ICP-MS Wood ICP Protocol

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June 26, 2017

Updated by Jim Dalling 6 Dec, 2021

Supplies needed

- Small glass vials (1 for each sample + 2 apple standards)
- 1 M HCl for acid washing
- Washing up gloves and tongs (preferably plastic)
- Miliq water
- 1 M nitric acid (see recipe below)
- Volumetric flasks for nitric acid preparation
- datasheet
- scale
- muffle furnace (in Wendy Yang's lab)
- 10 ml pump and base
- standards
- 15 ml tubes (1 for each sample + 2 apple standards + 2-3 standards + 1 blank) **Buy new for each sample.**

Sample preparation

Glass vials are needed for combustion of the samples and volumetric flasks to make up 1M nitric acid. These need to be acid washed in 1M HCl. The HCl solution is in a Nalgene 2G container. Add HCl to the flasks and leave overnight. After use, HCl solution can be returned to the container and reused. For vials, pour it out into a clean plastic washing up bowl. Use tongs to transfer vials to the bowl. Make sure the vials are submerged. Leave overnight. Use tongs to empty the vials back into the bowl (acid wash from the bowl can be reused). Submerge vials into a bowl of DI water (available in first floor prep room – white tap in the stainless steel sink). Each vial should then be rinsed three times with DI water. Place rinsed vials on Al foil in a clean metal tray and dry in the oven in 133 MH at ~70C for at least 2 hours. These are now clean and ready to go.

Mixing Nitric Acid

1 M nitric acid (1L)

- First add 750 ml of milliq water (from Yang lab) to a 1L glass bottle (*always add acid to water NOT water to acid*)
- add 64 ml of concentrated Nitric Acid
- add another ~186 ml of water

Tips:

- Always use the same acid for a single experiment. If you have to mix more acid, I would recommend re-running a few samples in the new acid solution.

Protocol:

1) Record sample info on datasheet, including the vial number
2) Tare glass bottle
3) Weigh 30mg (+/- 2 mg) of sample on the analytical balance in Dalling Lab.
4) Record Mass on datasheet
5) Cover vial with a square of Al foil (for transport of samples to the muffle furnace). Put vials in one of the cardboard vial trays for transport.
6) Don't forget the apple standard! It's in Jim's lab on the back-center bench, in a brown cylinder. You need 2 apple standards for each ICP run. Use the same mass of apple standard as I do other sample.
7) Put them in the muffle furnace in Yang lab at 550 C for 3 hours (the oven fits about 100 at a time)
8) **Don't forget to turn the oven off**, but then let samples cool for 2 hours
9) After samples have cooled, put 10 mL of 1 M nitric acid in the vials.
10) Let the samples site overnight.
11) Add 5 mL of miliq water to the vials. This dilutes the solution to 3% nitric acid.
12) Label the 15ml tubes that you will need for the run.
13) Put ~ 7-8 mL of solution in a **NEW** 15ml plastic tube

Standards and Blank

1. Making Blank – put 10ml of 1M HNO3 and 5 ml of miliq water in a 25 or 50 ml tube.
2. It's good to make new standards before a run of samples. See Dalling Lab Morrill 149 for standards (Ca, Mg, P, K, Na, Al, Mn).
3. Use Miliq water from either the Hu or Heath lab to make standards!
4. Label tubes low, med, and high.
5. Add 10ml of 1M HNO3 to the three 50 ml plastic tube.
6. Add standards to high and medium tubes with the following recipe.

Method 1:

****Updated June 26, 2017: These are the standards in 15 ml of 3% nitric acid.**

Stock	Standard 1 (Low)	Standard 2 (Med)	Standard 3 (High)	Final mg/L for low/high	ppm
1000 ppm P	1.5 uL	15 uL	150 uL	.1/1/10	.1/1/10
1000 ppm Ca	3 uL	30 uL	300 uL	.2/2/20	.2/2/20
1000 ppm K	3 uL	30 uL	300 uL	.2/2/20	.2/2/20
1000 ppm Mg	1.5 uL	15 uL	150 uL	.1/1/10	.1/1/10
100 ppm Mn	1.5 uL	15 uL	150 uL	.1/1/10	.1/1/10
1000 ppm Al	1.5 uL	15 uL	150 uL	.1/1/10	.1/1/10
add water	5ml	4.9ml	3.8ml		
1M HNO3	15 ml final volume	15 ml final volume	15 ml final volume		

Running the samples on the ICP-MS

List of supplies

- Samples in 15 ml tubes
- Apple standards in 15ml tubes
- Standards in 15 ml tubes
- Blank in 15 ml tubes.

Notes
- No need to dilute the samples!
- Program is called 2017-03-02 Dalling Wood 5
- Once the machine is up and running, the machine takes approximately 3 minutes / sample.

Calculations

Notes
- Manganese and Aluminum are both usually ~ 5-10% of what they should be.
- Potassium and Calcium usually run 75-85%
- Which Calcium isotope to use? You should check the apple standard and see which isotope is closest to the expected Caclium weight. Check 42, 43, 44, and 48. Gideon says 42 usually works the best. Do not use 46 because of interference with other elements. Also 44 and strontium interfere, and 48 and titanium interfere.

pph= % = 10,000 ppm
ppm = ug/g = 1000 ppb

Checking the apple standard

$$\frac{mass\ of\ sample\ (g) * \frac{known\ concentration\ (\%) * 10{,}000 \frac{ppm}{pph}}{\#\ of\ ml\ in\ digestion}}{ExtCal.Average\ (ppm)})$$

Phosphorus example:

$$.0296\ g * \frac{.1590\ \% * 10{,}000 \frac{ppm}{pph}}{15\ ml} = \frac{3.0582\ actual\ ppm}{3.1376\ ppm\ expected} = .9747$$

Calculating sample % weight from the ICP output

- Look for "ExtCal.Average". This is the average of the different replicates.

$$\frac{\frac{ppm(given\ by\ ICP\ as\ ExtCal.Average) * \#\ of\ ml\ in\ digestion}{mass\ of\ sample\ (g)}}{10{,}000\ ppm/pph}$$

For this protocol

$$\frac{\frac{ppm(given\ by\ ICP\ as\ ExtCal.Average) * 15\ ml}{mass\ of\ sample\ (g)}}{10{,}000\ ppm/pph}$$

Apple standard concentrations
P = .159%
K = 1.61%
Mg= .271%
Ca = 1.526%
Al = 286 (+/-9) ppm
Mn = 54 (+/-3) ppm

How to correct for actual nutrient concentration using the apple standard
NEED TO CHECK WITH GIDEON

% weight of element measured * proportion of element measured in apple standard = corrected % weight