ICP MS manual – Perkin Elmer Elan; Soil, Water and Plant Testing lab

Dec 2012 (FIRST DRAFT)

Note: when the machine is used regularly, it is preferable to keep it running with the vacuum on. Only the torch is lit and turned off, before and after use.

1. Check there is enough gas in the gas tanks (argon, nitrogen?, any reaction gas you may use), and open tanks if needed. The pressure of the Ar should be at least 65 psi.
2. Turn on the four switches on ICP-MS body, left side toward the back
3. Turn on vacuum switch on ICP-MS left side toward the back. It will take 5-10 min to pump the vacuum. When it is ready the green light on the front of the machine will stop blinking and stay lit
4. Turn on water chiller/recirculator if it is not already on (blue machine under autosampler)
5. Check the peristaltic pump tubing (a thin one for the sample and a thick on for the drain) for any flat parts. If needed, put on a new one. If it looks good, connect to peristaltic pump (in grooves) and close the clamps. Put the sample tubing in deionoized/distilled water
6. Open the Elan program from the computer desktop (double click)
7. Under [devices] choose peristaltic pump. Type 20 rpm under flow rate and then click start. Check that the pump is sucking up water evenly (follow an air bubble). Adjust clamp if possible. Check also drain tube underneath spray chamber and on peristaltic pump for good drainage.
8. To light the plasma torch, either hit the plasma start button (green) on the machine or under [instrument] choose plasma and click start
9. After about 2 minutes the torch will be lit. Check on the screen that all components of the system show up green on the image.
10. *Optional*: under smart tune check performance of nebulizer, autolens, and daily performance. For daily performance use e.g. mass cal solution containing e.g. Berillium, Indium, Uranium, Cobalt. See how many counts you get from those standards (should be e.g. >10,000 for In but can be 40,000 under the best conditions).
11. To create a new method: under method, choose new. Quantitative analytical method is most likely what you want, and peak hopping from element to element mass. Enter the specifics of which elements to analyze (right click shows periodic table, and also isotopes for different elements). Set the number of sweeps (e.g. 10-20), the dwell time on each mass (e.g. 50 – 500 ms) – the total of sweeps x dwell time should be about 1 second per peak, so e.g. 10 sweeps at 100 ms). That constitutes one replicate measurement. Choose # of reps, e.g. three. Consider adding an internal standard, e.g. Indium for Selenium or Berillium for lighter elements (or both). In the list of elements you can select groups, identify them, and then indicate which one in each group serves as internal standard, and what the concentration is. Choose detector to use: analog (for high conc, ppm) or pulse (low conc), or both (dual detector) if some are high and some low. The calibration will have to be for dual too then. (*Note*: in case of questions F1 = help). Under calibration choose standards. The Perkin Elmer standards are 1 ppb. Tell it where to find the standards on the autosampler, and what concentrations they are. The autosampler can be pulled up under [devices]. The big holes on the autosampler are for the blank and standards and are called positions 1-10. The positions in the racks start at number 11 and they

run left to right for each rack. Under sampling choose unit (ppb = microgram/L) for standards and for samples (can be different). Choose peristaltic pump settings: 24 rpm is standard, but flushing is done at 40 rpm. E.g.: sample flush (at 40) for 10 sec, read delay (at 24) for 15 sec, analysis (at 24) for … sec, and wash (at 40) for 2 seconds. Save method.

12. Under Report quant summary + automatic NetCDF (comma delimited file that can be opened in excel). Checking crosstab box should give table of samples vs element conc for all elements analyzed. Under [sample] click [batch] for large sample set. Enter samples and what to do with them. Right mouse click, choose run blank, standards, sample for the first sample, then for the next one only run sample, etc. Every so often you can re-run standards. You can also add quality controls. For that click [enable QC checking]. Choose QC actions, and indicate where the QCs are and what criteria to use to let it pass or fail the QC. For instance, let it run one of the standards as a QC and let it fail when it's off more than 20% (tell if what to do if it fails, e.g. restandardize and recheck QC, rerun last group of samples). Save the batchfile.
13. On the left, vertical taskbar click R to set up saving of the data, which will be done in separate folders, save dataset file(s). Under scheduler, choose analyze samples, details, choose saved batch file, choose dataset created under R. Choose post-run actions, e.g. wash 5 min then autostop. Then click start.
14. If a method is already saved, just open that method, and create the files for that day for the batch of samples, the files to be saved (R) and the run actions.

For specific questions you can ask Jim Self, who can in turn ask the Perkin Elmer contact, Randy Hergenreder.

Note: The machine is ok with acids, e.g. 10% nitric acid is fine (use trace metal grade acid). The ICP-MS is very sensitive (ppt levels) and should not be saturated with high element levels in samples (more than 1 ppm is already high). So in anticipation you can dilute them in blank and run the dilutions first.

Note2: For some elements there may be an atomic mass interference. For instance, Se has interference from Ar2 around mass 80. Therefore it needs oxygen (DRC mode, channel B) as a reaction gas. Before the run all lines need to be purged with gas from the oxygen tank (at 7 psi) at 0.2 L/min, and oxygen is used during the run to create a SeO complex, which will come out at around 96 dalton, which hopefully will have no interference (check for isotopes of interest). The method will have to be optimized (look for "conditioning…").