

ICPMS Standard Operating Procedures-Laser

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Introduction

This manual is made to be a comprehensive refresher for people who have previously been trained to use the instrument. It is divided into individual sections which are often interrelated and may not be next to each other. Refer to the concise operating procedures for either liquids or laser ablation. In the concise procedures, references will be given to in-depth instructions located in the document in case you need more information.

In case there are issues, see [Section 14. Troubleshooting and Error Help](#). Make sure you contact the lab manager with any issues or trouble you are having.

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See the sign outside the office
door for Kyle's current location.
ES 130 (down the hall from the lab)
Juliet (ES 131) can always
contact me if I am in the office.

The local login for the computer is
required due to proper instrument
file permissions. It is as follows:
Login: .IAMSEC
Password: AMSEC

Instrument Hazards and Best Practices: ICP-MS

This document will cover the inherent hazards when utilizing this piece of equipment as well as the best practices and procedures to avoid danger. These hazards will not include basic things that may be included in the basic safety training document that each user has attested to have reviewed at fom.wvu.edu/documents.

Lab coats are to be provided by the user unless special hazards exist in which case, they are located at the PPE station.

Hazards:

- Chemical exposure
- Compressed gases
- Ultraviolet light exposure
- Ultraviolet laser radiation

1. Required PPE

Appropriate laboratory attire is required at all times in the AMSEC laboratories. Whenever chemicals are being used, an additional requirement of a lab coat is required. Lab coats are to be provided by the user.

Whenever a user is in the AMSEC labs, the minimum requirement for eye protection is wrap around impact glasses. Anytime liquid chemicals are present in the same room as the user without a direct barrier, all users in the lab must wear chemical splash goggles. Splash goggles must be approved by State of Washington Administrative Code (WAC 296-24-078).

If chemicals being used are considered toxic, caustic, corrosive, flammable solvents, carcinogenic, mutagenic, or teratogenic, a minimum of disposable nitrile gloves is required. Avoid chemical transfer by taking off gloves when using anything other than the chemical(s).

2. Compressed gases

All users must have completed the compressed gas and cryogenic liquid training before accessing this equipment or touching any compressed gas cylinder or cryogenic liquid tank. All prudent practices dictated in that training as well as associated training documents must be strictly adhered to at all times.

3. Ultraviolet light exposure

The argon plasma associated with this instrument emits harmful levels and wavelengths of ultraviolet radiation that is harmful to the eyes. Every effort is taken to reduce the risk of ultraviolet light exposure; however, it is impossible to eliminate all possibilities, especially when an accessory item such as the laser ablation is being used which isn't designed with that in mind.

It is recommended to avoid eye contact with the plasma at all times unless using the properly shielded window located on the front of the instrument. Placing the protective orange plastic over the hole where the sample is introduced is also recommended to reduce the possibility of making eye contact.

4. Ultraviolet laser radiation

The ICP-MS is equipped with a laser ablation accessory system. It utilizes a high powered, 213 nm laser to ablate samples. This laser system is designed with many interlocks and safety devices in place to prevent users from ever being exposed to the laser. Under normal operation, the chances of being exposed to the laser are almost zero. If any interlocks are not satisfied and cannot be remedied by the user, the lab manager must be notified, and the user should cease use of the system immediately.

At no time should anyone tamper with or attempt to dismantle or repair any part of the instrument. Failure to follow these instructions could lead to serious injury or death.

1. Concise Operating Procedures

The concise operating procedures outline the necessary steps and reference relevant sections for more detailed instructions.

1. Check the hardware setup and change to laser ablation if necessary. [Section 3. Hardware Setup](#)
2. Setup sample in laser and purge cell with helium flowing. [Section 5.1. Loading a Sample](#) and [Section 5.4. Gas Flow](#)
3. Turn He flow to 50 before igniting plasma. [Section 5.4. Gas Flow](#)
4. Load your tune file [Section 10.1. Selecting a Tune File](#)
5. Ignite the plasma. [Section 4. Igniting the Plasma](#)
(Check Configuration File) [Section 6.1. Before Starting the Software](#)
6. Turn gas up to running flow rate. [Section 5.4. Gas Flow](#)
7. Load your method file. Edit as required. [Section 7. Creating/Editing a Method](#)
Choose Method → Load
8. Create your batch file. [Section 9.2.1 Creating a Batch File](#)
9. Setup your scan pattern(s) [Section 5.3.2. Software Layout](#)
10. Run your scans and acquire data [Section 5.5. Analyzing a Sample](#)
11. Export your data. [Section 9.3. Exporting and Viewing Data](#)
12. Turn off the instrument. [Section 12.2. Shutting Down](#)
 - a. Shut off plasma if not already off
 - b. Shut off helium flow and remove sample
 - c. Turn off laser power supply key
 - d. Shut off the argon and water chiller
16. Take your samples and clean up.

2. ICP-MS Theory and Operation

2.1. Instrument Operation

2.1.1. Plasma

Inductively coupled plasma-mass spectrometry (ICP-MS) is a technique that utilizes an extremely hot argon plasma of ~10,000 K for the purpose of atomizing/ionizing any sample for trace elemental analysis. The sample introduction method for this system is either a liquid autosampler capable of continuously running up to 200 samples or a laser ablation system which utilizes a 213 nm high powered laser to ablate a solid sample which a helium flow then takes to the plasma. Regardless of the sample introduction method, the operation of the instrument itself follows the sample principles.

The argon plasma is capable of breaking apart any molecule into atoms while simultaneously ionizing these atoms. Ionized atoms and molecules interact with magnetic fields due to an induced dipole from the loss of an electron, but we only want to measure atoms and not molecules. Therefore, a series of electromagnetic lenses have been devised to focus the ion beam and remove any polyatomics that may have been formed by the plasma or recombined after going through the plasma.

2.1.2. Interferences

The large majority of the ICP-MS system is dedicated to removing interferences. What exactly are interferences? This term refers to anything that can interfere with the analyte mass in question. The masses are measured at the end of the instrument by an ion counter. This counter doesn't know what is hitting it, it just counts the ions that hit. The quadrupole located directly before the ion counter is what filters out every ion except the one to be measured, and it is here that the effects of interferences are seen, but you don't know it. The quadrupole filters things by their mass to charge ratio. This means that if you are analyzing an ion of iron such as $^{56}\text{Fe}^+$ then the mass to charge ratio is 56/+1. There are other things that can result in the same mass to charge ratio such as argon oxide, $^{40}\text{Ar}^{16}\text{O}^+$. This will have the same mass to charge ratio and every one that makes it to the quadrupole will be detected as if it were ^{56}Fe . This is a simple example of an interference. You may be thinking that ArO should not be a stable molecule, especially in an ionized state, and you would be right. This doesn't apply in a high vacuum where the time from formation to measurement is very small. You can easily get an interference with three atoms as well. Four atoms is very unlikely. The other option is a doubly charged ion. An example of this is easily seen when analyzing a sample for ^{28}Si that also has iron in it. The silicon will have a mass to charge ratio of 28/+1, but the iron has the possibility of being doubly ionized to $^{56}\text{Fe}^{2+}$. This would give it a mass to charge ratio of 56/+2 which is simplified to 28/+1. This means any iron that gets doubly charged will have the same mass to charge as the silicon and will be counted as such.

