



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2790

Inorganic Constituents in Hardwood Biomass Material

This Standard Reference Material (SRM) is intended for use in validating methods for the determination of elements in hardwood biomass materials and similar matrices, and for qualifying in-house control materials. A unit of SRM 2790 consists of two bottles each containing approximately 30 g of material.

Certified Mass Fraction Values: Certified mass fraction values of elements in SRM 2790, reported on a dry-mass basis, and ash are provided in Tables 1 and 2, respectively. 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 [1]. Analyses for value assignment were performed by NIST. Certified mass fraction values were calculated as the mean of the mean values from NIST methods. The associated uncertainties are expressed at an approximately 95 % level of confidence [2–4].

Reference Mass Fraction Values: Reference mass fraction values for additional elements in SRM 2790, reported on a dry-mass basis, are provided in Table 3. A NIST reference mass fraction value is a noncertified value that is the best estimate of the true value based on available data; however, the values do not meet the NIST criteria for certification [1] and are provided with an uncertainty that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. The reference mass fraction values were derived from results reported by NIST.

Expiration of Certification: The certification of **SRM 2790** is valid, within the measurement uncertainty specified, until **30 June 2028**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Storage and Use”). The certification is nullified if the SRM is damaged, 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 or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was performed by L.J. Wood and R. Oflaz of the NIST Chemical Sciences Division.

Analytical measurements at NIST were performed by C.A. Barber, J.F. Browning, K.D. Chieh, S.E. Long, A.F. Marlow, A.J. Moors, J.M. Ness, R. Oflaz, R.L. Paul, R.S. Pugh, N.E. Sharp, and L.J. Wood of the NIST Chemical Sciences Division.

Statistical analysis was provided by J.H. Yen of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Certificate Issue Date: 11 June 2018

Steven J. Choquette, Director
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NOTICE TO USERS

SRM 2790 IS INTENDED FOR RESEARCH USE.

INSTRUCTIONS FOR STORAGE AND USE

Storage: The SRM should be stored in the original, unopened bottle at room temperature (20 °C to 25 °C) until required for use. The bottle can be closed and test portions removed and analyzed until the material reaches its expiration date.

Use: Prior to removal of a test portion for analysis, the contents of the bottle should be mixed thoroughly. Allow the contents to settle for one minute prior to opening to minimize the loss of fine particles. To relate analytical determinations to the certified values in this Certificate of Analysis, a test portion mass size of at least 0.2 g should be used when determining Ag, Al, As, Ce, Cl, Co, Cr, Cs, Fe, K, La, Mg, Mn, Na, Rb, Sb, Sc, Se, Sm, Th, V, W, and Zn; a test portion mass size of at least 0.25 g should be used when determining Hg; a test portion mass size of at least 0.5 g should be used when determining Cu; and a test portion mass size of at least 1 g should be used when determining B, C, H, and ash. Test portions should be analyzed as received and results converted to a dry-mass basis (See “Determination of Moisture”). Analytical results obtained in analyses should include their own estimates of uncertainty and can be compared to the listed values using procedures described in references 5 and 6.

Determination of Moisture: Moisture content of SRM 2790 was determined at NIST by (1) freeze-drying to constant mass over 7 d; (2) drying over magnesium perchlorate in a desiccator at room temperature for 14 d; and (3) drying in a forced air oven for 2 h at 90 °C. Unweighted results obtained using all three techniques were averaged to determine a conversion factor of (0.9700 ± 0.0072) gram dry-mass per gram as-received mass, which was used to convert data from an as-received to a dry-mass basis; the uncertainty shown on this value is an expanded uncertainty to represent a 95 % level of confidence. An uncertainty component for the conversion factor (0.4 %) obtained from the moisture measurements is incorporated in the uncertainties of the certified and reference values, reported on a dry-mass basis, that are provided in this certificate.

SOURCE, PREPARATION, AND ANALYSIS⁽¹⁾

Preparation: SRM 2790 was prepared from wood pellet fuel to support biomass energy, biomass technology developments, and environmental impact assessments. The material was purchased commercially as five 1 cu. ft. bags that were sent to the NIST Reference Material Production facility (ISO certified Class 7 clean room) in Charleston, SC. The material was first cryomilled and then cryohomogenized. The material was shipped to NIST Gaithersburg for jet milling, irradiation (Neutron Products, Inc., Dickerson, MD) to an absorbed dose of 7.9 kGy to 9.5 kGy, and bottling.

