Designation: D4574 – 06 (Reapproved 2017)

Standard Test Methods for Rubber Compounding Materials—Determination of Ash Content[1]

This standard is issued under the fixed designation D4574; 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 cover the determination of ash content of rubber chemicals.

1.2 The test methods include the following materials:

Material	Section
Sulfur	7 – 13
p-Phenylenediamine Antioxidants	14 – 22
Benzothiazole Sulfenamide Accelerators	14 – 22

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]

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D4676 Classification for Rubber Compounding Materials—Antidegradants

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 compounding materials. The test methods may be used for quality control, product acceptance, or research and development. Classification D4676 prescribes percent ash as an important characteristic of rubber antidegradants.

6. Apparatus

6.1 *Muffle Furnace,* capable of temperature regulation of ±25°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-cm^3 capacity.

6.5 *Porcelain Crucible,* high form, size 0, 15-cm^3 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 ± 2°C.

[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.

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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.

SULFUR

7. Scope

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

7.2 *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.*

8. Summary of Test Method

8.1 The ash content of sulfur is determined by the controlled burning of the sulfur, followed by ashing 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-cm^3 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 to 600 ± 25°C for at least 20 min. Cool in a desiccator and weigh.

11. Calculation

11.1 Calculate the percent ash as follows:

$$A = (B/C) \times 100 \quad (1)$$

where:

A = ash, %,
B = mass of ash, g, and
C = mass of sample, g.

12. Report

12.1 Report the following information:

12.1.1 Proper identification of the sample, and

12.1.2 Results of two individual determinations and their average reported as percent ash to the nearest 0.01 %.

13. Precision and Bias[3]

13.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical details.

13.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory programs as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

13.3 A Type 1 (interlaboratory) precision was evaluated in 1986. Both repeatability and reproducibility are short term. A period of a few days separates replicate test results. A test result is the mean value, as specified by this test method, obtained on two determinations or measurements of the property or parameter in question.

13.4 Three different materials were used in the interlaboratory program. They were tested in seven laboratories on two different days.

13.5 The results of the precision calculations for repeatability and reproducibility are given in Table 1, in ascending order of material average or level, for each of the materials evaluated.

NOTE 1—The percent ash values have been multiplied by 100 to avoid leading zeros in Table 1. The values of S_r, r, S_R, and R are influenced by this multiplication factor, for example: S_r (percent ash times 100)/100 = S_r (actual or true percent ash basis).

13.6 The precision of this test method may be expressed in the format of the following statements which 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 and R associated with a mean level in Table 1 closest to the mean level under consideration at any given time, for any given material in routine testing operations.

13.7 *Repeatability*—The repeatability, r, of this test method has been established as the *appropriate value* tabulated in Table 1. 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 nonidentical sample populations.

13.8 *Reproducibility*—The reproducibility, R, of this test method has been established as the *appropriate value* tabulated in Table 1. Two single test results obtained in two different laboratories, under normal test method procedures, that differ

TABLE 1 Precision Results—Ash, %, × 100

Material	Average	Within Laboratory[A]		Between Laboratory[A]	
		S_r	r	S_R	R
Insoluble Sulfur—A (Oil Treated, 90 %)	0.39	0.318	0.901	0.507	1.43
General Purpose Ground Sulfur	3.43	0.847	2.399	1.560	4.41
Insoluble Sulfur—B (Oil Treated, 90 %)	7.75	1.772	5.015	2.150	6.08
Pooled Values[B]	3.85	1.149	3.251	1.561	4.41

[A] S_r = repeatability standard deviation.
r = repeatability (2.83 × the square root of the repeatability variance).
S_R = reproducibility standard deviation.
R = reproducibility (2.83 × the square root of the reproducibility variance).
[B] No values omitted.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1050.

by more than the tabulated *R* (for any given level) must be considered to have come from different or nonidentical sample populations.

NOTE 2—The values of *r* and *R* are relatively large, whereas the average or mean test level is small (close to zero). This is typical for this type of precision measurement process. This should be kept in mind whenever use is made of *r* and *R*.

13.9 The relative repeatability (*r*) and reproducibility (*R*) have been omitted from Table 1 since the level of values tested was extremely low and approached the limits of sensitivity of the test method. Under these circumstances the relative values become trivial.

13.10 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values have not been evaluated for this test method. Bias, therefore, cannot be determined.