2.1.3. Cones

Upon going through the plasma, a portion of the sample is single, ionized atoms, a portion is ionized molecules, and a portion is neutral atoms and molecules. The first thing the sample stream comes into contact with is a set of nickel cones. The first cone is called the sample cone which is aligned such that only the hottest part of the plasma is sending sample into the lens chamber. This is beneficial since the hottest part has the highest amount of ionized atoms. The second cone known as the skimmer cone, has a smaller orifice which skims off even more of the stream that wasn't going in a straight line and reduces the sample stream to a smaller, more manageable size. Between the plasma chamber and the lens chamber, the pressure goes from atmospheric to a rough vacuum of $\sim 10^{-3}$ torr. This helps suck in the sample stream and allows for less collisions with gas molecules to increase the mean free path of the sample stream.

2.1.4. Lenses

After going through the set of cones, the sample stream is subjected to a set of lenses which focus the ion beam while allowing a large portion of neutrals to be sucked away by the vacuum. The next set of lenses deflects the ion beam by a couple millimeters. This is a key feature of the lens set since any neutral atoms/molecules that have made it this far will continue to fly straight and are ran into a plate and subsequently sucked into vacuum. The other reason for this deflection is to also remove any direct path for photons generated by the plasma to hit the electron multiplier tube at the end of the instrument as this would damage it. Up to this point so far, we have atomized and ionized a sample and refined and focused the beam into ionized atoms and ionized molecules.

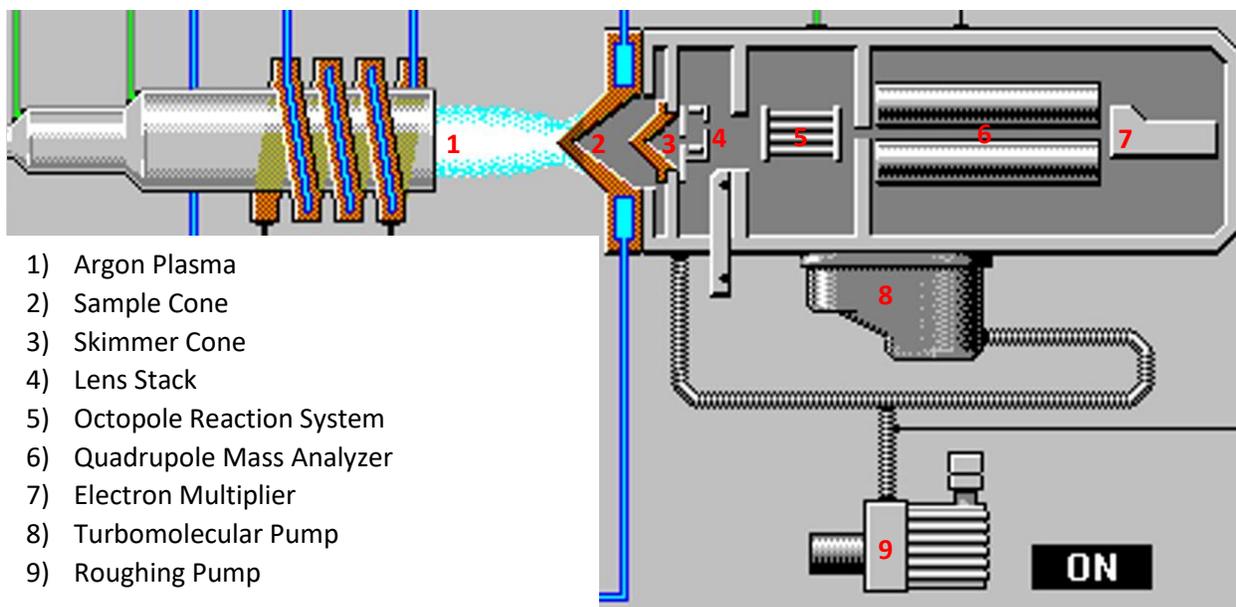
2.1.5. Octopole Reaction System

The rest of the analyzer chamber is at a vacuum of around 10^{-6} torr and the ion stream is pulled into the octopole reaction system (ORS). This is a specially designed cell with a set of eight small rods arranged in a cylinder shape. The rods have an electromagnetic field applied to keep the ion beam focused and flying straight. Just before entering the cell, the ion beam passes through an entrance pinhole which has a potential applied to it. Any ionized particle that doesn't have enough momentum cannot make it over this potential barrier is removed from the stream. Once the ion beam enters the ORS, there is one of three configurations that can be used.

The first is known as nogas mode. In this mode the ORS is under vacuum and simply focuses the beam to send it into the quadrupole mass analyzer. The second mode is helium mode. In this mode, also known as collision mode, the ORS is filled with helium gas. Statistically, larger ions, such as polyatomics, will collide with the helium much more frequently than the much smaller atoms that are passing through. This will either cause them to be ejected from the cell and sucked into a vacuum pump, or it will cause them to lose so much momentum that they will be unable to overcome the cell exit potential barrier. This mode is the most effective at removing polyatomic interferences.

The third mode is hydrogen mode, also known as reaction mode. Similar to helium mode, in hydrogen mode the cell is filled with hydrogen gas. Also similar to helium mode, collisions take place that remove polyatomics. The difference lies in the fact that the hydrogen can easily react with any ion and form a neutral molecule or a polyatomic with 1 amu added to the mass. Upon collision and reaction, the now neutral molecule will have changed course since it will not interact with the field generated by the octopole and will be sucked into a vacuum pump instead of heading into the quadrupole. Hydrogen mode and helium mode are often used for only very specific atoms which have specific interferences. Both of these modes severely lower the overall counts of everything due to collisions and reactions taking place with both atomic and polyatomic ions in the stream, but the interferences are reduced much more than the atomic ions.

The sample stream has now been refined to a very uniform ion beam of only single atoms. This beam now enters the quadrupole mass analyzer which is capable of utilizing strong electromagnetic fields to "filter" out every isotope that is not of interest. The quadrupole will scan across the entire range of masses selected by the user to give the mass spectrum of interest. The integration time as selected by the user, dictates how long signal is collected for each isotope. Once the isotope of interest is selectively chosen by the quadrupole, it then makes its way to the end of the instrument to the electron multiplier (EM). In the EM, every atom that hits the detector plate ejects some electrons, which then cascade into another plate, multiplying the signal by several orders of magnitude until they are finally read at the end as a current. This current is proportional to the number of ions detected and results in the counts that are seen on the screen.



2.2. Sample and Standard Preparation

Samples for ICP-MS must be kept as clean as possible. Things such as skin oils, dust, contamination from unclean sample holders, and contamination from user error are all things that contribute significantly to the error of the measurements obtained with this instrument. Proper analytical techniques must be applied to ensure the cleanliness and reproducibility of your measurements.

