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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 Mass Spec Solution

Catalog Number: MSW-10PPM
Lot Number: J2-W02030

Matrix: 0.14% (v/v) HNO3

0.2% (v/v) HF

Value / Analyte(s): 10 μg/mL ea:

W

Starting Material: W Powder

Starting Material Lot#: 1898

Starting Material Purity: 99.9953%

# 3.0 CERTIFIED VALUES AND UNCERTAINTIES

Certified Value:  $9.997 \pm 0.076 \mu g/mL$ 

Certified Density: 0.999 g/mL (measured at 20 ± 1 °C)

**Assay Information:** 

 ANALYTE
 METHOD
 NIST SRM#
 SRM LOT#

 W
 ICP Assay
 3163
 080331

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:

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

X<sub>a</sub> = mean of Assay Method A with standard uncertainty u<sub>char a</sub>

X<sub>b</sub> = mean of Assay Method B with standard uncertainty u<sub>char b</sub>

w<sub>a</sub> and w<sub>b</sub> = 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

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

u<sub>bb</sub> = bottle to bottle homogeneity standard uncertainty

ults = long term stability standard uncertainty (storage)

u<sub>sts</sub> = short term stability standard uncertainty (transportation)

#### 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:

X<sub>CRM/RM</sub> = mean of Assay Method A with standard uncertainty u<sub>char a</sub>

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<sub>char a</sub> = 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<sub>bb</sub> = bottle to bottle homogeneity standard uncertainty

u<sub>lts</sub> = long term stability standard uncertainty (storage)

u<sub>sts</sub> = 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	<	0.000742	M	Eu	<	0.000185	0	Na		0.000019	M	Se	<	0.022259	M	Zn	<	0.027823
М	Αl	<	0.037098	Ο	Fe	<	0.021300	M	Nb	<	0.020404	i	Si	<		Ο	Zr	<	0.006846
М	As	<	0.018549	М	Ga	<	0.000927	M	Nd	<	0.005565	M	Sm	<	0.000185				
0	Au	<	0.053249	М	Gd	<	0.000185	M	Ni	<	0.009274	M	Sn	<	0.004637				
М	В	<	0.007420	М	Ge	<	0.000927	n	Os	<		M	Sr	<	0.000371				
М	Ва	<	0.003710	M	Hf	<	0.003710	i	Р	<		M	Ta		0.000016				
М	Be	<	0.002782	i	Hg	<		M	Pb	<	0.000927	M	Tb	<	0.000185				
М	Bi	<	0.003710	М	Но	<	0.000185	M	Pd	<	0.000742	M	Te	<	0.004637				
0	Ca		0.000008	М	In	<	0.000927	M	Pr	<	0.000927	M	Th	<	0.032460				
М	Cd	<	0.000927	М	lr	<	0.000185	M	Pt	<	0.000927	M	Ti	<	0.002782				
М	Ce	<	0.000742	0	K		0.000475	M	Rb	<	0.013912	M	TI	<	0.001855				
М	Co	<	0.001855	М	La	<	0.000185	M	Re	<	0.126132	M	Tm	<	0.000185				
М	Cr	<	0.009274	0	Li	<	0.000400	M	Rh	<	0.000185	M	U	<	0.000927				
М	Cs		0.000005	М	Lu	<	0.000185	M	Ru	<	0.001855	M	V	<	0.009274				
М	Cu	<	0.003710	0	Mg	<	0.004004	i	S	<		s	W	<					
M	Dy	<	0.000185	M	Mn	<	0.004637	M	Sb	<	0.001855	M	Υ	<	0.000185				
M	Er	<	0.000185	M	Мо	<	0.004637	Ο	Sc	<	0.004004	M	Yb	<	0.000185				

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

n - Not Checked For s - Solution Standard Element

#### 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 - 183.85 +6 6,7,8,9 WOF5-2(chemical form as received)

Chemical Compatibility -W is very readily hydrolyzed requiring 0.1 to 1% HF solutions for stabile acidic solutions. The [WOF5]-2 is soluble in % levels of HCl and HNO3 provided it is in the [WOF5]-2 form. Stable at ppm levels with some metals provided it is fluorinated. <U>Do not mix with Alkaline or Rare Earths</U>. W is best to be mixed only with other fluorinated metals (Ti, Zr, Hf, Nb, Ta, Mo, Si, Sn, Ge). Look for yellow WO3 precipitate if mixed with other transitions at higher levels indicating instability. The yellow WO3 will form over a period of weeks even in trace HF, therefore, <U>HF levels of W multi-element blends should be &sim; 1%</U>.

**Stability** - 2-100 ppb levels stable (alone or mixed with all other metals that are at comparable levels) as the [WOF5]-2 for months in 1% HNO3 / LDPE container. 1-10,000 ppm single element solutions as the [WOF5]-2 chemically stable for years in 1% HF in a LDPE container.

**W** Containing Samples (Preparation and Solution) -Metal (Soluble in HF / HNO3); Oxide (soluble in HF or NH4OH); Organic Matrices (dry ash at 450 0C in Pt0 and dissolve oxide with HF).

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 182 amu	5 ppt	N/A	166Er16O						
ICP-OES 207.911 nm	0.03/0.001 µg/mL	1	Ru, In						
ICP-OES 209.475 nm	0.05/0.008 μg/mL	1	Мо						
ICP-OES 224.875 nm	0.05/0.005 μg/mL	1	Co, Rh, Ag						

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

September 18, 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:

James King Jr Product Documentation Supervisor

# **Certificate Approved By:**

Michael Booth QC Supervisor

Michael 2 Booth

### **Certifying Officer:**

Paul Gaines PhD., Senior Technical Director Paul R Laines