

Certificate of Analysis

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1.0 ACCREDITATION / REGISTRATION

INORGANIC VENTURES is accredited to ISO 17034, "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 (QSR Certificate Number QSR-1034).



2.0 PRODUCT DESCRIPTION

Product Code: Single Analyte Mass Spec Solution

 Catalog Number:
 MSOS-100PPM

 Lot Number:
 U2-OS732104

 Matrix:
 10% (v/v) HCI

Value / Analyte(s): 100 μg/mL ea:

Osmium

Starting Material: Ammonium Hexachloroosmate

Starting Material Lot#: 2203 and 2333

Starting Material Purity: 99.9955%

3.0 CERTIFIED VALUES AND UNCERTAINTIES

Certified Value: $99.93 \pm 0.75 \,\mu\text{g/mL}$

Density: 1.019 g/mL (measured at 20 ± 4 °C)

Assay Information:

ANALYTE METHOD Primary Certified Reference Material (PCRM''') PCRM™ LOT#
Os ICP Assay PCRM-OS-1000 T2-PCRMOS717957

-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 or More Methods

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

 $X_{CRM/RM} = \Sigma(w_i) (X_i)$

X_i = mean of Assay Method i with standard uncertainty u_{char i}

w_i = the weighting factors for each method calculated using the inverse square of

the variance:

 $\mathbf{w_i} = (1/\mathsf{u_{char\ i}})^2 \, / \, (\Sigma (1/(\mathsf{u_{char\ i}})^2)$

CRM/RM Expanded Uncertainty (±) = U_{CRM/RM} = k (u²_{char} + u²_{bb} + u²_{lts} + u²_{ts})^{1/2}

k = coverage factor = 2

 $\mathbf{u_{char}} = [\Sigma((\mathbf{w_i})^2 (\mathbf{u_{char}}_i)^2)]^{\frac{1}{2}}$ where $\mathbf{u_{char}}_i$ are the errors from each characterization method

u_{bb} = bottle to bottle homogeneity standard uncertainty

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

uts = transport stability standard uncertainty

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} = (X_a) (u_{char a})

X_a = mean of Assay Method A with

u_{char a} = the standard uncertainty of characterization Method A

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

k = coverage factor = 2

u_{char a} = the errors from characterization

 $\mathbf{u}_{\mathbf{b}\mathbf{b}}$ = bottle to bottle homogeneity standard uncertainty $\mathbf{u}_{\mathbf{l}\mathbf{t}\mathbf{s}}$ = long term stability standard uncertainty (storage)

u_{ts} = transport stability standard uncertainty

4.0 TRACEABILITY TO NIST

For this CRM, the accurate mass determinations and purity assessments realized traceability to the kilogram, a base unit of the SI.

The traceability was established through an unbroken chain of calibrations / comparisons, with their associated uncertainties, using accurate mass determinations by gravimetric reduction. Quantified uncertainties were determined by error budget analysis. Quantitative purity analyses were performed using ICP-OES and ICP-MS for Trace Metallic Impurities analysis (TMI) and inert gas fusion analysis for oxygen, nitrogen, and hydrogen.

The United State National Metrology Institute (NMI) is the National Institute of Standards and Technology (NIST). The NIST Policy on Metrological Traceability recommends adopting the definition of metrological traceability as stated in the most recent version of the International Vocabulary of Metrology (VIM)ⁱⁱ. The metrological traceability of this CRM was established by implementing the VIM definition. The VIM defines Metrological traceability as a "property of a measurement result whereby the result can be related to a reference (for this CRM the SI reference is the kilogram) through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty." In VIMⁱⁱⁱ, traceability to the SI is defined as "metrological traceability to a measurement unit of the International System of Units". Purity analysis and use of primary reference measurement procedures^{iv}, e.g., amount of substance, are at the apex of the hierarchy in establishing SI units.^V