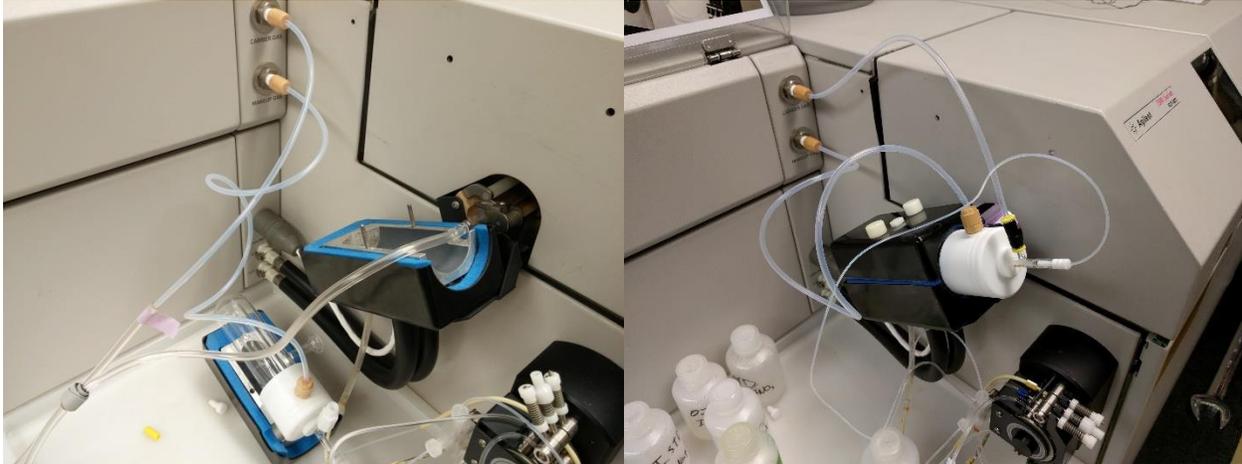
2.2.1. Laser Ablation

Samples for laser ablation commonly include thin sections of mineral deposits and other rock formations, samples deposited on a flat substrate, and any other relatively flat solid sample. Sample preparation for laser ablation is highly specialized to each user and can be discussed with the user's research advisor or the lab manager prior to using the ICP-MS. The main criteria are that the sample will fit in the laser ablation accessory sample chamber, and that the sample will have a uniform flow for the helium carrier gas. The uniform gas flow over the surface is very important to ensure reproducibility of measurements.

3. Hardware Setup

3.1. Current Setup

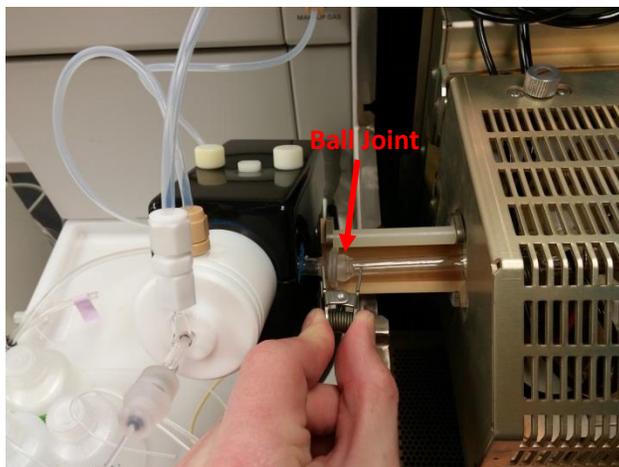
There are two possible setups for the ICP-MS. One is for laser ablation and the other is for liquids. The following two pictures display: Laser Ablation (left) and Liquids (right)



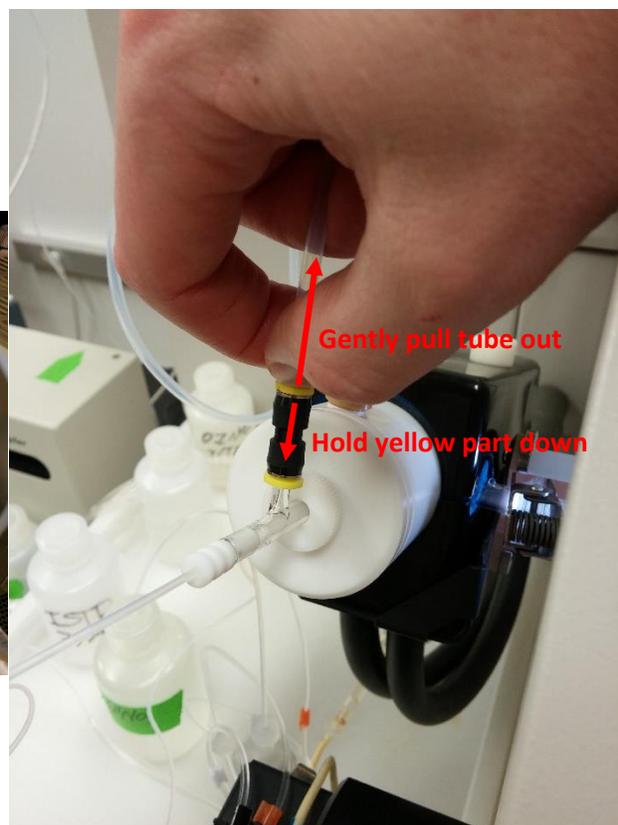
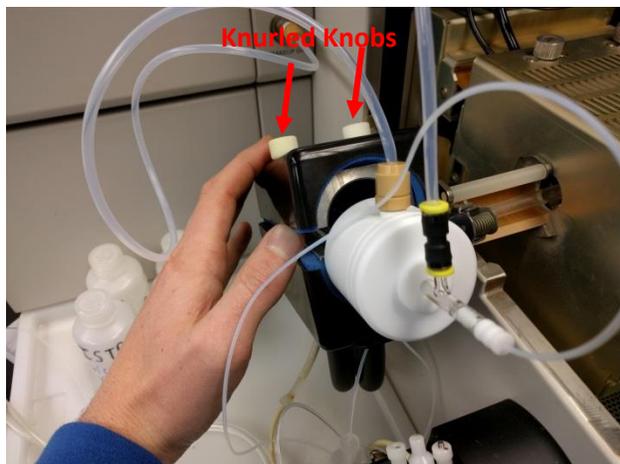
- The instructions for changing from liquids to laser ablation are on the following pages.

3.2. Liquids to Laser Ablation

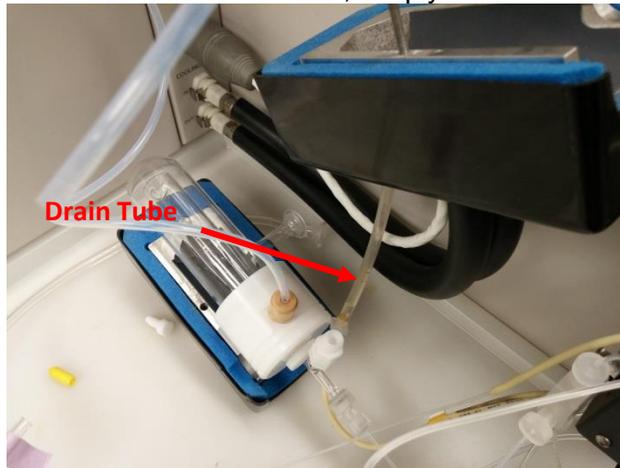
- 1) To change from the liquids setup to laser ablation you must first open the torch-box cover and disconnect the spray chamber ball joint by removing the spring clamp as seen:



