Designation: D5805 – 00 (Reapproved 2019)

Standard Test Methods for Rubber—Determination of Carbon Black in Masterbatches[1]

This standard is issued under the fixed designation D5805; 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 the amount of carbon black in a masterbatch and cover emulsion SBR-carbon black mixtures, but may be applicable to other polymers. Three test methods are included:

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Inert Atmosphere Pyrolysis	3 – 10
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NOTE 1—The nomenclature used in these test methods is in accordance with Practice D1418.

1.2 The values stated in SI units are to be regarded as the standard. The values given 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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]

D1418 Practice for Rubber and Rubber Latices—Nomenclature

D6370 Test Method for Rubber—Compositional Analysis by Thermogravimetry (TGA)

TEST METHOD A—CARBON BLACK

Inert Atmosphere Pyrolysis Test Method

3. Summary of Test Method

3.1 The prepared dried sample and a sample of standard SBR black determinate are tested in triplicate. The samples are weighed accurately into tared crucibles and then placed in a combustion tube at 550°C, which is continuously flushed with CO_2 until the rubber hydrocarbon is distilled. The crucibles and contents are cooled in a desiccator, weighed, then inserted into a muffle furnace at 550°C until all carbonaceous material is burned. The crucible and contents are again cooled in a desiccator and weighed. The percentage of carbon black is calculated from the weights.

4. Significance and Use

4.1 The carbon black content of an unvulcanized black or oil-black SBR masterbatch may be determined by this test method if an inert (CO_2) atmosphere and a determinate (see Note 2) of known carbon black content are available.

NOTE 2—Determinate black masterbatch may be prepared by careful addition of exact amounts of rubber and carbon black on a mill. An alternative would be a thorough blending of a quantity of black masterbatch and determination of the carbon black by use of a previously established determinate.

5. Apparatus

5.1 *Drying Oven.*

5.2 *Porcelain Crucibles,* No. 00000, tared.

5.3 *Weighing Bottles,* tared.

5.4 *Fused Alumina Combustion Boats.* [3]

5.5 *Combustion Tubes.* [4]

5.6 *Combustion Furnace.*

[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.

[3] Alundum No. 3 combustion boats have been found satisfactory for this purpose.

[4] The sole source of supply of the combustion tube known to the committee at this time is the McDaniel high-temperature combustion tube, 30 in. (762 mm) long by 1 in. (25.4 mm) in diameter available from Dunlap and Co., 623 Staring Lane, Baton Rouge, LA 70810. This tube will accommodate a maximum of two combustion boats containing six samples each within the heated area of the furnace. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7. Preparation of Sample

7.1 Blend 200 g of dried sample obtained in accordance with 4.1 or 9.1 by passing it five times between the rolls of a laboratory mill. Maintain the roll temperature at 50 ± 5°C (122 ± 9°F) and a distance between the rolls of 0.5 ± 0.13 mm (0.020 ± 0.005 in.). After blending, sheet the sample from the mill with the distance between the rolls set at 0.25 ± 0.05 mm (0.010 ± 0.002 in.). Dry approximately 2 g of the blended, thinly sheeted material for 1 h in the oven at 100 ± 5°C (212 ± 9°F). Keep the dried sheet in a desiccator until ready to weigh the samples.

8. Procedure

8.1 Into the tared porcelain crucibles accurately weigh three 0.3 to 0.5-g samples from the sheet of material being tested and three 0.3 to 0.5-g samples from a similarly prepared sheet of standard SBR black determinate (see Note 2). Due to the hygroscopic nature of the samples and the carbon black, weigh the crucibles and contents in the tared weighing bottles.

8.2 Place the six crucibles in a fused alumina combustion boat, or boats, alternating the samples of determinate and the test samples. Insert the combustion boat, or boats, into the combustion tube. Pass a stream of oxygen-free CO_2 through the tube at a flow rate of 150 to 240 cm^3 (atmospheric pressure)/min. After the tube has been swept free of oxygen (approximately 5 min is sufficient time), place it in the combustion furnace maintained at a temperature of 550 ± 25°C, and distill off the rubber hydrocarbon. Continue to pass CO_2 through the tube, while allowing the tube to remain in the furnace for 30 min. At the end of this period, remove the combustion tube from the furnace and allow it to cool, meanwhile continuing the flow of CO_2 through it. When it is cool, remove the boat, or boats, and place the crucibles in a desiccator until they cool to room temperature and are ready to be weighed. Accurately weigh the crucibles and contents.

