



# White Horse Reference Materials

## Certificate of Analysis

### WHRM-TJa

**ISSUE DATE:** July 01, 2017

**REVISION DATE:** Feb 6, 2020

### H<sub>2</sub> IN TITANIUM ALLOY

**INTENDED USE:** This Certified Reference Material (CRM) is intended primarily for use as a calibrant for calibrating instruments used to determine the hydrogen content in titanium alloys. In addition, it can be used for the demonstration of in-control status of a measurement process over time, to check instrumental performance, validation of analytical methods, especially regarding trueness, including repeatability, and harmonization among laboratories.

This material is fully characterized with traceability to the SI according to ISO 17034 requirements for Reference Material Producers. **\*\*[Qualified for accredited laboratory use as taught in ASTM E2972 Section 6.5 and ISO 17025 (A.3.1) and associated documents for such use.]** Please consult our [FAQs](#) to learn how to incorporate this in your quality system.

**DESCRIPTION:** One bottle consists of ~100 titanium pins of approximately 2.5 mm diameter by 4.5 mm length with a nominal weight of 0.1 g.

#### Mass Fraction    Expanded Uncertainty

<b>Hydrogen (H)</b>	<b>131 mg/kg</b>	<b>3.3 mg/kg</b>
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**VALUE ASSESSMENT:** The measurand listed is the amount of the element contained in Grade 5 titanium traceable to the derived SI unit of mass fraction expressed as milligrams per kilogram (mg/kg) realized using inert gas fusion method WHRM ME1447a along with Prompt Gamma Activation Analysis (PGAA) for hydrogen under repeatability conditions. The estimated true value for hydrogen is based on a statistical evaluation of data supplied by inert gas fusion (IGF) utilizing a thermal conductivity detector at White Horse Reference Materials (WHRM) and the National Institute of Standards and Technology (NIST) utilizing Prompt Gamma-ray Activation Analysis (PGAA).

The uncertainty in the amount of hydrogen used for calibration and analysis using a Standard Additions (SA) method has been evaluated by careful analysis of the equation of state, its measured parameters, fundamental constants, measurement precision and sample mass. All other sources of uncertainty were found to be negligible. The evaluation of the equation of state and the estimators of the uncertainty of hydrogen used for calibration were derived through MC simulation. The reported expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor  $k = 2$  such that the coverage corresponds to approximately 95 %.

**MINIMUM SAMPLE QUANTITY:** To relate analytical determinations to the assigned value on this Product Information Sheet, a minimum sample quantity of 0.2 g is recommended for hydrogen analysis. The property values and their associated uncertainty are only guaranteed if minimum sample quantity is respected.

**HETEROGENEITY** Material hydrogen heterogeneity was low and fit for purpose for value assignment. Heterogeneity testing was performed at WHRM using inert gas fusion with thermal conductivity detection using a LECO hydrogen analyzer following ASTM E1447. ANOVA demonstrated that bottle-to-bottle

variance was indistinguishable from the overall standard deviation calculated from of 50 sample analyses (selected from 10 bottles for 5 repetitions consisting of two pins each.)

**PERIOD OF VALIDITY:** The estimated true value(s) are valid, within the measurement uncertainty specified, indefinitely, provided the RM is handled and stored in accordance with the instructions given in this document (see “Instructions for Handling and Use”). This document is nullified if the RM is damaged, contaminated, or otherwise modified.

**STORAGE INSTRUCTIONS:** The material should be stored in its original, tightly capped bottle in a cool, dry location.

**INSTRUCTIONS FOR HANDLING AND USE:** The material does not require additional preparation prior to weighing, if stored as outlined per instructions given in this document (see “Storage Information”). Use a clean, dry tool to handle the pins, and do not touch the pins with any material likely to contaminate the surface.

**MAINTENANCE OF RM DOCUMENT:** WHRM will monitor this RM over the period of validity. If substantive technical changes occur that affect the value assignment, WHRM will publish on its website the revised document and make an effort to notify the end user.

**PREPARATION AND ANALYSIS:** The material for WHRM-TJa was obtained in the form of pins prepared by White Horse Technical Services (WHTS) using a proprietary process. The material was blended and bottled at WHTS. The starting material for preparation of WHRM-TJa was a Grade 5 titanium alloy, manufactured in the form of wire by Perryman Company (Houston, PA).

Quantitative hydrogen analysis of the material for WHRM-TJa was performed at WHRM using inert gas fusion with thermal conductivity detection. As required by ASTM E1447, each sample consisted of two pins for a mass per sample of approximately 0.2g. Additional quantitative analysis was performed by NIST employing PGAA instrumentation. The values reported were statistically evaluated to achieve the estimated mg/kg value and Expanded Uncertainty. The WHTS SA test method result of 131 mg/kg agrees well with the mean result of 131.8 mg/kg from (PGAA). The expanded uncertainty at approximately 95 % level of confidence is 3.3 mg/kg, which is 2.5 % relative. The highest magnitude uncertainty components are associated with the slope and the intercept of the standard additions line. The relative standard uncertainty for the amount hydrogen used for SA is just 0.022 % or 0.049 mg/kg determined using Monte Carlo simulation.

**QUALITY ASSURANCE:** For quality assurance, SRM 2454 was analyzed by WHRM at the same time and showed no detectable bias with an analysis result of 210.6 mg/kg being very close to the certified value of 211 mg/kg.

WHRM certifies that TJa was produced and is maintained in a manner compliant with the requirements of ISO 17034 and associated documents. WHRM remains committed to a responsible adherence to best practices as developed and demonstrated in both public and private sector organizations and as set forth in international Reference Material Producer requirements.

**HEALTH AND SAFETY INFORMATION:** This product is Non-Hazardous in solid form.

**STATISTICAL ANALYST:**   
**Curtis Vancura, Owner**