Designation: E2792 – 11

Standard Test Method for Determination of Hydrogen in Aluminum and Aluminum Alloys by Inert Gas Fusion[1]

This standard is issued under the fixed designation E2792; 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 aluminum and aluminum alloys in concentrations from 0.05 mg/kg to 1 mg/kg.

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.*

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

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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

E1914 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 E135 and E1914.

4. Summary of Test Method

4.1 The specimen, contained in a high-purity graphite crucible, is heated to just below the melting point to drive off the surface hydrogen. The sample is then heated to just beyond the melting point under a flowing carrier gas atmosphere. Hydrogen present in the sample is released as molecular hydrogen into the flowing gas stream. The released hydrogen is separated from other liberated gases such as carbon monoxide and subsequently measured in a thermal conductivity cell.

4.2 Calibration is made using gas dosing with either helium or hydrogen or reference materials of known hydrogen content.

4.3 This test method is written for use with commercial analyzers equipped to carry out the above operations automatically.

5. Significance and Use

5.1 This test method is intended for the routine testing of aluminum and aluminum alloys to qualitatively determine the concentration of hydrogen in aluminum and aluminum alloys. It is not intended to verify compliance with compositional specifications because of the lack of certified reference materials. 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 aluminum and aluminum alloys do not interfere.

7. Apparatus

7.1 *Fusion and Measurement Apparatus*—Automatic hydrogen analyzer, 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. 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*, machined from high-purity graphite. Use the crucible design(s) recommended by the manufacturer of the instrument.

7.3 *Quartz Crucibles*, for analysis of steel reference materials on some instrument types. Use the crucible design(s) recommended by the manufacturer of the instrument.

[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 E01.04 on Aluminum and Magnesium.

Current edition approved Nov. 1, 2011. Published December 2011. DOI: 10.1520/E2791-11.

[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.

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

8. Reagents and Materials

8.1 *Acetone, Low-Residue*.

8.2 *Sodium Hydroxide on Clay* (Commonly known as Ascarite II).

8.3 *High-Purity Gas (99.99 %)*—Argon, nitrogen, and helium or hydrogen (Note 1).

NOTE 1—Carrier and dosing gases vary by instrument model and include high-purity argon, nitrogen, helium or hydrogen. Gas purity requirements shall be specified by the instrument manufacturer.

8.4 *Magnesium Perchlorate* (commonly known as Anhydrone).

8.5 *Molecular Sieve*, as specified by the instrument manufacturer.

8.6 *Schutze Reagent*—Iodine pentoxide on granular silica, purity as specified by the instrument manufacturer.

8.7 *Copper Wire*, to convert CO to CO_2 in thermal conductivity cell instruments. Characteristics should be specified by the instrument manufacturer.

8.8 *Glass wool*, used to pack reagents.

8.9 *OMI™ Purifier Tube*—Organolithium polymer used by some instruments to remove O_2, water vapor, CO, CO_2, most sulfur compounds, most halogen compounds, alcohols, and phenols to less than 10 ppb from the carrier gas.

9. Hazards

9.1 Refer to Practices E50 for potential hazards present when using this test method.

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 Provide the manufacturer's recommended electrical power and gas requirements. Prepare the apparatus for operation in accordance with the instrument manufacturer's recommendations.

10.3 Set the instrument to the operational mode in accordance with the instrument manufacturer's recommendations.

10.4 Test the furnace and analyzer to ensure that the gas stream meets manufacturer's requirements for acceptable leak rate.

10.5 Optimize the crucible pretreatment power settings (commonly called outgas), the surface and analysis power settings, crucible pretreatment time and analysis integration time for aluminum alloys.

10.5.1 Most manufacturers offer application guidance on appropriate settings to achieve optimum performance for aluminum alloys. Refer to this application guidance literature for assistance in determining optimum settings.

10.5.2 If the instrument is capable; optimize the crucible pretreatment time and power settings to achieve a stable blank (see 12.2.2).

10.5.3 If the instrument is capable; optimize the analysis time and power settings to obtain the optimum signal to noise ratio for the analysis of aluminum alloys.

10.5.4 It will not be necessary to optimize the analysis set-up routinely. Store the settings into the instrument hardware or software for routine use.

11. Sampling and Sample Preparation

11.1 Samples can be taken either from molten aluminum during casting or from the appropriate areas of finished product.

11.1.1 Samples from molten aluminum should be taken using the procedure described by Ransley and Talbot.[3] Briefly, a ladle is used to pour molten metal into a copper sampler that is designed to minimize porosity, cracks, voids, pits, and other defects that may lead to erroneously high hydrogen results.

11.1.2 *Samples from Cast or Finished Product*—Samples from cast or finished product should be taken from an area that represents the nominal concentration of hydrogen in the piece being sampled. Hydrogen may segregate in product and may also accumulate around defects sometimes making it difficult to obtain a representative sample. It is incumbent on the user to insure that the area selected for sampling is satisfactory. A cubical piece should be cut from the product using a saw with a clean blade. Carbide tipped blades are recommended. The size of the cube needed depends on the final sample size required for the instrument.

11.2 Samples must be of an appropriate size to fit into the graphite crucible. In general, the sample should be as close to the maximum size for the crucible as possible. A sample size of at least 4 grams is recommended. Smaller samples may be analyzed, however, the amount of hydrogen generated will be smaller and the detection limit will be higher. Smaller samples also have a higher surface to bulk hydrogen ratio and the method parameters may not be ideal for separating the surface hydrogen from the bulk hydrogen.

11.3 The sample should be machined using a lathe or milling machine to the manufacturers recommended specifications. A fine surface is important for obtaining accurate results. Rough surfaces may lead to excessively high surface readings and may, in extreme cases, cause high bulk results. Diamond tipped tool bits and use of alcohol lubricant during machining may be used to improve the surface finish. The average surface roughness for samples machined using a diamond tipped tool bit and alcohol lubricant is typically 40 micro inches to 50 micro inches. Surface area of the sample will increase as the surface roughness increases. Increased surface area will result in higher surface hydrogen readings and in extreme cases may affect the bulk hydrogen analysis.

11.4 Specimens must be handled with crucible tongs or in a manner that prevents surface contamination. Samples may be rinsed in acetone if surface contamination is suspected.

[3] C.E. Ransley and D.E.J. Talbot, "The Routine Determination of the Hydrogen Content of Aluminum and Aluminum Alloys by the Hot-Extraction Method". Journal of the Institute of Metals, Vol. 84, 1955-1956, 445.