



Designation: E1941 – 10

Standard Test Method for Determination of Carbon in Refractory and Reactive Metals and Their Alloys by Combustion Analysis¹

This standard is issued under the fixed designation E1941; 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 applies to the determination of carbon in refractory and reactive metals and their alloys in quantities from 20 μg to 500 μg . This corresponds to mass fractions ranging from 0.004 wt % to 0.100 wt % for a 0.5 g sample (see [Note 1](#)).

NOTE 1—Actual quantitative range might vary from manufacturer to manufacturer and according to sample mass. Samples of higher mass may allow for proportionally lower detection limits provided complete combustion of the sample is assured.

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.* Specific precautionary statements are given in [Section 9](#).

2. Referenced Documents

2.1 *ASTM Standards:*²

[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](#)

[E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method](#)

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see [Terminology E135](#).

4. Summary of Test Method

4.1 The metal specimen, contained in a single-use ceramic crucible, is ignited (combusted) in an oxygen atmosphere in an

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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.

induction furnace. The carbon in the specimen is oxidized to carbon dioxide or carbon monoxide, or both, and is eventually carried to the analyzer/detector. The amount of carbon present is electronically processed and is displayed by the analyzer readout.

4.2 This test method is written for use with commercially available analyzers equipped to carry out the above operations and calibrated using commercially available reference materials of known carbon content.

5. Significance and Use

5.1 This test method is intended to test for compliance with compositional specifications. It is assumed that all who use this 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.

6. Interferences

6.1 The elements ordinarily present in these alloys do not interfere. Halides that are present in some sponge type samples will cause low carbon recovery.

7. Apparatus

7.1 *Combustion Furnace and Measurement Apparatus*, automatic carbon determinator, consisting of an induction furnace; a dust/debris removal trap; an analytical gas stream purification system; an infrared detection system; and an automatic readout (see [Note 2](#)).

NOTE 2—Several models of commercial carbon determinators are available and presently in use in industry. Each has its own unique design characteristics and operational requirements. Consult the instrument manufacturer's instruction manuals for operational details.

7.2 *Oxygen Tank and Regulator*.

7.3 *Ceramic Crucibles and Lids*, that meet or exceed the instrument manufacturer's specifications. Use of lids is optional. If they are used, they should have holes in them.

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

7.5 *Balance*, capable of weighing to the nearest milligram.

7.6 *Furnace*, capable of reaching and sustaining a temperature of at least 700 °C.

8. Reagents

8.1 *Acetone* (A.C.S. Reagent, or other suitable, degreasing reagents).

8.2 *Copper Accelerator*, low carbon (see [Note 3](#)).

8.3 *High Purity Iron Chip Accelerator*, low carbon (see [Note 3](#)).

8.4 *Magnesium Perchlorate (Anhydrone)*, purity as specified by equipment manufacturer.

8.5 *Oxygen*, high purity (as specified by equipment manufacturer).

8.6 *Tin Accelerator*, low carbon (see [Note 3](#)).

8.7 *Tungsten Accelerator*, low carbon (see [Note 3](#)).

8.8 *Sodium Hydroxide on Clay Base*, commonly known as Ascarite III (purity as specified by equipment manufacturer).

8.9 *Reference Materials*, with known carbon content.

NOTE 3—The total carbon content of all accelerators used must be sufficiently low to allow for blanking as described in [Section 12](#).

9. Hazards

9.1 For hazards to be observed in the use of certain reagents and equipment in this test method, refer to [Practices E50](#).

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

10. Preparation of Apparatus

10.1 Make a minimum of two determinations to condition the instrument as directed in [Section 12](#) before attempting to calibrate the system or determine the blank.

10.2 *Crucible and Lid Preparation*—Commercially available crucibles and lids ([Note 4](#)) often contain levels of carbon sufficient to adversely affect results. To minimize this problem, crucibles and lids may be heat treated prior to use to remove contamination. Heating to 700 °C to 800 °C for at least 2 h or to 900 °C to 1000 °C for at least 1 h. has been determined to be effective. Other heating conditions may be specified if there is data supporting the effectiveness of the time and temperature used on removing contaminants. Remove the crucibles and lids from the furnace and allow them to cool (see [Note 5](#)). Use of a desiccator or other covered container for storage is recommended to minimize the potential for contamination. Handle prepared crucibles only with clean crucible tongs.

NOTE 4—The use of lids is optional. If they are used, they should be prepared and stored in the same manner as the crucibles. If they are not used, references to them in this standard may be ignored.

NOTE 5—Crucibles and lids must be used within a specified time period of being removed from the furnace. Four hours has been found to be an acceptable period, but a longer time may be specified by the test facility if supporting data are kept on file. If crucibles or lids, or both, are not used within the specified time period after removing them from the furnace, they shall be reheated as described in [10.2](#).

11. Sample Preparation

11.1 The sample selected shall be representative of the material to be analyzed.

11.2 Nibble, drill, shear, or machine a clean sample so that pieces are uniform in size and will fit into the ceramic crucible without extending over the rim.

11.3 If necessary, wash the pieces in acetone or other solvents ([Note 6](#)) to remove any oil, grease, or cutting fluid contamination. For heavier contamination, the sample may be washed in a soap solution or in a sonic cleaner, or both, and rinsed with a solvent. Decant the solvent and dry the sample. Care should be taken to ensure complete removal of solvents or cleaners, especially from porous samples, which may trap cleaning liquids, biasing results. Store the clean dried samples in a manner that minimizes the potential for contamination.

NOTE 6—Other solvents may be alternative organic solvents, inorganic solvents or inorganic acids that are capable of removing oil, grease or machining fluids.

12. Calibration

12.1 *Calibration Reference Materials*—The calibration reference materials (RMs) will consist of one or more commercial RMs of known carbon content (the high RM value should slightly exceed that of the unknown). Use appropriate accelerators, for example one scoop (approximately 1 g) of iron chip accelerator and one scoop (approximately 1.5 g) of copper accelerator ([Note 7](#)) in a prepared crucible, plus a prepared crucible lid.

NOTE 7—Users of simultaneous carbon-sulfur determinators should be aware that copper accelerator will have a negative effect on the sulfur result caused by the formation of copper sulfide. Other accelerator combinations that allow for complete combustion without the use of copper may be used if data supporting the effectiveness of the alternate accelerators is available. The combination of 3 parts iron, 3 parts tungsten, and 2 parts tin has been found effective for carbon and sulfur.

12.2 *Crucible Blank*—The crucible blank will consist of a crucible and lid prepared the same way as those used for samples, containing the same accelerator as that used for samples.

12.2.1 Prepare four crucible blanks as described in [12.2](#).

12.2.2 Follow the blank calibration procedure as detailed in the manufacturer's instruction manual, using at least 3 blanks.

12.2.3 Analyze one additional blank to verify the blank calibration. The blank value should be within 5 µg of the adjusted zero.

12.2.4 Prepare at least three specimens of a reference material for each calibration point as directed in [Section 11](#) and [12.1](#). Calibrate the instrument in a manner consistent with the instructions in the manufacturer's operating manual.

12.2.5 Prepare at least one additional RM specimen to validate the calibration. 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 8](#)). Either acceptance limit criterion is acceptable for routine operation.

NOTE 8—See the instrument manufacturer's instructions concerning the troubleshooting and correcting of errant calibration.