National Institute of Standards and Technology
U.S. Department of Commerce

Date of Issue:
10 December 2020

Standard Reference Material® 2454a
Hydrogen in Titanium Alloy
(Nominal Mass Fraction 215 mg/kg H)
(pin form)

CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical and instrumental methods for determination of hydrogen in titanium and its alloys. It can be used to validate value assignment of in-house reference materials.

Description: A unit of SRM 2454a 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 per pin.

Certified Value: The certified value is the estimated mean mass fraction of the element hydrogen in titanium for all bottles of SRM 2454a. A certified value is the present best estimate of the true value [1]. The certified value is metrologically traceable to the SI derived unit of mass fraction, expressed as milligrams per kilogram. The expanded uncertainty is an interval calculated for nominal 95 % coverage using a Bayesian multilevel consensus model [2] in a manner consistent with the ISO/JCGM Guide [3-4], and it expresses contributions from all recognized sources of uncertainty.

Table 1. Certified Mass Fraction Value for SRM 2454a Hydrogen in Titanium Alloy

Constituent	Mass Fraction (mg/kg)	95 % Coverage Interval (mg/kg)
Hydrogen (H)	216.0	207.6 to 224.4

Period of Validity: The certification of **SRM 2454a** is valid, within the measurement uncertainty specified, until **30 June 2039**, provided the SRM is handled and stored in accordance with the instructions given in this certificate. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM to the end of the period of validity. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

Storage: 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.

Use: To relate analytical determinations to the assigned value on this Certificate of Analysis, a minimum sample quantity of 0.1 g (1 pin) is recommended. The material does not require preparation prior to weighing. To use the uncertainty interval given in this certificate in comparisons and calculations, it is recommended to use an approximation to the combined standard uncertainty, u_c, equal to one fourth of the width of the 95 % coverage interval given in Table 1.

Source and Preparation: The starting material for the preparation of SRM 2454a was a titanium alloy containing 6 % aluminum, and 4 % vanadium. The material for SRM 2454a was obtained in the form of pins prepared by White Horse Technical Services (Temple City, CA), using a proprietary procedure based on a process developed by NIST for development of SRMs 2452, 2453, and 2454 [5]. The material was bottled at NIST and sealed into aluminized polyester pouches. Homogeneity testing was performed at White Horse Technical Services using inert gas fusion with thermal conductivity detection following ASTM International E1447-09(2016) [6]. Material heterogeneity was low and fit for the purpose of value assignment with the standard deviation of a single determination of hydrogen equal to 0.8 mg/kg based on samples consisting of two pins each.

Quantitative analyses of the material for SRM 2454a were performed at NIST and at White Horse Technical Services. At NIST, prompt gamma-ray activation analysis (PGAA) was performed with each sample consisting of a single 0.10 g pin. At White Horse, inert gas fusion with thermal conductivity detection and standard additions calibration with pure H_2 gas was performed with each sample consisting of a single 0.10 g pin.

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sharpless, K.E.; Sieber, J.R.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2020); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2020.pdf (accessed Dec 2020).

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

[3] 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 (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2020); 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 Dec 2020).

[4] 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*; Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Dec 2020).

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

[6] ASTM E1447-09(2016); *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 (2019).

Certain commercial organizations, services, equipment, or materials may be identified in this Certificate of Analysis 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 organizations, services, materials, or equipment identified are necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, Maryland 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

* * * * * * * * * * * End of Certificate * * * * * * * * * * *