National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2453

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. SRM 2453 consists of 10 g of chips, each approximately 15 mg in size, contained in an amber glass bottle.

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 accounted for by NIST [3]. The certified value is based on cold-neutron prompt-gamma activation analysis (PGAA), neutron incoherent scattering (NIS) analysis, and volumetric measurements of H_2. The certified value is the sum of the residual concentration in the blank material (measured by PGAA and NIS) and the quantity of hydrogen added as determined by volumetric measurements

Table 1. Certified Value (mass fraction)
Hydrogen: 114 mg/kg ± 5 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 Guide [2]. The quantity u_c represents, at the level of one standard deviation, the potential combined effects of the uncertainty due to variability in PGAA, NIS, gravimetric and volumetric measurements, and material homogeneity. The quantity $k = 2$ is the coverage factor used to obtain an expanded uncertainty with an approximate confidence level of 95 %.

Expiration of Certification: The certification of this SRM is valid until **30 April 2014** within the measurement uncertainty specified, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Use"). This certification is nullified if the SRM is damaged, contaminated, or modified.

Maintenance of Certification: NIST will monitor representative portions from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Coordination of technical measurements leading to certification was performed under the direction of R.R. Greenberg of the NIST Analytical Chemistry Division.

Preparation of SRM 2453 was performed by R.M. Lindstrom of the NIST Analytical Chemistry Division. Prompt-gamma activation analysis and neutron incoherent scattering analyses were performed by R.L. Paul and H.H. Chen-Mayer, respectively, of the NIST Analytical Chemistry Division.

Statistical consultation was provided by J.J. Filliben of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 05 August 2005
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

INSTRUCTIONS FOR USE

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

PREPARATION AND ANALYSIS[1]

Twelve 10 cm × 30 cm sheets of Ti90/Al6/V4 alloy (Goodfellow) were cleaned, weighed, and degassed by heating to 800 °C in a vacuum system consisting of a quartz furnace and stainless-steel high-vacuum components. The degassed material was sampled by systematically punching from alternate sheets, then reweighed and returned to vacuum. A measured quantity of hydrogen was added to the system from a calibrated volume, and the material was heated to 400 °C. Reaction was completed by raising the temperature to 500 °C for 6 hours. Diffusion was encouraged by annealing at 400 °C overnight, then the material was cooled to room temperature, whereupon the pressure dropped to 10^{-2} Pa (10^{-7} atm). The sheets were reweighed and alternate sheets sampled again by punching. Analysis of these samples by cold-neutron prompt-gamma activation analysis (PGAA) and neutron incoherent scattering (NIS) indicated nonuniform loading, so the batch was chipped with an electric nibbler to a particle size of approximately 15 mg. Particles less than 0.425 mm were removed by shaking on a stainless steel sieve. The chipped material was cleaned by extraction with methanol, dried, blended, and bottled.

REFERENCES

[1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811, 1995 ed.; U.S. Government Printing Office: Washington, DC (1995).

[2] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; ISO: Geneva, Switzerland (1993); 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://physics.nist.gov/Pubs/.

[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.; *Definition of Terms and Modes Used at NIST for Value-Assessment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000).

Certificate Revision History: 05 August 2005 (This revision corrects the sample size description of the material); 06 January 2004 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

[1]Certain commercial equipment, instruments, or materials are identified in this certificate to specify adequately 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.