NIST Analytical Approach for Determination of Elements: Value assignment of the mass fractions of elements in SRM 2790 was based on the combination of measurements provided by NIST using inductively couple plasma mass spectrometry (ICP-MS), inductively couple plasma optical emission spectrometry (ICP-OES), isotope dilution cold vapor inductively coupled plasma mass spectrometry (ID/CV ICP-MS), instrumental neutron activation analysis (INAA), and prompt gamma neutron analysis (PGAA).

NIST Analyses for As, Cr, Co, Cs, Cu, Fe, K, Mg, Mn, Na, Se, V, and Zn using ICP-MS and ICP-OES: Mass fractions of arsenic, cesium, chromium, cobalt, copper, iron, magnesium, manganese, potassium, selenium, sodium, vanadium, and zinc were determined by either ICP-MS or ICP-OES in duplicate 0.5 g test portions taken from each of six bottles of SRM 2790. Samples were digested in a microwave sample preparation system using a HNO₃:HF solution. Quantification was based on the method of standard additions using calibration solutions prepared from the SRM 3100 series of single-element standard solutions.

NIST Analyses for Hg using ID/CV ICP-MS: The mass fraction of mercury was determined by ID/CV ICP-MS in single, nominal 0.25 g, test portions taken from each of eight bottles of SRM 2790. Samples were digested in a microwave sample preparation system using a HNO₃:HCl solution. Measurements were made using cold-vapor mercury generation coupled with ICP-MS. Quantification was based on reverse isotope dilution with a ²⁰¹Hg stock solution and SRM 1641d *Mercury in Water*.

NIST Analyses for Ag, Al, As, Ce, Cl, Co, Cr, Cs, Fe, K, La, Mg, Mn, Na, Rb, Sb, Sc, Se, Sm, Th, V, W, and Zn using INAA: Mass fractions of aluminum, antimony, arsenic, cerium, cesium, chlorine, chromium, cobalt, iron, lanthanum,

⁽¹⁾ Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

magnesium, manganese, potassium, rubidium, samarium, scandium, selenium, silver, sodium, thorium, tungsten, vanadium, and, zinc were determined by INAA using duplicate, nominal 0.2 g test portions taken from each of 12 bottles of SRM 2790. For the analysis of antimony, arsenic, cerium, cesium, chromium, cobalt, iron, lanthanum, rubidium, samarium, scandium, selenium, silver, thorium, tungsten, and zinc, powders were pressed into cylindrical pellets, and samples, standards, and controls were packaged individually in clean polyethylene bags and irradiated in a single rabbit at 20 MW for 16 h with a 180-degree inversion after 8 h. Nuclides were counted for 12 h after a 14 d decay (Ag, Ce, Co, Cr, Cs, Fe, Rb, Sb, Sc, Se, Th, Zn) or 2 h after a 5 d decay (As, La, Sm, W). For analysis of aluminum, chlorine, magnesium, manganese, potassium, sodium, and vanadium each sample, standard, or control material was individually irradiated together with one flux monitor foil for 120 s at a reactor power of 20 MW. Nuclides were counted for 5 min and 30 min after decays of 5 min and 15 min, respectively. Quantification is based on validated pure metal foils, pure compounds, or the SRM 3100 single element standard solutions.

NIST Analyses for B, C, K, and H using PGAA: Mass fractions of boron, carbon, potassium, and hydrogen were determined by PGAA using single, nominal 1 g test portions taken from each of eight bottles of SRM 2790. Samples were irradiated for 24 h. Gamma rays were measured using a reverse electrode high purity germanium detector. The 2223 keV, 477 keV, 770 keV, and 4945 keV peaks were integrated for hydrogen, boron, potassium, and carbon, respectively. Quantification was based on SRM 912a *Urea*, SRM 918b *Potassium Chloride Clinical Standard*, and SRM 3107 *Boron (B) Standard Solution*.

NIST Analytical Approach for Determination of Ash: Value assignment of the mass fraction of ash in SRM 2790 was based on the combination of measurements provided by NIST using thermogravimetric analyzer (TGA) (LECO Model 701) and muffle furnace. Nominal 1 g samples were held for 3 h at 575 °C using either TGA or a muffle furnace. Quantitation was based on the standard mass set maintained by the Inorganic Chemical Metrology Group.