8.3 After weighing the crucibles containing the carbon black residues, ignite the samples by either of the following methods:

8.3.1 Place the crucibles and contents in a combustion boat, or boats, and insert them into a second combustion tube. Place the tube in a combustion furnace maintained at 550 ± 25°C and pass a stream of oxygen or air through the tube.

8.3.2 Alternately, place the crucibles and contents in a muffle furnace maintained at 550 ± 25°C. After the carbon has been completely burned, remove the boat, or boats, from the muffle furnace and place the crucibles in a desiccator. When the crucibles have cooled to room temperature, weigh them.

9. Calculation

9.1 Calculate the percentage of ash, R, in the dry sample as follows:

$$R = [(F - E)/(D - E)] \times 100 \quad (1)$$

where:

F = mass of crucible plus ash after ignition of the carbon black, g,
E = mass of crucible, g, and
D = mass of original dry sample plus crucible, g.

9.2 Calculate the individual values, c_1, c_2, and c_3, for carbon black content of the material being tested on the dry, ash-free basis as follows:

$$c = 100(Q - F)/(D - E)(1 - 0.01R) \quad (2)$$

where:

Q = mass of crucible and residue after distillation of hydrocarbon, g, and F, D, E, and R are defined in 9.1.

9.3 Calculate the individual values, c_1', c_2', and c_3', for carbon black content of the standard SBR carbon black determinate (see Note 2) on the dry, ash-free basis in accordance with 3.1.2, using the appropriate values obtained on the samples of the determinate.

9.4 Calculate the corrected percentage of carbon black in the material being tested as follows:

$$\text{Carbon black, \%} = C + A - C' \quad (3)$$

where:

C = average of the three values of percentage carbon black (c_1, c_2, and c_3) on the dry, ash-free basis, obtained on the material being tested,
A = percentage of carbon black assigned as the standard value for the standard SBR carbon black determinate (see Note 2), and
C' = average of the three values of percentage carbon black, c_1', c_2', and c_3', on the dry, ash-free basis, obtained on the standard carbon black determinate.

10. Precision and Bias

10.1 Precision and bias have not been determined.

TEST METHOD B

Vacuum Pyrolysis Test Method

11. Summary of Test Method

11.1 The prepared dried sample is tested in triplicate. The samples accurately are weighed into tared crucibles and placed in a combustion tube and maintained under very low pressure at 600°C until the rubber is distilled. The samples are cooled

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

and returned to atmospheric pressure. The crucibles and contents are then further cooled in a desiccator and weighed. The percentage of carbon black is calculated on an ash-free basis.

12. Significance and Use

12.1 Carbon black in unvulcanized carbon black or oil-carbon black SBR masterbatch may be determined by this test method without the use of a determinate sample.

13. Apparatus

13.1 *Porcelain Crucible,* No. 00000.

13.2 *Combustion Boats,* sized to accommodate crucibles.

13.3 *Combustion Tube,* high-silica (see Fig. 1).[4]

13.4 *Combustion Furnace,* capable of controlling temperature at 600 ± 25°C.

13.5 *Vacuum Pump.*

13.6 *Vacuum Seal Lubricant* for ground-glass joint.

13.7 *Muffle Furnace,* capable of controlling at 550 ± 25°C.

13.8 *Drying Oven,* capable of controlling at 105 ± 5°C.

13.9 *Insertion Rod.*

13.10 *Trap,* to condense vapors coming from combustion tube (see Fig. 1).

