

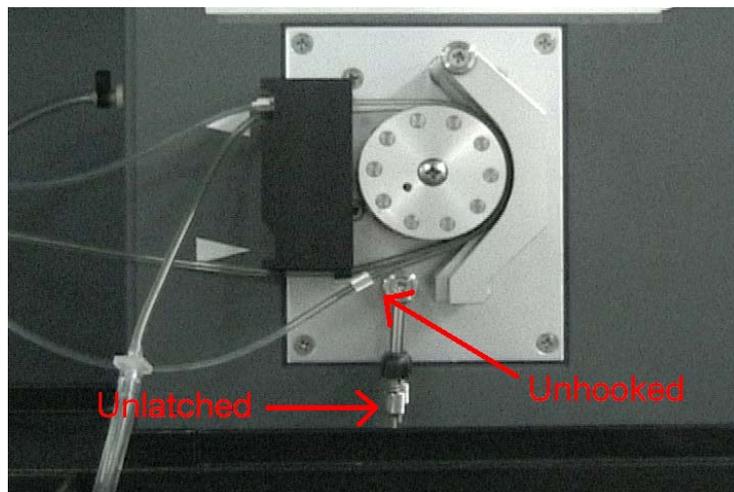
Basic ICP Use

Instrument Setup

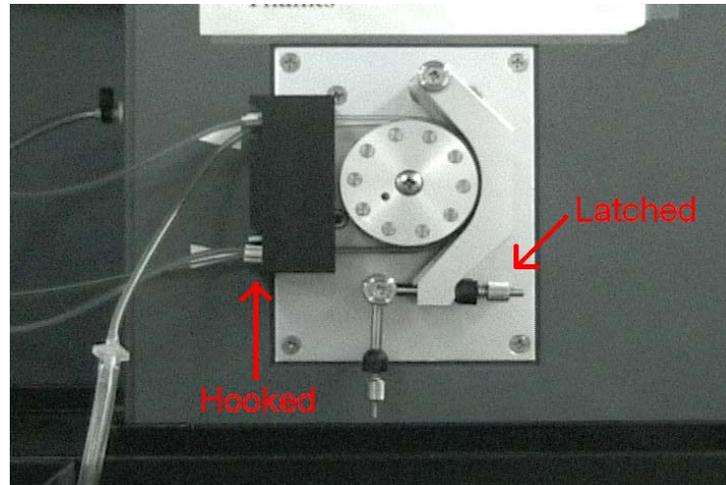
- 1.) On the ASL computer, log in to ICP-AES
- 2.) Check the pressure on the argon tank. It should always be on and set between 100-120. Turn the pressure knob to the left if necessary.



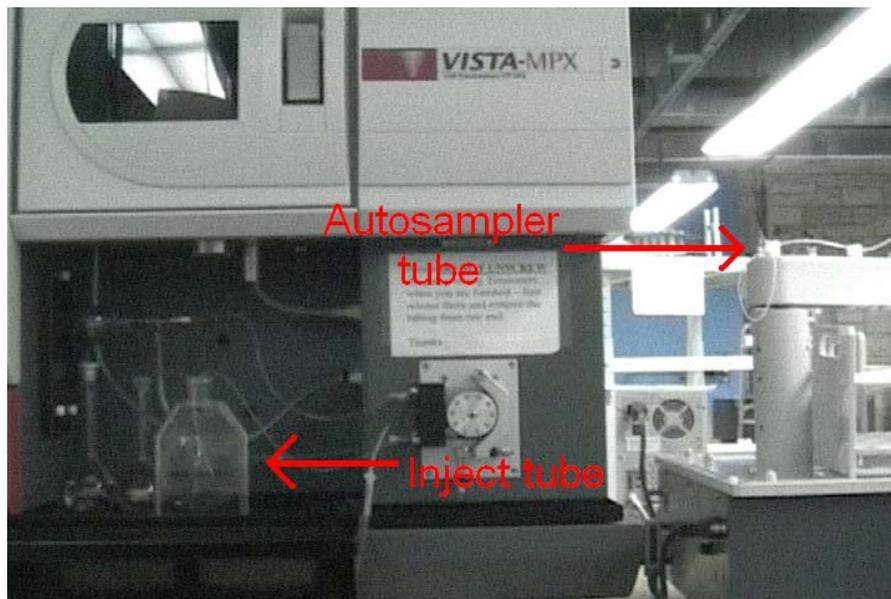
- 3.) On the automatic pump, snap the two plastic pump tubes into place by pulling on the hooks and snapping them into place.



4.) Lock the tubes with the hanging vertical levers. You only need to latch the outside and inside levers. You are free to lock the middle one but it doesn't do anything, so whatever. There are thumbscrews on the levers to adjust flow rate, but they already should be correctly adjusted. DO NOT MESS WITH THEM!



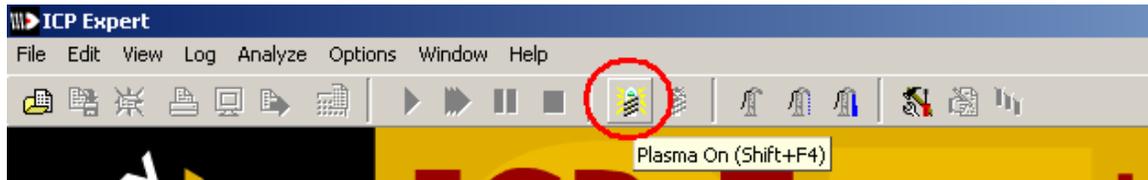
5.) If you are using the autosampler, connect the water tube (the tube that goes into the clear bottle full of liquid) into the autosampler tube.



