National Institute of Standards & Technology

Report of Investigation

Reference Material 8412

Corn (*Zea mays*) Stalk

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 corn stalk 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 8412 consists of approximately 34 g of dry powdered corn (*Zea mays*) stalk packaged in a glass bottle sealed in a foil bag.

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 8412 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.

M. Ihnat and W.R. Wolf of the U.S. Department of Agriculture (USDA) coordinated the interlaboratory analytical campaign to characterize the material. The material was prepared at Agriculture Canada under the direction of M. Ihnat, Centre for Land and Biological Resources Research (CLBRR).

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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Report Issue Date: 20 February 2008
See Report Revision History on Page 4

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NOTICE AND WARNING TO USERS

Storage: Until required for use, RM 8412 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 mass of 0.5 g should be taken for analysis. Moisture content should be determined on a separate test portion 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 and information concentrations listed in Table 2 is drying for 4 h 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 to prepare RM 8412 was obtained from the Central Experimental Farm, Agriculture Canada, Ottawa, Ontario, Canada. For the preparation of this RM, corn plants were manually harvested in the autumn of 1981, and manually separated into the component leaves, stalks, and ears. Stalks were washed free of any adhering solid, coarse-chopped, dried for at least 24 h at 80 °C in an air oven, and ground in a hammer mill. The resulting fine powder was sieved through nylon and Teflon monofilament sieve cloths supported in polyethylene holders, with material passing 210 µm (about - 70 mesh) collected as the RM. This fraction was blended in a V-type feed blender, packaged in glass bottles with linerless white polypropylene caps, and sterilized with ^{60}Co radiation to 2.0 megarads by Atomic Energy of Canada, Ltd. All preparatory work was performed in-house at the facilities of Agriculture Canada, Ottawa.

Assessment of Homogeneity: Homogeneity testing was performed on duplicate test portions of three masses (0.2 g, 0.5 g, and 1.0 g) taken from eight randomly selected units by two laboratories using acid digestion and flame atomic absorption spectrometry. No statistically significant heterogeneity was found for calcium (Ca), copper (Cu), iron (Fe), potassium (P), magnesium (Mg), manganese (Mn), sodium (Na), strontium (Sr), or zinc (Zn) in 0.5 g and 1.0 g samples. Due to low concentrations of some elements in the solutions resulting from the 0.2 g test portions, no concrete statements regarding material homogeneity at 0.2 g can be made for copper, iron, sodium, and zinc. Minimum test portions of 0.5 g should be used for measurement of all analytes. Data for all analytes have been treated as though they are homogeneous at the 0.5 g level, 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. Analytical methods are provided in Table 3. In the interlaboratory comparison exercise, analyses were performed by each participant on four test portions taken from two randomly selected jars. All analyses were performed on the material as received, with results converted to a dry-mass basis using moisture content determined at Agriculture Canada on test portions from separate units. Sample sizes used for analysis ranged from 0.1 g to 10 g, typically 0.2 g to 1.0 g. Reference values are based on results obtained by at least two, but typically several, independent analytical methods. Concentration estimates for four other 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.

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[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 [(b)]
Calcium	0.216 ± 0.008	A01, B04, D01
Chlorine	0.244 ± 0.014	D01, K04
Magnesium	0.160 ± 0.007	A01, B04, D01
Potassium	1.735 ± 0.047	A01, D01

Element	Mass Fraction (mg/kg)[(a)]	Methods[(b)]
Copper	8 ± 1	A01, B04, C10
Iron	139 ± 15	A01, B04, C06, D01
Manganese	15 ± 2	A01, B04, D01
Strontium	12 ± 2	A01, C10
Zinc	32 ± 3	A01, B04, C10, D01

(a) Reference values, expressed as mass fractions, are based on the dry material, dried according to instructions in this report, and are equally weighted means of results from different analytical methods applied by analysts in different laboratories. Uncertainties are imprecision estimates expressed 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, 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[(b)]
Fluorine	0.65	H04
Nitrogen	6970	I01
Selenium	0.016	C01, G01
Sodium	28	A01, D01

(a) These analytical values, on a dry-mass basis, are estimates given strictly for information only as they are based on results of limited determinations or only one method; 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), Sr, Zn
Dry ashing inductively coupled plasma atomic emission spectrometry	B04	Ca, Cu, Fe, Mg, Mn, Zn
Acid digestion isotope dilution mass spectrometry	C01	(Se)
Acid digestion isotope dilution spark source mass spectrometry	C10	Cu, Fe, Sr, Zn
Instrumental neutron activation analysis	D01	Ca, Cl, Fe, K, Mg, Mn, (Na), Zn
Acid digestion fluorometry	G01	(Se)
Oxygen flask combustion coulometric titrimetry	K04	Cl
Extraction ion selective electrode	H04	(F)
Kjeldahl method for nitrogen-volumetry	I01	(N)

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

Report Revision History: 20 February 2008 (Update of expiration date and editorial changes)**;** 03 April 2003 (Corrected number of bottles given in the unit size description); 24 September 2001 (This report revision reflects editorial changes); 18 February 1994 (Technical changes; method change for chlorine and potassium); 24 September 1993 (Editorial changes); 12 February 1986 (Original report date).

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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