National Institute of Standards and Technology
U.S. Department of Commerce

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Standard Reference Material® 1575a
Trace Elements in Pine Needles (*Pinus taeda*)
CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for use in the evaluation of techniques employed in the analysis of pine needles and materials of a similar matrix.

Description: A unit of SRM 1575a consists of approximately 50 g of dried, jet milled, radiation sterilized, and blended pine needles.

Certified Values: These values are traceable to the International System of Units (SI) [1]. The certified values for twelve elements, expressed as mass fractions [2] on a dry mass, 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 the United States Geological Survey (USGS) (Denver, CO).

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

Minor Constituents

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

Trace Elements

Elements	Mass Fraction (mg/kg)	Elements	Mass Fraction (mg/kg)
Aluminum (Al)	580 ± 30	Copper (Cu)	2.8 ± 0.2
Barium (Ba)	6.0 ± 0.2	Iron (Fe)	46 ± 2
Cadmium (Cd)	0.233 ± 0.004	Mercury (Hg)	0.0399 ± 0.0007
Chlorine (Cl)	421 ± 7	Rubidium (Rb)	16.5 ± 0.9
		Zinc (Zn)	38 ± 2

[(a)] The certified values are either the result of measurements from a single NIST primary method or the average values from two or more analytical methods. The uncertainty values represent expanded uncertainties, which include components of uncertainty from each method and for two or more analytical methods, a Type B distribution for between method uncertainty, combined according to the method described in reference 3 in compliance with the JCGM Guide [4]. The certified values are the measurands and are metrologically traceable to the SI derived unit of mass fraction expressed as percent (%) or as milligrams per kilogram (mg/kg). Analytical methods of analysis are listed in Table A2.

Non-Certified Values: Non-certified values are provided in Appendix A.

Additional Information: Additional information is provided in Appendix B.

Period of Validity: The certified values delivered by **SRM 1575a** are valid within the measurement uncertainty specified until **01 August 2032**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

Certificate Revision History on Page 2

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Storage and Use: SRM 1575a should be stored at temperatures between 10 to 30 ℃ in its original, tightly sealed bottle, away from sunlight and intense sources of radiation. SRM 1575a 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 mass fraction values provided.

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. At NIST, 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.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication (NIST SP) 260-136, 2021 edition; U.S. Government Printing Office: Washington, DC (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed May 2022).

[2] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008) available at https://www.nist.gov/pml/special-publication-811 (accessed May 2022).

[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.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, pp. 571–579 (2000).

[4] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at https://www.bipm.org/en/publications/guides (accessed May 2022); 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed May 2022).

If you use this SRM in published work, please reference:

Mackey E, Becker D, Oflaz R, Greenberg R, Lindstrom R, Yu L, Wood L, Long S. , Kelly W, Mann J, MacDonald B, Wilson S, Brown Z, Briggs P, Budhan J, Paul R (2004) Certification of NIST Standard Reference Material 1575a Pine Needles and Results of an International Laboratory Comparison, (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-156. https://www.nist.gov/publications/certification-nist-standard-reference-material-1575a-pine-needles-and-results

Certificate Revision History: 04 May 2022 (Change of period of validity; updated format; storage temperature added; editorial changes); 19 July 2017 (Updated title; editorial changes); 13 June 2012 (Extension of certification period; editorial changes); 17 September 2002 (Original certificate date).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control, but should not be used for calibrating or validating measurement procedures. Non-certified mass fraction values, expressed as mass fractions on a dry-mass basis, are provided in Table A1. The values are based on results obtained from a single NIST analytical method.

Table A1. Non-Certified Mass Fraction Values[(a)] (Dry-Mass Basis) for SRM 1575a

Minor Constituent

Elements	Mass Fraction (%)
Magnesium (Mg)	0.106 ± 0.017

Trace Elements

Elements	Mass Fraction (mg/kg)	Elements	Mass Fraction (mg/kg)
Arsenic (As)	0.039 ± 0.002	Manganese (Mn)	488 ± 12
Boron (B)	9.6 ± 0.2	Nickel (Ni)	1.47 ± 0.10
Cesium (Cs)	0.283 ± 0.009	Scandium (Sc)	0.0101 ± 0.0003
Cobalt (Co)	0.061 ± 0.002	Selenium (Se)	0.099 ± 0.004
Lead (Pb)	0.167 ± 0.015	Sodium (Na)	63 ± 1

[(a)] Non-Certified values in Table A1 are based on results of one analytical method at NIST. The uncertainty values represent expanded uncertainties and include Type A and Type B components combined according to the method described in the JCGM Guide [4]. However, NIST has not fully investigated all known or suspected sources of bias. As such, the provided uncertainties may not include all significant sources of uncertainty. The measurands are the mass fractions of the elements listed in Table A1 as determined by the methods in Table A2.

Additional Values of Interest to the User: 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. Cerium (Ce) was found to have a mass fraction value of 0.11 mg/kg, but insufficient information is available to provide an uncertainty associated with the value.

Period of Validity: The non-certified values delivered by **SRM 1575a** are valid within the measurement uncertainty specified until the same expiration date given in the certificate on page 1. The value assignments are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Reference Material Information Sheet and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Table A2. 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	Cold vapor isotope dilution 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

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APPENDIX B

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.

Coordination of the technical measurements leading to certification was performed by E.A. Mackey of the NIST Chemical Sciences Division. A complete list of analysts is given in Table B1.

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 Office of Reference Materials.

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

Table B1. Analysts for SRM 1575a

NIST Chemical Sciences 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

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

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