

## 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: CGSIO1

Lot Number: H2-SI03034

Matrix: 1% (v/v) HNO<sub>3</sub>  
tr. HF

Value / Analyte(s): 1 000 µg/mL ea:  
Silica

Starting Material: SiO<sub>2</sub>

Starting Material Lot#: 1551

Starting Material Purity: 99.9993%

## 3.0 CERTIFIED VALUES AND UNCERTAINTIES

**Certified Value:** 1001 ± 5 µg/mL

**Certified Density:** 1.005 g/mL (measured at 20 ± 1 °C)

### Assay Information:

**Assay Method #1**                      **1001 ± 5 µg/mL**  
ICP Assay NIST SRM 3150 Lot Number: 071204

**Assay Method #2**                      **1000 ± 5 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 Methods**

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

$$X_{CRM/RM} = [(w_a)(X_a) + (w_b)(X_b)]$$

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

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

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

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

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

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

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

$u_{char a\&b} = [(w_a)^2 (u_{char a})^2 + (w_b)^2 (u_{char b})^2]^{1/2}$  where  $u_{char a}$  and  $u_{char b}$  are the square root of the sum of the squares of errors from characterization which include instrument 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)

**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:

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

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

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 µm.

|                 |                 |                 |                 |                 |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| M Ag < 0.004820 | M Eu < 0.007230 | n Na <          | M Se < 0.019280 | O Zn < 0.000343 |
| O Al < 0.005154 | O Fe < 0.000334 | M Nb < 0.001205 | s Si <          | M Zr < 0.012050 |
| M As < 0.024100 | M Ga < 0.002410 | M Nd < 0.004820 | M Sm < 0.002410 |                 |
| M Au < 0.007230 | M Gd < 0.002410 | O Ni < 0.003951 | M Sn < 0.000328 |                 |
| n B <           | M Ge < 0.014460 | n Os <          | O Sr < 0.000120 |                 |
| M Ba < 0.024100 | M Hf < 0.004820 | O P < 0.008590  | O Ta < 0.010308 |                 |
| O Be < 0.000343 | O Hg < 0.018898 | M Pb < 0.007230 | M Tb < 0.000723 |                 |
| M Bi < 0.000964 | M Ho < 0.001205 | M Pd < 0.012050 | M Te < 0.072300 |                 |
| O Ca < 0.001503 | M In < 0.024100 | M Pr < 0.000723 | M Th < 0.002410 |                 |
| M Cd < 0.007230 | M Ir < 0.012050 | M Pt < 0.004820 | O Ti < 0.000334 |                 |
| M Ce < 0.012050 | O K < 0.001336  | M Rb < 0.002410 | M Tl < 0.002410 |                 |
| M Co < 0.007230 | M La < 0.001205 | M Re < 0.002410 | M Tm < 0.000964 |                 |
| O Cr < 0.002577 | O Li < 0.000034 | M Rh < 0.002410 | M U < 0.004820  |                 |
| M Cs < 0.000723 | M Lu < 0.000964 | M Ru < 0.004820 | O V < 0.001546  |                 |
| O Cu < 0.001718 | O Mg < 0.000100 | O S < 0.042950  | O W < 0.006872  |                 |
| M Dy < 0.014460 | M Mn < 0.009640 | M Sb < 0.001205 | M Y < 0.096400  |                 |
| M Er < 0.012050 | M Mo < 0.004820 | O Sc < 0.000343 | M Yb < 0.002410 |                 |

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  $20 \pm 4^\circ \text{C}$ . Do not pipette from the container. Do not return removed aliquots to container.

**Atomic Weight; Valence; Coordination Number; Chemical Form in Solution** -  $28.09 + 4 \text{ Si(OH)}_x(\text{F})_y$ -  
**Chemical Compatibility** -Soluble in HCl, HF, H<sub>3</sub>PO<sub>4</sub> H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> as the  $\text{Si(OH)}_x(\text{F})_y$ -. 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 with a tendency to hydrolyze forming silicic acid (silicic acid is soluble up to  $\sim 100$  ppm in water) in all dilute acids except HF.

**Stability** - 2-100 ppb levels - stability unknown - (alone or mixed with all other metals) as the  $\text{Si(OH)}_x(\text{F})_y$ -.  
1-10,000 ppm single element solutions as the  $\text{Si(OH)}_x(\text{F})_y$ - chemically stable for years in 2-5 % HNO<sub>3</sub> / trace HF in a LDPE container.

**SiO<sub>2</sub> Containing Samples (Preparation and Solution)** -Metal (Soluble in 1:1:1 H<sub>2</sub>O / HF / HNO<sub>3</sub>); Oxide - SiO<sub>2</sub>, amorphous (dissolve by heating in 1:1:1 H<sub>2</sub>O / HF / HNO<sub>3</sub>); Oxide - quartz (fuse in Pt<sub>0</sub> with Na<sub>2</sub>CO<sub>3</sub>); Geological Samples(fuse in Pt<sub>0</sub>with Na<sub>2</sub>CO<sub>3</sub> followed by HCl solution of the fuseate); Organic Matrices containing silicates and non volatile silicon compounds (dry ash at 4500C in Pt<sub>0</sub> and dissolve by gently warming with 1:1:1 H<sub>2</sub>O / HF / H<sub>2</sub>SO<sub>4</sub> or fuse / ash with Na<sub>2</sub>CO<sub>3</sub> and dissolve fuseate with HCl / H<sub>2</sub>O ); Silicone Oils - dimethyl silicones depolymerize to form volatile monomer units when heated (Measure directly in alcoholic KOH / xylene mixture where sample is treated first with the KOH at 60-1000C to "unzip" the Si-O-Si polymeric structure or digest with conc. H<sub>2</sub>SO<sub>4</sub> / H<sub>2</sub>O<sub>2</sub> followed by cooling and dissolution of the dehydrated silica with HF.) Note that the direct analysis of silicone oils in an organic solvent will result in false high results due to high vapor pressure of volatile monomer units like hexamethylcyclotrisiloxane. The KOH forms the  $\text{K}_2\text{Si}(\text{CH}_3)_2\text{O}$ = salt which is not volatile at room temperature.

**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 28 amu      | 4000 - 8000 ppt   | N/A   | N <sub>2</sub> , <u>12C16O</u>              |
| ICP-OES 212.412 nm | 0.02/0.01 µg/mL   | 1     | Hf, Os, Mo, Ta                              |
| ICP-OES 251.611 nm | 0.012/0.003 µg/mL | 1     | Ta, U, Zn, Th                               |
| ICP-OES 288.158 nm | 0.03/0.004 µg/mL  | 1     | Ta, Ce, Cr, Cd, Th                          |

**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

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**11.0 CERTIFICATION, EXPIRATION AND PERIOD OF VALIDITY**

**11.1 Certification Issue Date**

May 12, 2014

**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:**

Donna Senn  
Product Documentation Technician



**Certificate Approved By:**

Brian Alexander  
PhD., Technical Process Director



**Certifying Officer:**

Paul Gaines  
PhD., Senior Technical Director

