



Standard Test Method for Determination of Total Carbon in Mold Powders by Combustion-Infrared Absorption Method¹

This standard is issued under the fixed designation E 2050; 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 *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:

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications²

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals³

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials³

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory⁴

E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel and in Iron, Nickel, and Cobalt Alloys⁴

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method⁴

E 1763 Guide for Interpretation and Use of Results from the Interlaboratory Testing of Chemical Analysis Methods⁴

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E 135.

3.2 Definitions:

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 and carbon monoxide by combustion in a stream of oxygen. Full conversion of carbon monoxide to carbon dioxide occurs by the passage of sample gases through a catalytic heater assembly. The amount of carbon dioxide 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.

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 E 882.

6. Rounding Calculated Values

6.1 Calculated values shall be rounded in accordance with 3.4 to 3.6 of Practice E 29.

7. Interlaboratory Studies

7.1 This test method has been evaluated in accordance with Practice E 1601 and Guide E 1763. The lower limit in the scope of this test method specifies the lowest analyte content that may be analyzed with an acceptable error.

8. Interferences

8.1 Halogens, normally present in mold powders as fluoride, will interfere with this test method. A halogen trap, as described in 9.4, must be installed in the measure line between the furnace and analyzer to prevent this interference.

¹ This test method is under the jurisdiction of ASTM Committee E-1 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.

Current edition approved Nov. 10, 1999. Published February 2000.

² Annual Book of ASTM Standards, Vol 14.02.

³ Annual Book of ASTM Standards, Vol 03.05.

⁴ Annual Book of ASTM Standards, Vol 03.06.

9. Apparatus

9.1 *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 E 1019, which can be shown to give equivalent results may also be used for this test method.

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

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

9.4 *Halogen (Fluorine/Chlorine) Trap*⁵, 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.

9.5 *Metal Scoop*, for dispensing metal chips.

10. Reagents and Materials

10.1 *Iron Chip*, high purity (−6 to +20 mesh).

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

10.3 *Tungsten/Tin Accelerator*, high purity.

NOTE 1—Copper chip accelerator (−20 to +30 mesh) may be used in place of Tungsten/Tin.

11. Hazards

11.1 For precautions to be observed in the use of reagents and apparatus in this method, refer to Practices E 50 and Test Methods E 1019.

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

12. Sampling and Sample Preparation

12.1 *Materials Safety*—Samples must be prepared, stored, and disposed of in accordance with the materials and safety guidelines in Practices E 50.

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

13. Preparation of Apparatus

13.1 Test the furnace and analyzer to ensure the absence of leaks. Prepare the analyzer for operation in accordance with the manufacturer's instructions.

14. Calibration

14.1 *Calibration Reference Materials*—Tungsten carbide (6.10 % total carbon), NIST SRM 276b, or equivalent; silicon carbide (29.43 % total carbon), NIST SRM 112b, or equivalent.

14.2 Determination of Blank:

14.2.1 Enter 1.000-g weight into the weight stack, following the instrument manufacturer's recommended procedure.

14.2.2 Add 1.000 ± 0.005 g of accelerator and 1.000 ± 0.005 g of iron chip to the crucible.

14.2.3 Place the crucible on the furnace pedestal and analyze in accordance with the manufacturer's instructions.

14.2.4 Repeat 14.2.1-14.2.3 a minimum of three times.

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

14.3 Calibration Procedure:

14.3.1 Weigh ~ 0.25 -g tungsten carbide calibration reference material into a ceramic crucible and enter the weight into the weight stack, following the instrument manufacturer's recommended procedure.

14.3.2 Add 1.000 ± 0.005 g of accelerator and 1.000 ± 0.005 g of iron chip to the crucible.

14.3.3 Place the crucible on the furnace pedestal and analyze in accordance with the manufacturer's instructions.

14.3.4 Repeat 14.3.1-14.3.3 a minimum of three times and calibrate the instrument following the calibration procedure in accordance with the manufacturer's instruction manual.

14.3.5 Verify the calibration by analyzing the calibration reference material again. It should fall within the uncertainties of the certified value. If not, determine and correct the cause, and repeat 14.3.1-14.3.4.

14.3.6 Verify linearity of the instrument response by analyzing a silicon carbide (29.43 % total carbon) standard 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.

15. Procedure

15.1 Assemble the apparatus and condition it in accordance with Section 13.

15.2 Sample Analysis:

15.2.1 Weigh, to the nearest 0.1 mg, an appropriately sized sample into a ceramic crucible, in accordance with the following:

Carbon, %	Sample Weight, g
1 - 6	~ 0.25
6 - 10	~ 0.20
10 - 20	~ 0.15
20 - 25	~ 0.075

Enter the weight into the weight stack, following the instrument manufacturer's recommended procedure.

15.2.2 Add 1.000 ± 0.005 g of accelerator and 1.000 ± 0.005 g of iron chip to the crucible.

15.2.3 Place the crucible on the furnace pedestal and analyze.

15.2.4 Analyze each sample in duplicate, recording each value.

15.2.5 Check instrument calibration (see 14.3.5) after every 2 sets of duplicate sample runs.

16. Calculation

16.1 Most commercially available instruments calculate percent concentrations directly, including corrections for blank

⁵ Fluorine/Chlorine Trap Kit, Part Number 769-641, available from the LECO Corp., 820 Commonwealth Drive, Thornhill Industrial Park, Warrendale, PA 15086, has been found satisfactory for this purpose.

and sample weight, 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:

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

where:

A = DVM reading for sample,

B = DVM reading for blank,

C = weight compensator setting, and

D = sample weight, g.

16.2 Round the results and report to 2 decimal places for values less than 10 % carbon, and to 1 decimal place for values equal to or greater than 10 % carbon. Examples: 4.15 %, 15.6 %.

17. Precision and Bias ⁶

17.1 *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.

17.2 *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

TABLE 2 Predicted Reproducibility at Various Carbon Contents

Carbon, %	<i>R</i>	<i>R</i> _{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	0.329	3.3
20	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

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.

18. Keywords

18.1 carbon; combustion; infrared absorption; mold powders

⁶ A research report is available from ASTM headquarters. Request RR:E01-1029.

TABLE 1 Statistical Information, Carbon Combustion/Infrared Method

Test Material	Number of Laboratories	Carbon Found, %	Minimum SD (<i>S</i> _M , E 1601)	Reproducibility SD, (<i>S</i> _g , E 1601)	Reproducibility Index (<i>R</i> , E 1601)	<i>R</i> _{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

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).