Designation: E 1941 – 98

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Test Method for Determination of Carbon in Refractory and Reactive Metals and Their Alloys[1]

This standard is issued under the fixed designation E 1941; 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 concentrations from 0.004 to 0.100 % (see Note 1).

NOTE 1—Actual instrument range might vary from manufacturer to manufacturer and according to sample size.

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 8.

2. Referenced Documents

2.1 *ASTM Standards:*

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications.[2]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[3]

E 55 Practice for Sampling Wrought Nonferrous Metals and Alloys for the Determination of Chemical Composition[3]

E 1601 Practice for conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method[4]

E 456 Terminology for Statistical Methods[2]

E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, Oxygen, and Hydrogen in Steel and in Iron, Nickel, and Cobalt Alloys.[4]

3. Summary of Test Method

3.1 The metal specimen, contained in a single-use ceramic crucible, is ignited in an oxygen atmosphere in an 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.

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

4. Significance and Use

4.1 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.

5. Interferences

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

6. Apparatus

6.1 *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.

6.2 *Oxygen Tank and Regulator*.

6.3 *Ceramic Crucibles and Lids*, that meet or exceed the instrument manufacturer's specifications. Use lids with holes in them.

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

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

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

7. Reagents

7.1 *Acetone (A.R.*, or other suitable, degreasing reagents).

7.2 *Copper Accelerator*, (low carbon).

7.3 *Iron Chip Accelerator*.

7.4 *Magnesium Perchlorate (Anhydrone)*..

---

[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 E01.06 on Titanium, Zirconium, Tungsten, Molybdenum, Tantalum, Niobium, Hafnium, and Rhenium.

Current edition approved Feb. 10, 1998. Published August 1998.

[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.

7.5 *Oxygen*, high purity (99.5 % minimum).

7.6 *Sodium Hydroxide on Clay Base (8-20 Mesh)*, commonly known as Ascarite III.

7.7 *Reference Materials*, commercially available RM's of known carbon content.

8. Hazards

8.1 For hazards to be observed in the use of certain reagents and equipment in this test method, refer to Practices E 50.

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

9. Preparation of Apparatus

9.1 Assemble the apparatus as recommended by the manufacturer.

9.2 Test the furnace and analyzer to ensure the absence of gas leaks. Make the required electrical power connections. Prepare the apparatus for operation in accordance with the manufacturer's instructions. Make a minimum of two determinations as directed in 12.2 before attempting to calibrate the system or determine the blank.

9.3 *Crucible Preparation*—Heat crucibles in a muffle furnace at 700 to 800°C for at least 2 h or at 900 to 1000°C for at least 1 h. Remove the crucibles from the muffle furnace and allow them to cool in a covered container (see Note 3). Handle prepared crucibles only with clean crucible tongs.

NOTE 3—If crucibles or lids, or both, are not used within 4 h of removing them from the muffle furnace, they should be reheated as described in 9.3 and 9.4.

9.4 *Crucible Lid Preparation*—Crucible lids should be preheated in a muffle furnace at 700 to 800°C for at least 1 h or at 900 to 1000°C for at least 30 min. Remove the crucible lids from the muffle furnace and allow them to cool in a covered container. Handle prepared lids only with clean crucible tongs.

10. Sample Preparation

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

10.2 Nibble, drill, or machine a clean sample in such a manner the particles are uniform in size and do not exceed a diagonal length of 10 mm.

10.3 Wash in solvent to remove any oil or grease contamination. Decant the solvent and dry the sample on the low heat of an electric hot plate. Store the clean dried samples in covered glass beakers or vials.

11. Calibration

11.1 *Calibration Reference Materials*—Whenever possible, select RM's with matrices similar to that of the unknowns: the calibration RM's will consist of a commercial RM's of known carbon content (standard value should slightly exceed that of the unknown), one scoop (approximately 1 g) of iron chip accelerator and one scoop (approximately 1.5 g) of copper accelerator in a prepared crucible, plus a prepared crucible lid.

11.2 *Crucible Blank*—The crucible blank will consist of one scoop (approximately 1 g) of iron chip accelerator and one scoop (approximately 1.5 g) of copper accelerator in a prepared crucible plus a prepared crucible lid.

11.3 *Calibration Procedure*

11.3.1 Prepare at least four 0.50 g specimens of a carbon RM as directed in Section 10 and 11.1. Also prepare four crucible blanks as described in 11.2.

11.3.2 Follow the calibration procedure as detailed in the manufacturer's instruction manual. Calibrate with at least three RM's and three crucible blanks.

11.3.3 Analyze a fourth carbon RM and a fourth crucible blank. The obtained value should be within the allowable limits of the certificate value and the blank value should be within 5 µg of the adjusted zero.

12. Procedure

12.1 Assemble the apparatus and condition it as directed in Section 9.

12.2 *Procedure for Operation*:

12.2.1 Make any pre-operational instrument checks as recommended by the instrument manufacturer.

12.2.2 Set the analyzer to the operate mode.

12.2.3 Prepare a 0.50 g specimen as directed in Section 10 and place it in a prepared crucible (see 9.3), add one scoop each of iron chip and copper accelerator and cover with a prepared lid (see 9.4) (see Note 4).

NOTE 4—Users of simultaneous carbon-sulfur determinators should be aware that copper accelerator may have a negative affect on the sulfur result.

12.2.4 Place the crucible plus specimen on the induction furnace pedestal and close the furnace.

12.2.5 Enter the specimen weight as recommended by the manufacturer. If specimen identification feature is provided by manufacturer, enter identification.

12.2.6 Start the analysis cycle, referring to the manufacturer's recommended procedure.

13. Calculation

13.1 The carbon reading (result) will be direct if the blank and specimen weight have been correctly entered in the appropriate portion of the analyzer (see Note 5).

NOTE 5—If the analyzer does not offer these functions, calculate the carbon content by the following:

$$\text{carbon, ppm} = (A-B)/C \quad (1)$$

where:

A = µg of carbon in specimen,
B = µg, of carbon in blank, and
C = specimen weight (in g).

14. Precision and Bias

14.1 *Precision*—Nine laboratories cooperated in testing 15 samples representing 7 different matrices. The data obtained is presented in Table 1. The testing and statistical analysis were performed according to the provision of Practice E 1601.

14.2 *Bias*—No information on the accuracy of this test method is available because no suitable certified reference materials were available when the interlaboratory test was performed.

TABLE 1 Carbon in Refractory and Reactive Metals and Their Alloys

Metal	Identity	Carbon found, ppm	Minimum SD (S_M, Practice E 1601)	Repro-ducibility SD (S_R, Practice E 1601)	Repro-ducibility Index (R, Practice E 1601)	$R_{rel\%}$
Zirconium	3500701	24	3	6	17	71
	660b	346	7	15	42	12
	SRM 360a	151	6	10	27	18
Titanium	#1	654	8	32	90	14
	#2	72	3	9	27	37
	SRM 176	132	6	19	52	40
Hafnium	420b	194	12	26	74	38
Niobium	752	19	3	7	19	100
	SP5943	327	4	13	36	11
Molybdenum	5657	22	4	7	20	90
	5654	38	3	10	27	72
Tungsten	C-6	56	3	10	28	50
	C-3	111	3	9	24	21
Tantalum	A	119	3	7	20	17
	B	37	3	8	22	60

Nine laboratories contributed results for all the samples included in this study.

15. Keywords

15.1 carbon content; refractory metals