

3.0

# 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 Mass Spec Solution											
Catalog Number:	MSHF-10PPM											
Lot Number:	J2-HF01055R											
Matrix:	tr. HNO3 tr. HF											
Value / Analyte(s):	10 μg/mL ea: Hf											
Starting Material:	HfO2											
Starting Material Lot#:	RD-24633											
Starting Material Purity:	99.9992%											
CERTIFIED VALUES AND UNCERTAINTIES												
Certified Value:	10.000 ± 0.052 μg/mL											
Certified Density:	0.998 g/mL (measured at 20 ± 1 °C)											
Assay Information:												
ANALYTE Hf	<b>METHOD</b> ICP Assay	NIST SRM# 3122	SRM LOT# 000406									

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:

#### $\boldsymbol{X}_{\text{CRM/RM}} = [(\boldsymbol{w}_a) \; (\boldsymbol{X}_a) + (\boldsymbol{w}_b) \; (\boldsymbol{X}_b)]$

- $X_a$  = mean of Assay Method A with standard uncertainty  $u_{char a}$
- $x_b^a$  = mean of Assay Method B with standard uncertainty  $u_{char b}^a$
- $\vec{w_a}$  and  $\vec{w_b}$  = the weighting factors for each method calculated using the inverse square of the variance:
  - $\mathbf{w_a} = (1/u_{char a})^2 / ((1/u_{char a})^2 + (1/u_{char b})^2))$
  - $\mathbf{w_b} = (1/u_{char b})^2 / ((1/u_{char a})^2 + (1/u_{char b})^2))$

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

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

- uchar a&b = [(w<sub>a</sub>)<sup>2</sup> (u<sub>char a</sub>)<sup>2</sup> + (w<sub>b</sub>)<sup>2</sup> (u<sub>char b</sub>)<sup>2</sup>]<sup>1/2</sup> where u<sub>char a</sub> and u<sub>char b</sub> are the square root of the squares of errors from characterization which include instrument measurement, density, NIST SRM uncertainty, weighing, and volume
- ubb = bottle to bottle homogeneity standard uncertainty
- ults = long term stability standard uncertainty (storage)

usts = short term stability standard uncertainty (transportation)

#### 4.0 TRACEABILITY TO NIST

CRM/RM Expanded Uncertainty (±) =  $U_{CRM/RM} = k (u^2_{char a} + u^2_{bb} + u^2_{lts} + u^2_{sts})^{\frac{1}{2}}$ k = coverage factor = 2 in all cases at Inorganic Ventures

- uchar 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
- ubb = bottle to bottle homogeneity standard uncertainty
- ults = long term stability standard uncertainty (storage)
- usts = short term stability standard uncertainty (transportation)

- 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 µm.

М	Ag	<	0.007436	Μ	Eu	<	0.011155	0	Na	<	0.000724	Μ	Se	<	0.029747	Μ	Zn	0.000020
0	Al	<	0.000724	0	Fe	<	0.362335	0	Nb	<	0.144934	n	Si	<		0	Zr	0.000064
0	As	<	0.072467	М	Ga	<	0.003718	М	Nd	<	0.007436	Μ	Sm	<	0.003718			
0	Au	<	0.362335	М	Gd	<	0.003718	М	Ni	<	0.297479	Μ	Sn	<	0.018592			
Μ	В	<	0.260294	М	Ge	<	0.022310	n	Os	<		0	Sr	<	0.024638			
Μ	Ва	<	0.037184	s	Hf	<		i	Р	<		i	Та	<	0.026029			
Μ	Be	<	0.001859	0	Hg	<	0.108700	М	Pb	<	0.011155	Μ	Tb	<	0.001115			
Μ	Bi	<	0.001487	М	Ho	<	0.001859	М	Pd	<	0.018592	Μ	Те	<	0.111554			
0	Са		0.000029	М	In	<	0.037184	М	Pr	<	0.001115	Μ	Th	<	0.003718			
Μ	Cd	<	0.011155	М	lr	<	0.018592	0	Pt	<	0.362335	0	Ti	<	0.434802			
Μ	Ce	<	0.018592	0	К	<	0.362335	М	Rb	<	0.003718	Μ	ΤI	<	0.003718			
Μ	Co	<	0.011155	М	La	<	0.001859	М	Re	<	0.003718	Μ	Tm	<	0.001487			
0	Cr	<	0.014493	0	Li		0.000001	М	Rh	<	0.003718	Μ	U	<	0.007436			
Μ	Cs	<	0.001115	М	Lu	<	0.001487	М	Ru	<	0.007436	Μ	V	<	0.007436			
Μ	Cu	<	0.022310	0	Mg		0.000005	i	S	<		Μ	W	<	0.037184			
Μ	Dy	<	0.022310	М	Mn	<	0.014873	0	Sb	<	0.144934	0	Υ	<	0.007246			
М	Er	<	0.018592	Μ	Мо	<	0.007436	М	Sc	<	0.037184	0	Yb	<	0.072467			

M - Checked by ICP-MS O - Checked by ICP-OES i - Spectral Interference n - Not Checked For s - Solution Standard Element

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#### Characterization of CRM/RM by One Method Certified Value, X<sub>CRM/RM</sub>, where one method of characterization is used is the mean of individual results:

XCRM/RM = mean of Assay Method A with standard uncertainty uchar a

#### 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 - 178.49 +4 6,7,8 Hf(F)6-2 Chemical Compatibility - Soluble in concentrated HCl, HF, H2SO4 (very hot) and HNO3. Avoid H3PO4 and 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 but precipitation with phosphate, oxalate, and tartrate with a tendency to hydrolyze forming the hydrated oxide in all dilute acids except HF.

**Stability** - 2-100 ppb levels stable alone or mixed with all other metals that are at comparable levels as the Hf(F)6-2+Hf(OH)4F2-2 for months in 1% HNO3 / LDPE container. 1-10,000 ppm single element solutions as the Hf(F)6-2 chemically stable for years in 2-5% HNO3 / trace HF in a LDPE container.

Hf Containing Samples (Preparation and Solution) - Metal (Soluble in H2O / HF / HNO3); Oxide unlike TiO2 the HfO2 is best fused in one of the following ways (Na2O2 in Ni0, Na2CO3 in Pt0 or Borax in Pt0); Organic Matrices (dry ash at 450 EC in Pt0 and dissolve by fusing with Na2CO3 and dissolving in HF / HNO3 / H2O). Atomic Spectroscopic Information (ICP-OES D.L.s are given as <u>radial/axial</u> view):

Technique/Line	Estimated D.L.	Order	Interferences (underlined indicates severe)
ICP-MS 177amu	4 ppt	n/a	161Dy16O (fewer
			potential
			interferences on the
			177 vs 180 line)
ICP-OES 264.141 nm	0.02 / 0.002 µg/mL	1	Ba, Th, U
ICP-OES 273.876 nm	0.02 / 0.002 µg/mL	1	U, Mo
ICP-OES 277.336 nm	0.02 / 0.002 µg/mL	1	Nb, Cr, U

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

February 24, 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.

#### 12.0 NAMES AND SIGNATURES OF CERTIFYING OFFICERS

## **Certificate Prepared By:**

Zach Saunders Product Documentation Technician

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**Certificate Approved By:** 

Brian Alexander PhD., Technical Process Director

**Certifying Officer:** 

Paul Gaines PhD., Senior Technical Director