Designation: E 1447 – 01

Standard Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity Method[1]

This standard is issued under the fixed designation E 1447; 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 hydrogen in titanium and titanium alloys in concentrations from 0.0010 to 0.0200 %.

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.* For specific precautionary statements, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

C 696 Test Methods for Chemical, Mass Spectrometeric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets[2]

E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials[3]

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals[4]

3. Summary of Test Method

3.1 The specimen, contained in a small, single-use graphite crucible, is fused under a flowing carrier gas atmosphere. Hydrogen present in the sample is released as molecular hydrogen into the flowing gas stream. The hydrogen is separated from other liberated gases such as carbon monoxide and finally measured in a thermal conductivity cell.

3.2 This test method is written for use with commercial analyzers equipped to carry out the above operations automatically and is calibrated using standard samples of known hydrogen 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 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.

5. Interferences

5.1 The elements ordinarily present in titanium and its alloys do not interfere.

6. Apparatus

6.1 *Fusion and Measurement Apparatus*—Automatic hydrogen determinator, consisting of an electrode furnace or induction furnace; analytical gas stream impurity removal systems; thermal conductivity cell hydrogen measurement system; and auxiliary purification systems (Note 1).

NOTE 1—The apparatus and analysis system have been previously described in Test Methods C 696, Sections 142 to 149. Several models of commercial analyzers are available and presently in use in industry. Each has its own unique design characteristics and operational requirements. Consult the instrument manufacturer's instructions for operational details.

6.2 *Graphite Crucibles*—The crucibles are machined from high-purity graphite. Use the size crucibles recommended by the manufacturer of the instrument.

6.3 *Crucible Tongs*—Capable of handling recommended crucibles.

7. Reagents and Materials

7.1 *Acetone*, low-residue.

7.2 *Sodium Hydroxide on Clay Base*, commonly known as Ascarite II.

7.3 *High-Purity Carrier Gas (99.99 %)*—Argon, nitrogen (Note 2).

NOTE 2—Carrier gases vary by instrument model and include both high-purity argon and nitrogen. Consult instrument manufacturer's instructions for proper gas selection.

7.4 *High-Purity Tin Metal (Low Hydrogen)*—Use the purity specified by the instrument manufacturer.

7.5 *Magnesium Perchlorate, Anhydrone*.

[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 E01.06 on Ti, Zr, W, Mo, Ta, Nb, Hf.

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[2] *Annual Book of ASTM Standards*, Vol 12.01.

[3] *Annual Book of ASTM Standards*, Vol 03.05.

[4] Discontinued; see *1997 Annual Book of ASTM Standards*, Vol 03.05.

7.6 *Molecular Sieve*—Characteristics specified by the instrument manufacturer.

7.7 *Schutze Reagent*—Iodine pentoxide over silica gel.

8. Hazards

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

8.2 Use care when handling hot crucibles and operating electrical equipment 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 and make the required electrical power and water connections. Prepare the apparatus for operation in accordance with the manufacturer's instructions. Make a minimum of two determinations using a specimen as directed in 12.2 before attempting to calibrate the system or to determine the blank.

10. Sample Preparation

10.1 Use solid form specimens prepared as directed in 10.2. Specimens must be of an appropriate size to fit into the graphite crucible and should not exceed 0.30 g in weight.

10.2 Cut the specimen to the approximate size of 0.15 to 0.30 g (preferably by shearing). If necessary, abrade specimen surfaces with a clean file to remove contamination. Rinse the sample in acetone, and air dry. Weigh to ±0.001 g. After cleaning and weighing, specimens must be handled with tweezers or forceps to prevent contamination.

11. Calibration

11.1 *Calibration Standards*—Select only titanium or titanium alloy standards (Note 3).

NOTE 3—Gas dosing: it is satisfactory to calibrate the unit by dosing known volume(s) of hydrogen gas into the detection system. If the instrument has this feature built in, refer to the manufacturer's recommended procedure. In this case instrument response must always be verified by analyzing titanium or titanium alloy standards.

11.2 *Determination of Crucible/Tin Blank Reading*:

11.2.1 If the instrument is equipped with an electronic blank compensator, adjust to zero, and proceed with the determination of the blank value.

11.2.2 Make at least three blank determinations as directed in 12.2 using the weight of tin flux as recommended by the instrument manufacturer (Note 4). Use a fresh crucible each time.

NOTE 4—Flux weight is dependent upon the model of the instrument and the manufacturer's instruction. Refer to the manufacturer's instructions and recommendations.

