METAL Best Practices

Version 1 (1/12/21)

Data is only as good as the sample preparation. The best analytical measurements are useless if samples are poorly prepared. While lab staff will make every effort to assist users to implement best practices, lab staff will not be aware of all details of the accuracy, precision, and importance of different measurements to meet your analytical goals. Hence it is the **USER'S RESPONSIBILITY** to make sure your data is of sufficient quality to answer your scientific question.

∞ *See Appendices for Certified Standard Selections, Abbreviations & Definitions* ∞

Universal Best Practices

- Samples that produce overrange data should be diluted and re-run to fall within the calibration curve
- Detection limits established and abided by
- Second (independent) source quality control used as verification
- 10% of all samples run in triplicate when possible

Elemental concentrations by ICP-MS:

Minimum requirements for publishable data:

- Gravimetric determinations of sample weight at all procedural steps
- Sample digestion procedure, optimized and validated for sample type
- Proper sample dilution, optimized for instrument capability and elements of interest
- Suitable calibration standards, traceable to NIST standards
- Samples should be bracketed between calibration standards for elements of interest
- Suitable internal standard (preferably multi-element) added online to all samples, standards, and blanks
- Process blank carried through entire sample preparation protocol
- We strongly recommend data is archived close to the time of analysis through the KFLEB online database for traceability and audit purposes (not required in case of concerns about commercial proprietary information)

Better practices:

- Certified matrix-matched standards prepared in parallel with samples
- 10% of all samples prepared in triplicate (ideally, beginning with sample digestion)

Best practices:

- Process blanks should be processed with every batch of samples
- Certified matrix-matched standards (preferably at least two different standards) should be processed in parallel with every batch of samples. If certified matrix-matched standards are not available, they may be made by distributing in-house standards to at least two other reputable laboratories, or modifying certified standards by addition of known amounts of elements to more closely mimic anticipated sample elemental composition and ratios.

- Samples' and standards' identity should be blinded to the analyst. Data should not be re-associated with sample identity until *after* data processing is complete.
- Data should be reported with sufficient information for an outside researcher to replicate the analysis. This should include details of sample processing, accuracy and reproducibility of analytical standards, accuracy and reproducibility of standards processed in parallel with samples, and reproducibility of samples processed and analyzed in replicate.

Elemental Concentrations by ICP-OES

- Matrix matched standards and samples
- Samples are particulate free
- Samples are acidified according to elements of interest
- Acidified samples can be stored at room temperature, taking into consideration specific elemental stability in that matrix
- Internal standard (appropriate selection for samples)
- Light path is optimized for elements and wavelengths of interest (radial, axial, SVDV)
- If analyzing digests:
    - Certified QC material digested with sample run as recovery check in duplicate (triplicate preferred if possible)
        - QC material selected includes certified values for one or several elements of interest
        - QC selected is of similar material
    - Blanks digested with sample run (same acids) in duplicate (triplicate preferred if possible)
    - Proper sample weights used ensure complete digestion and target concentrations in range
    - Proper acids selected for complete digestion
- Wavelength calibration on instrument performed monthly (minimum)
- Wavelengths selected have been optimized for sample type
- Multiple wavelengths for element of similar concentration are best, but not always possible with interferences
- LODs & LOQs are determined
- CCV data is within 10% of known value (in some cases higher may be acceptable)

Isotope composition by MC-ICP-MS:

Note - Exact requirements will vary depending on element system and sample type

<u>Minimum</u> requirements for publishable data:

- Gravimetric determinations of sample weight at all important procedural steps (select exceptions exist)
- Sample digestion procedure, optimized and validated for sample type
- Sample ion exchange purification procedure, optimized and validated for sample and element type
- High chemical recovery (yield) and efficient purification through ion exchange chemistry validated by Q-ICP-MS measurement of post-column samples (may not be required for very specific sample types and applications)
- Proper sample dilution, optimized for instrument capability and element of interest
- Proper amount of element spike or double spike added if needed
- Suitable certified calibration standards, traceable to NIST standards
- Matrix-matched analytical standards run as check standards within each run

