National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1573a

Tomato 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 1573a consists of 50 g of dried tomato leaves.

Certified and Noncertified Values of Constituent Elements: The certified values of the constituent elements are given in Table 1. These values are based on the agreement of results from at least two independent analytical methods or the mean of results from a method of known accuracy. Noncertified values of constituent elements are provided for information only in Table 2. All values are reported as mass fractions [1].

NOTICE AND WARNINGS TO USERS

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

Stability: This material was radiation sterilized at an estimated minimum dose of 25 kGy (2.5 Mrads) for microbiological control. However, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes 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 °C 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 contents should be thoroughly mixed by rotating and/or rolling the bottle before each use. Allow the contents to settle for one minute prior to opening. A minimum sample of 150 mg of (dry mass - see "Instructions for Drying"), should be used to relate analytical determinations to the certified values in this certificate. Volatile elements (e.g., arsenic, mercury, and selenium) should be determined on samples as received; separate samples from the same bottle should be dried according to these instructions to obtain a correction factor for moisture. This factor is then to be used to correct the analytical results to a dry mass basis.

Dissolution: Digestion procedures should be designed to avoid loss of volatile elements. 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 the SRM and should be treated with a small amount of hydrofluoric acid to obtain total dissolution.

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

Statistical analysis of the experimental data was performed by W.F. Guthrie of the NIST Statistical Engineering Division.

The technical and support aspects involved in the certification and issuance of this SRM were coordinated through the Standard Reference Materials Program by R.A. Alvarez and T.E. Gills.

Gaithersburg, MD 20899
November 22, 1995
(Revision of certificate dated 10-19-93)

Thomas E. Gills, Chief
Standard Reference Materials Program

Instructions for Drying: Samples of this SRM must be dried only 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 mass losses and therefore are not recommended.

Homogeneity Assessment: Homogeneity was assessed by careful evaluation of the analytical data used for certification. No evidence of chemically or statistically significant inhomogeneity was observed.

Table 1. Certified Mass Fractions (w_B)

Element	w_B (in %)
Calcium	5.05 ± 0.09
Nitrogen (Total)	3.03 ± 0.15
Phosphorus	0.216 ± 0.004
Potassium	2.70 ± 0.05

Element	w_B (in mg/kg)	Element	w_B (in mg/kg)
Aluminum	598 ± 12	Mercury	0.034 ± 0.004
Antimony	0.063 ± 0.006	Nickel	1.59 ± 0.07
Arsenic	0.112 ± 0.004	Rubidium	14.89 ± 0.27
Boron	33.3 ± 0.7	Selenium	0.054 ± 0.003
Cadmium	1.52 ± 0.04	Sodium	136 ± 4
Chromium	1.99 ± 0.06	Vanadium	0.835 ± 0.010
Cobalt	0.57 ± 0.02	Zinc	30.9 ± 0.7
Copper	4.70 ± 0.14		
Iron	368 ± 7		
Manganese	246 ± 8		

Certified Values and Uncertainties: The certified values are equally weighted means of results from two or more different analytical methods or the mean of results from a method of known 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 2. Noncertified Mass Fractions (w_B)

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.

Element	w_B (in %)
Hydrogen	5.2
Magnesium	1.2
*Nitrogen (Kjeldahl)	2.92
Sulfur	0.96

Element	w_B (in mg/kg)	Element	w_B (in mg/kg)
Barium	63	Lanthanum	2.3
Bromine	1300	Molybdenum	0.46
Cerium	2	Samarium	0.19
Cesium	0.053	Scandium	0.1
Chlorine	6600	Silver	0.017
Gadolinium	0.17	Strontium	85
Hafnium	0.14	Thorium	0.12
Iodine	0.85	Uranium	0.035

*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 plant material for this SRM was collected and prepared under the direction of C.B. Smith, Plant Analysis Laboratory, The Pennsylvania State University, University Park, PA. The tomato leaves were selected from "Count II" tomato plants grown in three lime and fertilizer experiments covering about three acres at the Horticultural Research Farm at Rock Springs, PA. Mature leaves were selected primarily from guard plants which had not received any treatment in order to obtain as uniform material as possible. Twenty four batches of leaves were collected in paper or plastic containers. Since the leaves averaged only about 11 % dry mass, about three tons of leaves had to be collected. Fungicide sprays containing manganese, zinc, and copper were avoided in order to prevent trace element contamination of the leaves.

