



Designation: D865 – 99 (Reapproved 2005)

## Standard Test Method for Rubber—Deterioration by Heating in Air (Test Tube Enclosure)<sup>1</sup>

This standard is issued under the fixed designation D865; 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 deterioration induced by heating rubber specimens in individual test tube enclosures with circulating air. This isolation prevents cross contamination of compounds due to loss of volatile materials (for example, antioxidants) and their subsequent migration into other rubber compounds (specimens). The absorption of such volatile materials may influence the degradation rate of rubber compounds.

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:<sup>2</sup>

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D573 Test Method for Rubber—Deterioration in an Air Oven

D1349 Practice for Rubber—Standard Temperatures for Testing

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

<sup>1</sup> 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.

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<sup>2</sup> 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.

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

### 3. Summary of Test Methods

3.1 Specimens of vulcanized rubber are exposed to the deteriorating influence of air at specified elevated temperature 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. With this test method, these characteristics may be determined in a way that is free of some of the complications inherent in community-type aging devices, that is where numerous compounds (specimens) are aged in the same enclosure.

4.2 The isolation of compound (specimens) by the use of individual circulating air test tube enclosures prevents any cross contamination from volatile products and permits a more representative assessment of aging performance.

4.3 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. Apparatus

5.1 The apparatus for the heat aging shall consist of borosilicate glass test tubes 38 mm (1.52 in.) in diameter and 300 mm (12 in.) in length heated uniformly in suitable equipment

such as an oil or water bath or metal block so arranged as to comply with the following requirements:

5.1.1 No more than three specimens shall be used in each test tube, and all specimens in a single tube shall be from a single compound.

5.1.2 Provision shall be made for vertical suspension of the specimens as near the bottom of the test tube as possible without touching each other or the sides of the test tube during the test. A suggested means for holding the specimens is shown in Fig. 1.

5.1.3 The test tubes shall be provided with a tight-fitting cork or nonreactive composition stopper through which an inlet and an outlet tube shall be placed as shown in Fig. 1.

5.1.4 The heating equipment shall be provided with a thermostatic control that will maintain the temperature at the location of the specimens in the test tube within 1°C (1.8°F) of the prescribed temperature. If a liquid medium is used, suitable stirring or circulation shall be provided to assure uniformity of temperature.

5.1.5 The temperature should be automatically recorded over the entire test period using a temperature-measuring device capable of measuring at the specified temperature within  $\pm 1^\circ\text{C}$ . For apparatus not equipped with automatic recording capabilities, temperature shall be measured with sufficient frequency to ascertain that the temperature limits specified in 10.1 are adhered to. A check of the actual

temperature within the test tube should be made by placing a thermometer in the tube with the bulb in the location ordinarily occupied by the specimens and the stem extending through a third hole drilled in the cork stopper.

5.1.6 The lip of the test tube shall be within 50 mm (2 in.) of the heating medium during the test.

5.1.7 When an oil bath is used, means shall be provided to remove oil fumes from the oil bath so that they will not enter the inlet tube.

## 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 Prepare dumbbell-shaped specimens as described in the Test Methods D412. These 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. Make any adjustments that are necessary, for example, to thickness, prior to exposure.

7.2 Measure the cross-sectional dimensions of test specimens for calculating the physical properties prior to exposure in the aging chamber. Apply gage lines used for measuring elongations 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 Use at least three test specimens 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, make one test for tensile strength and elongation. If the results are below the specified requirements, prepare two additional specimens from the original sample and test. If the results of either of these tests are 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 a specification, it shall be permissible to determine the original properties required in 1

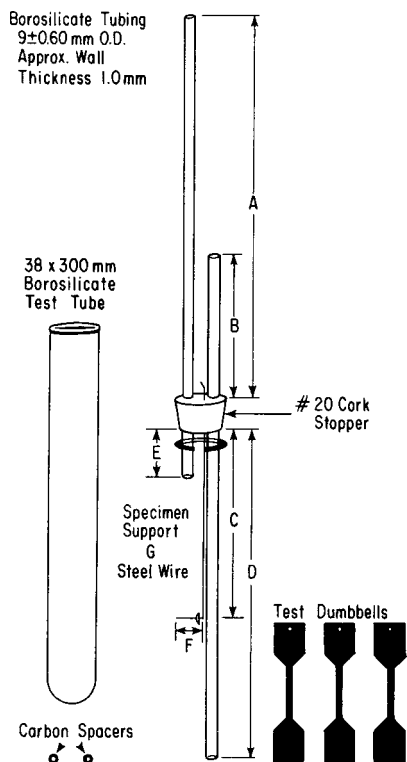


FIG. 1 Arrangement of Vent Tubes and Test Specimens