- Samples should be diluted to within the calibrated concentration range for certified standards
- Samples should be verified to be within the calibrated range of spike-sample ratios for double-spike analyses (including Ca, Mo, U)
- Process blank carried through entire sample preparation protocol
- We strongly recommend data is archived close to the time of analysis through the KFLEB online database for traceability and audit purposes (not required in case of concerns about commercial proprietary information)

Better practices:

- Certified matrix-matched standards prepared in parallel with samples
- 10% of all samples prepared in triplicate (ideally, beginning with sample digestion) certified standards, matrix-doped to match typical samples, should be analyzed within each run

Best practices:

- Process blanks should be processed with every batch of samples
- Certified matrix-matched standards (preferably at least two different standards) should be processed in parallel with every batch of samples. If certified matrix-matched standards are not available, they may be made by distributing in-house matrix-matched standards to at least two other reputable laboratories or modifying certified standards by addition of known amounts of elements to more closely mimic anticipated sample elemental composition and ratios.
- Samples' and standards' identity should be blinded to the analyst. Data should not be re-associated with sample identity until *after* data processing is complete.
- Data should be reported with sufficient information for an outside researcher to replicate the analysis. This should include details of sample processing, accuracy and reproducibility of analytical standards, accuracy and reproducibility of standards processed in parallel with samples, and reproducibility of samples processed and analyzed in replicate.

CHN (Perkin Elmer) Combustion Analysis for Carbon, Hydrogen & Nitrogen:

- Sample is dried and milled to homogenous powder
- Microbalance used for sample weight to 0.0000mg
- Sample weight in target range for sample type
- Instrument passes full calibration using acetanilide standard 3X and as a sample
- Appropriate QC (NIST std) used for sample type and verified prior to analysis
- K factor (acetanilide standard) and blank run every 10-12 samples
- Samples run in duplicate when possible

Shimadzu TOC-V/TN:

Dissolved Organic Carbon

- Samples are filtered through minimum 0.7micron filter
- Samples acidified to pH ≤2 with HCl prior to analysis
- Samples should be analyzed as soon as possible after collection, and in the case they need to be stored, should be stored at ≤4°C or acidified (≤28 days recommended holding time for organic carbon analysis when samples are frozen or acidified)
- Calibration is linear
- Second source QC used as verification and run every 10 samples

Total Dissolved Nitrogen

- Samples are filtered through minimum 0.7micron filter
- Samples should be analyzed as soon as possible after collection, and in the case they need to be stored, should be stored at ≤4°C (≤28 days recommended holding time for nitrogen analysis)
- Calibration is linear
- Second source QC used as verification and run every 10 samples
- Digestion QC check to confirm organic N species

Inorganic Carbon

- Samples are collected in a headspace free sealed vial (no exposure to air)
- Calibration is linear
- Second source QC used as verification and run every 10 samples
- Samples should be analyzed as soon as possible after collection, and in the case they need to be stored, should be stored at ≤4°C (14 days recommended holding time for inorganic carbon analysis)

Total Carbon

- Samples are collected in a headspace free sealed vial (no exposure to air)
- Samples should be analyzed as soon as possible after collection, and in the case they need to be stored, should be stored at ≤4°C (14 days recommended holding time for total carbon analysis)
- Calibration is linear
- Second source QC used as verification and run every 10 samples

Lachat Flow Injection Analysis & Seal AQ2 Discrete Analyzer:

N as Nitrate/Nitrite

- For dissolved - Samples filtered through 45mm GF/F filter
- For totals (digested) - Digestion process blank and secondary source standard check run and verified
- Samples should be analyzed as soon as possible after collection, and in the case they need to be stored, recommended holding time prior to analysis is 30 days when stored at ≤4°C (longer holding times can be achieved by acidifying samples to <2 pH with H_2SO_4)
- Sample pH should be between 5 & 9 (use HCl or NH_4OH to adjust)
- Matrix matched standards (water, KCl, persulfate digestion matrix)

