Designation: C 1179 – 91 (Reapproved 2005)

An American National Standard

Standard Test Method for Oxidation Mass Loss of Manufactured Carbon and Graphite Materials in Air[1]

This standard is issued under the fixed designation C 1179; 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 comparative oxidation mass loss of manufactured carbon and graphite materials in air.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 The test method determines mass loss characteristics of carbon and graphite articles in air as a function of temperature and time by subjecting standard size specimens to muffle furnace exposure and then comparing pre-test and post-test mass differentials.

3. Significance and Use

3.1 This test method is primarily concerned with the oxidation mass loss of manufactured carbon and graphite materials in air at temperatures from 371 to 677°C (700 to 1250°F).

3.2 The test method will provide acceptable results at preselected test temperatures that yield less than 10 % mass loss in 100 h. These results can be used to determine relative service temperatures.

4. Interferences

4.1 Results can be affected by materials released by the furnace walls or furniture. These materials may be present from previous oxidation test samples or other furnace use. It is recommended that the furnace be operated at 1093°C (2000°F) for 1 h prior to use for oxidation mass loss testing.

4.2 The validity of this test method depends upon the availability of oxygen to the test specimen. The door of the furnace shall be kept closed to maintain temperature control; however, the furnace door shall not be sealed, rendering the door airtight.

4.3 Due to factors beyond the scope of the test method that might cause significant differences, this test method is only useful for comparative results.

5. Apparatus

5.1 *Muffle Furnace*, with automatic temperature regulation with ±4°C (±7°F) precision. Furnace chamber volume 983 to 2458 cm^3 (60 to 150 $in.^3$).

NOTE 1—Commercially available muffle furnaces advertised to achieve ±11°C can be set up to achieve the necessary precision.

5.2 *Thermocouple*, K-type (chromel/alumel with Type 304 stainless steel sheath).

5.3 *Digital Thermometer*, for temperature readout.

5.4 *Crucible Tongs*.

5.5 *Glazed Crucibles*, flat bottom.[2]

5.6 *Quart Glass Tray,* or equivalent.

5.7 *Nylon Gloves*, lint-free.

5.8 *Paper*, lint-free.

5.9 *Analytical Balance*, with 0.001-g resolution.

5.10 *Desiccator*, charged with indicating desiccant.

6. Sample Preparation

6.1 All specimens are to be handled with lint-free nylon gloves or equivalent during all machining, handling, impregnation, and testing. Specimens should be kept free of contamination by placing them on clean nonmetallic surfaces during all operations. Once the specimens have been oxidized, direct handling is not recommended.

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

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[2] The sole source of supply of the crucible (Coors crucible 60048) known to the committee at this time is Coors Ceramics Co., 17750 W. 32nd Ave., Golden, CO 80401. 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.

6.2 Test specimens are to consist of right cylinders 25.4-mm diameter by 25.4 mm long ± 0.3 mm (1 in. by 1 in. ± 0.01 in.). Specimens are to be machined all over with carbide-tipped tools to achieve a surface roughness visually comparable to 32-µin. arithmetic average (AA).

6.3 Wipe specimens with lint-free paper to remove carbon machining dust.

6.4 Specimens and crucibles are to be clearly identified. To avoid sample contamination, use a carbide-tipped scribe to mark the specimens. Crucibles can be marked with ceramic ink.

6.5 Place each specimen to be tested on its side (cylindrical surface) in a dried, tared crucible. Specimens are to be kept in the same crucible for the test duration. Record the individual identification numbers for each specimen/crucible set.

6.6 Dry the specimen/crucible sets in a vented oven at 120 to 150°C (250 to 300°F) for a minimum of 2 h. Remove the specimen/crucible sets and cool in a desiccator for a minimum of 30 min to achieve room temperature.

7. Procedure

7.1 Before the running of this test method, the operator should practice the transferring of similar shaped specimens from furnace to desiccator. The specimen/crucible set should be transferred so that the specimen is exposed to the atmosphere for no longer than 30 s. The desiccator should be in the immediate vicinity of the furnace.

7.2 Preheat and stabilize the furnace to the designated test temperature as verified by a monitoring thermocouple extending 50 mm (2 in.) into the chamber from the rear furnace wall (see Fig. 1).

7.3 Just before testing, remove the specimen/crucible sets from the desiccator and immediately weigh on an analytical balance to the nearest 0.001 g. Record this data as pretest mass.

7.4 Place two preweighed replicate specimen/crucible sets on a quartz glass tray or equivalent and insert into the furnace chamber so that the monitoring thermocouple is approximately centered between the specimens. Specimens are to be 25 ± 6 mm (1 in. ± 0.24 in.) apart and centered on the monitoring thermocouple (see Fig. 1).

