



# 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 ( $\epsilon$ ) 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 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 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.

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.

<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](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

**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.

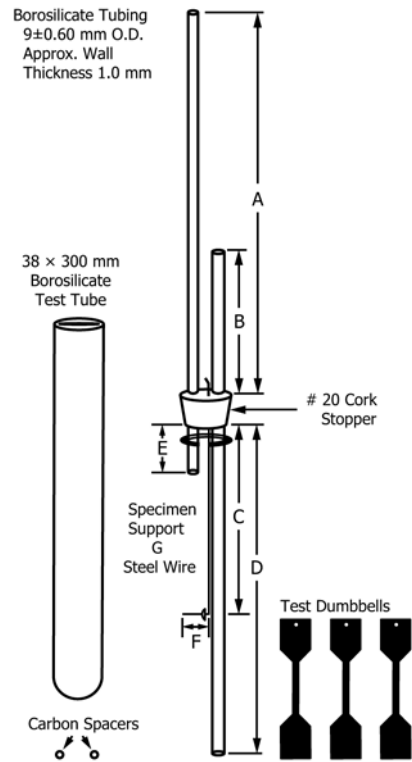
5.1.3 The test tubes shall be provided with a tight-fitting cork or nonreactive composition stopper through which an inlet and an outlet tube shall be placed as shown in Fig. 2.

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  $\pm 1^{\circ}\text{C}$  ( $\pm 1.8^{\circ}\text{F}$ ) 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  $\pm 1^{\circ}\text{C}$  ( $\pm 1.8^{\circ}\text{F}$ ). Apparatus that is not equipped with automatic recording capabilities, the temperature shall be



**FIG. 1 A Typical Metal Block Type Ageing Oven**



	A	B	C	D	E	F	G
mm	300	115	150	265	38	20	1.5
in.	12	4½	6	10½	1½	¾	⅙

**FIG. 2 Arrangement of Vent Tubes and Test Specimens**

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.

**6. Sampling**

6.1 The sample size shall be sufficient to allow for the determination of the original properties on three unexposed 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 ageing test.

6.2 When minimum requirements are specified, one test on three dumbbells shall be considered sufficient. 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 in 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. 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 ageing 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 unexposed 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, elongation, and durometer hardness. 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 Unexposed Specimens

9.1 Determine the stress-strain properties or tensile strength, ultimate elongation, durometer hardness (Test Method **D2240**), and any other desired properties of the original unexposed specimens within 96 h of the start of the ageing period. Disregard the results of specimens that are found to be unsatisfactory or statistical outliers 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 **9.1** simultaneously with the determination of the values after the first ageing period even though the elapsed time exceeds 96 h.

## 10. Procedure

10.1 Bring the heating medium to the temperature selected for the ageing test. When the temperature-measuring device indicates that this temperature is being maintained, immerse the test tube assembly containing the specimens in the fluid bath or metal block, as specified in **5.1.6** and **5.1.7**, and allow to heat for the desired period.

10.1.1 The selected temperature may be in the range from 70 to 250°C (158 to 482°F), and it is suggested that one of the standard temperatures shown in Practice **D1349** be used.

10.2 Start the ageing period at the time the specimens are placed in the heating medium. The selection of suitable periods of ageing will depend on the rate of deterioration of the particular material being tested.

10.2.1 Periods frequently used are 10, 22, 48, 70, and 168 h. Use ageing periods 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 ageing periods, while for routine tests of known materials fewer intervals may be employed.

10.3 At the termination of the ageing interval, remove the specimens from the test tubes, cool to the standard laboratory temperature on a flat, thermally non-conductive surface, and allow to equilibrate 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.

10.4 Clean the test tube and the circulation tubes carefully following each test or prior to each new test to remove any deposits of volatile material that may have condensed on the sides of the test tube or in the circulation tubes.

## 11. Physical Tests of Aged Specimens

11.1 Determine the stress-strain properties or tensile strength and ultimate elongation and other desired properties such as durometer hardness of the specimens aged for different periods as the intervals terminate in the progress of ageing, disregarding the fact that more specimens may still be ageing. In determining the physical properties after ageing, 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:

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

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

## 12. Calculations

12.1 Express the results of the ageing test (for each ageing period) as a percent change in each physical property (tensile strength, ultimate elongation, tensile stress, durometer hardness, or others), calculated as follows:

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

where:

$P$  = change in property, %,

$O$  = original value, and

$A$  = value after ageing.

## 13. Report

13.1 The report shall include the results calculated in accordance with **12.1** as follows:

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

13.1.2 Type of ageing test,

13.1.3 Ageing interval,

- 13.1.4 Ageing 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,
- 13.1.7 Dimensions of test specimens, and
- 13.1.8 Methods of determining hardness.

#### 14. Precision and Bias<sup>3</sup>

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 3 determinations or measurements of the property or parameter in question.

14.3 Six different materials were used in the interlaboratory program, these were tested in 3 laboratories on 2 different days. These precision results were obtained for a variety of compounds prepared in accordance with Method D15 prior to its removal from the *Annual Book of ASTM Standards*. Please see 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.

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

**TABLE 1 Precision Results—Ageing at 100°C**

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 both 48 and 96 h of ageing increases the DF for precision estimates.

Material or Compound	Mean Test Level	Part 1—Percent Change in Tensile Strength			
		Within Laboratories		Between Laboratories	
		$Sr$	$r$	$SR$	$R$
NR (1G)	−60.1	3.56	10.1	9.91	28.1
OESBR (10B3)	−16.0	5.19	14.7	5.08	14.4
SBR (9B)	−12.5	7.14	20.2	7.69	21.8
NBR (1F)	−8.3	8.30	23.5	8.92	25.2
CR (2D)	−4.5	3.13	8.9	4.84	13.7
IIR (2E)	0.53	2.05	5.8	2.08	5.9
Pooled Values	...	5.38	15.2	6.96	19.7

Material or Compound	Mean Test Level	Part 2—Percent Change in Elongation, Average of 48, 96 h Ageings			
		Within Laboratories		Between Laboratories	
		$Sr$	$r$	$SR$	$R$
NR (1G)	−65.0	2.95	8.35	4.82	13.64
SBR (9B)	−48.7	4.22	11.94	5.20	14.70
OESBR (10B3)	−42.4	3.12	8.83	3.23	9.14
NBR (1F)	−36.2	6.28	17.8	6.28	17.8
CR (2D)	−7.7	2.95	8.35	3.73	10.56
IIR (2E)	−7.6	2.81	7.95	4.77	13.50
Pooled Values	...	3.72	10.53	4.67	13.22

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 accelerated ageing; elevated temperature; heat ageing; oxidative ageing; rubber articles; rubber products; thermal ageing

<sup>3</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1057.

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