



Designation: D4004 – 06 (Reapproved 2017)

Standard Test Methods for Rubber—Determination of Metal Content by Flame Atomic Absorption (AAS) Analysis¹

This standard is issued under the fixed designation D4004; 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 These test methods cover the determination of lead, zinc, copper, and manganese in raw rubber and rubber compounds, vulcanized or unvulcanized. The level at which the metals are present is taken into account by suitable adjustments of sample mass and dilution.

1.2 Certain compounding ingredients, present in the rubber sample will dictate which of the methods should be used. Refer to Section 3. Five methods of determination are as follows:

Method	Sections
Method A	8
Method B	9
Method C	10
Method D	14 – 22
Method E	23 – 31

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.*

1.5 *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 These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and are the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

Current edition approved May 1, 2017. Published May 2017. Originally approved in 1981. Last previous edition approved in 2012 as D4004 – 06 (2012). DOI: 10.1520/D4004-06R17.

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.

[D1076 Specification for Rubber—Concentrated, Ammonia Preserved, Creamed, and Centrifuged Natural Latex](#)

[D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries](#)

[E663 Practice for Flame Atomic Absorption Analysis \(Withdrawn 1997\)³](#)

2.2 ISO Standards:

[ISO 1396 Rubber—Copper Content—Photometric Technique—Determination⁴](#)

[ISO 1655 Rubber—Manganese Content—Photometric Technique—Determination⁴](#)

3. Summary of Test Methods

3.1 *Method A*—Determination of lead and zinc in rubber not containing any halogen; in the case of lead, also not containing any silica filler. See Section 8.

3.1.1 In Method A, the rubber is furnace-dried in a platinum crucible at 250°C, followed by furnace ashing for 1 to 2 h at 550°C. The ash is dissolved with the aid of concentrated hydrochloric acid (HCl) and the resulting solution suitably diluted for Atomic Absorption Spectrometric (AAS) determination of the lead and zinc.

3.2 *Method B*—Determination of lead and zinc in rubber containing silica filler, but no halogen. See Section 9.

3.2.1 In Method B the rubber is ashed in platinum as in Method A. The ash is then fused with a lithium or sodium tetraborate or metaborate flux, after which the fused mixture is dissolved with the aid of HCl for subsequent AAS analysis.

3.3 *Method C*—Determination of lead and zinc in rubber containing halogen. See Section 10.

3.3.1 In Method C the rubber is wet-ashed with the aid of concentrated sulfuric acid (H₂SO₄) and nitric acid (HNO₃), evaporated to dryness, and further ashed in a muffle furnace at 550°C, after which the ash is dissolved as in Method A for subsequent AAS analysis.

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.