Ion on the Prize: Chasing Ultra Traces with ICP-MS at SNOLAB

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Outline

- Basics of ICP-MS functionality
- Equipment updates
- Low matrix, full metal suite method
- Isotopic dilution method

Basics of ICP-MS

There are 5 key components of the ICP-MS:

- Sample Introduction System
- Inductively Coupled Plasma
- Interface
- Analyzer
- Detector

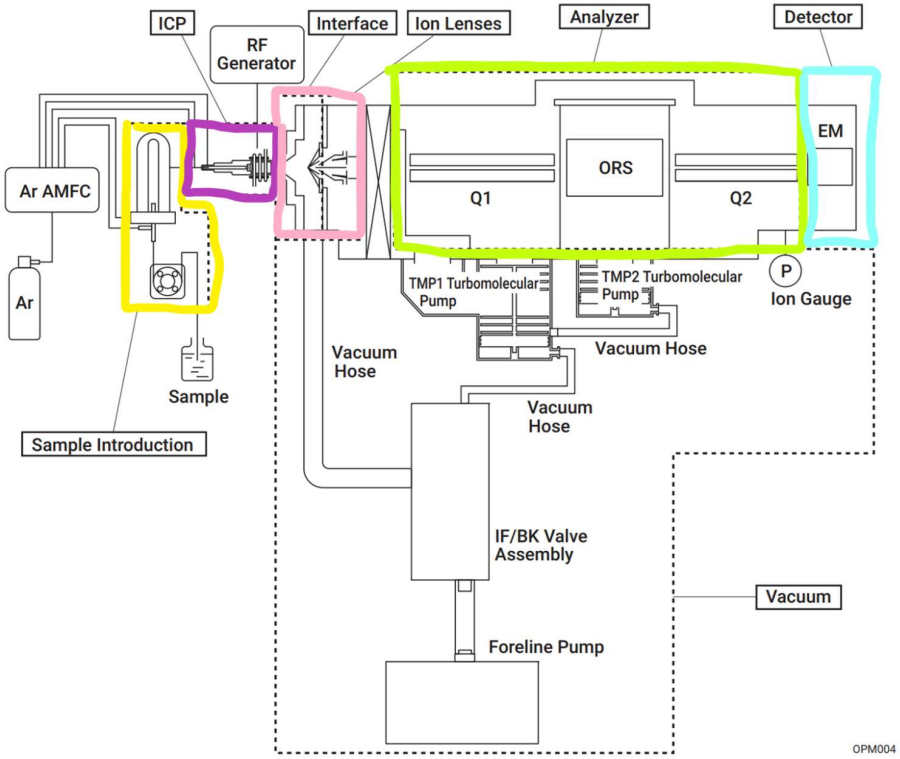
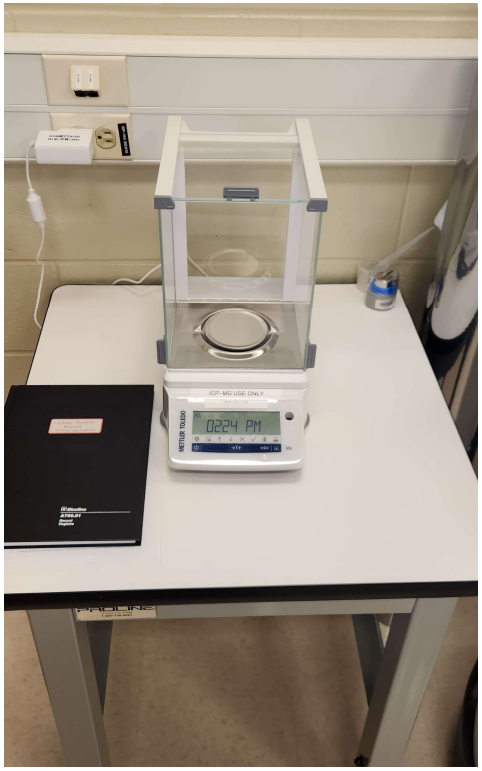


*Image source from Agilent Technologies (2022) pg. 17.

