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CERTIFICATE OF ANALYSIS

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1.0 ACCREDITATION / REGISTRATION

INORGANIC VENTURES is accredited to ISO Guide 34, "General Requirements for the Competence of Reference Material Producers" and ISO/IEC 17025, "General Requirements for the Competence of Testing and Calibration Laboratories". Inorganic Ventures is also an ISO 9001 registered manufacturer (SAI Global File Number 010105).



2.0 PRODUCT DESCRIPTION

Product Code: Single Analyte Custom Grade Solution

Catalog Number: CGNB2O510

Lot Number: J2-NB01070R

Matrix: 15% (v/v) HNO3

1.8% (v/v) HF

Value / Analyte(s): 10 000 μg/mL ea:

Nb

Starting Material: Nb2O5

Starting Material Lot#: 1845

Starting Material Purity: 99.9984%

3.0 CERTIFIED VALUES AND UNCERTAINTIES

Certified Value: $9954 \pm 54 \mu g/mL$

Certified Density: 1.093 g/mL (measured at 20 \pm 1 °C)

Assay Information:

Assay Method #1 9954 ± 53 μg/mL

ICP Assay NIST SRM 3137 Lot Number: 120629Nb

- The Calculated Value is a value calculated from the weight of a starting material that has been certified directly vs. a National Institute of Standards and Technology (NIST) SRM/RM. See Sec 4.2 for balance traceability.

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

Characterization of CRM/RM by Two Methods

Certified Value, $X_{CRM/RM}$, where two methods of characterization are used is the weighted mean of the two results:

 $\mathsf{X}_{\mathsf{CRM/RM}} = [(\mathsf{w}_{\mathsf{a}})\;(\mathsf{X}_{\mathsf{a}}) + (\mathsf{w}_{\mathsf{b}})\;(\mathsf{X}_{\mathsf{b}})]$

 X_a = mean of Assay Method A with standard uncertainty u_{char} a

X_b = mean of Assay Method B with standard uncertainty u_{char b}

 $\mathbf{w_a}$ and $\mathbf{w_b}$ = the weighting factors for each method calculated using the inverse square of the variance:

 $\mathbf{w_a} = (1/u_{\text{char a}})^2 / ((1/u_{\text{char a}})^2 + (1/u_{\text{char b}})^2))$ $\mathbf{w_b} = (1/u_{\text{char b}})^2 / ((1/u_{\text{char a}})^2 + (1/u_{\text{char b}})^2))$

CRM/RM Expanded Uncertainty (±) = $U_{CRM/RM} = k (u^2_{char} a \& b + u^2_{bb} + u^2_{lts} + u^2_{sts})^{1/2}$

k = coverage factor = 2 in all cases at Inorganic Ventures

 $\begin{array}{l} \textbf{u}_{\textbf{char a\&b}} = [(w_a)^2 \ (u_{\textbf{char a}})^2 + (w_b)^2 \ (u_{\textbf{char b}})^2]^{1/2} \ \ \text{where } u_{\textbf{char a}} \ \ \text{and} \ \ u_{\textbf{char b}} \ \ \text{are the square} \\ \text{root of the sum of the squares of errors from characterization which include instrument} \\ \text{measurement, density, NIST SRM uncertainty, weighing, and volume} \end{array}$

 $\mathbf{u_{bb}}$ = bottle to bottle homogeneity standard uncertainty

u_{lts} = long term stability standard uncertainty (storage)

u_{sts} = short term stability standard uncertainty (transportation)

Characterization of CRM/RM by One Method

Certified Value, X_{CRM/RM}, where one method of characterization is used is the mean of individual results:

X_{CRM/RM} = mean of Assay Method A with standard uncertainty u_{char a}

CRM/RM Expanded Uncertainty (±) = $U_{CRM/RM} = k (u^2_{char} a + u^2_{bb} + u^2_{lts} + u^2_{sts})^{1/2}$

k = coverage factor = 2 in all cases at Inorganic Ventures

u_{char a} = square root of the sum of the squares of the errors from characterization which include instrumental measurement, density, NIST SRM uncertainty, weighing, and volume

u_{bb} = bottle to bottle homogeneity standard uncertainty

u_{lts} = long term stability standard uncertainty (storage)

u_{sts} = short term stability standard uncertainty (transportation)

4.0 TRACEABILITY TO NIST

- This product is traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM/RM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRM/RM are available, the term 'in-house std.' is specified.