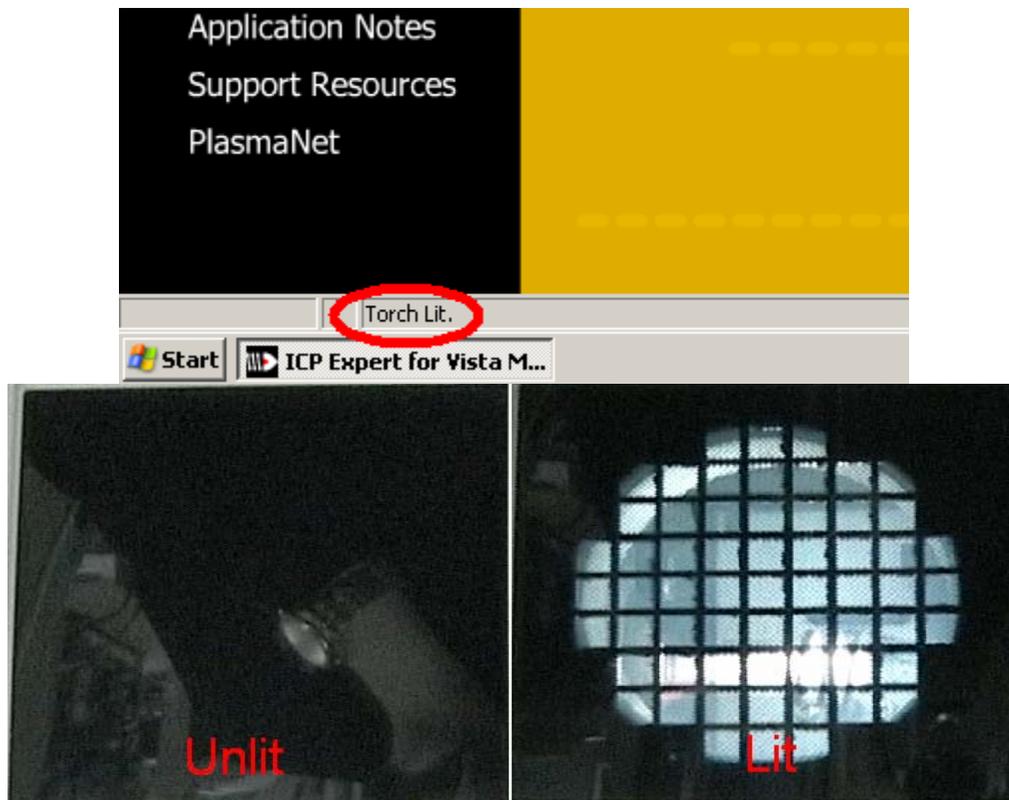
Computer Setup



1.) Whatever you do, **never ever shut the software on the computer down.** It takes very long to reboot.



2.) Turn plasma on by pressing the button in the tool bar.

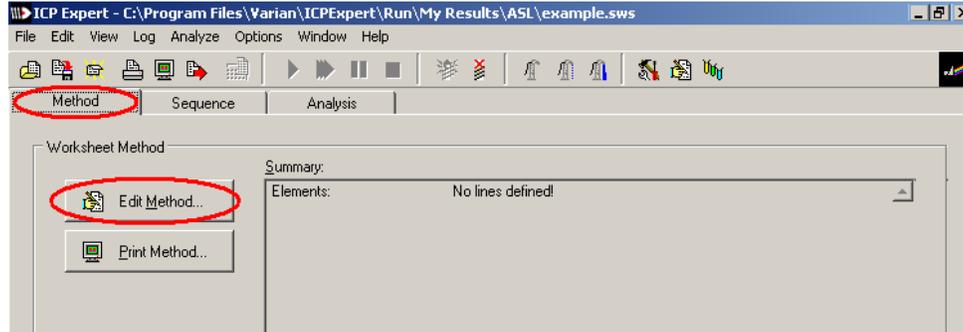


3.) The plasma will take a few minutes to turn on. You will know when it is completed because the status bar at the bottom of the computer screen will indicate that the plasma is on, and you will be able to see the light of the machine through the darkened window in the ICP.

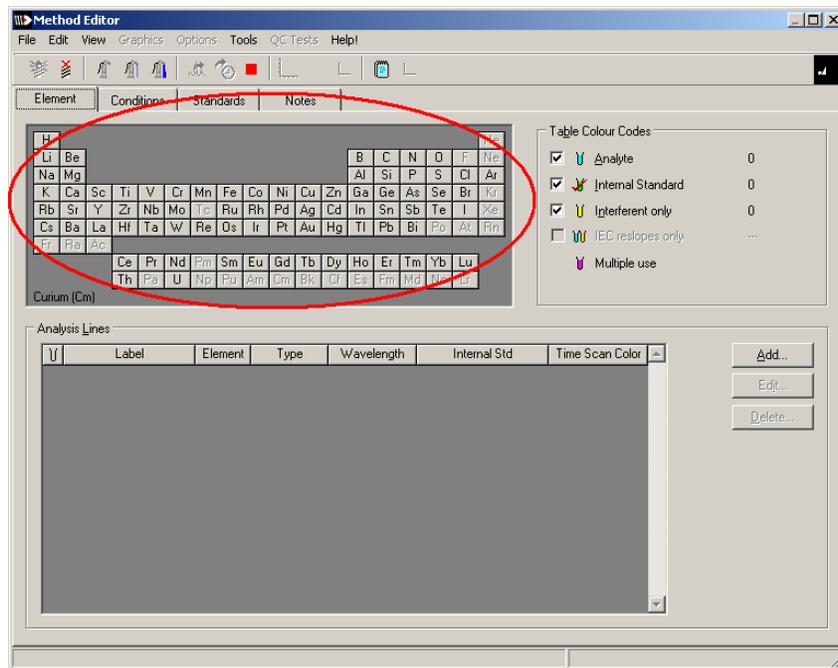


4.) After the plasma is on, click on the “Worksheet” button. Click “New”. Save the worksheet under your Lab’s folder under a name you can remember.

