



# Standard Test Methods for Carbon Black—Ash Content<sup>1</sup>

This standard is issued under the fixed designation D 1506; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope

1.1 These test methods cover the determination of the ash content of carbon black.

1.2 The values stated in SI units are to be regarded as the standard.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Sections 6 and 13.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 1799 Practice for Carbon Black—Sampling Packaged Shipments<sup>2</sup>

D 1900 Practice for Carbon Black—Sampling Bulk Shipments<sup>2</sup>

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries<sup>2,3</sup>

## 3. Summary of Test Method

3.1 A pre-dried sample (1 h at 125°C) is weighed before and after combustion for 16 h at 550°C. The remaining ash is weighed and calculated as a percent of the dried sample.

## 4. Significance and Use

4.1 The ash content of a carbon black is the amount of non-carbon components present after combustion. Primary contributors to ash are the manufacturing process water and the catalyst in the feedstock.

### Method A—Muffle Furnace Method

## 5. Apparatus

5.1 *Muffle Furnace*, capable of temperature regulation of  $\pm 25^\circ\text{C}$  at 550°C.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-24 on Carbon Black and is the direct responsibility of Subcommittee D24.31 on Non-Carbon Black Components of Carbon Black.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 09.01.

<sup>3</sup> Discontinued—See 1989 *Annual Book of ASTM Standards*, Vol 09.01.

## 5.2 Crucibles:

5.2.1 *Porcelain Crucible*, high-form, size O, rim 35 mm, height 29 mm, capacity 15 cm<sup>3</sup>, with cover size E.

5.2.2 *Porous Quartz Fiber Crucible*, rim 47 mm, height 14 mm, with disk.<sup>4</sup>

5.3 *Analytical Balance*, having a sensitivity of 0.1 mg.

5.4 *Desiccator*.

5.5 *Oven*, gravity-convection type, capable of temperature regulation within  $\pm 1^\circ\text{C}$  at 125°C and temperature uniformity within  $\pm 5^\circ\text{C}$ .

## 6. Hazards

### 6.1 Precautions:

6.1.1 Keep the door of the furnace open about 6 mm to admit air to support the combustion of organic material.

6.1.2 Exert care in removing ashed sample from furnace to desiccator.

6.1.3 Always keep the cover on the crucible when transferring it to and from the desiccator to prevent the loss of ash due to air currents.

6.1.4 After the sample has cooled in the desiccator, admit air slowly to avoid loss of ash from the crucible.

## 7. Sampling

7.1 Samples shall be taken in accordance with Practices D 1799 or D 1900.

## 8. Procedure

8.1 Ignite the crucible with cover in the muffle furnace at  $550 \pm 25^\circ\text{C}$  for 1 h. Place the crucible and cover in the desiccator. Cool to room temperature and weigh to nearest 0.1 mg.

8.2 Dry an adequate sample of carbon black for 1 h in the gravity-convection oven set at 125°C in an open container of suitable dimensions, so that the depth of the black is no more than 10 mm. Cool and store the sample in a desiccator.

8.3 Weigh  $2 \pm 0.1$  g of the dried carbon black into the ignited crucible and weigh to the nearest 0.1 mg. Place the uncovered crucible and its cover into the furnace (Note 1) at 550°C for 16 h (porcelain crucible) or 1.5 h (porous quartz fiber crucible—see 6.1.1). Cover the crucible containing the ash,

<sup>4</sup> Suitable quartz ashing dishes are Part Number 303040 available from CEM Corporation, P.O. Box 200, Matthews, NC 28106.

remove from the furnace to the desiccator, and allow to cool to room temperature. Weigh the covered crucible to the nearest 0.1 mg. Put the covered crucible back into the desiccator for additional 0.5 h. Reweigh to the nearest 0.1 mg. Repeat this weighing process until constant mass is obtained.

NOTE 1—For carbon blacks with very low ash content (such as high purity blacks) larger sample mass and longer ashing time may be required.

NOTE 2—If control testing is done at temperatures exceeding 550°C, some volatile salts may be lost from inorganic ash.

