National Research Council Canada | Conseil national de recherches Canada

Certificate of Analysis

Certified Reference Material

CACB-1

Calcium Carbonate Certified Reference Material for Lead and Cadmium

CACB-1 is a Calcium Carbonate Certified Reference Material (CRM) from the National Research Council Canada (NRC) with information on lead and cadmium content. A unit of CACB-1 consists of approximately 2.5 grams of calcium carbonate.

Table 1 shows the certified values established for CACB-1. Certified values are based on unweighted mean results from data generated by two independent methods. 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 JCGM Guide [1] and k is the coverage factor. The value of u_c is determined from the combined uncertainties of the various analytical methods (u_{char}) as well as uncertainties associated with homogeneity (u_{hom}). It is intended that U_{CRM} encompasses every aspect that reasonably contributes to the uncertainty of the measurement [2]. A coverage factor k=2 (approx. 95 % confidence) was applied for all elements.

Table 1: Mass fractions and expanded uncertainties (k = 2) for CACB-1

Element	Mass fraction, ng/g
cadmium	7.6 ± 1.1
lead	243 ± 14

Intended use

CACB-1 is intended for the calibration of instruments and evaluation of methods for the determination of Pb and Cd in calcium carbonate or materials of a similar matrix.

Storage and sampling

This material should be kept tightly closed in the original bottle and should be stored in a cool location. The contents should be well mixed by rotation and shaking prior of the bottle prior to use. The bottle should be tightly closed immediately after use.

Preparation of material

This material was obtained from a commercial producer. It was blended and bottled into precleaned glass bottles in a Class 100 clean room at the NRC.

Canada

Certified value

All measurements contributing to this certificate were conducted at NRC Ottawa. The certified values were calculated from the unweighted means of results from isotope dilution inductively coupled plasma mass spectrometry (ID-ICPMS) and electrothermal vapourization atomic absorption spectrometry (ETAAS). Both employed a preconcentration step. A column of iminodiacetate resin was used with ICP-MS whereas immobilized 8-hydroxyquinoline was used with ETAAS.

Guidelines for CRM producers suggest all sources relevant to the user of the material should contribute to the uncertainty of the certified value [2-6]. Included in the overall uncertainty estimate are uncertainties in the characterization (u_{char}), uncertainties related to possible between-bottle variation (u_{hom}) as well as instability derived from effects relating to long-term storage and transport (u_{stab}). Results for the uncertainty components used to calculate the certified values are shown in Table 2.

Characterization

The characterization uncertainties were calculated from the standard deviation of the laboratory means. The uncertainty components related to the characterization of CACB-1 are reported in Table 2 [3].

Homogeneity

The homogeneity components of the uncertainty in the certified values were derived from duplicate sub-samples (0.150 g) from twelve bottles of CACB-1. The results were evaluated using ANOVA [6]. Table 2 summarizes the resulting uncertainty components for homogeneity.

Stability

Instability is not expected in the Pb and Cd content of CACB-1 provided the CRM is handled and stored in accordance with the instructions. However, this CRM will continue to be monitored and users will be notified if any observed significant irregularity occurs prior to the expiry date.

Table 2: Uncertainty Components for CACB-1

Element	u_{char}/(ng/g)	u_{hom}/(ng/g)	u_c/(ng/g)	U_{CRM} (k=2)
cadmium	0.50	0.12	0.53	1.1
lead	7.1	1.0	7.2	14

Information Values

A lack of independent values precluded the certification of the elements listed in Table 3. Information values for these measurands are given below along with their associated standard deviations (s) derived from duplicate results from twelve bottles of CACB-1.

Table 3: Information Values for Trace Metals in CACB-1

Element	Mass fraction, ng/g	
	Mean	***s***
Ba	3.45	0.02
Sr	225	1
Ni	4.33	0.03
Cu	0.35	0.01
Zn	4.88	0.10
Mo	0.40	0.01

Metrological traceability

Results presented in this certificate are traceable to the International System of Units (SI) through CRMs produced by National Metrology Institutes and gravimetrically prepared standards of established purity. As such, CACB-1 serves as 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. For updates, please refer to doi.org/10.4224/crm.2005.cacb-1.

References

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Cited by

A list of scientific publications citing CACB-1 can be found at doi.org/10.4224/crm.2005.cacb-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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