



Certificate of Analysis

Certified Reference Material

DORM-5

Fish Protein Certified Reference Material

DORM-5 is a fish protein Certified Reference Material (CRM) from the National Research Council of Canada (NRC) with information on total trace element and species content. A unit of DORM-5 consists of approximately 18 grams of fish protein in an amber glass vial.

Table 1 shows the certified, reference and information values established for DORM-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 uncertainty ($k = 2$) for DORM-5

Analyte	Mass fraction, mg/kg	Type of value	International recognition of measurement capability (CMC)
aluminium (b,c,d,e)	250 ± 13	certified	TEB-01
antimony (a,c)	0.0062 ± 0.0024	reference	--
arsenic (b,c,d,e)	13.3 ± 0.7	certified	MEF-14
arsenobetaine (as As) (f,h)	11.8 ± 0.4	certified	MEF-15
inorganic arsenic (as As) (i,j)*	0.016 ± 0.001	certified	--
dimethylarsinic acid (as As) (i,j)	0.288 ± 0.027	reference	--
monomethylarsonic acid (as As) (i,j)	0.013 ± 0.001	reference	--
barium (a,c,d,e)	0.396 ± 0.023	certified	MEF-4
boron (a,c)	3.63 ± 0.16	certified	--
bromine (c)	50.7	information	--
cadmium (a,c)	0.148 ± 0.007	certified	MEF-16
calcium (b,c,d,e)	2010 ± 260	certified	MEF-17
chlorine (c)	12200	information	--
chromium (a,c,d,e)	0.515 ± 0.068	certified	MEF-18
cobalt (b,c)	0.063 ± 0.004	certified	MEF-19
copper (a,c,d,e)	3.30 ± 0.07	certified	MEF-20
iodine (c)	7.5 ± 1.4	reference	--
iron (a,c,d,e)	113 ± 8	certified	MEF-21
lead (a,c)	0.058 ± 0.006	certified	MEF-22
lithium (a,c)	0.391 ± 0.120	certified	--

Analyte	Mass fraction, mg/kg	Type of value	International recognition of measurement capability (CMC)
magnesium (b,c,d,e)	1030 ± 80	certified	MEF-23
manganese (b,c,d,e)	1.06 ± 0.04	certified	MEF-24
mercury (a,c)	0.316 ± 0.017	certified	MEF-25
molybdenum (a,c)	0.134 ± 0.023	certified	MEF-27
nickel (a,c,d,e)	0.44 ± 0.03	certified	MEF-28
phosphorus (b,c,d,e)	6230 ± 240	certified	--
potassium (b,c,d,e)	11600 ± 400	certified	MEF-29
rubidium (b)	2.76 ± 0.19	certified	--
selenium (a,c,d,e)	2.40 ± 0.11	certified	MEF-30
selenomethionine (as Se) (f)	0.62 ± 0.14	certified	--
silver (a,c)	0.135 ± 0.014	certified	MEF-31
sodium (b,c,d,e)	9200 ± 400	certified	MEF-32
strontium (a,c,d,e)	9.87 ± 0.23	certified	MEF-33
sulfur (b,d,e)	8400 ± 200	reference	--
tin (a,c)	0.077 ± 0.008	certified	--
uranium (a,c)	0.0163 ± 0.0039	certified	--
vanadium (b,c,d,e)	0.347 ± 0.029	certified	MEF-34
zinc (a,c,d,e)	28.7 ± 1.0	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 (SA-ICP-MS)
- c** Inductively-coupled plasma mass spectrometry (ICP-MS)
- d** Standard addition Inductively-coupled plasma atomic emission spectroscopy (SA-ICP-AES)
- e** Inductively-coupled plasma atomic emission spectroscopy (ICP-AES)
- f** Isotope dilution liquid chromatography ICP-MS (ID-LC-ICP-MS)
- g** Isotope dilution liquid chromatography mass spectrometry (ID-LC-MS)
- h** Standard addition liquid chromatography mass spectrometry (SA-LC-MS)
- i** Standard addition liquid chromatography ICP-MS (SA-LC-ICP-MS) [2]
- j** Liquid chromatography ICP-MS (LC-ICP-MS) [2]

Supplementary data

The accompanying datasheets (available from doi.org/10.4224/crm.2021.dorm-5) provide data from individual laboratories.

Certified values

Certified values are considered to be those for which the 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 true value and uncertainty.

Reference values

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

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

DORM-5 is intended for use in the method development, validation, and quality control for the analysis of trace and matrix constituents in marine fauna and similar sample matrices.

Storage and sampling

It is recommended that the material is stored at approximately $-20\text{ }^{\circ}\text{C}$ or below under typical freezer conditions. Each vial is packaged in a trilaminate foil pouch. Prior to use, the vial should be allowed to warm to room temperature and the contents should be well mixed, and tightly closed immediately thereafter. Certified values are based on a minimum 250 mg sub-sample.

Instructions for drying

To obtain dry weight a sample aliquot should be dried to a constant mass. The estimated moisture content of DORM-5 is approximately 0.04 g/g.

Preparation of material

This reference material was prepared from a commercial fish protein homogenate. The material was produced using an enzymatic hydrolysis procedure subsequent to removal of the bones and the majority of the oil. The protein hydrolysate was flash-pasteurized, spray dried, sieved to pass an 850 μm nylon screen, blended and bottled in amber glass vials. After bottling the material was sterilized by subjecting it to a minimum dose of 25 kGy gamma irradiation.

Stability

DORM-5 stability was assessed and deemed to be both physically and chemically stable in long term storage and transportation. Long term stability was assessed by proxy using similar CRMs and transportation stability study was carried out using an isochronous approach under elevated temperature.

Homogeneity

Homogeneity of the material using 250 mg sub-samples was assessed. Results from randomly selected bottles were evaluated using Bayesian analysis of variance (ANOVA) [3].

Uncertainty

Evaluation of the uncertainty associated with certified and reference values was carried out. Included in the overall combined uncertainty estimate are uncertainties in the batch characterization, uncertainties related to possible between-bottle variation, and uncertainties related to inconsistency between the various measurement methods [4,5]. Further information is presented in the supplementary datasheets doi.org/10.4224/crm.2021.dorm-5.

Metrological traceability

Results presented in this certificate are traceable to the SI through CRMs produced by National Metrology Institutes and gravimetrically prepared standards of established purity. As such, DORM-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.2021.dorm-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. van der Veen AMH (2017) Bayesian analysis of homogeneity studies in the production of reference materials. *Accred. Qual. Assur.* 22: 307-319. doi.org/10.1007/s00769-017-1292-6
4. Possolo A, Toman B (2007) Assessment of measurement uncertainty via observation equations. *Metrologia*, 44: 464-475. doi.org/10.1088/0026-1394/44/6/005
5. Thompson M, Ellison SLR (2011) Dark uncertainty. *Accred. Qual. Assur.* 16: 483-487. doi.org/10.1007/s00769-011-0803-0

Cited by

A list of scientific publications citing DORM-5 can be found at doi.org/10.4224/crm.2021.dorm-5.

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DORM-5

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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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