



## Standard Test Method for Carbon Black—Sieve Residue<sup>1</sup>

This standard is issued under the fixed designation D 1514; 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.

### 1. Scope

1.1 This test method describes the determination of the water wash sieve residue in regular untreated carbon blacks. It may not be applicable to oil-treated carbon blacks because the oil would prevent proper wetting of the black by water.

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

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 Section 6.

### 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</sup>

E 11 Specification for Wire-Cloth Sieves for Testing Purposes<sup>3</sup>

### 3. Summary of Test Method

3.1 A sample of carbon black is washed with water through a wire mesh screen of a specified size until all that remains is a non-carbon black residue. This residue is dried, weighed, and the amount of residue is expressed as mg/kg (ppm) of the original sample.

### 4. Significance and Use

4.1 The quantity of sieve residue of carbon black is important in some molded or extruded products as it may relate to the surface appearance of those products. The maximum residue in each application is normally determined and agreed to between the user and the producer.

### 5. Apparatus

5.1 *Sieve and Filtering Apparatus*,<sup>4</sup> as shown in Fig. 1.

5.2 *Balance*, with a sensitivity of 0.01 g.

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

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

5.5 *Sieves* of either phosphor bronze or stainless steel. The sieve shall conform to Specification E 11. The sieve to be used shall be agreed upon between the purchaser and the seller.

5.6 *Weighing Dishes*.

### 6. Precautions

6.1 Keep the apparatus clean at all times to prevent contamination.

6.2 Examine the sieves each time they are used to make sure that no cracks or holes have developed.

6.3 Examine the strainer periodically to ascertain that the filter screen is in good condition.

### 7. Sampling

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

### 8. Calibration

8.1 Calculate the sieve residue to the nearest mg/kg (ppm) as follows:

$$R = (W/S) \times 10^6 \quad (1)$$

where:

$R$  = sieve residue, mg/kg (ppm),

$W$  = mass of sieve residue, g, and

$S$  = mass of sample, g.

### 9. Procedure

9.1 Prior to making a test, clean the strainer by opening valves *A* and *B* and closing valve *C* as shown in Fig. 1. Allow the strainer to wash for 2 min.

9.2 Close valve *B* and open valve *C*. With valves *A* and *C* completely open, regulate the water pressure by means of a reducing valve. The recommended water pressure is  $207 \pm 34$  kPa ( $30 \pm 5$  lbf/in.<sup>2</sup>).

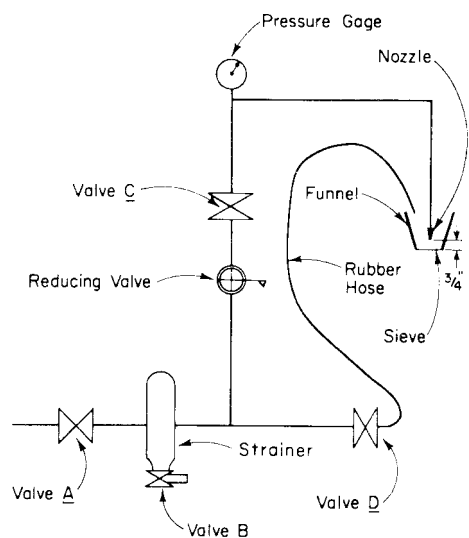
<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.31 on Non-Carbon Black Components of Carbon Black.

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

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>4</sup> Sieve and filtering apparatus, available from Titan Specialties, Inc., P.O. Box 2316, Pampa, TX 70066-2316, has been found suitable.



NOTE 1—Industrial/O.E.M. filter and pleated polyester fabric reusable cartridges have been found to be a satisfactory strainer. Housing No. 20 and cartridge No. 30 are available from Amtec, Plymouth Products Division, 562 Indiana Ave., Sheboygan, WI 53081.

NOTE 2—Corrosion-free piping must be used.

NOTE 3—Detailed drawings can be obtained from Test Method D 1514 – 88a.

NOTE 4—Spray nozzle: Part No. 460.746.30 BE available from Lechler Inc., 445 Kautz Rd., St. Charles, IL 60174, or Titan Specialties Inc., P.O. Box 2316, Pampa, TX 70066-2316.

**FIG. 1 Schematic Diagram of Apparatus for Sieve Residue Determination**

9.3 After the water pressure has been regulated, attach the sieve holder with a 50 mm (2 in.) diameter sieve to the funnel and allow the water to run through it for 3 min. Stop the water flow by closing valves A and C. Examine the sieve for particles; if none are observed, the apparatus is ready for use.

