



Designation: **E2792 – 11** **E2792 – 13**

Standard Test Method for Determination of Hydrogen in Aluminum and Aluminum Alloys by Inert Gas Fusion¹

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.

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.

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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.

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.

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

8. Reagents and Materials

8.1 *Acetone, Low-Residue. Acetone*—Reagent Grade or ACS certified grade or higher purity.

8.2 *Ethyl Alcohol*—Reagent Grade or ACS certified grade or higher purity.

8.3 *Isopropyl Alcohol*—Reagent Grade or ACS certified grade or higher purity.

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

8.5 *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.

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

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

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

8.9 *Copper Wire*, to convert CO to CO₂ in thermal conductivity cell instruments. Characteristics should be specified by the instrument manufacturer.

8.10 *Glass wool*, used to pack reagents.

8.11 *OMI™ Purifier Tube*—Organolithium polymer used by some instruments to remove O₂, water vapor, CO, CO₂, 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.