National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1575

Pine Needles

This Standard Reference Material (SRM) is intended primarily for evaluating the reliability of analytical methods for the determination of major, minor, and trace elements in botanical materials and other agricultural products. A unit consists of 70 grams of dried pine needles.

Certified Values of Constitutent Elements: The certified values for the constituent elements are shown in Table 1. They are based on results obtained either by reference methods of known accuracy or by two or more independent, reliable 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 will be invalid 5 years from date of shipment from NIST. Should it be invalidated before then, purchasers will be notified by NIST.

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: The bottled material should be mixed or rotated well before 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 on this certificate.

The original direction and coordination of the technical measurements leading to the certification were performed under the chairmanship of H.L. Rook. The overall coordination of the cooperative work performed by the Commission of European Communities, Joint Research Center, Ispra Estblishment, Italy, was by G. Rossi of the Chemistry Division.

This Certificate of Analysis has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certificate values or any technical data presented on this certificate.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by T.E. Gills.

Gaithersburg, MD 20899
February 24, 1993
(Revision of certificate dated 10-18-76)

William P. Reed, Chief
Standard Reference Materials Program

(over)

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

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

Note: Drying either in an oven at 105 °C or in a vacuum oven at 75 °C causes large losses of volatiles other than water and should *not* be used.

Additional Information Analyses: This SRM contains siliceous material which is an integral part of the sample. The analyses reported in Tables 1 and 2 were 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 of Michigan State University, East Lansing, MI. Its source was Maistee State Park, approximately 65 km north of Muskegon, MI. For the preparation of the SRM, the material was air-dried, and ground in a comminuting machine. After grinding, the material, was dried at 85 °C, thoroughly mixed in a feed blender, packaged in polyethylene-lined fiber drums, and sterilized in-situ with cobalt-60 radiation. The sterilization procedure was carried out at the U.S. Army Research and Development Command, Natick, MA under the direction of A. Brynjolfsson. At NIST, preliminary evaluation of the material homogeneity indicated that its improvement would be required to establish reliable certified values. Therefore, the material was resieved and the portion that passed a polypropylene sieve having openings of 0.25 mm (equivalent to a U.S. series 60 standard sieve) was retained for the SRM.

Homogeneity Assessment: Material homogeneity was evaluated by determining ten certified elements, P, Al, Fe, Mn, Rb, Cu, Cr, As, Hg, and U on samples of 500 mg or less taken at various locations within freeze-dried bulk material. The other certified elements, K, Ca, Sr, Pb, and Th were determined using sample weights not exceeding one gram. The uncertainties for the concentrations given in Table 1 include these results.

Table 1. Certified Values of Constituent Elements[a]

Major and Minor Constituents

Element	Content Wt. Percent
Calcium	0.41 ± 0.02
Potassium	0.37 ± 0.02
Phosphorus	0.12 ± 0.02

Trace Constituents

Element	Content µg/g			Element	Content µg/g		
Manganese	675	±	15	Copper	3.0	±	0.3
Aluminum	545	±	30	Chromium	2.6	±	0.2
Iron	200	±	10	Arsenic	0.21	±	0.04
Rubidium	11.7	±	0.1	Mercury	0.15	±	0.05
Lead	10.8	±	0.5	Thorium	0.037	±	0.003
Strontium	4.8	±	0.2	Uranium	0.020	±	0.004

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

The uncertainties of the values of the constituents shown in Table 1 include allowances for material inhomogeneity, method imprecision, and an estimate of possible biases of the analytical methods used.

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

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

Major Constituent

Element	Content Wt. Percent
Nitrogen	(1.2)

Trace Constituents

Element	Content µg/g	Element	Content µg/g
Bromine	(9)	Lanthanum	(0.2)
Nickel	(3.5)	Cobalt	(0.1)
Cerium	(0.4)	Thallium	(0.05)
Cadmium[b]	(<0.5)	Scandium	(0.03)
Antimony	(0.2)	Europium	(0.006)

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

[b] Cadmium was not sufficiently homogeneous for certification.

Analytical Methods

a. Atomic absorption spectroscopy
b. Isotope dilution mass spectrometry
c. Isotope dilution spark source mass spectrometry
d. Kjeldahl method for nitrogen
e. Neutron activation
f. Nuclear track technique
g. Optical emission spectroscopy
h. Spectrophotometry
i. Polarography

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