How to Operate the *ELEMENT2* SC-ICP-MS

A Quick Guide and Introduction

Solution mode

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For Analyses of Solutions

Before Turning "On" the ELEMENT2 SC-ICP-MS

1) Switch on both of the desktop monitors.

NOTE: the computer is usually already "**on**", but the monitor is normally "**off**" or in "sleep" mode

2) Sign onto the computer using the following keys:

Description	**Login Name**	**Password**
Administrator e.g. Laboratory Manager	Administrator	(see Bill McDonough)
General (starter) e.g. temporary users: graduate students, visiting faculty, etc.	Element2	Plasma$3685

3) Open the "**LAN Driver**" program as well as the "**Executive**" and "**Instrument**" programs

4) Before initiating the plasma ignition sequence, make sure to check the following parameters:

a) Vacuum conditions (High Vacuum < 2.00E-7 mbar)
b) Argon pressure (> 110 psi; Figs. 1 & 2)

NOTE: If P < 110 psi, check the Argon gas tanks in the closet outside, tank on the left is for the ELEMENT 2 Ar supply;

- Top valve → Fill level of tank; if below 1/4 → Tell Richard Ash and/or open back-up tanks
- Right valve → P inside tank, usually barely visible on the scale of the valve
- Left valve → Output P, shows value which can be read in the PlasmaLab

If the tank is new, P can be low because the tank is full of liquid and little vapour is available → Turn green valve (pressure builder) to allow warm air to warm up the interior of the tank, so that more Ar evaporates and P increases.
Attention: Close the pressure builder at the end of the day if you have opened it!

c) Condition of the sample and skimmer cones (replace with clean cones ~1-2x/week)

Procedure for Turning "On" the ELEMENT2 SC-ICP-MS

1) Turn on the Chiller. The power switch is on the back of the unit and the refrigeration switch is on the front (the refrigeration switch usually stays in the "on" position)

   NOTE: The unit operates at 4º C, so before running any solutions make sure the chiller has had adequate time to reach this temperature

2) Attach the spray chamber (cyclonic or Scott double-pass spray chamber) to the injector sleeve on the plasma torch and connect the gas line of the chamber to the argon gas supply on the ELEMENT2 (connection labeled "Ar GAS (0-2 L/min)"). Make sure that the sample injection tubing is prepared for sample uptake in the hood (keep it in Milli-Q until ready for sample analysis) and wrap the drainage tube around the rotary pump below the gas line

3) In the "**Instrument**" program press the "On" button to ignite the plasma. Pay close attention to the ignition sequence for the plasma:

   NOTE: After checking the peripump tubing and ensuring that the pump rotates in the proper direction for drainage (clockwise or counterclockwise, depending on the positioning of the inlet from the spray chamber and the drainage; Fig. 3), verify that the drain tubing is properly connected and click on "**Start Peristaltic pump**" in the prompted windows that pop-up when you start the plasma ignition sequence

   a) The *cooling system* box will turn yellow then green as the cooling water begins to flow through the system
   b) The coil and sample cone turn yellow then green as the coolant water begins to circulate and increases flow to its full flow rate
   c) The *main gas* meters will turn yellow, you should hear the sound of gas flow starting, and then the meters will become green as the gas flushes the system for approximately 2 minutes. In the "**Instrument**" program the gas levels should read as follows:
      i) Cool Gas → 16.0-16.5 L/min
      ii) Auxiliary Gas → ~1.0 L/min
      iii) Sample Gas → ~1.0 L/min
   d) After about 2 minutes, the *interface pump* box will turn yellow then green as the unit turns on (you should be able to hear the pump when it turns on, it sounds like a small motor)
   e) The *sample gas* meter will turn red as the sample gas pressure falls to 0.0 L/min as the torch moves in order to ignite the plasma

f) After a ~20 second delay, the *plasma* figure will turn yellow and a faint clicking sound will indicate the torch trying to ignite the plasma. The *plasma* figure will turn green and the blue shape of a flame will protrude from the figure when the plasma is ignite

NOTE: You can make sure the plasma is ignited by checking to see if there is a bluish hue radiating from the torch box of the ELEMENT2. If for any reason the clicking sounds stop and the plasma fails to light, hit the "**Stop**" button in the "**Instrument**" program to abort the plasma ignition sequence

g) The *sample gas* meter moves up from yellow to green as the sample gas is switched on after the plasma is ignited

i) When the preset pressure of the sample gas is reached, a blue spray will appear in the *spray chamber* box, indicating the readiness for sample intake

h) The *skimmer valve* box will turn yellow then green as the valve opens; you should hear the valve hiss as it open (it takes ~5 seconds)

NOTE: Failure to open the gate valve results in the plasma being extinguished (indicating a possible O-ring malfunction)

4) Allow the ELEMENT2 to warm up for about 20 minutes, and then you will be ready to tune the mass spectrometer

Creating a folder for data storage

A new data file for data storage is created every day.

