National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1547

Peach Leaves

This Standard Reference Material (SRM) is intended primarily for use as an analytical control material and for evaluating the reliability of analytical methods for the determination of major, minor, and trace elements in botanical materials, agricultural food products, and similar matrices. A unit of SRM 1547 consists of 50 grams of dried peach leaves of the Coronet variety.

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 the mean of results from a method of known accuracy. Noncertified values, for information only, are provided in Table 2.

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. Please return the attached registration form to facilitate notification.

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 shaking 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 the material, dried as described in the section on "Instructions for Drying" should be used to relate analytical determinations to the certified values in 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, and correct the analytical results to a dry weight basis.

Dissolution of SRM 1547: Digestion procedures should be designed to avoid loss of volatile elements, such as arsenic, mercury, etc. 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 dissolved with a small amount of hydrofluoric acid to obtain total dissolution.

Coordination of the analyses for certification 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.

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

Gaithersburg, MD 20899
July 2, 1991

William P. Reed, 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 hours over fresh anhydrous magnesium perchlorate. The sample depth should not exceed one cm.

2. Freeze drying for 24 hours 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 one cm in depth) at -40 °C or lower for at least one hour. At the end of the 24-hour period, samples are placed immediately in a desiccator with fresh anhydrous magnesium perchlorate. Samples are weighed after allowing a minimum of four hours 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 not recommended.

Homogeneity Assessment: Samples from randomly selected bottles of SRM 1547 were tested for homogeneity by instrumental neutron activation analysis. No evidence of chemically significant inhomogeneity was observed (Ref. 1).

Table 1. Certified Concentrations of Constituent Elements.

Element	Concentration, wt. percent
Calcium	1.56 ±.02
Magnesium	0.432 ±.008
Nitrogen (Total)	2.94 ±.12
Potassium	2.43 ±.03

Element	Concentration, µg/g	Element	Concentration, µg/g
Aluminum	249 ± 8	Mercury	0.031 ± 0.007
Arsenic	0.060 ± 0.018	Molybdenum	0.060 ± 0.008
Barium	124 ± 4	Nickel	0.69 ± 0.09
Boron	29 ± 2	Selenium	0.120 ± 0.009
Chlorine	360 ± 19	Sodium	24 ± 2
Copper	3.7 ± 0.4	Strontium	53 ± 4
Lead	0.87 ± 0.03	Vanadium	0.37 ± 0.03
Manganese	98 ± 3	Zinc	17.9 ± 0.4

Certified Concentrations and Uncertainties: The certified concentrations are equally weighted means of results from two or more 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 Concentrations of Constituent Elements

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	Concentration wt. percent
*Nitrogen (Kjeldahl)	(2.96)
Sulfur	(0.2)

Element	Concentration, μg/g	Element	Concentration, μg/g
Antimony	(0.02)	Lanthanum	(9)
Bromine	(11)	Neodymium	(7)
Cadmium	(0.03)	Rubidium	(19)
Cerium	(10)	Samarium	(1)
Chromium	(1)	Scandium	(0.04)
Cobalt	(0.07)	Terbium	(0.1)
Europium	(0.17)	Thorium	(0.05)
Gadolinium	(1)	Tin	(<0.2)
Iodine	(0.3)	Uranium	(0.015)
Iron	(220)	Ytterbium	(0.2)

*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 R.A. Isaac, Soil Testing & Plant Analysis Laboratory, The University of Georgia College of Agriculture. Leaves, representative of healthy Georgia peach trees, variety "Coronet" were picked from a field in Peach County, Georgia, approximately 150 miles south of Athens, Georgia. Fungicide and insecticide sprays were controlled to minimize heavy metal contamination. The leaves were dried and ground in a stainless steel mill to pass a 1 mm screen. 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 were irradiated with cobalt-60 radiation to a minimum absorbed dose of 25.0 kGy for microbiological control and bottled.

Table 3. Methods and Analysts for Certified Elemental Determinations.

Element	Method Code	Analyst Code	Element	Method Code	Analyst Code
Aluminum	ICP	19	Mercury	CVAAS	13, 22
	INAA	3		RNAA	22
Arsenic	HGAAS	13	Molybdenum	IDICPMS	9, 11
	RNAA	6, 15		RNAA	6, 15
Barium	IDICPMS	9, 11	Nickel	LEIS	5, 7, 8, 16
	INAA	3		IDICPMS	2, 9, 11
				RNAA	22
Boron	IDICPMS	2, 11	Nitrogen	KJEL	14
	PGAA	21		PGAA	21
			Potassium	IDTIMS	4, 9, 10
	PGAA	21		INAA	3
Calcium	IDTIMS	4, 9, 10	Selenium	HGAAS	13
	INAA	3		INAA	3
				RNAA	6, 15
Chlorine	INAA	3	Sodium	FAES	13
	PGAA	21		INAA	3, 25
Copper	POL	12	Nitrogen	KJEL	14
	RNAA	6,15		PGAA	21
Lead	IDTIMS	1, 9, 18	Strontium	IDICPMS	9, 11
				INAA	23
Magnesium	IDTIMS	4, 9, 10	Vanadium	INAA	3
	INAA	3		RNAA	22
	FAAS	24			
Manganese	LEIS	5, 7, 8, 16	Zinc	POL	12
	INAA	3		INAA	3
Copper	POL	12	Nitrogen	KJEL	14
	RNAA	6, 15		PGAA	21

Methods Used for Analysis of SRM 1547:

CVAAS = Cold-Vapor Atomic Absorption Spectrometry
FAAS = Flame Atomic Absorption Spectrometry
FAES = Flame Atomic Emission Spectrometry
GFAAS = Graphite Furnace Atomic Absorption Spectrometry
HGAAS = 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
KJEL = Kjeldahl Nitrogen Determination
INAA = Instrumental Neutron Activation Analysis
LEIS = Laser-Enhanced Ionization Spectrometry
PGAA = Prompt Gamma Activation Analysis
POL = Polarography
RNAA = Radiochemical Neutron Activation Analysis

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References

1. Becker, D.A., Homogeneity and evaluation of the new NIST leaf certified reference materials, in nuclear analytical methods in the life sciences, R. Zeisler and V.P. Guinn, eds. Clifton, NJ: Humana Press, 1990, 571-577. [Proceedings of the International Conference, "Nuclear Analytical Methods in the Life Sciences", held at NIST, Gaithersburg, MD, April, 1989.]