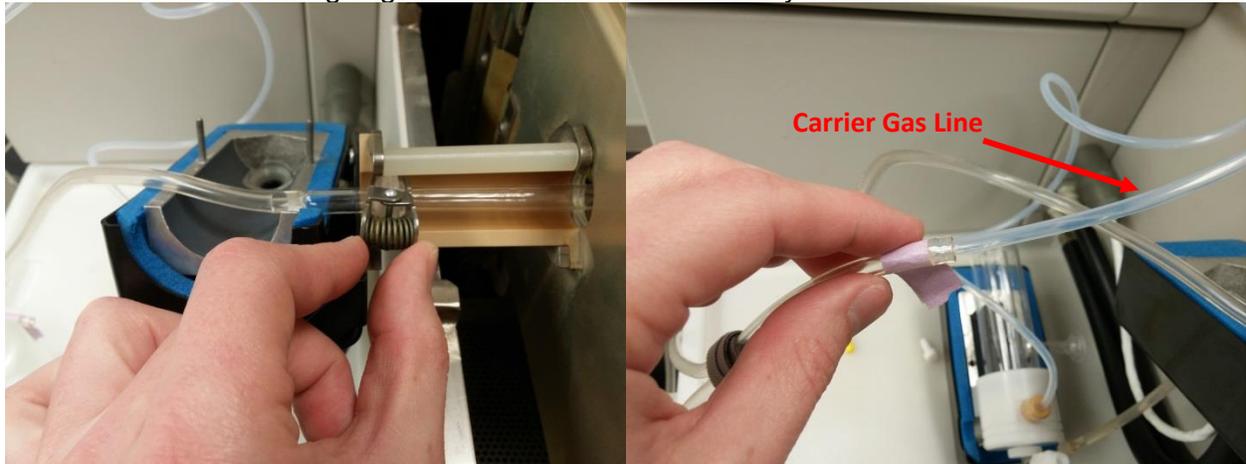
- 2) Unscrew the two white knurled nobs and remove the spray-chamber cover. Then disconnect the carrier gas line from the nebulizer (careful as the glass piece is very expensive) by pushing down on the plastic yellow ring while pulling on the tube, there should be instructions for this part posted on the instrument as well. Be very gentle as this setup costs ~\$1,000:



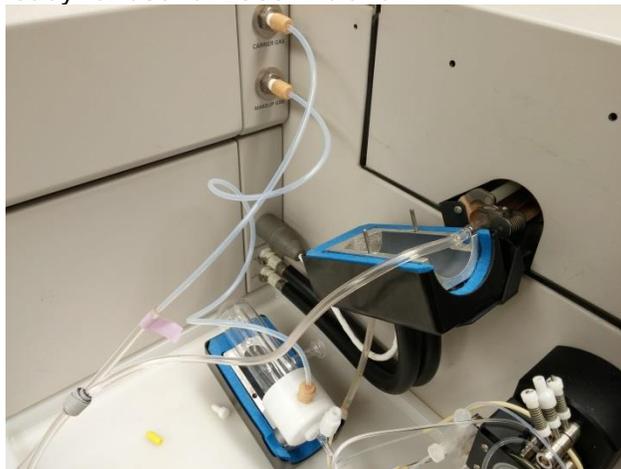
- 3) Carefully remove the spray chamber and set it down so that the tubing isn't stressed. If the drain tube comes out with the chamber, simply detach it.



- 4) Place the laser sample introduction ball joint in place and clamp with the spring clamp. Then insert the carrier gas line snugly into the other tube attached to the sample introduction tube going to the laser ablation accessory and close the torch-box cover:



- The ICP-MS is now ready for use for Laser Ablation!



4. Igniting the Plasma

4.1. Turn on the Water Chiller

- The water chiller is located in ES 128B. This is the small room just adjacent to the ICP-MS room. The chiller should turn on around room temperature and then cool to ~20°C.



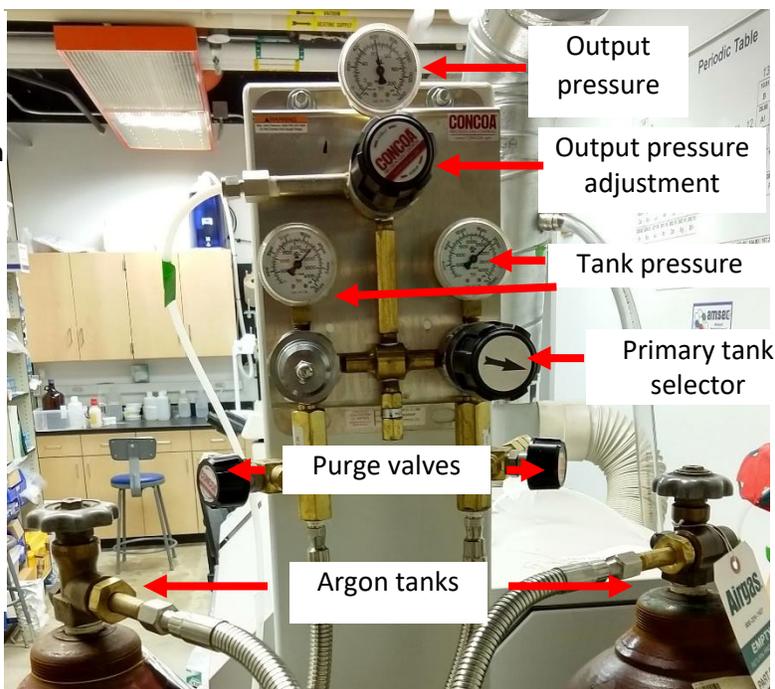
- You must turn on the water chiller before lighting the plasma. If you forget, it will not damage the instrument, but you will receive an insufficient water flow error.

4.2. Turn on the Argon

Argon Manifold Operation

When finished:

- Change tank(s), if required.
- Close both argon tank valves.



To operate:

- Open both argon tanks. Check pressure in each.
- If one tank is below 250 psi, change according to procedure below.
- Check that output pressure is between 50 and 100 psi. Adjust as necessary.

To change a tank:

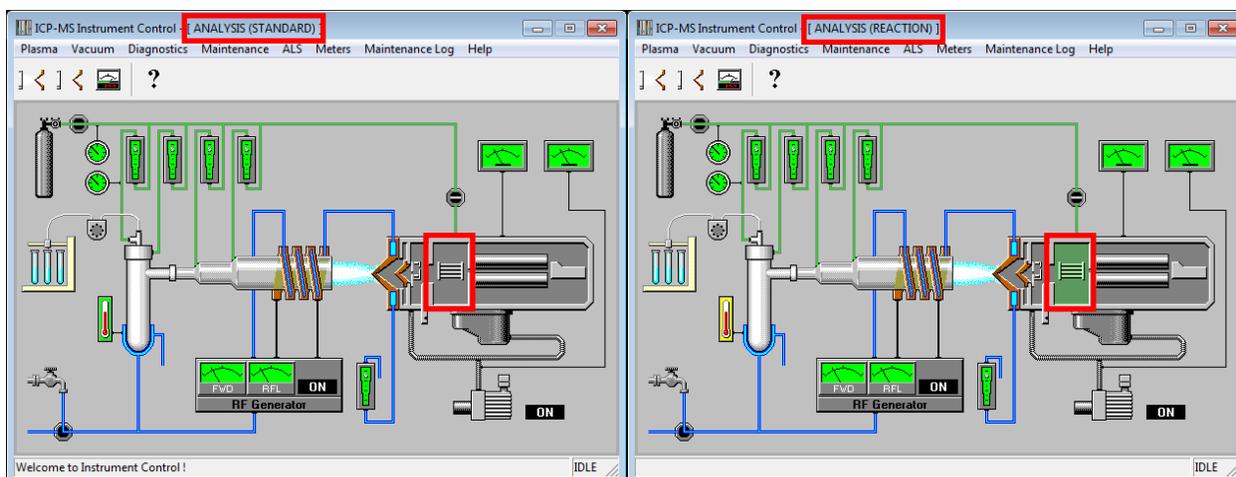
1. If a tank has dropped below 250 psi, it can be changed whether the argon is being used or not.
2. Turn arrow knob toward the tank with more argon.
3. Close tank valve of empty tank.
4. Disconnect nearly empty tank, cap it, and replace with a full one.
5. Strap full tank in, remove cap, and attach pigtail hose.
6. Open tank valve for the tank you just attached.
7. Slowly open purge valve for the tank you just attached and purge for 5 seconds. You do not have to purge at a high flow rate, just enough to get any air out of the system.

