Designation: D4218 – 15

Standard Test Method for Determination of Carbon Black Content in Polyethylene Compounds By the Muffle-Furnace Technique[1]

This standard is issued under the fixed designation D4218; 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 This test method covers the determination of black polyethylene compounds containing channel or furnace black. It is not applicable to thermal black.

1.2 This test method is not suitable for plastics that char on pyrolysis.

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.* Specific hazard statements are given in Section 7.

NOTE 1—There is no known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics

D1603 Test Method for Carbon Black Content in Olefin Plastics

D2741 Test Method for Susceptibility of Polyethylene Bottles to Soot Accumulation (Withdrawn 2011)[3]

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI): The Modern Metric System

3. Terminology

3.1 *Definitions*—For definitions of plastics terms used in this test method, see Terminology D883.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *channel black*—those carbon blacks produced by a process that operates in an open system in which a multitude of small diffusion flames burn in air and carbon is deposited on cooled surfaces.

3.2.2 *furnace black*—those carbon blacks produced using the principle of continuous oxidative combustion in closed systems.

3.2.3 *thermal black*—those carbon blacks based on a process that uses thermal decomposition in the absence of oxygen and operates in a closed system. Particles grow very slowly and can become very large and filamentous structures.

3.3 *Symbols*—Units and symbols used in this test method are those recommended in Practice IEEE/ASTM SI-10.

4. Summary of Test Method

4.1 Black polyethylene compound contained in a disposable aluminum weighing dish is pyrolyzed in a muffle furnace for a short period. During the pyrolysis of the polymer, the air in the muffle furnace becomes oxygen-deficient to prevent the combustion of the residual carbon black. Any soot, as defined in Test Method D2741, produced by the initial combustion of the gases is consumed before the carbon black itself.

4.2 After cooling and weighing for residual carbon black, the dish and contents are reinserted into the muffle furnace to determine ash content and the results used to determine true carbon black content. This is done only if the compound is suspected of containing mineral fillers as well as carbon black.

5. Significance and Use

5.1 This test method is capable of yielding duplicate test data, in 20 min or less, for a simple carbon black content determination.

5.2 This test method is suitable for manufacturing quality control, technical service, and research work.

5.3 For referee requirements, the number of replicate measurements is increased. Alternatively, a control sample of known carbon black content is tested with the unknown sample.

5.4 Test Method D1603 is available for referee testing.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.01).

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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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

6. Apparatus

6.1 *Fume Hood or Vent Hood.*

6.2 *Muffle Furnace,* approximately 100 mm in the three internal dimensions. Furnaces with larger internal dimensions have been found to be unsuitable. The seal at the door must be free of gaping cracks or chips that will allow air into the furnace. A muffle furnace with bare heating wires on the floor of the furnace must not be used without additional electrical safety precautions.

6.3 *Screen, 20-Mesh,* approximately 120 by 100 mm. Fold down approximately 10 mm of the long dimension to provide a raised platform to fit inside the muffle furnace.

6.4 *Thermocouple-Probe Temperature Controller or Equivalent*—This is an integral part of newer muffle furnaces or is purchased separately to provide improved control on older furnaces.

6.5 *Desiccator* (for example, 255 mm), with alumina or equivalent desiccant.

6.6 *Laboratory Interval Timer,* with alarm.

6.7 *Disposable Aluminum Weighing Dish*—Dishes with crimped sides and tabs on the rim are satisfactory.

6.8 *Long Tongs or Tweezers* (250 to 300 mm).

6.9 *Analytical Balance,* capable of measuring to 0.1 mg.

7. Hazards

7.1 Do not use a muffle furnace with bare heating elements on the floor of the furnace. Take steps to make the furnace electrically safe.

7.2 Do not attempt to test plastics that evolve corrosive fumes on pyrolysis.

7.3 Do not open the muffle furnace door during the test. Opening the door while fumes are being emitted from the door seal will cause ignition of the fumes.

7.4 This test must be conducted in a fume hood or under a vent hood to remove the resulting fumes.

8. Sample and Test Specimen

8.1 Samples are obtained from the manufacturer's or purchaser's sampling regimens or from a specifically identified part.

8.2 The test specimens are in the form of granules or pieces cut from an article such as pipe, jacket, film, molding, etc. Soiled articles must be washed, and printed articles, such as films, are wiped clean with a suitable solvent.

9. Conditioning

9.1 Conditioning is unnecessary.

10. Procedure

10.1 Place the muffle furnace in a fume hood or under a vent hood.

10.2 Set the controller indicator to 600°C and let it stabilize from 600 to 610°C.

10.3 Mark an aluminum weighing dish with an identifying impression on the tab.

10.4 Place the dish on the screen in the muffle furnace and burn off the surface oil for 2 min, as indicated by timer.

10.5 Transfer the dish to the desiccator and let it cool for at least 2 min.

10.6 Weigh the dish accurately on the analytical balance. Record the results as W_1.

10.7 Add about 1 g of specimen and reweigh the dish and contents accurately. Record the results as W_2.

10.8 Place the weighed dish and contents into the muffle furnace and set the interval timer for 3 min. If there are no known ashable fillers, then this time is sufficient. If there are other or additional fillers that will oxidize further, then follow the procedure at 10.11 to obtain a more complete pyrolysis.

