



Certificate of Analysis

Certified Reference Material

HICD-1

High Purity Cadmium Certified Reference Material for Cadmium Mass Fraction and Elemental Impurities

HICD-1 is a high purity cadmium Certified Reference Material (CRM). A unit of HICD-1 consists of approximately 1.0 g of high purity cadmium rod. This material is intended as a primary standard for the determination of mass fraction of cadmium.

Certified values for the mass fractions of cadmium and elemental impurities have been established for HICD-1 CRM. These values are listed in the Tables 1-2. Certified values are based on measurements carried out at the National Research Council Canada (NRC). The expanded uncertainty (U_{CRM}) in the certified values is equal to $U = k u_c$ where u_c is the combined standard uncertainty calculated according to the JCGM Guide [1] and k is the coverage factor. A coverage factor of two ($k = 2$) was applied which corresponds to approx. 95 % confidence. It is intended that U_{CRM} accounts for every aspect that reasonably contributes to the uncertainty of the measurement.

Table 1: Certified quantity value and expanded uncertainty ($k=2$) of mass fraction of cadmium in HICD-1

Quantity	Value	Expanded uncertainty
Mass fraction of cadmium, $w(\text{Cd})/(\text{kg}/\text{kg})$	0.999 984	0.000 062

Table 1 shows derived quantity that characterizes the property of the cadmium in HICD-1, and this quantity was calculated using the following mathematical expression:

$$w(\text{Cd}) = 1 - \sum_E w(\text{E})$$

Here E refers to the elements listed in Table 2. Other elements such as hydrogen, noble gases or any other elements not listed in Table 2 were not considered in the purity estimate.

Table 2 shows the measured quantities in HICD-1. Elemental impurities of cadmium were determined by glow discharge mass spectrometry (GDMS) using measurement models and methods with traceability to the International System of Units (SI) through a network of CRMs [2,3]. For the purposes of obtaining the mass fraction (purity) estimate of cadmium, all elemental impurities below detection limit were interpreted as half the detection limit (for example '<0.1 $\mu\text{g}/\text{kg}$ ' for lithium is interpreted as 0.05 $\mu\text{g}/\text{kg}$). A robust estimator (median) was used to summarize the observed values from 15 units reported in Table 2.

Table 2: Certified quantity values and expanded uncertainties ($k=2$) of mass fractions of elemental impurities in HICD-1 ($\mu\text{g}/\text{kg}$)

Impurity element, E	Mass fraction, w(E)	Expanded uncertainty	Impurity element, E	Mass fraction, w(E)	Expanded uncertainty
Li	<0.1	0.2	Pd	<10	20
Be	<0.1	0.1	Ag	<20	10
B	0.1	2	Cd	Matrix	Matrix
C	40	490	In	<210	810
N	10	70	Sn	<10	30
O	4000	40000	Sb	<1	1
F	<0.3	0.8	Te	<13	7
Na	10	50	I	<10	80
Mg	0.1	2	Cs	<4	5
Al	3	60	Ba	<40	50
Si	10	100	La	<0.7	0.7
P	0.1	2	Ce	<2	1
S	100	160	Pr	<2	6
Cl	6000	47000	Nd	<2	1
K	<30	30	Sm	<30	70
Ca	1	70	Eu	<0.1	0.3
Sc	<0.2	0.1	Gd	<0.4	1
Ti	0.5	3	Tb	<0.04	0.11
V	0.3	4	Dy	<0.2	0.4
Cr	50	500	Ho	<0.04	0.11
Mn	<30	60	Er	<0.1	0.3
Fe	200	2300	Tm	<0.04	0.11
Co	11	7	Yb	<0.1	0.4
Ni	700	280	Lu	<0.05	0.12
Cu	250	150	Hf	<0.3	0.3
Zn	40	100	Ta	<210	250
Ga	0.3	1	W	<0.4	0.9
Ge	<1	3	Re	<0.5	0.6
As	<90	60	Os	<3	5
Se	<4	2	Ir	<11	16
Br	<20	40	Pt	6	8
Rb	<0.4	0.5	Au	<150	80
Sr	<0.1	0.1	Hg	<5	4
Y	<0.2	0.3	Tl	1600	1600
Zr	<0.1	0.1	Pb	3000	2100
Nb	<0.6	0.5	Bi	90	610
Mo	<0.8	0.4	Th	<30	40
Ru	<0.5	1	U	<0.1	0.2
Rh	<1	1			

Supplementary data

The accompanying data sheets (available from doi.org/10.4224/crm.2022.hicd-1) provide elemental impurity results from the analyzed CRM units, the mass and chemical amount of cadmium in any given unit along with the mass of individual units and their expanded uncertainties. The serial number corresponding to each unit is located on the HICD-1 glass vial.

