



Designation: D8474 – 23

Standard Test Method for Recovered Carbon Black (rCB)—Compositional Analysis by Thermogravimetry (TGA)¹

This standard is issued under the fixed designation D8474; 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 provides a thermogravimetric (TGA) technique to determine the amounts of organic residue, overall carbon content, and ash content in recovered carbon black (rCB).

1.2 This test method uses previously calibrated, manual, or computer-assisted TGA instrumentation.

1.3 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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

[D1799 Practice for Carbon Black—Sampling Packaged Shipments](#)

[D1900 Practice for Carbon Black—Sampling Bulk Shipments](#)

[D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries](#)

[D8178 Terminology Relating to Recovered Carbon Black \(rCB\)](#)

[E473 Terminology Relating to Thermal Analysis and Rheology](#)

[E1953 Practice for Description of Thermal Analysis and Rheology Apparatus](#)

3. Terminology

3.1 The definitions relating to recovered carbon black (rCB) in this standard are in accordance with Terminology [D8178](#).

3.2 The definitions for thermal analysis in this standard are in accordance with Terminology [E473](#).

3.3 The descriptions of thermal analysis equipment in Practice [E1953](#) apply to this test method.

3.4 *Definitions of Terms Specific to This Standard:*

3.4.1 *ash, n*—inorganic substance with a decomposition temperature $>550^{\circ}\text{C}$.

3.4.2 *carbon content, n*—carbon black, carbonaceous residues, and other non-volatile oxidizable carbon containing substances at temperatures $\leq 550^{\circ}\text{C}$.

3.4.3 *organic residue, n*—volatile residuals from organic substances present in the feedstock at temperatures $\leq 550^{\circ}\text{C}$.

4. Summary of Test Method

4.1 The mass of the rCB sample, heated at a controlled, specified rate in a controlled, specified environment is recorded as a function of temperature. The mass loss over the specified temperature range provides a value for the amounts of organic residue, carbon content, and ash content of the sample.

5. Significance and Use

5.1 This test method is intended for use in quality control of production, for research and development purposes in which a compositional analysis or comparisons of different materials are needed.

5.2 This test method may not be suitable for rCB products containing either organic substances that decompose or oxidize only at temperatures of above 550°C or inorganic substances with decomposition temperatures $\leq 550^{\circ}\text{C}$.

¹ This test method is under the jurisdiction of ASTM Committee [D36](#) on Recovered Carbon Black (rCB) and is the direct responsibility of Subcommittee [D36.10](#) on Recovered Carbon Black.

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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.

6. Apparatus

6.1 *Thermogravimetric Analyzer (TGA)*—Apparatus capable of continuously weighing a test sample at a sensitivity of $\pm 2 \mu\text{g}$ and recording the change in mass of the test sample under atmospheric control over a specified temperature range.

7. Reagents and Materials

7.1 An inert compressed gas, such as argon or nitrogen, and a reactive gas, such as air or oxygen.

7.2 Compressed gases must be 99.99 % minimum purity.

7.3 The inert purge gas shall not contain more than $10 \mu\text{g/g}$ oxygen.

8. Calibration

8.1 Calibrate the apparatus with the empty crucible according to the prescribed procedure below, following similar holding times for each step to represent holding times for the rCB samples.

9. Procedure

9.1 Take 5 g of raw rCB or rCB, crush and homogenize with pestle and mortar. Dry the material at 125°C for a minimum of 1 h.

9.2 Cool the sample in a desiccator to room temperature and leave it there until ready to test.

9.3 Apply the manufacturer's recommended flow, using inert gas such as argon or nitrogen.

9.4 Tare an empty TGA-pan(s) with the flow rate from 9.3.

9.5 Weigh 20 to 40 mg of rCB or Raw rCB into a pre-tared TGA-pan and place the pan into the TGA.

9.6 Heat the TGA to 40°C and allow the instrument to equilibrate for a minimum of 2 min.

9.7 Heat the TGA from 40 to 550°C at $20^\circ\text{C}/\text{min}$ only using argon or nitrogen.

9.8 *Isothermal Step*—Hold for a minimum of 15 min after no change in first derivative can be observed and the mass loss is $< 5 \mu\text{g}/\text{min}$ ($< 0.025 \%/ \text{min}$) for the step to be considered complete.

9.9 Switch the purge gas to air or oxygen and purge at the manufacturer's recommended flow.

9.10 Hold for a minimum of 15 min after no change in the first derivative can be observed and the mass loss is $< 5 \mu\text{g}/\text{min}$ ($< 0.025 \%/ \text{min}$) for the step to be considered complete.

9.11 End of test.

10. Sampling

10.1 Samples shall be taken in accordance with Practices **D1799** or **D1900**.

11. Calculation

11.1 Record the percent mass loss for organics, carbon content, and ash as shown in **Fig. 1**.

12. Report

12.1 Report the following information:

12.1.1 Identification of the test sample, and

12.1.2 Organic residue, carbon content, and ash found, each to the nearest 0.1 %.

13. Precision and Bias

13.1 The precision and bias for the test method is being developed in accordance with Practice **D4483**.

14. Keywords

14.1 ash; carbon content; organic residue; rCB; recovered carbon black; thermogravimetry