National Research Council Canada | Conseil national de recherches Canada

Certificate of Analysis

Certified Reference Material

DOLT-5

Dogfish liver Certified Reference Material for trace metals and other Constituents

DOLT-5 is a dogfish (*Squalus acanthias*) liver Certified Reference Material (CRM) from the National Research Council of Canada with information on trace element and species content. A unit of DOLT-5 consists of approximately 20 grams of dogfish liver in an amber glass vial.

Table 1 shows the certified, reference and information values established for DOLT-5. The expanded uncertainties associated with the certified and reference 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 uncertainties *(k = 2)* for DOLT-5**

Analyte	Mass fraction, mg/kg	Type of Value	International recognition of measurement capability (CMC)
aluminium (c,d)	31.7 ± 4.2	reference	MEF-13
antimony (d)	0.013	information	--
arsenic (b,d)	34.6 ± 2.4	certified	MEF-14
arsenobetaine (as As) (g,i)	24.2 ± 0.8	certified	MEF-15
inorganic arsenic (as As) (g,h)*	0.034 ± 0.002	certified	--
dimethylarsinic acid (as As) (g,h)	3.09 ± 0.188	reference	--
monomethylarsonic acid (as As) (g,h)	0.201 ± 0.024	reference	--
cadmium (a,d)	14.5 ± 0.6	certified	MEF-16
calcium (c)	550 ± 80	certified	MEF-17
chromium (a,d)	2.35 ± 0.58	reference	MEF-18
cobalt (b,d)	0.267 ± 0.026	certified	MEF-19
copper (a,c,d)	35.0 ± 2.4	certified	MEF-20
iron (a,c,d)	1070 ± 80	certified	MEF-21
lead (a,d)	0.162 ± 0.032	certified	MEF-22
magnesium (c)	940 ± 100	certified	MEF-23
manganese (d)	8.91 ± 0.70	reference	MEF-24
mercury (a,e)	0.44 ± 0.18	certified	MEF-25
methylmercury (as Hg) (f)	0.119 ± 0.058	reference	MEF-26

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Analyte	Mass fraction, mg/kg	Type of Value	International recognition of measurement capability (CMC)
molybdenum (a,d)	1.41 ± 0.22	certified	MEF-27
nickel (a,d)	1.71 ± 0.56	reference	MEF-28
phosphorus (d)	11 500	information	--
potassium (c)	14 400 ± 3000	certified	MEF-29
selenium (a,d)	8.3 ± 1.8	certified	MEF-30
silver (a,d)	2.05 ± 0.08	certified	MEF-31
sodium (c)	9 900 ± 1600	certified	MEF-32
strontium (a,c,d)	3.73 ± 0.26	certified	MEF-33
thallium (d)	0.013	information	--
tin (a,d)	0.069 ± 0.036	certified	--
uranium (d)	0.082	information	--
vanadium (b,d)	0.51 ± 0.06	certified	MEF-34
zinc (a,c,d)	105.3 ± 5.4	certified	MEF-35

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

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)
- **g** Standard addition liquid chromatography ICP-MS (SA-LC-ICP-MS) [2]
- **h** Liquid chromatography ICP-MS (LC-ICP-MS) [2]
- **i** Isotope dilution liquid chromatography Orbitrap mass spectrometry

Certified values

Certified values are considered to be those for which the National Research Council of Canada (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 mean and uncertainty.

Reference values

Reference values are non-certified values for which insufficient data are available to provide a comprehensive estimate of uncertainty to permit their full certification.

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 and matrix constituents in marine fauna 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 °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 °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 [3].

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 2.

Table 2: Uncertainty components for DOLT-5

Element/compound	u_c, mg/kg	u_{char}, mg/kg	u_{hom}, mg/kg	u_{method}, mg/kg
aluminium	2.1	1.0	1.8	0.0
arsenic	1.2	0.8	0.9	0.0
arsenobetaine (as As)	0.4	0.3	0.2	0.0
cadmium	0.3	0.2	0.2	0.0
calcium	40	30	20	0
chromium	0.29	0.15	0.25	0.00
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
manganese	0.35	0.35	0.00	0.00
mercury	0.09	0.02	0.05	0.07
methylmercury (as Hg)	0.029	0.011	0.009	0.025
molybdenum	0.11	0.11	0.03	0.00
nickel	0.28	0.12	0.25	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 International System of Units (SI) through gravimetrically prepared standards of established purity and CRMs produced by National Metrology Institutes. As such, DOLT-5 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 (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 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.2014.dolt-5.

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. R. DerSimonian, N. Laird (1986) Meta-analysis in clinical trials. Controlled Clinical Trials 7: 177-188. doi.org/10.1016/0197-2456(86)90046-2

Cited by

A list of scientific publications citing DOLT-5 can be found at doi.org/10.4224/crm.2014.dolt-5

Authorship

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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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