

	Environmental Analysis Teaching and Research Laboratory	Date: 2/12/2018	Number: 70 v0.1
	Standard Operating Procedure	Title: ICP-MS Guide	
	Approved By: TBD	Revision Date: April 6, 2018	

1. Scope and Application

1.1 The scope of this SOP is train researchers...

1.2 The applications of this SOP are for...

2. Summary of Method

2.1 This SOP does this...

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3. Acknowledgements

3.1 This procedure requires XX minutes...

4. Personnel & Training Responsibilities

4.1 Researchers training is required before this the procedures in this method can be used...

4.2 Researchers using this SOP should be trained for the following SOPs:

- SOP01 Laboratory Safety
- SOP02 Field Safety

5. Health and Safety

Risks

5.1 Describe the risk...

Safety and Personnel Protective Equipment

6. Time Management

6.1 This section is to make users aware of the time commitment before running analyses.

Solutions Preparation

Methods Development

Instrument Start-up

Ignition Sequence and Warm Up

Performance Checks

Analysis and Data Retrieval

Instrument Shut-down

7. Sample and Standard Preparation

Acid Handling Guidelines

- **Never** mix organic solvents like ethanol or acetone with nitric acid (HNO_3) and do not store concentrated acid bottles around organic solvents. The two react violently with each other and create toxic fumes.

- Always add concentrated acid to a comparatively larger volume of deionized water first. **Do not** add water to concentrated acid.
- Be mindful of putting clothing or hands/arms over containers, particulates and dust will fall into them and could affect low level readings.
- Use non powdered gloves
- Acid solutions are diluted by volume. Use full strength nitric and/or hydrochloric acid (~70% and ~37%, respectively) and dilute directly into centrifuge tube or plastic test tube.

7.1 Example: to make a matrix solution of 1% nitric and 0.5% hydrochloric in a 50 ml centrifuge tube, use 0.5ml of ~70% HNO_3 and 0.25ml ~37% HCl . Then use 18 Ω deionized water to fill the rest of the tube (49.25mL).

Creating Standards

- Do not use glassware for ultra-trace applications
- Dilute and make standards directly in plastic containers/vials (HDPE or PTFE) whenever possible.
- Use plastic pipettes with no metal parts (metal can rust/corrode and contaminate solutions).
- **DO NOT** pipet out of the standard or acid containers, instead use an intermediate container to pipet out of to prevent contamination of the stock solution.

Tuning Solutions

- For most purposes, the internal standard tuning solution is diluted to 0.5 ppm and 1% HNO_3 .
- Tuning solution is diluted to 1 ppb at HNO_3 .
- P/A factor tuning solution is diluted to 1 ppb at HNO_3 .

8. Procedures

Creating a Batch

8.1 Think of a batch file as an all-inclusive file. It includes your method, tune profile, sample list, and other parameters all in one.

8.2 There are a few rules and limitations with the Masshunter software and batch files:

1. The MassHunter software requires you to make batch files in order to do about basically anything.
2. Once you have run an analysis, or sent your batch file to the queue, you must create a new one in order to run again.

Creating a Method

Notable Biases and Interferences

Instrument Start Up

- 8.3 Ensure there is enough internal tune solution, rinse, and autosampler rinse and the solutions and standards being used in the autosampler are uncapped.
- 8.4 Verify the Argon dewar has sufficient levels for analysis and make sure its valve is open.
- 8.5 Check gas supply regulator pressure, it should be at 100psi.
- 8.6 Inspect the PeriPump tubing and fittings and replace if needed.
- 8.7 Attach the PeriPump tubing and clamp them properly.
- 8.8 Clamp the PeriPump tubing located in the back of the autosampler.
- 8.9 Turn on the ICP chiller and verify it is functioning properly.
- 8.10 Turn on the Plasma by clicking the pulldown next to the [Plasma] gadget and click [Plasma on] When confirming to turn the plasma on make sure [Execute Configured Ignition Sequence] is **unchecked**. IMMEDIATELY record the plasma on-time in 24:00 hour time. E.g., 2:20 = 14:20.
- 8.11 Let the instrument “warm up” sufficiently by verifying the “SC Temp” is fluctuating between 1.9 and 2.0°C.

Gas	Pressure	Reorder #
Argon	100 psi	??
Oxygen		

- 8.12 Turn on chiller
- 8.13 Open argon valve
- 8.14 Connect drain and sample tubes to peristaltic pump and clamp.
- 8.15 Connect internal standard, should be diluted to 1 ppm or 1 μ /mL.

8.16 Check Settings, nebulizer, post rotate yes!

8.17 Turn on circulate water

8.18 Startup Configuration

8.19 Instrument set up – various tests done that should be checked.

8.20 Tuning solutions... Peripump, .5 uL solution.. internal standard concentration will be... speed to 0.3 because the tube stretches out. Stabilizes to 30s, acquisition speed... probe rinse...

8.21 Check Default Standard Setting

8.22 P/A solutions

8.23 Turn on Plasma Mode

8.24 Enable Configure Ignition Sequence is checked for liquid samples

8.25 Check meters

IF/Backing Pressure Analyzer Pressure Water Redirected Power Forced Power

8.26 Skip Warm-up

8.27 check for bubble moving into pump in tube

8.28 Autotuning solutions – DI water?

8.29 Check autoscale on 'Real Time Display'

8.30 Check Mainframe – performance report, record rsd $\pm 6\%$... check counts... oxides...cerium (mass 140/156) ± 2 double charges (mass 70 mass 40...) ± 3 , high matrix. check resolution axis around 7. peak width about 10%, .65 - .8, 6.9

Running a Batch

8.31 Prepare ...

8.32

9. Maintenance

Cleaning Nebulizer

9.1 Soak components in 5% nitric acid. Do not sonicate the nebulizer.

9.2 Nebulizer should be tight.

9.3 Replace jacket

Pump Oil

9.4 Replace pump oil every 3-4 months. Pump oil will break down and be the final resting place for all ions.

9.5

Checking Torch

9.6 Open cover

9.7 Shield can get ugly and needs to be replaced.

9.8 Don't seem to worry about finger prints on the outside.

9.9 Replace tab and torch bonnet stuff yearly

Sample and Skimmer Cone

9.10 Use software to "maintenance" and torch is moved.

9.11 Check and potentially Replace cones... depends on sample matrix, often a recently replaced cone are not stable.

9.12 Unscrew ring (use tool if needed)

9.13 Clean with sonicator, 1% Citronox dilute.

9.14 Use skimmer cone tool and unscrew it.

9.15 Be careful of the graphite o-ring

9.16 To replace, finger tighten skimmer cone.

9.17 Do not use skimmer cone tool until it's been finger threaded.

9.18 Replace sample cone

9.19 Initialize to put torch back in.

9.20 Close cover

Lenses

9.21 Using 3mm allen wrench...

9.22 Do not touch lens with hands w/o gloves

9.23 Loosen and pull them out.

9.24 Omega lens...

9.25 Cleaned as part of the PM (preventative maintenance).

9.26 Can check lens test via software.

10. Trouble Shooting

11. Definitions

12. References

12.1 APHA, AWWA. WEF. (2012) Standard Methods for examination of water and wastewater. 22nd American Public Health Association (Eds.). Washington. 1360 pp. (2014).

<https://crustal.usgs.gov/laboratories/icpms/intro.html>