



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1515

Apple 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 1515 consists of 50 grams of dried apple leaves of the Golden Delicious and Rome varieties.

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 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 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 1515: 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 this SRM and should be dissolved with a small amount of hydrofluoric acid to obtain total dissolution.

Coordination of the analyses 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

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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 1515 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.

<u>Element</u>	<u>Concentration, wt. percent</u>	
Calcium	1.526 ± 0.015	
Magnesium	0.271 ± 0.008	
Nitrogen (Total)	2.25 ± 0.19	
Potassium	1.61 ± 0.02	

<u>Element</u>	<u>Concentration,</u> <u>µg/g</u>	<u>Element</u>	<u>Concentration,</u> <u>µg/g</u>
Aluminum	286 ± 9	Mercury	0.044 ± 0.004
Arsenic	0.038 ± 0.007	Molybdenum	0.094 ± 0.013
Barium	49 ± 2	Nickel	0.91 ± 0.12
Boron	27 ± 2	Selenium	0.050 ± 0.009
Chlorine	579 ± 23	Sodium	24.4 ± 1.2
Copper	5.64 ± 0.24	Strontium	25 ± 2
Lead	0.470 ± 0.024	Vanadium	0.26 ± 0.03
Manganese	54 ± 3	Zinc	12.5 ± 0.3

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 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 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.

<u>Element</u>	<u>Concentration,</u> <u>wt. percent</u>
*Nitrogen (Kjeldahl)	(2.31)
Sulfur	(0.18)

<u>Element</u>	<u>Concentration,</u> <u>μg/g</u>	<u>Element</u>	<u>Concentration,</u> <u>μg/g</u>
Antimony	(0.013)	Neodymium	(17)
Bromine	(1.8)	Rubidium	(9)
Cadmium	(0.014)	Samarium	(3)
Cerium	(3)	Scandium	(0.03)
Chromium	(0.3)	Terbium	(0.4)
Cobalt	(0.09)	Thorium	(0.03)
Europium	(0.2)	Tin	(<0.2)
Gadolinium	(3)	Tungsten	(0.007)
Gold	(0.001)	Uranium	(0.006)
Iodine	(0.3)	Ytterbium	(0.3)
Lanthanum	(20)		
Iron	(80)		

*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, College of Agriculture, The Pennsylvania State University, University Park, PA. Mid-shoot leaves were collected from orchards adjacent to the Fruit Research Laboratory at Biglerville, PA and from orchards at the Horticultural Research Farm at Rock Spring, PA. Approximately equal amounts of leaves from Golden Delicious and Rome varieties were used. Fungicide and insecticide sprays were controlled to minimize heavy metal contamination. The leaves were dipped into a non-ionic detergent solution, rinsed in distilled water, dried in ovens at 60 °C, and ground to pass a 425 μm (40 mesh) screen in a stainless steel mill. 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.

<u>Element</u>	<u>Method Code</u>	<u>Analyst Code</u>	<u>Element</u>	<u>Method Code</u>	<u>Analyst Code</u>
Aluminum	ICP INAA	20 3	Mercury	CVAAS RNAA	14, 23 23
Arsenic	HAAS RNAA	14 6	Molybdenum	IDICPMS RNAA	9, 11 6, 23, 24
Barium	IDICPMS INAA	9,11 3	Nickel	LEIS IDICPMS RNAA	5, 7, 8, 17 2, 9, 11 23
Boron	IDICPMS PGAA	2, 11 22	Nitrogen	KJEL PGAA	15 22
Calcium	IDTIMS INAA	4, 9, 10 3, 26	Potassium	IDTIMS INAA	4, 9, 10 3
Chlorine	INAA PGAA	3 22	Selenium	HAAS INAA RNAA	14 3 6
Copper	POL RNAA	13 6, 16	Sodium	FAES INAA	14 3, 26
Lead	IDTIMS	1, 9, 19	Strontium	IDICPMS INAA	9, 11 24
Magnesium	IDTIMS INAA FAAS	4, 9, 10 3 25	Vanadium	INAA RNAA	3 23
Manganese	LEIS INAA XRF	5, 7, 8, 17 3 12, 15	Zinc	POL INAA	13 3

Methods Used for Analysis of SRM 1515:

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
 INAA = Instrumental Neutron Activation Analysis
 KJEL = Kjeldahl Nitrogen Determination
 LEIS = Laser-Enhanced Ionization Spectrometry
 PGAA = Prompt Gamma Activation Analysis
 POL = Polarography
 RNAA = Radiochemical Neutron Activation Analysis
 XRF = X-Ray Fluorescence Spectrometry

Analysts, National Institute of Standards and Technology

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|-------------------|-----------------|
| 1. I.L. Barnes | 12. P.A. Pella |
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Cooperating Analysts

22. D.L. Anderson, Center for Food Safety and Applied Nutrition, U.S. Food and Drug Administration, Washington, DC
23. A.R. Byrne, Jozef Stefan Institute, Ljubljana, Yugoslavia
24. J. Kucera, Nuclear Research Institute, Rez, Czechoslovakia
25. N. Miller-Ihli, Nutrient Composition Laboratory, U.S. Department of Agriculture, Beltsville, MD
26. B. Smodis, Jozef Stefan Institute, Ljubljana, Yugoslavia

Reference

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.]