ACCELERATORS AND ANTIDEGRADANTS

14. Scope

14.1 This test method describes the determination of the ash content of accelerators and antidegradants.

14.2 *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.*

15. Summary of Test Method

15.1 The ash content is determined by heating a known quantity of material over a gas burner to remove organic material leaving a carbonaceous mass. Ashing is completed in a muffle furnace. The remaining ash, measured by mass difference, is expressed as a percent of the original material.

16. Significance and Use

16.1 The ash content of a sample is the amount of all noncarbon components that remain after combustion, independent of chemical form. In effect, the analysis measures residual inorganic impurities that can remain with the product at low levels following the manufacturing process.

16.2 The quantity of ash in accelerators or antidegradants can affect the performance of these additives in rubber if critical levels are exceeded.

17. Apparatus

17.1 See Section 6.

18. Sampling

18.1 To ensure homogeneity, at least 250 g of the lot sample should be well blended prior to removing the test unit.

19. Procedure

19.1 Ignite the 15-cm^3 crucible in the muffle furnace at 750 ± 25°C for 30 min.

19.2 Transfer the crucible to the desiccator, cool to room temperature and weigh to the nearest 0.0001 g (B).

19.3 Weigh a 5-g test unit to the nearest 0.0001 g into the ignited crucible (C). Place the crucible in the clay triangle, and carefully heat the crucible and contents with the gas burner until all volatile material and pyrolysis products have been removed (gases may flame) and the residue has been carbonized.

19.4 Transfer the crucible to the muffle furnace at 750± 25°C and ignite for 2 h.

19.5 Carefully transfer the crucible containing the ash to the desiccator, cool to room temperature, and reweigh to the nearest 0.0001 g (D).

19.6 Repeat the procedure on a second test unit.

20. Calculation

20.1 Calculate the percent ash to the nearest 0.01 % as follows:

$$A = [(D - B)/(C - B)] \times 100 \quad (2)$$

where:

A = ash, %,
B = mass of crucible, g,
C = mass of crucible plus test unit, g, and
D = mass of crucible plus the ash, g.

21. Report

21.1 Report the following information:

21.1.1 Proper identification of the sample, and

21.1.2 Results obtained from two individual determinations and their average, reported to the nearest 0.01 %.

22. Precision and Bias[3]

22.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical details.

22.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory programs as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

22.3 A Type 1 (interlaboratory) precision was evaluated in 1987. Both repeatability and reproducibility are short term. A period of a few days separates replicate test results. A test result is the mean value, as specified by this test method, obtained on two determinations or measurements of the property or parameter in question.

22.4 Six different materials were used in the interlaboratory program. These were tested in seven laboratories on two different days.

22.5 The results of the precision calculations for repeatability and reproducibility are given in Table 2, in ascending order of material average or level, for each of the materials evaluated.

TABLE 2 Ash Content, PPD Antidegradants and Accelerators, %

Material	Average	Within Laboratory[A]		Between Laboratory[A]	
		S_r	r	S_R	R
M1-6PPD	0.02	0.012	0.033	0.013	0.036
M2-IPPD	0.01	0.004	0.013	0.009	0.026
M3-BMPPD	0.01	0.007	0.020	0.012	0.034
M4-DTPD	0.02	0.005	0.015	0.008	0.025
M5-DCBS	0.03	0.002	0.006	0.007	0.020
M6-TBBS	0.05	0.005	0.016	0.009	0.027
Pooled values[B]	0.02	0.007	0.020	0.010	0.028

[A] S_r = repeatability standard deviation.
r = repeatability (2.83 × the square root of the repeatability variance).
S_R = reproducibility standard deviation.
R = reproducibility (2.83 × the square root of the reproducibility variance).
[B] No values omitted.

22.6 The precision of this test method may be expressed in the format of the following statements which 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 Table 2 closest to the mean level under consideration at any given time, for any given material in routine testing operations.

22.7 *Repeatability*—The repeatability, r, of this test method has been established as the *appropriate value* tabulated in Table 2. 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 nonidentical sample populations.

22.8 *Reproducibility*—The reproducibility, R, of this test method has been established as the *appropriate value* tabulated in Table 2. 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 nonidentical sample populations.

22.9 The relative repeatability, (r), and reproducibility, (R), have been omitted from Table 2 since the level of values tested was extremely low and approached the limits of sensitivity of the test method. Under these circumstances the relative values become trivial.

22.10 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values have not been evaluated for this test method. Bias, therefore, cannot be determined.

23. Keywords

23.1 accelerators; antidegradants; ash; rubber compounding materials; sulfur