14. Preparation of Sample

14.1 Blend 200 g of the dried sample obtained in accordance with 4.1 or 9.1 by passing it five times between the rolls of a laboratory mill. Maintain the roll temperature at 50 ± 5°C (122 ± 9°F) and a distance between the rolls of 0.5 ± 0.13 mm (0.20 ± 0.005 in.). After blending, sheet the sample from the mill with the distance between the rolls set at 0.25 ± 0.05 mm (0.010 ± 0.002 in.). Dry approximately 2 g of the blended, thinly sheeted material for 1 h in the oven at 105 ± 5°C. Keep the dried sheet in a desiccator until ready to weigh the samples.

15. Procedure

15.1 Place into a tared crucible 0.3 to 0.5 g (to the nearest 0.1 mg) from the sheet of material being tested. Using the insertion rod, insert the boat containing the crucibles into the tube to maximum depth. Insert the ground-glass stopper coated properly with vacuum seal lubricant, turn on the vacuum pump, and evacuate the tube to less than 1.3 kPa.

15.2 Introduce the combustion tube into the combustion furnace maintained at 600 ± 25°C, using care not to rotate the tube or otherwise cause sample disturbance. Allow the tube to remain in the furnace for 10 min, maintaining maximum pressure of 1.3 kPa (10 mm Hg) during combustion. Remove the tube from the furnace, allow the tube and contents to cool for 7 min, and slowly allow pressure to increase to prevent blowing residue out of crucible.

15.3 Remove the boat and place it in a desiccator to cool to room temperature. Weigh each crucible and residue to the nearest 0.1 mg. Determine the ash content on a portion of sample.

16. Calculation

16.1 Calculate the percentage of carbon black as follows:

$$\text{Carbon black, } \% = [(A - B) - 0.01\,CD)/(C - 0.01\,CD) \times 100 \quad (4)$$

where:

A = mass of crucible and residue after distilling, g,
B = mass of crucible, g,
C = mass of original sample, g, and
D = percentage of ash.

