Designation: D454 – 04 (Reapproved 2015)

Standard Test Method for Rubber Deterioration by Heat and Air Pressure[1]

This standard is issued under the fixed designation D454; 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 U.S. Department of Defense.

1. Scope

1.1 This test method covers a procedure to determine the influence of elevated temperature and air pressure on the physical properties of vulcanized rubber. The results of this test may not give an exact correlation with service performance since performance conditions vary widely. The test may, however, be used to evaluate rubber compounds on a laboratory comparison basis. It will be most applicable to performance under conditions of increased temperature and air pressure.

NOTE 1—For evaluating rubber vulcanizates under less severe conditions that more nearly approach 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.*

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D15 Method of Compound and Sample Preparation for Physical Testing of Rubber Products (Withdrawn 1975)[3]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

D572 Test Method for Rubber—Deterioration by Heat and Oxygen

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 the deteriorating influence of air at specified elevated temperature and 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 allows these performance properties to be determined under the accelerated conditions of high air pressure and at elevated temperatures.

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

5. Apparatus

5.1 *Air-Pressure Chamber,* consisting of a metal vessel designed to maintain an internal atmosphere of air under

[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 July 1, 2015. Published October 2015. Originally approved in 1952. Last previous edition approved in 2010 as D454 – 04 (2010). DOI: 10.1520/D0454-04R15.

[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.

known pressure, with provisions for placing rubber specimens within it and subjecting them to controlled uniform temperature. The equipment shall conform to the following requirements:

5.1.1 The size and shape 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 operating temperature shall be 125 ± 1°C (257 ± 1.8°F) determined as described in 5.1.5. The temperature shall be automatically controlled by means of thermostatic regulation.

5.1.3 The source of heat is optional, but if located inside the aging chamber, shielding shall be provided so that direct radiation cannot reach the specimens. The temperature of the shield surfaces shall be within 1°C of the air temperature.

5.1.4 The heating medium is optional. Steam, air, or liquid media may be used. 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 organic fluids may be hazardous at the elevated temperature required, but if their use is necessary, they must have a flash point not lower than 200°C. For any one type of heat-transfer medium, complete immersion of the pressure vessel in the heating medium is recommended for referee purposes in order to assure uniformity of temperature inside the vessel.

5.1.5 To make certain that the operating temperature remains within the limits specified in 5.1.2, the temperature should be automatically recorded over the entire test period using a temperature measuring device capable of measurement within 1°C of the specified temperature. For apparatus not equipped with automatic recording capabilities, temperature should be measured with sufficient frequency to ascertain that the temperature limits specified in 5.1.2 are adhered to. If the pressure chamber is not completely immersed, the sensing element shall be placed in a thermometer well extending into the pressure chamber. The thermometer well should be filled with a nonvolatile liquid to a depth sufficient to cover the sensitive element, in order to facilitate heat transfer. In any case, it is desirable to verify the recorded temperature, and the uniformity of temperature distribution at different points within the pressure chamber, by checking with a temperature-indicating device having its sensitive element directly exposed to the air within the pressure chamber. 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.

5.1.6 The apparatus and method of heating shall be so designed that the interval required for the chamber to reach the operating temperature at the beginning of a test shall be as short as possible. By proper precautions, this lag may be reduced to less than 5 % of the usual minimum exposure periods. Provision shall also be made for rapid closing and opening of the apparatus for introduction or removal of specimens.

5.1.7 The air pressure shall be maintained at 550 ± 14 kPa (80 ± 2.0 psi) during the exposure periods. Automatic regulation is recommended.

5.1.8 Suitable provision shall be made by separation, filtration, or otherwise for removal of oil, dirt, and moisture from the air entering the pressure chamber. Care shall also be taken to avoid any other introduction of oil or grease into the pressure chamber.

5.1.9 No copper or brass parts shall be exposed to the atmosphere used in the pressure chamber.

5.1.10 The pressure chamber shall be equipped with a reliable safety valve or rupture diaphragm set for release at a pressure of not more than 1380 kPa (200 psi).

NOTE 2—**Caution:** Adequate safety provisions are important when heating oxidizable organic materials in air under pressure, since the rate of oxidation may become very rapid in some cases, particularly if a large surface area is exposed. If the same equipment is used for the air-pressure heat test and the oxygen-pressure test in accordance with Test Method D572, combustible heating media should not be used.

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.

6.2.1 The two additional specimens indicated in 6.2 may optionally be prepared and exposed simultaneously with the first three specimens exposed. They need not be tested if the median values of the first three specimens exposed and tested meet the minimum requirements. Testing five specimens is the norm for referee tests in accordance with 10.1.2.

7. Test Specimens

7.1 Dumbbell-shaped specimens prepared in accordance with 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, that is, to thickness are necessary, they shall 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. Tests of Original Specimens

8.1 The stress-strain properties or tensile strength and ultimate elongation, and any other required properties of the

original unaged specimens shall be determined within 96 h of the start of the aging period. Results on specimens that are found to be imperfect shall be discarded, and retests shall be made.