This system is designed to offer continuous, uninterrupted gas delivery. You can change a tank while the system is being used. The arrow knob indicates the primary tank. The primary tank will be used until it is around 200 psi, this will be considered empty. The system will then switch to the secondary tank.

You must check the pressure in each tank multiple times during a run. If you empty a tank, you must change it. Do not leave it for the next person to change.

4.3. Ignite the Plasma

- After the water chiller and argon tank has been turned on. You must make sure that your sample introduction is set to the desired configuration before lighting the plasma. See [Section 3. Hardware Setup](#) for more information.
- It is recommended to choose your tune file before igniting the plasma. See [Section 6.4.](#) on the tuning panel for more information.
- Go to the instrument control panel in ICP-MS Top and click Plasma On in the plasma menu or hit the Ignite Plasma icon.
- You will be prompted with two dialogue boxes. The first will ask if the exhaust system is working. Click yes.
- The second will ask to verify that the drain tubing is connected. Since you are using Laser Ablation, click yes and do not worry about this dialogue.
- The plasma should light after a few minutes. The status bar of the instrument control window will show standby --> analysis until it is ready for operation. Once it is ready for use the status bar will only say analysis mode. See the [Section 6.3. Instrument Control](#) for more information.

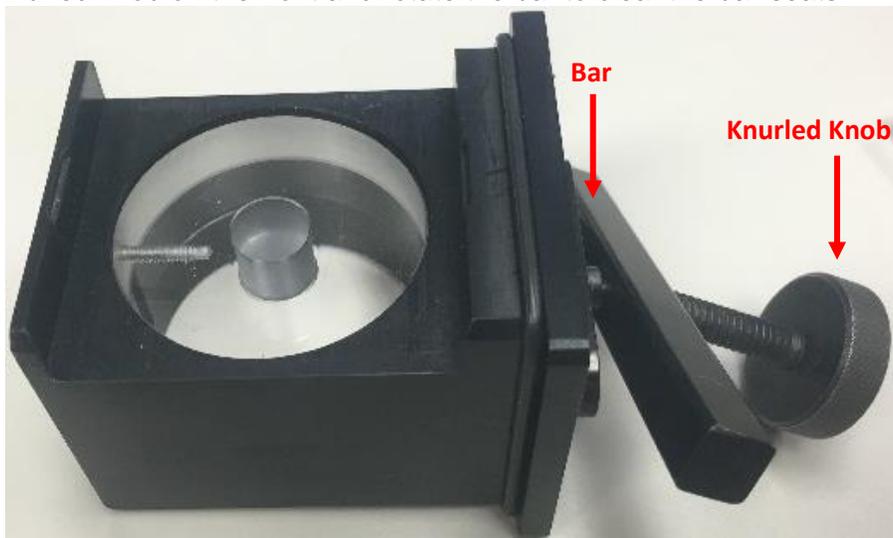


Left: Instrument control window in analysis mode without a reaction gas.
Right: Instrument control window in analysis mode with a reaction gas.

5. Laser Ablation

5.1. Loading a Sample

To load a sample, you must remove the sample stage from the laser ablation accessory. Simply unscrew the knurled knob on the front and rotate the bar to clear the bar-seats.



The stage will most likely have one of three inset holders installed as seen below. You will need to choose the appropriate type for your analysis. The left holder is for puck-style samples, the middle one is a deep-set slide holder, and the right one is a shallow slide holder with reference slots. Each of these have their own merits and uses. You want to try to fill as much volume as possible to ensure good aerosol flow through the chamber. Each holder has a rounded corner which means they can only be inserted in one orientation.



Once you have your sample loaded with the appropriate inset holder, simply replace the sample stage by reversing the steps to remove it. Ensure that your sample will not collide with the top of the chamber as you insert it. Occasionally, the o-ring on the stage will get caught and simply wiggle the stage up and down slightly will get it to seat properly. There is no need to overtighten the sample bar, it only needs to be snug.

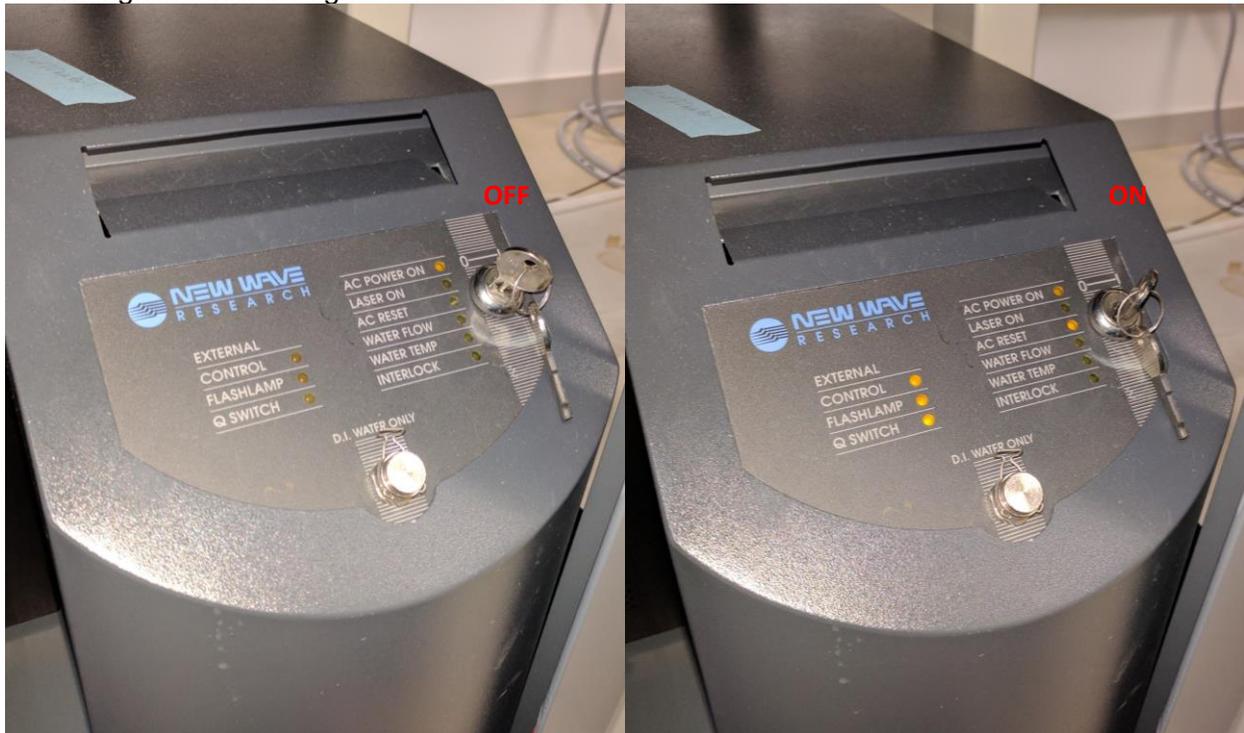
5.2. Setting up the Laser

We will discuss here what hardware to set up. There isn't a lot to do.

- First, make sure to turn on the laser itself. The software will remind you if you don't by popping up the following window if the key is not turned.



- Once you turn the key, the interlock status box will disappear as long as there are no other interlocks that are unsatisfied. See [Section 5.3.1.](#) on interlocks.
- The laser power supply is located right below the laser system. To turn it on, turn the key to the right so that the lights on the left come on.