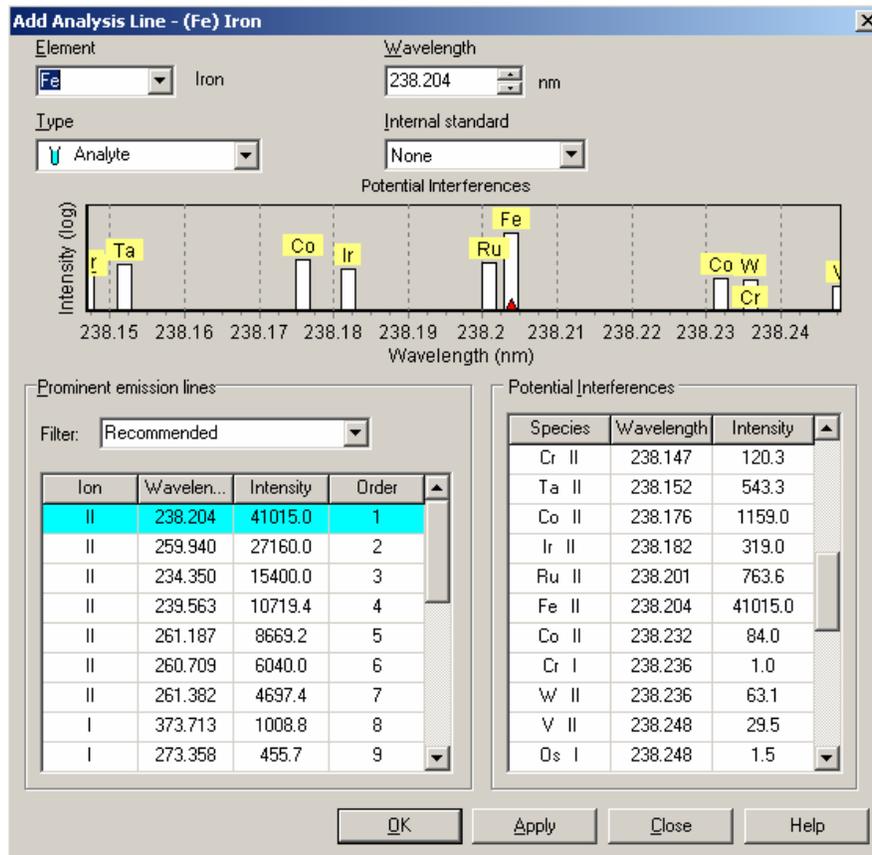
Edit Method



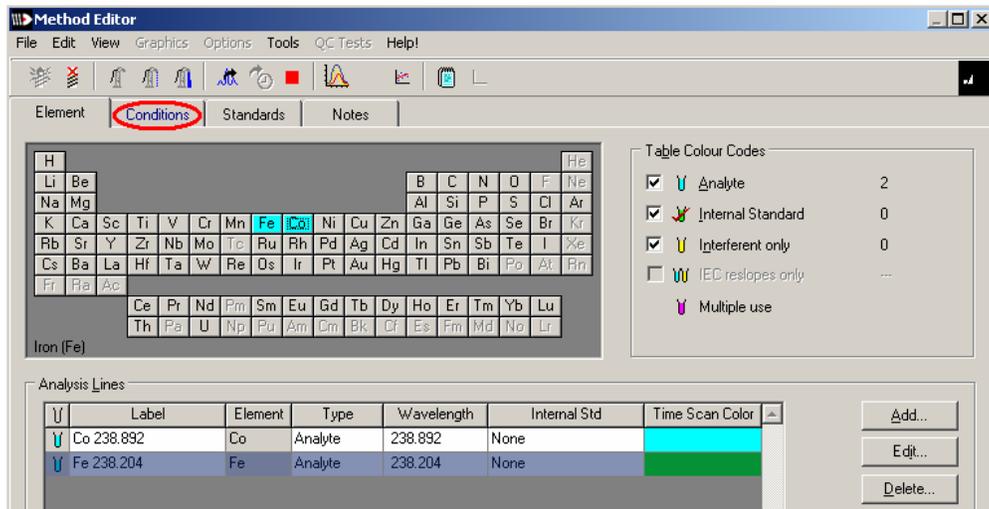
1.) Click Method->edit method



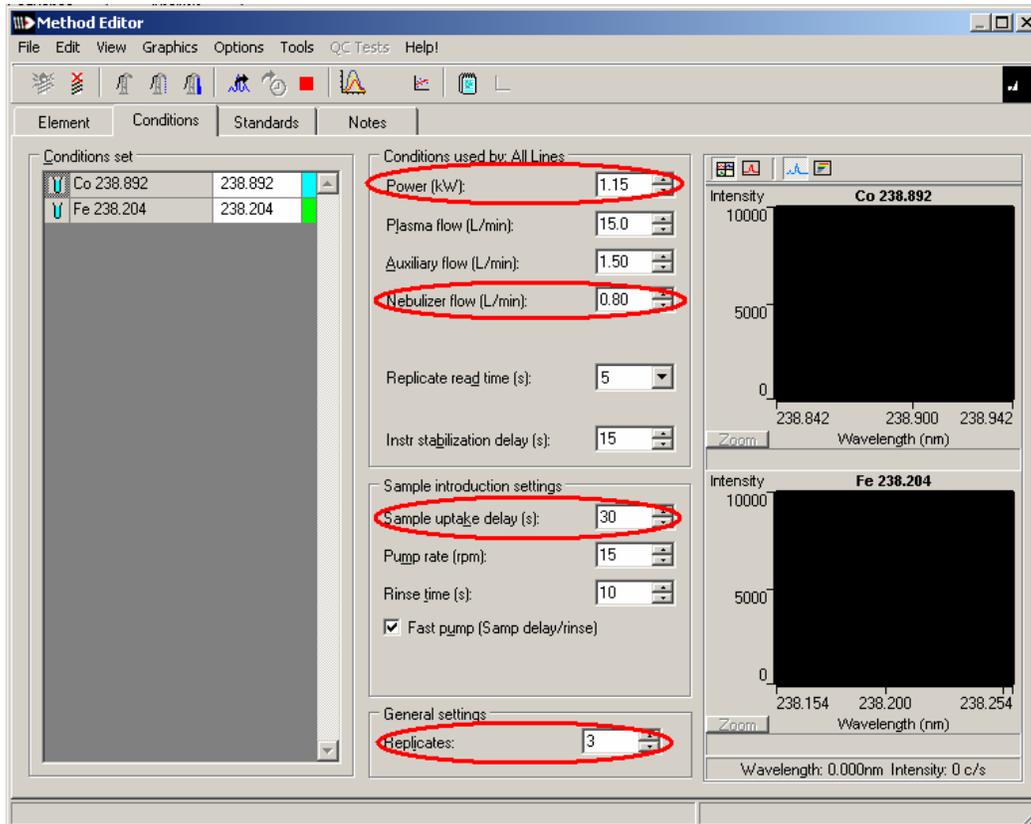
2.) Click on an element in the periodic table that you will be measuring.



- 3.) The new menu that opens up has three boxes, the bottom left “Prominent emissions line” lists the emissions for the selected element in order of intensity, the bottom right “Possible interferences” lists nearby elements that may interfere with your reading, the top is a graphical representation of both charts. Find a peak with a high intensity that isn’t likely to be interfered with by other frequencies.
- 4.) Select “apply” or “OK”. Be careful not to select both “apply” and “ok”, your scans will detect for that one peak twice and give faulty results for this element.
- 5.) Repeat steps 12-13 until you have identified peaks for every element that you are detecting for.



