National Institute of Standards and Technology
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Standard Reference Material® 695
Trace Elements in Multi-Nutrient Fertilizer
CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is intended primarily for the evaluation of techniques used in the analysis of multi-nutrient fertilizer materials and materials of a similar matrix.

Description: A unit of SRM 695 consists of approximately 70 g of jet-milled fertilizer in an amber bottle sealed in an aluminized polyester bag.

Certified Values: A NIST certified value is a value for which NIST has the highest confidence in that all known or suspected sources of bias and imprecision have been considered and any contributions they may make to measurement uncertainty have been quantified and are expressed in the reported uncertainty [1]. Certified mass fraction values for elements in SRM 695, reported on a dry-mass basis, are provided in Table 1. The measurands in Table 1 are total mass fractions for each analyte reported and metrological traceability is to the International System of Units (SI) derived unit for chemical mass fraction expressed as either milligrams per kilogram or as a percentage [2].

Table 1. Certified Mass Fraction Values (Dry-Mass Basis) for SRM 695[(a)]

Major and Minor Constituent Elements

Element	Mass Fraction (%)	Element	Mass Fraction (%)
Calcium (Ca)	2.26 ± 0.04	Manganese (Mn)	0.305 ± 0.005
Iron (Fe)	3.99 ± 0.08	Sodium (Na)	0.405 ± 0.007
Magnesium (Mg)	1.79 ± 0.05	Potassium (K)	11.65 ± 0.13
		Zinc (Zn)	0.325 ± 0.005

Trace Elements

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Arsenic (As)	200 ± 5	Lead (Pb)	273 ± 17
Cadmium (Cd)	16.9 ± 0.2	Mercury (Hg)	1.955 ± 0.036
Chromium (Cr)	244 ± 6	Molybdenum (Mo)	20.0 ± 0.3
Cobalt (Co)	65.3 ± 2.4	Nickel (Ni)	135 ± 2
Copper (Cu)	1225 ± 9	Vanadium (V)	122 ± 3

(a) Certified values for all elements except arsenic and mercury are the unweighted means of results from two or three analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance with a pooled, within method variance [3] following the ISO/JCGM Guide [4]. The certified values for As and Hg are each results from a single NIST method [instrumental neutron activation analysis (INAA) for As, and cold vapor, isotope dilution, inductively coupled plasma mass spectrometry (CV-ID-ICP-MS) for Hg] for which a complete evaluation of all sources of uncertainty has been performed. The uncertainty for each certified value is an expanded uncertainty with a coverage factor of 2, with uncertainty components combined following the ISO/JCGM Guide [4].

Period of Validity: The certified values delivered by **SRM 695** are valid within the measurement uncertainty specified until **01 April 2026**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

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Steven J. Choquette, Director
Office of Reference Materials

Certificate Revision History on Page 2

Non-Certified Values: Non-certified values and a table of analytical methods are provided in Appendix A.

Additional Information: Results from a test method developed by members of the Association of American Plant Food Control Officials (AAPFCO) (West Lafayette, IN) are provided as values of potential interest in Appendix B. Values of potential interest for additional trace elements and the source and preparation of SRM 695 can also be found in Appendix B.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Safety: SRM 695 is intended for research use. Please consult the Safety Data Sheet for this product.

Storage: The original unopened bottles of SRM 695 should be stored tightly sealed and away from sunlight, moisture, and intense sources of radiation at room temperature (20 °C ± 5 °C). An open bottle can be reused until the material reaches its expiration date, provided that the open bottle is resealed and stored at room temperature (20 °C ± 5 °C).

Use: Before it is sampled, the unit should be thoroughly mixed by carefully and repeatedly inverting and rotating the tightly sealed bottle horizontally. A minimum test portion of 200 mg should be used for analytical determinations to be related to the elemental certified mass fraction values and expanded uncertainties provided.

Drying Instructions: To relate their measurements directly to the certified and non-certified values, which are expressed on a dry-mass basis, users should determine a drying correction at the time of each analysis. The recommended drying procedure is desiccator drying of portions with a depth ≤0.5 cm, for two weeks, over fresh magnesium perchlorate. The average mass loss measured at NIST using this method for six portions of SRM 695 was 1.34 % (1 s = 0.06 %). Oven drying, even at relatively low temperatures (85 °C), can result in decomposition of the carbonate and ammonium (and possibly other) compounds in this blended fertilizer material. DO NOT dry SRM 695 in an oven to determine the dry-mass basis.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Sep 2023).

[2] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at https://www.nist.gov/pml/special-publication-811 (accessed Sep 2023).

[3] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, pp. 571–579 (2000); available at https://tsapps.nist.gov/publication/get_pdf.cfm?pub_id=151758 (accessed Sep 2023).

