9.1 simultaneously with the determination of the values after the first aging period even though the elapsed time exceeds 96 h.

10. Procedure for Accelerated Aging

10.1 Place the specimens for aging in the aging chamber after it has been preheated to the operating temperature. It is recommended that not more than 10 % of the volume of the pressure chamber be occupied by rubber or oxidizable substance. Avoid simultaneous aging of a mixed group of different compounds if possible. For instance, high-sulfur should not be aged with lower-sulfur compounds, and those containing antioxidants should not be aged with those having no antioxidants. Some migration is known to occur. When starting a test, flush the air out of the oxygen-pressure chamber by releasing the oxygen pressure and refilling, and check the chamber to make certain the apparatus does not leak.

10.2 The operating temperature shall be 70 ± 1°C (158 ± 1.8°F) determined in accordance with 5.1.5.

10.3 The pressure of oxygen supplied to the aging chamber shall be 2100 ± 100 kPa (300 ± 15 psi) as measured by a calibrated pressure gage.

10.4 Start the aging interval at the time the specimens are placed in the heated chamber and continue for a measured time interval. The selection of suitable intervals of aging will depend on the rate of deterioration of the particular materials being tested. Time intervals frequently used are 24, 48, 72, and 96 h.

10.5 The aging intervals used shall be such that the deterioration will not be so great as to prevent determination of the final physical properties. In experimental work, it is desirable to use a range of periods, while for routine tests of known materials, fewer intervals may be employed.

10.6 At the termination of the aging interval, remove the specimens from the aging chamber, cool to room temperature on a flat surface, and allow to rest not less than 16 h nor more than 96 h before determination of the physical properties. In relieving the pressure from the oxygen-pressure chamber preparatory to removing the aged specimens, it is essential that the release be slow and uniform, requiring at least 5 min so as to avoid possible formation of porosity in the specimen. Then apply to the specimens gage lines used for measuring elongations:

NOTE 3—**Caution:** For the evaluation of rubber compounds intended for service at elevated temperatures, the above test methods may be used with an operating temperature of 80 ± 1°C (176 ± 1.8°F), employing time intervals as suggested in 10.4, or as otherwise agreed upon. It should be noted that by increasing the aging temperature to 80°C (176°F) from 70°C (158°F) the rate of oxidation may be expected to be approximately double, and if the rubber compound property change may be expected to be approximately double, and if the rubber compound is of a rapid aging type, or if it is contaminated with such materials as copper or manganese, the rate of oxidation may be catalyzed to such extent as to become violent.

11. Test of Aged Specimens

11.1 Determine the tensile strength and ultimate elongation or the stress-strain properties of the specimens, aged for different intervals, as the intervals terminate, disregarding the fact that more specimens may still be aging. In determining the physical properties after aging, consider as the final values the median of results from three specimens except that under the following conditions expose and test two additional specimens and use the median of the values for the five specimens:

11.1.1 If one or more values do not meet the specified requirements when testing for compliance with specifications, or

11.1.2 If referee tests are being made. After completion of the tests, examine the broken specimens visually and manually and note their condition.

12. Calculations

12.1 Express the results of the aging test as a percent of the change in each physical property (tensile strength, ultimate elongation, or tensile stress), calculated as follows:

$$P = [(A - O)/O] \times 100 \quad (1)$$

where:

P = percentage change in property,
O = original value, and
A = value after aging.

12.2 Increases will be indicated as positive and decreases as negative.

13. Report

13.1 The report shall include the following results calculated in accordance with Section 12:

13.1.1 All observed and recorded data on which the calculations are based,

13.1.2 Type of aging test,

13.1.3 Aging interval,

13.1.4 Aging temperature,

13.1.5 Duration, temperature, and date of vulcanization of the rubber, if known,

13.1.6 Dates of original and final determinations of physical properties, and

13.1.7 Dimensions of test specimens.

14. Precision and Bias[4]

14.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical calculation details.

14.2 A Type 2 (interlaboratory) precision was evaluated in 1974. Both repeatability and reproducibility are short term, a period of a few days separates replicate test results. A test result is expressed on the basis of a median value, as specified by Test Methods D412 obtained on three determinations or measurements of the property or parameter in question.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1055.

14.3 Six different materials were used in the interlaboratory program, and were tested in three laboratories on two different days. These precision results were obtained for a variety of compounds prepared in accordance with Methods D15 prior to its removal from the *Annual Book of ASTM Standards.* Please see the Annex of Test Method D573 for more details on this work.

14.4 The results of the precision calculations for repeatability and reproducibility for both percent tensile strength change and percent elongation change are given in Table 1, in ascending order of material average or level, for each of the materials evaluated.

TABLE 1 Type 2—Precision Results—Aging at 70°C

Part 1—Percent Change in Tensile Strength, Aged 48 h at 70°C

Material or Compound	Mean Test Level	Within Laboratories		Between Laboratories	
		Sr	*r*	*SR*	*R*
CR (2D)	–0.4	3.61	10.2	8.48	24.0
NR (1G)	–77.6	1.41	4.0	...	...
SBR (9B)	–3.0	2.39	6.76	5.96	16.9
OESBR (10B3)	–2.8	7.20	20.4	10.1	28.6
IIR (2E)	–5.9	4.85	13.7	6.8	19.2
NBR (1F)	–5.6	15.7	44.4	15.1	42.7
Pooled Values:	...	7.55	21.4	9.83	27.8

Part 2—Percent Change in Elongation, Average of 48, 96 h Aging Precision Values

NR (1G)	–92.0	0.99	2.80	9.6	27.2
NBR (1F)	–15.5	9.4	26.6	9.4	26.6
SBR (9B)	–13.8	3.53	10.0	7.58	21.5
OESBR (10B3)	–7.8	5.54	15.7	7.54	21.3
CR (2D)	–0.0	5.32	15.1	7.59	21.5
IIR (2E)	–4.6	5.50	15.6	5.50	15.6
Pooled Values:	...	5.64	16.0	8.00	22.6

NOTE 1—
Sr = within laboratory standard deviation.
r = repeatability (in measurement units).
SR = between laboratory standard deviation.
R = reproducibility (in measurement units).

NOTE 2—Averaging both 48 and 96 h of aging for Part 2 increases the DF for precision estimates.

14.4.1 The precision of this test method may be expressed in the format of the following statements that use an appropriate value of *r*, *R*, (*r*), or (*R*), that is, that value to be used in decisions about test results (obtained with the test method). The *appropriate value* is that value of *r* or *R* associated with a mean level in the precision tables closest to the mean level under consideration at any given time, for any given material in routine testing operations.

14.5 *Repeatability*—The repeatability, *r*, of this test method has been established as the *appropriate value* tabulated in the precision tables. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or non-identical sample populations.

14.6 *Reproducibility*—The reproducibility, *R*, of this test method has been established as the *appropriate value* tabulated in the precision tables. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or non-identical sample populations.

14.7 The precision results indicate that the repeatability and reproducibility of both percent tensile strength change and percent elongation change are essentially the same. Also the value of *r* or *R*, or both, does not vary with the magnitude of percent elongation or percent tensile strength change. No values are given for (*r*) or (*R*) because of the near zero average values for some of the materials.

14.8 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias, therefore, cannot be determined.

15. Keywords

15.1 elevated temperature; oxidative aging; pressure chamber; pressure vessel; rubber articles; rubber products; thermal aging