- Samples high in Cl are dechlorinated prior to analysis with sodium thiosulfate
- Cadmium coil/column is producing >85% efficiency
- Calibration shows linearity
- QCS is run 3X after calibration to verify accuracy/precision
- Instrument performance check solution (CCV) and blank run every 10 samples

P as o-Phosphate

- For dissolved - Samples filtered through 45mm GF/F filter
- For totals (digested) - Digestion process blank and secondary source standard check run and verified
- For $NaHCO_3$ soil extracts – pH adjust samples to between 2 – 5.5 (use H_2SO_4)
- Samples should be analyzed as soon as possible after collection, and in the case they need to be stored, recommended holding time prior to analysis is 30 days when stored at ≤4°C (longer holding times can be achieved by acidifying samples to <2 pH with H_2SO_4)
- All reagent glassware and sample containers should be acid washed with HCl
- Matrix matched standards (water, $NaHCO_3$, persulfate digestion matrix)
- Calibration shows linearity
- QCS is run 3X after calibration to verify accuracy/precision
- Instrument performance check solution (CCV) and blank run every 10 samples

N as Ammonia

- It is <u>not</u> recommended to filter these samples prior to analysis, but centrifuge them and decant off the top into a secondary container
- Samples should be analyzed as soon as possible after collection, and in the case they need to be stored, recommended holding time prior to analysis is 28 days when stored at ≤4°C (longer holding times can be achieved by acidifying samples to <2 pH with H_2SO_4)
- Samples high in Cl are dechlorinated prior to analysis with sodium thiosulfate

Chloride

- Samples filtered through 45mm GF/F filter
- Samples should be analyzed as soon as possible after collection, and in the case they need to be stored, recommended holding time prior to analysis is 28 days when stored at ≤4°C

Green House Gas GC (Methane, Carbon Dioxide & Nitrous Oxide):

- Samples should be analyzed as soon as possible. Depending on the container, gases can have a holding time of up to 30 days.
- Calibration re-made & run daily
- Collecting directly into the 6mL vials that are used on the instrument minimizes loss of gases through transfer. These are provided to customers when needed.

BET for Surface Area & Porosity:

- Samples are dried and purged with nitrogen minimum 4 hours, preferably overnight.
- Drying temp should be appropriate for sample type, between 50-400 C.
- Carbon Black & Silica Alumina certified standards should be run regularly to ensure instrument is running normally
- Surface area: minimum = 1 sq. meter, recommended = > 5 sq. Meters
- Typical sample between .1 and 1.0 grams
- Pore size: minimum = 1.7 nanometers, recommended = 20-200 nanometers
- LN dewar should be filled up to the indicator mark on the dipstick (Do NOT fill above the mark)
- An isothermal jacket and a filler rod should be used. An isothermal jacket is used to maintain a constant temperature profile along the sample tube stem during an extended analysis. A filler rod is used to reduce the stem free-space volume, resulting in reduction of free-space error.

Ion Chromatograph for Anions (in water)

- Calibration curve run with every sample set
- Secondary source QC run every 10-12 samples
- Autosampler vial rinsed with sample/standard prior to filling
- Samples that contain particles larger than 0.45 microns require filtration to prevent damage to instrument columns and flow systems
- Anions in water have a holding time of 28 days when stored at ≤4°C

Appendix A – *Abbreviations*

NIST = The National Institute of Standards and Technology is a physical sciences laboratory and a non-regulatory agency of the United States Department of Commerce.

QC = Quality Control, generally a secondary source standard used for calibration verification

CCV = Continuing Calibration Verification (AKA Check Standards), generally a standard level from the calibration run again throughout the sequence to check stability

Calibration Blank = A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.

