Digital NIST pilot project: Transforming and Modelling Digital Reference Material Certificates

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Office of Reference Materials

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

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Acknowledgements

- **Pilot study participants:** Robert Hansich, James Fedchak, Steven Choquette, Catherine Rimmer, Dinis Camara, Catherine Cooksey, Katya Delak, Benjamin Long, Raymond Plante, Melissa Phillips, Jared Ragland, John Quintavalle, Manmohan Moondra, Gregory Cala, Damian Lauria, Jay Hendricks, *et al*.
- **PTB colleagues**
- **NIST Associate Director for Laboratory Programs**

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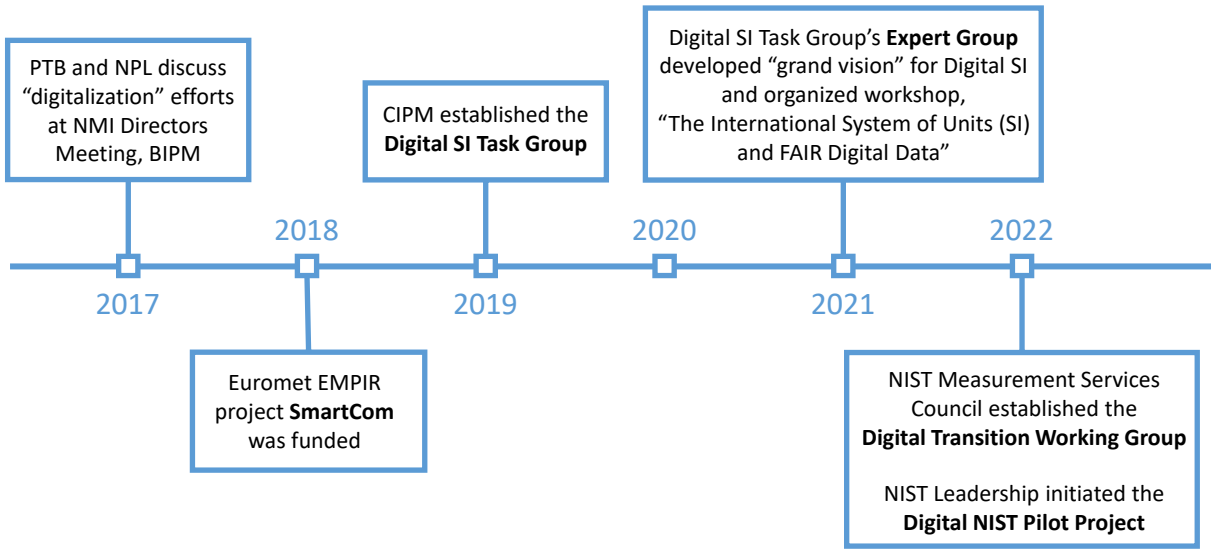
Movement for Digital Transformation of Metrology



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Digital Transformation

Joint Statement of Intent on the digital transformation in the international scientific and quality infrastructure

The joint statement is part of an ongoing initiative by the International Committee for Weights and Measures (CIPM) and its Task Group on the Digital SI (CIPM-TG-DSI) to develop and establish a world-wide uniform and secure data exchange format based on the International System of Units (SI).

Bureau International des Poids et Mesures

CIE INTERNATIONAL COMMISSION ON ILLUMINATION	IMEKO INTERNATIONAL MEASUREMENT CONFEDERATION
CODATA COMMITTEE ON DATA OF THE ISC	ISC INTERNATIONAL SCIENCE COUNCIL
IEC INTERNATIONAL ELECTROTECHNICAL COMMISSION	ISO INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ILAC INTERNATIONAL LABORATORY ACCREDITATION COOPERATION	OIML INTERNATIONAL ORGANIZATION OF LEGAL METROLOGY

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FAIR Data Principles

www.go-fair.org

Digital technologies will change the way we share metrological data

F **Findable:**
Described by sufficiently rich metadata and registered or indexed in a searchable resource

A **Accessible:**
through a well-defined and universally implementable protocol

I **Interoperable:**
use a formal, accessible, shared, and broadly-applicable language.

R **Reusable:**
rich metadata and documentation that meet relevant community standards.