6.) Click on “Conditions” tab.



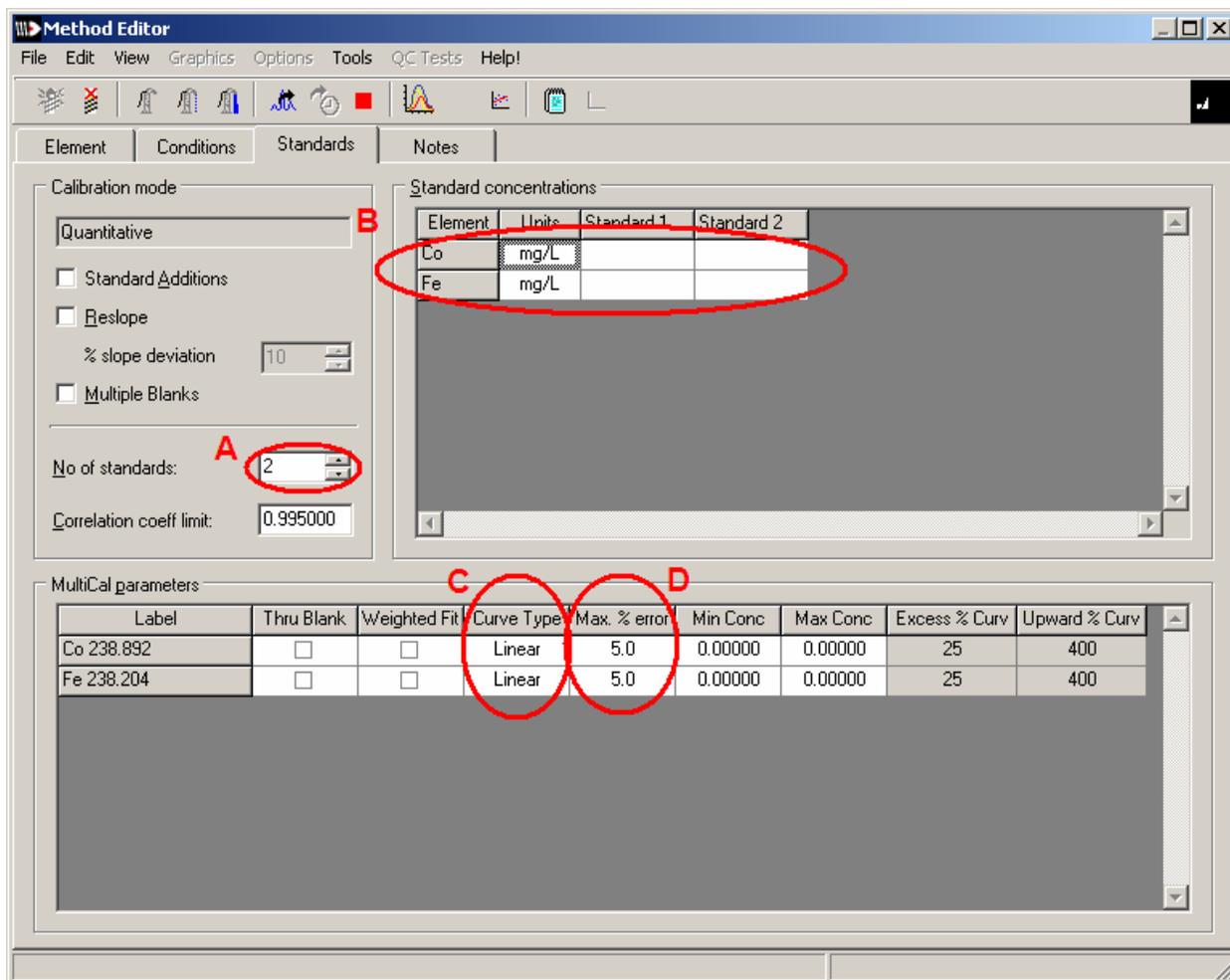
7.) Set the following conditions: **power** to 1.15kw, **nebulizer flow** to 0.80L/min.

Sample uptake is default at 30sec, feel free to change it between 25-30. Any lower than 25 and you will probably get systematically lower concentrations than you want.

Three **replicates** is standard, but feel free to increase this if you feel it's necessary. **DO NOT DECREASE THIS NUMBER.**

The conditions are used to maximize signal to noise ratio and get more repeatability in your results. Usually, the higher the number, the greater the S/N ratio but the longer the sample will take to run. Be prepared to tweak these settings if necessary to yield more precise results.

8.) Click Standards tab



9.) A: Change the number of standards to correct number. Column number should change accordingly.

