National Research Council Canada
Conseil national de recherches Canada

Certified Reference Material

CACB-1

Calcium Carbonate Certified Reference Material for Lead and Cadmium

The following table shows those elements for which certified values have been established for this calcium carbonate reference material (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 ISO 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 measurand [2]. A coverage factor of 2 was applied for all elements. The table below lists measurands certifed in CACB-1.

Certified Values

Cd	**7.6 ± 1.1 ng/g**
Pb	**243 ± 14 ng/g**

NRC·CNRC

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

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.

Expiration of Certification

The certified values for FEBS-1 are considered valid until April 2015, provided the CRM is handled and stored in accordance with instructions herein.

Preparation

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 National Research Council Canada.

Certified value

All measurements contributing to this certificate were conducted at NRCC Ottawa.

The certified values were calculated from the unweighted means of results from isotope dilution inductively coupled plasma mass spectrometry (ID-ICP-MS) and electrothermal vapourization atomic absorption specrometry (ETAAS).

Both employed a preconcentration step. A column of iminodiacetate resin was used with ICP-MS whereas immobilized 8-hydroxyquinoline was used with ETAAS.

Uncertainties

New 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 characterisation (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}). Expressed as standard uncertainties, these components can be combined as:

$$u^2_{c(CRM)} = u^2_{char} + u^2_{hom} + u^2_{stab} \qquad (1)$$

Results for the various uncertainty components used to calculate the certified values are shown in Table 2.

Characterisation

The characterisation uncertainties (u_{char}) were calculated in accordance with equations 2 and 3 [3],

$$u_c(l) = \frac{\sqrt{\sum [u_c]^2}}{l} \qquad (2)$$

where $u_c(l)$ is calculated from the method dependent uncertainties and l is the number of methods.

Since the method means were not completely identical, a residual component (u(R)) corresponding to the standard uncertainty of the average of the laboratory means was also considered:

$$u_c(R) = \frac{s_{betw}}{\sqrt{l}} \qquad (3)$$

where s_{betw} is the standard deviation of the laboratory means. The calculated uncertainty components related to the characterization of CACB-1 are reported in Table 2.

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.

For Cd and Pb, the situation depicted in equation 4 occurred:

$$s_{betw}^2 < \frac{s_{meas}^2}{n} \qquad (4)$$

where s_{meas} is the repeatability standard deviation for the method used in the homogeneity assessment and *n* is the number of replicates per unit.

In these cases, u_{hom} was calculated according to equation 5:

$$u_{\text{hom}} = \sqrt{\frac{MS_{within}}{n}} \sqrt[4]{\frac{2}{v_{MS_{within}}}} \qquad (5)$$

where MS_{within} represents the mean squares within groups and $v_{MS_{within}}$ is the number of degrees of freedom. [6]

Table 1 summarizes the resulting uncertainty components for homogeneity.

Stability

Instability is not expected in the Pb and Cd content of CALC-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.

Source	Uncertainty, ng/g Cd	Pb
u_{char}	0.50	7.1
u_{bb}	0.12	1.0
u_c	0.53	7.2
U_{CRM} (k=2)	**1.1**	**14**

Information Values

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

Table 3.

	Information Values, µg/g mean	S.D.
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

Updates

It is anticipated that as more data become available, reliable may be values assigned to more elements.

Our web site will contain any new information.

References

[1] Guide to the Expression of Uncertainty in Measurement, ISBN 92-67-10188-9, 1st ed. ISO, Geneva, Switzerland (1993).

[2] J. Pauwels, A. van der Veen, A. Lamberty, H. Schimmel, Evaluation of uncertainty of reference materials. Accred Qual Assur (2000) 5:95-99.

[3] J. Pauwels, A. Lamberty, H. Schimmel The determination of the uncertainty of reference materials certified by laboratory intercomparison. Accred Qual Assur (1998) 3: 180–184

[4] A. M.H.van der Veen and J. Pauwels, Uncertainty calculations in the certification of reference materials. 1.Principles of analysis of variance. Accred Qual Assur (2000) 5:464–469.

[5] A. M.H. van der Veen, T. P.J. Linsinger, H. chimmel, A. Lamberty and J. Pauwels, Uncertainty calculations in the certification of reference materials 4. Characterisation and certification. Accred Qual Assur (2001) 6:290–294.

[6] T.P.J. Linsinger, J. Pauwels, A.M.H. Van der Veen , H. Schimmel, A. Lamberty , Homogeneity and Stability of Reference Materials", Accred Qual Assur (2001), 6: 20-25.

Acknowledgements

The following members of staff of the Institute for National Measurement Standards, National Research Council Canada, participated in the preparation and certification: C. Scriver, S. Willie, L. Yang.

Certificate issued August 2005.
Date of expiry April 2015.

The results listed in this certificate are traceable to the SI through gravimetrically prepared standards of established purity and international measurement intercomparisons. As such, they serve as suitable reference materials for laboratory quality assurance programs, as outlined in ISO/IEC 17025.

Visit our web site at
http://inms-ienm.nrc-cnrc.gc.ca/calserv/chemical_metrology_e.html#certified

Comments, information and inquiries should be addressed to:

Dr. R.E. Sturgeon
National Research Council of Canada
Institute for National Measurement Standards
M-12, 1200 Montreal Road
Ottawa, Ontario, Canada K1A 0R6

Telephone (613) 993-2359
Facsimile (613) 993-2451
E-mail crm.inms@nrc-cnrc.gc.ca

Également disponible en français sur demande.

Canada