ICP-MS Equipment Updates



Dedicated analytical balance

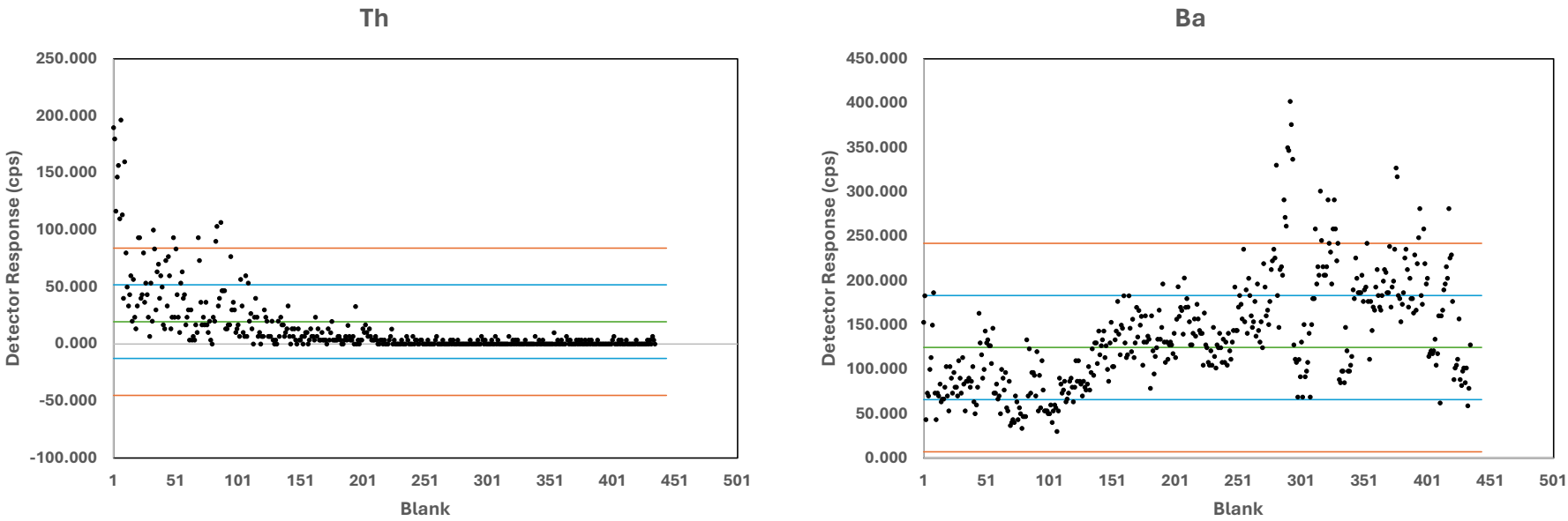
Dedicated ductless hood

UPS and generator tie-in

Low Matrix, Full Metal Suite Method

Method Name	Low Matrix, Full-Metal Suite
Target Analytes	Be, B, Na, Mg, Al, Si, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, As, Se, Rb, Sr, Y, Zr, Nb, Mo, Ru, Pd, Ag, Cd, Sn, Sb, Te, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Gd, Dy, Ho, Er, Tm, Yb, Hf, W, Re, Ir, Pt, Tl, Pb, Th, U
Key Detection Limits, 6σ (ng/L)	U = 0.07, Th = 1.5, K = 1750, Pb = 75
Sample Types	Aqueous, acidified with nitric acid, trace analysis only (ng/L) (TDS <0.001%)
Internal Standard (ISTD) Elements	Li, Sc, Ge, Rh, Tb, Lu, Bi
Applications	TeA plant UPW tank and nitric acid, trace contamination studies, leaching samples for butane diol plant materials, onsite UPW monitoring for surface and UG, distilled nitric acid QA, etc.

Are our backgrounds consistent?



Method blank control charts for thorium (left) and barium (right) in low matrix full metal suite method. Data collected between March 2024 and June 2025.

Background Monitoring

What?

- Distilled nitric acid (every LOT#)
- HDPE Autosampler vials (20%)
- PFA Sample Bottles (20%)
- Method backgrounds (each batch)

Why?

- Background contribution monitoring
- Confirm cleaning sufficiency
- Improve confidence in low-level results
- Minimize risk for unexpected memory effects

How?

- Prepare “blank” samples using the labware in the same manner it is used for samples
- Analyze samples at pre-determined time
- Compare results to pass/fail criteria to validate use of that LOT#