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Digital NIST Pilot Project

Objectives:

- Evaluate existing DCC schema and its applicability to NIST Calibration Reports and Certificates of Analysis for Standard Reference Materials® (SRMs)
- Assess resources required to deliver digital data for NIST measurement services

Deliverables:

- Fully digital NIST calibration report and certificate of analysis
- Human-readable versions produced from the digital report and certificate

Digital Calibration Certificates and Certificates of Analysis - Project Charter

General Project Information	
Project Name	Digital Calibration Certificates and Certificates of Analysis
Processes Impacted	NIST measurement services
Expected Start Date	10/1/21
Estimated Completion Date	9/30/22
Funding Estimate	[redacted]
Sponsors	
Executive Sponsors	Eric Lin, ADLP Greg Strouse, ADLP
Sponsors	Steve Choquette, Office of Reference Materials Jim Fedchak, Physical Measurement Lab and Calibration Services Robert Hanisch, Office of Data and Informatics Katrice Lippa, Office of Weights and Measures Kate Rimmer, Material Measurement Lab
Program/Project Manager	
Program/Project Manager	Kate Rimmer
Email Address | Phone Number	TBD@nist.gov | 240-426-4492
Organizational Unit	MML

Vision, Mission, Scope, Deliverables, and Success Criteria	
Vision and Mission	This project supports the modernization of NIST Measurement Services by evaluating existing Digital Calibration Certificates (DCCs) and assessing their applicability to NIST SRM Certificates of Analysis and NIST Calibration Reports. It is likely that some extensions to existing DCCs, such as those from the PTB, will be required to support NIST practices. The PTB DCC schema is encoded in XML and we will also evaluate whether a JSON implementation better suits NIST SRM and calibration services customers.
Scope	For both calibration reports and certificates of analysis, 3—5 samples will be chosen that represent the range of complexity, from a single measurand to more extensive measurands and parameters. A known shortcoming of PTB DCCs is that

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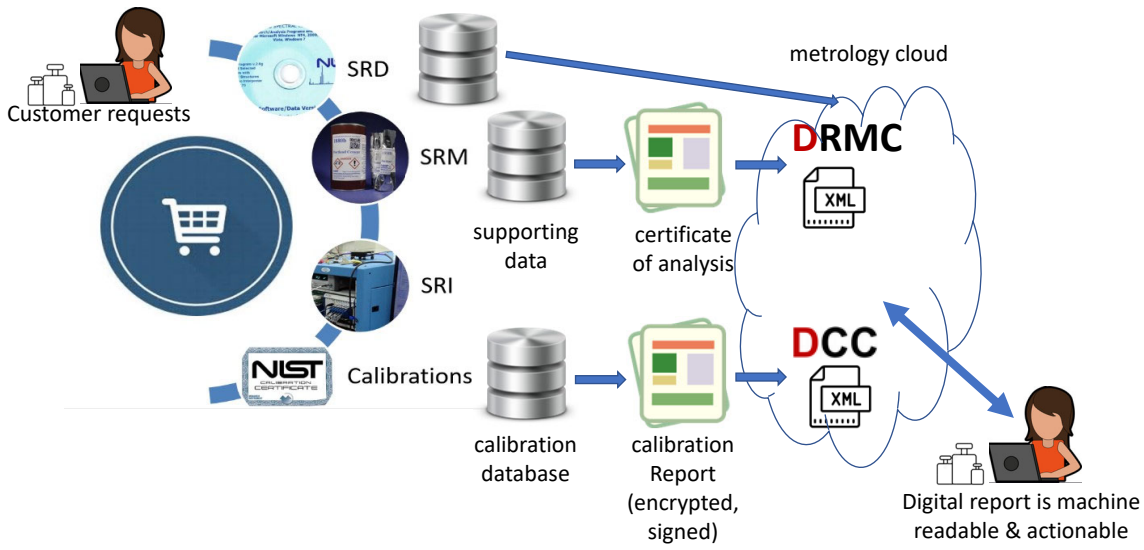
Digital NIST



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What is a Reference Material?

