Designation: D454 – 04 (Reapproved 2019)

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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D15 Method of Compound and Sample Preparation for Physical Testing of Rubber Products (Withdrawn 1975)[3]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

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials 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] The last approved version of this historical standard is referenced on www.astm.org.

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 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.