National Research Council Canada | Conseil national de recherches Canada

Certificate of Analysis

Certified Reference Material

KRIK-1

Cricket Flour Certified Reference Material

KRIK-1 is a cricket flour Certified Reference Material (CRM) from the National Research Council Canada (NRC) with information on total trace element and species content, as well as nutritional data listed in Supplementary datasheet. A unit of KRIK-1 consists of approximately 18 grams of cricket flour in an amber glass vial.

Table 1 shows the certified, reference and information values established for KRIK-1. 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 KRIK-1

Analyte	Mass fraction, mg/kg	Type of value	International recognition of measurement capability (CMC)
aluminium (c,d,e)	8.4 ± 1.4	certified	TEB-01
arsenic (b,c)	0.288 ± 0.018	certified	MEF-14
arsenobetaine (as As) (g,i)	0.21 ± 0.02	certified	--
inorganic arsenic (as As)(g,h)*	0.0059 ± 0.0010	reference	--
dimethylarsinic acid (as As) (g,h)	0.0209 ± 0.0015	reference	--
barium (c,e)	0.275 ± 0.020	certified	MEF-4
boron (c)	2.19 ± 0.62	reference	MEF-37
bromine (c)	15.1	information	--
cadmium (a,b,c)	0.0743 ± 0.0062	certified	MEF-16
calcium (c,d,e)	1490 ± 80	certified	MEF-17
chlorine (c)	5300	information	--
chromium (a,b)	0.0511 ± 0.0120	certified	MEF-18
cobalt (b)	0.0476 ± 0.0082	reference	MEF-19
copper (c,d,e)	45.7 ± 1.8	certified	MEF-20
iodine (c)	0.21	information	--
iron (a,b,c,d,e)	41.4 ± 1.4	certified	MEF-21
lead (a,b,c)	0.0160 ± 0.0038	certified	MEF-22
magnesium (b,c,d,e)	1110 ± 20	certified	MEF-23

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Analyte	Mass fraction, mg/kg	Type of value	International recognition of measurement capability (CMC)
manganese (c,d,e)	18.4 ± 0.4	certified	MEF-24
mercury (a,c,j)	0.110 ± 0.008	certified	MEF-25
molybdenum (c,d)	0.96 ± 0.08	certified	MEF-27
nickel (a,b,c)	0.215 ± 0.036	certified	MEF-28
phosphorus (b,c,d,e)	9870 ± 260	certified	--
potassium (b,c,d,e)	12 400 ± 200	certified	MEF-29
selenium (a,b,c)	0.84 ± 0.06	certified	MEF-30
selenomethionine (as Se) (f)	0.164 ± 0.030	certified	--
sodium (b,c,d,e)	3690 ± 100	certified	MEF-32
strontium (a,b,c,e)	2.04 ± 0.12	certified	MEF-33
sulfur (b,d,e)	5620 ± 380	reference	MEF-39
zinc (a,b,c,d,e)	150.2 ± 5.8	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) [5,6]
- **g** Standard addition liquid chromatography ICP-MS (SA-LC-ICP-MS) [4]
- **h** Liquid chromatography ICP-MS (LC-ICP-MS) [4]
- **i** Isotope dilution liquid chromatography mass spectrometry (ID-LC-MS)
- **j** Thermal decomposition atomic absorption spectrometry (TD-AAS)

Supplementary data

The accompanying datasheets (available from doi.org/10.4224/crm.2023.krik-1) provide measurement results that were used in this certification campaign. Additional data for total amino acids, free amino acids, fatty acid profile, protein, total carbohydrate, and total dietary fiber are also available.

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 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

KRIK-1 is intended for use in the method development, validation, and quality control for the analysis of trace and matrix constituents in high-protein materials.

Storage and sampling

It is recommended that the material is stored at nominal temperature of +4°C under typical refrigerator conditions. Each vial is packaged in a trilaminate foil pouch. Prior to use, the contents should be well mixed by rotation and shaking, and tightly closed immediately thereafter. Certified values are based on a minimum 250 mg sub-sample.

Instructions for drying

Determination of dry mass should be performed on a separate sample to avoid contamination. Sample should be dried to a constant mass. The estimated moisture content of KRIK-1 is approximately 0.02 g/g.

Preparation of material

This reference material was prepared from a commercial cricket flour. The material was 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 specified dose of 25 kGy gamma irradiation.

Stability

CRMs with similar matrix have been periodically analyzed for more than ten years at NRC and found to be both physically and chemically stable over this time interval. We expect similar results for KRIK-1. 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 Bayesian analysis of variance (ANOVA) [2] to determine both within-unit and between-unit heterogeneity components.

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 [3]. Further information is presented in the supplementary datasheets doi.org/10.4224/crm.2023.krik-1.

Metrological traceability

Results presented in this certificate are traceable to the International System of Units (SI) through CRMs produced by National Metrology Institutes and gravimetrically prepared standards of established purity. As such, KRIK-1 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.2023.krik-1.

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Cited by

A list of scientific publications citing KRIK-1 can be found at doi.org/10.4224/crm.2023.krik-1.

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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