Startup Procedure

•Log into FOM

•Before lighting the plasma, check...
1. Argon pressure (in hallway) ~90psi
2. Water circulator (in hallway) ~20°C
3. Exhaust (inside instrument)
4. Sample introduction- Clamp down pump tubing
    Empty waste
    Fill uptake container with *fresh* di-H_2O
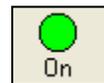   Fill Autosampler rinse bottle with 3% HNO_3

•Starting ICP-MS:
1. Turn on Autosampler (on lab cart) *Check rinse container: 2-3% acid
2. Open MS- Thermo Plasmalab Service (on taskbar near clock)
3. Start PlasmaLab (on desktop)

   •Go to 'Instrument' tab on left toolbar
   •Look for 'Vacuum ready'
   •Click the 'On' button (change to operate state) & wait
      until you see 'Operate'
   •Check task bar readings:

	Definition	Value	If	Then
Neb	nebulizer back pressure	~2bar	low	leak or broken neb
			high	clog
Fwd	Forward power- minor pg	1412W		Set based on how good rf generator is
Ref	Reflected power from plasma	< 5 W	high	automatically adjust load & tune
Load	rf power	not zero or 255		
Tune	rf power	not zero or 255		
Exp	Expansion pressure	2mbar	>2.5	hole in the cone is too big or sample cone is in wrong Or vac pump
Ana	Analyte pressure	2.9x10-7		
Speed	Turbo pump speed	1000Hz		
Load	What pump uses	0.8-1.2A	>3	vac leak

• Let the instrument settle for 10mins before tuning
• (Turn on CCT gas during these 10mins if you are using that mode
    -it's the 1st CCT-He slider bar in 'Add. Gases'
• (Turn off the CCT gas before tuning)

Autosampler Controls

•Make sure Autosampler is checked in Configurations (Cetac 260)

	Use	Model	Device Type	Description
Available Accessories and Devices:				
	☑	Cetac 260	Autosampler	serial port 9, baud rate 9600
	☐	Cetac 500	Autosampler	serial port 1, baud rate 9600
	☐	MicroProbe II	Laser	UP213 Local
	☐	Advantech PCI-1620	Trigger	Using un-filtered inputs
	☐	Cetac 500	Autosampler	serial port 1, baud rate 19200
	☑	Peri Pump	Peri-pump	operate at 34%

•Choose Accessories window button 🔲 (under Tune or Configurations)
•Choose 'Autosampler' tab
•Initialise the accessory unit 🔲

Go to Rinse station   Go to Sample   Start/Stop Rinse station pump

Choose Sample position

Accessory setup and control
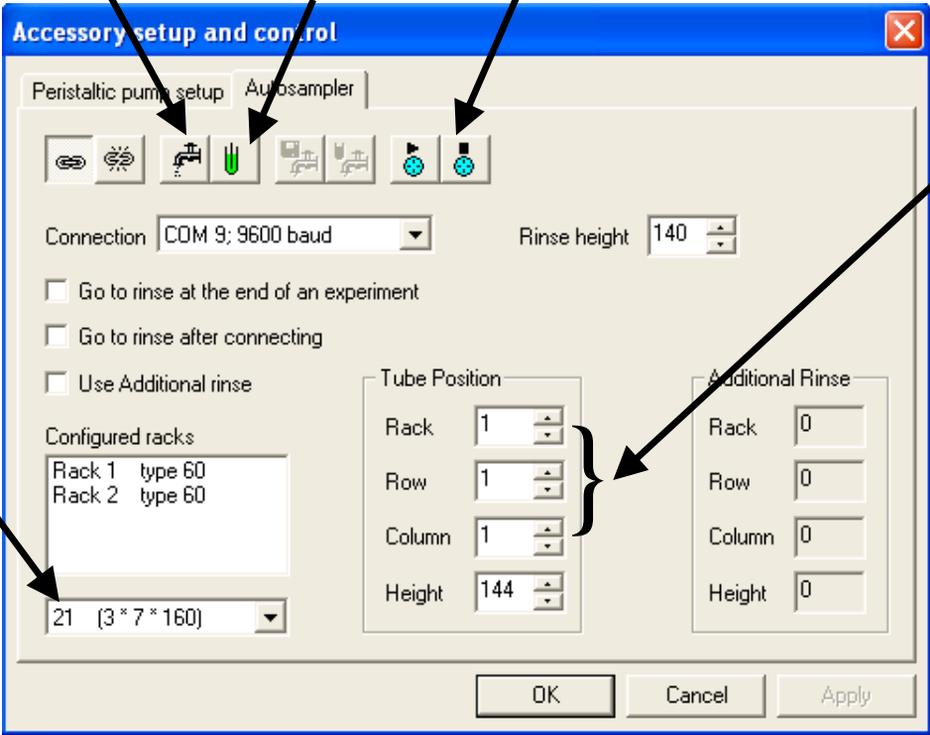
Peristaltic pump setup | Autosampler