[4] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Sep 2023); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at https://www.nist.gov/pml/nist-technical-note-1297 (accessed Sep 2023).

[5] Kane, P.F.; Hall, W.L., Jr.; *Analysis of Arsenic, Cadmium, Cobalt, Chromium, Lead, Molybdenum, Nickel, and Selenium by Microwave Digestion and ICP-OES Detection*: *Collaborative Study*; Paper 2006-17920 of the Office of Indiana State Chemist, Purdue University Agricultural Experiment Station, W. Lafayette, IN (2006).

Certificate Revision History: **18 September 2023** (Added values of potential interest to users for additional trace elements; updated format; editorial changes); **28 December 2015** (Change of expiration date; editorial changes); **26 June 2006** (Original certificate date).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the SI or other higher-order reference system [1].

Table A1. Non-Certified Mass Fraction Values (Dry-Mass Basis) for SRM 695[(a)]

Major and Minor Constituent Elements

Element	Mass Fraction (%)	Element	Mass Fraction (%)
Aluminum (Al)	0.61 ± 0.03	Nitrogen (N)	13.9 ± 0.4
Boron (B)	0.111 ± 0.002	Phosphorus (P)	7.2 ± 0.1

Trace Elements[(a)]

Element	Mass Fraction (mg/kg)
Selenium (Se)	2.1 ± 0.1

[(a)] Non-certified values for all elements except aluminum are based on results of one analytical method at NIST and the uncertainty values represent the expanded uncertainties which include the combined Type A and Type B uncertainties with a coverage factor of 2, following the ISO/JCGM Guide [4]. The non-certified value for aluminum is the unweighted mean of results from two analytical methods and the uncertainty listed is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance with a pooled, within method variance [3] following the ISO/JCGM Guide [4]. The measurand is the mass fraction for each element listed in Table A1 as determined by the method indicated.

Period of Validity: The non-certified values are valid within the measurement uncertainty specified until **01 April 2026**. The value assignments are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Appendix and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Table A2. Methods of Analysis for SRM 695

Element	Method(s)	Element	Method(s)
Al	INAA, XRF	Mg	INAA, XRF
As	INAA	Mn	PGAA, XRF, INAA
B	PGAA	Mo	ICP-OES, XRF
Ca	XRF, INAA	N	PGAA
Cd	ID-ICP-MS, PGAA	Na	INAA, XRF
Cl	INAA	Ni	ICP-OES, XRF
Co	INAA, XRF	P	XRF
Cr	INAA, XRF	Pb	ICP-OES, XRF
Cu	ID-ICP-MS, ICP-OES, XRF	Se	INAA
Fe	INAA, PGAA, XRF	Ti	XRF
Hg	CV ID-ICP-MS	V	INAA, XRF
K	PGAA, XRF	Zn	INAA, XRF

Methods:

CV ID-ICP-MS	Cold Vapor, Isotope Dilution, Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
ID-ICP-MS	Isotope Dilution Inductively Coupled Plasma Mass Spectrometry
INAA	Instrumental Neutron Activation Analysis
HG-AAS	Hydride Generation Atomic Absorption Spectrometry
PGAA	Prompt Gamma-ray Activation Analysis
XRF	X-ray Fluorescence Spectrometry

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APPENDIX B

Values of Potential Interest

Results from Test Method for Determination of Analysis of As, Cd, Co, Cr, Mo, Ni, Pb, and Se in Fertilizers

The certified and non-certified values presented in the Certificate of Analysis for SRM 695 represent the total elemental mass fractions. These values are obtained either from non-destructive methods such as INAA or methods that involve a complete dissolution of the material prior to performing measurements. For more routine analysis of metals in fertilizer materials, a method was developed and tested by members of AAPFCO. W. Hall initiated the development of this method and P. Kane coordinated and led this study in which ten laboratories participated. This test method involves microwave digestion with concentrated nitric acid followed by ICP-OES detection and is described in detail elsewhere [5].

Note that this digestion method does not completely dissolve this fertilizer material. Results indicate that only some elements are completely extracted. The results obtained using this method are shown in Table B1, together with the certified total elemental mass fractions, and the percent recovery defined as the ratio of the values obtained from the test method to certified total element mass fractions. Collaborating laboratories and analysts are listed in Table B2.