Certified values

Certified values are considered to be those for which the NRC has the highest confidence in accuracy and that all known and suspected sources of bias have been taken into account and are reflected in the stated expanded uncertainties. Certified values are the best estimate of the true value and uncertainty.

Intended use

This certified reference material is intended to be dissolved, in whole, to make a primary standard for the determination of the mass fraction of cadmium. If the procedure for preparing the primary standard solution involves weighing of the CRM, an appropriate cleaning should be performed to remove surface contamination.

Mass fractions of the impurities reported on this certificate are not intended for calibration purposes. This data is presented to allow users to compute/derive purity and assess the impact of concomitant impurities when a mixed element standard solution is prepared. However, if the impurity values are used in a measurement (X-ray fluorescence, laser ablation etc.) appropriate care should be taken to remove any surface contamination.

Storage

It is recommended that the material is stored at room temperature and the vials only be opened immediately prior to use in a clean area, taking precautions against contamination.

Preparation of material

The reference material was prepared from a high-purity cadmium metal rod of 2.3 mm diameter. It was cut into 1.0 g pieces of 29 mm length using a wire electrical discharge technique and bottled in 4 mL glass vials filled with argon.

Stability

Potential instabilities due to long-term storage and transport were considered, and such effects deemed to be negligible on the purity of the material. The material is deemed stable with respect to the certified values for ten years.

Homogeneity

The material was tested for homogeneity at NRC by analyzing 15 CRM units. See doi.org/10.4224/crm.2022.hicd-1 for data on each of these units.

Uncertainty

The overall combined uncertainty estimate includes the uncertainties in the batch characterization and uncertainties related to possible between-unit variation (homogeneity).

Sources of uncertainty considered for batch characterization include the primary standards, calibration model, and measurement repeatability.

Metrological traceability

Results of the elemental impurities and the mass fraction of cadmium (purity) presented in this certificate are traceable to the SI through a network of CRMs [2,3] supported by international measurement intercomparisons. As such, HICD-1 serves as a suitable reference material for laboratory quality assurance programs, as outlined in ISO/IEC 17025.

Quality Management System (ISO 17034, ISO/IEC 17025)

This material was produced in compliance with the NRC Metrology Quality Management System, which conforms to the requirements of ISO 17034 and ISO/IEC 17025. The Metrology Quality Management System supporting NRC Calibration and Measurement Capabilities, as listed in the *Bureau international des poids et mesures* (BIPM) Key Comparison Database (kcdb.bipm.org/), has been reviewed and approved under the authority of the Inter-American Metrology System (SIM) and found to be in compliance with the expectations of the *Comité international des poids et mesures* (CIPM) Mutual Recognition Arrangement. The SIM approval is available upon request.

Updates

For updates please refer to doi.org/10.4224/crm.2022.hicd-1

References

1. Evaluation of measurement data: Guide to the expression of uncertainty in measurement, JCGM 100:2008. <https://www.bipm.org/en/committees/jc/jcgm/publications>
2. Meija J, Methven B, Sturgeon R.E. (2017) Uncertainty of relative sensitivity factors in glow discharge mass spectrometry. *Metrologia*, 54: 796-804. doi.org/10.1088/1681-7575/aa83d6
3. Sturgeon RE, Methven B, Willie SN, Grinberg P. (2014) Assignment of purity to primary metal calibrants using a pin-cell VG 9000 glow discharge mass spectrometer - a primary method with direct traceability to the SI international system of units? *Metrologia* 51: 410-422. <http://dx.doi.org/10.1088/0026-1394/51/5/410>

Cited by

A list of scientific publications citing HICD-1 can be found at doi.org/10.4224/crm.2022.hicd-1.

Authorship

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HICD-1

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Approved by:



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This Certificate is only valid if the corresponding material was obtained directly from the NRC or an Authorized Reseller.

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