Connection COM 9; 9600 baud ▼   Rinse height 140

☐ Go to rinse at the end of an experiment

☐ Go to rinse after connecting

Choose Rack Type

☐ Use Additional rinse

Configured racks

Rack 1   type 60
Rack 2   type 60

21   (3 * 7 * 160) ▼

Tube Position
Rack   1
Row   1
Column   1
Height   144

Additional Rinse
Rack   0
Row   0
Column   0
Height   0

OK   Cancel   Apply

•Click OK when finished (do not un-initialise 🔲)

Peri-pump

• Normal pump speed is 25 rpm

<u>Tuning- All Users</u>

Standard Mode

• Wait about 10mins while the ICP warms up (*fresh* di-H2O uptake)
• Go to Configuration tab-
> Check <u>X Series Default</u> in 'Configurations'
> Check <u>Autosampler: Cetac 260</u>
>> and <u>Peri-Pump</u> in 'Accessories'
> Pick latest 'Instrument Settings' (top of the list)

• If the instrument was not in 'X Series Default' mode (ie. left in CCT)
> Go to Tune tab and click Reset button ⟲
> **Again, this is ONLY IF it was not in 'X Series Default'**

1a. <u>Autotuning Lens</u>: (everyday)

• Use Standard Tuning solution (Multi-Element Tune A)
• Click <u>Start the Autotune Wizard</u> button 🎵 (under Tune tab)
> Choose:
>> Sequence 'XSII Xt- without Co' (check desktop)
>> Introduce sample 'manually'
> It takes approximately 10 mins

⭐ • 'View report' to see if Autotune passed & click 'Finish' ⭐
• If it fails, find me.

Replace uptake tubing into di-H$_2$0 while setting up experiments

• Now is a good time to rinse out the autosampler lines. ⊞
• Refer to the information in the previous section
• Go to Rinse station, and watch your element of interest

Experienced users only:
Standard Mode- Performance & Calibration

2b. <u>Performance Report:</u> (everyday, if you manually checked performance)

•Use Standard Tuning solution (Tune A)

•Click <u>Start the Performance Report Wizard</u> button 📋 (under Tune)

       Choose: 'XSII Xt'

       Introduce sample 'manually'

       It takes approximately 5 mins

⭐•'View report' & scroll all the way to the bottom to see if Performance ⭐

       Report passed & click 'Finish'

•If it Fails because: sensitivity or interference problem, Autotune

           Mass Calibration fails, Find me

3. <u>Detector Cross Calibration:</u> (do once a month)

•Use Multi-Element Tune F solution

       Solution contains a high concentration of low mass elements
and low concentration of high mass elements to produce the
same number of counts on the detector.

•Click <u>Launch the Instrument Calibration Wizard</u> button 🔧 (under Tune)

       Choose: 'Detector cross calibration'

       Introduce sample 'manually'

       It takes approximately 5 mins

•Click 'Finish'

> Tune F stock soln ≈ 50ppm
> Dilute 1/1000
> TuneF working soln ≈ 50ppb

4. <u>Mass Calibrate the Quadripole:</u> (do once a month)

•Use Standard Tuning solution (Tune A)   takes about 3mins

•Click arrow next to <u>Instrument Calibration</u> button 🔧 ▾ (under Tune)

•Choose Mass Calibrate the Quadripole

       Choose Introduce sample 'manually'

•'View report' & click 'Finish'

•Go into file 'Calibrations>Mass Calibration' – Highlight <u>Standard</u>

       <u>Click off outliers</u>:

       Peak Width- 0.75 +/- 0.1AMU (0.65-0.85AMU)

       Error- Above 0.1 click off (right axis)

       Right click on graph to look at one or the other

Experienced users only:
Standard Mode Tune

1b. <u>Manually Check Performance</u>: (everyday, if you are experienced)

