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ABSTRACT

NIST recently released SRM 1575a Pine Needles to replace the original SRM 1575 Pine Needles, which was issued in 1976 and is now out of stock. This material is intended primarily for use in the evaluation of inorganic analytical techniques used to determine element content of botanical and agricultural materials that have a matrix similar to pine needles. The needles were collected in North Carolina from freshly felled loblolly pine (*Pinus taeda*) trees of about the same age. The needles were dried, ground, jet-milled, blended, sterilized, and bottled. Elemental analyses of this material were performed at NIST using five analytical techniques and at the U.S. Geological Survey in Denver, CO using three techniques. Selected data were used to provide certified mass fraction values for twelve elements, reference values for eleven elements, and information values for two elements. This material was also used as a test material for an international interlaborartory comparison exercise for the determination of elemental composition sponsored by the ASTM Task Group on Nuclear Methods of Chemical Analysis. A complete description of the material collection and preparation, the results of analyses, the methods used to assign certified, reference, and information values, and results of the interlaboratory comparison exercise are presented and discussed in this report.

Key Words: certified value, interlaboratory comparison, pine needles, reference material, round robin, Standard Reference Material (SRM), trace elements

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INTRODUCTION

In September of 2002, the National Institute of Standards and Technology (NIST) released Standard Reference Material (SRM) 1575a Pine Needles. This material was provided to replace NIST SRM 1575 Pine Needles, which is now out of stock. This renewal material is one of five NIST agricultural certified reference materials that are intended for use in the evaluation of analytical methods for the determination of element content of botanical materials. Analysis of SRM 1575a Pine Needles presents a different analytical challenge as compared with the other agricultural reference materials due to differences in both matrix and levels of some elements. For example, mass fractions of B, Ca, K, and Fe are significantly lower than in the other botanical SRMs whereas the mass fraction of Mn is significantly higher.

The material was collected, dried, and ground by the Forest Nutrition Cooperative of North Carolina State University and shipped to NIST for further processing and bottling. Determination of the elemental composition of the material was accomplished through a collaboration of NIST with the U.S. Geological Survey (USGS) Denver, CO. Scientists at NIST used inductively coupled plasma mass spectrometry (ICP-MS), cold vapor isotope dilution mass spectrometry (CV-IDMS), instrumental neutron activation analysis (INAA), prompt gamma-ray activation analysis (PGAA), and three different radiochemical neutron activation analysis (RNAA) procedures for the determination of 26 elements. Scientists at the USGS used INAA, ICP-MS, and inductively coupled plasma atomic emission spectrometry (ICP-AES) for the determination of 40 elements. Certified, reference, and information values were assigned according to the criteria described in detail by May et al. (2000). Complete descriptions of the collection, preparation, material analyses, data analysis, and certification of SRM 1575a Pine Needles are presented in this report.

Prior to release of SRM 1575a Pine Needles, this material was used as the test material for an international interlaboratory comparison exercise for the determination of element content. This round robin was conducted under the auspices of the ASTM Task Group on Nuclear Methods of Chemical Analysis. Each participant received one 6-g portion of SRM 1575a and one 6-g portion of SRM 1547 Peach Leaves for use as a control material. Participants were instructed to report mass fraction values based on the dry mass of the material and were instructed to determine the dry mass by desiccator drying of separate portions over fresh magnesium perchlorate. The two SRMs were shipped to the participants in March of 2002 and participants were asked to send results by the end of August (2002). Fifteen labs from eight countries participated in the study for the determination of element content and one additional laboratory provided results from determinations of two radioisotopes. The results from these laboratories and complete discussion of the study data also are presented in this report.

MATERIAL COLLECTION AND PREPRATION

The pine needles used to prepare SRM 1575a were collected, dried, and ground by members of the Forest Nutrition Cooperative of North Carolina State University. Approximately 70 kg of pine needles were collected in North Carolina from freshly felled loblolly pine (*Pinus taeda*) trees of about the same age. The pine needles were coarse-ground to pass through a 2-mm sieve and dried for 48 h at 70 °C prior to shipment to NIST. At NIST, the material was jet milled to pass a 100-µm sieve and blended over the course of two weeks. The material was radiation sterilized by exposure to ^{60}Co (2.5 Mrad) for 5 h. After irradiation, the material was apportioned into amber bottles containing approximately 50 g each. The material collected and processed filled about 1300 bottles. Bottles were randomly selected for certification analyses performed by NIST and USGS scientists. Each analyst was provided with six to twelve bottles of SRM 1575a. The bottles used for each analytical method are listed in Table 1. A total of 30 bottles were used for analyses performed by 14 analysts: ten analysts from NIST and four from the USGS Denver, CO.

Table 1. Bottle Numbers Used by Each for Certification Analyses.

Analyst Group	Technique (Elements)	Bottle Numbers
NIST	INAA (Na, K, Mn, Cl, Al, Mg, Ca)	29, 227, 260, 308, 522, 739, 803, 953, 986,1027, 1142, 1333
	INAA (As, Ba, Ce, Co, Cr, Cs, Fe, Rb, Sb, Sc, Se, Th, Zn)	66, 227, 308, 522, 606, 739, 953, 1142, 1333
	RNAA (Cu, Cd)	66, 227, 308, 739, 803, 986, 1027, 1142, 1333
	RNAA (P)	66, 522, 606, 739, 803, 865, 953, 986, 1027, 1224
	RNAA (Hg)	29, 66, 308, 522, 606, 1142, 1224
	PGAA (B, Cl, K)	66, 227, 308, 522, 739, 953, 986, 1027
	ICP-MS (Ba, Cd, Ni, Pb)	83, 189, 356, 472, 708, 1107
	CV-IDMS (Hg)	83, 189, 356, 472, 708, 1107
	Freeze-drying (moisture)	66, 227, 308, 522, 739, 953
USGS	ICP-AES	491, 619, 766, 824, 901, 1149, 1191, 1255
	ICP-MS	491, 619, 766, 824, 901, 1149, 1191, 1255

Prior to analysis, each analyst determined the moisture content of the material using separate portions from several different bottles. Two methods were used at NIST to dry portions removed from previously unopened bottles of SRM 1575a:

1.) desiccator drying over fresh $MgClO_4$ for 120 h,
2.) freeze-drying at a condenser temperature of -50 °C and a pressure of 1 Pa using a shelf temperature gradient beginning at -10 °C and increasing in 5 °C increments to a maximum of +5 °C over the course of one week.

A summary of the results of drying studies performed at NIST on those previously unopened bottles of SRM 1575a is shown in Table 2. For each method the number of portions (n) is listed together with the average relative mass lost (± 1 standard deviation, 1s). Results of these two drying methods were similar and indicated the moisture content of this material shortly after bottling was approximately 2.84% ± 0.17% (the average value ± 1s, for seventeen portions).

The value for moisture content will change with time, depending upon storage and local laboratory conditions. Pelletized portions of SRM 1575a stored for nine months in sealed polyethylene bags placed in plastic Petri dishes gained about 2.9% mass due to absorption of moisture. Other analysts using portions of SRM 1575a removed from previously opened bottles found that the moisture content of SRM 1575a had increased from approximately 2.8% (average for five portions with 1s of 0.1%) to 3.05% (average for 8 portions with 1s of 0.01%) and others found a change from 3.0% (average for six portions with 1s = 0.2%) to 3.6% (average for six portions with 1s = 0.2%). See Table 2. It is recommended that analysts determine the moisture content on separate individual portions from each bottle, for each use.

Table 2. Results of Initial Drying Study on Bottles of SRM 1575a

Technique	**n**	**Moisture Content of Unopened Bottles**
Freeze-Drying	6	2.76% ± 0.04%
Desiccator Drying ($MgClO_4$)	5	2.77% ± 0.11%
Desiccator Drying ($MgClO_4$)	6	2.98% ± 0.21%
	17	*2.84% ± 0.17% Average*
Technique	**n**	**Moisture Content of Previously Opened Bottles**
Desiccator Drying ($CaSO_4$)	8	3.05% ± 0.01% (previously opened bottles)
Desiccator Drying ($MgClO_4$)	6	3.6% ± 0.2% (previously opened bottles)

EXPERIMENTAL PROCEDURES

Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) at NIST

In preparation for ICP-MS, one 0.5-g portion from each of six bottles of SRM 1575a Pine Needles and two 0.5-g portions of SRM 1547 Peach Leaves were weighed into individual microwave cells. Then, 5 mL of HNO_3 and 1 mL of HF were added to each cell. Samples were digested in a microwave oven using an eight-step program in which the power was increased from 250 W to 550 W over the course of 40 min. The temperature in the cells ranged from 80 °C up to but not exceeding 90 °C. Each digest was quantitatively transferred to a pre-weighed polyethylene bottle and diluted to a total mass of 50 g after the addition of an internal standard consisting of 80 µg of Rh. From each diluted digest, 25 g were transferred to another polyethylene bottle. One gram of a spike solution containing 0.991 µg/g Ni, 0.247 µg/g Cd, 0.832 µg/g Ba, and 0.486 µg/g Pb was added to each.

Prior to the quantitative determination of the elements of interest, an ICP-MS semi-quantitative analysis was performed on un-spiked solutions of digested SRM 1575a and SRM 1547. These results were used to assess the presence or absence of interferences, and to determine the optimum concentrations for the spike. Molecular $^{44}Ca^{16}O$ was found to interfere with the measurement of ^{60}Ni, the isotope chosen for the quantification of Ni. A solution of 100 µg/g Ca was analyzed with the samples and the ratio of the intensities at masses 60 and 43 was calculated. This ratio was used to correct the measured intensity at mass 60 to obtain the intensity from ^{60}Ni. Similarly, $^{95}Mo^{16}O$ interfered with the determination of ^{111}Cd, the isotope used for quantification of Cd. A solution containing 50 µg/g Mo was analyzed with the samples and the ratio of the intensities of masses 111 and 97 was calculated. This ratio was used to correct the measured intensity at 111, to account for this interference in determining the mass fraction of Cd. No spectral interferences were found for the isotopes used to quantify Ba or Pb. Blanks were included in the analysis scheme and element mass fraction values were corrected for any amounts present in the blanks. Blank corrections were required for Ni, Ba, Cd, and Pb.

Cold Vapor-Isotope Dilution Mass Spectrometry (CV-IDMS) at NIST

Analysis of Hg was performed by CV-IDMS of samples prepared using a Carius tube sample digestion. This sample decomposition method has been described in detail elsewhere (Long, et al., 2002) and a brief description is included here. Approximately 0.25 g of sample from each bottle was weighed into a cleaned Carius tube and spiked with a weighed aliquot of ^{201}Hg followed by the addition of 5 mL of high-purity HNO_3. Each vessel was flame-sealed, placed in a steel cylinder along with 20 g of solid CO_2 for external pressurization, and heated in a convection oven at 240 °C for 12 h. After cooling to room temperature, each vessel was depressurized using a high-temperature flame, the contents transferred to polypropylene centrifuge tubes, and diluted to a concentration of approximately 0.25 ng/g Hg. The tubes were stored at 4 °C overnight to allow degassing of NO_2 and CO_2. Analyses were performed within 24 h using cold vapor Hg generation followed by ICP-MS isotope ratio measurements.

This CV-IDMS method has been described in detail elsewhere (Christopher, et al., 2001) and a brief description is included here. The Hg vapor was generated using a 10% (mass/volume) $SnCl_2$ in 7% (volume fraction) HCl reductant, and separated from the liquid phase using a commercially available reaction separator cell. The vapor was transferred to an ICP-MS system using 100 mL/min of $Ar_{(g)}$. The gas stream was mixed with the plasma injector gas stream using a plastic T piece. The ICP was operated in a dry plasma mode. The ^{201}Hg and ^{202}Hg isotopes were monitored in a pulse counting, Time-Resolved-Analysis mode to recover the individual ion count rates. Background corrected ratios were calculated from the isotope-time profiles. Instrument mass discrimination was measured by generation of Hg vapor from an isotopic calibration standard. The mass discrimination factor was close to unity during the measurement period

owing to the high mass of the isotopes and the lens settings used. The ^{201}Hg isotopic spike solution was calibrated by reverse isotope dilution using a high-purity primary standard, SRM 1641d Mercury in Water. Two separate stock solutions were prepared by serial dilution of the standard. Results of CV ICP-MS showed that the spike contained 0.5702 nmol/g Hg (± 0.095%, 1s). Results for samples were corrected for
42 pg of Hg in the procedural blank.

Instrumental Neutron Activation Analysis (INAA) at NIST

In preparation for INAA, one portion weighing between 200 mg and 250 mg from each bottle of SRM 1575a was formed into a disk-shaped pellet using a commercially available stainless steel die and hydraulic press. Each disk was doubly encapsulated in bags formed from acid-washed polyethylene film. Element standards consisted of filter papers onto which solutions containing a known amount of the analyte of interest had been deposited and dried. Two portions each of SRM 1547 Peach Leaves and SRM 1575 Pine Needles were included in the analysis for the purpose of quality control. These quality control SRMs were prepared and packaged in the same manner as the portions of SRM 1575a.

For analysis of short-lived products of neutron irradiation, one sample or control SRM or element standard was irradiated individually for 60 s in the NIST reactor using neutron irradiation tube facility RT-4, which exposed the sample, or sample and standard, to a thermal neutron fluence rate of 3.5×10^{13} $cm^{-2} \cdot s^{-1}$. After irradiation, the sample, control, or standard was removed from the irradiation container and polyethylene bag and repackaged in clean (un-irradiated) polyethylene film. Gamma spectrometry was performed on each using a system that consisted of a germanium detector, 16K channel, fixed conversion time, analog-to-digital converter linked to a multichannel analyzer. Gamma radiations were collected for 5 min, after a decay time of 5 min, at a distance of 20 cm from the end of the detector for the determination of Al, Ca, and Mg. Using the same detector and geometry, gamma radiations were collected again for 5 min, after 15 min of decay, for the determination of Cl, and for a third time for 1 h, after 1 h to 5 h of decay for the determination of Na, K, and Mn. Data reduction was accomplished using commercially available software to determine peak areas and to calculate the activity at the end of irradiation. Element mass fraction values were calculated based on comparison with standards.

