National Research Council Canada | Conseil national de recherches Canada

Certificate of Analysis

Certified Reference Material

UCHI-1

Natural Uranium Ore Concentrate (U_3O_8) Powder Certified Reference Material for Uranium Content, Uranium Isotope Ratios, and Trace Elements

UCHI-1 is a natural uranium ore concentrate powder Certified Reference Material (CRM) prepared at the National Research Council Canada (NRC). A unit of UCHI-1 consists of approximately 25 g of natural uranium ore concentrate. This CRM is intended for the calibration of measurement procedures and the development of methods for the determination of uranium content, uranium isotope ratios, and trace elements in uranium ore concentrate or similar matrices.

The following tables show those constituents for which certified, reference and information values have been established for this CRM. The expanded uncertainty (U_{CRM}) in the certified value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the principles of JCGM Guide 100 [1] and k is the coverage factor. A coverage factor of two (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. All values are expressed on an “as-is” basis, without dry weight correction. The moisture content in UCHI-1 is estimated at approximately 0.003 g/g.

Table 1: Uranium content and expanded uncertainty (*k*=2) in UCHI-1

Quantity	Value	Type
uranium content, *w*(U), mg/kg	833 000 ± 10 000	certified

Table 1 shows the consensus value of uranium content in UCHI-1. The primary method used was multi-collector inductively-coupled plasma mass spectrometry (MC-ICP-MS) using external calibration and isotope dilution approaches. Other methods were also used and the instrumentation used were thermal ionization mass spectrometry (TIMS), inductively-coupled plasma atomic emission spectroscopy (ICP-AES), electron microprobe analysis (EMPA), and potentiometric titration. The consensus value for uranium content was assigned from the interlaboratory study using Laplace random effects statistical model [2].

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Table 2: Uranium isotope ratios and expanded uncertainties (*k*=2) in UCHI-1

Quantity	Value	Expanded uncertainty	Type
Isotope ratio, $n(^{234}U)/n(^{238}U)$, mol/mol	0.000 054 75	0.000 000 02	certified
Isotope ratio, $n(^{235}U)/n(^{238}U)$, mol/mol	0.007 256 3	0.000 001 0	certified

Table 2 shows the consensus values of uranium isotope ratios in UCHI-1. Uranium isotope ratios were determined by MC-ICP-MS and TIMS using mass bias correction models which include gravimetric isotope mixture method, standard-sample bracketing, and exponential law. The consensus values for the isotope ratios were assigned from the interlaboratory study using Gaussian random effects model (DerSimonian-Laird) [2].

Table 3: Mass fractions and expanded uncertainties (*k*=2) for trace elements in UCHI-1

Element, E	Mass fraction, *w*(E), mg/kg	Mass fraction ratio, *w*(E)/*w*(U), mg/kg	Type	International recognition of measurement capability (CMC)
aluminium	4.1 ± 1.2	4.9 ± 1.5	certified	--
antimony	0.09	0.10	information	TES01
arsenic	161 ± 9	193 ± 18	certified	TES02
barium	0.29 ± 0.06	0.35 ± 0.08	certified	MYC01
bismuth	0.098 ± 0.009	0.118 ± 0.019	certified	MYC02
cadmium	6	7	information	TES04
calcium	68 ± 11	82 ± 15	certified	MES25
cerium	0.017 ± 0.007	0.020 ± 0.009	reference	MYC03
caesium	0.03	0.03	information	MYC04
chromium	0.34 ± 0.08	0.41 ± 0.10	certified	TES05
cobalt	0.199 ± 0.010	0.239 ± 0.031	certified	TES06
copper	0.87 ± 0.14	1.04 ± 0.20	certified	TES07
dysprosium	0.022 ± 0.006	0.026 ± 0.008	certified	--
erbium	0.0065 ± 0.0022	0.0078 ± 0.0029	certified	MYC05
europium	0.0017 ± 0.0016	0.0020 ± 0.0019	reference	MYC06
gadolinium	0.011 ± 0.004	0.013 ± 0.005	certified	MYC07
hafnium	0.065 ± 0.021	0.078 ± 0.027	reference	--
holmium	0.0030 ± 0.0010	0.0036 ± 0.0014	certified	MYC10
indium	0.006	0.007	information	MYC11
iron	27 ± 3	32 ± 4	certified	MES26
lanthanum	0.0045 ± 0.0027	0.0054 ± 0.0034	reference	MYC12
lead*	-	-	-	TES08
lithium	0.057 ± 0.016	0.069 ± 0.021	certified	TES09

