Certified Reference Material

DOLT-5

Dogfish Liver Certified Reference Material for Trace Metals and other Constituents

The following tables show those constituents for which certified, reference, and information values have been established for this dogfish (*Squalus acanthias*) liver CRM, along with their expanded uncertainty.

The expanded uncertainty (U_{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 $k = 2$ is the coverage factor. It is intended that U_{CRM} accounts for every aspect that reasonably contributes to the uncertainty of the measurement. All listed values are expressed on a dry mass basis.

Table 1: Certified Quantity Values

Element	Mass fraction, mg/kg
Arsenic (b,d)	34.6 ± 2.4
Cadmium (a,d)	14.5 ± 0.6
Calcium (c)	550 ± 80
Cobalt (b,d)	0.267 ± 0.026
Copper (a,c,d)	35.0 ± 2.4
Iron (a,c,d)	1070 ± 80
Lead (a,d)	0.162 ± 0.032
Magnesium (c)	940 ± 100
Mercury (a,e)	0.44 ± 0.18
Molybdenum (a,d)	1.41 ± 0.22
Potassium (c)	14 400 ± 3000
Selenium (a,d)	8.3 ± 1.8
Silver (a,d)	2.05 ± 0.08
Sodium (c)	9 900 ± 1600
Strontium (a,c,d)	3.73 ± 0.26
Tin (a,d)	0.069 ± 0.036
Vanadium (b,d)	0.51 ± 0.06
Zinc (a,c,d)	105.3 ± 5.4

Table 2: Reference Values

Element	Mass fraction, mg/kg
Aluminium (c,d)	31.7 ± 4.2
Nickel (a,d)	1.71 ± 0.56
Chromium (a,d)	2.35 ± 0.58
Methylmercury (as Hg) (f)	0.119 ± 0.058
Manganese (d)	8.91 ± 0.70

Table 3: Information Values

Element	Mass fraction, mg/kg
Antimony (d)	0.013
Phosphorus (d)	11 500
Thallium (d)	0.082
Uranium (d)	0.013

Coding

The coding refers to the instrumental method of analyte determination.

a - Isotope dilution inductively-coupled plasma mass spectrometry (ID-ICP-MS)
b - Standard addition inductively-coupled plasma mass spectrometry (ICP-MS)
c - Inductively-coupled plasma atomic emission spectroscopy (ICP-AES)
d - Inductively-coupled plasma mass spectrometry (ICP-MS)
e - Cold-vapour atomic emission spectroscopy (CV-AAS)
f - Isotope dilution gas chromatography ICP-MS (ID-GC-ICP-MS)

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 (Table 1).

Reference Values

Reference values are those for which insufficient data are available to provide a comprehensive estimate of uncertainty (Table 2).

Information Values

Information values are those for which insufficient data are available to provide any estimate of uncertainty (Table 3).

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 and matrix constituents in marine sediments and materials with similar matrices. A minimum sample mass of 250 mg is recommended.

Storage and Sampling

It is recommended that the material be stored in a cool, clean location. Each bottle is packaged in a trilaminate foil pouch which serves as an impermeable barrier to mercury vapour. Under conditions of high ambient levels of mercury vapour, mercury is able to penetrate the plastic cap of the bottle, thereby potentially contaminating the contents. The bottle contents should be well mixed by rotation and shaking prior to use, and tightly closed immediately thereafter. Certified values are based on a minimum 250 mg sub-sample from the bottle.

Instructions for Drying

Although initially free from moisture following the freeze drying, the materials have adsorbed moisture during subsequent operations. They should be dried to a constant mass before use. Drying for several hours at 105 ^{o}C is recommended as a relatively simple method to achieve a dry mass for most purposes. The moisture content is estimated at 0.043 g/g.

Preparation of Material

Frozen dogfish liver was sourced and prepared by Guelph Food Technology Center (Guelph, ON, Canada) where the liver was comminuted (50 µm), blended, partially defatted at 40 ^{o}C, and freeze-dried. The dried dogfish liver meal was then defatted by POS Bio-Sciences (Saskatoon, SK, Canada) using hexane to produce free-flowing powder. The defatted samples were bottled at NRC and radiation sterilized with a minimum dose of 25 kGy by Nordion Gamma Centre of Excellence (Laval, QC, Canada) to minimize any effects from biological activity.

Stability

The predecessor CRM, DOLT-4, 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 DOLT-5. Uncertainty components for long and short term stability were considered negligible and are thus not included in the uncertainty budget.

Homogeneity

The material was tested for homogeneity at NRC. Results from sub-samples (250 mg) were evaluated using the DerSimonian-Laird random effects model and included in the calculation of the certified values [2].

Uncertainty

Included in the overall combined uncertainty estimate (u_c) are uncertainties in the batch

characterization (u_{char}), uncertainties related to possible between-bottle variation (u_{hom}), and uncertainties related to inconsistency between the various measurement methods (u_{method}). Expressed as standard uncertainties, these components are listed in Table 4.

Table 4: Uncertainty Components for DOLT-5

Element	u_c, mg/kg	u_{char}, mg/kg	u_{hom}, mg/kg	u_{method}, mg/kg
Arsenic	1.2	0.8	0.9	0.0
Cadmium	0.3	0.2	0.2	0.0
Calcium	40	30	20	0
Cobalt	0.013	0.009	0.010	0.000
Copper	1.2	0.6	1.0	0.0
Iron	40	20	30	0
Lead	0.016	0.007	0.014	0.000
Magnesium	50	40	30	0
Mercury	0.09	0.02	0.05	0.07
Molybdenum	0.11	0.11	0.03	0.00
Potassium	1500	1400	400	0
Selenium	0.9	0.3	0.8	0.0
Silver	0.04	0.03	0.03	0.00
Sodium	800	700	300	0
Strontium	0.13	0.09	0.09	0.00
Tin	0.018	0.003	0.017	0.004
Vanadium	0.03	0.02	0.02	0.00
Zinc	2.7	1.9	1.9	0.0

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 with 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 (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 website at www.nrc.gc.ca/crm will contain any new information.

References

1. Evaluation of measurement data – Guide to the expression of uncertainty in measurement JCGM 100:2008
2. Rebecca DerSimonian, Nan Laird (1986) Meta-analysis in clinical trials. Controlled Clinical Trials 7: 177-188

Authorship

The following staff members of the Chemical Metrology Group at National Research Council Canada contributed to the production and certification of DOLT-5: L. Yang, S. Willie, P. Grinberg, I.P. Gedara, V. Clancy, P. Maxwell, J. Meija, and Z. Mester.

DOLT-5
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