For determination of long-lived products of neutron irradiation, separate 200-mg portions of SRM 1575a and control SRMs were prepared and packaged in the same manner. Element standards consisted of either pure metal foils of known mass or filter papers onto which solutions containing known amounts of the elements of interest had been deposited. All SRMs and standards were placed in one of two polyethylene irradiation containers. Iron foils were included in the top and bottom of the irradiation vessel to monitor any differences in neutron fluence over the length of the container. Each container was subjected to a neutron fluence rate of 3.5×10^{13} $cm^{-2} \cdot s^{-1}$ for 3 h. Halfway through the neutron irradiation, each container was inverted end-over-end and then reinserted into the reactor. This procedure serves to minimize differences in neutron exposure among the samples and standards due to a linear drop off of the neutron fluence rate with distance from the reactor core. All irradiated samples and standards were allowed to decay for 4 d to 5 d to eliminate or decrease the activity from short-lived isotopes. Then, each portion was removed from the irradiation vessel and from the irradiated polyethylene bags and placed in another polyethylene bag for gamma spectroscopy. Gamma-ray spectroscopy was performed using a germanium detector (40% efficiency relative to a standard-sized NaI crystal) and associated electronics. For the analysis of As, samples, standards, and controls were counted individually on the germanium detection system for 2 h to 8 h. After additional decay times of two to three weeks, each individual sample, control, or standard was counted for a minimum of 8 h and as long as 24 h for the analysis of Ba, Ce, Co, Cr, Cs, Fe, Rb, Sb, Sc, Se, Th, and Zn. Quantification was based on comparison with elemental standards. Count rates for the Fe fluence-rate monitors, corrected for decay time and mass differences, agreed within 0.5%, indicating that the two containers were exposed to the same neutron irradiation dose.

Prompt Gamma-Ray Activation Analysis (PGAA) at NIST

For PGAA, one portion weighing between 740 mg and 760 mg was removed from each of eight bottles. Each portion was formed into a disk using a commercially available stainless steel die and hydraulic press, and each disk was packaged in a bag formed from Teflon film. Two portions of SRM 1547 were prepared in the same manner and included for the purpose of quality control. Standards consisted of filter papers onto which solutions containing known amounts of the elements of interest had been deposited. The filter papers were formed into disks and packaged in bags formed from Teflon film so that the geometry of the samples and standards was identical. Each disk of the SRM was simultaneously irradiated and counted for 8 h to 24 h, and standards for 0.5 h to 1 h, in the new thermal neutron PGAA instrument at vertical beam tube VT-5. A Ti foil was irradiated before and after each sample to monitor any fluctuations in the neutron fluence rate of VT-5. Over the two-week interval required for this analysis, results of the Ti foil monitor irradiations showed that any variations in neutron fluence rate were ≤0.9%. Quantification was based on comparison with elemental standards. All data were corrected for the effects of the pile up of pulses at higher count rates. Results for B were corrected for the presence of this element in the background which was equivalent to 0.7 µg of B. The H content of the standards and the samples were nearly identical so that no corrections for neutron scattering were required.

Radiochemical Neutron Activation Analysis (RNAA) for Cu and Cd at NIST

Sample, control, and standard preparation were the same for RNAA as described for INAA at NIST. Two portions of SRM 1547 Peach Leaves and two portions of SRM 1570a Trace Elements in Spinach Leaves were included as control materials. The portions of all SRMs and element standards were placed in one of two polyethylene irradiation containers or "rabbits". Iron foils were included in the top and bottom of each rabbit to monitor any differences in neutron fluence over the length of the rabbit and between rabbits. Each rabbit was subjected to a neutron fluence rate of 3.5×10^{13} $cm^{-2} \cdot s^{-1}$ for 2 h. Halfway through the neutron irradiation, each rabbit was inverted end-over-end and then reinserted into the reactor to minimize differences in neutron exposure among the samples and standards.

This RNAA method has been described in detail elsewhere (Greenberg, 1986) and a brief description is included here. After a decay time of 52 h to 54 h, each portion was removed from the irradiation container and from the polyethylene bags, and placed in a Teflon beaker containing 10 mL of concentrated HNO_3, 10 mL of H_2O, and 0.1 mL of a carrier solution. The carrier solution contained 5 mg/mL Cu and 5 mg/mL Cd. Each Teflon beaker was placed on a hot plate with a surface temperature of 150 °C to 200 °C until the volumes were reduced to approximately 0.5 mL. An additional 10 mL of HNO_3 and 10 mL of $HClO_4$ were then added to each beaker and the beakers were covered with Teflon lids. After 1 h, the lids were removed and the surface temperature of the hot plate increased to approximately 180 °C to 220 °C. Beakers were removed from the hot plate when the volumes were reduced to approximately 0.5 mL. Again, 10 mL of HNO_3 and 10 mL of $HClO_4$ were added to each beaker. The beakers were covered and placed on a hot plate with a surface temperature of about 120 °C overnight. The following morning, the lids were removed and the surface temperature of the hot plate was increased to a temperature between 180 °C and 220 °C. After approximately 1 h, 1 mL of HF was added to each sample or standard. When volumes were reduced to 0.5 mL the beakers were removed from the hot plate.

Radiochemical separations consisted of adding 10 mL of 1 mol/L HNO_3 to each 0.5 mL digest and adjusting the pH to a value between 1.5 and 1.7. The diluted digest was added to a separatory funnel. The Cu and Cd were extracted with one 25 mL portion followed by one 5 mL portion of a solution containing 2 g/L of zinc diethyldithiocarbamate [$Zn(DDC)_2$] in chloroform. The $Zn(DDC)_2$ layer was drained into a second separatory funnel containing 25 mL of 2.5 mol/L HCl. This funnel was shaken for 30 s to back extract the Cd into the aqueous layer. The lower organic layer containing Cu was drained into a high-density polyethylene (HDPE) bottle and the upper aqueous layer containing Cd was transferred to another HDPE bottle. Gamma-ray spectroscopy was performed on each separated fraction using a germanium detector and associated electronics. For the assay of Cu, gamma radiations were collected for 0.5 h to 2 h. For the assay of Cd, gamma radiation was collected for 2 h to 8 h after allowing 24 h for the ^{115}Cd (half-life, $t_{1/2}$ = 53.4 h) and ^{115}In ($t_{1/2}$ = 4.49 h) isotopes to reach equilibrium. Quantification was based on comparison with standards. Count rates for the Fe fluence-rate monitors, corrected for decay time and mass differences, agreed within 2%, indicating that the two containers experienced the same neutron dose.

Radiochemical Neutron Activation Analysis (RNAA) for P at NIST

Standards were prepared by depositing solutions containing known amounts of P onto 40 mg pieces of aluminum foil, which were dried by heating under an infrared lamp. These were packaged in bags formed from clean polyethylene film. Sample and control SRMs were prepared and packaged as described for INAA. Samples, controls, and standards were subjected to a neutron fluence rate of 2.7×10^{13} $cm^{-2} \cdot s^{-1}$ for 5 min and were allowed to decay for approximately one week prior to radiochemical separation. Zinc foils were included in each irradiation container to monitor any differences in the neutron exposure within a container and among different containers.

Samples or controls were placed in beakers containing 7.02 mg of P (non-radioactive carrier), 10 mL concentrated HNO_3, and 10 mL H_2O, covered, and heated for about 16 h and then allowed to evaporate to near dryness. This procedure was followed by heating in covered beakers with 10 mL of HNO_3, 10 mL of $HClO_4$, and 10 drops of HF for 16 h, followed by evaporation to near dryness. Standards were subjected to a similar digestion procedure that also included the addition of 7 mL of HCl in the initial step. The residue from each was dissolved in 10 mL of HNO_3 and 30 mL of H_2O, and the standards were filtered to remove any insoluble material. To each sample, control or standard, 5 g of NH_4NO_3 and 20 mL of a solution containing10% (mass/volume) NH_4MoO_3 were added to precipitate $(NH_4)_3PO_4 \cdot 12MoO_3$. The precipitates were collected, washed twice with 15 mL of H_2O, and then dissolved in 10 mL of NH_4OH. To each were added 20 mL of H_2O, 10 mL of 20% (mass/volume) NH_4Cl and 15 mL of a magnesia reagent to precipitate $MgNH_4PO_4$ overnight. The precipitate was collected, washed, dried, and transferred to a planchet. Each sample, standard and control was counted using a β^- proportional counter. After counting, the precipitate was transferred to a crucible and heated at 650 °C for at least 1 h to convert the compound to $Mg_2P_2O_7$. The yields were determined gravimetrically from the masses of $MgNH_4PO_4$ and again from the masses of $Mg_2P_2O_7$.

Radiochemical purity was determined by both gamma-ray screening and by β^- analysis of activity as a function of time over the course of four half-lives. Count rates were corrected for the effects of self-absorption of β^- particles in the precipitate. Mass fractions were determined using decay-corrected count rates based on comparison with standards.

Radiochemical Neutron Activation Analysis (RNAA) for Hg at NIST

Portions of SRM 1575a weighing from 160 mg to 250 mg were removed from the bottles and flame-sealed in quartz vials. Control materials were prepared in the same manner. Standards were prepared by depositing solutions containing known amounts of Hg onto cysteine-impregnated filter paper. The filter papers were dried at room temperature and encapsulated in quartz vials. The standards, samples, and controls were exposed to a neutron flux of 7.7×10^{13} $cm^{-2} \cdot s^{-1}$ for 6 h.

After irradiation, the short-lived activities in the samples were allowed to decay before they were processed radiochemically. Each quartz vial was rinsed sequentially in HNO_3 and water, frozen in liquid nitrogen, wrapped in tissue, and crushed. In a quartz combustion tube, the package was burned in an unglazed porcelain boat with about 50 mg of HgO carrier. The sample and tissue were slowly ignited with a hand torch at an airflow of 100 mL/min and the oxidation completed in an oxygen flow of 100 mL/min for 10 min in a 700 °C furnace. The products of combustion were trapped at liquid nitrogen temperature, rinsed into a polyethylene bottle with 5 mL of HNO_3 and diluted to 50 mL for preliminary gamma assay. After adding 1 mg of Se holdback carrier, the solution was evaporated, taken up in 100 mL of hot 0.15 mol/L HNO_3, and mercury periodate precipitated with 1.0 g of $Na_3H_2IO_6$. After digestion, the precipitate was collected, washed, and mounted in a Petri dish for gamma spectrometry. Yields were determined based on the mass of dried $Hg_5(IO_6)_2$, and mass fractions of Hg were determined based on comparison with standards.

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) at the USGS

This procedure has been described in detail in U.S. Geological Survey Open File Report 02-223-I and a brief summary is included here. Samples and control materials (including SRM 1547 Peach Leaves) were decomposed using a mixture of HCl, HNO_3, $HClO_4$, and HF at low temperatures, brought to dryness, and then dissolved using 1 mL of $HClO_4$ and two drops of H_2O_2. Each digest was diluted with 19 mL of 1% (volume fraction) HNO_3, heated for 30 min, and further diluted 1:10 with 1% (volume fraction) HNO_3. Internal standards containing known amounts of Li, Rh, and Ir in 1% HNO_3 were added to each sample solution prior to analysis. A dual detector calibration and auto-lens adjustment were performed according to manufacturer's recommendations. Two multi-element solutions were used for calibration.

Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) at USGS

This procedure has been described in detail in U.S. Geological Survey Open File Report 02-223-G and a brief summary is included here. One 200-mg portion of each sample and control material (SRM 1547 Peach Leaves) was added to a Teflon vessel together with an internal standard consisting of 50 µg Lu in HCl. Samples and control materials were decomposed using a mixture of HCl, HNO_3, $HClO_4$, and HF at low temperatures and evaporated to dryness. The dried samples were dissolved in 1 mL of *aqua regia* and 9 mL of 1% (volume fraction) HNO_3. The ICP-AES instrument was calibrated prior to sample analysis using several single and multi-element standard solutions. The digested samples were aspirated into the ICP-AES discharge where the elemental emission signals were measured simultaneously for all elements determined. Method blanks were included, and blank subtractions performed where necessary. Inter-element correction factors and background corrections were applied using commercially available software.

RESULTS AND DISCUSSION

Quality Assurance

All analysts from NIST and the USGS included portions of either SRM 1575 Pine Needles or SRM 1547 Peach Leaves or both in the analysis scheme for the purpose of quality assurance. Portions of SRM 1570a were included for analyses of Cu and Cd at NIST. Results from these analyses agreed with the certified values within the associated uncertainties for all materials for all elements analyzed at NIST and for most elements analyzed at USGS. In a few cases where data did not agree with certified values for a given element, data for that element were not used to assign values for SRM 1575a Pine Needles. A summary of the results from analyses of SRMs 1575 and 1547 are shown in Table 3.

Analytical Results for SRM 1575a Pine Needles

Analyses were performed at NIST using INAA, RNAA, PGAA, ICP-MS, and CVIDMS. The results from analyses of individual portions expressed on a dry mass basis are listed in Appendix A. Analyses were performed at USGS Denver using ICP-MS, ICP-AES, and INAA. Selected data from USGS, obtained using methods independent of those used at NIST (i.e., methods other than those used at NIST) were used to calculate certificate values. Results from USGS from analyses of individual portions for data used to calculate certificate values are included in Appendix A. Additional data from USGS were used to confirm NIST values but are not included here. Where two NIST methods were available only those data were used to calculate the certificate values for SRM 1575a. The average values (± 1s) from each technique and each laboratory that were combined to provide certified values are listed in Table 4.

Material homogeneity was assessed based on results from INAA of 10 to 24 200 mg portions analyzed at NIST. These results indicated that SRM 1575a is not homogenous with respect to Cr content for this sample size. The range of values obtained from analyses of individual portions was 0.3 mg/kg to 0.5 mg/kg. Results from analyses performed at USGS were similar. No other elements were found to be inhomogeneous at this sample size.

CERTIFICATION AND DATA ANALYSIS

Certificate values for SRM 1575a are designated as certified, reference, or information according to the modes defined by May et al. (2000) in NIST SP 260-136. The modes used to provide certified, reference, and information values for this material are described briefly here. The certified value for Hg is based on results from one primary method (CV-IDMS). Results from RNAA confirm this value but were not included in the calculation to determine the certified value. All other certified values are based on results from two or more critically evaluated independent analytical techniques. 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 accounted for or investigated. Reference values for this material are based on results obtained from a single NIST analytical method, either INAA or ICPMS. Reference values are non-certified values that are the best estimate of the true values but do not meet NIST criteria for certification. Reference values are provided with associated uncertainties that may not include all sources of uncertainty. Information values were provided for two elements, Cr and Ce. These are noncertified values with no uncertainty assessed. The information value for Ce is based on results from one NIST method (INAA). Results from several different techniques indicated that Cr is distributed inhomogenously when measured in several 200 mg portions. For this reason, a Cr mass fraction range is given for information purposes and this range is based on results from both NIST and USGS.

Data from two or more techniques were combined to provide the certified values using the method described by Levenson et al. (2000). This method assumes a type B distribution of any bias between the methods. The formulae defined by Levenson et al. that were used to combine these results into a certified mass fraction value (X) and expanded uncertainty (U) are shown here:

1. $X = 1/2(x_1 + x_2)$ for two values or $1/3(x_1 + x_2 + x_3)$ for three values
2. $U = k[u(B)^2 + u(X)^2]^{0.5}$
3. $u(X) = [u(x_1)^2/4 + u(x_2)^2/4\,]^{0.5}$
4. $u(B) = |x_1 - x_2|/(2(3)^{0.5})$

In those formulae, *u(B)* is the standard uncertainty of the combined value assuming a rectangular distribution, and *u(X)* is the combination of the individual within-method uncertainties of the two methods. These combined mass fraction and uncertainty values are listed as certified values in the last column of Table 4. A copy of the Certificate of Analysis for SRM 1575a Pine Needles is included as Appendix B.

