

- a. Select Temperature. Check some parameters.
 CID temperature – ~-41.9 deg
 FPA temperature – ~28.3 deg
- b. Close the Plasma Control menu.

II. SAMPLES:

1. All samples should be prepared in 2% - 5% acid, e.g. HNO₃ with a total volume of ≥4 ml per sample. We suggest low density polyethylene tubes (LDPE). The Fisher Scientific part no. is 6250-9028. In January, 2009, the UCLA price for one package of 12 was \$8.13 and a case of twelve packages of 12 was \$97.58.

2. Place samples in autosampler rack:

6	7	8	9	waste	10	11	12	13	14	15	16	17	18	19		
5	1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8
	4	9	10	11	1	13	14	15	16	9	10	11	3	13	14	15
3		17	18	19	20	21	22	23	24	17	18	19	20	21	22	23
	2	1	2	3	4	5	6	7	8	1	2	3	4	5	6	7
1		9	10	11	2	13	14	15	16	9	10	11	4	13	14	15
		17	18	19	20	21	22	23	24	17	18	19	20	21	22	23

- a. For unknown samples, there are four racks with 24 positions each. For example, this is position **18** in rack **1**.
- b. The “L” rack to the left and top are to be used for blanks, calibration samples, and quality control samples.


IV. METHOD:


1. On the menu bar, select **Method**.
 - a. If you have an existing method, select Open.
 - b. Otherwise, select New.
 1. This will bring up a period table. If you single click on an element, all possible emission lines will be displayed on the right. Double click on an element to select it for your method and all the emission lines will be selected.
 2. Single click on an emission line and a popup window will display the relative intensity of the line along with all lines of other elements that may interfere a line. Use this information to decide which lines you want to observe.
 3. Double click any line to deselect it (or to re-select it). When done, select OK.
 4. Select Method, then “Save As” to save the new Method, taking care to NEVER store anything in anyone else’s method.

2. On the menu bar, select **Setup**. This will show a pulldown window of several items. Go through each. Saving the method often throughout this process is never a bad idea. CTRL S can be used as a shortcut to Save.
 - a. Analysis Preferences
 1. # of repeats = 3 (This will use 3.7 ml of sample.)
 2. Delay time = 0
 3. Sample flush time = 30 sec
 4. CID maximum integration time:
 - Low WL range = 25 sec
 - High WL range = 10 sec
 5. Select Auto-inc samplenames, if desired.
 - b. Source
 1. Check ICAP
 2. Do NOT check Hg lamp
 - c. Automated Output – (This section can be overridden in the protocol; see section 13.b. below.)
 1. The top section allows you to choose Store, Print, Virtual, and Export for Unknowns, QCs, Blanks, Standards, and Recoveries. Usually Virtual is No. For items you include in your analysis, the rest should be Yes. Anything you check as Yes needs to be set up in the Report preferences section described below. Right click on an item to toggle between Yes and No. For anything that is no, you do not need to be concerned with the corresponding Report preferences.
 2. Under Store to Database
 - a) Be sure to CHANGE THE FILENAME on the top line to avoid overwriting data!!
 - b) Be sure to use at least one letter or you will not be able to find the file later.
 - c) Check “Include repetitions”.
 - d) Check “Include imagedata”.
 3. Under Exported Data Formats, Comma-delimited Ascii should be checked. Also, right click under filename and enter a unique name (e.g. JS080808.TXT).
 4. Under Auto-Print Standardization Report
 - a) Check Slopes
 - b) Check Readbacks
 - c) Check Plots
 - d. Report preferences
 1. Unknowns
 - a) Under Output mode, select “Concentration”.
 - b) Under Options, check “Significant Figures?”
 - 1) Other options may be considered, e.g. “Blank Subtract”, “Line Switch”, and “Perform Limit Check”.
 - c) Under Correction Factor, set to 1.
 - d) Under Report type, select “Line”.
 - e) Under Report to, check “Screen”
 - f) Under Report on, check “Averages”, “Stats”, “Repeats”, “Errors”, “Wavelength”, “Int.Std. ref”, and “Units”.
 2. QC standards
 - a) Set these all the same as what you have for Unknowns.
 - b) “Perform Limit Checks” will be selected and you cannot change it. It would make sense to also select “Checks” in the section called Report on.
 - c) QC standards are probably other samples of known concentration.
 3. Blanks – Set these all the same as what you have for Unknowns.