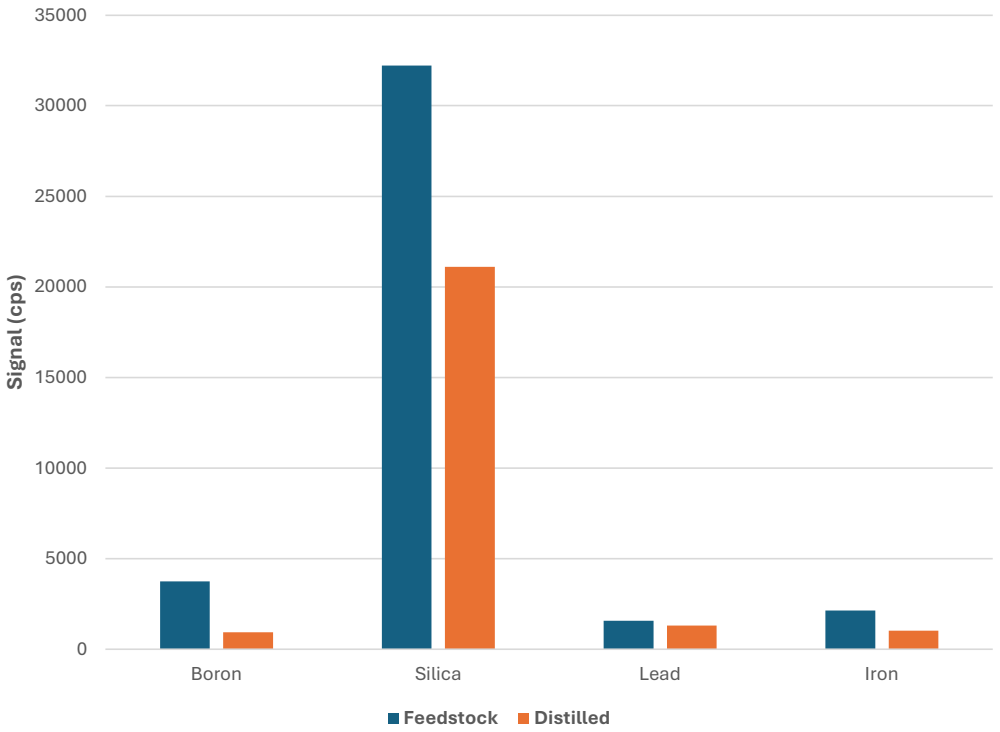
Future Plans

- Development work on bettering backgrounds from labware through improved cleaning
- Monitor the success of the program overtime and make adjustments to pass/fail criteria
- Add any additional materials used in sample preparation overtime

Reports here: https://www.snolab.ca/docushare/dsweb/View/Collection-11172

Distilled Nitric Acid QA

Sample Type	Purpose
5% (v/v) nitric acid feedstock	Assess initial source quality
5% (v/v) distilled nitric acid	Compare distilled to feedstock quality
3x 2% (v/v) distilled nitric acid	Confirm background contribution is consistent with MDLs
2% (v/v) distilled nitric acid (control charted)	Monitor trends in distillation backgrounds between LOTs
100 ng/L Spikes in 2% distilled nitric acid	Verify recoveries and assess potential matrix effects



Comparing select analytes for background signal from feedstock and distilled nitric acid.

Trace Contamination Testing

Sample	Al (ng/L)	K (ng/L)	Fe (ng/L)	Zn (ng/L)	Th (ng/L)	U (ng/L)
Ziplock (n=3)	991	<1750	<300	798	<1.50	<0.07
Blue Gloves (n=1)	<400	$4.0 * 10^5$	405	$2.5*10^4$	<1.50	0.34
Purple Gloves (n=1)	<400	$1.3*10^4$	<300	$2.6*10^3$	<1.50	0.23
Pink Plastic Sheeting (n=1)	$>1.0*10^5$	$1.5*10^4$	$5.6*10^3$	$>1.0*10^5$	<1.50	0.34
Clear Plastic Sheeting (n=1)	$1.2*10^3$	$4.5*10^3$	$8.0*10^3$	$1.4*10^4$	<1.50	0.23
2% HNO3 used for Sample Preparation	<400	<1750	<300	<190	<1.50	<0.07

* Samples are corrected for background of zip lock bags used for testing.

Areas for Continued Development

Data and Analysis

- Streamline data analysis using code
- Update detection limits
- Reporting MDL calculation method finalized

Backgrounds

- Labware cleaning improvements
- Filtration testing
- Implement statistical analyses that flag variations in background earlier

Trend Monitoring

- In-depth review of long-term trends to guide:
  - Inform areas of potential improvement
  - Identify flags for streamlined data to point analyst to problem areas
  - Instrument tuning needs

Reporting Standards

- Standardize error reporting for consistency and clarity

Data Reporting

Included	Not-Included
Calculated sample data	Internal Standard Recoveries
Quality Control standard results	Spike validation recoveries
Reference to QA testing on related sampling and sample preparation materials	Calibrations curves
Average method blank results (n=10)	Control Charts
Dilution information	Unprocessed data

