UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-0001

DATE: 30 March 2015

Product Identifier

SRM Number: 2453a
SRM Name Hydrogen in Titanium Alloy (Nominal Mass Fraction 125 mg/kg H)

Under the U.S. Department of Labor, Occupational Safety and Health Administration (OSHA) 29 CFR 1910.1200, this Standard Reference Material (SRM) is NOT classified as a physical hazard or a health hazard, a simple asphyxiant, combustible dust, pyrophoric gas, or hazard not otherwise classified. There are no hazard pictograms, hazard statements or signal word associated with it. Safety Data Sheet information is not required. This document may be used in conjunction with your hazard communication program.

This material is formed to a specific shape or design during manufacture which under normal conditions of use does not release more than very small quantities, e.g., minute or trace amounts of a hazardous chemical (as determined under paragraph (d) of 1910.1200), and does not pose a physical hazard or health risk to employees.

Description: This SRM is intended primarily for use in evaluating chemical and instrumental methods for determination of hydrogen in titanium alloys. A unit of SRM 2453a consists of one bottle containing 10 g of pins having an approximate mass per pin of 0.10 g and approximate dimensions of 2.5 mm diameter and 4.5 mm length.

Disposal: SRM 2453a components and derived products should be disposed of in accordance with local, state, and federal regulations.

Transport Information: This material is not regulated by the U.S. Department of Transportation (DOT) and/or International Air Transportation Association (IATA).

Disclaimer: This document was prepared carefully, using current references. Users of this SRM should ensure that this document and the corresponding Certificate of Analysis in their possession are current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srmmsds@nist.gov; or via the Internet at http://www.nist.gov/srm.

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2453a

Hydrogen in Titanium Alloy

(Nominal Mass Fraction 125 mg/kg H)

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Certified Mass Fraction Value: A certified mass fraction value for hydrogen in SRM 2453a is provided in Table 1 [1]. 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 investigated or taken into account. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories. Value assignment categories are based on the definitions of terms and modes used at NIST for certification of chemical reference materials [2].

Table 1. Certified Mass Fraction Value for SRM 2453a Hydrogen in Titanium

Constituent	Mass Fraction (mg/kg)	95 % Coverage Interval (mg/kg)
Hydrogen (H)	126.8	124.3 to 129.3

The certified value is the estimated mean mass fraction of the element hydrogen in titanium alloys of all bottles of SRM 2453a. The certified value is metrologically traceable to the SI unit of mass, expressed as milligrams per kilogram. The estimate comes from fitting a statistical model to the measurements made directly on the SRM 2453a material using two test methods as well as quality control measurements made on similar reference materials. The Bayesian inference paradigm was used for statistical inference [3]. The expanded uncertainty is an interval calculated in a manner consistent with the ISO/JCGM Guide [4,5], and it expresses contributions from all recognized sources of uncertainty, including differences between analytical methods, differences among samples, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration functions, assay of primary materials, and balance calibration. The nominal coverage for the interval is 95 %.

Expiration of Certification: The certification of **SRM 2453a** is valid, within the measurement uncertainty specified, until **31 October 2034**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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

Coordination of the technical measurements leading to the certification of SRM 2453a was under the direction of J.R. Sieber of the NIST Chemical Sciences Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 27 April 2015

Robert L. Watters, Jr., Director
Office of Reference Materials

Measurements for value assignment of SRM 2453a were performed by R.L. Paul and D. Sahin of the NIST Chemical Sciences Division. Additional measurements were performed by C. Vancura, White Horse Technical Services, (San Gabriel, CA).

Statistical consultation for this SRM was provided by 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.

INSTRUCTIONS FOR USE

To relate analytical determinations to the assigned values on this Certificate of Analysis, a minimum sample quantity of 0.2 g is recommended. The material does not require preparation prior to weighing. The material should be stored in its original, tightly capped bottle in a cool, dry location. Keep a new bottle sealed in the aluminized polyester pouch until time of use. Use a clean, dry tool to handle the pins, and do not touch the pins with any material likely to contaminate the surface with moisture or hydrocarbon compounds.

PREPARATION AND ANALYSIS[(1)]

The material for SRM 2453a was obtained in the form of pins prepared by White Horse Technical Services using a proprietary process based on a process developed by NIST for the development of SRMs 2452, 2453, and 2454 [6]. The material was blended and bottled at NIST and sealed into aluminized polyester pouches. The starting material for preparation of SRM 2453a was a titanium alloy containing 6 % Al and 4 % V manufactured in wire form by Perryman Company (Houston, PA).

Homogeneity testing was performed at White Horse Technical Services using inert gas fusion with thermal conductivity detection following ASTM International E1447-09 Standard Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity/Infrared Detection Method [7]. Material heterogeneity was low and fit for the purpose of value assignment with the standard deviation of a single determination equal to 1.2 mg/kg H based on samples consisting of two pins each.

Quantitative analyses of the material for SRM 2453a were performed at NIST using prompt gamma-ray activation analysis (PGAA) with each sample consisting of three pins for a mass per sample of approximately 0.3 g. Quantitative analyses of the material for SRM 2453a were performed at White Horse Technical Services using inert gas fusion with thermal conductivity detection and gas dosing calibration. As required by ASTM E1447-09, each sample consisted of two pins for a mass per sample of approximately 0.2 g.

[(1)] Certain commercial equipment, instrumentation, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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 http://www.nist.gov/pml/pubs/sp811/index.cfm (accessed Apr 2015).

[2] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://www.nist.gov/srm/upload/SP260-136.PDF (accessed Apr 2015).

[3] Gelman, A.; Carlin, J.B.; Stern, H.S.; Dunson, D.B.; Vehtari, A.; Rubin, D.B.; *Bayesian Data Analysis*; 3rd ed., CRC Press (2014).

[4] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2015); 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 http://www.nist.gov/pml/pubs/index.cfm (accessed Apr 2015).

[5] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the "Guide to the Expression of Uncertainty in Measurement" - Propagation of Distributions using a Monte Carlo Method*; JCGM (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Apr 2015).

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

[7] ASTM E1447-09; *Standard Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity/Infrared Detection Method*; Annu. Book ASTM Stand., Vol. 3.05, West Conshohocken, PA (2009); available at www.astm.org (accessed Apr 2015).

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