Process Blank = A blank of the representative sample matrix that has undergone the same prep/digestion/extraction processes as the samples

Internal Standard = An analyte that is not present in the samples that is added in a constant amount to the blank, the standards, and the samples. Internal standards are useful to compensate for changes in extraction efficiency, detector response due to sample loss during other sample preparation steps, fluctuations in sample analyzed, or changes in detector response due to different flow rates

SVDV = Synchronous Vertical Dual View, refers to the specific light path on the Agilent 5900 ICP-OES which allows for simultaneous measuring of radial & axial wavelengths

K Factor = Acetanilide standard used on the CHN for calibration

LOD = Limit of Detection (calculated as 3σ of the mean of the blank), the concentration at which the instrument can differentiate a signal from the background noise

LOQ = Limit of Quantification (calculated as 10σ of the mean of the blank), the lowest concentration of the analyte that can not only be detected but can be quantified within defined limits of certainty

BEC = Blank Equivalent Concentration, the blank value expressed in concentration units. It is generally obtained by dividing the signal in counts/second (c/s) obtained when aspirating a blank by the slope of the calibration curve in c/s/ppt.

Appendix B – *Certified Natural Standard Materials Currently Available (ICP)*

NOTE: NOT ALL STANDARDS ARE CERTIFIED FOR ALL MEASUREMENTS. IF LARGE AMOUNTS OF STANDARD ARE REQUIRED FOR A PROJECT, AT THE DISCRETION OF LAB STAFF, USERS MAY BE REQUIRED TO PURCHASE THEIR OWN STANDARD FROM NIST, USGS, OR THE IAEA.

Rocks / Sediments

NIST 2710 – Montana soil, Highly elevated trace element concentrations

Certified for: 64400 ppm Al, 12500 ppm Ca, 33800 ppm Fe, 8530 ppm Mg, 10100 ppm Mn, 1060 ppm P, 21100 ppm K, 289700 ppm Si, 11400 ppm Na, 2400 ppm S, 2830 ppm Ti, 38.4 ppm Sb, 626 ppm As, 707 ppm Ba, 21.8 ppm Cd, 2950 ppm Cu, 5532 ppm Pb, 32.6 ppm Hg, 14.3 ppm Ni, 35.3 ppm Ag, 76.6 ppm V, 6952 ppm Zn

NIST 2711 – Montana Soil, Moderately elevated trace element concentrations

Certified for: 65300 ppm Al, 28800 ppm Ca, 28900 ppm Fe, 10500 ppm Mg, 860 ppm P, 24500 ppm K, 304400 ppm Si, 11400 ppm Na, 420 ppm S, 3060 ppm Ti, 19.4 ppm Sb, 105 ppm As, 726 ppm Ba, 41.70 ppm Cd, 114 ppm Cu, 1162 ppm Pb, 638 ppm Mn, 6.25 ppm Hg, 20.6 ppm Ni, 1.52 ppm Se, 4.63 ppm Ag, 245.3 ppm Sr, 2.47 ppm Tl, 81.6 ppm V, 350.4 ppm Zn

NIST 2586 – Trace Elements in Soil Containing Lead from Paint

Certified for: 8.7 ppm As, 2.71 ppm Cd, 301 ppm Cr, 432 ppm Pb

NIST 1646 – Estuarine sediment

Certified for: 62500 ppm Al, 8300 ppm Ca, 33500 ppm Fe, 10900 ppm Mg, 540 ppm P, 11.6 ppm As, 0.36 ppm Cd, 76 ppm Cr, 10.5 ppm Co, 18 ppm Cu, 28.2 ppm Pb, 375 ppm Mn, 0.063 ppm Hg, 32 ppm Ni, 94 ppm V, 138 ppm Zn

USGS BIR-1 – Icelandic Basalt

Certified for: 223813 ppm Si, 82059 ppm Al, 95068 ppm Ca, 58489 ppm Mg, 79015 ppm Fe, 13503 ppm Na, 249 ppm K, 1355 ppm Mn, 92 ppm P, 5755 ppm Ti, 125 ppm Cu, 4 ppm Dy, 1.9 ppm Ce, 52 ppm Co, 370 ppm Cr, 0.55 ppm Eu, 1.8 ppm Gd, 0.6 ppm Hf, 2.5 ppm Nd, 170 ppm Ni, 0.63 ppm La, 3.6 ppm Li, 44 ppm Sc, 110 ppm Sr, 310 ppm V, 16 ppm Y, 1.7 ppm Yb, 70 ppm Zn, 18 ppm Zr.