## 9. Calculation

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

$$A = [(D - B)/(C - B)] \times 100 \quad (1)$$

where:

*A* = ash, %,

*B* = mass of crucible, g,

*C* = mass of crucible plus the sample, g, and

*D* = mass of crucible plus the ash, g.

## 10. Report

10.1 Report the following information:

10.1.1 Proper identification of the sample, and

10.1.2 A result obtained from an individual determination reported to the nearest 0.01 %.

## 11. Precision and Bias

11.1 These precision statements have been prepared in accordance with Practice D 4483. Refer to this practice for terminology and other statistical details.

11.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials used in the particular interlaboratory program described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method. Any appropriate value may be used from Table 1

11.3 A type 1 inter-laboratory precision program was conducted as detailed in Table 2. Both repeatability and reproducibility represent short term (daily) testing conditions. The testing was performed using two operators in each laboratory performing the test once on each of two days (total of four tests). A test result is the value obtained from a single determination. Acceptable difference values were not measured. The between operator component of variation is included in the calculated values for *r* and *R*.

**TABLE 1 Precision Parameters for D 1506 Ash Content, (Type 1 Precision)**

Units	Percent Ash				
	Mean Level	Sr	(r)	SR	(R)
Material					
IRB#6 (N330)	0.233	0.018	21.4	0.027	32.4
SRB D5 (N762)	0.258	0.015	16.0	0.030	32.6
N650	0.289	0.019	18.7	0.031	30.4
N550	0.436	0.025	16.1	0.037	23.7
SRB A5 (N135)	0.565	0.023	11.5	0.056	27.9
Average	0.356				
Pooled Values		0.020	16.0	0.037	29.7

**TABLE 2 Interlaboratory Precision Program**

Nominal Test Period	Material	Number of Laboratories
March 1996	N650	45
October 1996	IRB#6 (N330)	37
March 1997	SRB N762	48
September 1997	SRB A5 (N135)	43
March 1998	N550	46

11.4 The results of the precision calculations for this test are given in Table 1. The materials are arranged in ascending “mean level” order.

11.5 *Repeatability*—The pooled relative repeatability, (*r*), of this test has been established as 16.0 %. Any other value in Table 1 may be used as an estimate of repeatability, as appropriate. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the repeatability on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate action taken.

NOTE 3—Appropriate action may be an investigation of the test method procedure or apparatus for faulty operation or the declaration of a significant difference in the two materials, samples, etc., which generated the two test results.

11.6 *Reproducibility*—The pooled relative reproducibility, (*R*), of this test has been established as 29.7 %. Any other value in Table 1 may be used as an estimate of reproducibility, as appropriate. The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results produced in different laboratories that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

11.7 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

## Method B—Microwave Furnace Method

### 12. Apparatus

12.1 *Microwave Ashing Furnace*, capable of temperature regulation of  $\pm 25^\circ\text{C}$  at  $550^\circ\text{C}$ .

12.2 *Porcelain Crucible*, high-form, size 0, rim 35 mm, height 29 mm, capacity  $15\text{ cm}^3$ , with cover size E or quartz fiber ashing dish.

12.3 *Analytical Balance*, having a sensitivity of 0.1 mg.

12.4 *Desiccator*.

12.5 *Oven*, gravity-convection type, capable of temperature regulation within  $\pm 1^\circ\text{C}$  at  $125^\circ\text{C}$  and temperature uniformity within  $\pm 5^\circ\text{C}$ .

### 13. Hazards

#### 13.1 Precautions:

13.1.1 Always keep the sample covered when transferring it to and from the desiccator to prevent the loss of ash due to air currents.

13.1.2 After the test portion has cooled in the desiccator, admit air slowly to avoid loss of ash from the crucible.

### 14. Procedure

14.1 Pre-heat the furnace containing crucibles and covers to a set point of  $550^{\circ}\text{C} \pm 25^{\circ}\text{C}$  for 1 h. Place the crucible and cover in the desiccator and cool to room temperature.

14.2 Place an adequate sample of carbon black in an open container so that the depth of the black is no more than 10 mm. Dry the sample for 1 h in the gravity convection oven set at  $125^{\circ}\text{C}$ , cool and store the sample in a desiccator.