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 $0.3 \, \mu m$.

```
Μ
   Aq
            0.000035 M
                       Eu <
                                 0.000011 M
                                            Na
                                                     0.001421 M Se <
                                                                          0.000500 M
                                                                                     7n <
                                                                                              0.001300
Μ
   ΑI
            0.000152 M
                       Fe <
                                0.003500 M
                                            Nb <
                                                     0.000011 O
                                                                Si
                                                                          0.003045 M
                                                                                     Zr
                                                                                              0.000470
Μ
   As
            0.002335 M
                       Ga <
                                0.000041 M
                                            Nd <
                                                     0.000011 M
                                                                Sm <
                                                                          0.000011
Μ
   Au
            0.000083 M
                       Gd <
                                 0.000011 M
                                            Ni <
                                                     0.000082 M
                                                                Sn
                                                                          0.000074
М
   В
            0.001300 M
                       Ge
                                0.000056 s
                                            Os <
                                                             0
                                                                Sr <
                                                                          0.000110
М
   Ba
            0.000210 M
                       Hf <
                                 0.000011 O
                                            Р
                                                     0.021000 M
                                                                Ta <
                                                                          0.000011
                                0.000450 M Pb
М
   Be
            0.000061 M
                       Hg <
                                                     0.005100 M
                                                                Tb <
                                                                          0.000011
   Bi
М
            0.000131 M
                       Ho <
                                 0.000011 M
                                            Pd
                                                     0.000052 M
                                                                Te <
                                                                          0.000130
0
   Ca
            0.001624 M In <
                                            Pr
                                 0.000011 M
                                                     0.000011 M
                                                                Th <
                                                                          0.000270
М
   Cd <
            0.000011 M Ir
                                0.000802 M
                                            Pt
                                                     0.000385 M Ti <
                                                                          0.000230
М
   Ce <
            0.000041 O K <
                                            Rb
                                                     0.000041 M TI <
                                                                          0.082000
                                0.020000 M
Μ
   Co <
            0.000041 M La <
                                 0.000011 M
                                            Re <
                                                     0.000170 M Tm <
                                                                          0.000011
Μ
   Cr <
            0.002400 M Li <
                                0.000170 M Rh
                                                     0.000011 M U
                                                                          0.003100
Μ
   Cs
            0.000037 M Lu <
                                 0.000011 M Ru
                                                     0.000375 O
                                                                V
                                                                          0.002400
Μ
   Cu
            0.000012 O
                       Mg
                                0.000074 O
                                            S
                                                     0.110000 M
                                                                W
                                                                          0.000011
М
   Dy <
            0.000011 M
                       Mn <
                                 0.000110 M
                                            Sb
                                                     0.000111 M
                                                                Υ
                                                                          0.000011
Μ
   Er
            0.000011 M
                       Mo <
                                0.000041 M
                                            Sc
                                                     0.000051 M
                                                                Yb
                                                                          0.000011
