National Research Council Canada | Conseil national de recherches Canada

Certificate of Analysis

Certified Reference Material

KINO-1

Quinoa Flour Certified Reference Material for Trace Metals and Minerals

KINO-1 is a Certified Reference Material (CRM) prepared at the National Research Council of Canada (NRC) from food grade quinoa flour. A single unit of KINO-1 consists of 18 g of quinoa flour. This material is intended for the calibration of procedures and the development of methods for the determination of trace metals and minerals in quinoa flour or similar matrices.

The following tables show those constituents for which certified and reference values have been established for this CRM. 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 is the coverage factor. A coverage factor of two (2) was applied. 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: Mass fractions and expanded uncertainties (k = 2) for KINO-1

Analyte	Mass fraction, mg/kg	Type of Value	International recognition of measurement capability (CMC)
arsenic (b, c)	0.001 00 ± 0.000 38	certified	MEF-14
cadmium (a, c)	0.063 ± 0.006	certified	MEF-16
calcium (c, d)	720 ± 100	reference	MEF-17
iron (a, c, d)	84.1 ± 2.6	certified	MEF-21
magnesium (c, d)	1970 ± 300	reference	MEF-23
mercury (a)	0.000 97 ± 0.000 26	certified	MEF-25
lead (a)	0.0028 ± 0.0008	certified	MEF-22
potassium (c, d)	6010 ± 720	reference	MEF-29
selenium (a, c)	0.17 ± 0.02	certified	MEF-30
sodium (c, d)	37 ± 8	reference	MEF-32
strontium (a, c, d)	2.49 ± 0.18	certified	MEF-33
zinc (a, c, d)	30.6 ± 0.8	certified	MEF-35

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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** Standard addition inductively-coupled plasma atomic emission spectroscopy (SA-ICP-AES)

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 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.

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 CRM is intended for the calibration of procedures and the development of methods for the determination of major, minor and trace constituent elements in flour and other similar food, agricultural, or biological materials. This CRM is not intended for human consumption. A minimum sample mass of 250 mg is recommended for analysis.

Storage and sampling

It is recommended that the material be stored at room temperature in its original bottle, tightly capped, and not exposed to intense direct light or ultraviolet radiation. 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

A separate sample aliquot should be dried to a constant mass to obtain moisture content. KINO-1 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 or (2) vacuum drying (about 0.5 mm Hg) at room temperature for 24 hours. The estimated moisture content of KINO-1 is approximately 0.09 g/g.

Preparation of material

The source of material for KINO-1 was food-grade quinoa flour. The material was screened to pass a 600 µm (No. 30) screen, blended, and bottled at NRC. After bottling, the samples were radiation sterilized with a minimum dose of 25 kGy at the Nordion Gamma Centre of Excellence (Laval, QC, Canada) to minimize any effects from biological activity.

Stability

Potential instabilities due to long-term storage and transport were not considered as similar NRC CRM/RM food matrices have been periodically analyzed for more than fifteen years and found to be both physically and chemically stable over this time interval. We expect similar results for KINO-1. Uncertainty components for long and short term stability were 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 [2,3] and included in the calculation of the certified values.

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 KINO-1

Analyte	u_c, mg/kg	u_{char}, mg/kg	u_{hom}, mg/kg	u_{method}, mg/kg
arsenic	0.000 19	0.000 19	0	0
cadmium	0.003	0.003	0	0
calcium	50	10	40	20
iron	1.3	0.8	1.0	0
magnesium	150	40	140	10
mercury	0.000 13	0.000 13	0	0
lead	0.0004	0.0003	0.0003	0
potassium	360	290	210	0
selenium	0.010	0.005	0.009	0.001
sodium	4	4	0	0
strontium	0.09	0.06	0.07	0
zinc	0.4	0.4	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, KINO-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.2018.kino-1

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. DerSimonian R, Laird N (1986) Meta-analysis in clinical trials. *Controlled Clinical Trials* 7: 177-188. doi.org/10.1016/0197-2456(86)90046-2
3. Thompson M, Ellison S.L.R. Dark uncertainty. *Accred. Qual. Assur*. 2011; 16: 483-487 doi.org/10.1007/s00769-011-0803-0

Cited by

A list of scientific publications citing KINO-1 can be found at doi.org/10.4224/crm.2018.kino-1

Authorship

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Approved by:

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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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