- Wait about 15mins while the ICP warms up (di-H2O uptake)
- Go to Configuration tab-
  - Check <u>X Series Default</u> in 'Configurations'
  - Check <u>Autosampler</u> and <u>Peri-Pump</u> in 'Accessories'
  - Pick latest 'Instrument Settings' (top of the list)
- Go to Tune tab and click Reset button ⟲ (if above was not checked)
- Put uptake line into Standard 10ppb Tuning solution (Multi-Element Tune A) & restart Real Time Display ▶
- Look for Interference's (CeO & Ba++) Ratio<3%
  - Stability <10%
  - Sensitivity:

Analyte	Instrument Specification	Standard
7Li	>60,000	140,000
115In	>400,000	1,000,000
238U	>800,000	1,700,000
CeO/Ce	<0.02	0.015
Ba++/Ba+	<0.03	0.02

Co spec
150,000

- **If:** Sensit. is low/ Interfer. are high   **Then:** run autotune
  - Stability is high   (>10)   change uptake tubing
  - CeO/Ce is high   lower Nebulizer manually
  - Ba++ is high   raise Nebulizer manually
  - Li sensitivity is low   raise Focus manually
  - U sensitivity is low   lower Focus manually
- Save the settings after manually adjusting lenses 💾
- Run the Performance Report

Lens voltages:

Extraction	-100 to -300
Lens 1	-800 to -1400
Lens 2	-50 to -130
Focus	5 to 15
D1	-20 to -60
D2	-120 to -180
Pole Bias	0 to 0.5
Hexapole Bias	-4 to -3
Neb	0.84 mL/min
Lens 3	-180 to -200
DA	-30

Replace uptake tubing
into di-H_2O while setting
up experiments

CCT Users Only:
CCT Mode Tune

1. Run CCT1 Gas for 5-10 mins during instrument warm-up

2. Tune 1st in Standard Mode

3a. Tune CCT Mode (option 1):

   • Use Standard Tuning solution (Tune A) without As
   • Go to Configurations tab and check 'CCT 1 Default'
         Don't click Re-use to keep Std tunes settings ( )
   • Click Autotune button   (under Tune)

   Choose:
         'Quick CCT Tune'
         Introduce sample 'manually'
      It takes approximately 5 mins
   • 'View report.' If Report passed, click 'Finish'
   • If it doesn't pass, usually you can purge the CCT gas longer &
     it will pass. Find me if it doesn't.

CCT specs:

Analyte	Instrument Spec
78Se	< 20 cps
115In	> 100,000 cps

Lens voltages:

	Extraction	-100 to -300
	Lens 1	-800 to -1400
	Lens 2	-50 to -130
	Focus	0 to -5
	D1	-20 to -60
	D2	-120 to -180
*	Pole Bias	-14 (-9)
*	Hexapole Bias	-17 (-6)
	Neb	0.84 mL/min
	Lens 3	-180 to -200
*	DA	-20
*	CCT -He/H	2.8 to 4.0

*different from Std Mode

Replace uptake tubing into di-H$_2$0 while setting up experiments

Setting up an Experiment

•<u>When to use Std Mode:</u>
- •Use this setting for most analytes
- •Always use for elements below mass 23 (CCT is not good)
- •Watch out for:
  - -Analytes with IE (ionization energy) greater then 9 may be a problem. You may need a longer dwell time for better results. Default is 10ms so try 50ms.
  - -Analytes with low second IE, like Column I & II elements, may produce doubly charged ions (use cool plasma)
  - -In a high sodium solution, NaAr interferes with Cu-63. Use Cu-65.

•<u>When to use CCT Mode:</u>
- •To analyze elements that have polyatomic interferences.
- •Common elements: V, Cr, Fe, As, Se
  - 51V: In a solution of HCl, ClO interferes.
  - 52Cr: Watch out for ArC
  - 53Cr: In a solution of HCl, ClO interferes.
  - 56Fe: In a high calcium solution, CaO and ArO interferes.
  - 57Fe: In a high calcium solution, CaOH interferes.
  - 75As: In solution of HCl, ArCl interferes.
  - 77/80/82Se: Watch out for ArCl, Ar2, ArHArH
- •Other possible elements: Mg, Si, P, S, K, Ca, Ni, Co, Cu
  - 24Mg: High carbon soln, watch out for C2
  - 28Si: Watch out for N2
  - 31P: Watch out for NOH
  - 34S: Watch for O2, detection limit is still 60ppb at best.
  - 39/40K: Watch out for ArH, Ar
  - 40Ca: Watch out for Ar
  - 58/60/62Ni: High calcium soln, watch for CaO, CaOH
  - 59Co: High calcium soln, watch out for CaO, CaOH
  - 63Cu: High sodium soln, watch out for ArNa
  - 66Zn: Watch out for ArMg

Setting up an Experiment, cont.

