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.

Designation: E2050 – 17

Standard Test Method for Determination of Total Carbon in Mold Powders by Combustion[1]

This standard is issued under the fixed designation E2050; 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 total carbon in mold powders in the concentration range from 1 % to 25 %.

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 test method has been evaluated in accordance with Practice E1601 and Guide E1763. Unless otherwise noted in the precision and bias section, the lower limit in the scope of each method specifies the lowest analyte content that may be analyzed with acceptable error (defined as a nominal 5 % risk of obtaining a 50 % or larger relative difference in results on the same test sample in two laboratories).

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.*

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]

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods (Withdrawn 2015)[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *mold powder, n— in the continuous-casting of steel,* a metallurgical flux used to provide lubrication of the mold, enhance heat transfer at the strand-mold interface, and provide thermal insulation of the liquid metal surface to prevent unwanted solidification.

3.2.1.1 *Discussion*—Key chemical components of these powders are fluorides, the oxides of silicon and calcium, and carbon.

4. Summary of Test Method

4.1 Carbon in the test sample is converted in a furnace to a mixture of carbon dioxide (CO_2) and carbon monoxide (CO) by combustion in a stream of oxygen. Full conversion of CO to CO_2 occurs by the passage of sample gases through a catalytic heater assembly. The amount of CO_2 is measured by infrared absorption. Any interference from halogens in the sample is eliminated by placement of a halogen trap between the furnace and the analyzer.

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[1] This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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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.

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TABLE 1 Statistical Information, Carbon Combustion/Infrared Method

Test Material	Number of Laboratories	Carbon Found, %	Minimum SD (S_M, E1601)	Reproducibility SD, (S_g, E1601)	Reproducibility Index (R, E1601)	$R_{rel\%}$
B	7	1.2046	0.00601	0.02960	0.08288	6.88
A	7	3.1219	0.01269	0.04843	0.13559	4.34
C	7	6.5514	0.04774	0.09215	0.25803	3.94
D	7	10.5121	0.05788	0.10579	0.29620	2.82
E	7	15.1121	0.04964	0.14730	0.41244	2.73
F	7	19.7121	0.11949	0.25294	0.70824	3.59
G	7	29.4250	0.38830	0.60179	1.6850	5.73

5. Significance and Use

5.1 This test method for the determination of total carbon in mold powders is primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use this test method will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that the work will be performed in a properly equipped laboratory and that proper waste disposal procedures will be followed. Appropriate quality control practices must be followed such as those described in Guide E882.

6. Rounding Calculated Values

6.1 Calculated values shall be rounded in accordance with Practice E29.

7. Interferences

7.1 Halogens, normally present in mold powders as fluoride, will interfere with this test method. A halogen trap, as described in 8.4, must be installed in the measure line between the furnace and analyzer to prevent this interference.

8. Apparatus

8.1 *Combustion-Infrared Absorption Carbon Analyzer,* equipped with a combustion chamber, oxygen carrier stream, halogen trap, catalytic heater assembly, and infrared absorption detector, suitable for the analysis of carbon from 1 to 25 % in mold powders. Instruments, such as those in Test Methods E1019, which can be shown to give equivalent results may also be used for this test method.

8.2 *Crucibles*—Use ceramic crucibles that meet or exceed the specifications of those recommended by the manufacturer of the instrument.

8.3 *Crucible Tongs,* capable of handling recommended crucibles.

8.4 *Halogen (Fluorine/Chlorine) Trap,* available from the instrument manufacturer as a kit, consisting of the parts and necessary reagents for assembly. Follow the manufacturer's instructions for the assembly, installation, use, and proper maintenance of the trap.

8.5 *Metal Scoop,* for dispensing metal chips (accelerators).

9. Reagents and Materials

9.1 *Iron Chip,* high purity.

9.2 *Oxygen,* 99.5 % purity recommended. Other grades of oxygen may be used if low and consistent blank readings are obtained.

9.3 *Tungsten/Tin or Copper Accelerator,* high purity.

10. Hazards

10.1 For precautions to be observed in the use of reagents and apparatus in this method, refer to Practices E50 and Test Methods E1019.

10.2 Use care when handling hot crucibles and when operating furnaces to avoid personal injury by either burn or electrical shock.

11. Sampling and Sample Preparation

11.1 *Materials Safety*—Samples must be prepared, stored, and disposed of in accordance with the materials and safety guidelines in Practices E50.

11.2 *Prepared Sample*—Pulverize or grind the laboratory sample until 100 % passes a No. 100 (150-µm) sieve. Store in a suitable glass or plastic container.

12. Preparation of Apparatus

12.1 Test the furnace and analyzer to ensure the absence of leaks. Prepare the analyzer for operation as directed in the manufacturer's instructions.

13. Calibration

13.1 *Calibration Reference Materials*—Tungsten carbide certified for total carbon in the vicinity of 6.1 % C and silicon carbide certified for total carbon in the vicinity of 29.4 % C.

NOTE 1—As originally approved, this standard relied on National Institute of Standards and Technology SRM 276b Tungsten Carbide and SRM 112b Silicon Carbide as calibration materials. Those SRMs have been discontinued. Certified reference materials are available from other sources.

13.2 *Determination of Blank:*

13.2.1 Enter a specific mass (for example, 1.0 g) into the software, as it is recommended by the instrument manufacturer, and follow the recommended procedure.

13.2.2 Add accelerators to the crucible. Type and amount of accelerators depend on sample matrix. Optimum parameters are recommended by the instrument manufacturer. Some manufacturers provide scoops that dispense a specific amount of accelerator. Once it is verified that the scoop delivers this approximate mass, it is acceptable to use this device for routine dispensing of accelerator.