14.3 Determine and record the mass of the empty crucible. After the crucible has been conditioned, it is important not to touch or contaminate the cleaned crucibles.

14.4 Add approximately 2 g of carbon black to the crucible and determine its mass to 0.1 mg.

14.5 Place samples in the ashing chamber of the microwave furnace. Prop open the door of the ashing chamber approximately 6 mm to allow a flow of air. This is necessary to ensure that enough air flows into the ashing chamber to support the combustion of organic material.

14.6 In general, test portions should take 2 h to ash for quartz crucibles and 6 h for porcelain crucibles.

14.7 After the allotted time, remove the crucibles from the microwave furnace. Take care not to lose any ash while handling the crucibles. Place the crucible and test portion into a desiccator for cooling. Store in a desiccator until ready to determine the mass of the ashed test portion.

14.8 Determine the mass of the crucible and ash to the nearest 0.1 mg. If trying to determine the proper ashing time, place the crucible back into the microwave furnace and heat for an additional hour. If there is less than 0.5 mg loss in mass, the black has been adequately heated. If the change is greater than 0.5 mg, continue this procedure until the mass change is less than 0.5 mg.

14.9 Repeat the procedure on subsequent test portions.

NOTE 4—For carbon blacks with very low ash content (such as high purity blacks) larger sample mass and longer ashing time may be required.

### 15. Calculation

15.1 Calculate the percentage of 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 the test portion, g, and
- $D$  = mass of crucible plus the ash, g.

### 16. Report

16.1 Report the following information:

- 16.1.1 Proper identification of the sample, and
- 16.1.2 Results obtained from a single determination reported to the nearest 0.01%.

### 17. Precision and Bias

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

17.2 The precision results in this precision and bias give an estimate of the precision 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.

17.3 A Type 1 inter-laboratory precision program was conducted in 1990. Both repeatability and reproducibility represent short term testing conditions. Three laboratories tested five carbon blacks (A, B, C, D, and E) twice on two different days. Therefore,  $p = 3$ ,  $q = 5$ , and  $n = 4$ . Acceptable difference values were not measured.

17.4 The result of the precision calculations are given in Table 3 with the material arranged in ascending mean level order.

**TABLE 3 ASTM Test Method Precision—Type 1 Carbon Black—Ash Content (Method B—Microwave Furnace)<sup>4</sup>**

NOTE 1—Symbols are defined as follows:

- $S_r$  = Within laboratory standard deviation.
- $r$  = Repeatability (in measured units).
- ( $r$ ) = Repeatability (relative).
- $S_R$  = Between laboratory standard deviation.
- $R$  = Reproducibility (in measured units).
- ( $R$ ) = Reproducibility (relative).

Material	Mean Level of Ash, %	Within Laboratories			Between Laboratories		
		$S_r$	$r$	( $r$ )	$S_R$	$R$	( $R$ )
Sample A	0.155	0.015	0.044	28.22	0.015	0.044	28.22
Sample E	0.335	0.027	0.077	16.78	0.027	0.077	16.78
Sample B	0.458	0.040	0.113	19.06	0.040	0.113	19.06
Sample C	0.591	0.039	0.110	14.11	0.039	0.110	14.11
Sample D	0.782	0.024	0.068	20.21	0.033	0.093	27.78
Pooled or averaged values	0.464	0.028	0.079	16.98	0.029	0.083	17.88

<sup>4</sup>This is short-term precision (days) with:  
 $p = 3$ ,  $q = 5$ , and  $n = 4$ .

17.5 The precision for the pooled values for ash content may be expressed as follows:

17.6 *Repeatability*— The repeatability,  $r$ , of the ash content result has been established as 0.08 % ash. Two single test results (or determinations) that differ by more than 0.08 % ash must be considered suspect and dictates that some appropriate investigative action be taken.

17.7 *Reproducibility*— The reproducibility,  $R$ , of the ash content result has been established as 0.08 % ash. Two single test results (or determinations) produced in separate laboratories that differ by more than 0.08 % ash must be considered

suspect and dictates that some appropriate investigative or technical/commercial action be taken.

17.8 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

## 18. Keywords

18.1 ash; carbon black; microwave furnace

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