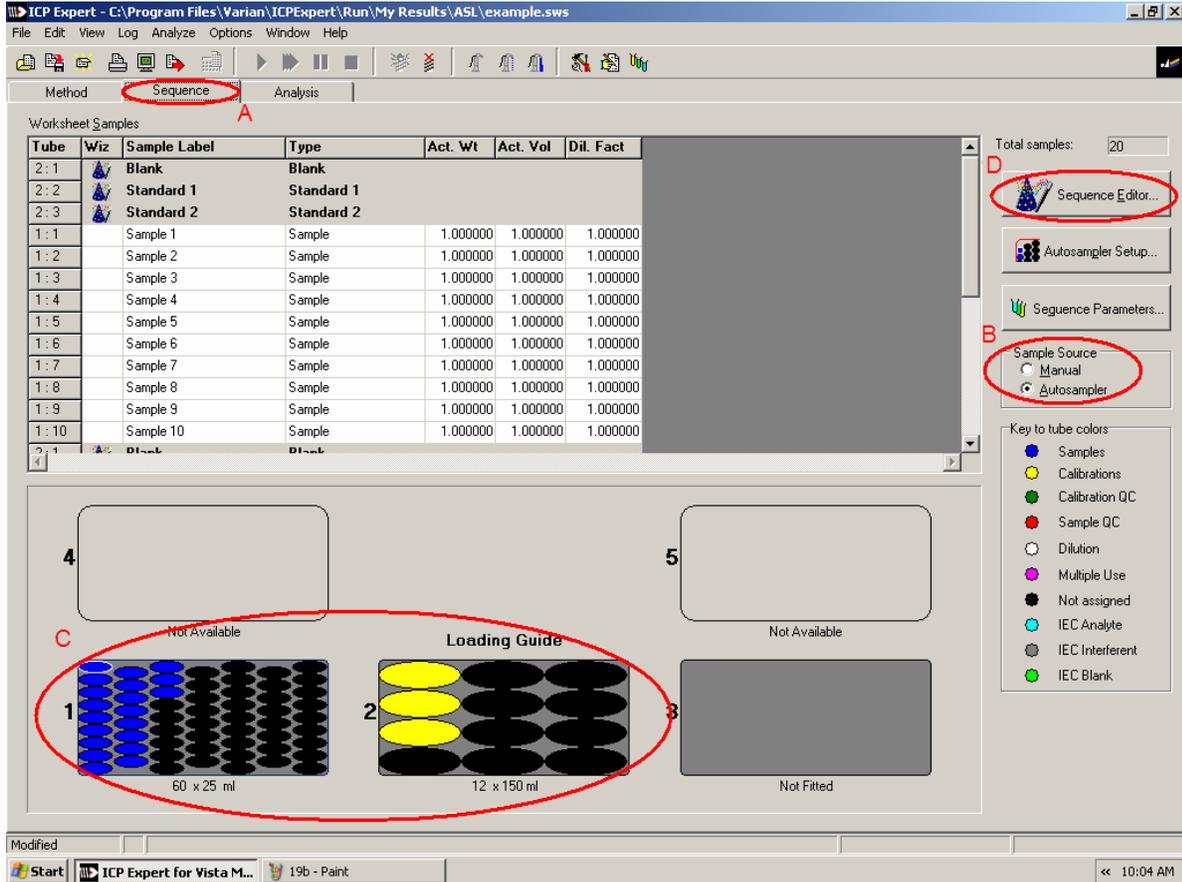
B: For each of your standards, enter the correct concentration (including unit of measurement) of each of the elements that you are measuring. A lack of any number is assumed to be zero. **At this point, it is important to keep your standards in order.**

C: Choose whether your calibration curves will be generated based on linear or quadratic data fits. (usually linear)

D: Change your percent error if necessary, 5% is usually standard. You may need to return and change these settings if your spectra get calibration errors and you're too lazy to remake your standards.

10.) Close the method editor and update the worksheet.

Sequence Editor

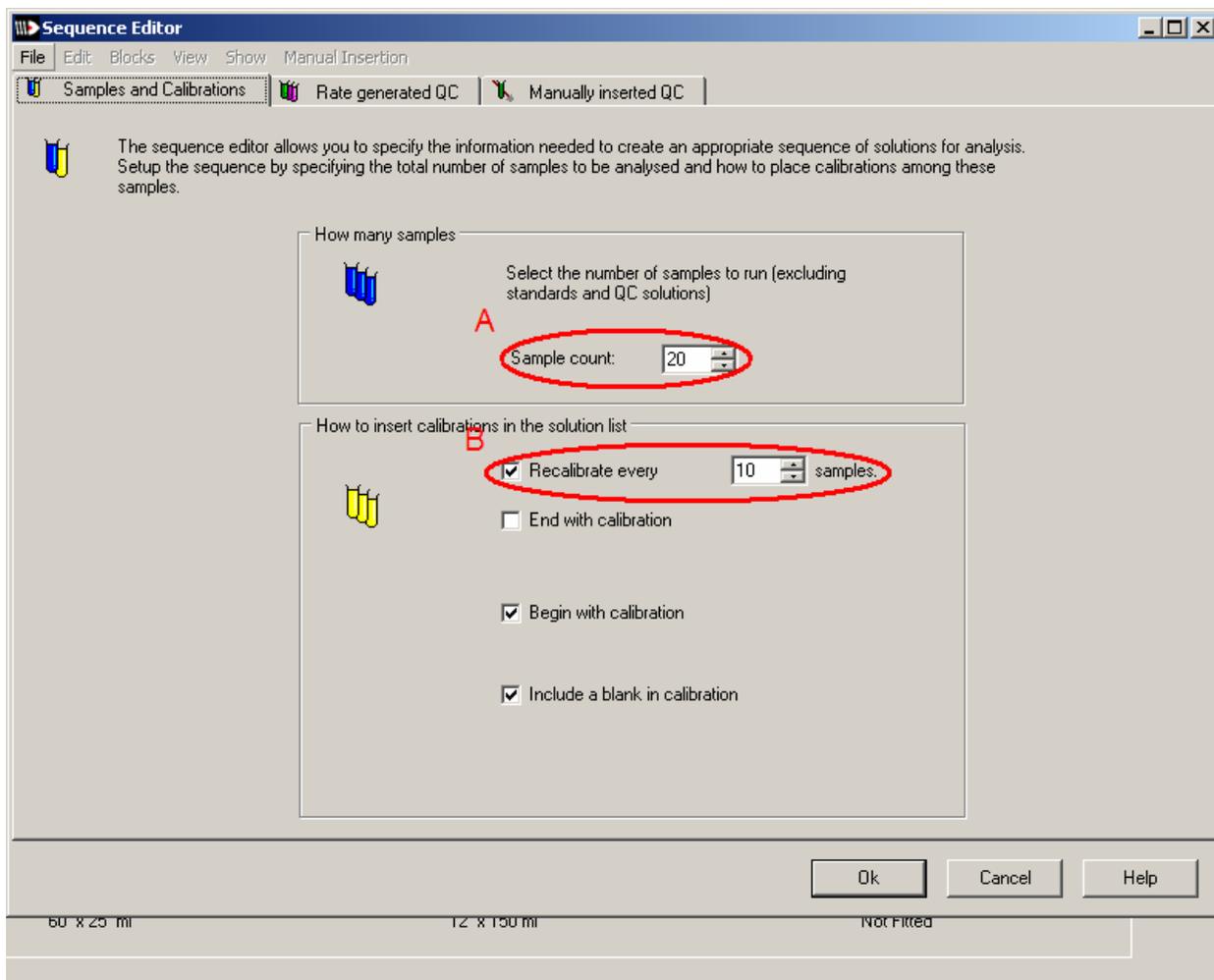


1.) A: Click on Sequence tab.

B: Choose between autosampler and manual.

C: If you are using the **autosampler**, now is the time to arrange your samples on the grid exactly as shown (especially the standards)

D: Click sequence editor.



2.) A: Choose your **sample count** (it never hurts to overestimate).

B: Choose the interval between recalibrations (10 is recommended).

3.) Exit the sequence editor.