Table 3. Results from Analyses of SRMs 1575 Pine Needles and 1547 Peach Leaves Included with Analyses of SRM 1575a. Methods are listed in Table 4.

	SRM 1575			**SRM 1547**		
Element (units)	**Method 1**	**Method 2**	**Certificate Values**	**Method 1**	**Method 2**	**Certificate Value**
Al (µg/g)	603 ± 20	590	545 ± 30	250 ± 9	270	249 ± 8
As (µg/g)	0.20 ± 0.02		0.21 ± 0.04	0.065 ± 0.014		0.060 ± 0.018
B (µg/g)	not analyzed			27.8 ± 1.3		29 ± 2
Ba (µg/g)	7.4 ± 0.1	not analyzed		128 ± 10	127 ± 4	124 ± 4
Cd (µg/g)	See 1570a below	not analyzed	<0.5 (non-certified)	0.032 ± 0.012	0.026 ± 0.003	0.026 ± 0.003
Ca (%)	0.41 ± 0.02	0.413	0.41% ± 0.02%	1.57 ± 0.07	1.63	1.56% ± 0.02%
Ce (µg/g)	0.27		0.4 (non-certified)	11.3 ± 0.5		10 (non-certified)
Cl (µg/g)	not analyzed	293 ± 31		354 ± 11	349 ± 29	360 ± 19
Co (µg/g)	0.109 ± 0.003		0.1 (non-certified)	0.071 ± 0.002		0.07 (non-certified)
Cr (µg/g)	2.52 ± 0.15	2.84	2.6 ± 0.2	1.33 ± 0.09	0.55	1 (non-certified)
Cs (µg/g)	0.118 ± 0.004			0.079 ± 0.003		
Cu (µg/g)	See 1570a below	2.8	3.0 ± 0.3	3.72 ± 0.20	3.6	3.7 ± 0.4
Fe (µg/g)	186 ± 6	190	200 ± 10	224 ± 6	220	218 ± 14
Hg (µg/g)	0.1415		0.15 ± 0.05	0.0336		0.031 ± 0.007
K (%)	not analyzed	0.37 ± 0.04	0.37% ± 0.02%	2.49 ± 0.07	2.54 ± 0.11	2.43% ± 0.03%
Mg (%)	0.122 ± 0.029			0.442 ± 0.016		0.432% ± 0.008%
Mn (µg/g)	692 ± 21		675 ± 15	99 ± 4		98 ± 3
Na (µg/g)	40.1 ± 3.0			25.7 ± 10		24 ± 2
Ni (µg/g)	not analyzed		3.5 (non-certified)	0.73 ± 0.09		0.69 ± 0.09
P (%)	0.110 ± 0.004	0.117	0.12% ± 0.02%	0.132 ± 0.002	0.147	0.137% ± 0.007%
Pb (µg/g)	not analyzed		10.8 ± 0.5	0.87 ± 0.03		0.87 ± 0.03
Rb (µg/g)	11.7 ± 0.3	11.4	11.7 ± 0.1	20.0 ± 0.6	19.8	19.7 ± 1.2
Sc (µg/g)	0.040 ± 0.001		0.03 (non-certified)	0.044 ± 0.001		0.04 (non-certified)
Se (µg/g)	0.061 ± 0.027			0.117 ± 0.004		0.120 ± 0.009
Zn (µg/g)	65 ± 10	60.3		18.3 ± 1.3	18.7	17.9 ± 0.4
	SRM 1570a					
Element (units)	**Method 1**		**Certificate Values**			
Cu (µg/g)	12.5 ± 0.6		12.2 ± 0.6			
Cd (µg/g)	2.86 ± 0.26		2.89 ± 0.07			

Table 4. Average Mass Fraction Values Used to Provide Certificate Values for SRM 1575a Pine Needles.

Element	Method 1	Method/Lab	Method 2	Method/Lab	Method 3	Method/Lab	Certified Value
Al (µg/g)	575 ± 16	INAA/NIST	589 ± 29	ICPAES/USGS			580 ± 30
Ba (µg/g)	5.96 ± 0.45	INAA/NIST	6.06 ± 0.43	ICPMS/NIST			6.0 ± 0.2
Cd (µg/g)	0.233 ± 0.009	RNAA/NIST	0.234 ± 0.003	ICPMS/NIST			0.233 ± 0.004
Ca (%)	0.248 ± 0.007	INAA/NIST	0.250 ± 0.013	ICPMS/USGS	0.258 ± 0.012	ICPAES/USGS	0.25 ± 0.02
Cl (µg/g)	417 ± 3	PGAA/NIST	425 ± 8	INAA/NIST			421 ± 7
Cu (µg/g)	2.89 ± 0.03	RNAA/NIST	2.70 ± 0.17	ICPAES/USGS			2.8 ± 0.2
Fe (µg/g)	45.9 ± 0.5	INAA/NIST	46.0 ± 2.3	ICPAES/USGS			46 ± 2
K (%)	0.4137 ± 0.0037	PGAA/NIST	0.4209 ± 0.0128	INAA/NIST			0.417 ± 0.007
Hg (µg/g)	0.0399 ± 0.0004	CVIDMS/NIST	0.0414 ± 0.0028	RNAA/NIST			0.0399 ± 0.0007
P (%)	0.102 ± 0.002	RNAA/NIST	0.112 ± 0.005	ICPAES/USGS			0.107 ± 0.008
Rb (µg/g)	16.55 ± 0.14	INAA/NIST	16.36 ± 0.81	ICPMS/USGS			16.5 ± 0.9
Zn (µg/g)	38.0 ± 0.2	INAA/NIST	37.7 ± 1.8	ICPAES/USGS			38 ± 2
							Reference Value
As (µg/g)	38.9 ± 1.2	INAA/NIST					0.039 ± 0.002
B (µg/g)	9.6 ± 0.2	PGAA/NIST					
Cs (µg/g)	283 ± 3	INAA/NIST					0.283 ± 0.009
Co (µg/g)	61.4 ± 0.8	INAA/NIST					0.061 ± 0.002
Pb (µg/g)	0.167 ± 0.013	ICPMS/NIST					0.167 ± 0.015
Mg (%)	0.106 ± 0.007	INAA/NIST					0.106 ± 0.017
Mn (µg/g)	488 ± 6	INAA/NIST					488 ± 12
Ni (µg/g)	1.47 ± 0.10	ICPMS/NIST					1.47 ± 0.10
Sc (µg/g)	0.0101 ± 0.0002	INAA/NIST					0.0101 ± 0.0003
Se (µg/g)	0.099 ± 0.004	INAA/NIST					0.099 ± 0.004
Na (µg/g)	63 ± 2	INAA/NIST					63 ± 1
							Information Value
Cr (µg/g)	0.375 ± 0.065	INAA/NIST	0.32 ± 0.02	ICPAES/USGS	0.39 ± 0.03	ICPMS/USGS	0.3 - 0.5
Ce (µg/g)	0.114 ± 0.010	INAA/NIST					0.11

INTERLABORATORY COMPARISON METHODS

The ASTM Task Group for Nuclear Methods of Chemical Analysis has sponsored interlaboratory comparison exercises for the last 20 y to provide laboratories with the opportunity to assess their analytical capabilities. Fifteen laboratories participated in this comparison exercise for analysis of inorganic constituents of SRM 1575a Pine Needles and one additional laboratory submitted values for the isotopes ^{232}Th and ^{238}U. All participants were provided one bottle containing about 6 g of SRM 1575a Pine Needles and another containing about 6 g of SRM 1547 Peach Leaves for use as a control material. The instructions included a request to the laboratories to analyze a minimum of three portions weighing at least 200 mg. Analysts were also instructed to determine the material dry mass using three separate portions not used for elemental analysis and to report the results of analysis on a dry-mass basis. The list of participants is included in Appendix C.

When data were received, each lab was assigned (randomly) a number between 1 and 24 for identification purposes. Labs that provided values from more than one analytical technique were given alphabetical additions to the number codes, e.g., Laboratory 13a, 13b, and 13c, or Laboratory 15a and 15b. These data were not combined into a single value from that one laboratory, but rather were treated as if they were from separate laboratories. Most participants used INAA to determine element content but a few laboratories provided additional information using other techniques (e.g., RNAA, flame atomic absorption spectrometry [AAS], or graphite furnace AAS) and one lab used ASTM methods. The analytical methods used by each laboratory for each element are listed in Table 5 together with the laboratory number code.

RESULTS OF THE ASTM INTERLABORATORY COMPARISON

The results from each laboratory are listed in Appendix D identified only by laboratory number code and results for each element are listed in Appendix E. All data from the study participants were compiled and average and standard deviation values were calculated where possible. Values from individual laboratories were then compared with certificate values where available and with the average value from all participants where no certificate value was available. These comparisons were done in the form of z-scores that were calculated according to the following equation:

5. $z = (x_n - x_{ref})/(sx_{ref})$

where x_n is the value for the participating laboratory, 10% was selected as the relative standard deviation s, and x_{ref} is either the certificate value or the ASTM average (where no certificate value was available). Outlier rejection was based on z-score values >+3 or <-3, indicating that the reported value differed from the reference value by greater than 30%. After any outliers were identified, the ASTM average was recalculated and the z-scores were re-evaluated so that the final values listed reflect this iterative approach. Results that were rejected based on this criteria have been shaded in the tables in Appendices D and E. Average and z-score values were not calculated in cases where no certificate value was available *and* the number of values was ≤ 3. Average and z-scores were not calculated for either Hf or Au because the range of values was too great, and therefore agreement too poor, to permit meaningful data analysis. Where no statistical analyses were performed, cells in the tables have been filled with diagonal hatch marks.

Table 5. Methods Used by Participants in the ASTM Interlaboratory Comparison Exercise for Determination of Element Content of SRM 1575a Pine Needles

Lab No.	Elements	Methods
2	^{232}Th, ^{238}U	Radioisotope Analysis
3	Al, Sb, As, Ba, Br, Ca, Cd, Ce, Cs, Cl, Cr, Co, Cu, F, Au, Hf, Fe, La, Mg, Mn, Hg, K, Rb, Sm, Sc, Se, Na, Sr, Th, Zn	INAA
4	Br, Co, Eu, Fe, La, Rb, Sm, Sc, Se, Zn	INAA
6	Sb, As, Ba, Br, Ca, Ce, Cs, Cl, Cr, Co, Fe, La, Mg, Mn, K, Rb, Sm, Sc, Na, Th, Zn	INAA
8	Al, Sb, As, Ba, Br, Ca, Cs, Cl, Cr, Co, Eu, Au, Fe, La, Mg, Mn, Ni, K, Rb, Sm, Sc, Se, Sr, Ta, Th, U, V, Zn	INAA
10	Co, Mo, Rb, Sc, U, Zn	INAA
13a	As, Ba, Br, Cs, Cr, Co, Au, Hf, Fe, La, Mn, P, K, Rb, Sc, Na, Th, Zn	INAA-1 RNAA (P only)
13b	Sb, Ba, Br, Ce, Cs, Cr, Co, Hf, Fe, La, K, Rb, Sm, Sc, Se, Th, Yb, Zn	INAA-2
13c	Ca, Fe, Mg, Mn, K, Na, Zn	AAS
15a	Br, Mn, K, Na	INAA
15b	Al, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Ag, Na, Zn	FAAS/GFAAS
16	Sb, Ba, Br, Ca, Cd, Ce, Cs, Cr, Co, Eu, Au, Hf, Fe, La, Lu, Hg, Ni, K, Rb, Sm, Sc, Se, Na, Sr, Ta, Tb, Th, Tb, Zn	INAA
17	Al, Sb, As, Br, Ca, Cs, Cl, Cr, Co, Fe, Mg, Mn, K, Rb, Sc, Na, Zn	INAA
18	Br, Ca, Ce, Co, Fe, La, Nd, K, Rb, Sm, Sc, Na, Tb, Zn	INAA
19	As, Ba, Cs, Cr, Co, Fe, La, K, Rb, Sc, Na, Zn	INAA
20	Sb, As, Ba, Br, Ca, Ce, Cs, Cr, Co, Eu, Au, Hf, Fe, La, Lu, Hg, K, Rb, Sm, Sc, Se, Na, Sr, Ta, Tb, Th, Yb, Zn	INAA

21	Br, Ca, Cs, Co, Fe, K, Rb, Sc, Na, Zn	INAA
23	Ca, Cu, Fe, Mg, Mn, Total Kjeldahl N, P, K, S, Zn	E-6010 ASTM#1402-07 (TKN, P) ASTM# 1552-90 (S)
24	Al, Sb, As, Br, Ca, Cl, La, Mg, Mn, Mo, Ni, K, Sm, Na	INAA

A summary of the interlaboratory comparison data is presented in Table 6 together with the certified values. Note that the number of values for each element differed greatly, ranging from 16 values to just one value. Because outlier rejection was based on comparison with the certificate values, the interlaboratory averages calculated after rejection of outliers generally agree better with the certificate values. In all cases, standard deviation values were reduced by outlier rejection. Participants also provided mass fraction values or limits of detection for 23 elements (Au, Br, Eu, F, Ga, Hf, I, In, Ir, La, Mo, total Kjeldahl N, S, Sb, Sm, Sr, Ta, Tb, Th, W, U, and Yb) that were not included on the certificate of analysis for SRM 1575a. A summary of these results is shown in Table 7. The number of values for these elements is generally smaller but ranged from 13 values for Br to a single value for the following eight elements: F, Ga, In, I, Ir, Total Kjeldahl N, S, W. The average values before and after outlier rejection were calculated except in cases where there was only one value, or in cases where agreement among the values was too poor to permit meaningful data analysis.

***Nota Bene*: Results of analyses from this ASTM interlaboratory comparison exercise are included in this report to provide a complete description of the study results. Any values in this report that are not included on the Certificate of Analysis have not been evaluated for accuracy and should not be used as certificate values.**

Table 6. Results of the ASTM Interlaboratory Comparison Exercise for Determination of Element Content of SRM 1575a Pine Needles Compared with Certificate Mass Fraction Values.