- In the "**Instrument"** program (Window "Executive – Thermo ELELMENT), go to "Customize" and choose "Directories": The window "Customize Directories" opens
- Go to "Data", "Browse", "Data" and choose "Create new folder"; name new folder after the current date, e.g. …Aug1_05
- Press " OK" twice

Tuning the ELEMENT2 SC-ICP-MS

1) Open the "**Tune**" program:

2) Open a tune parameter file (tpf) (it is stored in the "**idsc**" folder, which should automatically open if you are in the appropriate user account).

   a) From the file menu select "**open**".

   b) Select the file you want and click "**Open**".

      For Li analysis: Choose the file Li_tune_Cycl_Normal.tpf OR ScotDP_Li.tpf, depending on what you are using.

3) Select a scan list for tuning.

   a) Click the yellow chart icon (2 peaks) on the toolbar → "Scan List" window appears

   b) Enter elements and isotopes manually in the top half of the window

      or

      click "**load**" in the bottom half of the window to choose a saved scan list, e.g the file SolTune.scl (Sc, In, U).

      For Li analysis: File "Tune_Lithium+Na.scl"

      or

      click **"load"** and choose "Tune_range Ox" to check interferences with oxides

   c) Click "**OK**" when satisfied.

4) Click "**start scan**" (green arrow) to accumulate data on the selected mass stations.

   For Li analysis: Check the Li background in 2% HNO_3 using the "Tune_Lithium+Na.scl" file. Background for ^{7}Li should be < 3000 cps, for ^{6}Li < 400 cps; be aware that with a low Li background, you might have higher abundances of U oxides; vice versa, tuning for low abundances of U oxides usually results in a higher background for Li.
   Note: A low Li background can be achieved by adjusting both the sample and the auxiliary gas flow to the same value, e.g. 1.12;

5) Introduce tuning solution (1 ppb UMD-1)

6) Adjust the gas flow rates. Do not change the "**cool gas**", but adjust the auxiliary, sample and additional gas 1. Optimize the torch position ("**x**" and "**y**" values only) such that it gives a flat mass response curve and maximum signal intensity. It's better to click on + and – and not to move the bar.

- To change gas flow rates, open the *Plasma Parameters* dialog box by right clicking on the plasma-torch icon next to the gas flow panel.
- To change the torch position, right click on the icon in the torch position panel.
- Do NOT modify the "**z**" torch position or the values for cooling gas flow rate (for most application, use the –5.000 position).

7) When machine is well tuned, note cps for Sc, In and U in lab book

Note: The signal using the Scott DP is generally lower than using the cyclonic spray chamber,U should have a higher signal than In because it is heavier; U produces oxides easily, so U oxides can be a problem.

Ex. cyclonic spray chamber: ^{115}In ~ 1.2 x 10^6 and ^{238}U ~ 1.5 to 2 x 10^6 is ok
Ex. Scott DP: ^{45}Sc ~ 500000, ^{115}In ~ 900000 and ^{238}U ~ 1.1 x 10^6 is ok

For Li analysis: ^{6}Li ~ 18000, ^{7}Li ~ 250000 for cyclonic spray chamber and ^{7}Li ~ 120 000 for ScotDP are reasonable signals.

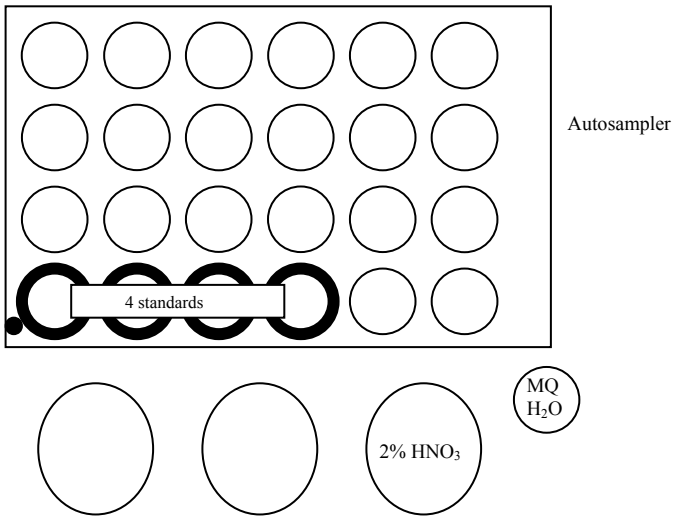
8) Click on the disk symbol "**Save Current Parameters**" when changing to other files

9) Click "**Stop**" (red square symbol) when finished with tuning

10) See the Lab Manager if you need to run a mass calibration.

Preparation of the autosampler

To select correct autosampler holder, go to "Executive" → "Customize" → "Autosampler" and click & drag.



How to Setup up a Method/Element Menu

1) Open the "**Method Editor**" program.
2) Open the "**View**" menu.
3) Select "**Spread Sheet**"
4) Choose elements/isotopes manually by entering them in the form "**Ca43**" in the *Isotope* column. Or, you can load an existing element menu by choosing "File -> open".

