National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1570a

Trace Elements In Spinach Leaves

This Standard Reference Material (SRM) is intended primarily for use in evaluating the reliability of analytical methods for the determination of major, minor, and trace elements in botanical materials, agricultural food products, and materials of similar matrix. A unit of SRM 1570a consists of 60 g of finely powdered dried spinach leaves.

Certified and Noncertified Concentrations of Constituent Elements: The certified concentrations of the constituent elements are given in Table 1. These concentrations are based on the agreement of results from at least two independent analytical methods or from a method of known accuracy. Noncertified concentrations of constituent elements are provided for information only in Table 2.

NOTICE AND WARNINGS TO USERS

Expiration of Certification: This certification is valid for five (5) years from the date of shipment from NIST. Should any of the certified values change before the expiration of the certification, purchasers will be notified by NIST. Please return the attached registration card to facilitate notification.

Stability: This material was radiation sterilized at an estimated minimum dose of 27.8 kGy for microbiological control; however, its stability has not been rigorously assessed. Spinach leaves have a tendency to rapidly bleach and to turn a tan or light brown color in the presence of visible light. Based on 15 years experience with the original SRM 1570, there is no evidence documenting any change in elemental concentrations as a result of that color change. However, NIST will monitor this material and will report any substantive changes in certification to the purchaser.

Storage: The material should be kept tightly closed in its original bottle and stored in the dark at a temperature between 10 and 30 °C. It should not be exposed to intense sources of radiation. Ideally, the bottle should be kept in a desiccator under the conditions indicated above.

Use: The bottle should be thoroughly mixed by rotating and/or rolling before each use. Allow the contents to settle for 1 min prior to opening to minimize the loss of fine dust particles. A minimum sample weight of 150 mg of the material, dried as described in the section on "Instructions for Drying", should be used to relate analytical determinations to the certified values on this certificate. In some cases, especially for volatile elements such as mercury, it is preferable to analyze samples from the bottle without drying, determine the moisture content on a separate sample from the same bottle taken at the same time, and correct the analytical results to a dry weight basis.

Digestion procedures should be designed to avoid loss of volatile elements, such as arsenic and mercury. Digestion of the SRM in nitric and perchloric acids was found to be incomplete, with a small residue of siliceous material remaining. This residue must be considered an integral part of this SRM and should be dissolved with a small amount of hydrofluoric acid to obtain total dissolution. All certified values are based on the total dissolution.

The original technical and support aspects involved in the certification and issuance of this SRM were coordinated through the Standard Reference Materials Program by R. Alvarez and T.E. Gills. Revision of this certificate was coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
July 15, 1996
(Revision of certificate dated 10-20-94)

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

Coordination of all analytical measurements used in the characterization of this SRM was performed by D.A. Becker of the NIST Inorganic Analytical Research Division.

Statistical analysis of the experimental data was performed by W. Guthrie and S.B. Schiller of the NIST Statistical Engineering Division.

Instructions for Drying: Samples of this SRM must be dried by one of the following two procedures:

1. Drying in a desiccator at room temperature (approximately 22 °C) for 120 h over fresh anhydrous magnesium perchlorate. The sample depth should not exceed 1 cm.

2. Freeze drying for 24 h at a pressure of 13.3 Pa or lower and a shelf temperature of -5 °C or lower after having frozen the sample (not to exceed 1 cm in depth) at -40 °C or lower for at least 1 h. At the end of the 24-h period, samples are placed immediately in a desiccator with fresh anhydrous magnesium perchlorate. Samples are weighed after allowing a minimum of 4 h to establish temperature equilibrium.

Note: Vacuum drying at room temperature and oven drying at elevated temperatures have resulted in excessive weight losses and therefore are <u>not</u> recommended.

Homogeneity Assessment: Samples from randomly selected bottles of SRM 1570a were tested for homogeneity by instrumental neutron activation analysis (INAA). No evidence of chemically significant inhomogeneity was observed.

Certified Concentrations and Uncertainties: The certified concentrations are equally weighted means of results from two or more different analytical methods or the mean of results from a single method of known high accuracy. In the case of two or more methods, each uncertainty is the sum of a 95% confidence limit and an allowance for systematic error between the methods used. In the case of a method of known accuracy, each uncertainty is the sum of a 95% confidence limit and the known systematic error of the method.