Homogeneity Assessment: To address issues of possible inhomogeneity of the SRM, analyses of variance with 5 % significance level and graphical analyses were run on NIST data where box information was available. For As, La, Mn, Na, Sm, and W, the uncertainty incorporates a component for possible inhomogeneity based on the standard deviation.

Value Assignment: The certified mass fraction values in this SRM were derived from results of analyses performed by NIST using the mean of the mean values from NIST results. The uncertainty of the combined mean is estimated using a bootstrap procedure based on a Gaussian random effects model for the between-method effects [2–4].

Certified Mass Fraction Values: Each certified mass fraction value listed in Table 1 is the combined mean from each set of analyses by NIST. Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty of the certified value. The true value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with 95 % confidence. To propagate this uncertainty, treat the certified value as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/2$ [2–4]. The measurand is the total mass fraction for each analyte listed in Table 1, on a dry-mass basis. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram).

Table 1. Certified Mass Fraction Values for Elements in SRM 2790

	Mass Fraction (mg/kg)		
Arsenic (As) ^(a,b)	0.0335	±	0.0089
Cesium (Cs) ^(a,b)	0.0199	±	0.0010
Chromium (Cr) ^(a,b)	1.59	±	0.15
Cobalt (Co) ^(a,b)	0.098	±	0.012
Iron (Fe) ^(b,c)	82.9	±	5.5
Magnesium (Mg) ^(b,c)	189	±	11
Manganese (Mn) ^(b,c)	52.4	±	1.9
Mercury (Hg) ^(d)	0.01323	±	0.00020
Potassium (K) ^(b,c,e)	1040	±	130
Selenium (Se) ^(a,b)	0.0102	±	0.0023
Sodium (Na) ^(b,c)	26.0	±	5.3
Vanadium (V) ^(a,b)	0.243	±	0.011
Zinc (Zn) ^(b,c)	9.26	±	0.90

^(a) ICP-MS

^(b) INAA

^(c) ICP-OES

^(d) ID/CV ICP-MS

^(e) PGAA

Certified Mass Fraction Value for Ash: The certified mass fraction value for ash is the mean of the means from the sets of analyses by NIST using TGA and muffle furnace. Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty of the certified value. The true value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with 95 % confidence [2]. To propagate this uncertainty, treat the certified value as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/2$ [2–4]. The measurand is the total mass fraction for ash listed in Table 2. Metrological traceability is to the SI derived unit for mass fraction (expressed as grams per 100 grams).

Table 2. Certified Mass Fraction Value for Ash in SRM 2790

	Mass Fraction (g/100 g)		
Ash	0.604	±	0.084

Reference Mass Fraction Values for Elements: Each reference mass fraction value is the mean from each set of analyses by NIST. Values are expressed as $x \pm U_{95\%}(x)$, where x is the reference value and $U_{95\%}(x)$ is the expanded uncertainty of the value. The method-specific value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with about a 95 % confidence [2–4]. The measurand is the mass fraction for each element listed in Table 3, on a dry-mass basis, as determined by the method indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as either percent or milligrams per kilogram) as realized by the method used.

Table 3. Reference Mass Fraction Values for Elements in SRM 2790

	Mass Fraction (%)		
Carbon (C) ^(a)	51.73	±	0.75
Hydrogen (H) ^(a)	6.291	±	0.088
	Mass Fraction (mg/kg)		
Aluminum (Al) ^(b)	92.06	±	0.89
Antimony (Sb) ^(b)	0.0397	±	0.0016
Boron (B) ^(a)	3.210	±	0.039
Cerium (Ce) ^(b)	0.5523	±	0.0098
Chlorine (Cl) ^(b)	35.40	±	0.64
Copper (Cu) ^(c)	1.545	±	0.030
Lanthanum (La) ^(b)	0.643	±	0.063
Rubidium (Rb) ^(b)	3.11	±	0.099
Samarium (Sm) ^(b)	0.0507	±	0.0052
Scandium (Sc) ^(b)	0.0111	±	0.0004
Silver (Ag) ^(b)	0.0079	±	0.0010
Thorium (Th) ^(b)	0.0146	±	0.0005
Titanium (Ti) ^(b)	11.1	±	1.0
Tungsten (W) ^(b)	0.225	±	0.029

^(a) PGAA

^(b) INAA

^(c) ICP-OES

REFERENCES

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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: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at <https://www.nist.gov/srm>.