ISO 17034

Reference Material

material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process

Certified Reference Material

reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a reference material certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

NIST CRM are called **Standard Reference Material® (SRM)**

- Has at least one **certified value**
  - Highest confidence
  - Complete uncertainty analysis

NIST Reference Material (RM)

- Has at the most only non-certified values
- May just be a material, homogeneous and stable with respect to a specific measurand

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NIST SRM Overview

>1100 SRM Products

- Ferrous Metals
- Nonferrous Metals
- Microanalysis
- High Purity Materials
- Health and Industrial Hygiene
- Inorganics
- Primary Gas Mixtures
- Fossil and Alternative Fuels
- Organics
- Food and Agriculture
- Geological Materials and Ores
- Ceramics and Glasses
- Cement
- Engine Wear Materials
- Forensics
- Ion Activity
- Polymeric Properties
- Thermodynamic Properties
- Optical Properties
- Radioactivity
- Electrical Properties
- Metrology, Liquids and Glasses
- X-Ray Diffraction
- Sizing
- Surface Finish
- Fire Research
- Nanomaterials
- Miscellaneous Performance Engineering Materials

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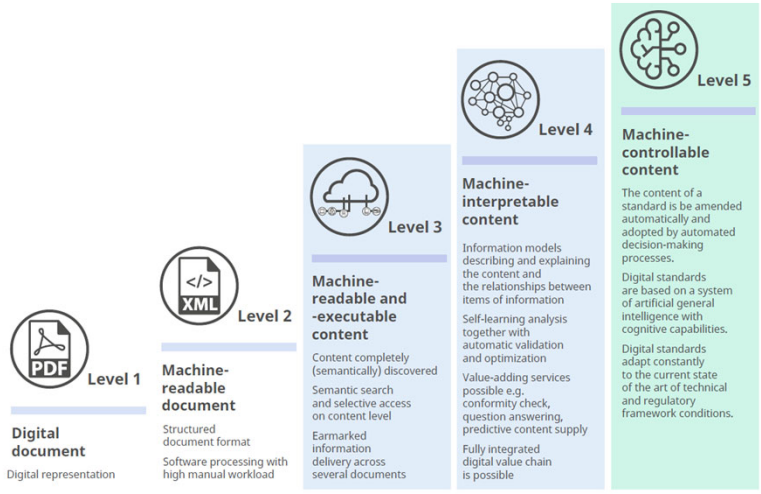
Where are we?



IDIS Whitepaper, “SCENARIOS FOR DIGITIZING STANDARDIZATION AND STANDARDS”, 2021

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Models, Modeling, and Interactions

What is a "model"?

- A **model**
  is a representation (definition, description, specification) of what something is and does
- Like a **theory**, it can, ideally,
  be used to predict and think about its behavior (life processes) from one moment to the next

We focus on models of data and process for system implementation.

- However, these concepts apply to any other kind of modeling (knowledge development, research) activity
  And this will be important for the task of translating knowledge from one representation to another

What makes the story of models meaningful is that they are part of a knowledge lifecycle

- A process wherein we learn as much as possible about something and organize all that knowledge in one place

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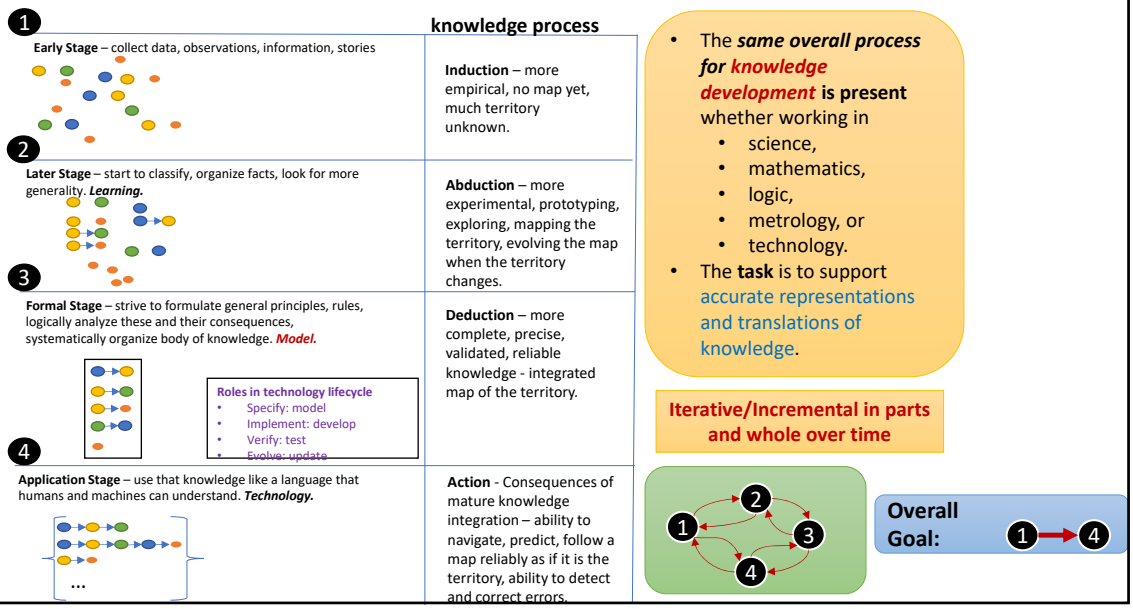
View of Models and Modeling: Knowledge Lifecycle