4.1 Thermometer Calibration

- All thermometers are NIST traceable through thermometers that are calibrated by an accredited calibration laboratory.

4.2 Balance Calibration

- All analytical balances are calibrated by an accredited calibration laboratory and procedure. The weights used for testing are annually compared to master weights and are traceable to NIST.

4.3 Glassware Calibration

- An in-house procedure is used to calibrate all Class A glassware used in the manufacturing and quality control of CRM/RMs.

5.0 TRACE METALLIC IMPURITIES (TMI) DETERMINED BY ICP-MS AND ICP-OES (µg/mL)

CRM/RMs are tested for trace metallic impurities by Axial ICP-OES and ICP-MS. The result from the most sensitive method for each element, is reported below. Solutions tested by ICP-MS were analyzed in an ULPA-Filtered Clean Room. An ULPA-Filter is 99.9985% efficient for the removal of particles down to $0.3 \, \mu m$.

M	Ag	<	0.028939	M	Eu	<	0.000193	0	Na	<	0.100000	M	Se	<	0.038585	0	Zn	<	0.050000
0	Αl	<	0.050000	0	Fe	<	0.050000	s	Nb	<		0	Si	<	0.200000	М	Zr		0.009108
M	As	<	0.005788	M	Ga	<	0.000193	M	Nd	<	0.000193	M	Sm	<	0.001929				
М	Au	<	0.009646	М	Gd	<	0.000193	0	Ni	<	0.030000	M	Sn	<	0.005788				
М	В	<	0.003858	М	Ge	<	0.000965	n	Os	<		0	Sr	<	0.001000				
М	Ba	<	0.003858	М	Hf	<	0.003858	0	Р	<	0.100000	M	Ta		0.135705				
М	Be	<	0.000965	i	Hg	<		M	Pb	<	0.003858	M	Tb	<	0.000193				
М	Bi	<	0.007717	М	Но	<	0.000193	M	Pd	<	0.000193	M	Te	<	0.115755				
0	Ca		0.023604	М	In	<	0.000193	M	Pr	<	0.000193	M	Th	<	0.003858				
М	Cd	<	0.000193	М	lr	<	0.000193	M	Pt	<	0.000193	M	Ti	<	0.009646				
М	Ce	<	0.000193	0	K	<	0.100000	M	Rb	<	0.000193	M	TI	<	0.000193				
0	Co	<	0.020000	М	La	<	0.000193	M	Re	<	0.000193	M	Tm	<	0.000579				
М	Cr	<	0.003858	М	Li	<	0.005788	M	Rh	<	0.000193	M	U	<	0.000193				
М	Cs	<	0.005788	М	Lu	<	0.000193	M	Ru	<	0.000193	M	V	<	0.001929				
М	Cu	<	0.038585	0	Mg		0.007081	i	S	<		M	W		0.002550				
М	Dy	<	0.000193	M	Mn	<	0.003858	M	Sb		0.035520	M	Υ	<	0.000193				
М	Er	<	0.000193	M	Мо	<	0.001929	0	Sc	<	0.002000	M	Yb	<	0.000193				

M - Checked by ICP-MS O - Checked by ICP-OES i - Spectral Interference

6.0 INTENDED USE

- For the calibration of analytical instruments and validation of analytical methods as appropriate.

