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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Rubber Chemicals—Determination of Ash Content¹

This standard is issued under the fixed designation D 4574; 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.

1. Scope

1.1 These test methods describe the determination of ash content of rubber chemicals.

1.2 The test methods include the following materials:

Material	Section
Sulfur	7-13
<i>p</i> -Phenylenediamine Antioxidants	14-22
Benzothiazole Sulfenamide Accelerators	14-22

1.3 The values stated in SI units are to be regarded as the 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²

D 4676 Classification for Rubber Compounding Materials—Antidegradants²

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 “*lot*” sample—a production sample representative of a standard production unit, normally referred to as “the sample.”

3.1.2 *test unit*—the actual material used in the analysis. It must be representative of the “lot” sample.

4. Summary of Test Methods

4.1 The ash content is determined by heating a known quantity of the rubber chemical on a hot plate or over a gas burner to volatilize the sample and then heating in a muffle furnace to complete the ashing process.

4.2 Sample preparation, procedures, calculations, and precision statements will be found in each section dealing with a particular rubber chemical.

5. Significance and Use

5.1 These test methods are suitable for the determination of the ash content of rubber chemicals. The test methods may be used for quality control, product acceptance, or research and development. Classification D 4676 prescribes percent ash as an important characteristic of rubber antidegradants.

6. Apparatus

6.1 *Muffle Furnace*, capable of temperature regulation of $\pm 25^{\circ}\text{C}$ between 500 and 800°C.

6.2 *Hot Plate* (or laboratory gas burner).

6.3 *Laboratory Fume Hood*.

6.4 *Porcelain Combustion Crucible*, capsule form, 25-cm³ capacity.

6.5 *Porcelain Crucible*, high form, size 0, 15-cm³ capacity.

6.6 *Clay Triangle*.

6.7 *Steel Crucible Tongs*.

6.8 *Heat Resistant Gloves*.

6.9 *Desiccator*.

6.10 *Analytical Balance*, sensitive to 0.0001 g.

6.11 *Air Circulating Oven*, capable of $70 \pm 2^{\circ}\text{C}$.

SULFUR

7. Scope

7.1 This test method is used for the determination of the ash content of sulfur.

8. Summary of Test Method

8.1 The ash content of sulfur is determined by the controlled burning of the sulfur, then ashing of the specimen in a furnace at 600°C.

9. Apparatus

9.1 See Section 6.

10. Procedure

10.1 Dry 6 g or more of sulfur in an oven at 70°C for 2 h. Cool in a desiccator.

10.2 Weigh a 5-g specimen to the nearest 0.0001 g into a previously ignited, weighed, 25-cm³ porcelain crucible. In a well-ventilated hood, place the crucible on a hot plate and heat to 400 to 500°C, burning off all of the sulfur; or burn off the sulfur by moderate heating over a gas burner. When all of the sulfur is gone, transfer the crucible to a muffle furnace and heat

¹ These test methods are under the jurisdiction of ASTM Committee D-11 on Rubber and are the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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² *Annual Book of ASTM Standards*, Vol 09.01.