

National Institute for Environmental Studies

Certificate of Analysis

NIES CRM No. 15 Scallop

This environmental certified reference material (CRM) was developed and certified by the National Institute for Environmental Studies (NIES) for the determination of tributyltin [TBT: $\text{Sn}(\text{C}_4\text{H}_9)_3^+$], triphenyltin [TPT: $\text{Sn}(\text{C}_6\text{H}_5)_3^+$], and total tin in scallops and materials of similar matrix.

Certified Values

Compound	Mass fraction			Analytical method *
	Unit	Certified value	Uncertainty	
TBT ($\text{Sn}(\text{C}_4\text{H}_9)_3^+$)	mg/kg	0.404	0.027	GC-MS, LC-MS
TPT ($\text{Sn}(\text{C}_6\text{H}_5)_3^+$)	mg/kg	0.0170	0.0017	GC-MS, LC-MS

The uncertainty attached to the certified values is the expanded uncertainty using a coverage factor $k = 2$, corresponding to the half-width of a confidence interval of approximately 95 %.

All certified values were determined based on dry mass.

* GC-MS, gas chromatography-mass spectrometry

LC-MS, liquid chromatography-mass spectrometry

Compound	Mass fraction			Analytical method *
	Unit	Certified value	Uncertainty	
Total Tin (Sn)	mg/kg	0.179	0.021	ICP-MS, ID-ICP-MS, INAA

The uncertainty attached to the certified values is the expanded uncertainty using a coverage factor $k = 2$, corresponding to the half-width of a confidence interval of approximately 95 %.

All certified values were determined based on dry mass.

* ICP-MS, inductively coupled plasma-mass spectrometry

ID-ICP-MS, isotope dilution-inductively coupled plasma-mass spectrometry

INAA, instrumental neutron activation analysis

Characterization

The property values of the material were statistically determined based on chemical analyses by 21 organizations using a wide range of methods. A property value satisfying the following conditions was accepted as a certified value:

- 1) the relative standard deviation associated with the mean of the laboratory means was 5 % or less,

- 2) the number of laboratories contributing to the mean of the laboratory means was at least six, and
- 3) the number of methods contributing to the mean of the laboratory means was at least two.

The uncertainty attached to the certified values is the expanded uncertainty using a coverage factor $k = 2$, corresponding to the half-width of a confidence interval of approximately 95 %. Property values for the elements except tin are given as reference values. All certified and reference values were determined based on dry mass.

Reference Values

Element	Mass fraction		Analytical method *
	Unit	Reference value	
Magnesium (Mg)	%	0.179	ICP-OES, INAA, XRF
Phosphorus (P)	%	0.956	ICP-OES, XRF
Potassium (K)	%	1.65	ICP-OES, INAA, XRF
Sodium (Na)	%	0.561	ICP-OES, INAA
Arsenic (As)	mg/kg	3.76	AS, F-AAS, HR-ICP-MS, ICP-MS, INAA, MIP-MS, XRF
Bromine (Br)	mg/kg	43.7	INAA, XRF
Copper (Cu)	mg/kg	0.889	ICP-MS, ICP-OES
Iron (Fe)	mg/kg	10.8	ICP-MS, ICP-OES
Zinc (Zn)	mg/kg	56.1	ICP-MS, ICP-OES, INAA

All reference values were determined based on dry mass.

* AS, absorption spectrophotometry

F-AAS, furnace-atomic absorption spectrometry

HR-ICP-MS, high resolution-inductively coupled plasma-mass spectrometry

ICP-MS, inductively coupled plasma-mass spectrometry

ICP-OES, inductively coupled plasma-optical emission spectrometry

INAA, instrumental neutron activation analysis

MIP-MS, microwave induced plasma-mass spectrometry

XRF, X-ray fluorescence spectrometry

Description of the Material

The material consists of freeze dried and powdered adductor muscle of scallop (20 g) in a vacuum-packed in a plastic bag.

Homogeneity

TBT and TPT, and the elements were determined in material taken from 10 bags randomly selected from the 1380 bags, using LC-MS, and ICP-OES or ICP-MS, respectively. The between-bottle variation evaluated by a one-way analysis of variance (ANOVA) showed the relative standard deviations between bags for the analytes to be less than 1 %. The material, therefore, is sufficiently homogeneous for its intended use as a reference material.

Instructions for Use

1. This CRM should be kept tightly closed in its original bag and stored at ≤ -20 °C.
2. Prior to weighing aliquots for analysis, the contents of the bag should be shaken gently.
3. For convenience of handling a sample intake of 1 g is recommended.
4. Precautions should be taken to avoid inhalation of the material.
5. This CRM should not be used for purposes other than research. When disposing of the material, local laws concerning processing and disposal of waste materials should be strictly adhered to.
6. The mass fractions of elements in this CRM are reported on a dry mass basis. This CRM, as received, contains 2-5 % water. Correction to dry mass should be determined by drying a separate sub-sample for 4 hours at 85 °C.
7. This CRM contains 1.65 % potassium, 0.956 % phosphorus, 0.561 % sodium, and 0.179 % magnesium. An appropriate analytical method should be selected.

Expiry Date of Certification

The expiry date for the certified values of this CRM is November 2030 assuming that the recommended storage conditions are adhered to. NIES will notify via its website if any changes in the contents are recognized within the term of validity.

Collaborating Laboratories in Analysis

The certified and reference values for this CRM were based on the analytical values from the following participating organizations:

National Institute for Environmental Studies; Chiba Institute of Technology; Environmental Control Center Co., Ltd.; Environmental Research Center Co., Ltd.; Green Blue Corporation; Gunma University; IDEA Consultants, Inc.; Japan Atomic Energy Research Institute; Japan Food Research Laboratories; Kanagawa Industrial Technology Center; Kaneka Techno Research Co., Ltd.; Miura Co., Ltd.; Murata Keisokuki Service Co., Ltd.; Nippon Telegraph and Telephone Corporation; Nittech Research Corporation; Osaka Prefecture University; Rigaku Corporation; Shimadzu Techno-Research, Inc.; Tokyo City University; Tokyo Metropolitan Industrial Technology Research Institute; University of Tokyo.

Technical Information

Technical information and the latest reports regarding this material can be obtained from the website.

<http://www.nies.go.jp/labo/crm-e/index.html>

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