13.2.3 Place the crucible on the furnace position and analyze in accordance with the manufacturer's instructions.

13.2.4 Repeat 13.2.1 – 13.2.3 a minimum of three times.

13.2.5 Enter the average blank following the routine outlined in the manufacturer's instruction manual.

13.3 *Calibration Procedure:*

13.3.1 Transfer 0.25 g of tungsten carbide calibration reference material, weighed to the nearest 1 mg, into a ceramic crucible and enter the mass into the software, following the instrument manufacturer's recommended procedure.

13.3.2 Add the same type and amount of accelerators, which are used in 13.2.2, to the crucible.

13.3.3 Place the crucible on the furnace position and analyze as directed in the manufacturer's instructions.

13.3.4 Repeat 13.3.1 – 13.3.3 a minimum of three times and calibrate the instrument following the calibration procedure as directed in the manufacturer's instruction manual.

13.3.5 Verify the calibration by analyzing the calibration reference material again. The obtained value shall agree with the certificate value within the range given by the published uncertainty or it shall agree within the limits of a prediction interval calculated using Eq 1. The prediction interval is defined as the range of values bounded by the analysis value $-p$ and the analysis value $+p$. If the prediction interval does not encompass the certified value, determine and correct the cause, and repeat calibration (Note 2). Either acceptance limit criterion is acceptable for routine operation.

NOTE 2—See the instrument manufacturer's instructions concerning the troubleshooting and correcting of errant calibration.

$$p = t \times \left(1 + \frac{1}{\sqrt{n}} \right) \times s \tag{1}$$

where:

p = magnitude of the one-sided, prediction interval,

n = number of replicates used in 13.3.4,

t = student's t variate chosen for a one-sided interval at the 95 % confidence level for degrees of freedom $df = (n - 1)$; a table may list this variate as t.95. The values are:

$t = 2.92$ for $n = 3$ $(df = 2)$

$t = 2.35$ for $n = 4$ $(df = 3)$

$t = 2.13$ for $n = 5$ $(df = 4)$

s = standard deviation of n replicates in 13.3.4 (Note 3).

NOTE 3—Here, s should be comparable to S_m, the repeatability standard deviation, given in Table 1. If s much greater than S_m, there is evidence that the repeatability of the particular instrument is not acceptable for use with this test method. The user should determine and correct the cause, and repeat 13.3.4 through 13.3.5.

13.3.6 Verify linearity of the instrument response by analyzing a silicon carbide (29.43 % total carbon) certified reference material. Results should fall within the uncertainties of the certificate value expected tolerance. If not, identify and correct the problem before proceeding with the analysis of samples.

14. Procedure

14.1 Assemble the apparatus and condition it as directed in Section 12.

14.2 *Sample Analysis:*

14.2.1 Weigh, to the nearest 0.1 mg, an appropriate mass of sample into a ceramic crucible, as follows:

Carbon, %	Approximate Sample Mass, g
1 - 6	0.25
6 - 10	0.20
10 - 20	0.15
20 - 25	0.075

Enter the sample mass into the software, following the instrument manufacturer's recommended procedure.

14.2.2 Add 1.000 g ± 0.005 g of accelerator and 1.000 g ± 0.005 g of iron chip to the crucible (see 13.2.2).

14.2.3 Place the crucible on the furnace position and analyze.

14.2.4 Analyze each sample in duplicate, recording each value.

14.2.5 Check instrument calibration (see 13.3.5) after every two sets of duplicate sample runs.

15. Calculation

15.1 Most commercially available instruments calculate the total carbon content directly, including corrections for blank and sample mass, so calculations by the analyst are not required. If, however, the analyzer does not compensate for blank and sample values, then use the following formula:

$$\text{Carbon, \%} = \frac{(A - B) \times C}{D} \tag{2}$$

where:

A = instrument reading for sample,

B = instrument reading for blank,

C = weight compensator setting, and

D = sample weight, g.

15.2 Round the results and report to two decimal places for values less than 10 % carbon, and to one decimal place for values equal to or greater than 10 % carbon. Examples: 4.15 %, 15.6 %.

16. Precision and Bias[4]

16.1 *Precision*—Seven laboratories cooperated in testing this test method. Table 1 summarizes the precision information. Table 2 displays the reproducibility expected for results at a number of carbon values.

16.2 *Bias*—The accuracy of this test method could not be evaluated because certified reference materials of mold powders were unavailable. Table 3 compares the means from this test method with results on the same materials by the combustion/gravimetric carbon test method. The differences show that the combined bias of both test methods does not exceed ±0.1 % carbon.

17. Keywords

17.1 carbon; combustion; infrared absorption; mold powders

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[4] A research report is available from ASTM International headquarters. Request RR:E01-1029.

TABLE 2 Predicted Reproducibility at Various Carbon Contents

Carbon, %	R	$R_{ref\ \%}$
0.5	0.079	15.7
1.0	0.083	8.3
2.0	0.109	5.4
5.0	0.178	3.6
10.0	0.329	3.3
20.0	0.645	3.2

TABLE 3 Comparison Between IR and Gravimetric Carbon

Test Material	IR Carbon, %	Gravimetric Carbon, %	IR - Gravimetric Carbon, %
B	1.205	1.30	–0.095
A	3.122	3.17	–0.048
C	6.551	6.57	–0.019
D	10.512	10.51	0.002
E	15.112	15.07	0.042
F	19.712	19.60	0.112

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