•Check pump speed: should be 25 rpm (Inst. tab, Accessories window 🔲)

•<u>Starting an Experiment from an Existing Experiment</u>

   •Go to 'Experiment' tab on the left toolbar
   •<u>Create an experiment from an existing experiment</u>
   •Locate the file that you want to use as a template
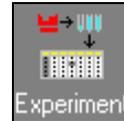
•<u>Timings Pop-up</u>

   • When it asks 'Do you want to change the timings?' Say 'No'
   • This is the Uptake/Washout delay (25 sec under maximum delay)

•<u>Skip directly to the Sample List</u>: (check the other tabs for accuracy)
   •<u>Sample List</u>:
       •Rack: 0- Standard Rack (All row 1)
                1- Farthest Sample Rack
                2- Closest Sample Rack
       •Row/Column: Each rack is labeled, columns go <u>right</u> to <u>left</u>
       •Show advanced tab: Remove Int. Stds. here if they look wrong in results.
                Make a new Int. Std list. Choose this new list for each
                sample that has a problem in the list.

⭐•<u>Fully Quantitative Concentrations</u>:   ⭐
       •Fill in the your standard concentrations for each analyte

•**<u>Connect the Uptake line to the Autosampler:</u>**
                Go to the rinse station for a couple mins,
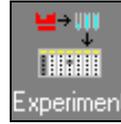   if you have not already done so.

To start your run: press the Queue button,
save your file name, and press Append.

Setting up an Experiment, cont.

• Check pump speed: should be 25 rpm (Inst. tab, Accessories window 🔲)

• <u>Starting a new ICP experiment:</u>
  • Go to 'Experiment' tab on the left toolbar
  • Create a <u>new blank experiment</u> (usually choose 'Continuous')
    - Continuous (ICP only)
    - TRA- time resolved analysis (profile for LA & HPLC)
  • Select a <u>database</u> (usually choose 'default'):
    - default (standard ICP settings)
    - default cct (Collision Cell ICP settings)
    - multiple mode database 2005 (both std & cct settings)
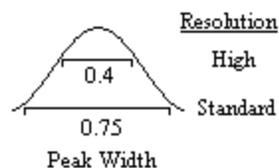• <u>Setup:</u>
  • <u>Configuration Editor:</u> should already be highlighted correctly & unchecked
    - X Series Default (std mode) or CCT 1 Default (CCT mode)
    - Autosampler & Peri-pump in Instrument tab
    - Latest Instrument settings are highlighted
  • <u>Timings:</u> default is 25 sec delay- under 'maximum delay'
    *Au, Ag, and Pt need 60 sec washout. You should have HCl too.
    (Check Uptake/Washout if you want to monitor an element.
    Make sure to include a minimum delay if you check this.)
  • <u>Analyte:</u> -Double click both your <u>analytes</u> and <u>internal standards</u>
    -Single click on an analyte to choose multiple isotopes
    -Elements that have known interferences will automatically turn
    them on. You will see a blue dot for those elements.
    -Pb: Percentages vary so edit equation to add 1x each isotope
  • <u>Acquisition Parameters:</u>
    -Survey: 10 sweeps, 0.6ms dwell, 10 channels, std resolution
    -Main-Peak jump: 100 sweeps, 10ms dwell, 1 channel, std res.
    -To increase resolution on main runs, you could:
      Lower sweeps to 50 & raise dwell to 50ms
    -For high Na samples, use 5ms dwell and high resolution.
    -To reduce interferences in your analyte peak, you could:
      Change resolution to 'High' to change peak width

Setting up an Experiment, cont.

•<u>Setup, cont</u>:
  •<u>Internal Standards</u>: One by one, choose your Internal Standards from the
        left list and move to the right list [≫]
        -Good internal standards are made to produce 100,000 to
        300,000 counts. You may need to increase the concentration of
        the lower mass elements in the internal standard set to achieve
        this. Typically 2- **5ppb** are used.
        -Technique: <u>Interpolate</u>- applies mass curve using all the internal
                standards and corrects analyte based on curve. If
                one int. std starts to drop too low and therefore
                drift (Ex- low mass elements at the wrong
                concentration), you can take that std out per
                sample in the Sample List.
              <u>115In</u>- corrects analyte based on In internal std only
                This std should be <u>within 60 mass units of analyte</u>.
                CCT mode: Int std should have similar IE.
    •<u>Isotope Ratio</u>: not usually necessary

•<u>Instrument Calibrations</u>:
    •Current Mass Calibration & Detector Cross Calibration are automatically
    used so you do not need to do anything on this page.