USGS BCR-2 – Columbia River Basalt

Certified for: 71400 ppm Al, 50900 ppm Ca, 96500 ppm Fe, 14900 ppm K, 21600 ppm Mg, 23400 ppm Na, 1500 ppm P, 253000 ppm Si, 13500 ppm Ti, 683 ppm Ba, 53 ppm Ce, 37 ppm Co, 18 ppm Cr, 2 ppm Eu, 23 ppm Ga, 6.8 ppm Gd, 25 ppm La, 1520 ppm Mn, 248 ppm Mo, 28 ppm Nd, 48 ppm Rb, 33 ppm Sc, 346 ppm Sr, 6.2 ppm Th, 1.69 ppm U, 416 ppm V, 37 ppm Y, 3.5 ppm Yb, 127 ppm Zn, 188 ppm Zr

USGS GSP-2 – Granodiorite, Silver Plume, Colorado

Certified for: 78800 ppm Al, 15000 ppm Ca, 34300 ppm Fe, 44800 ppm K, 5800 ppm Mg, 20600 ppm Na, 1300 ppm P, 311000 ppm Si, 4000 ppm Ti, 1340 ppm Ba, 410 ppm Ce, 7.3 ppm Co, 20 ppm Cr, 43 ppm Cu, 2.3 ppm Eu, 22 ppm Ga, 180 ppm La, 320 ppm Mn, 27 ppm Nb, 200 ppm Nd, 17 ppm Ni, 42 ppm Pb, 245 ppm Rb, 6.3 ppm Sc, 27 ppm Sm, 240 ppm Sr, 105 ppm Th, 2.4 ppm U, 52 ppm V, 28 ppm Y, 1.6 ppm Yb, 120 ppm Zn, 550 ppm Zr

USGS Nod-A1 – Manganese nodule, Atlantic

Certified for: 17780 ppm Si, 20488 ppm Al, 109083 ppm Fe, 110078 ppm Ca, 28702 ppm Mg, 185065 ppm Mn, 7419 ppm Na, 4979 ppm K, 3177 ppm Ti, 6113 ppm P, 1670 ppm Ba, 3110 ppm Co, 1100 ppm Cu, 448 ppm Mo, 6360 ppm Ni, 846 ppm Pb, 1750 ppm Sr, 770 ppm V, 590 ppm Zn

USGS Nod-P1 – Manganese nodule, Pacific

Certified for: 64867 ppm Si, 25412 ppm Al, 58038 ppm Fe, 22159 ppm Ca, 19898 ppm Mg, 291148 ppm Mn, 16323 ppm Na, 9962 ppm K, 2997 ppm Ti, 2008 ppm P, 3350 ppm Ba, 2240 ppm Co, 11500 ppm Cu, 760 ppm Mo, 13400 ppm Ni, 560 ppm Pb, 680 ppm Sr, 570 ppm V, 1600 ppm Zn

USGS SCo-1 – Cody Shale

Certified for: 293067 ppm Si, 72529 ppm Al, 35871 ppm Fe, 18728 ppm Ca, 16401 ppm Mg, 6677 ppm Na, 22995 ppm K, 917 ppm P, 3777 ppm Ti, 12 ppm As, 72 ppm B, 570 ppm Ba, 7.8 ppm Cs, 1.8 ppm Be, 62 ppm Ce, 11 ppm Co, 68 ppm Cr, 29 ppm Cu, 770 ppm F, 30 ppm La, 45 ppm Li, 410 ppm Mn, 1.4 ppm Mo, 26 ppm Nd, 27 ppm Ni, 31 ppm Pb, 110 ppm Rb, 630 ppm S, 2.5 ppm Sb, 11 ppm Sc, 170 ppm Sr, 9.7 ppm Th, 130 ppm V, 26 ppm Y, 100 ppm Zn, 160 ppm Zr.

