



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 values for twelve elements, expressed as mass fractions [1] 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 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-mass 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]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1575a** is valid, within the measurement uncertainty 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.

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 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 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.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 19 July 2017
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

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 inhomogeneously 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^(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 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 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).

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

| Minor Constituents | | | | | | |
|--------------------|-------------------|---|-------|--|--|--|
| 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) 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 the JCGM Guide [4]. The measurands are the concentrations of the elements listed in Table 2 as determined by the methods in Table 4. Metrological traceability is the SI derived units for mass fraction, expressed as percent or milligrams per kilogram.

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

| Elements | Mass Fraction (mg/kg) |
|-------------------------------|-----------------------|
| Cerium (Ce) | 0.11 |
| Chromium (Cr) (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 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

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

REFERENCES

- [1] 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/physical-measurement-laboratory/special-publication-811> (accessed July 2017).
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- [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 (JCGM) (2008); available at http://www.bipm.org/utls/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed July 2017); 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 July 2017).

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| Certificate Revision History: 19 July 2017 (Updated title; editorial changes); 13 June 2012 (Extension of certification period; editorial changes); 17 September 2002 (Original certificate date). |
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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 at: telephone (301) 975-2200; fax (301) 948-3730, email srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.