Scanning and Analysis

The screenshot shows the ICP Expert software interface. The 'Analysis' tab is selected and circled in red. A red letter 'A' points to the 'Analysis' tab. The 'Method' and 'Sequence' tabs are also visible. The main window displays a table of sample labels and their corresponding concentrations for Co 238.892 mg/L and Fe 238.204 ppt. To the right, two spectral intensity plots are shown: one for Co 238.892 and one for Fe 238.204. The plots show intensity versus wavelength (nm) with a scale from 0 to 10000. The bottom status bar indicates 'Wavelength: 0.000nm', 'Intensity: 0.0 c/s', and 'All Replicates'.

Tube	Sample Labels	Co 238.892 mg/L	Fe 238.204 ppt
1	Blank	0.000000	0.000000
2	Standard 1	10.0000	
3	Standard 2		100.000
4	Sample 1		
5	Sample 2		
6	Sample 3		
7	Sample 4		
8	Sample 5		
9	Sample 6		
10	Sample 7		
11	Sample 8		
12	Sample 9		
13	Sample 10		
14	Blank	0.000000	0.000000
15	Standard 1	10.0000	
16	Standard 2		100.000
17	Sample 11		
18	Sample 12		

1.) A: Go to analysis tab.

B: Hit Play button. This activates the pump (it should start spinning pretty fast) and starts the sequence that runs the samples.

If you are using the autosampler, the ICP will begin scanning on its own. Make sure you've arranged your samples and standards according to the directions under the sequence editor section and skip to step 5.

For Manual

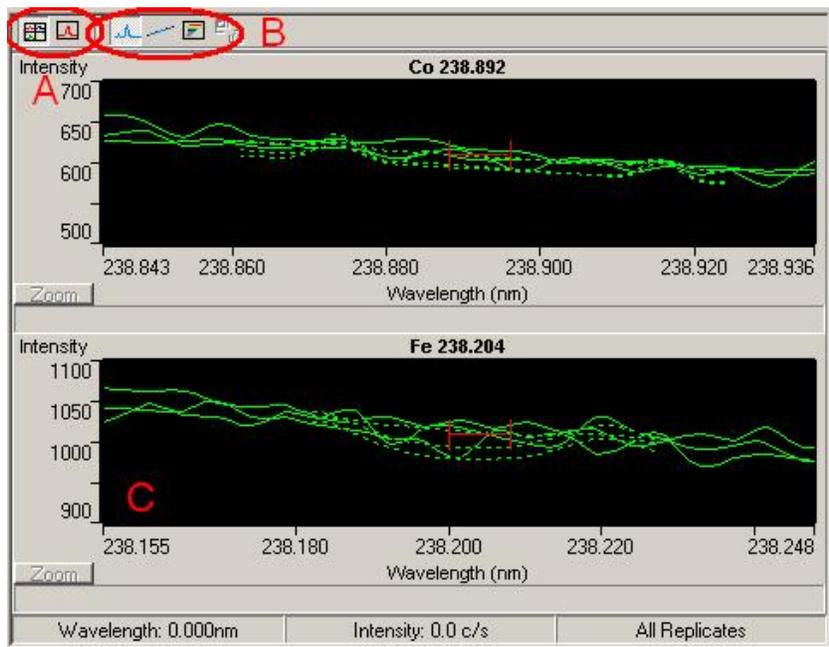
2.) Check the tube that pulls water out of the water bottle and into the ICP for a constant stream of liquid. If you see that the injection tube is taking up air bubbles, loosen the thumbscrews on the levers of the pump until the flow rate is smooth.

3.) The computer should be prompting you for a blank. Remove the tube from the DI water bottle, stick it into your blank and click "OK".

4.) The ICP will scan the blank three times and then prompt you for your first reference. Continue with other references and samples as prompted.

For everyone

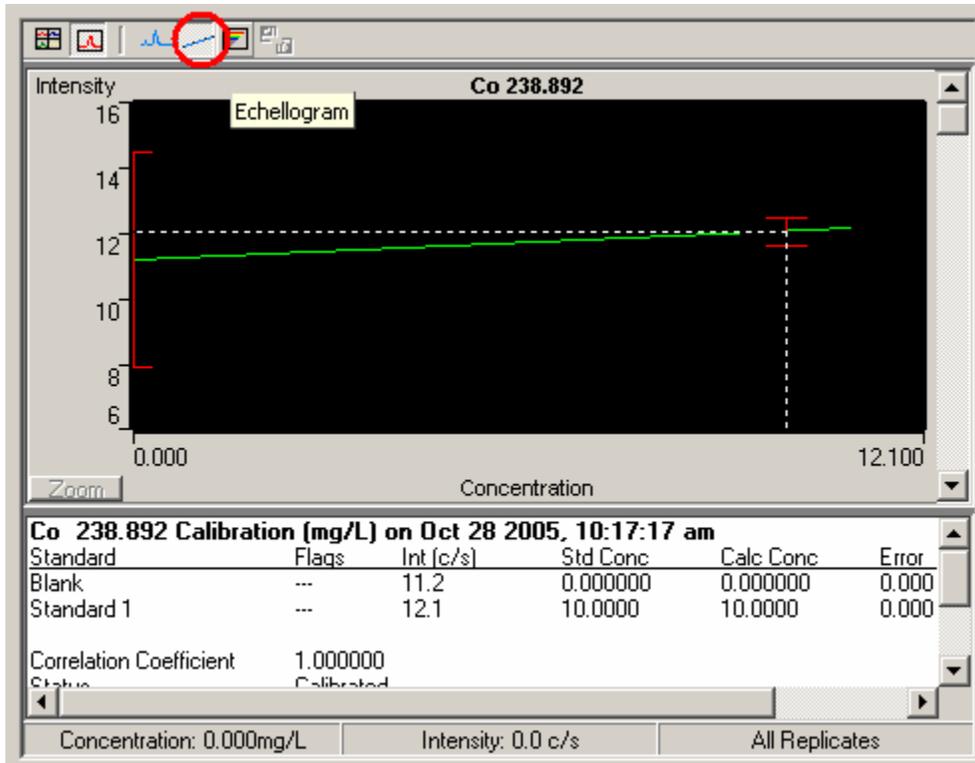
5.) If you are running your standards and you see a “calibration failed”, you have a problem with either your signal to noise ratio (it’s too low) or your sample. Please see step 7 for advice.