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What we observe here

The digital transformation process at work

- In one metrological organization (NIST), a part of the global metrological eco-system
- With complex, variable metrological data
- Performing incremental modeling/translation of data and workflow into digital infrastructure

Important considerations for anyone engaging this kind of process

- **Individual teams sorting out specialized data/modeling considerations**
  (i.e., exploring what data structures/types best represent the data, and how to organize that to meet a number of objectives)
- **Searching for unifying abstractions across them all**
  (i.e., looking for patterns that illuminate and unify the most essential aspects of operations)
- **Individual, distributed components of infrastructure coming into being**
  (e.g., prototyping tools, editors, data, platforms, etc., that can work together in standalone and distributed processes)
- **Needs for synchronization and graceful evolution throughout this developmental phase**
  (i.e., organizing teams and processes to align and integrate data with evolving schemas)
- **Needs for pushing into the deeper, broader context**
  (i.e., observing how these pieces begin to connect together both within and between organizations)

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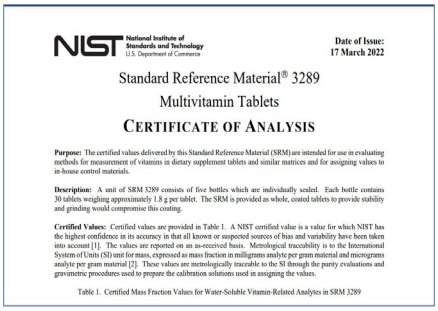
SRM Certificates and Modeling


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

Date of Issue:
17 March 2022

Standard Reference Material® 3289

Multivitamin Tablets

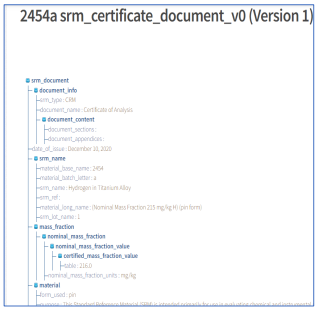
CERTIFICATE OF ANALYSIS

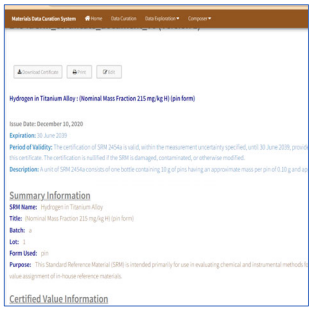
Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for use in evaluating methods for measurement of vitamins in dietary supplement tablets and similar matrices and for assigning values to in-house control materials.

Description: A unit of SRM 3289 consists of five bottles which are individually sealed. Each bottle contains 30 tablets weighing approximately 1.8 g per tablet. The SRM is provided as whole, coated tablets to provide stability and grinding would compromise this coating.

Certified Values: Certified values are provided in Table 1. 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 and variability have been taken into account [1]. The values are reported on an as-received basis. Metrological traceability is to the International System of Units (SI) unit for mass, expressed as mass fraction in milligrams analyte per gram material and micrograms analyte per gram material [2]. These values are metrologically traceable to the SI through the purity evaluations and gravimetric procedures used to prepare the calibration solutions used in assigning the values.

Table 1. Certified Mass Fraction Values for Water-Soluble Vitamin-Related Analytes in SRM 3289





Original RM Certificate → XML data → Human readable RM Certificate

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What Goes in a DRMC

Current Draft of Schema

- Organization Identification
- Reference Material Identification
- Material Information
  - Unit Description
  - Usage Information
  - Measurement Information
  - Additional Information
- Context
- Provenance
- References

Based On:

ISO 17034

ISO Guide 31

NIST Quality Manual

NIST Policy

Customer Input

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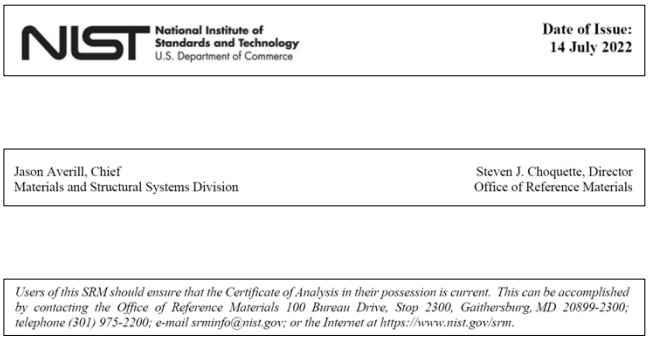
Organization Identification

- **Information to identify organization(s) responsible for creating the material**
  - Organization Name
  - Organization Type
  - Contact Details
  - Approving Official & Role



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

Date of Issue: 14 July 2022

Jason Averill, Chief
Materials and Structural Systems Division

Steven J. Choquette, Director
Office of Reference Materials

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, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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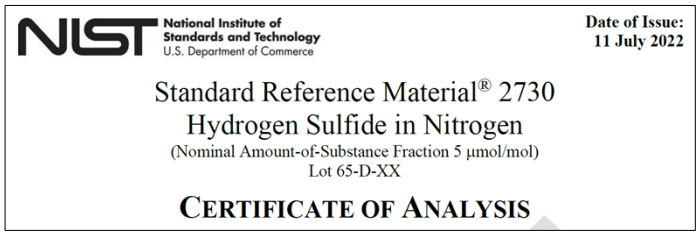
Reference Material Identifier

- **Information to uniquely identify the material in a catalog**
  - Number
  - Batch
  - Lot
  - Serial Number
  - Document Type
  - Title
  - Version Number


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

Date of Issue: 11 July 2022

Standard Reference Material® 2730
Hydrogen Sulfide in Nitrogen
(Nominal Amount-of-Substance Fraction 5 µmol/mol)
Lot 65-D-XX

CERTIFICATE OF ANALYSIS

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Distribution

Homogeneity applying across multiple units

- **One certificate applies to multiple units**
- **Certificate is publicly available**

Homogeneity applying to one unit

- **Each unit has its own certificate**
- **Certificate is only available to the purchaser**

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Material Information

- **Information about the material and its properties**
  - Unit Description
  - Usage Information
  - Measurement Information
  - Additional Information

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Material Information

- **Unit Description**
  - How many parts make up the reference material
  - Unique identifier for each part
  - How the parts are packaged
  - How much material is provided

Description: A unit of SRM 3148a consists of five 10 mL sealed borosilicate glass ampoules of an acidified aqueous solution prepared gravimetrically from high-purity scandium oxide to contain a known mass fraction of scandium. The solution contains nitric acid at a concentration (molarity) of approximately 1.6 mol/L.

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Material Information

- **Usage Information**
  - Storage information
  - Handling information
  - Specific instructions when using the material

INSTRUCTIONS FOR STORAGE AND USE

Storage: The SRM should be stored in its original bottle, tightly sealed and away from sunlight and intense sources of radiation, under normal laboratory conditions.

Use: Before it is sampled, the unit should be thoroughly mixed by carefully inverting and rotating the tightly sealed bottle. A minimum test portion mass of 30 mg for aluminum, calcium, chlorine, chromium, copper, iron, magnesium, manganese, potassium, silicon, sodium, strontium, sulfur, titanium, vanadium, and zinc; 100 mg for barium and scandium; 200 mg for antimony, arsenic, boron, bromine, cerium, cesium, cobalt, europium, rubidium, selenium, and uranium; 250 mg for mercury; 400 mg for nickel; and 750 mg for hydrogen should be used for analytical determinations.

Drying Instructions: To relate measurements directly to the certified and reference values, which are expressed on a dry-mass basis, users should determine a drying correction at the time of the analysis. The correction is determined by oven-drying a separate 1 g sample in a nitrogen atmosphere at 107 °C ± 3 °C to a constant mass [3] or equivalent technique. Attainment of constant mass is defined according to the ASTM thermogravimetric (TG) method as either a mass loss of ≤ 0.05 %, relative, over a nine-minute period or the mass loss after one hour of heating [3]. At NIST, the mass losses determined in both manners, and in both nitrogen and air, were similar.

The mass loss determined in both a nitrogen and air atmosphere, which is reported ***for information purposes only***, was nominally 2 %. The mass loss determined by the user may be different, depending on ambient conditions when the bottle is sampled.

