National Institute of Standards & Technology

Report of Investigation

Reference Material 8438

Soft Winter Wheat Flour

Agriculture Canada

Distributed by the National Institute of Standards and Technology

This Reference Material (RM) is intended for use in evaluating analytical methods and instruments used for the determination of major, minor, and trace constituent elements in flour and other similar food, agricultural, and biological materials. This material can also be used for quality assurance when assigning values to in-house control materials. RM 8438 consists of 50 g of flour packaged in two glass bottles containing 25 g each.

Reference Concentration Values: Reference concentration values for major, minor, and trace constituent elements are provided in Table 1. These reference values were derived from results reported in an interlaboratory comparison exercise. Reference values are noncertified values that are the best estimates of the true values; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods.

Information Concentration Values: Information concentration values for additional elements are provided in Table 2. These are noncertified values with no reported uncertainties as there is insufficient information to assess uncertainties. The information values are given to provide additional characterization of the material. Use of this RM to quantitatively monitor method performance for analytes other than those with reference concentration values in Table 1 is not warranted.

Expiration of Report: The Report of Investigation of RM 8438 is valid, within the measurement uncertainty specified, until **31 August 2011**, provided the RM is handled in accordance with instructions given in this report (see "Instructions for Use"). This report is nullified if the RM is damaged, contaminated, or otherwise modified.

Maintenance of RM Value Assignment: NIST will monitor this RM over the period of its value assignment. If substantive technical changes occur that affect the value assignment before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The material was prepared at Agriculture Canada under the direction of M. Ihnat, Centre for Land and Biological Resources Research (CLBRR), who also coordinated the interlaboratory analytical campaign to characterize the material. Statistical support was provided by M.S. Wolynetz, Statistical Research Section, Research Program Service, Agriculture Canada.

Support aspects involved in the issuance of this RM were coordinated through the NIST Measurement Services Division.

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See Report Revision History on Page 5

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NOTICE AND WARNING TO USERS

Storage: Until required for use, RM 8438 should be stored at room temperature in its original bottle, tightly capped, and **NOT** exposed to intense direct light or ultraviolet radiation.

Warning: For laboratory use only. **NOT** for human consumption.

Instructions for Use: Prior to each use, contents of the bottle should be well mixed by gentle shaking and rolling of the container. A recommended minimum subsample mass of 0.5 g should be taken for analysis. Moisture content should be determined on a separate subsample for conversion of analytical results to a dry-mass basis. The recommended method of drying to relate analytical results to the reference values listed in Table 1 is drying for 4 hours in an air oven at 85 °C. Values reported in Tables 1 and 2 represent total concentrations of elements in this RM. Dissolution procedures should be capable of rendering a completely dissolved sample and should be designed to avoid losses of elements by volatilization or by retention on decomposition and processing containers and measuring equipment. Analytical methods should be capable of measuring total levels of analytes for comparison with reference values.

PREPARATION AND ANALYSIS[1]

Preparation: The material used for preparation of RM 8438 was unbleached, enriched soft winter wheat flour obtained from Maple Leaf Mills, Almonte, Ontario, Canada. All preparatory work following acquisition of the commercial product was performed at the facilities of Agriculture Canada, Ottawa [1]. The dry bulk powder was sterilized with ^{60}Co gamma radiation to 2.0 megarads by Atomic Energy of Canada Ltd. All subsequent processing was performed using plastic equipment. The material was sieved through nylon monofilament sieve cloths supported in high density white polyethylene holders. Pairs of sieves with openings of approximately 150 μm and 50 μm were used to yield suitable narrow middle cuts constituting the RM. This fraction was blended in a polymethylmethacrylate V-configuration blender and packaged into clean 70 mL brim capacity, colorless glass bottles with triseal (polyethylene)-lined black polypropylene screw caps.

Assessment of Homogeneity: Homogeneity testing was performed on duplicate samples taken from four randomly selected units by one laboratory. No statistically significant heterogeneity was found for calcium, copper, iron, potassium, magnesium, manganese, sodium, or zinc in 2 g samples [2,3]. Data for all analytes have been treated as though they are homogeneous, although the homogeneity of other analytes has not been investigated.

Value Assignment: Reference concentrations of elements were determined using results from the homogeneity assessment described above and an interlaboratory comparison exercise involving laboratories shown in Appendix A [4]. Analytical methods are provided in Table 3. In the interlaboratory comparison exercise, analyses were performed by each participant on duplicate subsamples from randomly selected (usually four) units of material using subsample masses and methods left to the discretion of the analyst. Subsample sizes ranged from 0.001 g to 5 g, typically 1 g. Elemental determinations were performed on "as received" material, with conversion of results to a dry-mass basis using moisture values determined on separate 2 g subsamples dried by the procedure specified in this report. Reference values provided in Table 1 are based on results generally obtained by at least two, but typically several, independent analytical methods. Concentration estimates for additional elements are provided in Table 2 as information values only, as they are based on results of limited determinations or only one analytical method, or may lack sufficient statistical agreement among multiple analytical methods.

[1]Certain commercial materials and equipment are identified in order to adequately specify the experimental procedure. Such identification does not imply a recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are the best available for the purpose.