9.4 Weigh 100.0 g of the carbon black on the balance.

9.5 Attach the proper sieve to the funnel and start the water flow by opening valves A and C.

9.6 Add the carbon black slowly to the funnel to prevent plugging of the sieve.

NOTE 1—If desired, it shall be permissible to disperse the carbon black in water using a high speed stirring apparatus. It shall also be permissible

to utilize wetting agents to facilitate dispersion. Care must be taken to ensure that all of the solid material is transferred from the vessel used for dispersing the black into the funnel of the washing apparatus.

9.7 Use a gentle stream of water from the hose attached to valve D to wash down any carbon black on the sides of the funnel.

9.8 Continue washing until the wash water coming through the sieve is clear.

9.9 Remove the sieve holder and sieve from the funnel and rub the residue lightly with the finger to disaggregate any hard carbon black lumps which may not have been thoroughly wet by the water.

9.10 Replace the sieve holder and sieve and wash for an additional 2 min.

9.11 Remove the sieve holder and sieve and dry for 1 h at 125°C.

9.12 Transfer the dried residue to a piece of smooth white bond paper and rub gently to remove any carbon black remaining on the residue. Rub until the white paper no longer shows any smears.

NOTE 2—The purpose of gently rubbing the residue is to remove any black that may be clinging to the gritty residue. Do not apply excessive force when rubbing, as this action may fracture the residue allowing it to escape, or may trap the residue in the paper causing a low grit determination. Similarly, do not wear gloves when performing this step as the residue may be trapped in the glove material, giving a low result.

9.13 Brush all the loose residue to a tared weighing dish and weigh to the nearest 0.1 mg using the analytical balance.

**10. Report**

10.1 Report the following information:

- 10.1.1 Proper identification of the sample,
- 10.1.2 Identification of the sieve number, and
- 10.1.3 Result of the determinations reported to the nearest mg/kg (ppm).

**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

**TABLE 1 Test Method D 1514 Test Method Precision—Type 1 (Sieve Residue Using a No. 325 Sieve)<sup>A</sup>**

Material	Mean Level, mg/kg (ppm)	Within Laboratories <sup>B</sup>			Between Laboratories <sup>B</sup>		
		<i>S<sub>r</sub></i>	<i>r</i>	( <i>r</i> )	<i>S<sub>R</sub></i>	<i>R</i>	( <i>R</i> )
B	9.3	2.62	7.42	79.5	7.09	20.08	215
A	52.9	8.02	22.70	42.9	28.79	81.49	154
C	137.0	19.16	54.22	39.6	42.29	119.68	87.3
Average	66.4						
Pooled values		12.09	34.20	51.5	29.82	84.39	127

<sup>A</sup> This is short-term precision (days) with outliers removed from the data set.

<sup>B</sup> Symbols are defined as follows:

- S<sub>r</sub>* = within-laboratory standard deviation,
- r* = repeatability (in measurement units),
- (*r*) = repeatability (in percent),
- S<sub>R</sub>* = between-laboratory standard deviation,
- R* = reproducibility (in measurement units), and
- (*R*) = reproducibility (in percent).

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 2. A type 1 inter-laboratory precision program was conducted as detailed in Table 3. 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).

11.3 The results of the precision calculations for this test are given in Table 2. The materials are arranged in ascending “mean level” order.

11.4 *Repeatability*—The pooled relative repeatability, (r), of this test has been established as 80.8 %. Any other value in Table 2 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 2 Precision Parameters for D 1514 Sieve Residue, (Type 1 (No. 325 Sieve) Precision)**

Units	mg/kg (ppm)				
Material	Mean Level	Sr	(r)	SR	(R)
IRB#6 (N330)	18.3	6.0	93.3	13.1	203
N762	18.3	7.4	114	9.7	150
SRB A5 (N135)	20.8	5.1	69.5	9.7	132
N550	64.3	10.4	45.9	24.7	109
N650	75.7	20.2	75.6	46.7	174
Average	39.5				
Pooled Values		11.3	80.8	25.1	180

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**TABLE 3 Interlaboratory Precision Program**

Nominal Test Period	Material	Number of Laboratories
March 1996	N650	49
October 1996	IRB#6 (N330)	41
March 1997	N762	45
September 1997	SRB A5 (N135)	42
March 1998	N550	47

Table 2 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.

11.5 *Reproducibility*—The pooled relative reproducibility, (R), of this test has been established as 180 %. Any other value in Table 2 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 2 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

11.6 *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.

## 12. Keywords

12.1 carbon black; non-carbon black residue; sieve residue; water wash sieve residue