Table 1. Certified Concentrations of Constituent Elements

Element	Concentration Wt %*
Calcium	1.527 ± 0.041
Nitrogen (Total)	5.90 ± 0.25
Phosphorus	0.518 ± 0.011
Potassium	2.903 ± 0.052
Sodium	1.818 ± 0.043

*Wt % = mg/kg x 10^{-4}

Element	Concentration (mg/kg)	Element	Concentration (mg/kg)
Aluminum	310 ± 11	Mercury	0.030 ± 0.003
Arsenic	0.068 ± 0.012	Nickel	2.14 ± 0.10
Boron	37.6 ± 1.0	Selenium	0.117 ± 0.009
Cadmium	2.89 ± 0.07	Strontium	55.6 ± 0.8
Cobalt	0.39 ± 0.05	Thorium	0.048 ± 0.003
Copper	12.2 ± 0.6	Vanadium	0.57 ± 0.03
Manganese	75.9 ± 1.9	Zinc	82 ± 3

Elements other than those certified are present in this material. Those that were determined, but not certified, are provided as additional information on the composition. Although total nitrogen is certified, nitrogen determined by the Kjeldahl procedure is not.

Table 2. Noncertified Concentrations of Constituent Elements

Element	Concentration Wt %*
Magnesium	(0.89)
Nitrogen (Kjeldahl)†	(5.74)
Sulfur	(0.46)

*Wt % = mg/kg x 10^{-4}

Element	Concentration (mg/kg)	Element	Concentration (mg/kg)
Europium	(0.0054)	Rubidium	(13)
Lead	(0.20)	Scandium	(0.055)
		Uranium	(0.15)

†**Method Reference:** Official Methods of Analysis of the Association of Official Analytical Chemists, Arlington, VA, 14th Ed., 1984, p. 16, Nitrogen (Total) in Fertilizers, Kjeldahl Method (Final Action): Method 2.057, Improved Method for Nitrate Free Samples. Samples were dried as described in procedure 1 under "Instructions for Drying".

Source and Preparation of Material: The material, (approximately 2270 kg) for this SRM was obtained from a commercial supplier by Oregon Freeze-Drying Corp., Albany, OR. It consists of a U.S. Grade A chopped frozen spinach. The material was thawed, placed in a ribbon mixer, thoroughly mixed, and blended. After mixing, the spinach was freeze-dried. The freeze-dried material was then ground in a stainless steel grinder and shipped to NIST. At NIST, the freeze-dried material was sieved through a polypropylene sieve having openings of 0.25 mm (equivalent to a U.S. Series 60 standard sieve). The sieved material was then jet milled and air classified to a particle size of approximately 75 μm (200 mesh). After mixing in a large blender, the spinach was irradiated with cobalt-60 radiation to a minimum absorbed dose of approximately 27.8 kGy for microbiological control and bottled.

Table 3. Methods and Analysts for
Certified Elemental Determination

Element	Method Code	Element	Method Code
Aluminum	ICP INAA	Nitrogen	KJEL PGAA
Arsenic	FI-HGAAS RNAA	Phosphorus	COLOR ICP
Boron	IDICPMS PGAA	Potassium	IDTIMS INAA
Cadmium	RNAA IDICPMS PGAA	Selenium	FI-HGAAS INAA RNAA
Calcium	IDTIMS INAA	Sodium	PGAA INAA
Cobalt	INAA RNAA	Strontium	IDTIMS INAA
Copper	ICP RNAA	Thorium	INAA RNAA
Manganese	LEAFS INAA	Vanadium	INAA IDTIMS
Mercury	CVAAS RNAA	Zinc	ICP INAA
Nickel	IDICPMS RNAA		

Analytical Methods:

CVAAS = Cold-Vapor Atomic Absorption Spectrometry
FI-HGAAS = Flow Injection Hydride Generation Atomic Absorption Spectrometry
ICP = Inductively-Coupled Plasma Emission Spectrometry
IDICPMS = Isotope Dilution, Inductively Coupled Plasma Mass Spectrometry
IDTIMS = Isotope Dilution, Thermal Ionization Mass Spectrometry
INAA = Instrumental Neutron Activation Analysis
KJEL = Kjeldahl Nitrogen Determination
LEAFS = Laser-Excited Atomic Fluorescence Spectrometry
PGAA = Prompt Gamma Activation Analysis
RNAA = Radiochemical Neutron Activation Analysis

Analysts, National Institute of Standards and Technology:

1. Beary, E.S.
2. Becker, D.A.
3. Beck, C.M., II
4. Epstein, M.S.
5. Fassett, J.D.
6. Garrity, K.M.
7. Greenberg, R.R.
8. Lindstrom, R.M.
9. Mackey, E.A.
10. Morales, P.
11. Murphy, K.E.
12. Paulsen, P.J.
13. Rush, T.A.
14. Sara Swati, R.
15. Smeller, J.M.
16. Turk, G.C.
17. Vocke, R.D.
18. Watters, R.L., Jr.
19. Wood, L.J

Cooperating Analysts:

Anderson, D.L., Center for Food Safety and Applied Nutrition, U.S. Food and Drug Administration, Washington, DC.

Byrne, A.R., Nuclear Chemistry Department, Jozef Stefan Institute, Ljubljana, Slovenia.

Kucera, J., Nuclear Physics Institute, Academy of Sciences of Czech Republic, Rez, Czechoslovakia.