

Designation: D865 - 11 (Reapproved 2018)

Standard Test Method for Rubber—Deterioration by Heating in Air (Test Tube Enclosure)¹

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 U.S. 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, 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

- 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 Conditions for Testing

- D2240 Test Method for Rubber Property—Durometer Hardness
- 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 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 unexposed 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 in 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 or if otherwise agreed upon between customer and supplier or between laboratories.

4. Significance and Use

4.1 Rubber and rubber products must resist the deterioration of physical properties with time caused by oxidative and thermal ageing. This test method determines these characteristics in a way that is free of some of the complications inherent in community-type ageing devices, that is where numerous compounds (specimens) are aged in the same enclosure.

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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.

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

5. Apparatus

5.1 The apparatus for heat ageing 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 (see Fig. 1) so arranged as to comply with the following requirements:

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

5.1.2 Provisions 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. 2.

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. 2.

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 $\pm 1^{\circ}$ C



FIG. 1 A Typical Metal Block Type Ageing Oven



FIG. 2 Arrangement of Vent Tubes and Test Specimens

 $(\pm 1.8^{\circ}\text{F})$ 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 $\pm 1^{\circ}$ C ($\pm 1.8^{\circ}$ F). Apparatus that is not equipped with automatic recording capabilities, the temperature shall be measured with sufficient frequency to assure compliance with the temperature limits specified in 10.1.

5.1.6 Prior to commencing a test, or series of tests, a check of the actual temperature within the test tube shall be made by placing a thermometer in the tube with the bulb in the location ordinarily occupied by the specimens. Alternatively, a digital temperature measuring device may be used in place of a liquid-in-glass thermometer, wherein the probe is placed in a position ordinarily occupied by the specimens. The thermometer or probe shall be inserted through either the tubes or through a third hole in the stopper which shall be securely closed during the test.

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

5.1.8 When an oil bath is used, a means shall be provided to remove oil fumes from the oil bath, typically using a reflux condenser, so that they will not enter the inlet tube.