National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 2452

Hydrogen in Titanium Alloy
(Nominal Mass Fraction 60 mg/kg H)
(chip form)

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and the calibration of equipment used in the determination of hydrogen in titanium and titanium alloys. SRM 2452 consists of one bottle containing 10 g of titanium alloy chips.

Certified Mass Fraction Value: The certified value for hydrogen, expressed as a mass fraction, is provided in Table 1 [1,2]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been accounted for by NIST [3]. The certified value is based on inert gas fusion (IGF) measurements using standard additions calibration and IGF using calibration by dosing with pure hydrogen gas. The measurand is the total amount of hydrogen, and the certified value is metrologically traceable to the derived SI unit for mass fraction, expressed as milligrams per kilogram.

Table 1. Certified Mass Fraction Value for SRM 2452

Constituent	Value (mg/kg)	Expanded Uncertainty (mg/kg)
Hydrogen (H)	55.9	2.2

The uncertainty in the certified value for hydrogen is expressed as an expanded uncertainty, $U = ku_c$, calculated according to the methods in the ISO/JCGM Guides [2]. The quantity u_c represents, at the level of one standard deviation, the combined effects of the uncertainty due to variance among IGF measurement methods and material heterogeneity. The quantity $k = 2.0$ is the coverage factor used to obtain an approximate confidence level of 95 %.

Expiration of Certification: The certification of this SRM is valid until **30 April 2023**, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage, Handling, and Use"). The certification is nullified if the SRM is damaged, contaminated, or modified.

Coordination of the technical measurements leading to certification of this SRM was performed by R.R. Greenberg and J.R. Sieber of the NIST Chemical Sciences Division.

Maintenance of Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Statistical consultation was provided by J.J. Filliben and A.L. Pintar of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 08 June 2018
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR STORAGE, HANDLING, AND USE

A minimum sample mass of 100 mg should be used for analysis. When not in use, the bottle should be kept tightly capped. Use clean, dry, steel tools to sample material from the bottle and transfer it for weighing and measurement. Do not touch the metal with plastic or organic materials that may transfer hydrogen-containing compounds. The chips require no preparation prior to analysis. Store unused material in its tightly-capped original bottle in a cool, dry location.

PREPARATION AND ANALYSIS

Preparation of SRM 2452 was performed by R.M. Lindstrom and R.L. Paul of the NIST Chemical Sciences Division [4]. A total of 2.2 kg of titanium alloy (nominally 6 % Al and 4 % V) was taken from the same lot of chipped material used for SRM 173c. The material was divided in half for processing. Each portion was weighed and degassed at 700 °C in a vacuum system consisting of a quartz furnace and stainless-steel, high-vacuum components for 4 d to 5 d, by which time the hydrogen pressure shown by a residual gas analyzer was less than 10^{-4} Pa. After cooling to room temperature, a measured quantity of dry hydrogen was added to the system from a calibrated volume, and the material was heated to 500 °C. The reaction to incorporate the hydrogen as chemically bound species was completed by raising the temperature to 600 °C for 4 d to 5 d. After cooling, the residual hydrogen pressure was less than 10^{-7} Pa. The two batches were combined and passed through a 4.75 mm stainless-steel sieve. The material was then blended and bottled.

SRM 2452 was originally certified in 2005, using results from prompt gamma-ray activation analysis and the material preparation process described above, during which a known quantity of hydrogen was chemically bound into the alloy matrix. In 2013, the original certified value was questioned, and new analyses were initiated, using inert gas fusion methods performed at NIST and expert collaborating laboratories. The new work resulted in the new certified value reported in this certificate of analysis. Evidence suggests the material has been stable since preparation, and the original certified hydrogen value was biased high.

Analyses for certification were performed at NIST by J.R. Sieber of the NIST Chemical Sciences Division. Additional analyses were performed by the following laboratories: P.M. Cole, ATI Specialty Materials (Monroe, NC); J.F. Jeanson, Bureau Veritas Laboratories (Cergy-Pontoise, France); C. Chezy, Centre Régional d'Innovation et de Transfert de Technologie (Charleville-Mézières, France); L. Dilks. Laboratory Testing, Inc. (Hatfield, PA); M.J. Smith, ATI Flat Rolled Products (Natrona Heights, PA); K. Vonscio, Perryman Co. (Houston, PA); D. Tullis, Westinghouse Electric Co. (Ogden, UT); and C. Vancura, White Horse Technical Services (San Gabriel, CA).

REFERENCES

[1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI);* NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at https://www.nist.gov/pml/special-publication-811 (accessed June 2018).

[2] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement (*ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed June 2018); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at https://www.nist.gov/pml/nist-technical-note-1297 (accessed June 2018).

[3] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at https://www.nist.gov/srm/publications.cfm (accessed June 2018).

[4] Paul, R.L.; Lindstrom, R.M.; *Preparation and Certification of Hydrogen in Titanium Alloy Standard Reference Materials*; Metall. Mater. Trans. A, Vol. 43A, Issue 12, pp. 4888–4895 (2012).

Certificate Revision History: 08 June 2018 (Revised value and uncertainty estimate for hydrogen; title change; revisions to instructions for storage, handling, and use; change of expiration date; editorial changes); 25 April 2005 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 948-3730, email srminfo@nist.gov; or via the Internet at https:/www.nist.gov/srm.