Designation: C561 – 16

An American National Standard

Standard Test Method for Ash in a Graphite Sample[1]

This standard is issued under the fixed designation C561; 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.

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

1. Scope*

1.1 This test method provides a practical determination for the ash content in a graphite sample.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C562 Test Method for Moisture in a Graphite Sample

3. Terminology

3.1 *Definitions:*

3.1.1 *ash, n—in carbon and graphite technology*, residue remaining after oxidation of a carbon or graphite.

4. Significance and Use

4.1 This test method provides a practical estimate of nonburnable residues in commercially available graphite materials. The ash values determined by this test method are of use in comparing the relative purity of various grades of graphite. To facilitate use, this test method institutes simplifications that preclude the ability to determine absolutely the ash values of the test graphite material due to uncontrolled sources of trace contamination.

4.2 This test method is not intended for use in determining the ash content of purified graphites, for example, nuclear materials. The relationship between the mineral content of a graphite sample and the ash content of that sample is unknown and is not determined by the application of this test method.

5. Interferences

5.1 Although permitted within the scope of this test method, the use of alumina ceramic crucibles may affect results due to difficulties in obtaining repeatable or proper weights, or both, because of (*1*) the hygroscopic nature of some ceramic crucibles, and (*2*) the possible chemical combination of trace elements with the ceramic crucible.

5.2 Any ash or trace elements introduced to the sample will influence results. Contamination can occur during drilling to obtain the sample and during pulverization. (See 7.1.)

6. Apparatus

6.1 *Alumina Ceramic or Platinum Crucible or Dish,* suitable for holding sample (subsequently called sample holder).

6.2 *Analytical Balance,* capable of weighing to ±0.0002 g.

6.3 *Muffle Furnace,* capable of reaching 950 °C with controller capable of maintaining a temperature of 950 °C ± 20 °C.

6.4 *Platinum or Stainless Steel Wire.*

6.5 *Desiccator,* charged with indicating desiccant.

6.6 *Drying Oven,* air convection type, capable of being controlled to 110 °C ± 2 °C.

7. Sampling

7.1 Samples may be solid or particulate. Solid bodies may be sampled by removing one or more solid pieces from the body by, for example, sawing, turning, milling, or fracturing. Particulate samples may be generated from solid bodies by drilling, using a carbide drill to minimize contamination, or by other crushing and grinding methods.

8. Procedure

8.1 Dry the sample in accordance with Test Method C562, or for a minimum of 16 h in a drying oven at 110 °C ± 2 °C, and allow the sample to cool to room temperature in the desiccator.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

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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.

***A Summary of Changes section appears at the end of this standard**

8.2 Tare a dried sample holder using an analytical balance to ±0.002 g. As soon as the sample has cooled to room temperature, remove it from the desiccator and weigh a 25 g to 50 g sample into the tared sample holder. Reweigh the sample and sample holder to ±0.002 g.

8.3 Introduce a slow stream of air into the muffle furnace (Note 1). Place the sample holder containing the sample into the furnace, then heat the furnace so that the sample reaches 500 °C in 1 h and 750 °C in 2 h.

NOTE 1—The stream of air into the furnace should be carefully adjusted so that the sample or ash will not be blown from the dish. The air stream may be admitted by leaving the door of the furnace open a slight amount. Exercise care when placing a sample into a warm furnace so that no sample material will be lost due to drafts.

8.4 To aid in oxidation, stir the sample periodically with the wire. The absence of black flecks indicates that ashing is complete; at that time raise the furnace temperature to 950 °C for 1 h.

8.5 Remove the dish containing the ash from the furnace, (see Note 1) and cool to room temperature in the desiccator. Following removal from the desiccator, weigh the dish with the ash as rapidly as possible.

8.6 Replace the sample in the furnace at 950 °C for 30 min and repeat the procedure prescribed in 8.5 until a constant weight is obtained to ±0.002 g.

9. Calculation

9.1 Calculate the percentage of ash as follows:

$$\text{Ash, } \% = [(C - A)/(B - A)] \times 100 \quad (1)$$

where:

A = weight of the sample holder,
B = weight of the sample holder and dried sample,
C = weight of the sample holder and ash.

10. Report

10.1 The report shall include the following:

10.1.1 Proper identification of the sample, and

10.1.2 Results obtained from at least two ash determinations and their averages.

11. Precision and Bias[3]

11.1 The precision of this test method was determined during a round-robin test among four laboratories testing samples split from a common sample which was ground to pass a No. 60 (250 µm) sieve. This sampling method was used for the round-robin to minimize variations among samples provided to participating laboratories.

11.2 On this sample the mean, standard deviation between laboratories and standard deviation within laboratories were, respectively:

Mean	0.7286 %
Between laboratories	0.0159 %
Within laboratories	0.0127 %

11.3 No bias was noted during this round-robin test; however, the method is empirical and there is no suitable reference material that can be used to determine the bias of this test method.

12. Keywords

12.1 ash; carbon; graphite

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C05-1008.

SUMMARY OF CHANGES

Subcommittee D02.F0 has identified the location of selected changes to this standard since the last issue (C561 – 91 (2010)$^{\varepsilon 1}$) that may impact the use of this standard. (Approved Jan. 1, 2016.)

(1) Added new Section 3, Terminology.