Table B1. Values of Potential Interest Results from Test Method for Determination of As, Cd, Co, Cr, Mo, Ni, Pb, and Se in Fertilizers[(a)]

Element	Test Method Results Average Mass Fraction (1*s*); Range (mg/kg)	Recovery Average; Range (%)	Total Elemental Mass Fraction[(b)] (mg/kg)
As	193 (19); 171 – 235	96 %; 85 % – 117 %	200 (5)
Cd	16.1 (2.9); 12.4 – 23.2	95 %; 74 % – 137 %	16.9 (0.2)
Co	47.5 (12.3); 27.4 – 65.7	73 %; 42 % – 101 %	65.3 (2.4)
Cr	174 (19); 136 – 192	71 %; 56 % – 79 %	244 (6)
Mo	14.0 (2.0); 10.2 – 16.8	70 %; 51 % – 84 %	20.0 (0.3)
Ni	112 (15); 85 – 131	83 %; 63 % – 97 %	135 (2)
Pb	257 (15); 231 – 281	94 %; 85 % – 103 %	273 (17)

(a) Selenium values are not included because the mass fraction of Se in SRM 695 is below the method detection limit.
(b) The values shown in this column are the certified total element mass fraction values, with expanded uncertainties inside the parentheses, from Table 1 of the Certificate of Analysis.

Table B2. Collaborating Laboratories and Analysts

J. Bartos	Division of Regulatory Services, University of Kentucky (Lexington, KY)
R. Boles	Experiment Station, University of Missouri (Columbia, MO)
M. Dupuis	Ottawa Lab (Carling) Canadian Food Inspection Agency (Ottawa, ON)
E. Hasty	CEM Corporation (Matthews, NC)
W. Hall, C. Kinsey, and K. Sakyi-Amfo	The Mosaic Company (Tampa, FL)
P. Kane, S. Mullins, and N. Newlon	Office of the Indiana State Chemist (West Lafayette, IN)
J. Purkiss	Michigan Department of Agriculture (Grand Rapids, MI)
C. Rivera	Varian Inc. (Palo Alto, California)
W. Robarge	North Carolina State University (Raleigh, NC)
C. Seeley	Teledyne Leeman Labs (Hudson, NH)
S. Seigel	CF Industries (Washington, DC)
M. Svee	Montana Department of Agriculture (Helena, MT)
T. Van Erem	South Dakota State University (Brookings, SD)
A. Vindiola	Office of the Texas State Chemist (College Station, TX)

Mass Fraction Value of Interest: The mass fraction values in Tables B3, B4 and B5 are given as additional information on the matrix and are provided without uncertainty estimates because insufficient information is available to give a reasonable estimate of each uncertainty.

Table B3. Mass Fraction Values of Interest (Dry-Mass Basis) Determined by One Analytical Method at NIST

Element	Mass Fraction
Chlorine (Cl)	4.6 %
Titanium (Ti)	310 mg/kg

Table B4. Mass Fraction Values of Interest (As Is Basis) for SRM 695 Determined Externally by Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Antimony (Sb)	4	Holmium (Ho)	1	Silicon (Si)	2164
Barium (Ba)	49	Iodine (I)	16	Silver (Ag)	2
Beryllium (Be)	1	Lanthanum (La)	5	Strontium (Sr)	128
Bromine (Br)	697	Lithium (Li)	4	Thallium (Tl)	1
Cerium (Ce)	5	Lutetium (Lu)	1	Thorium (Th)	9
Dysprosium (Dy)	2	Neodymium (Nd)	3	Tin (Sn)	22
Erbium (Er)	2	Niobium (Nb)	1	Tungsten (W)	5
Europium (Eu)	32	Praseodymium (Pr)	1	Uranium (U)	282
Gadolinium (Gd)	1	Rubidium (Rb)	30	Ytterbium (Yb)	4
Gallium (Ga)	4	Samarium (Sm)	1	Yttrium (Y)	39
Germanium (Ge)	1	Scandium (Sc)	4	Zirconium (Zr)	19

Table B5. Elements Not Detected by ICP-MS That May be Present at Mass Fractions Below 1 mg/kg

Element	Element	Element	Element
Bismuth (Bi)	Indium (In)	Platinum (Pt)	Tantalum (Ta)
Cesium (Cs)	Iridium (Ir)	Rhenium (Re)	Tellurium (Te)
Gold (Au)	Osmium (Os)	Rhodium (Rh)	Terbium (Tb)
Hafnium (Hf)	Palladium (Pd)	Ruthenium (Ru)	Thulium (Tm)

Source and Preparation of Material: This multi-nutrient blended fertilizer was developed in collaboration with members of AAFPCO and The Fertilizer Institute (TFI) [Arlington, VA]. The material used to prepare SRM 695 was provided to NIST by W.L. Hall (The Mosaic Company, Tampa, FL). The material consists of urea, diammonium hydrogen phosphate, calcium carbonate, potassium chloride, potassium nitrate, and potassium magnesium sulfate, and various other metal sulfates. The material was ground and shipped to NIST where the Office of Reference Materials jet-milled, blended, and bottled it.

Analyses of this material used for certification were performed at NIST (Gaithersburg, MD) and at the United States Geological Survey. The analytical techniques used for each element are listed in Table A2.

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