17. Precision and Bias

17.1 Precision and bias have not been determined.

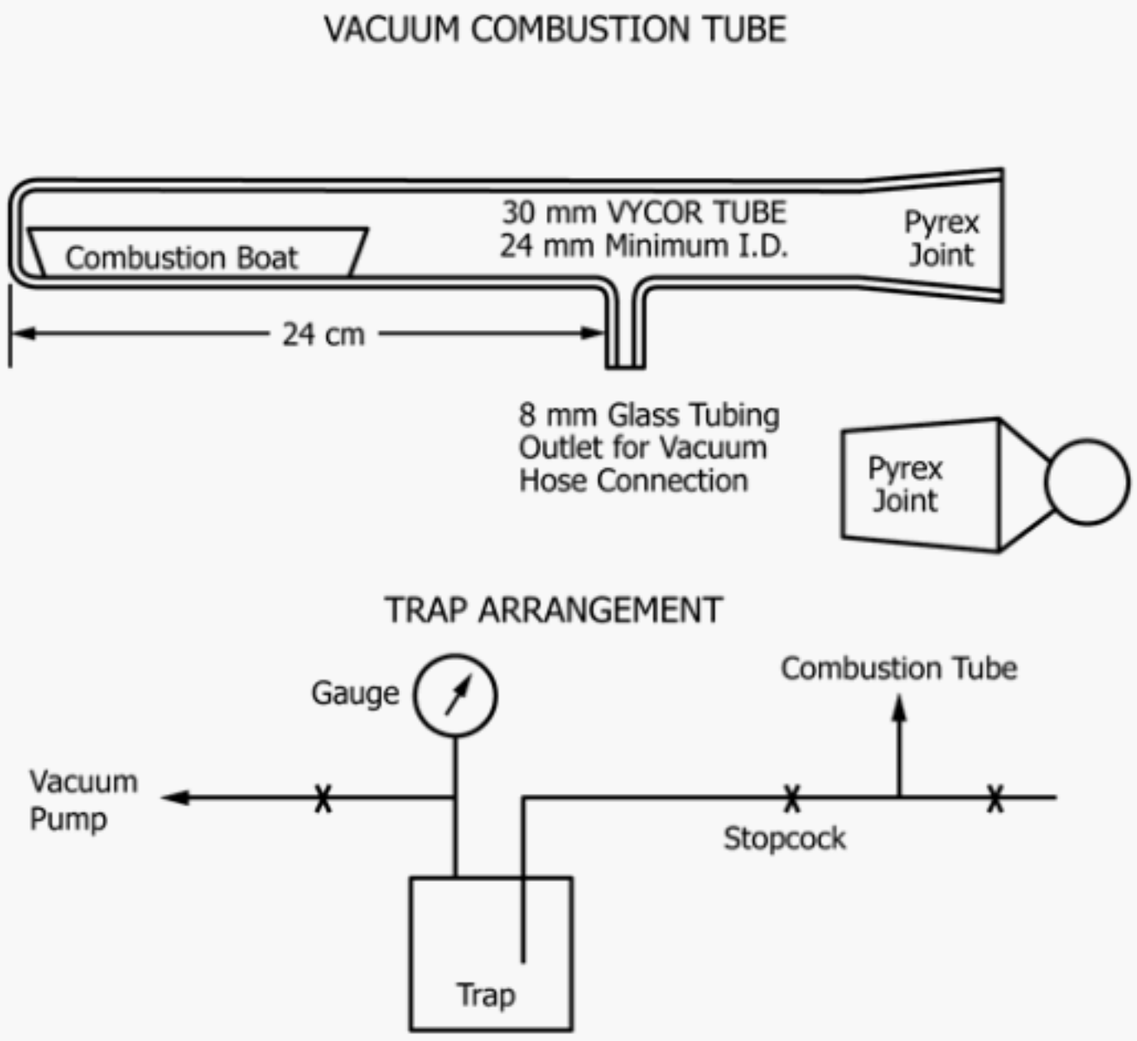

FIG. 1 Vacuum Combustion Tube and Trap Arrangement

TEST METHOD C
Thermogravimetric Analyzer

18. Summary of Test Method

18.1 The prepared, dried sample is placed in a calibrated thermogravimetric analyzer (TGA) where it is weighed using the integral balance (See Test Method D6370). The sample is heated to 575°C in an inert atmosphere to pyrolyze the rubber and other softeners. The atmosphere is then switched to air, combusting the carbon black. The difference in mass before and after the switch of gases is the mass of the carbon black contained in the sample.

19. Significance and Use

19.1 The carbon black content of any vulcanized or unvulcanized compound may be determined by this method without use of a determinate sample.

19.2 Expertise in thermogravimetric analysis (TGA) is necessary to the successful application of this test method.

20. Apparatus

20.1 *Thermogravimetric Analyzer,* maintained and operated in accordance with manufacturer's instructions.

21. Preparation of Sample

21.1 Blend 200 g of dried sample obtained in accordance with 4.1 or 9.1 by passing it five times between the rolls of a laboratory mill. Maintain the roll temperature at 50 ± 5°C (122 ± 9°F) and a distance between the rolls of 0.5 ± 0.13 mm (0.020 ± 0.005 in.). After blending, sheet the sample from the mill with the distance between the rolls set at 0.25 ± 0.05 mm (0.010 ± 0.002 in.). Dry approximately 2 g of the blended, thinly sheeted material for 1 h in the oven at 100 ± 5°C (212 ± 9°F). Keep the dried sheet in a desiccator until ready to weigh the samples.

22. Procedure

22.1 Set zero on the analyzer following manufacturer's instructions.

22.2 Place in the instrument pan 10 ± 3 mg of the prepared sample. Weigh accurately using integral instrument balance.

22.3 Under inert atmosphere heat the sample to 575 ± 50°C and hold until constant mass.

22.4 Switch atmosphere to air and hold until constant mass.

23. Calculation

23.1 Calculate the percentage of carbon black in the samples as follows:

$$C = (A - B)(100)/I \quad (5)$$

where:

C = % carbon black in the sample,
A = constant mass of sample at 575°C under inert gas,
B = constant mass of sample at 575°C under air, and
I = initial mass of sample.

23.2 Alternatively, the instrument may perform this calculation automatically.

24. Precision and Bias

24.1 Precision and bias have not been determined.

25. Keywords

25.1 carbon black; masterbatch; thermogravimetric analysis (TGA)