USGS SDO-1 – Devonian Ohio Shale

Certified for: 64959 ppm Al, 7505 ppm Ca, 65310 ppm Fe, 27798 ppm K, 9286 ppm Mg, 325 ppm Mn, 2819 ppm Na, 480 ppm P, 229973 ppm Si, 4260 ppm Ti, 68.5 ppm As, 397 ppm Ba, 79.3 ppm Ce, 46.8 ppm Co, 66.4 ppm Cr, 6.0 ppm Dy, 1.6 ppm Eu, 16.8 ppm Ga, 38.5 ppm La, 134 ppm Mo, 11.4 ppm Nb, 36.6 ppm Nd, 99.5 ppm Ni, 8.9 ppm Pr, 126 ppm Rb, 13.2 ppm Se, 7.7 ppm Sm, 75.1 ppm Sr, 48.8 ppm U, 160 ppm V, 40.6 ppm Y, 3.4 ppm Yb, 64.1 ppm Zn, 165 ppm Zr.

USGS G-2 – Analyzed Granite

Certified for: 81476 ppm Al, 14010 ppm Ca, 18620 ppm Fe, 37191 ppm K, 4522 ppm Mg, 232 ppm Mn, 30271 ppm Na, 611 ppm P, 322653 ppm Si, 2880 ppm Ti, 1880 ppm Ba, 2.5 ppm Be, 160 ppm Ce, 4.6 ppm Co, 1.34 ppm Cs, 2.4 ppm Dy,

1.4 ppm Eu, 1280 ppm F, 23 ppm Ga, 7.9 ppm Hf, 89 ppm La, 34 ppm Li, 55 ppm Nd, 30 ppm Pb, 170 ppm Rb, 3.5 ppm Sc, 7.2 ppm Sm, 478 ppm Sr, 25 ppm Th, 36 ppm V, 11 ppm Y, 0.8 ppm Yb, 86 ppm Zn, 309 ppm Zr.

USGS RGM-1 – Glass Mountain Rhyolite

Certified for: 342533 ppm Si, 72529 ppm Al, 13020 ppm Fe, 279 ppm Mn, 30197 ppm Na, 35696 ppm K, 1619 ppm Ti, 8220 ppm Ca, 1688 ppm Mg, 0.11 ppm Ag, 3 ppm As, 28 ppm B, 810 ppm Ba, 2.4 ppm Be, 1.3 ppm Br, 47 ppm Ce, 510 ppm Cl, 2 ppm Co, 9.6 ppm Cs, 12 ppm Cu, 4.1 ppm Dy, 0.66 ppm Eu, 340 ppm F, 15 ppm Ga, 3.7 ppm Gd, 24 ppm La, 57 ppm Li, 0.4 ppm Lu, 280 ppm Mn, 2.3 ppm Mo, 8.9 ppm Nb, 19 ppm Nd, 24 ppm Pb, 150 ppm Rb, 1.3 ppm Sb, 4.4 ppm Sc, 4.3 ppm Sm, 4.1 ppm Sn, 110 ppm Sr, 0.95 ppm Ta, 15 ppm Th, 5.8 ppm U, 13 ppm V, 1.5 ppm W, 2.6 ppm Yb, 220 ppm Zr.

USGS COQ-1 – Carbonatite

Certified for: 1959 ppm Al, 16193 ppm Si, 20580 ppm Fe, 7537 ppm Mg, 345246 ppm Ca, 1328 ppm K, 899 ppm Ti, 11352 ppm P, 3330 ppm Mn, 1000 ppm Ba, 1.2 ppm Be, 1700 ppm Ce, 0.2 ppm Cs, 7 ppm Er, 15 ppm Eu, 6 ppm Ga, 50 ppm Gd, 3 ppm Ho, 750 ppm La, 3900 ppm Nb, 480 ppm Nd, 13 ppm Ni, 150 ppm Pr, 3 ppm Sc, 56 ppm Sm, 12000 ppm Sr, 4 ppm Tb, 10 ppm Th, 11 ppm U, 110 ppm V, 81 ppm Y, 6 ppm Yb, 87 ppm Zn, 65 ppm Zr.

