Designation: E 1447 – 05

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

1. Scope

1.1 This test method applies to the determination of hydrogen in titanium and titanium alloys in concentrations from 0.0006 to 0.0260 %.

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 9.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

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

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

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

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

E 1914 Practice for Use of Terms Relating to the Development and Evaluation of Methods for Chemical Analysis

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology E 135 and E 1914.

4. Summary of Test Method

4.1 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.

4.2 Alternately, hydrogen is converted to H_2O by passing the gas stream over heated copper oxide and subsequently measuring in an appropriate IR cell.

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

6. Interferences

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

7. Apparatus

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

NOTE 1—The apparatus and analysis system have been previously described in the Apparatus and Apparatus and Equipment sections of Test Methods C 696. 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.

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

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

[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] 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.

8. Reagents and Materials

8.1 *Acetone*, low-residue.

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

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

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

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

8.5 *Magnesium Perchlorate, Anhydrone*.

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

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

8.8 *Copper Oxide Wire*—To convert H_2 to H_2O in IR detection instruments. Characteristics specified by the instrument manufacturer.

9. Hazards

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

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

10. Preparation of Apparatus

10.1 Assemble the apparatus as recommended by the manufacturer.

10.2 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 13.2 before attempting to calibrate the system or to determine the blank.

11. Sample Preparation

11.1 Use solid form specimens prepared as directed in 11.2. Specimens must be of an appropriate size to fit into the graphite crucible and should not exceed 0.30 g in weight.

11.2 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.

12. Calibration

12.1 *Calibration Reference Materials*—Select only titanium or titanium alloy reference materials (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, refer to the manufacturer's recommended procedure. In this case instrument response must always be verified by analyzing titanium or titanium alloy reference materials.

12.2 *Determination of Crucible/Tin Blank Reading*:

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

12.2.2 Make at least three blank determinations as directed in 13.2 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.

12.2.3 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 12.2.1 and 12.2.2 (Note 5).

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

12.2.4 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 10).

12.3 *Calibration Procedure*:

12.3.1 Prepare at least four 0.15 to 0.30 g specimens of a titanium hydrogen reference material as directed in 11.2 (Note 7). 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 (0.0006 to 0.0260 %).

NOTE 7—The certificate of analysis may specify a minimum mass per specimen greater than 0.15 to 0.30 g. In that case use the quantity specified on the certificate but not to exceed 0.35 g. None of the reference material requirements used in the ILS fell outside of the .15 to 0.30 g range.

12.3.2 Follow the calibration procedure recommended by the manufacturer. Analyze at least three reference material specimens to determine the calibration slope. Treat each specimen as directed in 13.2 before proceeding to the next one (Note 8).

NOTE 8—Some instruments have expanded computer capabilities that allow multi-point calibration which may improve the accuracy and precision of the calibration over the single-point calibration methodology as tested in the current interlaboratory study.

12.3.3 Confirm the calibration by analyzing the fourth specimen of the titanium hydrogen reference material (Note 9). The ILS used an acceptance criterion where the value fell within the allowable limits of the certified value. An alternate procedure can be implemented where this value should agree with the certified value 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 12.3.1 and 12.3.2 (Note 10).

NOTE 9—Confirmation of the calibration does not ensure accuracy. The accuracy of this test method is largely dependent upon the absence of bias