After each collection, the leaves were transported to the Plant Analysis Laboratory and washed as soon as possible (usually the same day). Most of the soil contamination was removed in a water spray and then the leaves were dipped in a detergent solution, and rinsed in tap water and three successive rinses of distilled water.

The washed leaves were drained and then placed in large pasteboard trays for drying in ovens at 60 °C to 70 °C. Drying had to be done quickly to avoid decomposition. The leaves were then ground to pass a 40-mesh screen in a Wiley Mill. A representative sample was taken from each batch for analysis using an autoanalyzer with manual digestion for nitrogen and an ICP emission spectrometer for twelve other elements. These analyses allowed for a check on each batch before it was mixed with others.

The leaves were placed in six 55-gallon drums with plastic liners for shipment to NIST. Each drum contained an equal portion from each of the 24 batches.

At NIST, the ground leaves were jet milled and air classified to a particle size of approximately 75 μm (200 mesh). After mixing in a large blender, the leaves in bulk were sent to a private company to be irradiated with cobalt-60 radiation to a minimum absorbed dose of 25 kGy for microbiological control then returned to NIST and bottled.

Table 3. Methods and Analysts for Certified Elemental Determinations

Element	Method Code	Element	Method Code
Aluminum	ICP-AES INAA	Mercury	CVAAS RNAA
Antimony	INAA RNAA	Nickel	ID-ICPMS RNAA
Arsenic	FIA-HAAS RNAA	Nitrogen	KJEL PGAA
Boron	ID-ICPMS PGAA	Phosphorus	COLOR ICP-AES
Cadmium	ID-ICPMS PGAA RNAA	Potassium	INAA PGAA
Calcium	ID-TIMS INAA	Rubidium	ID-TIMS INAA
Chromium	INAA RNAA	Selenium	FIA-HAAS INAA RNAA
Cobalt	INAA RNAA	Sodium	FAES INAA
Copper	ICP-AES RNAA	Vanadium	ID-TIMS INAA
Iron	ICP-AES INAA	Zinc	ICP-AES INAA
Manganese	LEAFS INAA		

Methods:

COLOR	Colorimetry
CVAAS	Cold-vapor atomic absorption spectrometry
FAES	Flame atomic emission spectrometry
FIA-HAAS	Flow injection-hydride generation atomic absorption spectrometry
ICP-AES	Inductively-coupled plasma atomic emission spectrometry
ID-ICPMS	Isotope dilution, inductively coupled plasma mass spectrometry
ID-TIMS	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

NIST Analysts

E.S. Beary	K.E. Murphy
C.M. Beck II	P.J. Paulsen
D.A. Becker	T.A. Rush
D.S. Braverman	R. Saraswati
M.S. Epstein	J.M. Smeller
J.D. Fassett	G.C. Turk
K.M. Garrity	T.W. Vetter
R.R. Greenberg	R.D. Vocke
R.M. Lindstrom	R.L. Watter, Jr.
E. Mackey	L.J. Wood
J.R. Moody	

Cooperating Analysts

D.L. Anderson, Center for Food Safety and Applied Nutrition, U.S. FDA, Washington, DC
A.R. Byrne, Jozef Stefan Institute, Ljubljana, Slovenia
J. Kucera, Nuclear Research Institute, Rez, Czech Republic
B. Smodis, Jozef Stefan Institute, Ljubljana, Slovenia

REFERENCE

[1] Taylor, B.N., Guide for the use of the International System of Units (SI), NIST Special Publication 811, 1995 Ed., (April 1995).