



Certificate of Analysis

Certified Reference Material

TORT-3

Lobster Hepatopancreas Certified Reference Material for Trace Metals

TORT-3 is a lobster hepatopancreas Certified Reference Material (CRM) from the National Research Council of Canada (NRC) with information on total trace element and species content. A unit of TORT-3 consists of approximately 15 g of lobster hepatopancreas in an amber glass vial.

Table 1 shows the certified and information values established for TORT-3. The expanded uncertainties associated with the certified 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 TORT-3

Analyte	Mass fraction, mg/kg	Type of value	International recognition of measurement capability (CMC)
arsenic (a,b)	59.5 ± 3.8	certified	SIM-QM-CA-000001E8-1
arsenobetaine (as As) (c,g)	54.9 ± 2.5	certified	SIM-QM-CA-000001E9-1
inorganic arsenic (as As) (g,h)*	0.596 ± 0.029	certified	--
dimethylarsinic acid (as As) (g,h)	3.44 ± 0.41	reference	--
monomethylarsonic acid (as As) (g,h)	0.307 ± 0.034	reference	--
cadmium (b,d)	42.3 ± 1.8	certified	SIM-QM-CA-000001EA-1
chromium (b,d)	1.95 ± 0.24	certified	SIM-QM-CA-000001EC-1
cobalt (a)	1.06	information	SIM-QM-CA-000001ED-1
copper (a,b,d)	497 ± 22	certified	SIM-QM-CA-000001EE-1
iron (a,b)	179 ± 8	certified	SIM-QM-CA-000001EF-1
lead (a,d)	0.225 ± 0.018	certified	SIM-QM-CA-000001EG-1
manganese (a,d)	15.6 ± 1.0	certified	SIM-QM-CA-000001EI-1
mercury (d)	0.292 ± 0.022	certified	SIM-QM-CA-000001EJ-1
Methylmercury (as Hg) (e,f)	0.137 ± 0.012	certified	SIM-QM-CA-000001EK-1
molybdenum (a,d)	3.44 ± 0.12	certified	SIM-QM-CA-000001EL-1
nickel (a,d)	5.30 ± 0.24	certified	SIM-QM-CA-000001EM-1
selenium (a,d)	10.9 ± 1.0	certified	SIM-QM-CA-000001EO-1

Analyte	Mass fraction, mg/kg	Type of value	International recognition of measurement capability (CMC)
strontium (b,d)	36.5 ± 1.6	certified	SIM-QM-CA-000001ER-1
tin (a,d)	0.029	information	SIM-QM-CA-00000NR9-1
vanadium (a,d)	9.1 ± 0.4	certified	SIM-QM-CA-000001ES-1
zinc (b,d)	136 ± 6	certified	SIM-QM-CA-000001ET-1

*inorganic arsenic is the sum of As(III) and As(V)

Coding

The coding refers to the instrumental method of analyte determination.

- a - Inductively coupled plasma mass spectrometry (ICP-MS)
- b - Inductively coupled plasma atomic emission spectrometry (ICP-AES)
- c - Isotope dilution liquid chromatography orbitrap mass spectrometry (ID-LC-MS)
- d - Isotope dilution inductively coupled plasma mass spectrometry (ID-ICP-MS)
- e - Gas chromatography inductively coupled plasma mass spectrometry (GC-ICP-MS)
- f - Gas chromatography mass spectrometry (GC-MS)
- g - Standard addition liquid chromatography ICP-MS (SA-LC-ICP-MS) [2]
- h - Liquid chromatography ICP-MS (LC-ICP-MS) [2]

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.

Information values

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

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 a food matrix can be found in the BIPM database at <https://www.bipm.org/kcdb/>.

Intended use

This reference material is primarily intended for use in the calibration of procedures and the development of methods for the determination of trace metals in marine fauna and materials of similar matrix.

Storage and sampling

This material should be stored in a cool and dark location. Prior to use, the bottle should be rotated and shaken to ensure the contents are well mixed. The bottle should be tightly closed thereafter. Certified values are based on a minimum 0.200 g sub-sample from the bottle.

Instructions for drying

Determination of dry mass should be performed on a separate sample to avoid contamination. TORT-3 can be dried to constant mass by: (1) drying at reduced pressure (i.e., 50 mm Hg) at room temperature in a vacuum desiccator over magnesium perchlorate for 24 hours; (2) vacuum drying (about 0.5 mm Hg) at room temperature for 24 hours.

Preparation of material

This reference material was prepared from edible grade lobster tomalley. The material was homogenized, spray dried, defatted with hexane and vacuum dried. The resulting powder was bottled at the NRC. After bottling the material was sterilized by subjecting it to a minimum dose of 25 kGy gamma irradiation.

Stability

The predecessor CRM, TORT-2, has been periodically analyzed for more than ten years and found to be both physically and chemically stable over this time interval. We expect similar results for TORT-3. Uncertainty components for long and short term stability were considered negligible and are thus not included in the uncertainty budget.

The methylmercury content of TORT-3 is continually monitored by NRC. Studies indicate that methylmercury content in similar materials has been stable for more than ten years. This is not expected to change, provided the material is stored as noted above.

Homogeneity

The material was tested for homogeneity at NRC. Results from sub-samples (0.200 g) from thirty bottles were evaluated using ANOVA [3] and included in the calculation of the certified values.

Uncertainty

Included in the overall uncertainty estimate (u_c) are uncertainties in the batch characterization (u_{char}) and uncertainties related to possible between-bottle variation (u_{hom}). Expressed as standard uncertainties these components are listed in Table 2.

Table 2: Uncertainty components for TORT-3

Analyte	u_c , mg/kg	u_{char} , mg/kg	u_{hom} , mg/kg
As	1.9	1.7	0.8
AsBet(As)	1.23	0.96	0.77
Cd	0.9	0.5	0.8
Cr	0.12	0.06	0.10
Cu	11	9	6
Fe	4	4	3
Pb	0.009	0.004	0.009

Analyte	u_c , mg/kg	u_{char} , mg/kg	u_{hom} , mg/kg
Mn	0.5	0.5	0.2
Hg	0.011	0.005	0.010
MeHg(Hg)	0.006	0.004	0.005
Mo	0.06	0.04	0.05
Ni	0.12	0.10	0.06
Se	0.5	0.4	0.3
Sr	0.8	0.6	0.5
V	0.2	0.2	0.2
Zn	3	2	2

Metrological traceability

Results presented in this certificate are traceable to the SI through CRMs produced by National Metrology Institutes and gravimetrically prepared standards of established purity. As such, TORT-3 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 (bipm.org/kcdb), 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.2013.tort-3.

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. Gajdosechova Z, Grinberg P, Kubachka et al. (2023) Determination of inorganic As, DMA and MMA in marine and terrestrial tissue samples: a consensus extraction approach. *Environmental Chemistry*. 20: 5-17. doi.org/10.1071/EN23006
3. van der Veen AMH. Bayesian analysis of homogeneity studies in the production of reference materials. *Accred Qual Assur.* (2017), 22 (6): 307-319. doi.org/10.1007/s00769-017-1292-6

Cited by

A list of scientific publications citing TORT-3 can be found at doi.org/10.4224/crm.2013.tort-3.

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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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