National Research Council Canada | Conseil national de recherches Canada

Certificate of Analysis

Certified Reference Material

UCLO-1

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

UCLO-1 is a natural uranium ore concentrate powder Certified Reference Material (CRM) prepared at the National Research Council Canada (NRC). A unit of UCLO-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 UCLO-1 is estimated at approximately 0.005 g/g.

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

Quantity	Value	Type
uranium content, w(U), mg/kg	838 000 ± 9000	certified

Table 1 shows the consensus value of uranium content in UCLO-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 UCLO-1

Quantity	Value	Expanded uncertainty	Type
Isotope ratio, $n(^{234}U)/n(^{238}U)$, mol/mol	0.000 053 04	0.000 000 07	certified
Isotope ratio, $n(^{235}U)/n(^{238}U)$, mol/mol	0.007 256 3	0.000 001 3	certified

Table 2 shows the consensus values of uranium isotope ratios in UCLO-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 UCLO-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	2.7 ± 0.8	3.3 ± 1.0	certified	--
antimony	0.2	0.3	information	TES01
arsenic	0.5 ± 0.4	0.5 ± 0.4	reference	TES02
barium	0.14	0.17	information	MYC01
bismuth	48 ± 2	57 ± 5	certified	MYC02
cadmium	4.3 ± 0.3	5.1 ± 0.6	certified	TES04
calcium	6	7	information	MES25
cerium	0.13 ± 0.06	0.15 ± 0.07	reference	MYC03
caesium	0.07	0.08	information	MYC04
chromium	0.45 ± 0.18	0.54 ± 0.22	reference	TES05
cobalt	0.029 ± 0.005	0.034 ± 0.007	certified	TES06
copper	39 ± 2	47 ± 4	certified	TES07
dysprosium*	-	-	-	--
erbium	0.006	0.008	information	MYC05
europium	0.01	0.02	information	MYC06
gadolinium	0.011	0.013	information	MYC07
gallium	0.09	0.10	information	MYC08
germanium	0.2	0.2	information	MYC09
hafnium*	-	-	-	--
holmium	0.0025	0.0030	information	MYC10
indium	0.216 ± 0.009	0.258 ± 0.033	certified	MYC11
iridium	0.027	0.032	information	--
iron	20.7 ± 1.2	24.8 ± 2.6	certified	MES26

Element, E	**Mass fraction, *w*(E), mg/kg**	**Mass fraction ratio, *w*(E)/*w*(U), mg/kg**	**Type**	**International recognition of measurement capability (CMC)**
lanthanum	0.07 ± 0.04	0.08 ± 0.05	reference	MYC12
lithium	0.020 ± 0.010	0.024 ± 0.013	reference	TES09
magnesium*	-	-	-	MES28
manganese	0.31 ± 0.07	0.37 ± 0.09	certified	TES10
molybdenum	72 ± 4	86 ± 8	certified	TES12
neodymium	0.029 ± 0.011	0.034 ± 0.014	reference	MYC14
nickel	0.34 ± 0.28	0.40 ± 0.33	reference	TES13
niobium	12.9 ± 1.2	15.4 ± 2.0	certified	MYC15
platinum	0.09	0.11	information	--
potassium	13.3 ± 1.8	15.8 ± 2.6	certified	MES27
praseodymium	0.010 ± 0.006	0.012 ± 0.007	reference	--
rhenium	0.0085 ± 0.0024	0.0101 ± 0.0033	reference	MYC18
rubidium	0.09 ± 0.04	0.10 ± 0.05	reference	MYC17
ruthenium*	-	-	-	--
samarium	0.011 ± 0.007	0.013 ± 0.009	reference	MYC19
scandium	0.74 ± 0.21	0.89 ± 0.27	reference	MYC20
silicon*	-	-	-	MES33
silver	7.4 ± 2.2	8.8 ± 2.7	certified	TES15
sodium	145 ± 5	173 ± 15	certified	MES29
strontium	0.13	0.15	information	TES16
sulfur	3000 ± 230	3580 ± 349	certified	MES31
tantalum	0.015	0.018	information	MYC21
tellurium*	-	-	-	MYC22
terbium*	-	-	-	MYC23
tin	0.74 ± 0.11	0.88 ± 0.16	certified	--
titanium	1.5	1.8	information	--
tungsten	1.37 ± 0.12	1.64 ± 0.22	certified	MYC25
yttrium	0.033 ± 0.016	0.039 ± 0.020	reference	MYC27
zinc	1140 ± 60	1360 ± 115	certified	--
zirconium	1120 ± 60	1340 ± 118	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.uclo-1.

Table 3 shows the consensus values of trace element impurities in UCLO-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.uclo-1 showing all the reported mass fractions for UCLO-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.uclo-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

UCLO-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 UCLO-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, UCLO-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 UCLO-1 can be found at doi.org/10.4224/crm.2020.uclo-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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