



LUTS-1

Non Defatted Lobster Hepatopancreas Reference Material for Trace Metals

The following table shows the eighteen metals 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 uncertainties represent 95 percent confidence limits for an individual subsample. That is, 95 percent of samples from any bottle would be expected to have concentrations within the specified range 95 percent of the time.

	Certified Values (milligrams/kilogram)			
	As Bottled		Dry Weight	
Arsenic (d,g,h,n,s)*	2.83	± 0.13	19.0	± 0.9
Cadmium (g,i,p)	2.12	± 0.15	14.2	± 1.0
Calcium (f,i,n)	203	± 33	1360	± 220
Chromium (c,g)	0.079	± 0.012	0.53	± 0.08
Cobalt (d,g,n)	0.051	± 0.006	0.34	± 0.04
Copper (f,g,i,n,p)	15.9	± 1.2	107	± 8
Iron (d,f,g,i,n)	11.6	± 0.9	77.8	± 6.0
Lead (g,p,t)	0.010	± 0.002	0.069	± 0.011
Magnesium (f,i,n)	89.5	± 4.1	601	± 28
Manganese (d,f,g,i,n)	1.20	± 0.13	8.02	± 0.86
Nickel (d,g,k,p)	0.200	± 0.034	1.34	± 0.23
Potassium (e,f,n)	948	± 72	6360	± 480
Selenium (c,g,h)	0.641	± 0.054	4.30	± 0.36
Silver (g,i,n,p)	0.580	± 0.049	3.89	± 0.33
Strontium (f,i,p)	2.46	± 0.28	16.5	± 1.9
Zinc (f,i,n,p)	12.4	± 0.8	82.9	± 5.4

* see next page for key to coding

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.

- a - Anion exchange/cold vapour atomic absorption spectrometry
- 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
- l - 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

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.

The metal concentrations of unopened bottles are warranted for a two year period from the shipping date. Studies have shown that the material is stable with respect to total trace metal concentrations for at least five years.

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 ± 0.05 g of reference material. The moisture content of the bottled material is 85.10 ± 0.07 percent. 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 weight by:

- (1) heating at 105°C for 2 hours.
- (2) vacuum drying (about 0.5 mm Hg) at room temperature for 24 hours.

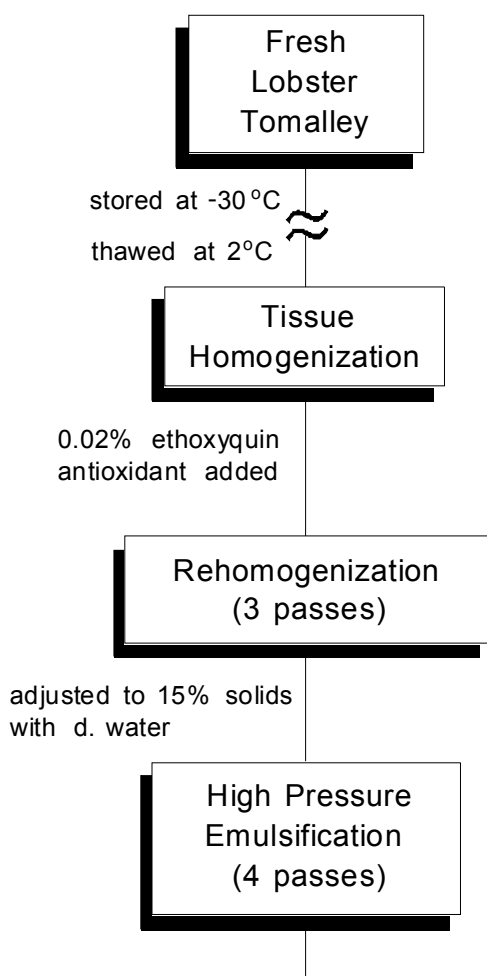
Both of these methods were used to obtain a conversion factor to produce the "dry weight" results listed on the first page.

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 ml of sample weighs approximately 0.9g.
- (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.

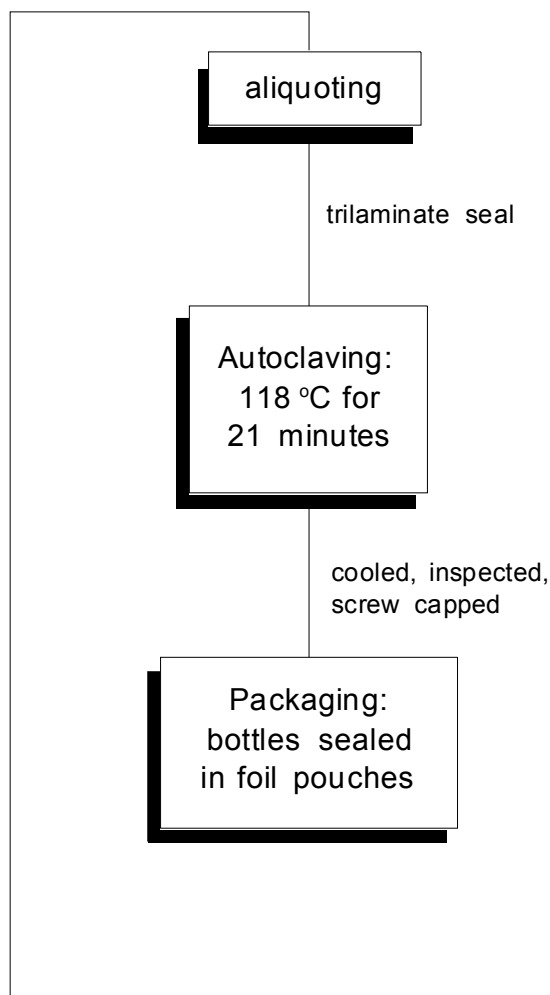
Preparation of Materials

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. The preparation scheme is described below in the schematic drawing. For a full description of the preparation process see S.S. Berman and R.E. Sturgeon, *Fresenius Z. Anal. Chem.* 332, 546-548 (1988).



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.



Certification

The majority of the certification work was done within the Chemical Metrology Group of the Institute for National Measurement Standards, National Research Council of Canada. Five external expert laboratories cooperated in the certification process.

Acknowledgements

This material was prepared following the advice of the NRC Committee on Marine Analytical Chemistry. The guidance of the members of the committee is much appreciated.

These members of staff of the Institute for National Measurement Standards, National Research Council of Canada, participated in the analyses: 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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Stability

No loss of integrity of this material has been discerned during a 12 year monitoring period. If substantive changes occur that affect the certified values, NRC will notify all users. This certificate is valid provided the material is handled and stored in accordance with the instructions given herein.

Updates

It is anticipated that as more data become available the established values may be updated and reliable values assigned to more elements. Updates will be posted on our web site at http://inms-ienm.nrc-cnrc.gc.ca/calserv/chemical_metrology_e.html.

Comments from users will be welcomed.

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revised August 1995

revised January 2005, *Hg and MeHg removed*

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. This CRM is registered at the Bureau International des Poids et Mesures (BIPM) in Appendix C of the Comité International des Poids et Mesures database listing Calibration and Measurement Capabilities accepted by signatories to the Mutual Recognition Arrangement of the Metre Convention.

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