Element, E	**Mass fraction, *w*(E), mg/kg**	**Mass fraction ratio, *w*(E)/*w*(U), mg/kg**	**Type**	**International recognition of measurement capability (CMC)**
lutetium	0.001	0.001	information	MYC13
magnesium	10.2 ± 1.2	12.3 ± 1.9	certified	MES28
manganese	1.62 ± 0.09	1.95 ± 0.23	certified	TES10
mercury	4	5	information	TES11
molybdenum	5100 ± 400	6200 ± 629	certified	TES12
neodymium*	-	-	-	MYC14
nickel	2.9 ± 0.3	3.4 ± 0.5	certified	TES13
niobium	0.54 ± 0.02	0.64 ± 0.08	certified	MYC15
palladium*	-	-	-	--
phosphorus	5.8 ± 2.8	7.0 ± 3.4	reference	MES30
potassium	15.0 ± 1.7	18.0 ± 2.6	certified	MES27
praseodymium*	-	-	-	--
rhenium	0.0036 ± 0.0021	0.0043 ± 0.0025	reference	MYC18
rubidium	0.051	0.061	information	MYC17
ruthenium	0.021	0.025	information	--
samarium	0.019	0.022	information	MYC19
scandium*	-	-	-	MYC20
silicon*	-	-	-	MES33
silver*	-	-	-	TES15
sodium	297 ± 8	357 ± 28	certified	MES29
strontium	0.44 ± 0.03	0.53 ± 0.07	certified	TES16
sulfur	3700 ± 300	4500 ± 470	certified	MES31
tantalum	0.0034 ± 0.0010	0.0040 ± 0.0013	certified	MYC21
tellurium*	-	-	-	MYC22
terbium	0.0030 ± 0.0010	0.0037 ± 0.0014	reference	MYC23
thallium*	-	-	-	--
thorium	0.0075 ± 0.0021	0.0089 ± 0.0028	reference	--
thulium	0.0009 ± 0.0007	0.0010 ± 0.0008	reference	MYC24
tin*	-	-	-	--
titanium	1.13 ± 0.26	1.36 ± 0.35	reference	--
tungsten	220 ± 13	264 ± 26	certified	MYC25
vanadium	0.151 ± 0.016	0.181 ± 0.030	certified	--
ytterbium	0.0035 ± 0.0007	0.0042 ± 0.0010	certified	MYC26
yttrium	0.075 ± 0.012	0.090 ± 0.018	certified	MYC27
zinc	1.4 ± 0.4	1.7 ± 0.5	certified	--
zirconium	139 ± 12	167 ± 19	certified	MYC28

*No consensus was obtained for the mass fraction of these elements. Reported data are provided in the supplementary document available at: doi.org/10.4224/crm.2020.uchi-1.

Table 3 shows the consensus values of trace element impurities in UCHI-1. The primary method used was ICP-MS using external calibration, standard addition and isotope dilution approaches. A total reflection X-ray fluorescence (TXRF) spectrometer was also used to obtain mass fraction of trace element impurities. The consensus values for the mass fractions of all elements were assigned from the interlaboratory study using Laplace random effects model [2].

A supplementary data file is also available at doi.org/10.4224/crm.2020.uchi-1 showing all the reported mass fractions for UCHI-1 that were used to obtain the consensus values.

International recognition of measurement capability

The measurement capabilities supporting these results are registered at the Calibration and Measurement Capabilities (CMC) database of the *Bureau international des poids et mesures* (BIPM) indicating recognition of the measurement certificates by National Metrology Institutes (NMIs) participating in the Mutual Recognition Arrangement (MRA) with the corresponding identifiers. Lists of all registered measurement capabilities in sediments, soils, ores and particulates matrices can be found in the BIPM database at https://www.bipm.org/kcdb/

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.

Reference values

Reference values are those for which insufficient data are available to provide a comprehensive estimate of uncertainty.

Information values

Information values are those for which insufficient data are available to provide any estimate of uncertainty.

Additional data

For elements where no consensus was obtained for the mass fraction, reported data are provided in the supplementary document available at: doi.org/10.4224/crm.2020.uchi-1.

Intended use

This CRM is primarily intended for the calibration of procedures and the development of methods for the determination of uranium content, uranium isotopic ratios and trace elements in uranium ore concentrate or similar matrices. A minimum sample mass of 250 mg is recommended to analyze the uranium content, uranium isotope ratios, and trace elements.

Storage and sampling

It is recommended that the material is stored at room temperature and the vials opened immediately prior to use in a clean area with precautions taken against contamination. Prior to each use, contents of the bottle should be well mixed by gentle shaking and rolling of the container, and tightly closed immediately thereafter.

Preparation of material

UCHI-1 is an industrial-grade uranium ore concentrate. The material was homogenized and bottled in 60 mL amber glass bottles.

Stability

Similar NRC CRMs have been monitored for trace elements for more than ten years and found to be both physically and chemically stable over this time. We expect similar behaviour of UCHI-1. Effects from potential instabilities due to long-term storage and transport were deemed to be negligible on the isotopic composition, the trace element impurities and the uranium content.

Homogeneity

The material was tested for homogeneity at NRC. Results from 10+ sub-samples (250 mg) were evaluated using DerSimonian-Laird random effects model and the resulting relative uncertainty due to homogeneity was regressed against the mass fraction of the element using Horwitz-type power law. The obtained trend was used to assign uncertainty due to homogeneity for all elements with the exception of uranium.

Uncertainty

Included in the overall combined uncertainty estimate are the uncertainties in the batch characterization, uncertainties related to possible between-unit variation, and uncertainties related to inconsistency between the various measurement methods/laboratories. The latter is estimated as the heterogeneity in the random effects model fitted to the results of individual methods, also known as the dark uncertainty [3,4]. Standard random effects model (DerSimonian-Laird) was adopted for isotope ratios. Measurements of trace elements and uranium content, however, were challenging in this material and consequently the consensus building necessitated a more robust statistical model. As a result, we selected Laplace random effects model.

Metrological traceability

Results presented in this certificate are traceable to the SI through gravimetrically-prepared standards of established purity, CRM and international measurement intercomparisons. As such, UCHI-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

Users should ensure that the certificate they have is current. Our website at www.nrc.gc.ca/crm will contain any new information.

References

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Cited by

A list of scientific publications citing UCHI-1 can be found at doi.org/10.4224/crm.2020.uchi-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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