Element (unit)	*Certificate Mass Fraction Value*	*Interlaboratory Average ± 1s*	*n*	*Interlaboratory (after outlier rejection) Average ± 1s*	*n*
Aluminum (µg/g)	580 ± 30	583 ± 128	5	533 ± 69	4
Barium (µg/g)	6.0 ± 0.2	6.2 ± 1.9	8	5.4 ± 0.4	5
Cadmium (µg/g)	0.233 ± 0.004	0.208 ± 0.178	3	0.252	1
Calcium (%)	0.25 ± 0.02	0.253 ± 0.072	12	0.24 ± 0.02	10
Chlorine (µg/g)	421 ± 7	399 ± 22	5	399 ± 22	5
Copper (µg/g)	2.8 ± 0.2	2.9 ± 1.0	3	3.0	1
Iron (µg/g)	46 ± 2	56 ± 42	14	46 ± 3	12
Potassium (%)	0.417 ± 0.007	0.421 ± 0.529	13	0.407 ± 0.016	12
Mercury (µg/g)	0.0399 ± 0.0007	0.024 ± 0.018	3	0.0341 ± 0.0071	2
Phosphorus (%)	0.107 ± 0.008	0.118 ± 0.008	2	0.118 ± 0.008	2
Rubidium (µg/g)	16.5 ± 0.9	15.1 ± 4.8	12	16.5 ± 0.7	11
Zinc (µg/g)	38 ± 2	37 ± 5	14	36 ± 4	13
Element (unit)	*Reference Mass Fraction Value*	*Interlaboratory Average ± 1s*	*n*	*Interlaboratory (after outlier rejection) Average ± 1s*	*n*
Arsenic (µg/g)	0.039 ± 0.002	0.048 ± 0.023	8	0.040 ± 0.005	7
Cesium (µg/g)	0.283 ± 0.009	0.276 ± 0.029	10	0.284 ± 0.012	9
Cobalt (µg/g)	0.061 ± 0.002	0.101 ± 0.132	12	0.063 ± 0.007	11
Lead (µg/g)	0.167 ± 0.015	0.115 ± 0.031	1	no values	0
Magnesium (%)	0.106 ± 0.017	0.100 ± 0.018	8	0.105 ± 0.010	7
Manganese (µg/g)	488 ± 12	458 ± 31	8	458 ± 31	8
Nickel (µg/g)	1.47 ± 0.10	1.21 ± 0.27	4	1.31 ± 0.22	3
Scandium (µg/g)	0.0101 ± 0.0003	0.018 ± 0.025	12	0.0104 ± 0.0015	11
Selenium (µg/g)	0.099 ± 0.004	0.093 ± 0.044	6	0.118 ± 0.015	4
Sodium (µg/g)	63 ± 1	71 ± 20	11	63 ± 5	9
Element (unit)	*Information Values*	*Interlaboratory Average ± 1s*	*n*	*Interlaboratory (after rejection of selected data) Average ± 1s*	*n*
Chromium (µg/g)	0.3 - 0.5	0.502 ± 0.507	10	0.334 ± 0.039	8
Cerium (µg/g)	0.11	0.208 ± 0.261	6	0.10 ± 0.02	5

Table 7. Results of the ASTM Interlaboratory Comparison Exercise for Determination of Element Content of SRM 1575a Pine Needles: elements not included on the Certificate of Analysis.

Element (units)	Range	Interlaboratory Average ± 1s or single value	n*	Range (after outlier rejection)	Interlaboratory (after outlier rejection) Average ± 1s	n
Antimony (mg/kg)	0.0062 - 0.033	0.015 ± 0.009	8	0.0062 - 0.0099	0.009 ± 0.001	4
Bromine(mg/kg)	2.10 - 3.91	2.95 ± 0.42	13	2.10 - 3.38	2.87 ± 0.32	12
Europium(mg/kg)	<0.002 - 0.0017 (n = 5)	0.0014 ± 0.0003	3			
Fluorine(mg/kg)		389	1			
Gallium(mg/kg)		<1	1			
Gold (mg/kg)	0.00026- 0.0026		5			
Hafnium(mg/kg)	<0.015 - 0.12		5			
Indium(mg/kg)		<0.003	1			
Iodine(mg/kg)		<1.5	1			
Iridium(mg/kg)		<0.0001	1			
Lanthanum(mg/kg)	0.042 - 0.39	0.101 ± 0.106	10	0.042 - 0.066	0.053 ± 0.008	8
Molybdenum (mg/kg)	<0.1 - 0.0124 (n = 3)	0.0124	1			
Neodymium (mg/kg)		0.0283	1			
Nitrogen (TKN)%		1.11	1			
Samarium(mg/kg)	0.0059 - 0.042	0.012 ± 0.011	9	0.0059 - 0.0090	0.0077 ± 0.0009	8
Strontium(mg/kg)	4.9 - 8.5	6.8 ± 1.5	4	no data rejected	no data rejected	4
Sulfur(%)		0.102	1			
Tantalum (mg/kg)	0.0011 - 0.0028, <0.005 (n = 1)	0.0018 ± 0.0009	3			
Terbium(mg/kg)	0.00053, 0.008		2			
Thorium(mg/kg)	0.010 - 0.205	0.068 ± 0.090	7	0.014 - 0.018	0.016 ± 0.001	4
^{232}Th (mg/kg)		0.027	1			
Tungsten(mg/kg)		0.065	1			
Uranium (mg/kg)	0.007 - 0.012 (<0.04, <0.08)	0.087 ± 0.003	3			
^{238}U (mg/kg)		0.0049	1			
Vanadium (mg/kg)	<0.08 - <0.34		3			
Ytterbium (mg/kg)	0.002 - 0.025		3			

*The values included in the average do not include values that were reported as limits of detection. The value for "n" represents the number of laboratories, not number of portions analyzed.

***Nota Bene*: Results of analyses from this ASTM interlaboratory comparison exercise are included in this report to provide a complete description of the study results. Any values in this report that are not included on the Certificate of Analysis have not been evaluated for accuracy and should not be used as certificate values.**

References

Christopher SJ, Long SE, Rearick MS, Fassett JD. Anal. Chem. 73, 2190-2199, 2001

Greenberg RR. Anal. Chem. 58, 2516-2523, 1986.

Levenson MS, Banks DL, Eberhardt KR, Gill LM, Guthrie WF, Liu HK, Vangel MS, Yen JH, Zhang NF, J Res. NIST 105(2000) 571-579.

Long SE, Kelly WR, Anal. Chem. 74, 1477-1483, 2002.

May W, Parris R, Beck C, Fassett J, Greenberg R, Guenther F, Kramer G, Wise S, Gills T, Colbert J, Gettings R, MacDonald B. NIST SP 230-136 “Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements” U.S. Government Printing Office Washington, pp. 12, 2000.

U.S. Geological Survey Open File Report 02-0223-G Analytical Methods for Chemical Analysis of Geological and Other Materials U.S. Geological Survey. Chapter I “The Determination of 40 Elements in Geological Materials by Inductively Coupled Plasma-Atomic Emission Spectrometry.” PH Briggs, pp. 18, 2002.

U.S. Geological Survey Open File Report 02-0223-I, Analytical Methods for Chemical Analysis of Geological and Other Materials U.S. Geological Survey, Chapter I “The Determination of 42 Elements in Geological Materials by Inductively Coupled Plasma-Mass Spectrometry.” PH Briggs and AL Meier, pp. 14, 2002.

Appendix A Results of Certification Analyses of Individual Portions of SRM 1575a Performed at NIST and at USGS Presented by Technique and Laboratory

NIST-CVIDMS		NIST-ICPMS			
Bottle No.	Hg (ng/g)	Ba (µg/g)	Pb (µg/g)	Ni (µg/g)	Cd (µg/g)
83		5.78	0.157	1.478	0.235
189	40.35	5.82	0.156	1.539	0.232
356	40.21	6.84	0.189	1.500	0.233
472	40.11	5.79	0.161	1.430	0.240
708	39.46	5.82	0.164	1.587	0.233
1107	39.52	6.29	0.173	1.313	0.232
average	39.9	6.06	0.167	1.475	0.234
std dev	0.4	0.43	0.013	0.095	0.003
n	5	6	6	6	6

NIST-RNAA	
Bottle No.	P (%)
66	0.101
522	0.101
606	0.103
739	0.100
803	0.102
865	0.103
953	0.104
986	0.106
1027	0.102
1224	0.101
average	0.102
std dev	0.002
n	10

RNAA		
Bottle No	Cu (µg/g)	Cd (ng/g)
66	2.93	231
227	2.86	229
308	2.89	226
739	2.90	245
803	2.89	223
986	2.92	237
1027	2.88	247
1142	2.86	232
1333		224
average	2.89	233
std dev	0.03	9
n	8	9

NIST-PGAA			
Bottle No.	B (µg/g)	Cl (µg/g)	K (µg/g)
66	9.66	410	4153
227	9.60	414	4127
308	9.44	417	4107
522	9.62	424	4206
739	9.62	416	4108
953	9.63	414	4108
986	9.63	418	4173
1027	9.55	424	4115
average	9.59	417	4137
std dev	0.07	5	37
n	8	8	8

NIST- INAA Bottle No.	As (ng/g)	Ba (µg/g)	Co (ng/g)	Cr (ng/g)	Cs (ng/g)	Fe (µg/g)	Zn µg/g)
1333	38.8	6.7	62.0	460	285	46.4	38.1
739	37.6	6.0	61.1	340	280	46	37.9
522	40.8	5.9	60.3	358	283	46.2	38.1
308	39.5	5.4	61.7	359	283	46.5	38.2
66	36.8	6.3	62.5	354	281	45.4	37.5
1142	38.8	5.6	60.6	349	283	46.1	38.0
953	--	5.4	61.9	524	281	45.4	37.8
606	39.5	6.5	62.0	326	287	45.4	38.3
308	--	5.7	60.6	355	284	45.3	38.1
227	39.3	6.1	60.8	323	278	45.8	37.9
average	38.9	5.96	61.4	374.8	283	45.9	38.0
std dev	1.2	0.45	0.8	64.8	3	0.5	0.2
n	8	10	10	10	10	10	10

NIST INAA Bottle No.	Ce (ng/g)	Rb (µg/g)	Sb (ng/g)	Sc (ng/g)	Se (ng/g)	Th (ng/g)
1333	110.0	16.7	7.2	10.3	105	17.1
739	104.0	16.5	7.2	9.87	104	17.3
522	103.0	16.7	7.2	10.0	101	14.9
308	107.0	16.6	7.0	10.3	101	15.7
66	122.0	16.4	6.3	9.87	95	14.2
1142	108.0	16.7	7.1	10.1	95	16.5
953	114.0	16.4	7.3	10.1	95	16.3
606	116.0	16.7	7.9	10.0	98	15.4
308	124.0	16.4	7.0	10.1	96	17.6
227	132.0	16.4	6.8	10.0	99	19.7
average	114.0	16.6	7.1	10.1	99	16.5
std dev	9.5	0.1	0.4	0.2	4	1.6
n	10	10	10	10	10	10

NIST-INAA short-lived Bottle No.	Na µg/g	K µg/g	Mn µg/g	Cl µg/g	Al µg/g	Mg µg/g	Ca %
1027	61.1	4340	490	429	562	1011	0.253
1027	62.3	4190	485	440			0.271
1142	62.4	4210	488	425	562	1031	0.254
1142	60.2	3840	463	430	564	1072	0.240
133	64.5	4000	489	436	560		0.246
133	61.1	4380	486	432	587	976	0.244
227	64.1	4260	491	420	564	1057	0.247
227	63.2	4170	489	414	581	1195	0.247
260	67.2	4010	490	428	557	909	0.241
260	63.8	4160	489	415	586	1019	0.248
29	62.1	4320	491	417	561	979	0.243
29	63.7	4210	488	432	591	1108	0.245
308	61.5	4340	491	428	584	980	0.251
308	61.7	4240	488	429	608	1050	0.242
522	62.4	4330	491	428	564	1063	0.251
52	64.6	4340	493	420	614	1193	0.256
739	64.4	4290	491	424	561	1114	0.257
739	63.4	4330	487	420	573	1154	0.245
803	61.7	4230	488	429	568	1035	0.255
803	61.2	4170	487	426	593	1015	0.248
953	61.7	4220	491	416	565	1158	0.236
953	64.1	4160	486	407	575	1062	0.240
986	62.1	4190	491	425	571	1006	0.242
986	60.4	4080	485	422	585	1080	0.251
average	62.7	4209	488	425	575	1058	0.248
std dev	1.6	128	6	8	16	73	0.007
n	24	24	24	24	23	22	24

NIST RNAA Bottle No.	Hg (ng/g)
29	37.61
66	43.53
308	45.18
522	43.13
606	39.61
1142	42.61
1224	39.01
average	41.5
std dev	2.8
n	7

USGS	ICP40	ICP40	ICPMS_ACID	ICP40	ICP40	ICP40	ICPMS_ACID	ICP40
Bottle No.	Al mg/kg	Ca %	Ca %	Cu mg/kg	Fe mg/kg	P %	Rb mg/kg	Zn mg/kg
766a	630	0.27499	0.221	2.72	48.8	0.115	14.7	40.1
901a	590	0.26378	0.237	2.69	46.2	0.112	15.4	37.8
1191a	625	0.27178	0.264	2.84	48.5	0.118	17.2	39.4
1255b	600	0.26001	0.247	2.60	46.4	0.111	16.2	37.4
491a	605	0.27216	0.244	2.67	47.1	0.114	15.9	38.8
1255a	593	0.25272	0.247	2.92	46.4	0.115	16.1	38.4
1149a	539	0.23976	0.249	2.49	42.2	0.103	16.3	34.6
824b	578	0.25546	0.263	2.63	45.8	0.111	17.2	37.4
619b	571	0.2491	0.26	2.63	44.4	0.109	16.9	36.2
491b	607	0.2646	0.247	2.67	47.4	0.114	16.2	38.6
824a	569	0.2444	0.264	2.70	42.5	0.109	17.3	36.9
619a	578	0.25064	0.259	2.68	45.3	0.111	17.1	37.1
1191b	612	0.26924	0.267	2.79	48.3	0.117	17.5	39.1
1149b	528	0.2331	0.251	2.38	41.7	0.101	16.5	34.0
766b	622	0.26728	0.23	3.14	49.1	0.122	15.2	40.3
901b	576	0.25168	0.245	2.61	46.0	0.110	16.1	37.0
Avg	589	0.258	0.250	2.70	46.0	0.112	16.36	37.7
Std Dev	29	0.012	0.013	0.17	2.3	0.005	0.82	1.8
n	16	16	16	16	16	16	16	16