4. Standards – Set these the same except select “Int. Ratio” which will gray out any options.
5. Recoveries
 - a) Set these all the same as what you have for Unknowns.
 - b) “Perform Limit Checks” will be selected and you cannot change it. It would make sense to also select “Checks” in the section called Report on.
 - c) Recoveries are probably the standards used for the calibration.
- e. Element Preferences
 1. For each element and each wavelength, select Report. (These are usually already right.)
 - a) For element name, this should automatically include the element and wavelength.
 - b) Specify the units to be used, e.g. ppm.
 - c) Set Sig. Figs. to 4.
 2. For each element and wavelength, select Standards.
 - 1) Input a name and concentration. Right click on the left half to change the name. Right click on the right half to change the concentration.
 - 2) Once this is set up for a particular wavelength, select Copy All.
 - 3) Select the next wavelength and select Paste All. Do this for all wavelengths.
 - 4) Select OK.
- f. Standards (If the previous step has been done correctly, this part should already be right.)
 1. Under Standards, give a name that includes the concentration.
 2. For each Standard listed, under Element, select the element to display the Lines. Double click to check (or uncheck) the element and under Conc., right click to edit the concentration.
 3. Under Lines, for each element, double click to check (or uncheck) the particular line.
 4. Select OK.
- g. Plasma control
 1. Under Nebulizer Pump
 - Flush Pump rate = 130 rpm
 - Analysis Pump rate = 75 rpm
 - Relaxation Time = 0 sec
 - Pump Tubing Type = Tygon-Orange
 2. Nebulizer Pressure = 32.06 psi
 3. Auxiliary gas – Check “Low”.
 4. RF Power – Check value appropriate to element, e.g. 1150 watts
- h. You MUST save the Method after having changed anything in Setup. CTRL S can be used as a shortcut to Save.

V. SETTING UP A RUN:

1. On the menu bar, select **Run**. This will show a pulldown window of several items. Select “Automated Analysis”. Then go through the following.
 - a. Select “Samples”.
 1. Select the icon to add samples. 
 2. Input all required information for a sample.
 3. Select Save. This will increment the sample name and allow you to input information for the next sample, etc.
 4. When all samples have been input, select Done.
 5. Select “Samples” on the menu bar, then Save As to create a new sample file or Save to overwrite your existing file.
 - b. Select “Advanced”. Under Protocol on the tool bar, either open an existing protocol or select New.

1. Under Options on the toolbar, if “Override method” is checked, set up the Automated output options as described in 12.c. above. The problem is that if you select “Override method” to look at the Automated options this way, it will end up being checked. If you even look at this, make sure that you select Options again, just to see if “Override method” is checked.
2. There are three parts to a protocol: Initial checks, Analysis, and Final checks. The first three icons on the left of the row below the tool bar will display these three parts, respectively.
 - a) Initialization: – You can choose items from the Actions menu on the toolbar. Reasonable choices are: Run a blank, Standardization, etc. depending on what you have in your method.
 - b) Analysis – The most likely choice here is Run unknowns. Select the Run unknowns square and right click, then select modify. Enter the correct number of samples to be run. (Be sure to NOT include your calibration standards in this number.) You could also select run QC’s, etc.
 - c) Final Checks – Either leave this as is, or choose Alarm.
3. Be sure to Save the protocol, again taking care to NEVER store anything in anyone else’s protocol.
- c. Either select a table from the list, then Edit, or select New.
 1. Select Edit Set. This is an icon near the middle that looks like this: 
 2. Find your saved Method in the pulldown menu. Find your file of samples in the pulldown menu. Find the protocol you have stored in the pulldown menu.
 3. Check Auto-Rinse. Set the Rinse time to 60.0 sec.
 4. Select OK.
 5. Select “Table” on the menu bar, then Save As to create a new sample file or Save to overwrite your existing table.

VI. RUNNING SAMPLES:

1. All of the above steps under **Method**, **Setup**, and **Run** can be completed prior to setting up samples in the autosampler, if so desired. If the tubes have been capped, remove all caps now!
2. Put the sipper back onto the autosampler. The depth should be such that the black mark on the sipper tube is just at the top of the hole it goes through. Make sure the tubing is securely in the clamp so it is out of the way when moving around.
3. Under **Run**, “Automated Analysis”:
 - a. Select Run.
 - b. To start the full analysis, select Run.

VII. WHEN DONE:

1. Select the Plasma Control button (looks like a flame). In the “Plasma Control” panel:
 - a. Make sure the Controller Status and Plasma Status are blue, then select SHUTDOWN.
 - b. Wait for the Controller Status and Plasma Status messages to turn blue again.
2. Close the ThermoSpec software.
3. Raise the sipper such that it is just inside the waste tube and retighten the screw to hold it there. Do not forget this step because leaving the sipper in the waste tube will cause liquid to collect in the nebulizer!
4. Open the tubing holders on the peristaltic pump.
5. Close the Ar valve at the back of the instrument and at the tank.

Additional Notes:

1. If you get an error message when opening the software, you may need to do a reset.
 - a. On the menu bar, select Instrument, then Reset controller...
 - b. A Soft Reset may be performed anytime before starting your analysis while the plasma is on.
 - c. A Hard Reset must be done before starting analysis and with the plasma turned off.

2. RF Power:
1750 watts for elements that are hard to ionize
950 watts for Ca
1350 watts will do Fe, but will over-ionize Ca

3. IEC means “Interfering Element Correction”.