



# White Horse Reference Materials

## Certificate of Analysis

### WHRM-TMa

**ISSUE DATE:** Oct 9, 2019

**REVISION DATE:** Oct 9, 2019

#### 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, including repeatability, and harmonization among laboratories.

**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

Hydrogen (H)	<b>216.2 mg/kg</b>	<b>2.5 mg/kg</b>
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**MASS FRACTION VALUE:** The values listed are the amount of analyte contained in Grade 5 titanium expressed as mass fraction realized using inert gas fusion method WHTS ME1447a along with Prompt Gamma Activation Analysis (PGAA) for hydrogen. 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.

**MINIMUM SAMPLE SIZE:** To relate analytical determinations to the assigned values on this Product Information Sheet, a minimum sample quantity of 0.2 g is recommended for hydrogen analysis, and 0.1g for nitrogen and oxygen analysis.

**PERIOD OF VALIDITY:** The statement of WHRM-TMa estimated value is 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.

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

**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”).

**STORAGE INFORMATION:** The material should be stored in its original, tightly capped bottle in a cool, dry location. Use a clean, dry tool to handle the pins, and do not touch the pins with any material likely to contaminate the surface.

**PREPARATION AND ANALYSIS:** The material for WHRM-TMa 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-TMa was a Grade 5 titanium alloy, manufactured in the form of wire by Perryman Company (Houston, PA).

H2 Heterogeneity testing was performed at WHRM using inert gas fusion with thermal conductivity detection in-house using a LECO hydrogen analyzer following ASTM E1447. Material hydrogen heterogeneity was low and fit for purpose for value assignment with the standard deviation of a single determination equal to 0.74 mg/kg. Hydrogen calculated from 50 sample analyses selected from 10 bottles for 5 repetitions consisting of two pins each.

Quantitative hydrogen analysis of the material for WHRM-TMa 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 216.2 mg/kg is nearly identical to the reported mean result of 216.4 mg/kg from (PGAA). The expanded uncertainty at approximately 95 % level of confidence is 2.5 mg/kg, which is 1.1 % 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.

#### **QUALITY ASSURANCE:**

For quality assurance, SRM 2454 was analyzed 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 TMa 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.

**INTERPRETATION OF VALUE ASSIGNMENT:** The value assignment, sigma and uncertainty derived during WHRM-TMa characterization of hydrogen in this material lot was performed in near ideal circumstances. A study in your laboratory with this material is advised to determine your process precision and bias.

**STATISTICAL ANALYST:** 

**TITLE:** Owner