Designation: D 4571 – 02

Standard Test Methods for
Rubber Chemicals—Determination of Volatile Material[1]

1. Scope

1.1 These test methods describe the determination of the loss of volatile materials in various rubber chemicals based on the mass loss upon heating at 70°C.

1.2 These test methods for the determination of volatile matter include the following materials:

	Section
Sulfur	7–14
p-Phenylenediamine antidegradents	15–23

1.3 The values stated in SI units are to regarded as the standard. The values given in parentheses are for information only.

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 Industries[2]

D 4676 Classification for Rubber Compounding Materials—Antidegradents[2]

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 rubber chemicals are weighed before and after heating at 70°C.

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 volatile material in rubber chemicals. The test methods may be used for quality control, product acceptance, or research and development. Classification D 4676 prescribes percent volatile material as an important characteristic of rubber antidegradents.

6. Apparatus

6.1 *Weighing Bottle*, low form, 82 cm^3 capacity.

6.2 *Explosion-proof, Vented Air Circulating Oven*, capable of temperature regulation of 70 ± 2°C.

6.3 *Analytical Balance*, sensitive to 0.1 mg.

6.4 *Desiccator*.

SULFUR

7. Scope

7.1 This test method covers the measurement of the loss of volatiles from sulfur at 70°C. The volatile components consist of moisture, low boiling organic components, and sulfur.

8. Summary of Test Method

8.1 The sulfur specimen is weighed before and after heating for 2 h at 70°C. The measured loss of mass is the volatile material content.

9. Significance and Use

9.1 This test method determines the volatile material in sulfur at 70°C. By ascertaining that the volatiles are lower than a predicted maximum limit, it may safely be concluded that moisture (water) is below that limit and is not present in excessive amounts.

9.2 Testing at temperatures higher than 70°C results in greater volatilization of sulfur.

10. Apparatus

10.1 See Section 6.

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

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

11. Procedure

11.1 Weigh the weighing bottle to the nearest 0.1 g (A). Then add approximately 5 g sulfur and weigh again (B). Make sure the sulfur covers the base of the bottle evenly.

11.2 Place the uncovered bottle in the 70°C oven for 2 h. Remove the specimen, cover, and cool in a desiccator for 1 h (C). Weigh the cooled specimen to the nearest 0.1 mg.

12. Calculation

12.1 The volatile material is given by the following equation:

$$V = [(B - C) / (B - A)] \times 100 \tag{1}$$

where:
V = volatiles, %,
A = original specimen mass, and
B = heated specimen mass.

13. Report

13.1 Report results obtained from two individual determinations and their average to the nearest 0.01 %.

14. Precision and Bias [3]

14.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical details.

14.2 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.

14.3 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.

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

14.5 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.

14.6 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 1 closest to the mean level under consideration at any given time, for any given material in routine testing operations.

TABLE 1 Type 1 Precision Results—Volatile Materials, Percent

Material	Average	Within Laboratory[A]		Between Laboratory[A]	
		S_r	r	S_R	R
General Purpose Ground Sulfur	0.07	0.018	0.053	0.045	0.127
Oil Treated, 90 % Insolule Sulfur—A	0.26	0.025	0.073	0.130	0.368
Oil Treated, 90 % Insoluble Sulfur—B	0.32	0.042	0.119	0.151	0.427
Pooled Values[B]	0.22	0.030	0.087	0.119	0.338

[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.

14.7 *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 sample populations.

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

NOTE 1—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*.

14.9 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.

14.10 *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.

p-PHENYLENEDIAMINE ANTIDEGRADENT

15. Scope

15.1 This test method describes the determination of volatile materials in *p*-phenylenediamine antidegradents and is based on the mass loss upon heating at 70°C.

15.2 The measured loss on heating represents volatile organics plus moisture.

16. Summary of Test Method

16.1 The *p*-phenylenediamine test unit is weighed before and after heating for 3 h at 70°C. The measured difference in mass is the volatile material.

17. Significance and Use

17.1 The quantity of volatile material in *p*-phenylenediamines can affect performance of these antidegradents in rubber if they exceed critical levels.

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[3] Supporting data are available from ASTM Headquarters. Request RR: D11-1047.

17.2 Since the loss on heating is an empirical and nonspecific method, it is essential that the prescribed analysis conditions (sample container, oven temperature, oven air flow, cooling conditions, and so forth) be controlled so that results are meaningful.

17.3 Prolonged heating or overheating of the sample or the test unit can result in high heat loss values due to oxidative generation of volatile degradation products.

18. Apparatus

18.1 See Section 6.

19. Sampling

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

20. Procedure

20.1 Dry a clean weighing bottle and stopper (stopper removed) for 30 min in the specified oven set at 70°C. Place the bottle and stopper in the desiccator and allow them to cool to room temperature. Weigh the bottle with a stopper to the nearest 0.1 mg (A).

20.2 Weigh a nominal 20-g test unit into the weighing bottle to the nearest 0.1 mg (B).

20.3 Place the weighing bottle containing the test unit and the stopper (with the stopper removed) in the specified oven, which has equilibrated at 70°C, for 3 h.

20.4 After the heating period, replace the stopper and transfer the bottle to the desiccator for a period of time sufficient for the assembly to equilibrate at room temperature. Reweigh the bottle to the nearest 0.1 mg (C).

20.5 Repeat the procedure on a second test unit.

21. Calculation

21.1 Calculate the percent heating loss to the nearest 0.1 % as follows:

$$H = [(B - C)/(B - A)] \times 100 \qquad (2)$$

where:

H = heating loss, %,
A = mass of weighing bottle and stopper, g,
B = mass of weighing bottle, stopper, and test unit before heating, g, and
C = mass of weighing bottle, stopper, and test unit after heating, g.

22. Report

22.1 Report the following information:

22.1.1 Proper identification of the sample, and

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

23. Precision and Bias [3]

23.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical details.

23.2 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.

23.3 Four different materials were used in the interlaboratory program. These were tested in six laboratories on two different days.

23.4 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 Volatile Materials of *p*-Phenylenediamine, Percent

Material	Average	Within Laboratory[A]		Between Laboratory[A]	
		S_r	r	S_R	R
M1-6PPD	0.06	0.022	0.062	0.029	0.083
M2-IPPD	0.08	0.004	0.011	0.026	0.075
M3-BMPPD	0.19	0.034	0.098	0.073	0.026
M4-DTPD	0.04	0.002	0.005	0.019	0.055
Pooled Values[B]	0.09	0.021	0.061	0.045	0.129

[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.

23.5 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.

23.6 *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.

23.7 *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.

23.8 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.

23.9 *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.

24. Keywords

24.1 antidegradents; rubber chemicals; sulfur; volatile content

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