National Research Council Canada | Conseil national de recherches Canada

Certificate of Analysis

Certified Reference Material

SOPH-1

Marine Sediment Certified Reference Material for the Determination of Butyltin Compounds

SOPH-1 is a marine Sediment Certified Reference Material (CRM) from the National Research Council of Canada (NRC) for butyltin compounds content. A unit of SOPH-1 consists of approximately 12 grams of marine sediment. This material is intended for use in the calibration of procedures and the development of methods for the determination of butyltin compounds in marine sediments and materials with similar matrices.

Table 1 shows the certified and information values established for SOPH-1. The expanded uncertainties associated with the certified and reference values were calculated according to the JCGM Guide [1] and correspond to approx. 95 % confidence (k = 2). All listed values are expressed on a dry mass basis.

Table 1: Mass fractions and expanded uncertainty (k = 2) for SOPH-1

Compound	Mass fraction, ng/g	Type
dibutyltin (as Sn)	174 ± 9	certified
monobutyltin (as Sn)	170	information
tributyltin (as Sn)	125 ± 7	certified

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.

The certified values for SOPH-1 are based on the unweighted mean of results from data submitted by laboratories participating in several *Comité Consultatif pour la Quantité de Matière* (CCQM) comparisons.

Information Values

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

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Background

CCQM-P18, CCQM-P43 and CCQM-K28 were undertaken in 2002 and 2003 to assess the current capabilities of interested National Metrology Institutes (those which are members of the Consultative Committee for Amount of Substance) and selected outside expert laboratories to quantitate tributyltin and dibutyltin in a prepared marine sediment. These exercises were sanctioned by the CCQM as an activity of the Inorganic Analysis Working Group, and were jointly piloted by the National Research Council Canada (NRC) and LGC Ltd (UK). A detailed report addressing CCQM-P18 is published in Metrologia [2].

Characterization

Mean values submitted by selected laboratories participating in CCQM P-18, CCQM P-43 and CCQM K-28 were used to calculate the original certified value and the uncertainty of characterization (u_{char}) [3].

Intended Use

This reference material is primarily intended for use in the calibration of procedures and the development of methods for the determination of $(C_4H_9)_3Sn^+$ (tributyltin, TBT) and $(C_4H_9)_2Sn^{2+}$ (dibutyltin, DBT) in sediments or materials of a similar matrix. A minimum sample mass of 500 mg is recommended.

Storage and Sampling

To ensure the stability of the organotin species it is necessary to store these materials at a temperature of –20 °C. Prior to use, the bottle should be well mixed by rotation, shaken and tightly closed immediately thereafter.

Instructions for Drying

A separate aliquot of the sediment should be used to obtain a dry weight correction factor. Drying for two hours at 105 °C has proved to be a relatively simple method to achieve a dry mass for most purposes.

Preparation of Material

SOPH-1 was prepared in-house at NRC following gravimetric blending of two NRC CRM marine sediments, PACS-2 and HISS-1, the latter containing negligible concentrations of TBT and thus serving as a solid, inert diluent. Table 2 and Figure 1 provide information values about the particle size distribution of SOPH-1.

Table 2: Particle Size of SOPH-1

SOPH-1	Particle size, µm
Median	12.0
Mean	13.8
Mode	18.6

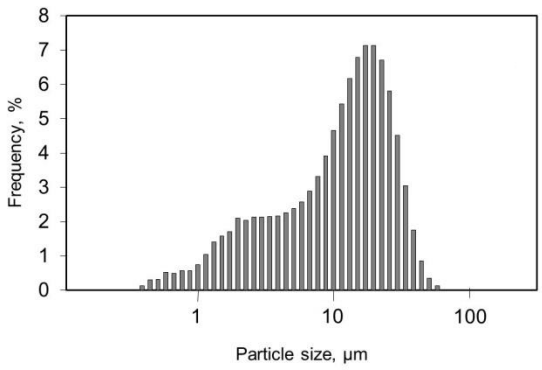

Figure 1: Particle Distribution for SOPH-1

Stability

An uncertainty component associated with the short and long term stabilities were evaluated and considered insignificant.

Homogeneity

These materials were tested for homogeneity at the NRC. Also, randomly bottles were selected for the analysis by the participating laboratories. The homogeneity is warranted for samples of 500 mg and above.

Metrological traceability

Results presented in this certificate are traceable to the International System of Units (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.

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.2004.soph-1

References

1. Evaluation of measurement data: Guide to the expression of uncertainty in measurement JCGM100:2008. https://www.bipm.org/en/publications/guides/gum.html

2. R. Sturgeon and R. Wahlen, Metrologia, (2002),39, *Tech. Suppl.*, 08003. https://doi.org/10.1088/0026-1394/39/1A/21

3. A.M.H. van der Veen, T.P.J. Linsinger, H.Schimmel, A. Lamberty and J. Pauwels, Accred Qual Assur (2001) 6:290–294. https://doi.org/10.1007/PL00010459

Cited by

A list of scientific publications citing SOPH-1 CRM can be found at doi.org/10.4224/crm.2004.soph-1.

Authorship

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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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