•<u>Calibration Method</u>:
    •Method:☆<u>None</u>- Internal Standards
            <u>Semi-quant</u>- if you want to analyze an element and don't have a
                    standard for it (maybe use in survey run)
                    Accurate within 20%
            <u>Addition</u>- Standard addition
        ☆<u>Fully-quant</u>- Normal calibration method for your analytes.
                    -When semi-quant is checked on the right, this analyte
                    will be used to generate the semi-quant curve.
                    -Uncheck Semi-quant for Pb if you use an equation to
                    calculate results (see setup>analyte).

- QC Setup:
  - This section is only used in environmental labs.

- Sample List:
  - Sample List:
    - Type: Blank- di-H_2O + acid + Internal Standards
      Fully Quant Standard- Multiple ppb standards + acid +
        Internal Standards
      Unknown- Your samples + acid + I.S.
    - Rack: 0- Standard Rack (All row 1)
      1- Farthest Sample Rack
      2- Closest Sample Rack
    - Row/Column: Each rack is labeled, columns go right to left
    - Show advanced tab: Remove Int. Stds. here if they look wrong in results.
      Make a new Int. Std list. Choose this new list for each
      sample that has a problem in the list.

- Fully Quantitative Concentrations:
  - Fill in the your standard concentrations for each analyte

- Connect the Uptake line to the Autosampler:
  Go to the rinse station for a couple mins,
  if you have not already done so.

To start your run: press the Queue button, save your file name, and press Append.

Monitoring your Run

Results tab:

•Calibration Data: Check your calibration curves for contaminations
Click on a point in the graph to remove a standard
from the curve
Intercept Conc- Detection Limit

•Numerical Results:
-Analyte Dilution Conc- Corrected for internal standards and
dilution (specified in sample list)
Watch Internal Standards through out the run (≈ 0.7-2.0)
If Internal Standard Correction goes above 2.0, the cones are
probably getting clogged & results may be off.
-Mass Uncorrected ICPS- Watch Internal Standard Raw counts
(115In $\approx 300,000$ for 5ppb)
Record Raw Indium counts at the beginning and end of run in
the Log Book

•Spectra: -Check to make sure impurity peaks are not overlapping with
your analyte peaks (see Setup>Acq. Parameters>Resolution)
-When you see a peak half way between mass units, you
probably have doubly charged ions from an element at 2x that
mass number.
-The dotted line represents the analog signal and the solid line
is the digital. They should match if the detector is calibrated
correctly.

Reports

Two options:

•Generate a Report using the PlasmaLab software
Go to Report tab
Choose: Fully Quant Calibrations
Dilution Corrected Concs, etc.
Save or Print
• Generate an Excel file:
Right click on your Numerical Results
Export as csv or copy

<u>Shutdown</u>

- **<u>Rinse:</u>**
  - Rinse in Autosampler rinse station
  - Go to Instrument>Tune>Accessories window button 　⊞

    Go to Peri-pump tab>Initialise 　⊗

    Click on Rabbit speed button 🐰 -do not hit OK when in rabbit

    Run for a couple mins, then click play ▶ (25 rpm) & OK
  - Disconnect tubing to Autosampler & put uptake tubing into di-H_2O

  > - If using CCT mode, go to 'Configurations' & check 'X Series
  >   Default.' Then go to 'tune' and click ↺ to reset settings

STOP HERE if someone is on after you

- **<u>Turn Off:</u>**
  - Take tube out of di-H2O and run for a couple mins

    It is recommended to turn off the instrument dry.
  - Turn the instrument to 'Vacuum state' ⊗ Off

  - DO NOT switch the instrument to 'Shutdown State'; this
    happens if you press the 'Off' button twice.

- **<u>Before you leave:</u>**
  - ⭐ Release the tension on the peri-pump tubing & unhook the tubes
    from the tabs on the pump.
  - Turn off Autosampler
  - Empty waste container
  - (closing Plasmalab will not shut off the ICP)
  - Clean up area of any sample tubes, paper, etc.
  - ⭐ Turn off the Argon gas or liquid tank
  - ⭐ Log off FOM

Call me if you have any problems:
Becky: 7-7282 lab
Thank you!!