U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards Certificate of Analysis

Standard Reference Material 1571

Orchard Leaves

This Standard Reference Material is intended for use for the calibration of apparatus and the validation and/or verification of methods used in the analysis of agricultural and other botanical materials for major, minor, and trace elements.

Certified Values of Constituent Elements: The certified values for the constituent elements are shown in Table 1. The analytical techniques used and the names and affiliations of the analysts are shown in Table 3. Certified values are based on results obtained by reference methods of known accuracy and performed by two or more analysts; or alternatively, from results obtained by two or more independent, reliable analytical methods. Non-certified values are given for information only in Table 2. All values are based on a minimum sample size of 250 mg of the dried material.

Notice and Warnings to Users:

Expiration of Certification: This certification is invalid after 5 years from the date of shipping. Should it be shown invalid prior to that time, 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, including ultraviolet lamps or sunlight. Ideally, the bottle should be kept in a desiccator in the dark at the temperature indicated.

Use: A minimum sample of 250 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.

The overall direction and coordination of the technical measurements leading to this certificate were performed under the chairmanship of P. D. LaFleur. The overall coordination of the cooperative work performed by the Commission of European Communities, Joint Research Center, Ispra Establishment, Italy, was by G. Rossi of the Chemistry 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 T. W. Mears.

Washington, D.C. 20234
(January 28, 1971)
(Revised August 15, 1976)
(Revised August 31, 1977)

J. Paul Cali, 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 $\mu g/g$ and 44 $\mu g/g$, 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

Table 1. Certified Values of Constituent Elements[a]

Major Constituents		Minor Constituents	
Element	Content Wt. Percent	Element	Content Wt. Percent
Nitrogen	2.76 ± 0.05	Magnesium	0.62 ± 0.02
Calcium	2.09 ± 0.03	Phosphorus	0.21 ± 0.01
Potassium	1.47 ± 0.03		

The uncertainties shown above include both the imprecision, expressed as the standard deviation of a single measurement, and an allowance for unknown sources of systematic error.

Trace Constituents[a]

Element	Content μg/g	Element	Content μg/g
Iron	300 ± 20	Antimony	2.9 ± 0.3
Manganese	91 ± 4	Chromium	2.6 ± 0.3
Sodium	82 ± 6	Nickel	1.3 ± 0.2
Lead	45 ± 3	Molybdenum	0.3 ± 0.1
Strontium	37 ± 1	Mercury	0.155 ± 0.015
Boron	33 ± 3	Cadmium	0.11 ± 0.01
Zinc	25 ± 3	Selenium	0.08 ± 0.01
Copper	12 ± 1	Thorium	0.064 ± 0.006
Rubidium	12 ± 1	Uranium	0.029 ± 0.005
Arsenic	10 ± 2	Beryllium	0.027 ± 0.010

The uncertainties shown above are the imprecisions expressed as either two standard deviations of a single determination (commonly, but perhaps incorrectly, called the "95 percent confidence limit"), or the entire range of observed results - whichever of the two is larger. No additional allowance for the uncertainty from unknown sources of systematic error has been included, since these are considered to be small relative to the imprecision as expressed.

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

Table 2. Non-certified Values for Trace Constituent Elements[a]

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

Element	Content μg/g	Element	Content μg/g
Sulfur	(1900)	Cobalt	(0.2)
Chlorine	(690)	Iodine	(0.17)
Barium	(44)	Bismuth	(0.1)
Bromine	(10)	Gallium	(0.08)
Fluorine	(4.)	Cesium	(0.04)
Lithium	(0.6)	Tellurium	(0.01)

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

Table 3 Methods and Analysis*

METHOD / ELEMENT	A	B	C	D	E	F	G	H	I	J	K	L	M	N
Sb								19				25		
As	1,2							18,19,20, 22,31,34				25		27
Ba					9,11									
Be	1,3												28	
Bi												25		
B					10,15				5	32,33				
Br								19						
Cd	1,3							30				25		
Ca	1,3	1,3												
Cs								30						
Cl								19,30						
Cr					8,10			18						
Co								19						
Cu						14,16,17		19,20,29, 30		32				
F				7										
Ga								30						
I								23						
Fe	1,2					14,16,17		19,30,31				25		
Pb						14,16,17					24	25		
Li														
Mg	1,3,29							30		32				
Mn	1,3							18,20,30						
Hg	1,3					14,16,17		19,23,29, 30						
Mo					9,12			21,30						
Ni						14,16,17		30				25		
N							4,7		5					
P			4,7					30		32,33				
K	1,3	1,3						30						
Rb		1,3,29			9,11,12			30,31						
Se						14,16,17		23,30						
Na		1,3						20,30,31						
Sr					9,12									
S			5,6											
Te								23						
Th					10,12									
U					12,13			18	5					
Zn	1,3					14,16,17		18,29,30, 31		32,33				

*Numbers in body of table refer to analysts named above.