7.0 INSTRUCTIONS FOR THE CORRECT USE OF THIS REFERENCE MATERIAL

7.1 Storage and Handling Recommendations

- Keep cap tightly sealed when not in use. Store and use at $20 \pm 4^{\circ}$ C. Do not pipette from the container. Do not return removed aliquots to container.

Atomic Weight; Valence; Coordination Number; Chemical Form in Solution - 92.91 +5 6,7,8 NbOF5-2 Chemical Compatibility -Soluble in concentrated HCl and dilute HF / HNO3. Avoid neutral to basic media. Unstable at ppm levels with metals that would pull F- away (i.e. Do not mix with Alkaline or Rare Earths or high levels of transition elements unless they are fluorinated). Stable with most inorganic anions provided it is in the chemical form shown above.

Stability - 2-100 ppb levels stable (alone or mixed with all other metals that are at comparable levels) as the NbOF5-2 for 5 months in 1% HNO3 / LDPE container. 1-10,000 ppm single element solutions as the NbOF5-2 chemically stable for years in 2-5% HNO3 / trace HF in a LDPE container.

Nb Containing Samples (Preparation and Solution) - Metal (Soluble in HF / HNO3); Oxide - very resistant to all acids including HF (Fusion with K2S2O7,, KOH, or Na2CO3); Organic Matrices (Dry ash at 450EC in Pt0 and dissolve by fusing with Na2CO3 or K2S2O7).

Atomic Spectroscopic Information (ICP-OES D.L.s are given as radial/axial view):

Technique/Line	Estimated D.L.	Order	Interferences (underlined indicates severe)					
ICP-MS 93 amu	1 ppt	n/a	77Se16O,					
			74Se16O, [186X+2					
			(where X = W,Os)					
ICP-OES 269.706 nm	0.07 / 0.002 μg/mL	1	Th, Co					
ICP-OES 295.088 nm	0.08 / 0.001 μg/mL	1	Hf, U					
ICP-OES 309.418 nm	0.04 / 0.002 μg/mL	1						

HF Note: This standard should not be prepared or stored in glass.

8.0 HAZARDOUS INFORMATION

- Please refer to the Safety Data Sheet for information regarding this CRM/RM.

9.0 HOMOGENEITY

- This solution was mixed according to an in-house procedure and is guaranteed to be homogeneous. Homogeneity data indicate that the end user should take a minimum sample size of 0.2 mL to assure homogeneity.

10.0 QUALITY STANDARD DOCUMENTATION

10.1 10CFR50 Appendix B - Nuclear Regulatory Commission

- Domestic Licensing of Production and Utilization Facilities

10.2 10CFR21 - Nuclear Regulatory Commission

- Reporting defects and Non-Compliance

10.3 ISO 9001 Quality Management System Registration

- SAI Global File Number 010105

10.4 ISO/IEC Guide 17025 "General Requirements for the Competence of Testing and Calibration Laboratories"

- Chemical Testing - Accredited / A2LA Certificate Number 883.01

10.5 ISO/IEC Guide 34 "General Requirements for the Competence of Reference Material Producers"

- Reference Material Producer - Accredited / A2LA Certificate Number 883.02

11.0 CERTIFICATION, EXPIRATION AND PERIOD OF VALIDITY

11.1 Certification Issue Date

June 22, 2015

11.2 Expiration Date

11.3 Period of Validity

- The certification is valid within the measurement uncertainty specified provided the CRM/RM is handled and stored in accordance with instructions given in Sec 7.0 and used prior to the date given in Sec 11.2. This certification is nullified if the CRM/RM is damaged, contaminated, or otherwise modified.

Book & From Bleglists
Parl R Laine

12.0 NAMES AND SIGNATURES OF CERTIFYING OFFICERS

Certificate Prepared By:

Brenda Francis

Product Documentation Technician

Certificate Approved By:

Brian Alexander

PhD., Technical Process Director

Certifying Officer:

Paul Gaines

PhD., Senior Technical Director