Appendix B Copy of the SRM1575a Pine Needles Certificate of Analysis

Appendix C. List of Participants in ASTM Interlaboratory Comparison Exercise

Participant/Analyst(s)	**Affiliation**	**City, Country**
Ljudmila Benedik, Urska Repinc	Dept. of Environmental Sciences Jozef Stefan Institute	Ljubljana, Slovenia
Maria Carmo-Fritas	Instituto Tecnológico E Nuclear	Sacavem, Portugal
Sara Resnizky	Comisión Nacional de Energía Atómica	Buenos Aires, Argentina
Elisabete Fernandes, Cláudio Luiz Gonzaga	CENA, Laboratorio de Radioisotopos University of Sao Paulo	Sao Paulo, Brazil
Craig Stuart	Becquerel Laboratories	Mississauga, Ontario, Canada
Gregory Kennedy, Jean St-Pierre	Écolé Polytechnique Montréal Slowpoke Laboratory	Centre-Ville, Montréal, Canada
Jan Kucera	Czech Academy of Sciences	Rez near Prague, Czech Republic
M. Sundersanan, S. R. Kayatsth, K. K. Swain	Government of India, Bhabha Atomic Research Centre, Analytical Chemistry Division	Trombay, Mumbai, India
E. Wallich, Maxine Ranta	Weyerhaeuser Analytical and Testing Services	Federal Way, Washington, USA
Raymund Gwozdz	TraceChem	Copenhagen, Denmark
Achim Berger, Wolf Goerner	Lab 1.43, Activation Analysis BAM	Berlin, Germany
Marina Frontasyeva	Joint Institute for Nuclear Research	Dubna, Russia
Bozena Danko	Institute for Nuclear Research	Warsaw, Poland
Amarnath Garg, Ashok Kumar	Radioanalytical Laboratory Department of Chemistry Indian Institute of Technology	Roorkee, India
Xileio Lin Richard Henkelmann	Technical University of Munich	Munich, Germany
Sophie Ayrault	Laboratory Pierre Sue, Commissariat á l'Energie Atomique, CNRS	Saclay, France

Appendix D Analytical Results From Each Participant in the ASTM Interlaboratory Comparison Coded by Participant Numbers

Element	Lab No.	Average	1s	n	z-score	Method
Aluminum (mg/kg)	Lab 3	580	19	2	0.0	INAA
Antimony (mg/kg)	Lab 3	0.0120	0.001	1	3.3	INAA
Arsenic (mg/kg)	Lab 3	0.047	0.009	2	2.1	INAA
Barium (mg/kg)	Lab 3	5.2	1	2	-1.3	INAA
Bromine (mg/kg)	Lab 3	3.16	0.1	2	1.0	INAA
Cadmium (mg/kg)	Lab 3	0.252	0.014	2	0.8	INAA
Calcium (%)	Lab 3	0.252%	0.014%	2	0.0	INAA
Cerium (mg/kg)	Lab 3	0.114	0.017	2	0.4	INAA
Cesium (mg/kg)	Lab 3	0.30	0.01	2	0.6	INAA
Chlorine (mg/kg)	Lab 3	389	17	2	-0.8	INAA
Chromium (mg/kg)	Lab 3	0.36	0.03	2	0.8	INAA
Cobalt (mg/kg)	Lab 3	0.074	0.003	2	2.1	INAA
Copper (mg/kg)	Lab 3	3.9	0.6	2	3.9	INAA
Fluorine (mg/kg)	Lab 3	389	20	2		INAA
Gallium (mg/kg)	Lab 3	<1		2		INAA
Gold (mg/kg)	Lab 3	0.00056	0.00018	2		INAA
Hafnium (mg/kg)	Lab 3	0.014	0.02	2		INAA
Indium (mg/kg)	Lab 3	<0.003		2		INAA
Iodine (mg/kg)	Lab 3	<1.5		2		INAA
Iridium (mg/kg)	Lab 3	<0.0001		2		INAA
Iron (mg/kg)	Lab 3	51.4	2.2	2	1.2	INAA
Lanthanum (mg/kg)	Lab 3	0.0565	0.0050	2	0.6	INAA
Magnesium (%)	Lab 3	0.109%	0.012%	2	0.3	INAA
Manganese (mg/kg)	Lab 3	502	16	2	0.3	INAA
Mercury (mg/kg)	Lab 3	0.029	0.007	2	-0.2	INAA
Molybdenum (mg/kg)	Lab 3	<0.1		2		INAA
Nickel (mg/kg)	Lab 3	<1.3		2		INAA
Potassium (%)	Lab 3	0.404%	0.015%	2	-0.3	INAA
Rubidium (mg/kg)	Lab 3	16.4	0.5	2	-0.1	INAA
Samarium (mg/kg)	Lab 3	0.0078	0.0004	2	0.1	INAA
Scandium (mg/kg)	Lab 3	0.0116	0.0005	2	1.5	INAA
Selenium (mg/kg)	Lab 3	0.13	0.02	2	3.1	INAA
Silver (mg/kg)	Lab 3	<0.03		2		INAA
Sodium (mg/kg)	Lab 3	63.0	2.4	2	0.0	INAA
Strontium (mg/kg)	Lab 3	7.2	1.3	2	0.5	INAA
Tantalum (mg/kg)	Lab 3	<0.005		1		INAA
Thorium (mg/kg)	Lab 3	0.016	0.002	2	0.0	INAA
Uranium (mg/kg)	Lab 3	<0.04		1		INAA
Vanadium (mg/kg)	Lab 3	<0.26		2		INAA
Zinc (mg/kg)	Lab 3	38.5	2.0	2	0.1	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Bromine (mg/kg)	Lab 4	2.966	0.248	6	0.4	INAA
Cobalt (mg/kg)	Lab 4	0.0686	0.0064	6	1.2	INAA
Europium (mg/kg)	Lab 4	0.0017	0.0006	6		INAA
Iron (mg/kg)	Lab 4	50.0	5.6	6	0.9	INAA
Lanthanum (mg/kg)	Lab 4	0.0565	0.0057	6	0.6	INAA
Rubidium (mg/kg)	Lab 4	15.8	2.8	6	-0.4	INAA
Samarium (mg/kg)	Lab 4	0.0080	0.00071	6	0.3	INAA
Scandium (mg/kg)	Lab 4	0.0106	0.0008	6	0.5	INAA
Selenium (mg/kg)	Lab 4	0.113	0.010	6	1.4	INAA
Zinc (mg/kg)	Lab 4	40.2	2.5	6	0.6	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Antimony (mg/kg)	Lab 6	0.0099	0.0035	4	1.0	INAA
Arsenic (mg/kg)	Lab 6	0.037	0.008	4	-0.5	INAA
Barium (mg/kg)	Lab 6	5.7	2.3	4	-0.5	INAA
Bromine (mg/kg)	Lab 6	2.84	0.07	4	-0.1	INAA
Calcium (%)	Lab 6	0.240%	0.024%	4	-0.4	INAA
Cerium (mg/kg)	Lab 6	0.119	0.028	4	0.8	INAA
Cesium (mg/kg)	Lab 6	0.286	0.012	4	0.1	INAA
Chlorine (mg/kg)	Lab 6	396	21	4	-0.6	INAA
Chromium (mg/kg)	Lab 6	0.290	0.051	4	-1.3	INAA
Cobalt (mg/kg)	Lab 6	0.060	0.005	4	-0.2	INAA
Europium (mg/kg)	Lab 6	<0.002		4		INAA
Iron (mg/kg)	Lab 6	43.3	3.0	4	-0.6	INAA
Lanthanum (mg/kg)	Lab 6	0.066	0.012	4	2.4	INAA
Magnesium (%)	Lab 6	0.096%	0.018%	4	-1.0	INAA
Manganese (mg/kg)	Lab 6	475.1	6.4	4	-0.3	INAA
Potassium (%)	Lab 6	0.3971%	0.0081%	4	-0.5	INAA
Rubidium (mg/kg)	Lab 6	17.1	0.5	4	0.4	INAA
Samarium (mg/kg)	Lab 6	0.0080	0.0016	4	0.4	INAA
Scandium (mg/kg)	Lab 6	0.0097	0.0005	4	-0.4	INAA
Sodium (mg/kg)	Lab 6	56.1	1.0	4	-1.1	INAA
Thorium (mg/kg)	Lab 6	0.0178	0.0055	4	1.1	INAA
Zinc (mg/kg)	Lab 6	37.6	1.0	4	-0.1	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Aluminum (mg/kg)	Lab 8	553	55	6	-0.5	INAA
Antimony (mg/kg)	Lab 8	0.0083	0.0012	6	-0.8	INAA
Arsenic (mg/kg)	Lab 8	0.037	0.006	6	-0.5	INAA
Barium (mg/kg)	Lab 8	5.9	0.6	6	-0.2	INAA
Bromine (mg/kg)	Lab 8	3.1	0.3	6	0.8	INAA
Calcium (%)	Lab 8	0.243%	0.036%	6	-0.3	INAA
Cerium (mg/kg)	Lab 8	<0.16		6		INAA
Cesium (mg/kg)	Lab 8	0.30	0.03	6	0.6	INAA
Chlorine (mg/kg)	Lab 8	438	44	6	0.4	INAA
Chromium (mg/kg)	Lab 8	0.33	0.1	6	-0.1	INAA
Cobalt (mg/kg)	Lab 8	0.067	0.010	6	1.0	INAA
Europium (mg/kg)	Lab 8	<0.03		6		INAA
Gold (mg/kg)	Lab 8	0.00260	0.0005	6		INAA
Hafnium (mg/kg)	Lab 8	<0.015		6		INAA
Iron (mg/kg)	Lab 8	48	6	6	0.4	INAA
Lanthanum (mg/kg)	Lab 8	0.058	0.009	6	0.9	INAA
Magnesium (%)	Lab 8	0.102%	0.017%	6	-0.3	INAA
Manganese (mg/kg)	Lab 8	465	46	6	-0.5	INAA
Nickel (mg/kg)	Lab 8	1.29	0.19	6	-1.2	INAA
Potassium (%)	Lab 8	0.426%	0.085%	6	0.2	INAA
Rubidium (mg/kg)	Lab 8	18.1	1.8	6	1.0	INAA
Samarium (mg/kg)	Lab 8	0.0082	0.0008	6	0.6	INAA
Scandium (mg/kg)	Lab 8	0.0126	0.0025	6	2.5	INAA
Selenium (mg/kg)	Lab 8	0.13	0.03	6	3.1	INAA
Strontium (mg/kg)	Lab 8	8.5	0.8	6	2.4	INAA
Tantalum (mg/kg)	Lab 8	0.0028	0.0007	6		INAA
Thorium (mg/kg)	Lab 8	0.016	0.002	6	0.0	INAA
Tungsten	Lab 8	0.065	0.019	6		INAA
Uranium (mg/kg)	Lab 8	0.0068	0.0014	6		INAA
Vanadium (mg/kg)	Lab 8	<0.27		6		INAA
Zinc (mg/kg)	Lab 8	38.2	3.8	6	0.1	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Cobalt (mg/kg)	Lab 10	0.0598	0.0048	3	-0.2	INAA
Molybdenum (mg/kg)	Lab 10	0.0124	0.0012	4		INAA
Rubidium (mg/kg)	Lab 10	16.18	1.62	3	-0.2	INAA
Scandium (mg/kg)	Lab 10	0.0117	0.0009	3	1.6	INAA
Uranium (mg/kg)	Lab 10	0.0071	0.0011	4		INAA
Zinc (mg/kg)	Lab 10	39.3	2.0	3	0.3	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Arsenic (mg/kg)	Lab 13a	0.104	0.019	2	16.7	INAA
Barium (mg/kg)	Lab 13a	9.5	1.7	3	5.8	INAA
Bromine (mg/kg)	Lab 13a	3.08	0.16	5	0.7	INAA
Cesium (mg/kg)	Lab 13a	0.270	0.017	3	-0.5	INAA
Chromium (mg/kg)	Lab 13a	1.33	0.33	6	29.9	INAA
Cobalt (mg/kg)	Lab 13a	0.501	0.117	3	72.1	INAA
Gold (mg/kg)	Lab 13a	0.00150	0.0004	3		INAA
Hafnium (mg/kg)	Lab 13a	0.116	0.026	3		INAA
Iron (mg/kg)	Lab 13a	192	19	6	32	INAA
Lanthanum (mg/kg)	Lab 13a	0.335	0.022	3	53.0	INAA
Manganese (mg/kg)	Lab 13a	506	47	4	0.4	INAA
Phosphorus (%)	Lab 13a	0.112%	0.009%	10	0.5	INAA
Potassium (%)	Lab 13a	0.431%	0.048%	7	0.3	INAA
Rubidium (mg/kg)	Lab 13a	16.4	0.5	3	-0.1	INAA
Scandium (mg/kg)	Lab 13a	0.097	0.012	3	86.0	INAA
Sodium (mg/kg)	Lab 13a	111	36	7	7.6	INAA
Thorium (mg/kg)	Lab 13a	0.205	0.032	3	118	INAA
Zinc (mg/kg)	Lab 13a	51.5	2.9	3	3.6	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Antimony (mg/kg)	Lab 13b	0.0330	0.007	3	26.7	INAA
Barium (mg/kg)	Lab 13b	8.8	2.4	3	4.7	INAA
Bromine (mg/kg)	Lab 13b	3.91	0.43	3	3.6	INAA
Cerium (mg/kg)	Lab 13b	0.74	0.10	3	57.3	INAA
Cesium (mg/kg)	Lab 13b	0.293	0.023	3	0.4	INAA
Chromium (mg/kg)	Lab 13b	2.37	0.2	3	61.0	INAA
Cobalt (mg/kg)	Lab 13b	0.541	0.130	3	78.7	INAA
Hafnium (mg/kg)	Lab 13b	0.122	0.025	3		INAA
Iron (mg/kg)	Lab 13b	208	19	3	35	INAA
Lanthanum (mg/kg)	Lab 13b	0.389	0.03	3	63.1	INAA
Potassium (%)	Lab 13b	0.467%	0.096%	3	1.2	INAA
Rubidium (mg/kg)	Lab 13b	17.2	1.3	3	0.4	INAA
Samarium (mg/kg)	Lab 13b	0.0420	0.003	3	44.5	INAA
Scandium (mg/kg)	Lab 13b	0.098	0.009	3	87.0	INAA
Selenium (mg/kg)	Lab 13b	0.017	0.002	3	-8.3	INAA
Thorium (mg/kg)	Lab 13b	0.195	0.029	3	112	INAA
Ytterbium (mg/kg)	Lab 13b	0.025	0.005	3		INAA
Zinc (mg/kg)	Lab 13b	50.4	7.0	3	3.3	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Calcium (%)	Lab 13c	0.457%		1	8.2	AAS
Iron (mg/kg)	Lab 13c	49.9		1	0.8	AAS
Magnesium (%)	Lab 13c	0.110%			0.4	AAS
Manganese (mg/kg)	Lab 13c	309			-3.7	AAS
Potassium (%)	Lab 13c	0.289%			-3.1	AAS
Sodium (mg/kg)	Lab 13c	133			11.1	AAS
Zinc (mg/kg)	Lab 13c	49.9			3.1	AAS

Element	Lab No.	Average	1s	n	z-score	Method
Bromine (mg/kg)	Lab 15a	3.38	0.16	1	1.8	AAS
Manganese (mg/kg)	Lab 15a	452	13	1	-0.7	AAS
Potassium (%)	Lab 15a	0.398%	0.078%	1	-0.5	AAS
Sodium (mg/kg)	Lab 15a	73.2	5.5	1	1.6	AAS

