

ANEXOS

Determinación de plata en muestras biológicas
mediante técnicas de espectrometría atómica
(ETAAS).

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Trabajo Fin de Grado

Anexo I

DOLT-4

Dogfish liver certified reference material for trace metals

Elements for which certified values have been established for this dogfish (*Squalus acanthias*) liver CRM, along with their expanded uncertainty ($UCRM = kuc$, where uc is the combined standard uncertainty calculated according to the JCGM Guide [1] and k is the coverage factor) are listed below. It is intended that $UCRM$ encompasses every aspect that reasonably contributes to the uncertainty of the certified mass fraction [2]. Values are based on dry mass.

Table 1. Certified quantity values for DOLT-4

Element	Mass fraction, (mg/kg)
Arsenic (d,e,h)	9.66 \pm 0.62
Cadmium (d,e,i,p)	24.3 \pm 0.8
Copper (d,e,l,p)	31.2 \pm 1.1
Iron (d,i)	1833 \pm 75
Lead (d,e,p)	0.16 \pm 0.04
Mercury (c,d,p)	2.58 \pm 0.22
Nickel (d,e,l,p)	0.97 \pm 0.11
Selenium (e,h)	8.3 \pm 1.34
Silver (d,e,p)	0.93 \pm 0.07
Zinc (d,l,p)	116 \pm 6
Methylmercury (as Hg) (g,s,t)	1.33 \pm 0.12

Coding

The coding refers only to the instrumental method of analyte determination.

c - Cold vapour atomic absorption spectrometry.

d - Inductively coupled plasma mass spectrometry.

e - Electrothermal vaporization atomic absorption spectrometry (ETAAS).

g - Solid phase microextraction (SPME) isotope dilution gas chromatography mass spectrometry.

h - Hydride generation atomic absorption spectrometry

i - Inductively coupled plasma atomic emission spectrometry.

p - Isotope dilution inductively coupled plasma mass spectrometry (ID-ICPMS).

s - SPME isotope dilution gas chromatography ICPMS.

t - Ethylation cold vapor atomic fluorescence spectrometry.

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 0.250 g sub-sample from the bottle.

Instructions for drying

Determination of dry mass should be performed on a separate sample to avoid contamination. DOLT-4 can be dried to constant mass by: (1) drying at reduced pressure (e.g., 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.

Information values

The following table presents information values for elements which could not be certified because of insufficient information to accurately assess uncertainties.

Table 2: Information values for DOLT-4

Element	Mass fraction, (mg/kg)
Na	6800
Mg	1500
Al	200
K	9800
Ca	680
V	0.6
Cr	1.4
Co	0.25
Sr	5.5
Mo	1
Sn	0.17

Preparation of DOLT-4

This reference material was processed at the Guelph Food Technology Center, Guelph Ontario. The preparation sequence is illustrated on the following page. The final product was sterilized by gamma irradiation (minimum dose of 25 kGy) at the Canadian Irradiation Centre, Laval, Quebec.

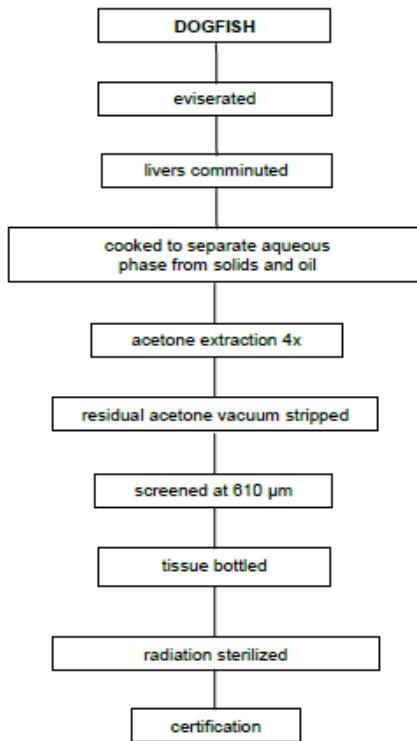


Figure 1: Preparation steps for DOLT-4

Certified values

DOLT-4 was provided as an unknown sample to a group of laboratories participating in an annual intercomparison for trace metals in marine samples coordinated by NRC [3]. Data generated by NRC were also included in the pool of intercomparison results. Laboratories were requested to provide triplicate results using an analytical method of choice based on total digestion of the sample. DOLT-3 was provided as a quality control sample.

Data were returned to NRC for evaluation. Results from a select sub-group of participants were used for the certification of DOLT-4. Such laboratories were selected based on their performance history in previous intercomparisons.

Included in the overall uncertainty estimate are uncertainties in the batch characterization (u_{char}) and uncertainties related to possible between-bottle variation (u_{hom}). Expressed as standard uncertainties these components can be combined as:

$$\text{Equation 1: } u_c(\text{CRM}) = u_{\text{char}} + u_{\text{hom}} + u_{\text{stab}}$$

Based on NRC's experience with similar materials, uncertainty components for long and short term stability were considered negligible and are thus not included in the uncertainty budget.

Expiration of certificate

A predecessor CRM, DOLT-2, has been periodically analyzed for more than nine years and found to be both physically and chemically stable over this time interval. We expect similar characteristics from DOLT-4. The certified values for DOLT-4 are considered valid until April 2014, provided the CRM is handled and stored in accordance with instructions herein.

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. 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.

Accreditation

The Chemical Metrology laboratory is compliant to ISO 17025 and ISO Guide 34, with approval by The Inter-American Metrology System (SIM). The 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-cnrc.gc.ca> will contain any new information.

References

1. Evaluation of measurement data – Guide to the expression of uncertainty in measurement
JCGM 100:2008.
1. ISO Guide 35:2006, Reference materials — General and statistical principles for certification, Geneva, Switzerland (2006)
2. S. Willie, Twentieth Intercomparison for Trace Elements in Marine Sediments and Biological Tissues, NRC No. 50099, October 2007.

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The following laboratories participated in the certification of DOLT-4:

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Australian Nuclear Science and Technology, Organization, Environmental Science Program
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Battelle Pacific Northwest, Sequim Bay Road, Sequim, WA

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San Francisco Public Utilities Commission, Southeast Laboratory, San Francisco, CA,

Texas A. & M. College of Veterinary Medicine, Trace Element Research Laboratory, College
Station, TX

Texas Parks and Wildlife, Environmental Contaminants Laboratory, San Marcos, TX U.S. Customs Laboratory,
Savannah, GA

USGS-WRD, 3039 Amwiler Road, Atlanta, GA

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