Isotopic Dilution Method

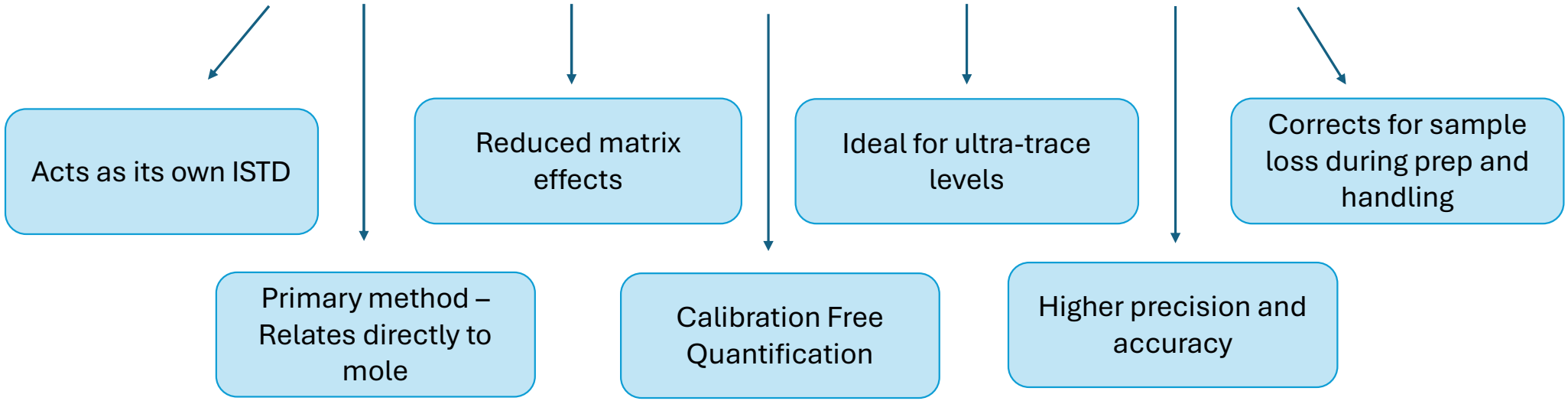
U and Th Improvements

	Uranium	Thorium
Process Blank Average (pg/L) n=12	0.06	0.09
Process Blank Std. Dev. (pg/L) n=12	0.46	0.87
Est. DL 6σ (pg/L)	2.76	5.22
40 pg/L QC	43 ± 15	51 ± 16
7 pg/L QC	8 ± 5	5 ± 7

Data Provided is in He Mode, data taken on 2025-01-16 (Run #3 of Method). Data is based on external calibration using volumetric dilution. The DL is also based on UPW with distilled nitric acid only.

Isotopic Dilution (ID) ICP-MS

ID-ICPMS is a technique where a known amount of an isotopically enriched tracer is added to a sample to accurately measure the concentration of a given isotope of the same element. This is done through isotopic ratio comparison.



Basic Isotopic Dilution Equation

$$C_{sample} = C_{spike} * \frac{R_{mix} - R_{sample}}{R_{spike} - R_{mix}} * \frac{m_{spike}}{m_{sample}}$$

$C_{sample} = concentration\ of\ the\ analyte\ in\ sample$
$C_{tracer} = concentration\ of\ the\ analyte\ in\ tracer$
$R_{mix} = measured\ isotope\ ratio\ in\ the\ mixture$
$R_{sample} = isotope\ ratio\ in\ the\ natural\ sample$
$R_{spike} = istope\ ratio\ in\ the\ tracer$
$m_{tracer} = mass\ of\ tracer\ solution\ added$
$m_{sample} = mass\ of\ sample\ analyzed$

ID-ICPMS Plans

Foundation Work Completed

- Characterization of natural abundance standard
- Sensitivity improvements
- Source procurement and handling plans

Next Steps

- Receive and verify tracer purity and concentration
- Initial tracer recovery and ratio validation
- Initial method dev for prep (low matrix)

Initial Validation

- Spike recovery studies and reproducibility testing, QA protocol
- Detection limit and uncertainty quantification
- Interlaboratory comparison
- Method documentation and SOP creation

Future Priorities

- Initial digestion samples will be dust and copper
- Resin testing and validation to reduce matrix load
- Cleaning studies for labware and equipment, extend QA testing to include backgrounds for this method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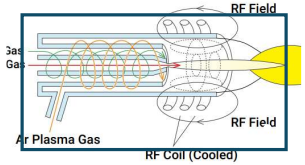
The Impossible Takes A Little Longer

Questions?


RF Field
RF Field
Ar Plasma Gas
RF Coil (Cooled)