NIST 2702 – Inorganics in Marine Sediment

Certified for: 84100 ppm Al, 45.3 ppm As, 397.4 ppm Ba, 123.4 ppm Ce, 0.817 ppm Cd, 27.76 ppm Co, 352 ppm Cr, 79100 ppm Fe, 0.438 ppm Hg, 20540 ppm K, 73.5 ppm La, 1757 ppm Mn, 6810 ppm Na, 75.4 ppm Ni, 127.7 ppm Rb, 1552 ppm P, 132.8 ppm Pb, 5.6 ppm Sb, 25.9 ppm Sc, 119.7 ppm Sr, 20.51 ppm Th, 8840 ppm Ti, 0.8267 ppm Tl, 357.6 ppm V, 485.3 ppm Zn

NIST 1632d – Coal (Bituminous)

Certified for: 5.1% H, 1.462% S, 0.1094% K, 0.1142% Cl, 0.749% Fe, 76.88% C, 1.59% N, 1.65% Si, 0.912% Al, 0.144% Ca, 0.455 ppm Sb, 40.42 ppm Ba, 3.424 ppm Co, 5.83 ppm Cu, 3.845 ppm Pb, 0.0928 ppm Hg, 7.36 ppm Rb, 296.9 ppm Na, 63.5 ppm Sr, 1.428 ppm Th, 477 ppm Ti, 0.517 ppm U, 23.74 ppm V

NIST 8704 – Buffalo River Sediment

Certified for: 6.1% Al, 2.641% Ca, 3.351% C, 3.97% Fe, 1.2% Mg, 2.001% K, 0.553% Na, 0.457% Ti, 3.07 ppm Sb, 413 ppm Ba, 2.94 ppm Cd, 66.5 ppm Ce, 5.83 ppm Cs, 121.9 ppm Cr, 13.57 ppm Co, 1.31 ppm Eu, 8.4 ppm Hf, 150 ppm Pb, 544 ppm Mn, 42.9 ppm Ni, 11.26 ppm Sc, 9.07 ppm Th, 3.09 ppm U, 94.6 ppm V, 408 ppm Zn

Waters

NRC SLEW-3 – Estuarine Water

Certified for: 0.00134 ppm As, 0.000047 ppm Cd, 0.000181 ppm Cr, 0.00004 ppm Co, 0.00153 ppm Cu, 0.000561 ppm Fe, 0.000009 ppm Pb, 0.00159 ppm Mn, 0.00121 ppm Ni, 0.00254 ppm V, 0.000198 ppm Zn.

NRC CASS-5 – Nearshore seawater reference material for trace metals

Certified for: 0.00121 ppm As, 0.0000210 ppm Cd, 0.000103 ppm Cr, 0.000371 ppm Cu, 0.00140 ppm Fe, 0.000011 ppm Pb, 0.00256 ppm Mn, 0.00959 ppm Mo, 0.000322 ppm Ni, 0.00311 ppm U, 0.00128 ppm V, 0.000702 ppm Zn

NRC NASS-5 – Seawater reference material for trace metals

Certified for: 0.00127 ppm As, 0.000023 ppm Cd, 0.000110 ppm Cr, 0.000011 ppm Co, 0.000297 ppm Cu, 0.000207 ppm Fe, 0.000008 ppm Pb, 0.000919 ppm Mn, 0.0096 ppm Mo, 0.000253 ppm Ni, 0.000102 ppm Zn