   For Li analysis: Open folder "Li research" and choose the "Li_escan3.met" spreadsheet
5) In the *Mass Window* column select an appropriate value.
6) In the *Settling Time* column enter appropriate values.
7) Enter the amount of time per cycle to spend on each isotope in the *Sample Time* column.
8) Enter how many times to sample each isotope per cycle in the *Samples Per Peak* column.
9) Save your element menu/method file. (This file is saved in the desc folder.)

How to shutdown the ELEMENT 2

1) Put tube in Milli-Q H_2O for 5 min
2) Put tube in air for 5 min
3) In the Instrument-Window, click "Off" and wait until the automatic shut-down is finished
4) Go to "Instrument" and click on "Peristaltic Pump Off"
5) Switch off the chiller
6) Unhook tube from peristaltic pump
7) Switch off the autosampler

Analyses of Lithium in aftercuts

How to run a Li analysis

1. Blank

1) Open the 'Sequence Editor" Program

2) Go to "Customize" → "Templates" → "Blanks"

3) Click on yellow icon to open the "Template Gallery", choose "**MMLi blank**"

4) In the window "Methods used by BLK analyte", use the following parameters:

- Data file: Name after date, e.g. "20July_2005_"
- Method: "Li_escan3.met"
- Tune parameters: ScotDP.tpf OR Cycl_Normal.tpf, depending on which spray chamber you are using;
- Report: "Just_Intensities.arcf"
- Quantification type: Intensities
- Take-up time: 45 secs (measure the take-up time before you start)
- Wash time: 30 secs for Li measurements in after cuts; a longer wash time is needed for samples with more Li in them!
- Pumping speed: 5

5) Drag green BLK (blank) box to the sequence between Start & Stop

6) Drag green Blank1 box from sequence to the large #1 autosampler position, which is 2% HNO_3 → Blank 1 00/01 appears in the green box

7) Save sequence file (File → Save → July_20_)

Note: It is recommended to have just one sequence file for blank, standards and samples (up to about 24)

8) To check and run the sequence, click on blue flag symbol; Attention: De-click print boxes at the bottom of the window!

9) Click "check".

- If "errors" occur: Sequence does not run
- If "warnings" occur: Possibly boxes for "Sampling" and "Internal Standards" not filled in; this is no problem for solution work;

10) To look at the blank measurement, see the blank file in the "Results" program

2. Standards

1) For Li analysis, 4 standards should be on the sample holder (0.1, 0.5, 1 and 5 ppb Li)
2) Go to "Customize" → "Templates" → "Standards"
3) In the window "Template Gallery", choose "**MMStd**"
4) Use the following parameters:
   - Data file: Name_Li100ppt, Name_Li500ppt, etc.
   - Method: "Li_escan3.met" (in the folder "Li_research")
   - Tune parameters: "ScotDP.tpf" OR "Cycl_Normal.tpf"
   - Blank: Browse and open the blank you've just measured before
   - Calibration: Type in file name, e.g. "Licalib" (a calibration file is created automatically when the calibration is finished)
   - Report: "FQ_Intensities_Concentrations.arcf"
   - Quantification type: Calib. (EXT.CALIB)
   - Standard: Browse and choose the correct standard in the folder "Li-research"
5) Drag standards from blue window STD into e.g. June23.seq window to add standards to analysis and change the name of standards accordingly. Drag standards from the sequence into the positions where they are in the sample holder;
6) Run the sequence
7) Look at calibration curve in the e.g. June23 folder, using the RESULT program; you must look for files of the type "CalibFiles"

3. Samples

1) Go to "Customize" → "Templates" → "Samples"
2) Choose "**MM Li samples**"
3) Use the following parameters:
   - Data file: June23a_1
   - Method: Li_escan3 (in Folder "Li_research")
   - Tune parameters: "ScotDP.tpf" OR "Cycl_Normal.tpf"
   - Blank: e.g. "Blank1.dat" (browse for the blank file of this day)
   - Calibration: e.g. "LiCal.dat" (browse for the calibration file of this day)

- Report: “FQ_Intensities_Concentrations.arcf”
- Quantification type: Quant. (EXT. CALIB)
- Take-up time: 45 secs
- Wash-time: 30 secs

4) To fill the sequence, click on the “Fill down” symbol and put in the following information:
   - Start ID: First number after blank, standards and the first sample
   - Rack No: 1 (0 = Milli-Q, HNO_3, etc.; 1= samples)
   - Vial Position: Vial corresponding to the starting ID sample
5) Save sequence
6) If you use a second rack, start a new sequence and remember refilling H_2O and HNO_3 if necessary.
7) Watch the “Spectrum Online” (sub-window of “Show-Thermo ELEMENT) during analysis
8) To check the results, open the respective files in the “Results” program; click on “C” to obtain concentrations.