Table 1. Reference Concentrations of Constituent Elements

Element	Mass Fraction (%)[a]	Methods
Nitrogen	1.756 ± 0.029	I01, J01, J02
Phosphorus	0.108 ± 0.006	B02, F01, F02
Potassium	0.148 ± 0.021	A01, A03, D01
Sulfur	0.126 ± 0.019	B02, J02, J04, M02

Element	Mass Fraction (mg/kg)[a]	Methods
Calcium	240 ± 23	A01, B02, D01
Chlorine	640 ± 60	D01, F02, K03
Magnesium	214 ± 16	A01, B02, D01
Manganese	5.4 ± 0.6	A01, A03, B02, D01
Selenium	0.076 ± 0.009	C01, C04, G01

[a] Reference values, expressed as mass fractions on a dry-mass basis, are equally weighted means of results from generally at least two, but typically several, different analytical methods applied by analysts in different laboratories. The exception to the approach involving at least two independently different analytical methods for establishing reference values for this RM is the acceptance of data for zinc by essentially a single reliable atomic absorption method applied in two laboratories with suitable quality control. Uncertainties are imprecision estimates expressed either as a 95 % confidence interval, based on a sample mass of at least 0.5 g. These uncertainties, based on between-method, between-laboratory, between-unit, and within-unit estimates of variances, include measures of analytical method and laboratory imprecisions and biases and material inhomogeneity. (NIST has replaced the previously used term "best estimate" with "reference value.")

[b] Analytical method codes and descriptions are provided in Table 3.

Table 2. Information Concentrations of Constituent Elements

Element	Mass Fraction (mg/kg)[a]	Methods
Aluminum	2.3	B02, D01
Barium	1	B02
Boron	0.1	B02
Cadmium	0.03	A06
Chromium	0.032	A06, C05
Copper	1.2	A01, C06, D01
Fluorine	0.04	H04
Iron	29	A01, A03, B02
Mercury	0.002	A09
Molybdenum	0.29	B02, C06, F01
Sodium	7	A01, D01
Vanadium	0.03	B02, D01
Zinc	5.8	A01, A03

[a] These analytical values, on a dry-mass basis, are estimates given strictly for information as they are based on results of limited determinations or only one method, or may lack sufficient statistical agreement among multiple analytical methods; no uncertainties are provided. Use of this RM to quantitatively monitor method performance for elements other than those with reference concentration values in Table 1 is **NOT** warranted.

[b] Analytical method codes and descriptions are provided in Table 3.

Table 3. Analytical Methods Used to Determine Reference and Information Concentration Values[a]

Analytical Method	Code	Elements Determined
Acid digestion flame atomic absorption spectrometry	A01	Ca, (Cu), (Fe), K, Mg, Mn, (Na), (Zn)
Dry ashing flame atomic absorption spectrometry	A03	(Fe), K, Mn, (Zn)
Dry ashing electrothermal atomic absorption spectrometry	A06	(Cd), (Cr)
Acid digestion cold vapor atomic absorption spectrometry	A09	(Hg)
Acid digestion inductively coupled plasma atomic emission spectrometry	B02	(Al), (B), (Ba), Ca, (Cu), (Fe), Mg Mn, (Mo), P, S, (V)
Acid digestion isotope dilution mass spectrometry	C01	Se
Acid digestion dry ashing hydride generation isotope dilution inductively coupled plasma mass spectrometry	C04	Se
Dry ashing acid digestion isotope dilution mass spectrometry	C05	(Cr)
Acid digestion isotope dilution inductively coupled plasma mass spectrometry	C06	(Cu), (Mo)
Instrumental neutron activation analysis	D01	(Al), Ca, Cl, (Cu), K, Mg, Mn, (Na), (V)
Acid digestion light absorption spectrometry	F01	(Mo), P
Dry ashing light absorption spectrometry	F02	Cl, P
Acid digestion fluorometry	G01	Se
Extraction ion selective electrode	H04	(F)
Kjeldahl method for nitrogen -volumetry	I01	N
Combustion elemental analysis -thermal conductivity	J01	N

Combustion elemental analysis with chromatographic separation -thermal conductivity	J02	N
Combustion elemental analysis -fluorometry	J04	S
Combustion volumetry	K03	Cl
Dry ashing gravimetry	M02	S

[(a)] Letter codes refer to classes of similar methods; number codes refer to specific variants. Elements in parentheses have only informational values in this RM.

REFERENCES

[1] Ihnat M.; *Preparation of Twelve Candidate Agricultural Reference Materials*; Fresenius' J. Anal. Chem., Vol. 332, pp. 539–545 (1988).

[2] Ihnat, M.; *High Reliability Atomic Absorption Spectrometry of Major and Minor Elements in Biological Materials*; Fresenius Z. Anal. Chem. Vol. 326, pp. 739–741 (1987).

[3] Ihnat, M.; *Reliable Measurement of Major, Minor, and Trace Elemental Nutrients*; J. Res. Nat. Bur. Stds. Vol. 93, pp. 354–358 (1988).

[4] Ihnat, M.; Stoeppler, M.; *Preliminary Assessment of Homogeneity of New Candidate Agricultural/Food Reference Materials*; Fresenius' J. Anal. Chem., Vol. 338, pp. 455–460 (1990).

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Users of this RM should ensure that the report in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

APPENDIX A

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