

ICP-MS Operating Instructions

Version 2.1

1/3/13

Preparing instrument to run:

- 1) Check the Argon gas pressure in the cylinder room to make sure there is enough gas for the run. An average run of 40 samples will use approximately 500 psi of gas. The manifold is set up so that one tank is fully used up before it switches automatically to the other tank. An alarm will sound when a tank is empty; this can be silenced by pushing the silver button on the panel. See the lab manager for assistance, if necessary.
- 2) Turn the auto-sampler on. The switch is located on the back, in the lower right-hand corner.
- 3) Snap the clip closed on the back of the auto-sampler (located on the lower left-hand side).
- 4) Place the tubing on the peristaltic pump in the two center positions and pull it tight to place the tubing clips into the clip holders.
- 5) Close the two center arms over the tubing, making sure that the tubing is in the center of the arm and not being pinched.
- 6) Ensure that there is enough rinse solution (de-ionized water) for analysis. If not, fill the bottle from the Purelab water system in the lab. Place the rinse tube, which is the large tube without the small tubing taped to it, into the rinse solution. Place the waste tubes into the waste bottle. Empty the waste bottle, if necessary, into the drain inside the fume hood. Run the tap water in the drain for several minutes after dumping waste to dilute it. The sink drains into an acid neutralization chamber which will effectively neutralize the waste.
- 7) Launch PlasmaLab software if it is not already running. Record the Analyzer Pressure, which is located in the upper right-hand side of the Control tab.
- 8) At the top of the PlasmaLab screen, click *On*→*Yes* to put the instrument into an operational state.
- 9) Wait about 1 minute for the instrument to go through various checks and for the plasma to ignite. The peristaltic pump will start turning and the “On” button will turn green on the Control tab when the instrument is ready to perform analyses.

Checking instrument tune:

- 1) Go to *Tune*→*Manual*→*Sampling*→*Control*. Hit *Initialize*. This will enable the previously grayed-out buttons.
- 2) Set the rack to “0” by using the arrow keys, set the column and row to “1”, and enter “150” for depth.
- 3) Place the tuning solution (usually 1 ppb Indium) in the far left position of the back rack (the rack that holds the larger tubes), and remove the cap.
- 4) Hit *Go to tube position*.

- 5) Monitor the Indium intensity and stability. The optimal intensity will depend upon the age of the detector; typically the counts should be 60,000 + and stability should be less than 3. Once the Indium counts are stable, record the value in the logbook. If the intensity is low (relative to recent values) and/or stability is high, the instrument may need optics and/or torch box tuning. Perform tuning if necessary, but do not use the auto-tune for the torch box, as this causes the torch box to lose alignment over time. Please seek assistance if you are not experienced at tuning.
- 6) Hit *Go to rinse station*. Allow system to rinse until the tuning solution has been washed out. This can be monitored graphically or by looking at the numerical Indium counts. After the tuning solution has been washed out, hit *Pump Off* and *Initialize*.

Creating an experiment:

- 1) Click the “Experiment” button on the left side of the PlasmaLab main screen. A blank experiment can be created, or an experiment can be created based on a template or an existing experiment. Choose your preference; it is usually easiest to create an experiment based on an existing experiment. An existing experiment can also be selected from this window for data review.
- 2) If prompted for the analyte database for experiment, choose “Default”.
- 3) In *Setup*→*Analyte*, choose desired analytes by clicking on them in the periodic table. Select the desired isotopes for each analyte by clicking the check box next to them. Double-clicking on the analytes when selecting them in the periodic table will automatically select the most commonly analyzed isotope for that analyte. To remove analytes, right-click on the desired analyte and select “Remove analyte” from the pop-up menu. The internal standard(s) will appear on this screen in yellow.
- 4) In *Setup*→*Acquisition Parameters*, select *Continuous* from the drop-down menu.
- 5) In *Setup*→*Internal Standards*, choose internal standards (usually 1 ppb Indium, sometimes also Bismuth or other elements).
- 6) In *Setup*→*Isotope Ratio*, specify isotope ratios (if desired).
- 7) In *Instrument Parameters*→*Configuration Editor*, select “PQ Excell CCT Sim Default” under *Configurations*. Select the most recent tune (at the top of the list) under *Instrument Settings*.
- 8) In *Instrument Parameters*→*Timings*, type in 120 for the uptake and washout delays.
- 9) In *Calibration Method*, all analytes use the “fully quant” method except for internal standards, which use “none”. Make sure the *External drift correction* checkbox is unchecked if using internal standards.
- 10) In *Sample List*→*Sample List*, list all standards and samples in the following order: the blank, the standards from lowest to highest concentration, and the unknowns. The blank is the matrix used for the standards and samples (usually internal standard solution). The standards are typically multi-element solutions with

- various ppb-level concentrations and a NIST 2709 sample solution that is run as an unknown (this is a check standard). A 2709 sample should be run every 15 samples for quality control. For “type”, blank=blank, standard=fully quant, and unknown=unknown. Make sure that the samples are in the same order in both the sample list and the auto-sampler. Enter the rack, row, and column information. The Height is 150 mm. Enter 3 for Main Runs; this will analyze the sample 3 times and take the mean as the concentration.
- 11) In *Sample List*→*Fully Quantitative Concentrations*, make sure all of the standard concentrations are correct. Also make sure the units are correct at the top of the columns.
 - 12) Save the experiment by going to *File*→*Save As*, then your experiment name. Make sure the experiment is saved in the correct folder.
 - 13) To start the run, hit the “Queue” button at the top of the PlasmaLab screen. Hit *Append*→*OK*, and the samples should start running after a brief delay.
 - 14) The total run time can be viewed by selecting the “Technician” icon on the left side of the PlasmaLab main screen. Also in this window, the instrument can be set to go to Vacuum when the run(s) is completed by selecting “Vacuum” from the drop-down menu labeled “Queue Empty Action”. Once the “Vacuum” command is selected, click on another part of the screen so that the shutdown procedure cannot be changed by scrolling the mouse button. **DO NOT SELECT “SHUTDOWN”** in the “Queue Empty Action” drop-down menu because this will break the instrument vacuum and turn the instrument completely off.
 - 15) Record the experiment name, number of samples, etc. in the logbook.

Shutting down instrument:

- 1) If the instrument has just finished analyzing a sequence, skip to Step 3.
- 2) Go to *Tune*→*Manual*→*Sampling*→*Control*. Hit *Go to rinse station*. Watch the screen until the sample counts drop to near zero.
- 3) Take the tube out of the rinse bottle, hit *Pump On*, and allow the residual water in the tube to be pumped out.
- 4) Hit *Pump Off*, then *Initialize*. This turns off the auto-sampler pump and resets the auto-sampler probe.
- 5) Hit the “Off” button at the top of the PlasmaLab screen if the plasma has not already been shut off by the “Vacuum” command in the Technician window. This places the instrument into a “vacuum ready” state and turns off the peristaltic pump after a short time. **DO NOT** hit the “Shutdown” tab located on the Control tab, as this will break the vacuum of the mass spectrometer and completely shut the instrument down.
- 6) Place the rinse and waste tubes from the respective bottles into the respective beakers. Cap both bottles.
- 7) Release the arms from the tubing on the peristaltic pump and loosen the tubing by releasing the tubing clips from the holders.
- 8) Release the clip on the auto-sampler pump and turn the auto-sampler off.