7.5 Monitor the thermocouple reading and maintain furnace chamber temperature within ±4°C (±7°F) of the designated test temperature. Record the temperature readings from the monitoring thermocouple on the data sheet for the following times for each test period:

7.5.1 Just before inserting samples into furnace,

7.5.2 1 h after start, and

7.5.3 Just before removing samples for weighing.

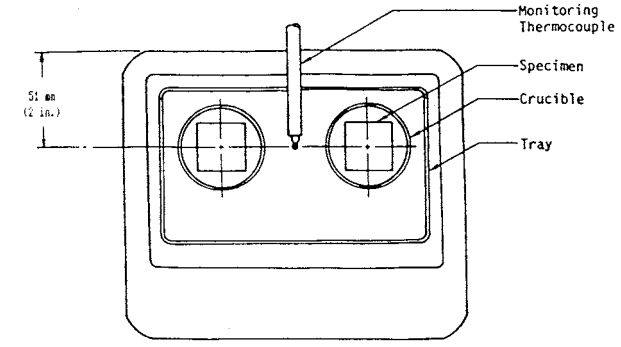
7.6 Remove the specimen/crucible sets from the furnace every 25 ±1 h, for a total test time of 100 h, ±15 min. Cool in a desiccator for a minimum of 30 min. Weigh the specimen/crucible set on an analytical balance to the nearest 0.001 g and record data as post-test mass for the specific test time interval at the designated test temperature.

7.7 Repeat 7.4 through 7.6 for 100 h total test duration.

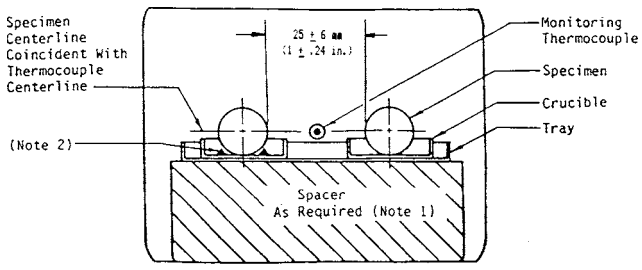
8. Calculation

8.1 Calculate the percent mass loss after each time interval and post test as follows:

$$\%\ \text{mass loss} = 100 \times \left[1 - \left(\frac{B - T}{A - T}\right)\right]$$



(a) Furnace Chamber—Top View



(b) Furnace Chamber—Cross Section View

NOTE 1—A medium weight insulating firebrick has been found to be satisfactory for this purpose.
NOTE 2—Small wedges of ceramic (broken) crucible pieces can be used to hold samples in place.

FIG. 1 Views of Furnace Chamber

where:
A = pre-test specimen/crucible set mass,
B = post-test specimen/crucible set mass, and
T = crucible tare mass.

9. Report

9.1 Report the following information:

9.1.1 Specimen identification, including material designation and process lot number.

9.1.2 Percent mass loss results for each specimen of 25, 50, 75, and 100 h at designated test temperatures.

10. Precision and Bias [3]

10.1 *Precision*—The precision of this test method was determined via a round robin test among six laboratories. Two different carbon materials were selected to verify the validity of the test method at lower and higher test temperatures. Material A, a carbon-graphite, was evaluated at 427°C (800°F) and Material B, an electrographite, was evaluated at 538°C (1000°F). All specimens of each material were from the same processing lot, with six specimens of each material evaluated by each laboratory. Evaluations were conducted in muffle furnaces from various manufacturers with chamber volumes ranging from 983 to 2458 cm^3 (60 to 150 $in.^3$).

10.2 The coefficient of variation for Material A within each laboratory was 0.1144 and between laboratories was 0.1140. For Material B, the coefficient of variation within each laboratory was 0.1804 and between laboratories was 0.4120.

10.3 Testing was deliberately conducted with different materials to demonstrate capabilities of the test method for both lower and higher temperatures. The data shows that more variability exists at the higher test temperatures. At these more reactive temperature regimes, furnace differences, temperature control variations, and the availability of oxygen must be carefully controlled to ensure consistent results.

10.4 *Bias*—No statement on bias can be made because the true value of the oxidation mass loss has not been established.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C05-1009.

11. Keywords

11.1 carbon; graphite; oxidation-air

APPENDIX

(Nonmandatory Information)

X1. OXIDATION TEST DATA SHEET

X1.1 Fig. X1.1 is an example of a typical report sheet for test data.

Test # _____
End Date _____
Initials _____

Specimens:

Conditions: _____°C in air, 100 hours total
_____(°F)

I. Data (mass in grams)

	25 hours	50 hours	75 hours	100 hours
Date/Time In				
Temperature - Before samples in				
Temperature - 1 hour after				
Temperature - Before samples out				
Date/Time Out				

Specimen Ident	Pos. In Furn.	Pre-Test Dish Tare(T)	Pre-Test Set wt.(A)	Post-test specimen/crucible set wts (B)			
	L						
	R						

Remarks:

II. Calculated Results (Percent mass loss at end of period)

$$\% \text{ mass loss} = 100 \times \left[1 - \left(\frac{B-T}{A-T}\right)\right]$$

Specimen Ident.	25 hours	50 hours	75 hours	100 hours
Average				

Conclusions:

FIG. X1.1 Sample Oxidation Test Data Sheet