- The last part of setting up the hardware is to turn on the helium tank. Remember this section only covers the laser ablation hardware. For ICPMS hardware setup, see [Section 4. Igniting the Plasma](#).
- Simply open the tank in the middle on the end of the ICPMS table. Do not adjust the output pressure or close the small valve on the regulator. When finished you should only close the tank valve.



5.3. Software

The software for running the laser is on a different computer than the ICPMS software. There are three monitors on the desk. The left one is for the laser computer and the right two are for the ICPMS. You can use the one mouse and keyboard to control both computers. Whatever monitor the mouse is on will dictate what the keyboard controls.

If the computer is off or isn't logged in, you will have to use the following credentials:

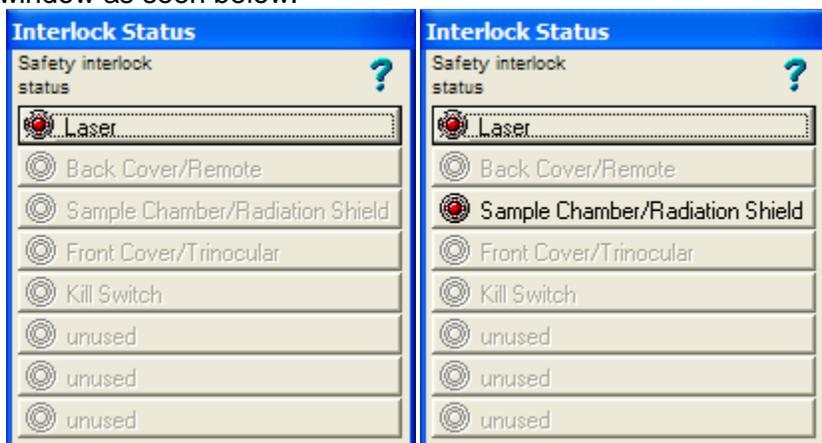
Login: AMSEC

Password: AMSEC

Domain: AMSEC (this computer)

5.3.1. Interlocks

The laser system has interlocks in place to protect the user. These interlocks are usually some sort of micro switch or magnetic sensor. If these interlocks are not satisfied, then you will get an interlock status window as seen below.

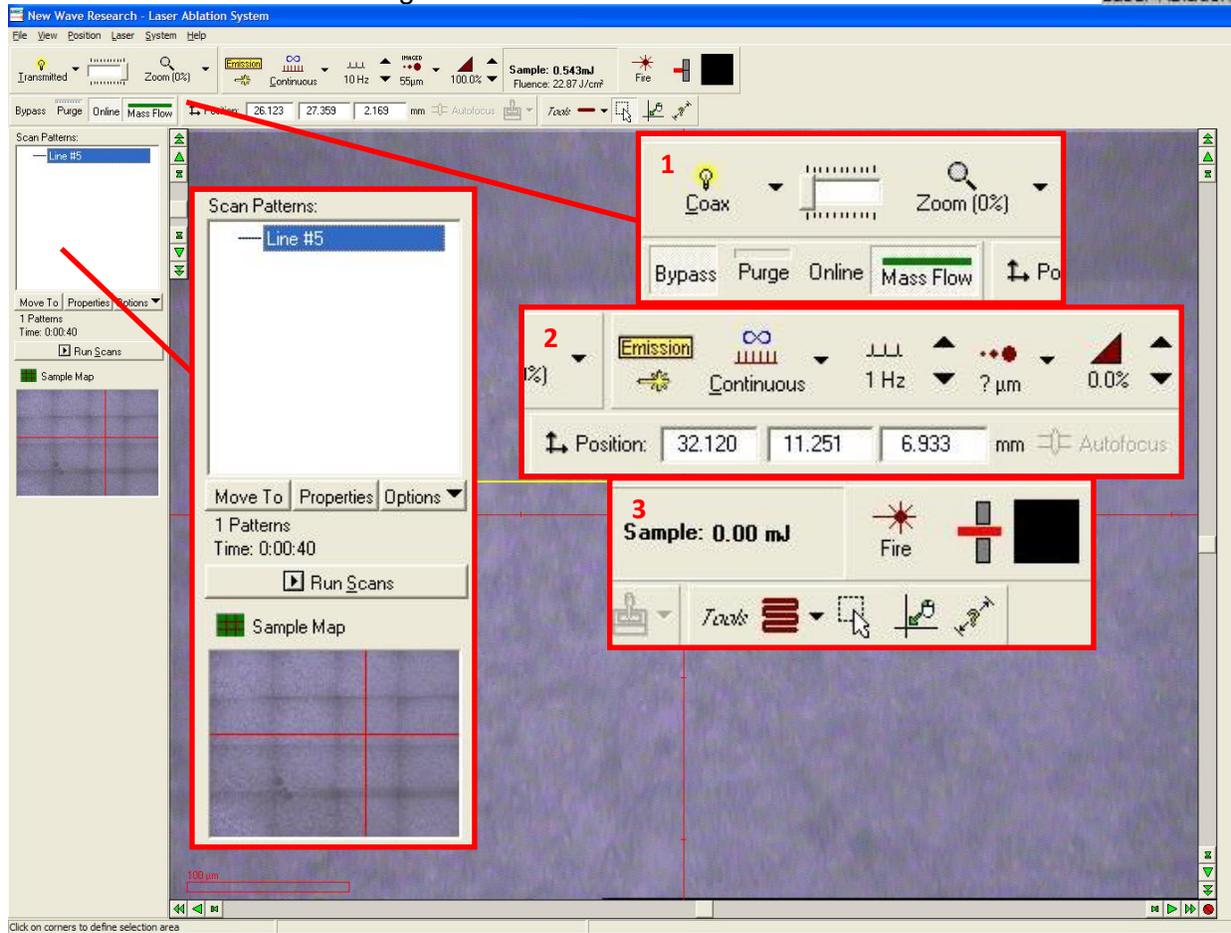


These two are the only interlocks that you should ever see. If any others show up, contact the lab manager.

- The laser interlock means that the key on the laser power supply is turned off. See [Section 5.2.](#) on setting up the laser. Once the power supply is switched on, the interlock should go away on its own. Occasionally you need to click on the interlock to get it to go away.
- The sample chamber interlock means that the sample door is open and/or not secured properly. This can be remedied by closing and clamping the door properly. See [Section 5.1.](#) for more information.
- The interlocks will only prevent the laser from firing, the sample can still be viewed, and scans can be set up.

5.3.2. Software Layout

On the desktop, double click the laser ablation icon to open the software. The window that opens will look as seen below. There are zoomed in regions in red boxes below for ease of viewing.



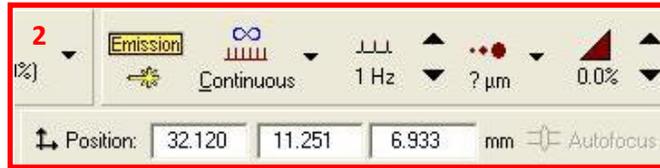
In the red boxes from left to right we will discuss the buttons of the program.

Scan Pattern Box:

- The white area will show all of your scan patterns.
- **Move To:** moves the crosshairs to the start of the selected scan.
- **Properties:** opens the properties pane of a selected scan. [Section 5.3.6.](#) has more info.
- **Options:** displays a list of options that will be discussed in [section 5.3.5. Pattern Options](#)
- **Run Scans:** how you ablate a pattern.
- The sample map displays the map if you have made one and you can navigate by clicking it. This doesn't always display properly.