11.2.3 If the average blank value exceeds 0.0000 ± 0.0001 %, or a standard deviation for the three consecutive values exceeds ±0.0001 %, then determine the cause, make necessary corrections, and repeat 11.2.1 and 11.2.2 (Note 5).

NOTE 5—Refer to the instrument manufacturer's instructions concerning the troubleshooting and correction of blank determinations not meeting the above criterion.

11.2.4 Enter the average blank value in the appropriate mechanism of the analyzer (Note 6), and refer to the manufacturer's instruction. This mechanism will electronically compensate for the blank value.

NOTE 6—If the unit does not have this function, the average blank must be subtracted from the total result (see Note 9).

11.3 *Calibration Procedure*:

11.3.1 Prepare at least four 0.15 to 0.30-g specimens of a titanium hydrogen reference material as directed in 10.2. This titanium hydrogen reference material should have a hydrogen content greater than or approximately equal to the unknown samples within the scope of this test method (0.0010 to 0.0200 %).

11.3.2 Follow the calibration procedure recommended by the manufacturer. Analyze at least three standard specimens to determine calibration slope. Treat each specimen as directed in 12.2 before proceeding to the next one.

11.3.3 Confirm the calibration by analyzing the fourth titanium hydrogen reference material (Note 7). This value should be within the allowable limits of the certified value. If not, determine and correct the cause, and repeat 11.3.1 and 11.3.2 (Note 8).

NOTE 7—The accuracy of this test method is largely dependent upon the accuracy of the hydrogen values assigned to the reference materials and upon the homogeneity of these materials. Thus, wherever possible, the titanium hydrogen calibration standard should be a NIST Standard Reference Material or other certified reference material.

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

11.3.4 Confirm calibration linearity by analyzing a mid range titanium hydrogen reference material that should yield results within the allowable limits of the reference material. If not, determine the cause of non-linearity and repeat 11.3.1 through 11.3.4.

12. Procedure

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

12.2 *Procedure for Operation*:

12.2.1 Set the analyzer to operate mode.

12.2.2 Prepare a 0.15 to 0.30-g specimen as directed in 10.2.

12.2.3 Place a 0.15 to 0.30-g specimen in the loading device. If the instrument does not have this feature, refer to the manufacturer's recommended procedure regarding entry of sample.

12.2.4 Enter the sample weight as recommended by the manufacturer.

12.2.5 Place a crucible containing the equal weight of high-purity tin as measured in 11.2.2 on the furnace crucible pedestal assembly and close the furnace.

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

13. Calculation

13.1 The reading will be direct if the blank and weight have been entered correctly into the appropriate portion of the analyzer (Note 9).

NOTE 9—If the analyzer does not offer these functions, calculate the

hydrogen content by the following method: Dial the sample weight or a multiple of the sample weight on the weight compensator and use the following formula for the calculation of the result:

$$\text{Hydrogen, \%} = \frac{(A - B) \times C}{D} \quad (1)$$

where:
A = sample DVM reading,
B = blank DVM reading,
C = weight compensator setting, and
D = sample weight, g.

14. Precision and Bias

14.1 *Precision*[5]—Ten laboratories cooperated in testing three samples from NIST and one from the European Communities Community Bureau of Reference. The data are presented in Table 1. The testing and statistical analysis was performed in accordance with the provisions of Practice E 173.

TABLE 1 Hydrogen in Titanium Metal Statistical Information

Standard	Certified Value, µg/g H	Certified Precision, µg/g H	Interlaboratory Testing Results (10 Laboratories)		
			Average, µg/g H	R_1,[A] µ g/g H	R_2,[A] µg/g H
NBS 354[B]	215	6	215	6.3	11.7
NBS 353[B]	98	5	95	11.2	11.9
NBS 352[B]	32	2	28	2.7	6.8
BCR 318[C]	12.1	0.8	12.7	3.3	4.8

[A] $m = 1$; see Practice E 173.
[B] NBS Standard Series 352 to 354 supplied through the courtesy of Mr. J. I. Schultz of NIST. The series is no longer available commercially.
[C] BCR 318, a standard of the Commission of the European Communities, is available from Brammer Standard Co., Inc., Houston, TX 70069.

14.2 *Bias*—Information on the accuracy of this test method is incomplete at this time. The accuracy of this test method may be judged by comparing the results obtained from certified reference materials with their certified values for hydrogen.

15. Keywords

15.1 hydrogen; inert gas fusion; titanium; titanium alloys

[5] Supporting data are available from ASTM Headquarters. Request RR: E01.0600.