

# Certificate of Analysis

**NRC-CNRC**

Certified Reference Material

## LUTS-1

Non Defatted Lobster Hepatopancreas Certified Reference Material for Trace Metals

The following table shows those constituents for which certified values have been established for LUTS-1.

Certified values are based on the results of determinations by at least two independent methods of analysis. The expanded uncertainty ( $U_{\text{CRM}}$ ) 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. It is intended that  $U_{\text{CRM}}$  accounts for every aspect that reasonably contributes to the uncertainty of the measurement. A coverage factor of  $k = 2$  was applied for all elements.

**Table 1: Certified quantity values for LUTS-1**

Element	As bottled, mg/kg	Dry-weight corrected, mg/kg
arsenic (d,g,h,n,s)	$2.83 \pm 0.13$	$19.0 \pm 0.9$
cadmium (g,i,p)	$2.12 \pm 0.15$	$14.2 \pm 1.0$
calcium (f,i,n)	$203 \pm 33$	$1360 \pm 220$
chromium (c,g)	$0.079 \pm 0.012$	$0.53 \pm 0.08$
cobalt (d,g,n)	$0.051 \pm 0.006$	$0.34 \pm 0.04$
copper (f,g,i,n,p)	$15.9 \pm 1.2$	$107 \pm 8$
iron (d,f,g,i,n)	$11.6 \pm 0.9$	$77.8 \pm 6.0$
lead (g,p,t)	$0.010 \pm 0.002$	$0.069 \pm 0.011$
magnesium (f,i,n)	$89.5 \pm 4.1$	$601 \pm 28$
manganese (d,f,g,i,n)	$1.20 \pm 0.13$	$8.02 \pm 0.86$
nickel (d,g,k,p)	$0.200 \pm 0.034$	$1.34 \pm 0.23$
potassium (e,f,n)	$948 \pm 72$	$6360 \pm 480$
selenium (c,g,h)	$0.641 \pm 0.054$	$4.30 \pm 0.36$
silver (g,i,n,p)	$0.580 \pm 0.049$	$3.89 \pm 0.33$
strontium (f,i,p)	$2.46 \pm 0.28$	$16.5 \pm 1.9$
zinc (f,i,n,p)	$12.4 \pm 0.8$	$82.9 \pm 5.4$



## Coding

The coding refers only to the ultimate method of analyte determination. No mention is made here regarding the various methods of sample preparation, decomposition and possible analyte separation prior to determination within each coded method.

- c** Isotope dilution gas chromatography/mass spectrometry
- d** Inductively coupled plasma mass spectrometry
- e** Flame atomic emission spectrometry
- f** Flame atomic absorption spectrometry
- g** Graphite furnace atomic absorption spectrometry (GFAAS)
- h** Hydride generation atomic absorption spectrometry
- i** Inductively coupled plasma atomic emission spectrometry
- k** Adsorptive accumulation voltammetry
- n** Instrumental neutron activation
- p** Isotope dilution inductively coupled plasma mass spectrometry
- s** Hydride generation/in-situ concentration/GFAAS determination
- t** Ethyllead generation/in-situ concentration/ GFAAS determination

## Certified values

Certified values are considered to be those for which 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 uncertainty and mean (Table 1).

## Intended use

This reference material is primarily intended for use in the calibration of procedures and the development of methods used for the analysis of biological materials, especially those with a high lipid content.

## Storage and sampling

It is recommended that the material be stored in a cool, clean location. The bottles should be opened only in a clean area with precautions taken against contamination during sampling.

Each plastic bottle of LUTS-1 contains  $10.30 \pm 0.05$  g of material. The moisture content of the bottled material is  $0.8510 \pm 0.0007$  g/g. The rigid control afforded to the mass of material per bottle and the moisture content enables the analyst to quantitatively sample directly or prepare a slurry from any bottle and immediately utilize it for reference purposes without the need to establish a dry weight on the sample.

LUTS-1 can be dried to constant mass by:

1. Heating at 105 °C for 2 hours.
2. Vacuum drying (about 0.5 mm Hg) at room temperature for 24 h.

Both of these methods were used to obtain a conversion factor to produce the “dry weight” results listed on the first page.

Two methods of sampling are recommended. The first is preferred in this laboratory because of its simplicity:



- 1) The bottle is thoroughly shaken or sonicated and a pipetted sample is delivered to a tared vessel and weighed. One millilitre of sample weighs approximately 0.9 g.
- 2) The contents of a bottle are transferred to a volumetric flask with water. Aliquots for analysis may be taken after thorough shaking or sonication. Acids must not be used for the dilution or the fats will hydrolyse and precipitate in the flask. If solids settle in the flask they can be easily resuspended by shaking.

### **Preparation of materials**

LUTS-1 is a second generation reference material prepared from edible grade lobster tomalley. The processing was performed at the Canadian Institute for Fisheries Technology, Technical University of Nova Scotia, Halifax, Nova Scotia. The material has not been defatted or dried during its preparation like the lobster hepatopancreas reference material TORT-2. Except for the addition of some water and a small quantity of antioxidant, the sample is a natural biological material containing 55 percent lipids on a dry weight basis. For a full description of the preparation process see reference 1.

### **Stability**

No loss of integrity of this material has been discerned during monitoring. This certificate is valid provided the material is handled and stored in accordance with the instructions given herein.

### **Homogeneity**

The materials were tested for homogeneity at the National Research Council (NRC) in Ottawa. Also, randomly selected bottles were used for the analytical determinations by NRC and collaborating laboratories. Results from different bottles indicated no significant differences compared to results from sub-samples within bottles. It is assumed, then, that all bottles of these materials have essentially the same composition.

### **Metrological Traceability**

Results presented 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.

### **Quality management (ISO/IEC 17025, ISO Guide 34)**

This material was produced in compliance with the documented NRC MSS Quality System, which conforms to the requirements of ISO/IEC 17025 and ISO Guide 34.

The 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](http://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 certificate of approval is available upon request.



## Updates

Users should ensure that the certificate they have is current. Our web site at <http://www.nrc.ca/crm> will contain any new information.

## References

1. Evaluation of measurement data: Guide to the expression of uncertainty in measurement JCGM100:2008
2. S.S. Berman and R.E. Sturgeon, Fresenius Z. Anal. Chem. 332, 546-548 (1988).

## Authorship

The following staff members of the Measurement Science and Standards portfolio at the NRC contributed to the production and certification of LUTS-1: S.S. Berman, V.J. Boyko, V.P. Clancy, J. Lam, P. Maxwell, J.W. McLaren, M. Miedema, K.W.M. Siu, R.E. Sturgeon and S.N. Willie.

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**LUTS-1**

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**Approved by:**



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**This Certificate is only valid if the corresponding product was obtained directly from NRC or one of our qualified vendors.**

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