8.2 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 8.1 simultaneously with the determination of the values after the first aging period, even though the elapsed time exceeds 96 h.

9. Procedure

9.1 Suspend the specimens for exposure vertically in the pressure 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 an oxidizable substance. Avoid simultaneous exposure of a mixed group of different compounds, if possible. For instance, high-sulfur compounds should not be exposed with low-sulfur compounds, nor those containing antioxidants with those that do not, since some migration is known to occur under the conditions of the test.

9.2 Consider the exposure period to start when the specimens are placed in the heated chamber, after which close the chamber immediately and apply air pressure. This entire operation shall not take longer than 3 min. However, keep a record of the time interval elapsing from the starting time until the temperature of the chamber reaches 125°C (257°F). If this interval exceeds 5 % of the total exposure time, make approximate correction by adding one half of the interval to the exposure period. The exposure shall be continuous for the specified time without pressure reduction or opening of the chamber for introduction or removal of specimens.

9.3 Select suitable periods of exposure depending on the rate of deterioration of the particular material being tested. The periods shall be such that the deterioration will not be so great as to prevent determination of the final physical properties. Intervals frequently used are 3, 5, 8, 12, 20, and 30 h. In experimental work, it is desirable to employ a range of periods so as to determine the rate of deterioration, but for routine tests of known materials and for purchase acceptance purposes, fewer intervals or even a single period may suffice.

9.4 At the termination of the exposure period, release the air pressure gradually, this operation requiring at least 5 min to avoid possible formation of porosity in the specimens. Remove specimens from the pressure chamber immediately. Cool the specimens to room temperature on a flat surface and allow to rest not less than 16 nor more than 96 h before determination of the physical properties. Apply gage lines used for measuring elongations to the specimens.

10. Physical Tests of Exposed Specimens

10.1 Determine the tensile stress at a specified elongation, or tensile strength and ultimate elongation, or both, of the specimens exposed for different periods as the intervals terminate, except that it shall be permissible to accumulate specimens for testing together when this does not conflict with the specified rest period. In determining the physical properties after aging, the final values shall be the median of results from three specimens, except that under the following conditions, two additional specimens shall be exposed and tested and the median of the values for the five specimens shall be used:

10.1.1 If one or more values do not meet the specified requirements when testing the compliance with specifications.

10.1.2 If referee tests are being made. After completion of the tests, the broken specimens shall be examined visually and manually and their condition noted.

11. Calculations

11.1 Express the results of the air pressure heat test for each exposure period as a percentage change in each physical property (tensile strength, ultimate elongation, or tensile stress) calculated as follows:

$$P = \frac{A - O}{O} \times 100 \quad (1)$$

where:

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

In this type of expression, negative percent values indicate a reduction in the value of the property.

12. Report

12.1 The report shall include the following results calculated in accordance with Section 10.

12.1.1 All observed are recorded data on which the calculations are based,

12.1.2 Description of the apparatus,

12.1.3 The exposure period,

12.1.4 Statement of condition of exposed specimens,

12.1.5 Dimensions of test specimens,

12.1.6 The duration, temperature, and date of vulcanization of the rubber, if known, and

12.1.7 Dates of original and final determination of physical properties.

13. Precision and Bias[4]

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

13.2 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.

13.3 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

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D11-1054.

its removal from the *Annual Book of ASTM Standards.* Please see the Annex of Test Method D573 for more details on this work.

13.4 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.

13.4.1 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.

TABLE 1 Type 2—Precision Results: Aging at 125°C

Material or Compound	Mean Test Level	Within Laboratories		Between Laboratories	
		Sr	*r*	*SR*	*R*
Part 1—Percent Change in Tensile Strength, Average of 8, 30 h Aging					
NR (1G)	−86.5	6.56	18.6	11.1	31.4
OESBR (10B3)	−34.0	4.60	13.0	11.5	32.6
SBR (9B)	−32.3	3.96	11.2	14.2	40.1
NBR (1F)	−27.3	4.23	12.0	17.4	49.2
IIR (2E)	−18.3	3.17	8.96	26.0	73.6
CR (2D)	−16.9	4.33	12.3	11.4	32.2
Pooled Values:	...	4.48	12.7	15.3	43.2
Part 2—Percent Change in Elongation, Average of 8, 30 h Aging					
NR (1G)	−74.9	2.0	5.8	18.9	53.5
SBR (9B)	−54.4	0.92	2.6	11.6	32.8
NBR (1F)	−52.2	2.55	7.2	24.1	68.2
OESBR (10B3)	−51.1	1.30	3.7	8.94	25.3
CR (2D)	−9.0	1.85	5.2	13.7	38.8
IIR (2E)	−6.7	4.72	13.4	15.3	43.3
Pooled Values:	...	2.22	6.3	15.4	43.7

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

NOTE 2—Averaging the 8 and 30 h aging results gives increased DF estimates of precision.

13.5 *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.

13.6 *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.

13.7 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.

13.8 *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.

14. Keywords

14.1 elevated temperature; pressure chamber; pressure vessel; rubber articles; rubber products