National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 1575a

Trace Elements in Pine Needles

(*Pinus Taeda*)

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of techniques employed in the analysis of pine needles and materials of a similar matrix. A unit of SRM 1575a consists of approximately 50 g of dried, jet-milled, radiation sterilized, and blended pine needles.

Certified Values: The certified concentrations for twelve elements, expressed as mass fractions [1] on a dry basis, are provided in Table 1. The certified value for mercury is based on results from a single NIST primary method, cold vapor isotope dilution inductively coupled plasma mass spectrometry, and was confirmed by radiochemical neutron activation analysis at NIST. Certified values for other elements are based on results from two or more critically evaluated independent analytical techniques. Analyses were performed at NIST and at United States Geological Survey (USGS), Denver, CO. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [2].

Reference Values: The reference values for eleven constituents, expressed as mass fractions on a dry basis, are provided in Table 2. The reference values are based on results obtained from a single NIST analytical method. Reference values are noncertified values that are estimates of the true value. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty [2].

Information Values: Information values for two elements are provided in Table 3. An information value is a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2].

Expiration of Certification: The certification of **SRM 1575a** is valid, within the measurement uncertainties specified, until **01 August 2022** provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). This certification is nullified if the SRM is contaminated, contaminated or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to certification was performed by E.A. Mackey of the NIST Analytical Chemistry Division. A complete list of analysts is given in Table 5.

Statistical analyses were provided by H-k. Liu and J. Lu of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

The protocol used for obtaining the pine needles was developed by D.A. Becker of the NIST Analytical Chemistry Division. The needles were collected by members of the Forest Nutrition Cooperative of North Carolina State University.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 13 June 2012
Certificate Revision History is on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

Preparation of SRM 1575a was performed by C.N. Fales of the NIST Standard Reference Materials Group.

INSTRUCTIONS FOR USE

Sampling: The SRM should be thoroughly mixed by repeatedly inverting and rotating the bottle horizontally before sampling. A minimum sample mass of 250 mg should be used for analytical determinations to be related to elemental concentration values provided. The SRM should be stored in its original, tightly sealed bottle away from sunlight and intense sources of radiation.

Drying: In order to relate measurements to the certified and reference values that are expressed on a dry mass basis, users should determine a drying correction at the time of each analysis by desiccator drying for five days over fresh magnesium perchlorate or equivalent. Freeze-drying for six days at 1 Pa with a condenser temperature of 50 °C, initial shelf temperature of –10 °C and final shelf temperature of 5 °C provided equivalent results. The average mass loss measured at NIST using these two methods for SRM 1575a was 2.9 % (1 s = 0.2 %, n = 14). No significant difference between these two methods was observed. The amount of moisture in this material may vary depending on storage and environmental conditions.

SOURCE, PREPARATION, AND ANALYSIS

The pine needles for this SRM were collected from loblolly pine trees (*Pinus taeda*) in North Carolina from freshly felled trees of approximately the same age and origin. The needles were dried at 70 °C for 48 h, coarse ground to pass through a 2 mm sieve and shipped to NIST where the material was jet-milled to pass a 100 µm sieve, blended, radiation sterilized, and bottled. Measurements confirmed that the jet-milling process resulted in a powder with particle sizes equivalent to spheres ranging in diameter from approximately 1 µm to 100 µm.

Chromium was found to be inhomogenously distributed in this material (for a sample size of 250 mg). Analysis of a total of 58 portions yielded chromium mass fraction values ranging from approximately 0.3 mg/kg to 0.5 mg/kg.

Analyses of this material used for certification were performed at NIST and at USGS. The analytical techniques used for each element are listed in Table 4 and the analysts are listed in Table 5.

Table 1. Certified Mass Fraction Values (Dry Basis) in SRM 1575a

Minor Constituents

Elements	Mass Fraction (%)
Phosphorus	0.107 ± 0.008
Potassium	0.417 ± 0.007
Calcium	0.25 ± 0.01

Trace Elements

Elements	Mass Fraction (mg/kg)	Elements	Mass Fraction (mg/kg)
Aluminum	580 ± 30	Copper	2.8 ± 0.2
Barium	6.0 ± 0.2	Iron	46 ± 2
Cadmium	0.233 ± 0.004	Mercury	0.0399 ± 0.0007
Chlorine	421 ± 7	Rubidium	16.5 ± 0.9
		Zinc	38 ± 2

The certified values are the average values from two or more analytical methods and the uncertainty values represent expanded uncertainties, which include components of uncertainty from each method with a Type B distribution for between method uncertainty, combined according to the method described in reference 3 in compliance with the ISO Guide [4].

