



Designation: D572 – 23

Standard Test Method for Rubber—Deterioration by Heat and Oxygen¹

This standard is issued under the fixed designation D572; 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 relative deterioration resistance of vulcanized rubber in a high temperature and high pressure oxygen environment. There may be no exact correlation between this accelerated test and natural aging of rubber because of the varied conditions of natural aging. This accelerated test is suitable for laboratory compound or product comparisons.

NOTE 1—For evaluating rubber vulcanizates under less severe conditions more nearly approaching natural aging, the use of Test Methods **D573** and **D865** is recommended. For evaluating the aging characteristics of rubber vulcanizates used in oxygen enriched atmospheres under conditions simulating natural and accelerated aging at ambient pressure, the use of Practices **G114** 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.* (For specific precautionary statement, see **Note 2**.)

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

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.

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

D454 Test Method for Rubber Deterioration by Heat and Air Pressure

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

G63 Guide for Evaluating Nonmetallic Materials for Oxygen Service

G94 Guide for Evaluating Metals for Oxygen Service

G114 Practices for Evaluating the Age Resistance of Polymeric Materials Used in Oxygen Service

2.2 *United States Pharmacopeial Convention Standard:*⁴

USP 24-NF 19 Oxygen monograph

3. Summary of Test Method

3.1 Specimens of vulcanized rubber are exposed to a deteriorating influence at a specified elevated temperature and oxygen 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 The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from U.S. Pharmacopeia (USP), 12601 Twinbrook Pkwy., Rockville, MD 20852.

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 provides a way to assess these performance characteristics of rubber, under certain accelerated conditions as specified.

4.2 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. Oxygen Pressure Vessel

NOTE 2—Caution: Adequate safety provisions are important when heating oxidizable organic materials in oxygen since the rate of reaction may become very rapid in some cases, particularly if large surface area is exposed, and very high pressures may be developed. If the same equipment is used for the oxygen-pressure test and the air-pressure heat test, Test Method **D454**, care must be exercised to see that the thermostatic controls are properly set, since the specimens may react very rapidly with oxygen at the temperature of the air-pressure heat test. Liquids acceptable as heating media for one test may be hazardous when used for the other test.

5.1 The oxygen-pressure chamber shall consist of a metal vessel designed to retain an internal atmosphere of oxygen gas under pressure, with provisions for placing rubber specimens within it and subjecting the whole to controlled uniform temperature. Because of the superior temperature control and heat transfer, metal vessels completely immersed in a liquid medium are recommended for purposes of referee tests. The apparatus shall conform to the following requirements:

5.1.1 The size 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 source of heat is optional but shall be located outside of the aging chamber proper.

5.1.3 The heating medium is optional. Water, air, or other fluids known to be safe in the presence of oxygen may be used. Water has an advantage because of its rapid heat transfer and noncombustible nature. 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 fluids are extremely hazardous in the presence of oxygen and should not be used as heating media for this test.

5.1.4 Automatic temperature control of the heating medium by means of thermostatic regulation shall be used. The regulation system shall be provided with power failure protection and over-shoot protection to prevent accidental runaway temperature increase.

5.1.5 The temperature should be automatically recorded over the entire test period using a temperature-measuring device capable of measuring at the specific temperature to 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 **11.2** are adhered to. 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. If the pressure chamber is not completely immersed in the heating medium, the sensing element may be placed in a thermometer well extending into the pressure chamber. The thermometer well should be filled with water, or oil, to a depth sufficient to cover the element, in order to facilitate heat transfer. If it is confirmed by actual check that the temperature of the oxygen within the chamber is the same as that of the heating medium, the temperature may be taken in the heating medium instead of in the thermometer well. If air is used as the heating medium, a check of the oven temperature shall be made by means of temperature-indicating devices placed in various parts of the oven to verify the uniformity of heating. In any case, it is desirable to verify the recorded temperature by checking with a temperature-indicating device having its sensitive element directly exposed to the oxygen within the pressure chamber.

5.1.6 Positive, rapid, and complete circulation of the heating medium shall be maintained so as to assure accurate, uniform heating.

5.1.7 The pressure chamber shall be equipped with a reliable safety valve or rupture diaphragm set for release at 3450 kPa (500 psi) of pressure.

NOTE 3—Caution: Per Guides **G63** and **G94**, oxygen pressure effects on nonmetals (for example, rubbers) and metals can range from relatively mild below 70 kPa (10 psi) to extremely severe above 20 000 kPa (3000 psi). Furthermore, per Guide **G63**, when evaluating polymeric materials such as rubbers, any mixture with oxygen exceeding atmospheric concentration at pressures higher than 101 kPa (14.7 psi) should be evaluated from the hazard point of view for possible significant increase in material combustibility. Conversely, as oxygen concentration decreases from 100 %, the likelihood of rapid or catastrophic deterioration of rubber test materials and accompanying adverse effects on metallic pressure vessel materials of construction will diminish.

5.1.8 No parts made from copper or copper-containing alloys such as brass shall be exposed to the atmosphere, nor used in the pressure chamber and tubing or valves leading to it.

NOTE 4—Copper has been reported to accelerate aging of natural rubber (NR).⁵ In addition, NR has been reported to react with copper to form cuprous sulfide, which may lead to depolymerization.⁶ For polymers in general, including rubbers covered by this Test Method, trace quantities of transition metals such as Co, Cu, Fe, and Mn have been reported to accelerate oxidation because they can be potent catalysts for hydroperoxide decomposition and, thus, greatly reduce the activation energy for oxidative aging or deterioration.⁷

⁵ Cassagne, P., "The Determination of Copper in Crude Rubber," *Rubber Chemistry and Technology*, Vol. 20, No. 1, 1947, pp. 308–312. <https://meridian.allenpress.com/rct/article-abstract/20/1/308/88046/The-Determination-of-Copper-in-Crude-Rubber?redirectedFrom=fulltext>.

⁶ Buchan, S. and Rae, W. D., "Chemical Nature of the Rubber-To-Brass Bond," *Rubber Chemistry and Technology*, Vol. 19, No. 4, 1946, pp. 968–986. <https://meridian.allenpress.com/rct/article-abstract/19/4/968/87489/Chemical-Nature-of-the-Rubber-To-BrassBond?redirectedFrom=fulltext>.

⁷ Grassie, N. and Scott, G., *Polymer Degradation and Stabilisation*, Cambridge University Press, Cambridge, 1988. <http://polymerdatabase.com/polymer%20chemistry/Autooxidation.html>.