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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

- **6.1** This standard is intended for the calibration of analytical instruments and validation of analytical methods as appropriate. This CRM may be used in connection with EPA Methods 6010, 6020 (all versions), Standard Methods 3120 B and USP <232> / ICH Q3D.
- **6.2** For products attaining traceability through Inorganic Ventures' Primary Certified Reference Materials (PCRM™) see the Limited License to Use PCRM™ in the Inorganic Ventures <u>Terms and Conditions of Sale</u>. https://www.inorganicventures.com/terms-and-conditions-sale. The Terms and Conditions contain information on the use of materials traceable to PCRM™ certified reference materials. This Limited License agreement is especially pertinent for laboratories accredited under ISO:17034.

7.0 INSTRUCTIONS FOR THE CORRECT USE OF THIS REFERENCE MATERIAL

7.1 Storage and Handling Recommendations

- Store between approximately 4° 30° C while in sealed TCT bag.
- While stored in the sealed TCT bag, transpiration of this CRM/RM is negligible. After opening the sealed TCT bag transpiration of the CRM/RM will occur, resulting in a gradual increase in the analyte concentration(s). It is the responsibility of the user to account for this effect. When the bottle is weighed both before and after being placed in storage, the mass difference observed will be a measure of transpiration mass loss.
- After opening the sealed TCT bag, keep cap tightly sealed when not in use and store between 4° 24° C to minimize the effects of transpiration. Use at $20^{\circ} \pm 4^{\circ}$ C to minimize volumetric dilution error when using the reported density. Do not pipette from the container. Do not return removed aliquots to container.
- For more information, visit www.inorganicventures.com/TCT

Atomic Weight; Valence; Coordination Number; Chemical Form in Solution - 190.20 +4 4,5,6,8 OsCl62-Chemical Compatibility - Stable in HCI. Stable with most metals and inorganic anions as the OsCl62- in dilute HCI media. DO NOT EXPOSE TO NITRIC ACID - FORMATION OF THE VERY VOLATILE AND TOXIC OsO4 WILL RESULT. Any oxidizing condition must be avoided.

Stability - 2-100 ppb levels are not stable in 1% HNO3 / LDPE container. The stability of HCl solutions at ppb levels has not been determined by our laboratory. 1-10,000 ppm solutions are presumed chemically stable for years in 10% HCl / LDPE container, stability studies have not been performed.

Os Containing Samples (Preparation and Solution) -Oxides (fuse with KOH / KNO3 in a Ag0 crucible and dissolve in water being sure to avoid addition of any acid); Ores (See Oxides); Organics (The OsO4 is volatile and acidic oxidizing preparations should be used with caution. The preferred approach is the KOH / KNO3 fusion and dissolution of the fuseate in water. Our laboratory has used APDC to help stabilize Os solutions but more work is required to validate effectiveness.)

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 192 amu	1 ppt	n/a	176Yb16O,
			176Lu16O,
			176Hf16O,
			192Pt.Please note -
			The presence of the
			OsO4 will give false
			high results due to
			its enhanced
			nebulization
			efficiency (volatility).
			Only dilutions in HCl
			should be made.
			The use of nitric
			acid should be
			strictly avoided.
			Preparations from
			caustic nitrate
			fusions should be
			diluted in water.
CP-OES 225.585 nm	0.03 / 0.001 μg/mL	1	Fe, Ta, Ge, Ir, Cr

8.0 HAZARDOUS INFORMATION

- Please refer to the Safety Data Sheet for information regarding this CRM/RM. Avoid dilutions with oxidizing acids such as concentrated HNO3, due to the formation of toxic osmium tetroxide.

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 ISO 9001 Quality Management System Registration

- QSR Certificate Number QSR-1034

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

- Chemical Testing - Accredited / A2LA Certificate Number 883.01

10.3 ISO 17034 "General Requirements for the Competence of Reference Material Producers"

- Reference Material Producer - Accredited / A2LA Certificate Number 883.02

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11.0 CERTIFICATION, LOT EXPIRATION AND PERIOD OF VALIDITY

11.1 Certification Issue Date

April 24, 2023

- The certification is valid within the measurement uncertainty specified provided the CRM/RM is stored and handled in accordance with instructions given in Sec 7.1. This certification is nullified if instructions in Sec 7.1 are not followed or if the CRM/RM is damaged, contaminated, or otherwise modified.

11.2 Lot Expiration Date

- April 24, 2028
- The date after which this CRM/RM should not be used.
- The lot expiration date reflects the period of time that the stability of a CRM/RM can be supported by long term stability studies conducted on properly stored and handled CRM/RMs. Lot expiration is limited primarily by transpiration (loss of water from the solution) and infrequently by chemical stability.

11.3 Period of Validity

Sealed TCT Bag Open Date:	

- This CRM/RM should not be used longer than one year (or six months in the case of a 30 mL bottle) from the date of opening the aluminized bag or after the date given in Sec. 11.2, whichever comes first. This is contingent upon the CRM/RM being stored and handled in accordance with the instructions given in Sec. 7.1.

12.0 NAMES AND SIGNATURES OF CERTIFYING OFFICERS Certificate Prepared By:

Uyen Truong
Supervisor, Product Documentation

Certificate Approved By:

Nicholas Plymale Lead Quality Control Technician

Certifying Officer:

Paul Gaines

Chairman / Senior Technical Director

Didl

https://www.nist.gov/calibrations/traceability, Created February 12, 2010, Updated April 20, 2021

ⁱⁱ Joint Committee for Guides in Metrology (2012, 3rd Edition, International Bureau of Weights and Measures (BIPM)). VIM, 2.41, p.29. https://www.bipm.org/en/committees/jc/jcgm/publications

iii Ibid. VIM, 2.43, p.30

iv Ibid. VIM, 5.4, p.47

v https://www.nist.gov/mml/csd/organic-chemical-metrology/primary-focus-areas/fundamental-chemical-metrology/si