10.9 After the elapsed time, remove the dish from the muffle furnace and place it in the desiccator to cool for at least 2 min. Longer cooling periods do not affect the test.

10.10 Reweigh the dish and remaining carbon black accurately. Record the results as W_3.

10.11 If ashable mineral fillers are suspected to be present in the compound, replace the dish and contents into the muffle furnace for a period of 10 min or longer until only light-colored ash remains.

10.12 Transfer the dish and contents from the muffle furnace to the desiccator and let it cool for 2 min.

10.13 Reweigh the dish and ash accurately. Record the results as W_4.

10.14 A minimum of two determinations is made for each sample.

11. Calculation

11.1 Calculate the percent carbon black content as follows:

$$\text{Carbon black, \%} = \frac{W_3 - W_1}{W_2 - W_1} \times 100 \quad (1)$$

or

$$\text{Carbon black, \%} = \frac{W_3 - W_4}{W_2 - W_1} \times 100 \quad (2)$$

where W_1, W_2, W_3, and W_4 are described in Section 10.

12. Report

12.1 Report the following information:

12.1.1 Complete identification of material tested, including type, source, manufacturer's code number, form, previous history, etc.

12.1.2 Specific location of the specimen, if significant, for example, from articles.

12.1.3 Value of each determination and average. When the specimen requires the longer time for complete ashing, then report the time and the ash content as part of the result.

12.1.4 If more than one apparatus is available, identification of the apparatus used.

12.1.5 Value obtained from a control sample, if tested.

12.1.6 Date.

13. Precision and Bias[4]

13.1 *Precision*—Six laboratories participated in an interlaboratory evaluation of this test method. Each test sample was repaired by roll milling natural polyethylene and black masterbatch to achieve 2, 2.5, or 3 % level of carbon black content. The milled crepe was reduced to fine particle size on a grinder. The ground material was blended before sampling for distribution. Six compounds were prepared in the above manner. Three compounds were based on polyethylene to give carbon black contents of 2, 2.5, and 3 %. A similar set of three compounds was prepared using polyethylene containing 3 % vinyl acetate and a different grade of carbon black. Duplicate determinations of carbon black content (uncorrected for ash) were made on six materials that had been prepared with known target values of carbon black. Table 1 summarizes the results obtained for S_r, S_L, and S_R as these parameters are defined in Practice E691:

NOTE 2—**Caution:** The following explanations of *r* and *R* (13.2 – 13.2.3) are only intended to present a meaningful way of considering the approximate precision of this test method. Do not apply the data in Table 1 rigorously to acceptance or rejection of material, as those data are specific to the round robin and are not representative of other lots, conditions, materials, or laboratories. Apply the principles outlined in Practice E691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 13.2 – 13.2.3 would then be valid for such data.

13.2 *Concept of r and R*—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing one specimen:

TABLE 1 Precision Data

Material	Target Carbon Black	Average	S_r [A]	S_R [B]	r [C]	R [D]
D	2.00	2.0142	0.0272	0.03884	0.07698	0.10992
A	2.00	2.0433	0.0342	0.06117	0.09679	0.17311
E	2.50	2.5392	0.0250	0.05963	0.07075	0.16874
B	2.50	2.5517	0.0410	0.05808	0.11603	0.16437
F	3.00	3.0242	0.0364	0.06892	0.10301	0.19506
C	3.00	3.0710	0.0270	0.0613	0.07641	0.18149

[A] S_r = within-laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviations of the test results from all of the participating laboratories.
[B] S_R = between-laboratories reproducibility, expressed as standard deviation, for the indicated material.
[C] r = within-laboratory repeatability limit = 2.8 S_r.
[D] R = between-laboratories reproducibility limit = 2.8 S_R.

13.2.1 *Repeatability Limit, r*—(Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day)—The two test results are not equivalent if they differ by more than the *r* value for that material.

13.2.2 *Reproducibility Limit, R*—(Comparing two test results for the same material, obtained by different operators using different equipment in different laboratories)—The two test results are not equivalent if they differ by more than the *R* value for that material.

13.2.3 Any judgement in accordance with 13.2.1 or 13.2.2 would have an approximate 95 % (0.95) probability of being correct.

13.3 *Bias*—There are no recognized standards by which to estimate the bias of this test method.

14. Keywords

14.1 carbon black; muffle furnace; polyethylene

[4] Supporting data are available from ASTM Headquarters. Request RR:D20-1100.