National Research Council Canada | Conseil national de recherches Canada

Certificate of Analysis

Certified Reference Material

HIPC-1

High Purity Carbon Certified Reference Material for Mass Fraction of Carbon, Carbon Isotope Delta and Elemental Impurities

HIPC-1 is a high purity carbon Certified Reference Material (CRM). A unit of HIPC-1 consists of approximately 0.25 g of high purity carbon rod. This material is intended as a primary standard for the determination of the mass fraction of carbon, and for carbon isotope delta measurements.

Certified values for the mass fraction of carbon, carbon isotope delta and mass fractions of elemental impurities have been established for HIPC-1. These values are listed in Tables 1-2. Certified values for the mass fraction of carbon and elemental impurities are based on measurements carried out at the National Research Council Canada (NRC). The certified value for carbon isotope delta was determined by combining the measurement results from three participating expert laboratories, including the NRC, using a random laboratory effects statistical model. The expanded uncertainty (U_{CRM}) in the certified values is equal to $U_{CRM} = ku_c$ 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 approximately 95 % confidence.

Table 1: Certified quantity values and expanded uncertainties ($k = 2$) of mass fraction of carbon and carbon isotope delta value in HIPC-1

Quantity	Value	Expanded uncertainty
Mass fraction of carbon, w(C)	0.999 89 kg/kg	0.000 27 kg/kg
Carbon isotope delta, $\delta_{VPDB}(^{13}C)$	–26.32 ‰	0.11 ‰

The mass fraction of carbon was calculated using the following mathematical expression:

$$w(C) = 1 - \sum_{E} w(E)$$

where 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.

The certified value for carbon isotope delta in HIPC-1 is expressed on the Vienna Peedee Belemnite (VPDB) scale with a value of –46.6 ‰ assigned to LSVEC and +1.95 ‰ to NBS19 [2].

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Table 2: Certified mass fractions and expanded uncertainties (k = 2) of elemental impurities in HIPC-1 (µg/kg)

Impurity element, E	Mass fraction, w(E)	Expanded uncertainty	Impurity element, E	Mass fraction, w(E)	Expanded uncertainty
Li	<8	19	Pd	<150	380
Be	<3	8	Ag	<24	61
B	1500	1800	Cd	<13	6
C	Matrix		In	<13	33
N	21 000	211 000	Sn	<110	270
O	45 000	139 000	Sb	<35	28
F	<4200	10 800	Te	<40	360
Na	680	3200	I	<9	22
Mg	<360	200	Cs	<39	22
Al	340	1000	Ba	<33	18
Si	5700	78 400	La	<5	6
P	90	490	Ce	<11	9
S	6700	6700	Pr	<9	24
Cl	2100	2100	Nd	<35	89
K	<2400	1400	Sm	<47	24
Ca	14 100	24 000	Eu	<12	11
Sc	<14	8	Gd	<39	19
Ti	<86	51	Tb	<9	24
V	<6	4	Dy	<35	21
Cr	<1700	1000	Ho	<9	24
Mn	<60	39	Er	<28	72
Fe	600	1800	Tm	<10	25
Co	<11	7	Yb	<30	76
Ni	<28	19	Lu	<10	25
Cu	6800	32 600	Hf	<62	50
Zn	<350	730	Ta	<300	770
Ga	<57	34	W	<91	50
Ge	<40	120	Re	<70	170
As	90	330	Os	<160	400
Se	<150	90	Ir	<110	280
Br	<170	430	Pt	<60	170
Rb	<70	60	Au	<180	460
Sr	<100	70	Hg	<310	160
Y	<6	3	Tl	<26	67
Zr	<10	120	Pb	<60	50
Nb	<14	35	Bi	<16	42
Mo	<67	40	Th	<45	27
Ru	<130	330	U	<45	25
Rh	<50	510			

Table 2 shows the measured quantities in HIPC-1. Elemental impurities 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 [3,4]. For the purpose of obtaining the mass fraction (purity) estimate of carbon, all elemental impurities below detection limit were interpreted as half the detection limit (for example '<8 µg/kg' for lithium is interpreted as 4 µg/kg). A robust estimator (median) was used to summarize the observed values from 15 units reported in Table 2.