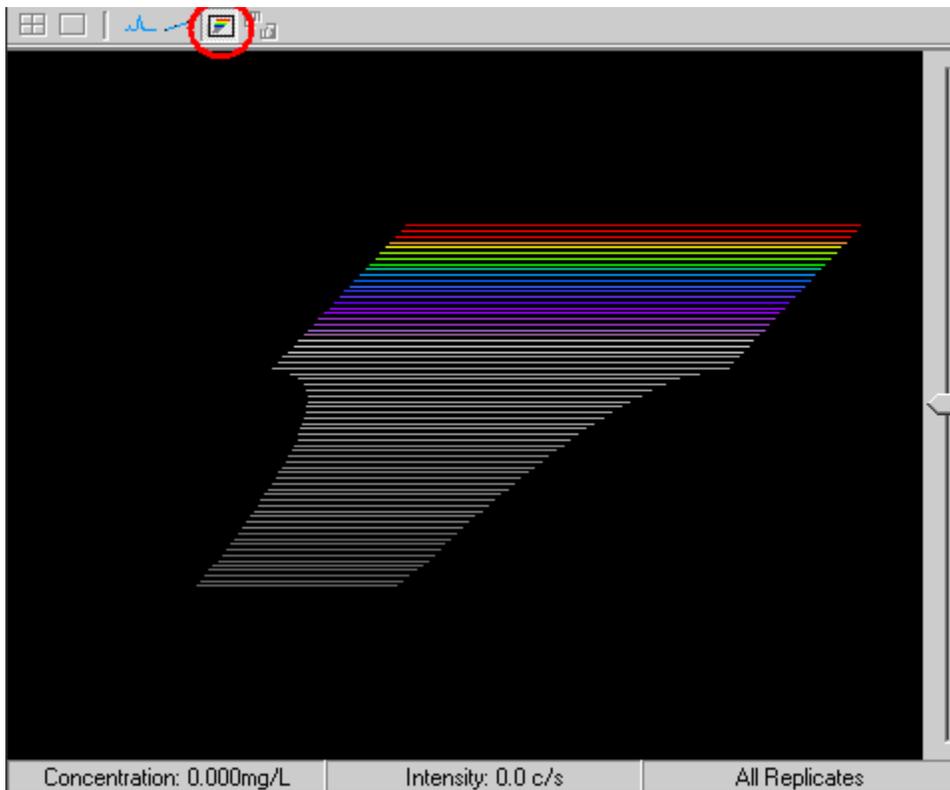
6.) After your samples start running, you will be able to look at a variety of spectra for each sample on the right of the analysis screen (shown above).

A: toggle between examining all elements vs. one element

B: Each of the three tabs corresponds to a graph option: overlaid spectra from each replicate (shown), calibration curve, emission spectrum (both below).



calibration curve



full spectrum view

15	Standard 1	10.0000	
16	Standard 2		100.0
17	Sample 11		
18	Sample 12		

10/28/2005 9:51:43 AM Worksheet Created
10/28/2005 9:51:43 AM Calibration Failed

ICP Expert - C:\Program Files\Varian\ICPEXPERT\Run\My R

File Edit View Log Analyze Options Window Help

Method Sequence Analysis

Tube	Sample Labels	Co 238.892 mg/L	Fe 238.204 ppt
1	Blank	0.000000	0.000000
2	Standard 1	10.0000	
3	Standard 2		100.000
4	Sample 1	-25.4956u	uncal
5	Sample 2		
6	Sample 3		

7.) Bad calibration or bad data points (both above) suggest a lack of correlation between replicates. You have two options.

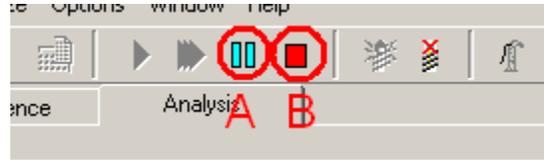
Tube	Sample Labels	Co 238.892 mg/L	Fe 238.204 ppt
1	Blank	0.000000	0.000000
2	Standard 1	10.0000	
3	Standard 2		
4	Sample 1		
5	Sample 2		
6	Sample 3		
7	Sample 4		
8	Sample 5		
9	Sample 6		
10	Sample 7		
11	Sample 8		

Copy
Copy Special
Edit Replicates... Enter
QC Actions
Delete All Results...
Sort Columns
Column Properties ...

The faster (and more scientifically incorrect) way: left click on the cell you want to edit, right click and select “edit replicates” to mask data points.

Otherwise, return to the method editor-> conditions and/or standards tab to increase your delay time parameters or increase your data-fit allowable error percentages.

8.) If you want to stop scanning, you have two options.

