Designation: E1447 – 22

Standard Test Method for Determination of Hydrogen in Reactive Metals and Reactive Metal Alloys by Inert Gas Fusion with Detection by Thermal Conductivity or Infrared Spectrometry[1]

This standard is issued under the fixed designation E1447; 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 reactive metals and reactive metal alloys, particularly titanium and zirconium, with mass fractions from 9 mg⁄kg to 320 mg⁄kg.

1.2 This method has been interlaboratory tested for titanium and zirconium and alloys of these metals and can provide quantitative results in the range specified in 1.1. It may be possible to extend the quantitative range of this method provided a method validation study, as described in Guide E2857, is performed and the results of the study show the method extension meets laboratory data quality objectives. This method may also be extended to alloys other than titanium and zirconium provided a method validation study, as described in Guide E2857, is performed.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazards, see Section 9.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E2857 Guide for Validating Analytical Methods

E2972 Guide for Production, Testing, and Value Assignment of In-House Reference Materials for Metals, Ores, and Other Related Materials

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology E135.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *drift correction, n*—a procedure for normalizing the instrument response to account for drift of sensitivity or blank, or both.

3.2.1.1 *Discussion*—A drift correction procedure is used to adjust the response of an instrument to maintain the viability of an existing calibration. A full correction of drift encompasses both the blank and the sensitivity. A full drift correction requires measurements of two points to establish correction factors, one for blank and one for sensitivity. A single correction for sensitivity may provide enough adjustment in instrument response to restore statistical control. Drift correction assumes the linearity of the instrument response has not changed.

4. Summary of Test Method

4.1 The specimen is added to a small, single-use graphite crucible with tin or nickel flux, or both, and fused under a flowing carrier gas atmosphere. Hydrogen present in the specimen is released as molecular hydrogen into the flowing gas stream.

[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, Re.

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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.

4.1.1 The detection of hydrogen is different based on the instrument manufacturer. Either hydrogen is isolated from nitrogen in a helium or an argon stream with a molecular sieve and measured by a thermal conductivity (TC) cell, or hydrogen is measured directly by a thermal conductivity cell using nitrogen as the carrier gas

4.1.2 For infrared detection, hydrogen is converted to water by passing the gas stream over heated copper oxide or other appropriate oxidizing reagent and subsequently measured in an infrared (IR) cell.

4.2 This test method is written for use with commercial instruments equipped to perform the above operations automatically and calibrated using reference materials (RMs) of known hydrogen content.

5. Significance and Use

5.1 This test method is intended for the routine analysis of reactive metals and reactive metal alloys to verify compliance with compositional specifications such as those specified by Committees B09 and B10. It is expected 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 zirconium and their alloys do not interfere.

7. Apparatus

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

7.1.1 Several models of commercial instrument are available and presently in use in industry. Each has unique design characteristics and operational requirements. Consult the manufacturer's instructions for operational details.

7.2 *Graphite Crucibles*—The crucibles are manufactured from high-purity graphite. Use crucibles specified for the model of instrument in use.

7.3 *Crucible Tongs*—Tongs or forceps capable of handling specified crucibles.

7.4 *Tweezer, Scoops, or Forceps*—For contamination-free sample handling.

8. Reagents and Materials

8.1 *Acetone,* reagent grade or equivalent. Equivalency is defined as the required purity documented by the laboratory to not bias test results.

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

8.3 *High-Purity Carrier Gas*—Carrier gases vary by instrument model and include high-purity argon, helium, and nitrogen. Use a gas that meets or exceeds the instrument manufacturer's specifications.

8.4 *Flux (Low Hydrogen) - Tin or Nickel Metal*—Use metal that meets or exceeds the instrument manufacturer's specifications. Flux may come in various forms, including chips, pellets, baskets, or capsules. Tin and nickel may be used individually or together.

8.5 *Drying Agent - Magnesium Perchlorate or Phosphorus Pentoxide.*

8.6 *Molecular Sieve*—Use material that meets or exceeds the instrument manufacturer's specifications. Not required for all measurement apparatus types - consult the manufacturer's literature for operational detail.

8.7 *Iodine Pentoxide Over Silica Gel,* commonly known as Schutze Reagent.

8.8 *Copper Oxide*—Use material that meets or exceeds the instrument manufacturer's specification.

8.9 *Copper*—Use material that meets or exceeds the manufacturer's specification. Not required for all measurement apparatus types.

9. Hazards

9.1 For hazards that may be encountered using this test method, refer to Practices E50, to the safety data sheets for reagents, and to the instrument manufacturer's literature.

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

10. Preparation of Apparatus and Measurement Conditions

10.1 Instrumentation shall be installed in a manner consistent with the manufacturer's recommendations.

10.2 Using the instrument software, create an analytical method with measurement parameters. Instrument manufacturers typically provide application notes with suggested measurement parameters. Refer to application notes as a starting point for method development. Measurement parameters that must be specified are detailed in 10.2.1 and 10.2.2. For implementation of this test method on an existing instrument that is currently producing analyses that meet laboratory data quality objectives, the performance of steps 10.3 – 10.6 may be unnecessary and is therefore optional.

10.2.1 *Instrument Measurement Parameters*

10.2.1.1 Gas flow.

10.2.1.2 Purge time.

10.2.1.3 Measurement times (integration time(s), delay time(s), ramp time(s)).

10.2.1.4 Furnace parameters.

10.2.1.5 *Calibration Protocol*—Only first order calibration models may be used with this test method. See Section 12 for calibration requirements. See Section 14 for further instructions on calibration line calculation.