Element	Lab No.	Average	1s	n	z-score	Method
Aluminum (mg/kg)	Lab 15b	786	67	4	3.6	FAAS/GFAAS
Cadmium (mg/kg)	Lab 15b	0.360	0.040	2	5.5	FAAS/GFAAS
Calcium (%)	Lab 15b	0.194%	0.029%	4	-2.3	FAAS/GFAAS
Chromium (mg/kg)	Lab 15b	0.32	0.06	2	-0.4	FAAS/GFAAS
Cobalt (mg/kg)	Lab 15b	<0.035		1		FAAS/GFAAS
Copper (mg/kg)	Lab 15b	1.86	0.18	2	-3.4	FAAS/GFAAS
Iron (mg/kg)	Lab 15b	43.5	5.6	4	-0.5	FAAS/GFAAS
Lead (mg/kg)	Lab 15b	0.115	0.031	4	-3.1	FAAS/GFAAS
Magnesium (%)	Lab 15b	0.123%	0.009%	4	1.6	FAAS/GFAAS
Manganese (mg/kg)	Lab 15b	405	40	4	-1.7	FAAS/GFAAS
Nickel (mg/kg)	Lab 15b	1.10	0.04	2	-2.5	FAAS/GFAAS
Potassium (%)	Lab 15b	0.44%	0.02%	4	0.6	FAAS/GFAAS
Silver (mg/kg)	Lab 15b	<0.05		1		FAAS/GFAAS
Sodium (mg/kg)	Lab 15b	52.6	5.0	4	-1.7	FAAS/GFAAS
Zinc (mg/kg)	Lab 15b	29.9	3	4	-2.1	FAAS/GFAAS

Element	Lab No.	Average	1s	n	z-score	Method
Antimony (mg/kg)	Lab 16	0.0062	0.0012	11	-3.1	INAA
Barium (mg/kg)	Lab 16	4.0	0.3	8	-3.3	INAA
Bromine (mg/kg)	Lab 16	2.1	0.4	8	-2.7	INAA
Cadmium (mg/kg)	Lab 16	0.0119	0.0025	5	-9.5	INAA
Calcium (%)	Lab 16	0.15%	0.01%	10	-4.0	INAA
Cerium (mg/kg)	Lab 16	0.0783	0.0015	7	-2.9	INAA
Cesium (mg/kg)	Lab 16	0.1991	0.0096	8	-3.0	INAA
Chromium (mg/kg)	Lab 16	0.268	0.006	9	-2.0	INAA
Cobalt (mg/kg)	Lab 16	0.0452	0.0028	8	-2.6	INAA
Europium (mg/kg)	Lab 16	0.0011	0.0002	5		INAA
Gold (mg/kg)	Lab 16	0.00070	0.0001	9		INAA
Hafnium (mg/kg)	Lab 16	0.0063	0.0007	7		INAA
Iron (mg/kg)	Lab 16	32.4	0.1	3	-3.0	INAA
Lanthanum (mg/kg)	Lab 16	0.0416	0.0049	8	-2.2	INAA
Lutecium (mg/kg)	Lab 16	0.0006	0.0001	7		INAA
Mercury (mg/kg)	Lab 16	0.0035	0.0003	6	-9.1	INAA
Neodymium (mg/kg)	Lab 16	0.0283		2		INAA
Nickel (mg/kg)	Lab 16	0.9	0.1	6	-3.9	INAA
Potassium (%)	Lab 16	0.59%	0.17%	3	4.1	INAA
Rubidium (mg/kg)	Lab 16	0.0115	0.0004	8	-10.0	INAA
Samarium (mg/kg)	Lab 16	0.0059	0.0008	9	-2.3	INAA
Scandium (mg/kg)	Lab 16	0.0074	0.0001	7	-2.7	INAA
Selenium (mg/kg)	Lab 16	0.0696	0.0066	8	-3.0	INAA
Silver (mg/kg)	Lab 16	0.0118		2		INAA
Sodium (mg/kg)	Lab 16	72.5	9.3	4	1.5	INAA
Strontium (mg/kg)	Lab 16	4.9	0.7	4	-2.8	INAA
Tantalum (mg/kg)	Lab 16	0.0011	0.0001	5		INAA
Terbium (mg/kg)	Lab 16	0.0008	0.0001	4		INAA
Thorium (mg/kg)	Lab 16	0.010	0.0002	7	-3.8	INAA
Uranium (mg/kg)	Lab 16	0.0122		2		INAA
Ytterbium (mg/kg)	Lab 16	0.0033	0.0007	6		INAA
Zinc (mg/kg)	Lab 16	27.3	0.3	9	-2.8	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Aluminum (mg/kg)	Lab 17	567	20	3	-0.2	INAA
Antimony (mg/kg)	Lab 17	0.0230	0.002	3	15.6	INAA
Arsenic (mg/kg)	Lab 17	0.038	0.002	3	-0.3	INAA
Bromine (mg/kg)	Lab 17	2.7	0.1	3	-0.6	INAA
Calcium (%)	Lab 17	0.254%	0.013%	3	0.1	INAA
Cesium (mg/kg)	Lab 17	0.28	0.01	3	-0.1	INAA
Chlorine (mg/kg)	Lab 17	391	15	3	-0.7	INAA
Chromium (mg/kg)	Lab 17	0.37	0.02	3	1.1	INAA
Cobalt (mg/kg)	Lab 17	0.062	0.003	3	0.2	INAA
Iron (mg/kg)	Lab 17	45	3	3	-0.2	INAA
Magnesium (%)	Lab 17	0.096%	0.003%	3	-0.9	INAA
Manganese (mg/kg)	Lab 17	474	15	3	-0.3	INAA
Potassium (%)	Lab 17	0.408%	0.015%	3	-0.2	INAA
Rubidium (mg/kg)	Lab 17	16.2	0.05	3	-0.2	INAA
Scandium (mg/kg)	Lab 17	0.0108	0.0004	3	0.7	INAA
Sodium (mg/kg)	Lab 17	67	3	3	0.6	INAA
Zinc (mg/kg)	Lab 17	38.3	2.0	3	0.1	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Bromine (mg/kg)	Lab 18	2.75	0.22	6	-0.4	INAA
Calcium (%)	Lab 18	0.232%	0.044%	5	-0.8	INAA
Cerium (mg/kg)	Lab 18	0.097	0.025	6	-1.2	INAA
Cobalt (mg/kg)	Lab 18	0.060	0.004	6	-0.2	INAA
Iron (mg/kg)	Lab 18	42.7	4.6	6	-0.7	INAA
Lanthanum (mg/kg)	Lab 18	0.049	0.011	6	-0.8	INAA
Potassium (%)	Lab 18	0.402%	0.032%	6	-0.4	INAA
Rubidium (mg/kg)	Lab 18	15.3	1.6	6	-0.7	INAA
Samarium (mg/kg)	Lab 18	0.0078	0.0008	6	0.1	INAA
Scandium (mg/kg)	Lab 18	0.0086	0.0007	6	-1.5	INAA
Sodium (mg/kg)	Lab 18	61	5	6	-0.3	INAA
Zinc (mg/kg)	Lab 18	34.7	3.1	6	-0.9	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Arsenic (mg/kg)	Lab 19	0.036	0.005	6	-0.8	INAA
Barium (mg/kg)	Lab 19	5.4	0.4	6	-1.0	INAA
Cesium (mg/kg)	Lab 19	0.289	0.009	6	0.2	INAA
Chromium (mg/kg)	Lab 19	0.366	0.02	6	1.0	INAA
Cobalt (mg/kg)	Lab 19	0.065	0.001	6	0.7	INAA
Iron (mg/kg)	Lab 19	47.3	1.3	6	0.3	INAA
Lanthanum (mg/kg)	Lab 19	0.050	0.004	6	-0.6	INAA
Potassium (%)	Lab 19	0.383%	0.003%	6	-0.8	INAA
Rubidium (mg/kg)	Lab 19	16.1	0.2	6	-0.2	INAA
Scandium (mg/kg)	Lab 19	0.0110	0.0004	6	0.9	INAA
Sodium (mg/kg)	Lab 19	57	1	6	-0.9	INAA
Zinc (mg/kg)	Lab 19	37.2	0.5	6	-0.2	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Antimony (mg/kg)	Lab 20	0.0086	0.00046	3	-0.5	INAA
Arsenic (mg/kg)	Lab 20	0.040	0.003	3	0.3	INAA
Barium (mg/kg)	Lab 20	4.96	0.30	3	-1.7	INAA
Bromine (mg/kg)	Lab 20	2.72	0.14	3	-0.5	INAA
Calcium (%)	Lab 20	0.247%	0.012%	3	-0.2	INAA
Cerium (mg/kg)	Lab 20	0.0981	0.0057	3	-1.1	INAA
Cesium (mg/kg)	Lab 20	0.266	0.014	3	-0.6	INAA
Chromium (mg/kg)	Lab 20	0.365	0.020	3	0.9	INAA
Cobalt (mg/kg)	Lab 20	0.0645	0.0037	3	0.6	INAA
Europium (mg/kg)	Lab 20	0.00144	0.00012	3		INAA
Gold (mg/kg)	Lab 20	0.00026	0.000042	3		INAA
Hafnium (mg/kg)	Lab 20	0.0128	0.0016	3		INAA
Iron (mg/kg)	Lab 20	46.4	2.7	3	0.1	INAA
Lanthanum (mg/kg)	Lab 20	0.0479	0.0028	3	-1.0	INAA
Lutecium (mg/kg)	Lab 20	0.00055	0.00006	3		INAA
Mercury (mg/kg)	Lab 20	0.0391	0.0028	3	-2.7	INAA
Potassium (%)	Lab 20	0.422%	0.019%	3	0.1	INAA
Rubidium (mg/kg)	Lab 20	16.73	0.76	3	0.1	INAA
Samarium (mg/kg)	Lab 20	0.0070	0.00044	3	-1.0	INAA
Scandium (mg/kg)	Lab 20	0.0104	0.0006	3	0.3	INAA
Selenium (mg/kg)	Lab 20	0.0985	0.0063	3	-0.1	INAA
Sodium (mg/kg)	Lab 20	64.7	3.1	3	0.3	INAA
Strontium (mg/kg)	Lab 20	6.76	0.41	3	-0.1	INAA
Tantalum (mg/kg)	Lab 20	0.00135	0.00012	3		INAA
Terbium (mg/kg)	Lab 20	0.00053	0.00010	1		INAA
Thorium (mg/kg)	Lab 20	0.0142	0.0007	3	-1.1	INAA
Ytterbium (mg/kg)	Lab 20	0.00215	0.00026	3		INAA
Zinc (mg/kg)	Lab 20	37.6	2.0	3	-0.1	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Bromine (mg/kg)	Lab 21	2.76	0.09	6	-0.4	INAA
Calcium (%)	Lab 21	0.243%	0.010%	6	-0.3	INAA
Cesium (mg/kg)	Lab 21	0.276	0.012	6	-0.2	INAA
Cobalt (mg/kg)	Lab 21	0.0628	0.0030	6	0.3	INAA
Iron (mg/kg)	Lab 21	46.2	1.5	6	0.0	INAA
Potassium (%)	Lab 21	0.397%	0.006%	6	-0.5	INAA
Rubidium (mg/kg)	Lab 21	17.0	0.5	6	0.3	INAA
Scandium (mg/kg)	Lab 21	0.0103	0.0002	6	0.2	INAA
Sodium (mg/kg)	Lab 21	61.8	2.0	6	-0.2	INAA
Zinc (mg/kg)	Lab 21	36.5	0.6	6	-0.4	INAA

Element	Lab No.	Average	1s	n	z-score	Method
Boron (mg/kg)	Lab 23	<20		3		E-6010
Calcium (%)	Lab 23	0.246%	0.001%	3	-0.2	E-6010
Copper (mg/kg)	Lab 23	3	0	3	0.7	E-6010
Iron (mg/kg)	Lab 23	41	0	3	-1.1	E-6010
Magnesium (%)	Lab 23	0.100%	0.001%	3	-0.6	E-6010
Manganese (mg/kg)	Lab 23	473	2	3	-0.3	E-6010
Nitrogen (%) [TKN]	Lab 23	1.11%	0.01%	5		ASTM#1402-07
Phosphorus (%)	Lab 23	0.124%	0.001%	5	1.6	ASTM#1402-07
Potassium (%)	Lab 23	0.393%	0.003%	3	-0.6	E-6010
Sulfur (%)	Lab 23	0.102%	0.001%	3		ASTM#1552-90
Zinc (mg/kg)	Lab 23	38	0	3	0.0	E-6010

Element	**Lab No.**	**Average**	**1s**	**n**	**z-score**	**Method**
Aluminum (mg/kg)	Lab 24	430	17	4	-2.6	INAA
Antimony (mg/kg)	Lab 24	0.0200	0.006	4	12.2	INAA
Arsenic (mg/kg)	Lab 24	0.047	0.006	4	2.1	INAA
Bromine (mg/kg)	Lab 24	2.83	0.07	4	-0.1	INAA
Calcium (%)	Lab 24	0.273%	0.041%	4	0.9	INAA
Chlorine (mg/kg)	Lab 24	382	32	4	-0.9	INAA
Lanthanum (mg/kg)	Lab 24	0.222	0.070	4	31.7	INAA
Magnesium (%)	Lab 24	0.062%	0.027%	1	-4.2	INAA
Manganese (mg/kg)	Lab 24	436	14	4	-1.1	INAA
Molybdenum (mg/kg)	Lab 24	<0.4		4		INAA
Nickel (mg/kg)	Lab 24	1.54	0.21	4	0.5	INAA
Potassium (%)	Lab 24	0.435%	0.003%	4	0.4	INAA
Samarium (mg/kg)	Lab 24	0.0090	0.002	4	1.7	INAA
Sodium (mg/kg)	Lab 24	93.0	4.1	4	4.8	INAA
Uranium (mg/kg)	Lab 24	<0.08		4		INAA
Vanadium (mg/kg)	Lab 24	<0.34		4		INAA
Vanadium (mg/kg)	Lab 24	<0.08		4		INAA

Appendix E Analytical Results for Each Element Reported by Participants Compared with Certificate Values and Interlaboratory Averages

Element	Lab No.	Average	1s	n	z-score	Method
Aluminum (mg/kg)	Lab 15b	786	67	4	3.6	FAAS/GFAAS
	Lab 17	567	20	3	-0.2	INAA
	Lab 24	430	17	4	-2.6	INAA
	Lab 3	580	19	2	0.0	INAA
	Lab 8	553	55	6	-0.5	INAA
all ASTM data	Avg ± 1s	583	128	5		number rejected
after rejection	Avg ± 1s	533	69	4		**1**
	NIST	575	16	23		INAA
	USGS	589	29	16		ICP-AES
Certificate Value	**Certified**	**580 ± 30**				

