



Designation: E2792 – 21

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 mass fractions 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.3 *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](#)

[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 Develop-](#)

[ment and Evaluation of Methods for Chemical Analysis \(Withdrawn 2016\)](#)³

[E2857 Guide for Validating Analytical Methods](#)

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology [E135](#) and Practice [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 (CO) and subsequently measured in a thermal conductivity cell ([Note 1](#)).

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.

NOTE 1—The current ILS data was gathered from technology described in this test method. Other units and detection systems exist that are not represented here due to availability during the ILS. This test method provides guidance for appropriate use of these units, but users of other technologies should perform in-house method validation per [Guide E2857](#).

5. Significance and Use

5.1 This test method is intended for the routine testing of aluminum and aluminum alloys to quantitatively determine the mass fraction 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

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.

³ The last approved version of this historical standard is referenced on www.astm.org.

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, 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*—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 *NaOH on Clay*.

8.5 *High-Purity Gas (99.99 %)*—Argon, nitrogen, and helium or hydrogen (Note 2).

NOTE 2—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* ($MgClO_4$, commonly known as Anhydron).

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

8.8 *Schutze Reagent*—Iodine pentoxide (I_2O_5) on granular silica, purity as specified by the instrument manufacturer.

8.9 *Copper Wire*, to convert CO to CO_2 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 O_2 , water vapor, CO, CO_2 , most sulfur compounds, most halogen compounds, alcohols, and phenols to less than 10 ng/g 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 regularly optimize the analysis set-up. Store the settings into the instrument hardware or software for frequent 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.⁴ 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 mass fraction 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

⁴ Ransley, C. E., and Talbot, D. E. J., "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, p. 445.

ensure 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 mass of at least 4 g 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 ethyl alcohol or isopropyl 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, ethyl alcohol, or isopropyl alcohol if surface contamination is suspected.

12. Calibration

12.1 Calibration can be done by two different methods: gas dosing or by the use of certified reference materials.

12.2 Blank Determination Procedure:

12.2.1 Both gas dose and reference material calibration require an accurate blank determination. The blank value may be included in the calibration curve or subtracted from all subsequent determinations.

12.2.2 Make at least three blank determinations using an empty graphite crucible following the manufacturer's guidelines.

12.2.3 If the standard deviation of three consecutive blank determinations exceeds 0.005 mg/kg, or the blank readings are too high, repeat 12.2.2. 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 into the instrument software following the manufacturers recommended practice. The software will automatically compensate for the blank value in subsequent calibration and sample analysis.

12.3 *Calibration by Gas Dosing*—Prepare at least four gas dose determinations as directed by the instrument manufacturer. Gas doses should provide the equivalent mass of hydrogen greater than or approximately equal to the mass of

hydrogen from the highest mass fraction unknown samples within the scope of this test method, viz. *the total H in 4 g to 7 g quantities of aluminum containing 0.05 mg/kg to 1.0 mg/kg total H.*

12.3.1 Follow the gas dose calibration procedure recommended by the manufacturer. Some instruments have software capabilities that allow multi-point gas dose calibration which may improve the accuracy and precision of the calibration over the single-point calibration methodology. The gas dose procedure should be repeated before analysis of each group of samples or if the instrument has not been in use for 4 h or the carrier gas has been changed, or both.

12.4 *Calibration Using Reference Material*—Instrumentation should be calibrated using aluminum-matrix reference materials (Note 3). Refer to the manufacturer's application specifications for the power and time settings for each base metal. It is not necessary to perform the reference material calibration procedure each time that the analysis procedure is performed. However, the calibration must be verified when a different lot of crucibles is used or the system has not been in use for 4 h, or the carrier gas supply has been changed, or more than one of these are changed. Verification should consist of analyzing at least one aluminum control material or at least one steel, nickel, titanium or other reference material (12.5) and at least one linearity check reference material (12.6).

NOTE 3—With an increased availability of elevated H₂ in Al RMs available in the industry, instrument calibration with Al-matrix calibration reference materials is recommended.

12.4.1 Prepare at least four, 4 g or greater aluminum reference materials; or four approximately 1 g specimens of a steel, nickel reference materials; or four approximately 0.25 g of titanium reference material; or four other metal reference materials for each mass fraction level to be included in the calibration. The specimens must be of an appropriate size to fit into the quartz or graphite crucible. The steel, nickel, titanium or other metal reference materials should have mass fraction of hydrogen greater than or approximately equal to the unknown samples within the scope of this test method (0.05 mg/kg to 1.0 mg/kg).

12.4.2 Follow the calibration procedure recommended by the manufacturer. If the instrument is capable of this functionality, enter the mass fraction of the hydrogen content of the reference material into the instrument/software. Analyze at least three of the reference material specimens prepared in 12.4.1.

12.4.3 Use the average instrument response obtained for the reference materials to set the calibration slope in accordance with the manufacturer's recommendations.

12.4.4 Confirm the stability of the instrument/calibration by analyzing the fourth specimen of the hydrogen reference material (Note 4).

NOTE 4—Confirmation of the calibration does not ensure accuracy. The accuracy of this test method is largely dependent upon the absence of bias in the hydrogen values assigned to the reference materials and upon the homogeneity of these materials.

12.5 Confirm the calibration by analyzing a specimen of aluminum control material or with steel, nickel, titanium or