

Agilent ICP-MS 7500 ce
Standard Operating Procedure
(Revised 08-17-2010)

I. Start Up (From Standby Mode to Analysis Mode)

1. Sign into the logbook.
2. Make sure vacuum pump is on.
3. Make sure the vent is working.
4. Check that sample waste & rinse waste containers are empty.
(Sample waste goes into the glass amber waste bottle)
5. Check that A/S rinse supply container is full. (Milli-Q H2O only)
6. **Check that the internal standard containers are full (10 ppb solution, Acid blank, or Milli-Q)**
Put the internal standard line in the acid blank.
7. Turn on Heat Exchanger
8. Open Argon gas cylinders valves – When using Liquid Ar DO NOT use the smaller compressed Ar tanks (outlet pressure should be 100 psi)
9. Reboot computer – not always necessary but recommended occasionally.
10. Click on “TOP” software and make sure the instrument is in **Standby Mode** [Note: There are three modes: Standby, Analysis, and Shutdown. Do not go to Shut Down Mode].
11. Record the analyzer pressure and interface/backing pressure on the user form.
12. Check if peristaltic pump tubing sections are in good condition (no flat sides). Clamp the **three** tubing sections into the pump. Check that the direction of flow for all three is correct.

**** Note the standard addition line sits in the middle pump tubing position****
13. Go to ALS and move the sampling probe to the Acid Blank source.
(Usually 2% HNO₃, 0.5% HCL)
14. Go to PLASMA, → “Plasma On” → “Yes.” Instrument will ask you “Exhaust system operating?” → “Yes”. Instrument will ask you “Verify drain tubing connection” → “Yes”. Instrument will change from **Standby Mode** to **Analysis Mode**.
15. Confirm that the waste is flowing into the waste bottle container.
16. Warm up the instrument.
 - Check your method as ICP-MS warms up
 - After 40 minutes, record the analyzer and interface/backing pressures and the reflected power on the user form.

II. Tuning and Semi-quantitative Analysis of Blank

17. Select “Instrument” → “Tune.”
18. Go to ALS and move sampling probe to the tuning solution; wait for uptake of the tuning solution to reach the nebulizer (4-6 minutes), or increase peripump speed until tuning solution reaches nebulizer.
19. The last tuning file is automatically downloaded as “atune.u”. Use these conditions to confirm counts are similar to previous tunings.
20. **Parameter changes must be approved in advance by Patty Wurm @ 935-4832.**
Normally no changes are needed, but after approval, you may adjust tuning parameters to maximize signal and minimize background.
Operations Manual (4-39).
21. After tuning the instrument, generate printouts of your tune reports and file them in the folder provided. Go to “File” → “Generate Report”

Please note on report any changes in parameters or unusual conditions.
22. Go to ALS and move sample probe to rinse – allow to rinse for about 1 min. (You must rinse sample probe to avoid contaminating the blank solution.)
23. Run a semi-quantitative analysis on the 2% HNO₃, 0.5% HCL blank solution using the new method created for ALL Users. We need everyone to use the same method so that we can track the blank counts/concentration from day to day.
24. Click “Method” → “Load and Run Method” → Choose
“D:\Everyone_methods\SemiQ_PT.M” → OK → Choose
“D:\Everyone_Calibration\SemiQ_PT.C” → OK.
25. Type in the vial # for 2% HNO₃, 0.5% HCL blank solution. You may type in sample name and comments (like before or after sample run).
26. Change Data Filename to → **D:\your name\data\your semiQ data file name.B** and click “Run Method” button.
27. Take printouts and file them in the folder provided.

Please note on report any changes in parameters or unusual conditions.

III. Analysis

28. Load your Method & Calibration that have been previously approved.
They should be saved as →
D:\your name\method\your method name.M
D:\your name\calibration\your cal name.C
D:\your name\sequence\your seq name.S

Ex: D:\Patty\Methods\1-ModeAu.M

29. Load your Sequence and remember to manually save it, there is no prompt to save it when closing the file.
30. Check sample rack type- You must exit ICP-MS Chem Station .
Then go to desk top → Configuration → Auto Sampler Setup → Rack ID
-choose rack type you will be using (21, 24, 40, 60 or 90).

31. Save your data to → D:\your name\data\your data file name.B

Ex: D:\Patty\data\07-19-09 patty.B

32. Click on <Run Sequence>.
33. All samples must be approved by Patty before analysis and follow the guidelines listed at the end of this SOP!!!
(If your first few samples have concentrations above these limits, run blanks until you flush any contamination, dilute all samples, and then continue your run.)

IV. Turning Off The Plasma (From Analysis Mode to Standby Mode)

34. Move ALS and Internal std line to acid blank and flush lines, repeat with milli Q. (leave tubing in milli Q to prevent lines from drying out)
35. Re-run a semi quantitative analysis on a 2% HNO₃, 0.5% HCL blank solution (Follow steps 26-30).
36. If counts are not comparable to those at start-up, run 2% HNO₃, 0.5% HCL blank solution until a semi-quant gives lower counts. If no decrease after 30 minutes, go to the next step and inform Patty (5-7671) of the issue.
37. Go to “Instrument” → “Plasma Off.”
38. Make sure instrument is in Standby Mode.
39. After 10-15 minutes (or when Exhaust temperature is below 30°C), turn off the heat exchanger.
40. Close Ar gas valves. Replace any empty tanks for next user.
41. Unclamp the tubings on the peristaltic pump.
42. Empty sample waste & rinse waste containers.
43. Sign out in the logbook.

Data Spreadsheet option:

44. Save your data spreadsheet to → D:\Userdataarchive\your name\file name.xls

Upper Concentration Levels for standard curves & samples

200ug/L or ppb for: Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Tl,
V, Zn, Th, U, Pd, Pt, Rh, Au

20,000ug/L or ppb for: Fe, K, Ca, Na, Mg

Total Dissolved Solid Limits

< 0.5% or 5g/L for all samples = For example, about 0.1 M NaCl

No suspended solids – this will definitely clog sample lines and/or nebulizer
(Please filter if needed with 0.45 um filter)

Agilent 7500 ICP-MS ChemStation Operator's Manual
Tuning

**Typical values of Tuning Parameters in the Standard Mode
 (with ShieldTorch, Micro Mist Nebulizer)**

Parameter	Typical Value	Adjustment
RF Power (W)	1500	Normally used 1500
Sampling Depth (mm)	8	7 to 10
Carrier Gas (L/min)	0.9	Normally used 0.9
Makeup Gas (L/min)	0.15	0.1 to 0.3
PeriPump 1 (rps)	0.1	
S/C temp (°C)	2	Normally used at 2°C

Typical values of Sensitivity (Standard Mode)

Mass	Count/1 ppb Integration time = 0.1 sec.	RSD
⁷ Li	>2000	<15%
⁸⁹ Y	>6000	<15%
²⁰⁵ Tl	>3000	<15%
Metal Oxides	156/140	< 2%
Double Charged	70/140	< 3%
Resolution Axis	Mass ± 0.05	(Adjust Axis Gain)
W- 10%	0.65 - 0.80	(Adjust AMU Gain)