Table 2. Reference Mass Fraction Values (Dry Basis) for SRM 1575a

Minor Constituent

Element	Mass Fraction (%)
Magnesium	0.106 ± 0.017

Trace Elements

Elements	Mass Fraction (mg/kg)	Elements	Mass Fraction (mg/kg)
Arsenic	0.039 ± 0.002	Manganese	488 ± 12
Boron	9.6 ± 0.2	Nickel	1.47 ± 0.10
Cesium	0.283 ± 0.009	Scandium	0.0101 ± 0.0003
Cobalt	0.061 ± 0.002	Selenium	0.099 ± 0.004
Lead	0.167 ± 0.015	Sodium	63 ± 1

Reference values are based on results of one analytical method at NIST and the uncertainty values represent the expanded uncertainties, which include the combined Type A, and Type B with a coverage factor, combined according to the method described in reference 5 in compliance with the ISO Guide [4].

Table 3. Information Mass Fraction Values (Dry Basis) for SRM 1575a

Elements	Mass Fraction (mg/kg)
Cerium	0.11
Chromium (inhomogeneous)	0.3 - 0.5

Table 4. Methods of Analysis for SRM 1575a

Element	Method
Aluminum	INAA, ICP-AES [USGS]
Arsenic	INAA
Barium	INAA, ICP-MS
Boron	PGAA
Cadmium	ICP-MS, RNAA (and confirmed by ICP-AES[USGS], ICP-MS [USGS])
Calcium	INAA, ICP-MS [USGS], ICP-AES [USGS]
Cerium	INAA
Cesium	INAA
Chlorine	PGAA, INAA
Chromium	INAA, ICP-MS [USGS], ICP-AES [USGS]
Cobalt	INAA
Copper	RNAA, ICP-AES[USGS]
Iron	INAA, ICP-AES[USGS]
Lead	ICP-MS
Magnesium	INAA (and confirmed by ICP-AES[USGS])
Manganese	INAA
Mercury	CV-ID-ICP-MS (and confirmed by RNAA)
Nickel	ICP-MS
Phosphorus	RNAA, ICP-AES[USGS]
Potassium	INAA, PGAA (and confirmed by ICP-AES[USGS])
Rubidium	ICP-MS[USGS], INAA
Scandium	INAA
Selenium	INAA
Sodium	INAA
Zinc	INAA, ICP-AES [USGS]

Methods:

ICP-AES [USGS]	Inductively coupled plasma atomic emission spectrometry at USGS
ICP-MS[USGS]	Inductively coupled plasma mass spectrometry at USGS
INAA[USGS]	Instrumental neutron activation analysis at USGS
CVAAS[USGS]	Cold vapor atomic absorption spectrometry at USGS
ICP-MS	Inductively coupled plasma mass spectrometry at NIST
CV ID-ICP-MS	Isotope dilution, cold vapor, inductively coupled plasma mass spectrometry at NIST
INAA	Instrumental neutron activation analysis at NIST
PGAA	Prompt gamma-ray activation analysis at NIST
RNAA	Radiochemical neutron activation analysis at NIST

Table 5. Analysts for SRM 1575a

NIST Analytical Chemistry Division

W.R. Kelly	R.D. Oflaz
R.M. Lindstrom	B.J. Porter
S.E. Long	R.L. Paul
E.A. Mackey	L.J. Wood
J.L. Mann	L.L. Yu

USGS, Denver, CO

S.A. Wilson	P.H. Briggs
Z.A. Brown	J. Budahn

REFERENCES

[1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; 1995 Ed. (1995); available at http://www.nist.gov/pml/pubs/index.cfm/ (accessed June 2012).

[2] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G..; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260–136; U.S. Government Printing Office: Washington, DC (2000); available at http://www.nist.gov/srm/publications.cfm (accessed June 2012).

[3] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.-k.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; J. Res. NIST 105, pp. 571–549 (2000).

[4] G*uide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed June 2012).

[5] Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed June 2012).

Certificate Revision History: 13 June 2012 (Extension of certification period; editorial changes); 17 September 2002 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.