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Material Information

- **Measurement Information**
  - Period of Validity
  - Traceability
  - Measurand
  - Values
  - Units
  - Uncertainties
  - Type of value (Certified vs. Non-Certified)
  - Measurement Procedures

Expiration of Certification: The certification of **SRM 2940a** is valid, within the measurement uncertainty specified, until **01 September 2028**, provided the SRM is handled and stored in accordance with the instructions given here (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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Values

- **Single Value with uncertainty**
- **Multiple values and uncertainties**
- **DNA sequences**
- **Information about absence of a measurand (e.g., < 0.005 g)**
- **Other**

Flexibility in delivering values in the units preferred by our stakeholders

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

Table 1. Certified Human mtDNA Sequence Differences from the Revised Cambridge Reference Sequence for SRM 2932 Component CHR.

Site	rCRS	CHR	Comment
64	C	Y = CT	C/T heteroplasmy
73	A	G	
195	T	C	
204	T	C	
207	G	A	
263	A	G	
309.1		C	insertion
315.1		C	insertion
709	G	A	
750	A	G	
1438	A	G	
1719	G	A	

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Material Information

- **Additional Information**
  - Figures
  - Pictures
  - Formulas
  - Other

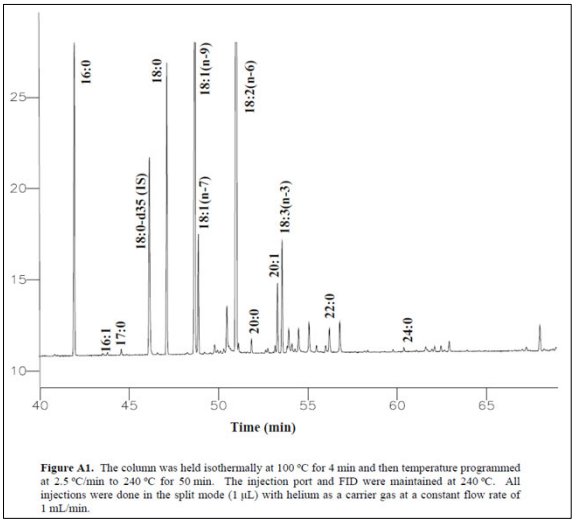

Figure A1. The column was held isothermally at 100 °C for 4 min and then temperature programmed at 2.5 °C/min to 240 °C for 50 min. The injection port and FID were maintained at 240 °C. All injections were done in the split mode (1 µL) with helium as a carrier gas at a constant flow rate of 1 mL/min.

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Context

- **Information related to the support of the material by the producer**
  - Legal Notices
  - Maintenance Statements

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of the value delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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Provenance

- **Information related to revision history of the material**
  - Dates of updates
  - Type of change
  - Description of updates
  - Specific changes

Certificate Revision History: 14 September 2022 (Change of expiration date; editorial changes); 16 December 2015 (Addition of parenthesis to equation four, alternate wavelength models; editorial changes); 31 August 2012 (Original certificate date).

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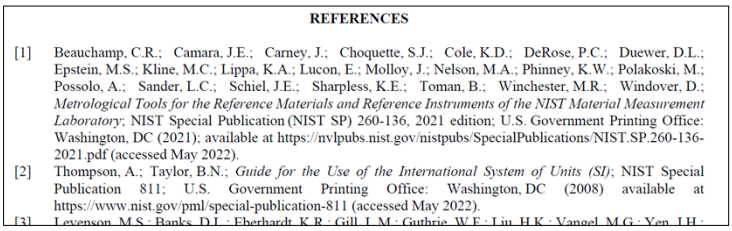
References

- **Information related to citations for the Reference Material**
  - Author(s)
  - Title
  - Place of Publication
  - Date of Publication



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.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; 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 (NIST SP) 260-136, 2021 edition; U.S. Government Printing Office: Washington, DC (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed May 2022).

[2] Thompson, A.; 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 (2008) available at https://www.nist.gov/pml/special-publication-811 (accessed May 2022).