Element	Lab No.	Average	1s	n	z-score	Method
Antimony (mg/kg)	Lab 13b	0.0330	0.007	3	26.7	INAA
	Lab 16	0.0062	0.0012	11	-3.1	INAA
	Lab 17	0.0230	0.002	3	15.6	INAA
	Lab 20	0.0086	0.00046	3	-0.5	INAA
	Lab 24	0.0200	0.006	4	12.2	INAA
	Lab 3	0.0120	0.001	1	3.3	INAA
	Lab 6	0.0099	0.0035	4	1.0	INAA
	Lab 8	0.0083	0.0012	6	-0.8	INAA
all ASTM data	Avg ± 1s	0.015	0.009	8		number rejected
after rejection	Avg ± 1s	0.009	0.001	4		**4**

Element	Lab No.	Average	1s	n	z-score	Method
Arsenic (mg/kg)	Lab 13a	0.104	0.019	2	16.7	INAA
	Lab 17	0.038	0.002	3	-0.3	INAA
	Lab 19	0.036	0.005	6	-0.8	INAA
	Lab 20	0.040	0.003	3	0.3	INAA
	Lab 24	0.047	0.006	4	2.1	INAA
	Lab 3	0.047	0.009	2	2.1	INAA
	Lab 6	0.037	0.008	4	-0.5	INAA
	Lab 8	0.037	0.006	6	-0.5	INAA
all ASTM data	Avg ± 1s	0.0483	0.023	10		number rejected
after rejection	Avg ± 1s	0.0403	0.005	9		1
	NIST	0.0389	0.00123	8		INAA
Certificate Value	**Reference**	**0.039 ± 0.002**				

Element	Lab No.	Average	1s	n	z-score	Method
Barium (mg/kg)	Lab 13a	9.5	1.7	3	5.8	INAA
	Lab 13b	8.8	2.4	3	4.7	INAA
	Lab 16	4.0	0.3	8	-3.3	INAA
	Lab 19	5.4	0.4	6	-1.0	INAA
	Lab 20	4.96	0.30	3	-1.7	INAA
	Lab 3	5.2	1	2	-1.3	INAA
	Lab 6	5.7	2.3	4	-0.5	INAA
	Lab 8	5.9	0.6	6	-0.2	INAA
all ASTM data	Avg ± 1s	6.2	1.9	8		number rejected
after rejection	Avg ± 1s	5.4	0.4	5		3
	NIST	5.96	0.45	10		INAA
	NIST	6.06	0.43	6		ICP-MS
Certificate Value	**Certified**	**6.0 ± 0.2**				

Element	Lab No.	Average	1s	n	z-score	Method
Boron (mg/kg)	Lab 23	<20		3		E-6010
	NIST	9.59	0.07	8		PGAA
Certificate Value	**Reference**	**9.6 ± 0.2**				

Element	Lab No.	Average	1s	n	z-score	Method
Bromine (mg/kg)	Lab 13a	3.08	0.16	5	0.7	INAA
	Lab 13b	3.91	0.43	3	3.6	INAA
	Lab 15a	3.38	0.16	1	1.8	AAS
	Lab 16	2.1	0.4	8	-2.7	INAA
	Lab 17	2.7	0.1	3	-0.6	INAA
	Lab 18	2.75	0.22	6	-0.4	INAA
	Lab 20	2.72	0.14	3	-0.5	INAA
	Lab 21	2.76	0.09	6	-0.4	INAA
	Lab 24	2.83	0.07	4	-0.1	INAA
	Lab 3	3.16	0.1	2	1.0	INAA
	Lab 4	2.966	0.248	6	0.4	INAA
	Lab 6	2.84	0.07	4	-0.1	INAA
	Lab 8	3.1	0.3	6	0.8	INAA
all ASTM data	Avg ± 1s	2.95	0.42	13		number rejected
after rejection	Avg ± 1s	2.87	0.32	12		1

Element	Lab No.	Average	1s	n	z-score	Method
Cadmium (mg/kg)	Lab 15b	0.360	0.040	2	5.5	FAAS/GFAAS
	Lab 16	0.0119	0.0025	5	-9.5	INAA
	Lab 3	0.252	0.014	2	0.8	INAA
all ASTM data	Avg ± 1s	0.208	0.178	3		number rejected
after rejection	only Lab 3	0.252	0.014	1		2
	NIST	0.233	0.009	9		RNAA
	NIST	0.234	0.003	6		ICP-MS
Certificate Value	**Certified**	**0.233 ± 0.004**				

Element	Lab No.	Average	1s	n	z-score	Method
Calcium (%)	Lab 13c	0.457%		1	8.2	AAS
	Lab 15b	0.194%	0.029%	4	-2.3	FAAS/GFAAS
	Lab 16	0.15%	0.01%	10	-4.0	INAA
	Lab 17	0.254%	0.013%	3	0.1	INAA
	Lab 18	0.232%	0.044%	5	-0.8	INAA
	Lab 20	0.247%	0.012%	3	-0.2	INAA
	Lab 21	0.243%	0.010%	6	-0.3	INAA
	Lab 23	0.246%	0.001%	3	-0.2	E-6010
	Lab 24	0.273%	0.041%	4	0.9	INAA
	Lab 3	0.252%	0.014%	2	0.0	INAA
	Lab 6	0.240%	0.024%	4	-0.4	INAA
	Lab 8	0.243%	0.036%	6	-0.3	INAA
all ASTM data	Avg ± 1s	0.253%	0.072%	12		number rejected
after rejection	Avg ± 1s	0.242%	0.020%	10		2
	NIST	0.248%	0.007%	24		INAA
	USGS	0.250%	0.013%	16		ICP-MS
	USGS	0.258%	0.012%	16		ICP-AES
Certificate Value	**Certified**	**0.25% ± 0.02%**				

Element	Lab No.	Average	1s	n	z-score	Method
Cerium (mg/kg)	Lab 13b	0.74	0.10	3	57.3	INAA
	Lab 16	0.0783	0.0015	7	-2.9	INAA
	Lab 18	0.097	0.025	6	-1.2	INAA
	Lab 20	0.0981	0.0057	3	-1.1	INAA
	Lab 3	0.114	0.017	2	0.4	INAA
	Lab 6	0.119	0.028	4	0.8	INAA
	Lab 8	<0.16		6		INAA
all ASTM data	Avg ± 1s	0.208	0.261	6		number rejected
after rejection	Avg ± 1s	0.101	0.016	5		1
	NIST	0.11	0.01	10		INAA

Certificate Value **Information** **0.11**

Element	Lab No.	Average	1s	n	z-score	Method
Cesium (mg/kg)	Lab 13a	0.270	0.017	3	-0.5	INAA
	Lab 13b	0.293	0.023	3	0.4	INAA
	Lab 16	0.1991	0.0096	8	-3.0	INAA
	Lab 17	0.28	0.01	3	-0.1	INAA
	Lab 19	0.289	0.009	6	0.2	INAA
	Lab 20	0.266	0.014	3	-0.6	INAA
	Lab 21	0.276	0.012	6	-0.2	INAA
	Lab 3	0.30	0.01	2	0.6	INAA
	Lab 6	0.286	0.012	4	0.1	INAA
	Lab 8	0.30	0.03	6	0.6	INAA
all ASTM data	Avg ± 1s	0.276	0.029	10		number rejected
after rejection						0
	NIST	0.283	0.003	10		INAA

Certificate Value **Reference** **0.283 ± 0.009**

Element	Lab No.	Average	1s	n	z-score	Method
Chlorine (mg/kg)	Lab 17	391	15	3	-0.7	INAA
	Lab 24	382	32	4	-0.9	INAA
	Lab 3	389	17	2	-0.8	INAA
	Lab 6	396	21	4	-0.6	INAA
	Lab 8	438	44	6	0.4	INAA
all ASTM data	Avg ± 1s	399	22	5		number rejected
after rejection						0
	NIST	417	4.9	8		PGAA
	NIST	425	7.6	24		INAA
Certificate Value	**Certified**	**421 ± 7**				

Element	Lab No.	Average	1s	n	z-score	Method
Chromium (mg/kg)	Lab 13a	1.33	0.33	6	29.9	INAA
	Lab 13b	2.37	0.2	3	61.0	INAA
	Lab 15b	0.32	0.06	2	-0.4	FAAS/GFAAS
	Lab 16	0.268	0.006	9	-2.0	INAA
	Lab 17	0.37	0.02	3	1.1	INAA
	Lab 19	0.366	0.02	6	1.0	INAA
	Lab 20	0.365	0.020	3	0.9	INAA
	Lab 3	0.36	0.03	2	0.8	INAA
	Lab 6	0.290	0.051	4	-1.3	INAA
	Lab 8	0.33	0.1	6	-0.1	INAA
all ASTM data	Avg ± 1s	0.637	0.686	10		number rejected
after rejection		0.334	0.039	8		2
	NIST	0.375	0.065	10		INAA
	USGS	0.32	0.02	16		ICP-AES
	USGS	0.39	0.03	16		ICP-MS
Certificate Value	**Information**	**0.03 - 0.05**				

Element	Lab No.	Average	1s	n	z-score	Method
Cobalt (mg/kg)	Lab 10	0.0598	0.0048	3	-0.2	INAA
	Lab 13a	0.501	0.117	3	72.1	INAA
	Lab 13b	0.541	0.130	3	78.7	INAA
	Lab 15b	<0.035		1		FAAS/GFAAS
	Lab 16	0.0452	0.0028	8	-2.6	INAA
	Lab 17	0.062	0.003	3	0.2	INAA
	Lab 18	0.060	0.004	6	-0.2	INAA
	Lab 19	0.065	0.001	6	0.7	INAA
	Lab 20	0.0645	0.0037	3	0.6	INAA
	Lab 21	0.0628	0.0030	6	0.3	INAA
	Lab 3	0.074	0.003	2	2.1	INAA
	Lab 4	0.0686	0.0064	6	1.2	INAA
	Lab 6	0.060	0.005	4	-0.2	INAA
	Lab 8	0.067	0.010	6	1.0	INAA
all ASTM data	Avg ± 1s	0.133	0.172	13		number rejected
after rejection		0.063	0.007	11		2
	NIST	0.0614	0.0008	10		INAA
Certificate Value	**Reference**	**0.061 ± 0.002**				

Element	Lab No.	Average	1s	n	z-score	Method
Copper (mg/kg)	Lab 15b	1.86	0.18	2	-3.4	FAAS/GFAAS
	Lab 23	3	0	3	0.7	E-6010
	Lab 3	3.9	0.6	2	3.9	INAA
all ASTM data	Avg ± 1s	2.920	1.022	3		number rejected
after rejection	only Lab 23	3	0	1		2
	NIST	2.89	0.03	8		RNAA
	USGS	2.70	0.17	16		ICP-AES
Certificate Value	**Certified**	**2.8 ± 0.2**				

Element	Lab No.	Average	1s	n	z-score	Method
Europium (mg/kg)	Lab 16	0.0011	0.0002	5		INAA
	Lab 20	0.00144	0.00012	3		INAA
	Lab 4	0.0017	0.0006	6		INAA
	Lab 6	<0.002		4		INAA
	Lab 8	<0.03		6		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Fluorine (mg/kg)	Lab 3	389	20	2		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Gallium (mg/kg)	Lab 3	<1		2		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Gold (mg/kg)	Lab 13a	0.00150	0.0004	3		INAA
	Lab 16	0.00070	0.0001	9		INAA
	Lab 20	0.00026	0.000042	3		INAA
	Lab 3	0.00056	0.00018	2		INAA
	Lab 8	0.00260	0.0005	6		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Hafnium (mg/kg)	Lab 13a	0.116	0.026	3		INAA
	Lab 13b	0.122	0.025	3		INAA
	Lab 16	0.0063	0.0007	7		INAA
	Lab 20	0.0128	0.0016	3		INAA
	Lab 3	0.014	0.02	2		INAA
	Lab 8	<0.015		6		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Indium (mg/kg)	Lab 3	<0.003		2		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Iodine (mg/kg)	Lab 3	<1.5		2		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Iridium (mg/kg)	Lab 3	<0.0001		2		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Iron (mg/kg)	Lab 13a	192	19	6	32	INAA
	Lab 13b	208	19	3	35	INAA
	Lab 13c	49.9		1	0.8	AAS
	Lab 15b	43.5	5.6	4	-0.5	FAAS/GFAAS
	Lab 16	32.4	0.1	3	-3.0	INAA
	Lab 17	45	3	3	-0.2	INAA
	Lab 18	42.7	4.6	6	-0.7	INAA
	Lab 19	47.3	1.3	6	0.3	INAA
	Lab 20	46.4	2.7	3	0.1	INAA
	Lab 21	46.2	1.5	6	0.0	INAA
	Lab 23	41	0	3	-1.1	E-6010
	Lab 3	51.4	2.2	2	1.2	INAA
	Lab 4	50.0	5.6	6	0.9	INAA
	Lab 6	43.3	3.0	4	-0.6	INAA
	Lab 8	48	6	6	0.4	INAA
all ASTM data	Avg ± 1s	65.8	54.8	15		number rejected
after rejection	Avg ± 1s	45.2	4.9	13		2
	NIST	45.85	0.45	10		INAA
	USGS	46.01	2.31	16		ICP-AES
Certificate Value	**Certified**	**46 ± 2**				

Element	Lab No.	Average	1s	n	z-score	Method
Lanthanum (mg/kg)	Lab 13a	0.335	0.022	3	53.0	INAA
	Lab 13b	0.389	0.03	3	63.1	INAA
	Lab 16	0.0416	0.0049	8	-2.2	INAA
	Lab 18	0.049	0.011	6	-0.8	INAA
	Lab 19	0.050	0.004	6	-0.6	INAA
	Lab 20	0.0479	0.0028	3	-1.0	INAA
	Lab 24	0.222	0.070	4	31.7	INAA
	Lab 3	0.0565	0.0050	2	0.6	INAA
	Lab 4	0.0565	0.0057	6	0.6	INAA
	Lab 6	0.066	0.012	4	2.4	INAA
	Lab 8	0.058	0.009	6	0.9	INAA
all ASTM data	Avg ± 1s	0.125	0.128	11		number rejected
after rejection	Avg ± 1s	0.053	0.008	8		3

Element	Lab No.	Average	1s	n	z-score	Method
Lead (mg/kg)	Lab 15b	0.115	0.031	4	-3.1	FAAS/GFAAS
	NIST	0.167	0.013	6		ICP-MS
Certificate Value	**Reference**	**0.167 ± 0.015**				

