

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 Custom Grade Solution

Catalog Number: CGZR10

Lot Number: U2-ZR729358

Matrix: 1.1% (v/v) HF

Value / Analyte(s): 10 000 µg/mL ea:

Zirconium

Starting Material: Zr Metal

Starting Material Lot#: 2512

Starting Material Purity: 99.9533%

3.0 CERTIFIED VALUES AND UNCERTAINTIES

Certified Value: 9994 \pm 66 μ g/mL

Density: 1.015 g/mL (measured at 20 ± 4 °C)

Assay Information:

Assay Method #1 9992 ± 63 μg/mL

ICP Assay NIST SRM traceable to SRM#3169 Lot Number: S2-ZR707913

Assay Method #2 9996 ± 61 μg/mL

Calculated NIST SRM Lot Number: See Sec. 4.2

- 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

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

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

k = coverage factor = 2

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

ubb = bottle to bottle homogeneity standard uncertainty ults = 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:

 $X_{CRM/RM} = (X_a) (u_{char} a)$

Xa = mean of Assay Method A with

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

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

k = coverage factor = 2

u_{char a} = the errors from characterization

 $\mathbf{u_{bb}}$ = bottle to bottle homogeneity standard uncertainty u_{lts} = long term stability standard uncertainty (storage)

u_{ts} = transport stability standard uncertainty

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.

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

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.

| i | Ag | < | | М | Eu | < | 0.000500 | М | Na | | 0.099552 | М | Se | < | 0.018000 | М | Zn | | 0.119463 |
|---|----|---|----------|---|----|---|----------|---|----|---|----------|---|----|---|----------|---|----|---|----------|
| M | Αl | | 0.398210 | М | Fe | | 2.654737 | М | Nb | < | 0.240000 | 0 | Si | | 0.309719 | s | Zr | < | |
| M | As | < | 0.006000 | М | Ga | < | 0.015000 | М | Nd | < | 0.000500 | М | Sm | < | 0.000500 | | | | |
| M | Au | < | 0.000910 | М | Gd | < | 0.000500 | М | Ni | | 0.243350 | М | Sn | | 0.002875 | | | | |
| M | В | | 0.035396 | М | Ge | < | 0.000500 | М | Os | < | 0.000460 | M | Sr | < | 0.007000 | | | | |
| M | Ва | | 0.039821 | М | Hf | | 0.165921 | 0 | Р | < | 0.220000 | M | Ta | | 0.009291 | | | | |
| M | Ве | < | 0.003000 | М | Hg | < | 0.000910 | М | Pb | < | 0.007000 | M | Tb | < | 0.000500 | | | | |
| M | Bi | | 0.001880 | М | Но | < | 0.000500 | М | Pd | < | 0.002800 | M | Те | < | 0.024000 | | | | |
| M | Ca | | 0.068580 | М | In | < | 0.000500 | М | Pr | < | 0.000500 | M | Th | < | 0.002000 | | | | |
| M | Cd | < | 0.065000 | М | lr | < | 0.000460 | М | Pt | < | 0.005000 | M | Ti | | 0.057519 | | | | |
| M | Ce | < | 0.000500 | 0 | K | | 0.042033 | М | Rb | < | 0.002500 | M | TI | < | 0.000500 | | | | |
| M | Co | | 0.006194 | М | La | < | 0.000500 | М | Re | < | 0.000500 | M | Tm | < | 0.000500 | | | | |
| M | Cr | | 0.619438 | М | Li | < | 0.006000 | М | Rh | < | 0.000500 | M | U | | 0.006858 | | | | |
| M | Cs | < | 0.002000 | М | Lu | < | 0.000500 | М | Ru | < | 0.002800 | M | V | | 0.018583 | | | | |
| M | Cu | | 0.044245 | М | Mg | | 0.009070 | 0 | S | < | 0.022000 | M | W | | 0.001548 | | | | |
| M | Dy | < | 0.000500 | М | Mn | | 0.073005 | М | Sb | < | 0.003000 | M | Υ | < | 0.000500 | | | | |
| M | Er | < | 0.000500 | М | Мо | | 0.014379 | 0 | Sc | < | 0.027000 | М | Yb | < | 0.000500 | | | | |
| | | | | | | | | | | | | | | | | | | | |

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 - 91.22 +4 6,7,8 Zr(F)6-2 Chemical Compatibility -Soluble in concentrated HCl, HF, H2SO4 (very hot) and HNO3. Avoid H3PO4and 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 Zr(F)6-2 + Zr(OH)4F2-2 for months in 1% HNO3 / LDPE container. 1-10,000 ppm single element solutions as the Zr(F)6-2 chemically stable for years in 2-5% HNO3 / trace HF in a LDPE container.

Zr Containing Samples (Preparation and Solution) -Metal (Soluble in H2O / HF / HNO3); Oxide unlike TiO2 the ZrO2 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 0C 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 radial/axial view):

| Technique/Line | Estimated D.L. | Order | Interferences (underlined indicates severe) | | | | | |
|--------------------|--------------------|-------|---------------------------------------------|--|--|--|--|--|
| ICP-MS 90 amu | 2 ppt | N/A | 74Ge16O, | | | | | |
| | | | 74Se16O, [180X+2 | | | | | |
| | | | (where X = Hf, Ta, | | | | | |
| | | | W)] | | | | | |
| ICP-OES 272.261 nm | 0.018/0.001 μg/mL | 1 | Cr, V, Th, W | | | | | |
| ICP-OES 339.198 nm | 0.008/0.0007 μg/mL | 1 | Th, Mo | | | | | |
| ICP-OES 343.823 nm | 0.007/0.0004 µg/mL | 1 | Hf, Nb | | | | | |

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 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

February 08, 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

- February 08, 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 Approved By:

Thomas Kozikowski Manager, Quality Control

Certifying Officer:

Paul Gaines Chairman / Senior Technical Director Paul R. Sains