[3] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.;

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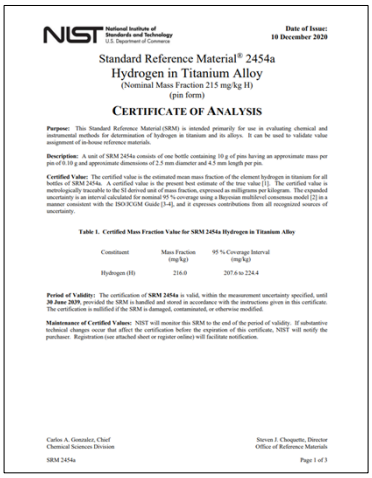
Additional Considerations

- **Greek Letters and Symbols**
- **Superscripts and Subscripts**
- **Creation of Human-Readable Certificates**
- **Separating data from text**



NIST 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

SRM 2454a

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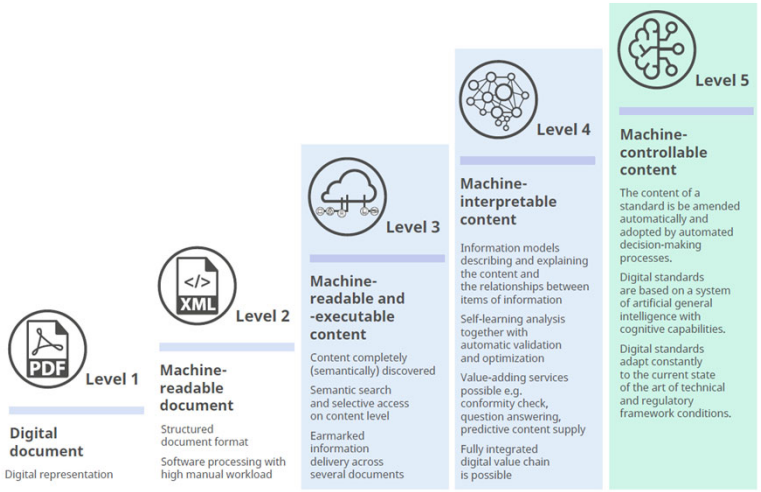
Where are we going?



IDIS Whitepaper, "SCENARIOS FOR DIGITIZING STANDARDIZATION AND STANDARDS", 2021

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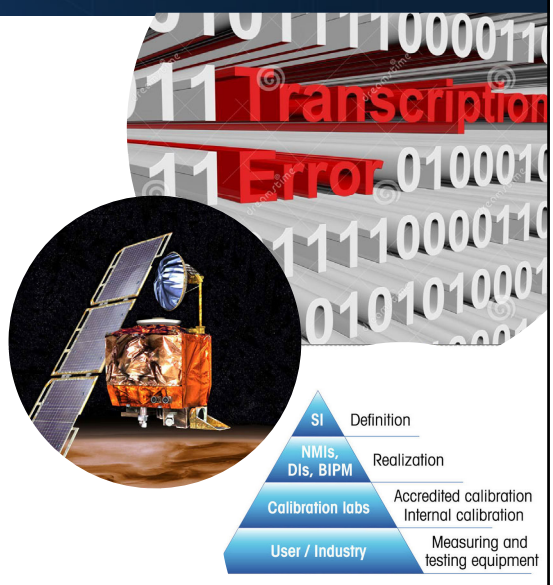
Advantages of Digital Products

- Eliminates transcription errors
- Interoperable data and unit formats
- Secure: use of digital signatures, persistent identifiers
- Enables a secure digital traceability chain
- Long-term preservation of information
  - Searchable database
  - Analyze data history using NIST web-based or other tools
- Provides a foundation for innovation in instruments
  - What if an instrument could tell you that your sample size is too small?
- Enables shared web-based measurement services
  - Customer data on NIST calibrated artifact can be directly compared to NIST data
- Customizable human readable reports and certificates
  - Formats, units, data presentation
- Allows access to larger, more complete data sets



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Next Steps

- **Continue to work on model for NIST**
- **Convert existing "paper based" data to be digital**
- **Work with SIM members**

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SIM MWG14 Sub-Working Group on DRMCs

- Initial Goal – Share information regarding DRMC development
- Work on DRMC schema
  - Model and rules for schema usage
  - Protocol for support and distribution
- Present recommendations to MWG14 after 1 year
- Informal format
- NIST led

Attendance to the meetings is open to all SIM members. It is asked that a representative from each participating SIM country be identified so that information from the group can be communicated to that individual and vice versa.

If you are interested in joining, contact me or your NMI's representative on MWG14
dinis.camara@nist.gov
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Thank You!
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digitalNIST@nist.gov
NIST
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