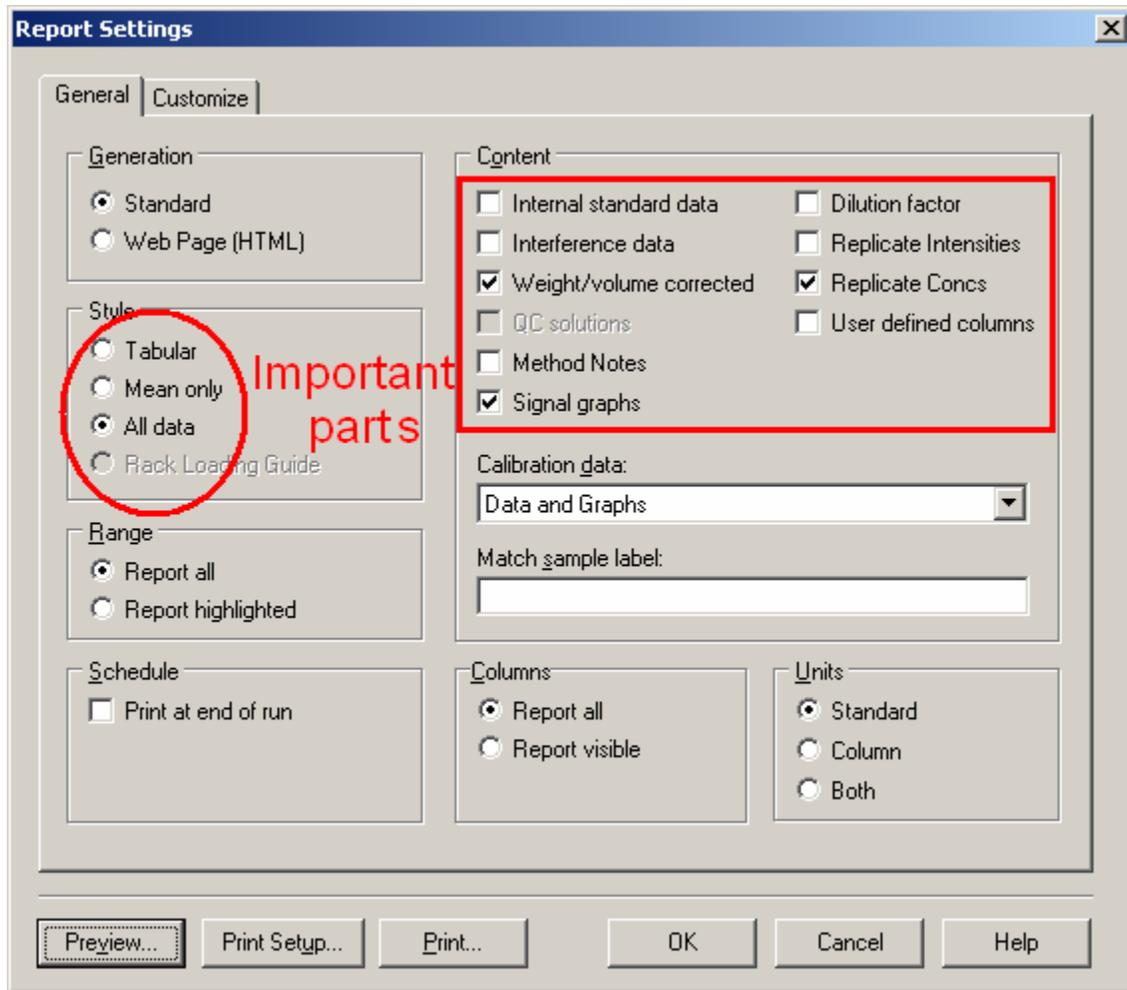


A: To finish a sample, record the data and then halt, press pause button.

B: To stop immediately and dump all data from the current sample, hit stop.

You can do either of these at any time regardless of whether you are running manual or auto which it why it doesn't hurt to over-estimate your samples.

9.) When you are finished, if you want to print your data, **DO NOT go directly to print**. Instead go to "File -> report settings".



In the window that opens, under the “style” box choose between “mean only” or “all data” if you want to print all of your sample runs. Under the “content” box, check “Internal standard data” for calibration info, “Replicate conc” for calculated concentrations and “Method notes” for a log of instrument activity during your run. Select what you want and print accordingly.

10.) To export the data, go to File->Export. Unless told otherwise, select “comma separated variable” or “.csv”. This sort of file is recognized by Microsoft Excel and will be easily used for most analysis programs. Save onto your disk (the ICP-AES has both a CD burner and a floppy drive, take your pick) and save a spare copy on your space on the ASL server.

For shut down:

- 1.) Save your work (file-> save)



- 2.) Run fast pumps for about a minute to clean out the system (button on tool bar, above).

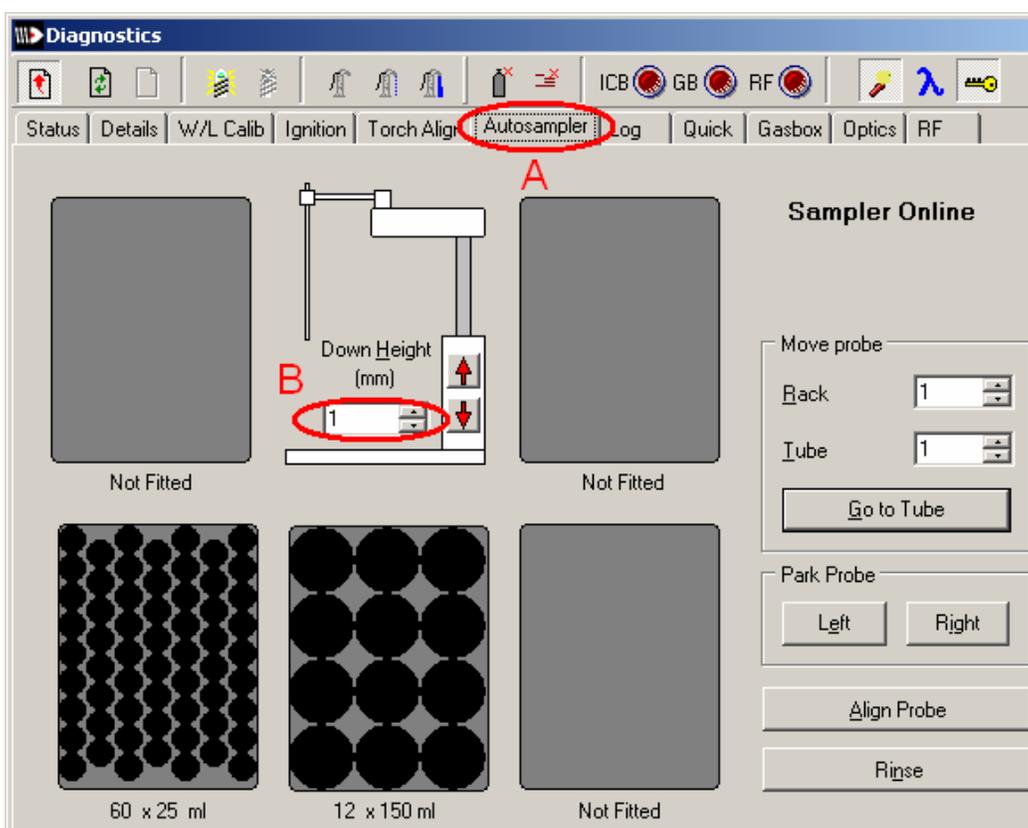


3.) Turn off the plasma with the button on the tool bar (above).



4.) If you used the autosampler, select the wrench and hammer button on the tool bar (above).

A new window opens:



A: Select the Autosampler tab.

B: Set the Down Height to 1.

This is to ensure that the autosampler head is cleaned off.

5.) Unsnap the levers and tubes [picture]. Return sample tube to water [picture].

6.) Close workbook (File -> close). **Be careful not to close the entire program** (file->exit =bad!!!)

7.) Record work in logbook. (should be next to computer). List notable chemicals you worked with (elements you were studying and any potentially dangerous reagents, like HF) as well as any problems you encountered.

8.) Log out at ASL computer