OSIL IAPSO – Standard seawater calibrated for salinity

Certified for: 0.174 ppm Li, 4.5 ppm B, 27.6 ppm C, 0.42 ppm N, 1.3 ppm F, 10770 ppm Na, 1290 ppm Mg, 0.00054 ppm Al, 2.8 ppm Si, 0.07 ppm P, 904 ppm S, 19354 ppm Cl, 399 ppm K, 412 ppm Ca, 0.000014 ppm Mn, 0.000055 ppm Fe, 0.0005 ppm Ni, 0.00025 ppm Cu, 0.0004 ppm Zn, 0.0017 ppm As, 67 ppm Br, 0.12 ppm Rb, 7.9 ppm Sr, 0.00008 ppm Cd, 0.05 ppm I, 0.00029 ppm Cs, 0.014 ppm Ba, 0.000001 ppm Hg, 0.000002 ppm Pb, 0.0033 ppm U

Organic materials

NIST 1400 – Bone ash

Certified for: 381800 ppm Ca, 6840 ppm Mg, 179100 ppm P, 660 ppm Fe, 9.07 ppm Pb, 186 ppm K, 249 ppm Sr, 181 ppm Zn

NIST 1575a – Trace Elements in Pine Needles

Certified for: 1070 ppm P, 4170 ppm K, 2500 ppm Ca, 580 ppm Al, 6.0 ppm Ba, 0.233 ppm Cd, 421 ppm Cl, 2.8 ppm Cu, 46 ppm Fe, 0.0399 ppm Hg, 16.5 ppm Rb, 38 ppm Zn

NIST 1947 – Lake Michigan fish tissue

Certified for: 0.732 ppm As, 0.411 ppm Cu, 3.79 ppm Fe, 0.254 ppm Hg including isotope ratios, 0.076 ppm Mn, 4.51 ppm Rb, 0.475 ppm Se, 2.66 ppm Zn

NIST SRM 1515 - Apple Leaves

Certified for: 1.526% Ca, 0.271% Mg, 2.25% N, 0.159% P, 1.61% K, 0.18% S, 286 ppm Al, 0.038 ppm As, 49 ppm Ba, 27 ppm B, 0.013 ppm Cd, 579 ppm Cl, 5.64 ppm Cu, 83 ppm Fe, 0.47 ppm Pb, 54 ppm Mn, 0.044 ppm Hg, 0.094 ppm Mo, 0.91 ppm Ni, 10.2 ppm Rb, 0.05 ppm Se, 24.4 ppm Na, 25 ppm Sr, 0.26 ppm V, 12.5 ppm Zn

NIST SRM 1547 – Peach Leaves

Certified for: 1.56% Ca, 0.432% Mg, 2.94% N, 0.137% P, 2.43% K, 249 ppm Al, 0.06 ppm As, 124 ppm Ba, 29 ppm B, 0.026 ppm Cd, 360 ppm Cl, 3.7 ppm Cu, 218 ppm Fe, 0.87 ppm Pb, 98 ppm Mn, 0.031 ppm Hg, 0.06 ppm Mo, 0.69 ppm Ni, 19.7 ppm Rb, 0.12 ppm Se, 24 ppm Na, 53 ppm Sr, 0.37 ppm V, 17.9 ppm Zn

NIST SRM 1577c – Bovine Liver

Certified for: 5.9 ppb Ag, 19.6 ppb As, 131 ppm Ca, 97 ppb Cd, 0.3 ppm Co, 53 ppb Cr, 275.2 ppm Cu, 197.94 ppm Fe, 1.023% K, 620 ppm Mg, 10.46 ppm Mn, 3.3 ppm Mo, 0.2033% Na, 44.5 ppb Ni, 62.8 ppb Pb, 0.749% S, 2.031 ppm Se, 95.3 ppb Sr, 8.17 ppb V, 181.1 ppm Zn

Appendix C – *Certified Natural Standard Materials Currently Available (Other Instruments/Applications)*

Acetanilide (for use on CHN)

Certified for: 71.1% Carbon (w/w) ± 0.23%, 6.71% Hydrogen (w/w) ± 0.07%, 10.34% Nitrogen (w/w) ± 0.09%

EML B2162 – Algae (Spirulina)

Certified for: 47.21% Carbon (w/w) ± 0.39%, 6.89% Hydrogen (w/w) ± 0.12%, 10.81% Nitrogen (w/w) ± 0.25%