National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1572

Citrus Leaves

This Standard Reference Material (SRM) is intended primarily for use in calibrating instrumentation and evaluating the reliability of analytical methods for the determination of major, minor, and trace elements in botanical materials, agricultural food products, and similar matrices.

Certified Values of Constituent Elements: The certified values for the constituent elements are shown in Table 1. They are based on results obtained either by definitive methods of known accuracy or by two or more independent analytical methods. Non-certified values, which are given for information only, appear in Table 2.

Notice and Warnings to Users:

Expiration of Certification: This certification is invalid 5 years after the shipping date. Should it be invalidated before then, purchasers will be notified by NBS.

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

Use: The bottle should be shaken well before each use. A minimum sample of 500 mg of the dried material (see Instructions for Drying) should be used for any analytical determination to be related to the certified values of this certificate.

Statistical consultation was provided by K. Kafadar of the Statistical Engineering Division.

The overall direction and coordination of the analyses leading to this certification were performed under the chairmanship of E.L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234
December 20, 1982
(Revision of Certificate
dated 2-22-82)

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

Additional Information on Analyses: Digestion procedures should be designed to avoid loss of volatile elements such as arsenic, mercury, etc. It was found that digestion of the orchard leaves in nitric and perchloric acids was incomplete, a small residue of siliceous material remaining. This residue must be considered an integral part of this Standard Reference Material. Therefore, dissolution procedures must be capable of complete dissolution of the leaves, but must not result in losses of volatile elements.

Iron and lead in the nitric-perchloric acid soluble portion were determined to be 270 μg/g and 44 μg/g, respectively. These two values, not to be confused with the *total* material values given in Table 1, are not certified, but given for information only.

Source and Preparation of Material: The orchard leaves for this Standard Reference Material were collected and prepared under the direction of A. L. Kenworthy of Michigan State University. These leaves were hand picked from an orchard near Lansing, Michigan, and air dried. The dried leaves were ground in a comminuting machine to pass a 40-mesh sieve (about one-third passing a 60-mesh sieve). After grinding the material, it was dried at 85° C and thoroughly mixed in a feed blender. The prepared leaves were packaged in polyethylene-lined fiber drums and sterilized *in situ* with 4.9 megarad of cobalt-60 radiation. The sterilization procedure was carried out at the U. S. Army Natick Laboratories under the direction of A. Brynjolfsson.

Homogeneity Assessment: The homogeneity of this material was established on the premise that the minimum sample size be 250 milligrams. Assessment of homogeneity was made using analyses for nitrogen, potassium, and magnesium. A statistical analysis of the data shows that there is evidence for a small degree of variability between samples with respect to potassium. The data for the other elements do not reveal such an effect. Statistical design and analysis of data were performed by J. Mandel of the NBS Institute for Materials Research.

Instructions for Drying: Before weighing, samples of this Standard Reference Material *must* be dried by either:

1. Drying in air in an oven at 85 °C for at least 4 hours.
2. Lyophilization using a cold trap at or below −50 °C at a pressure *not greater* than 30 Pa (0.2 mm Hg) for at least 24 hours.

NOTE: Drying at 135 °C results in large losses and discoloration and should *not* be used.

Analysts and Analytical Methods Used

Analytical Methods

A. Atomic absorption spectroscopy
B. Flame emission spectrometry
C. Gravimetry
D. Intersociety Committee Method 12204-01-68T for fluorine
E. Isotope dilution mass spectrometry
F. Isotope dilution spark source mass spectrometry
G. Kjeldahl method for nitrogen
H. Neutron activation
I. Nuclear track technique
J. Optical emission spectroscopy
K. Photon activation
L. Polarography
M. Spectrofluorimetry
N. Spectrophotometry

Analytical Methods Used and Analyses

Analytical Methods:

A. Atomic absorption spectrometry
B. Atomic emission spectrometry, flame
C. Atomic emission spectrometry, inductively coupled plasma
D. Ion chromatography
E. Isotope dilution thermal source mass spectrometry
F. Isotope dilution spark source mass spectrometry
G. Kjeldahl method for nitrogen
H. Neutron activation
I. Photon activation
J. Polarography
K. Spectrophotometry

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Table 1. Certified Values of Constituent Elements

Major and Minor Constituents

Element	Content,[1] (Wt. Percent)
Calcium	3.15 ± 0.10
Magnesium	0.58 ± 0.03
Phosphorus	0.13 ± 0.02
Potassium*	1.82 ± 0.06
Sulfur	0.407 ± 0.009

Trace Constituents

Element	Content,[1] μg/g	Element	Content,[1] μg/g
Aluminum	92 ± 15	Manganese	23 ± 2
Arsenic	3.1 ± 0.3	Mercury	0.08 ± 0.02
Barium	21 ± 3	Molybdenum	0.17 ± 0.09
Cadmium	0.03 ± 0.01	Nickel	0.6 ± 0.3
Chromium	0.8 ± 0.2	Rubidium*	4.84 ± 0.06
Copper	16.5 ± 1.0	Sodium	160 ± 20
Iodine	1.84 ± 0.03	Strontium*	100 ± 2
Iron	90 ± 10	Zinc	29 ± 2
Lead*	13.3 ± 2.4		

[1] Based on dry weight: For drying instructions, see the section of this certificate on Instructions for Drying. The uncertainties are based on judgment and represent an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples weighing 500 mg or more.

*For those elements determined by definitive methods, the uncertainties are given as 95%/95% statistical tolerance intervals. See The Role of Standard Reference Materials in Measurement Systems, NBS Monograph 148, 1975 p 14.

Table 2. Non-certified Values for Constituent Elements

NOTE: The following values are not certified because they are not based on the results of either a definitive method of known accuracy or two or more independent methods. These values are included for information only.

Major Constituent

Element	Content,[1] (Wt. Percent)
Nitrogen	(2.86)

Trace Constituents

Element	Content,[1] µg/g	Element	Content,[1] µg/g
Antimony	(0.04)	Samarium	(0.052)
Bromine	(8.2)	Scandium	(0.01)
Cerium	(0.28)	Selenium	(0.025)
Cesium	(0.098)	Tellurium[2]	(0.02)
Chlorine	(414)	Thallium	(≤0.01)
Cobalt	(0.02)	Tin	(0.24)
Europium	(0.01)	Uranium	(≤0.15)
Lanthanum	(0.19)		

[1]Analytical values are based on the "dry weight" of material (See Instructions for Drying).

[2]Not sufficiently homogeneous for certification.

Instructions for Drying: Samples of this SRM must be dried before weighing and analysis by either of the following procedures:

1. Drying for 2 hours in air in an oven at 85 °C.
2. Drying for 24 hours at 20 to 25 °C and at a pressure not greater than 30 Pa (0.2 mm Hg).

Additional Information on Analyses: This SRM contains siliceous material, which is an integral part of the sample. The values in Tables 1 and 2 are based on analyses performed on the *entire* sample. Therefore, dissolution procedures should be capable of complete dissolution of the sample but should not result in losses of volatile elements, such as arsenic and mercury.

Source and Preparation of Material: The plant material for this SRM was collected and prepared under the direction of A. L. Kenworthy, Michigan State University. Its source was the Lake Alfred area of central Florida. The material was air-dried, ground in a comminuting machine to pass a 425-µm (No. 40) sieve, dried at 85 °C, and thoroughly mixed in a feed blender. After packaging the material in polyethylene-lined fiber drums, it was sterilized in situ with cobalt-60 radiation. The sterilization procedure was carried out at the U.S. Army Research and Development Command, Natick, Mass. under the direction of A. Brynjolfsson.

Analysts

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