Element	Lab No.	Average	1s	n	z-score	Method
Lutecium (mg/kg)	Lab 16	0.0006	0.0001	7		INAA
	Lab 20	0.00055	0.00006	3		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Magnesium (%)	Lab 13c	0.110%			0.4	AAS
	Lab 15b	0.123%	0.009%	4	1.6	FAAS/GFAAS
	Lab 17	0.096%	0.003%	3	-0.9	INAA
	Lab 23	0.100%	0.001%	3	-0.6	E-6010
	Lab 24	0.062%	0.027%	1	-4.2	INAA
	Lab 3	0.109%	0.012%	2	0.3	INAA
	Lab 6	0.096%	0.018%	4	-1.0	INAA
	Lab 8	0.102%	0.017%	6	-0.3	INAA
all ASTM data	Avg ± 1s	0.100%	0.018%	8		number rejected
after rejection	Avg ± 1s	0.105%	0.010%	7		1
	NIST	0.1058%	0.0074%	22		INAA
Certificate Value	**Reference**	**0.106% ± 0.017%**				

Element	Lab No.	Average	1s	n	z-score	Method
Manganese (mg/kg)	Lab 13a	506	47	4	0.4	INAA
	Lab 13c	309			-3.7	AAS
	Lab 15a	452	13	1	-0.7	AAS
	Lab 15b	405	40	4	-1.7	FAAS/GFAAS
	Lab 17	474	15	3	-0.3	INAA
	Lab 23	473	2	3	-0.3	E-6010
	Lab 24	436	14	4	-1.1	INAA
	Lab 3	502	16	2	0.3	INAA
	Lab 6	475.1	6.4	4	-0.3	INAA
	Lab 8	465	46	6	-0.5	INAA
all ASTM data	Avg ± 1s	449.7	57.6	10		number rejected
after rejection	Avg ± 1s	465.3	31.4	9		1
	NIST	488	6	24		INAA
Certificate Value	**Reference**	**488 ± 8**				

Element	Lab No.	Average	1s	n	z-score	Method
Mercury (mg/kg)	Lab 16	0.0035	0.0003	6	-9.1	INAA
	Lab 20	0.0391	0.0028	3	-2.7	INAA
	Lab 3	0.029	0.007	2	-0.2	INAA
all ASTM data	Avg ± 1s	0.024	0.018	3		number rejected
after rejection	Avg ± 1s	0.034	0.007	2		1
	NIST	0.03993	0.00041	5		CV-IDMS
	NIST	0.0414	0.0029	7		RNAA
Certificate Value	**Certified**	**0.0399 ± 0.0007**				

Element	Lab No.	Average	1s	n	z-score	Method
Molybdenum (mg/kg)	Lab 10	0.0124	0.0012	4		INAA
	Lab 24	<0.4		4		INAA
	Lab 3	<0.1		2		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Neodymium (mg/kg)	Lab 16	0.0283		2		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Nickel (mg/kg)	Lab 15b	1.10	0.04	2	-2.5	FAAS/GFAAS
	Lab 16	0.9	0.1	6	-3.9	INAA
	Lab 24	1.54	0.21	4	0.5	INAA
	Lab 3	<1.3		2		INAA
	Lab 8	1.29	0.19	6	-1.2	INAA
all ASTM data	Avg ± 1s	1.208	0.273	4		number rejected
after rejection	Avg ± 1s	1.310	0.221	3		1
	NIST	1.475	0.095	6		ICP-MS
Certificate Value	**Reference**	**1.47 ± 0.10**				

Element	Lab No.	Average	1s	n	z-score	Method
Nitrogen (%) [TKN]	Lab 23	1.11%	0.01%	5		ASTM#1402-07

Element	Lab No.	Average	1s	n	z-score	Method
Phosphorus (%)	Lab 13a	0.112%	0.009%	10	0.5	INAA
	Lab 23	0.124%	0.001%	5	1.6	ASTM#1402-07
all ASTM data	Avg ± 1s	0.118%	0.849%	2		number rejected
after rejection	Avg ± 1s	0.118%	0.849%	2		0
	NIST	0.1023%	0.0020%	10		RNAA
	USGS	0.1120%	0.0050%	16		ICP-AES
Certificate Value	**Certified**	**0.107% ± 0.008%**				

Element	Lab No.	Average	1s	n	z-score	Method
Potassium (%)	Lab 13a	0.431%	0.048%	7	0.3	INAA
	Lab 13b	0.467%	0.096%	3	1.2	INAA
	Lab 13c	0.289%			-3.1	AAS
	Lab 15a	0.398%	0.078%	1	-0.5	AAS
	Lab 15b	0.44%	0.02%	4	0.6	FAAS/GFAAS
	Lab 16	0.59%	0.17%	3	4.1	INAA
	Lab 17	0.408%	0.015%	3	-0.2	INAA
	Lab 18	0.402%	0.032%	6	-0.4	INAA
	Lab 19	0.383%	0.003%	6	-0.8	INAA
	Lab 20	0.422%	0.019%	3	0.1	INAA
	Lab 21	0.397%	0.006%	6	-0.5	INAA
	Lab 23	0.393%	0.003%	3	-0.6	E-6010
	Lab 24	0.435%	0.003%	4	0.4	INAA
	Lab 3	0.404%	0.015%	2	-0.3	INAA
	Lab 6	0.3971%	0.0081%	4	-0.5	INAA
	Lab 8	0.426%	0.085%	6	0.2	INAA
all ASTM data	Avg ± 1s	0.418%	0.060%	16		number rejected
after rejection	Avg ± 1s	0.415%	0.023%	14		2
	NIST	0.4137%	0.0037%	8		PGAA
	NIST	0.4209%	0.0128%	24		INAA
Certificate Value	**Certified**	**0.417% ± 0.007%**				

Element	Lab No.	Average	1s	n	z-score	Method
Rubidium (mg/kg)	Lab 10	16.18	1.62	3	-0.2	INAA
	Lab 13a	16.4	0.5	3	-0.1	INAA
	Lab 13b	17.2	1.3	3	0.4	INAA
	Lab 16	0.0115	0.0004	8	-10.0	INAA
	Lab 17	16.2	0.05	3	-0.2	INAA
	Lab 18	15.3	1.6	6	-0.7	INAA
	Lab 19	16.1	0.2	6	-0.2	INAA
	Lab 20	16.73	0.76	3	0.1	INAA
	Lab 21	17.0	0.5	6	0.3	INAA
	Lab 3	16.4	0.5	2	-0.1	INAA
	Lab 4	15.8	2.8	6	-0.4	INAA
	Lab 6	17.1	0.5	4	0.4	INAA
	Lab 8	18.1	1.8	6	1.0	INAA
all ASTM data	Avg ± 1s	15.3	4.6	13		number rejected
after rejection	Avg ± 1s	16.5	0.7	12		1
	NIST	16.55	0.143	10		INAA
	USGS	16.36	0.81	16		ICP-MS
Certificate Value	**Certified**	**16.5 ± 0.9**				

Element	Lab No.	Average	1s	n	z-score	Method
Samarium (mg/kg)	Lab 13b	0.0420	0.003	3	44.5	INAA
	Lab 16	0.0059	0.0008	9	-2.3	INAA
	Lab 18	0.0078	0.0008	6	0.1	INAA
	Lab 20	0.0070	0.00044	3	-1.0	INAA
	Lab 24	0.0090	0.002	4	1.7	INAA
	Lab 3	0.0078	0.0004	2	0.1	INAA
	Lab 4	0.0080	0.00071	6	0.3	INAA
	Lab 6	0.0080	0.0016	4	0.4	INAA
	Lab 8	0.0082	0.0008	6	0.6	INAA
all ASTM data	Avg ± 1s	0.012	0.011	9		number rejected
after rejection	Avg ± 1s	0.0077	0.0009	8		1

Element	Lab No.	Average	1s	n	z-score	Method
Scandium (mg/kg)	Lab 10	0.0117	0.0009	3	1.6	INAA
	Lab 13a	0.097	0.012	3	86.0	INAA
	Lab 13b	0.098	0.009	3	87.0	INAA
	Lab 16	0.0074	0.0001	7	-2.7	INAA
	Lab 17	0.0108	0.0004	3	0.7	INAA
	Lab 18	0.0086	0.0007	6	-1.5	INAA
	Lab 19	0.0110	0.0004	6	0.9	INAA
	Lab 20	0.0104	0.0006	3	0.3	INAA
	Lab 21	0.0103	0.0002	6	0.2	INAA
	Lab 3	0.0116	0.0005	2	1.5	INAA
	Lab 4	0.0106	0.0008	6	0.5	INAA
	Lab 6	0.0097	0.0005	4	-0.4	INAA
	Lab 8	0.0126	0.0025	6	2.5	INAA
all ASTM data	Avg ± 1s	0.024	0.033	13		number rejected
after rejection	Avg ± 1s	0.0104	0.0015	11		2
	NIST	0.01006	0.00015	10		INAA
Certificate Value	**Reference**	**0.0101 ± 0.0003**				

Element	Lab No.	Average	1s	n	z-score	Method
Selenium (mg/kg)	Lab 13b	0.017	0.002	3	-8.3	INAA
	Lab 16	0.0696	0.0066	8	-3.0	INAA
	Lab 20	0.0985	0.0063	3	-0.1	INAA
	Lab 3	0.13	0.02	2	3.1	INAA
	Lab 4	0.113	0.010	6	1.4	INAA
	Lab 8	0.13	0.03	6	3.1	INAA
all ASTM data	Avg ± 1s	0.093	0.044	6		number rejected
after rejection	Avg ± 1s	0.094	0.022	3		3
	NIST	0.099	0.004	10		INAA
Certificate Value	**Reference**	**0.099 ± 0.004**				

Element	Lab No.	Average	1s	n	z-score	Method
Silver (mg/kg)	Lab 15b	<0.05		1		FAAS/GFAAS
	Lab 16	0.0118		2		INAA
	Lab 3	<0.03		2		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Sodium (mg/kg)	Lab 13a	111	36	7	7.6	INAA
	Lab 13c	133			11.1	AAS
	Lab 15a	73.2	5.5	1	1.6	AAS
	Lab 15b	52.6	5.0	4	-1.7	FAAS/GFAAS
	Lab 16	72.5	9.3	4	1.5	INAA
	Lab 17	67	3	3	0.6	INAA
	Lab 18	61	5	6	-0.3	INAA
	Lab 19	57	1	6	-0.9	INAA
	Lab 20	64.7	3.1	3	0.3	INAA
	Lab 21	61.8	2.0	6	-0.2	INAA
	Lab 24	93.0	4.1	4	4.8	INAA
	Lab 3	63.0	2.4	2	0.0	INAA
	Lab 6	56.1	1.0	4	-1.1	INAA
all ASTM data	Avg ± 1s	74.3	23.9	13		number rejected
after rejection	Avg ± 1s	62.9	6.7	10		3
	NIST	62.7	1.63	24		INAA
Certificate Value	**Reference**	**63 ± 1**				

Element	Lab No.	Average	1s	n	z-score	Method
Strontium (mg/kg)	Lab 16	4.9	0.7	4	-2.8	INAA
	Lab 20	6.76	0.41	3	-0.1	INAA
	Lab 3	7.2	1.3	2	0.5	INAA
	Lab 8	8.5	0.8	6	2.4	INAA
all ASTM data	Avg ± 1s	6.8	1.5	4		number rejected
after rejection	Avg ± 1s					0

Element	Lab No.	Average	1s	n	z-score	Method
Sulfur (%)	Lab 23	0.102%	0.001%	3		ASTM#1552-90

Element	Lab No.	Average	1s	n	z-score	Method
Tantalum (mg/kg)	Lab 16	0.0011	0.0001	5		INAA
	Lab 20	0.00135	0.00012	3		INAA
	Lab 3	<0.005		1		INAA
	Lab 8	0.0028	0.0007	6		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Terbium (mg/kg)	Lab 16	0.0008	0.0001	4		INAA
	Lab 20	0.00053	0.00010	1		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Thorium (mg/kg)	Lab 13a	0.205	0.032	3	118	INAA
	Lab 13b	0.195	0.029	3	112	INAA
	Lab 16	0.010	0.0002	7	-3.8	INAA
	Lab 20	0.0142	0.0007	3	-1.1	INAA
	Lab 3	0.016	0.002	2	0.0	INAA
	Lab 6	0.0178	0.0055	4	1.1	INAA
	Lab 8	0.016	0.002	6	0.0	INAA
all ASTM data	Avg ± 1s	0.068	0.0905	7		number rejected
after rejection	Avg ± 1s	0.016	0.001	4		3

Element	Lab No.	Average	1s	n	z-score	Method
Tungsten (mg/kg)	Lab 8	0.065	0.019	6		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Uranium (mg/kg)	Lab 10	0.0071	0.0011	4		INAA
	Lab 16	0.0122		2		INAA
	Lab 24	<0.08		4		INAA
	Lab 3	<0.04		1		INAA
	Lab 8	0.0068	0.0014	6		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Vanadium (mg/kg)	Lab 24	<0.34		4		INAA
	Lab 24	<0.08		4		INAA
	Lab 3	<0.26		2		INAA
	Lab 8	<0.27		6		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Ytterbium (mg/kg)	Lab 13b	0.025	0.005	3		INAA
	Lab 16	0.0033	0.0007	6		INAA
	Lab 20	0.00215	0.00026	3		INAA

Element	Lab No.	Average	1s	n	z-score	Method
Zinc (mg/kg) A334	Lab 10	39.3	2.0	3	0.3	INAA
	Lab 13a	51.5	2.9	3	3.6	INAA
	Lab 13b	50.4	7.0	3	3.3	INAA
	Lab 13c	49.9			3.1	AAS
	Lab 15b	29.9	3	4	-2.1	FAAS/GFAAS
	Lab 16	27.3	0.3	9	-2.8	INAA
	Lab 17	38.3	2.0	3	0.1	INAA
	Lab 18	34.7	3.1	6	-0.9	INAA
	Lab 19	37.2	0.5	6	-0.2	INAA
	Lab 20	37.6	2.0	3	-0.1	INAA
	Lab 21	36.5	0.6	6	-0.4	INAA
	Lab 23	38	0	3	0.0	E-6010
	Lab 3	38.5	2.0	2	0.1	INAA
	Lab 4	40.2	2.5	6	0.6	INAA
	Lab 6	37.6	1.0	4	-0.1	INAA
	Lab 8	38.2	3.8	6	0.1	INAA
all ASTM data	Avg ± 1s	39.1	6.6	16		number rejected
after rejection	Avg ± 1s	36.4	3.7	13		3
	NIST	38.0	0.2	10		INAA
	USGS	37.7	1.8	16		ICP-AES
Certificate Value	**Certified**	**38 ± 2**				

Isotope	Lab No.	Average	1s	n	z-score	Method
U-238	Lab 2	0.00490	0.00083	6		Isotope Analysis
Th-232	Lab 2	0.0270	0.0055	6		Isotope Analysis