National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 2454

Hydrogen in Titanium Alloy

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 alloy. A unit of SRM 2454 consists of one bottle containing 10 g of titanium alloy chips.

Certified Values: The certified value for hydrogen, expressed as 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 investigated or taken into account [3]. The certified value is based on cold-neutron prompt-gamma activation analysis (PGAA) and volumetric measurements of hydrogen.

Table 1. Certified Value (mass fraction)

Hydrogen:	211 mg/kg ± 4 mg/kg

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 Guide [2]. The quantity u_c represents, at the level of one standard deviation, the potential combined effects of the uncertainty due to variability both within and between PGAA and volumetric measurements (combined in quadrature) and material homogeneity. The quantity $k = 2$ is the coverage factor used to obtain an expanded uncertainty with an approximate confidence level of 95 %. The measurand is the total mass fraction of hydrogen. The certified value is metrologically traceable to the SI unit of milligrams per kilogram.

Expiration of Certification: The certification of **SRM 2454** is valid, within the measurement uncertainty specified, until **30 April 2023**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage and Use"). This 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 this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate this notification.

Coordination of the technical measurements leading to the certification of this SRM was performed under the direction of R.R. Greenberg of the NIST Chemical Sciences Division.

Preparation of SRM 2454 was performed by R.M. Lindstrom, and PGAA was performed by R.L. Paul, both of the NIST Chemical Sciences Division.

Statistical consultation was provided by J.J. Filliben 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: 20 February 2014
Certificate Revision History on Last Page

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

INSTRUCTIONS FOR STORAGE AND USE

A minimum sample mass of 200 mg should be used for analysis. When not in use, the bottle should be kept tightly capped.

PREPARATION AND ANALYSIS

A total of 2.8 kg of titanium alloy (nominally 6 % Al and 4 % V) was taken from the same lot of chipped material intended 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. The degassed material was weighed, returned to vacuum, and heated to 500 °C. A measured quantity of dry hydrogen was added to the system from a calibrated volume, and the system held at 500 °C for a day. After cooling, the residual hydrogen pressure was less than 10^{-7} Pa. The two batches were combined and passed through a No. 4 (4.75 mm) stainless-steel sieve. The material was then blended and bottled.

PGAA was used to determine the hydrogen concentration on six portions of the final material. The certified value is the sum of the residual concentration in the degassed blank material (measured by PGAA) and the quantity of hydrogen added as determined by measurements of the pressure, volume, and temperature.

REFERENCES

[1] 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 (1995).

[2] 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 Feb 2014); 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 Feb 2014).

[3] 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.; *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: http://www.nist.gov/srm/publications.cfm (accessed Feb 2014).

Certificate Revision History: 20 February 2014 (Extension of certification period; editorial changes); 03 May 2005 (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: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.