Supplementary data

The accompanying datasheets (available from doi.org/10.4224/crm.2022.hipc-1) provide elemental impurity results from the analyzed CRM units, the mass and chemical amount of carbon along with the mass of individual units and their expanded uncertainties. The serial number corresponding to each unit is located on the HIPC-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

It is recommended to use solid sampling technique for the determination of the mass fraction of carbon.

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 to 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.

For carbon isotope delta measurements, HIPC-1 is intended to be used with at least one other reference material for calibration to the VPDB scale. A minimum sample mass of 100 µg is recommended.

Storage

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

Preparation of material

HIPC-1 was prepared from a 3 mm diameter, high-purity carbon rod. It was cut into 0.25 g pieces of 20 mm length using a Teflon blade 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

For the mass fraction of carbon, the material was tested for homogeneity at the NRC by analyzing 15 CRM units. See doi.org/10.4224/crm.2022.hipc-1 for data on each of these units.

For carbon isotope delta measurements, the homogeneity was assessed by analyzing 30 CRM units. The uncertainty due to homogeneity was evaluated using Bayesian random effects model and is included in the uncertainty budget.

Uncertainty

The overall combined standard uncertainty estimate (u_c) for mass fraction of carbon, elemental impurities and carbon isotope delta measurements includes the uncertainties in the batch characterization (u_{char}) and uncertainties related to between-unit variations (u_{hom}). For the mass fraction of carbon and elemental impurities, sources of uncertainty considered for batch characterization include the primary standards, calibration model, and measurement repeatability. For carbon isotope delta, these components are u_c = 0.05 ‰, u_{char} = 0.026 ‰, and u_{hom} = 0.047 ‰.

Metrological traceability

Results of the elemental impurities and the mass fraction of carbon (purity) presented in this certificate are traceable to the SI through a network of CRMs [3,4] supported by international measurement intercomparisons. As such, HIPC-1 serves as a suitable reference material for laboratory quality assurance programs, as outlined in ISO/IEC 17025.

The *Comité international des poids et mesures* (CIPM) has noted that isotope delta measurements that cannot be traceable to the SI should be made traceable to materials recognized as international standards by the IUPAC Commission on Isotopic Abundances and Atomic Weights. The carbon isotope delta value in HIPC-1 is traceable to such internationally recognized reference materials [5] (see Table 3) and serves as a suitable reference material for laboratory quality assurance programs, as outlined in ISO/IEC 17025.

Table 3: Re-assigned carbon isotope delta values and the associated standard uncertainties for the reference materials used to calibrate HIPC-1

Reference Material	$\delta_{VPDB}(^{13}C)$
IAEA-CH-6	–10.450(49) ‰
USGS65	–20.290(49) ‰
IAEA-600	–27.770(49) ‰
NBS22	–30.030(58) ‰
USGS61	–35.050(49) ‰
IAEA-603	+2.474(23) ‰
IAEA-610	–9.145(19) ‰
IAEA-611	–30.925(21) ‰
IAEA-612	–36.878(26) ‰
NBS19	+1.95 ‰

The standard uncertainties associated with IAEA-CH-6, USGS65, IAEA-600, NBS22 and USGS61 were revised by adding the uncertainty associated with the consistency between the reference materials (u = 0.029 ‰) [6] to their reported standard uncertainties [5,7-9]. Additionally, IAEA-603, IAEA-610, IAEA-611, and IAEA-612 were used as calibrators. However, the isotope delta values for these materials are certified relative to the VPDB with no reference to LSVEC [10,11]. Consequently, these values were converted to the VPDB scale as defined by NBS19 and LSVEC [12]. The reassessment of IAEA-603, IAEA-610, IAEA-611, and IAEA-612 did not include the aforementioned additional uncertainty due to lack of coherence because this suite of reference materials has been calibrated independent of the other internationally recognized reference materials.

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 CIPM Mutual Recognition Arrangement. The SIM approval is available upon request.

Updates

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

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Cited by

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

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HIPC-1

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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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