Red Box 1:

- The light source can be changed by click either the box or the dropdown. Play around with the light sources to get the best illumination.
- The light intensity can be changed by using the slider bar to the right of the light selector.
- **Zoom:** change camera zoom. This isn't very useful.
- **Bypass, Purge, and Online:** located below the light selector. These are discussed elsewhere in [Section 5.4.2.](#)
- **Mass Flow:** opens the window to change the flow of helium through the cell. [Section 5.4.](#) has more info.



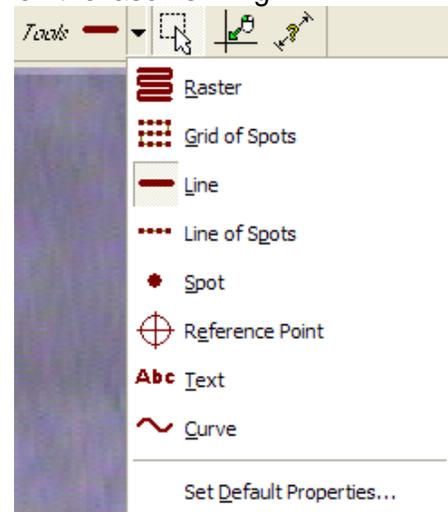
Red Box 2:

- **Continuous:** selects between different laser firing modes. Continuous, burst, and single. Continuous is most commonly used.
- **Hertz:** Selects the firing frequency between 1 and 20 Hz. 10 is most commonly used.
- **μm Selector:** Selects the laser spot size between 4 μm and 100 μm. All spots are round except for the 65 μm square spot.
- **% Selector:** Selects the power of the laser in percentage. 0 – 100% can be selected in increments of 5.
- **Position:** Shows the current x, y, and z position of the crosshair.



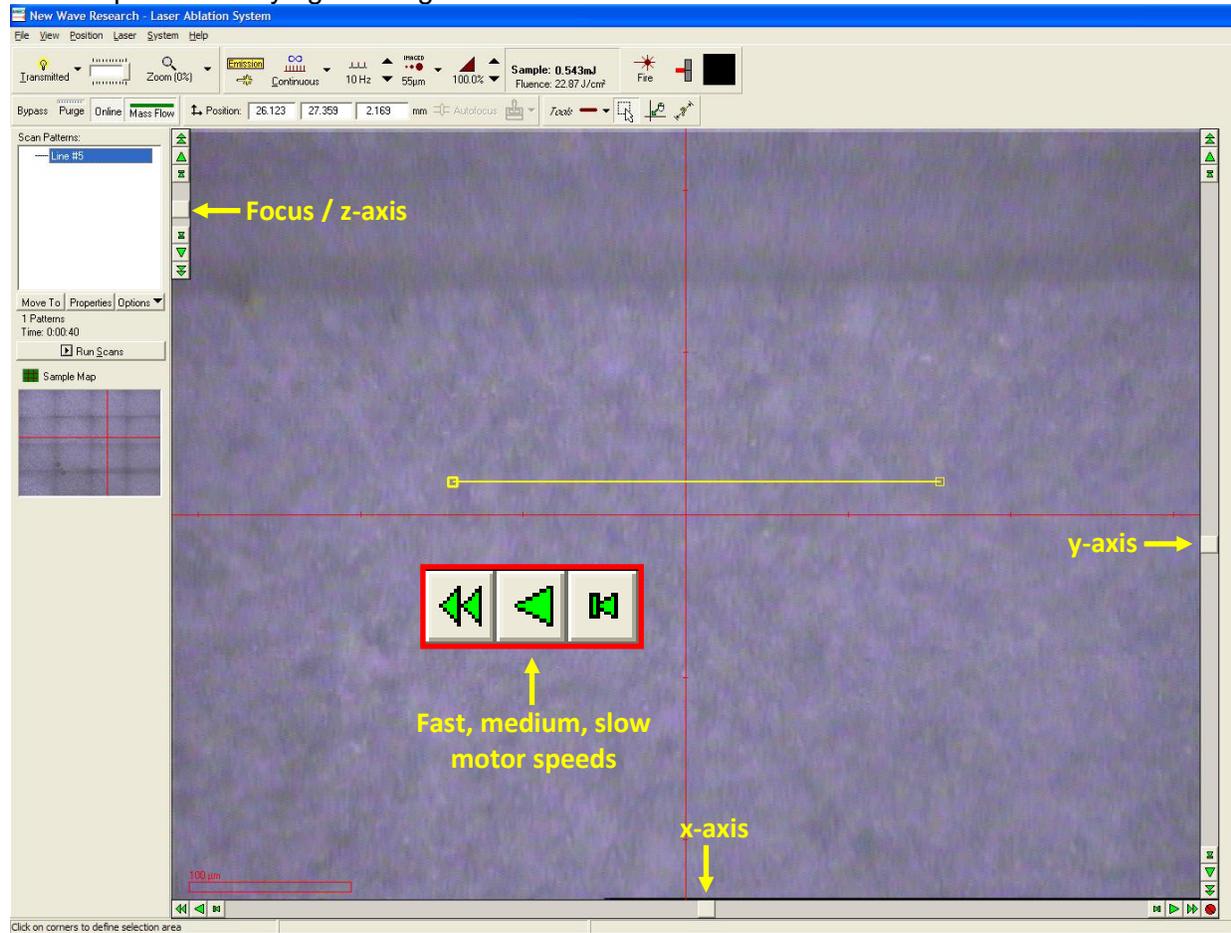
Red Box 3:

- **Sample:** This is not a button, but a readout of the power of the laser to the sample.
- **Fire:** fire the laser manually.
- **Shutter:** manual shutter operation to open or closed. This is overridden by any scan that is ran.
- **Black Box:** The black box will display an animation when the laser is firing.
- **Patterns:** This changes the mouse tooltip to a pattern drawing tool. The options for this are show to the right. They are fairly self-explanatory. Play around with drawing things if you are curious. To draw things, you must select the type of pattern and then left click on the screen, drag to the size and then left click again. If you would like to continue drawing, then keep dragging. If you want to quit simply right click on the end point.
- **Mouse Box:** changes the mouse tooltip to a selector tool to move patterns around.
- **Mouse Crosshair:** changes the mouse tooltip to move the crosshair to the clicked position.
- **Measure ?:** changes the mouse tooltip to a measurement device. The measurement shows up at the bottom of the window.



5.3.3. Navigation and Focus

Navigation of your sample is probably the most important part of using the laser. You need to know where you are and what you are seeing and ablating. Make sure to properly illuminate the sample before trying to navigate.



Focus:

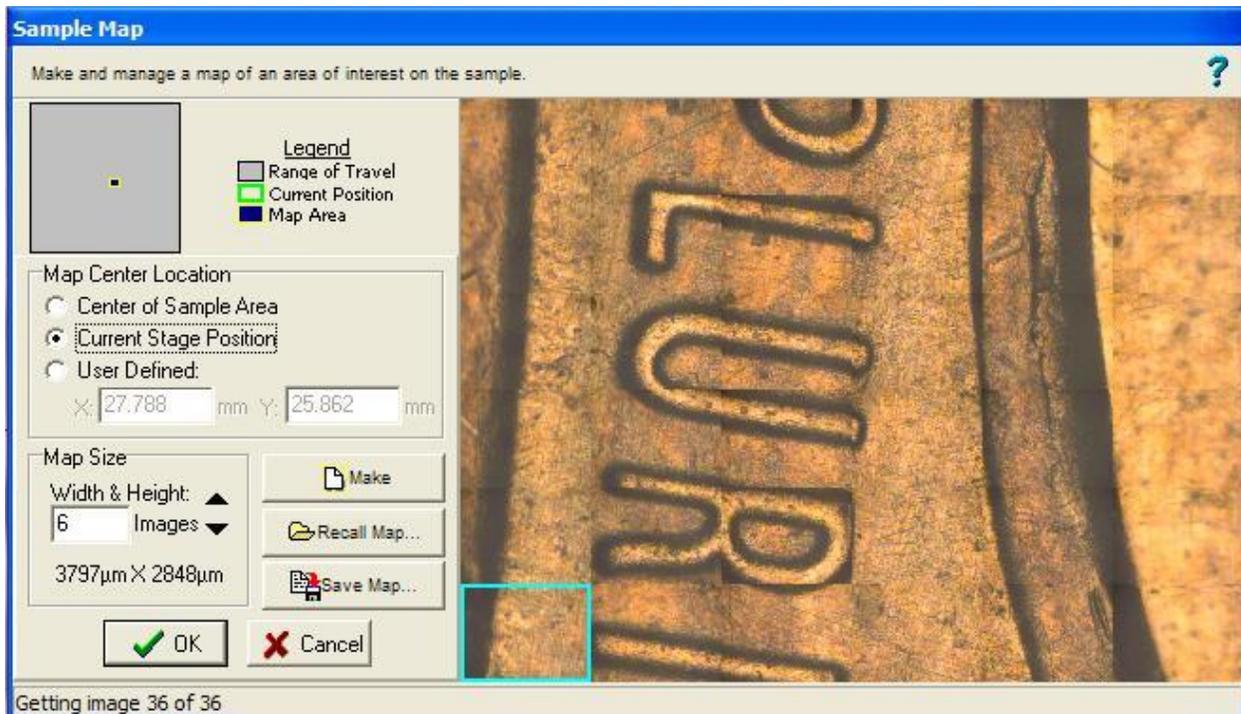
The z-axis motor must be used to focus the camera and laser on the sample surface. When the software is started, it will default the focus to the highest point, which is often far away from the surface. If finding the surface is difficult and it would not be detrimental to the study, the laser can be manually fired at the surface to create a crater to focus on. The laser will have to be fired while focusing if it is very out of focus.

Navigation:

Navigation takes place via the x and y-axis controls as labeled above. The arrows can be used to control the speed and direction. Alternatively, the slider bar may be dragged, and the motors will bring the position to that point. Another useful tool are reference points that are placed via the pattern tooltip. Experiment with what works for you. The next section on Sample Maps is also relevant to navigation. The mini map on the left can be clicked on to move around your sample map. You can also keep the sample map window open to navigate as well.

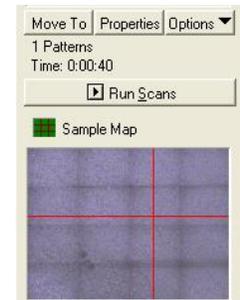
5.3.4. Sample Maps

The sample maps window can be accessed from the “view” dropdown at the top of the screen or from right under the “Run Scans” button on the left side of the window.



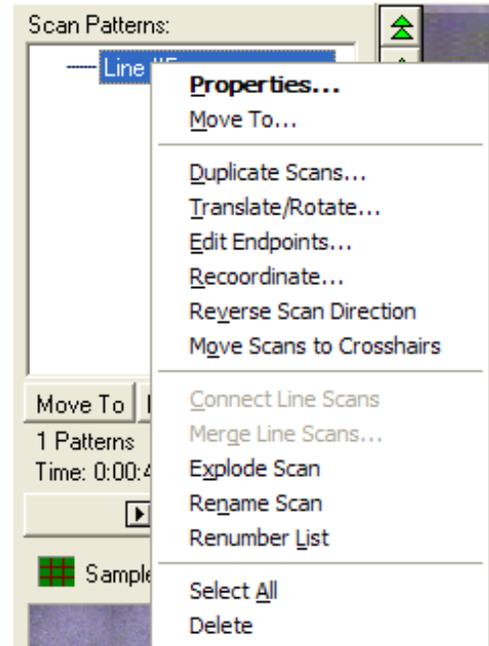
- The sample map window has a couple options that are critical. The main one being the “Map Center Location” area. The default option is “Center of Sample Area” which means it will go to the center of the x and y-axes and start there. Normally “Current Stage Position” is selected as the center of the map.
- The width and height is also important under “Map Size”. It is typically done in images and usually anywhere from 2 – 10 is chosen. Depending on the feature size of the sample to be analyzed, the setting will need to be altered. Once a map is made, it is easy to make another one. The image produced can be save as well.
- This window can be left open and the image can be clicked on for navigation, or the smaller image on the left can be used.

NOTE: It is very common for the image not to appear on the left hand of the screen under the “Run Scans” button. The image fills in sporadically as the crosshair moves on the screen. Therefore, using the sample map window is typical over trying to use the small image.



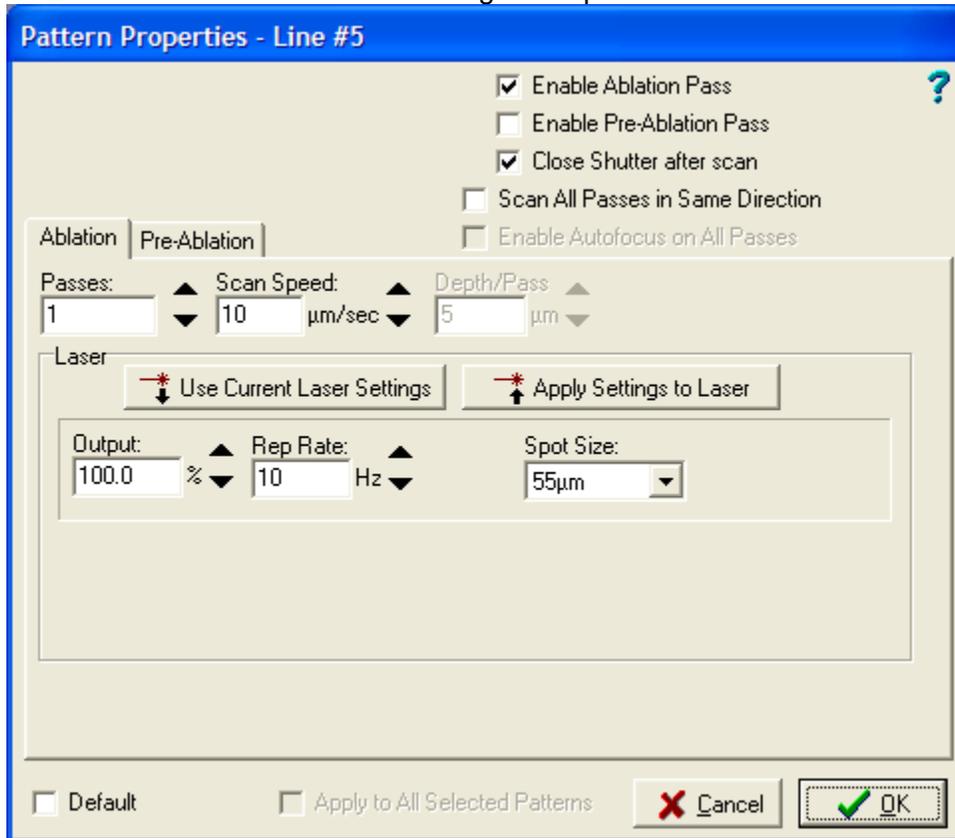
5.3.5. Pattern Options

- The options for the patterns can be accessed by right clicking on the pattern, or by selecting the pattern and clicking the options button below the scan patterns box.
- Most of the options are self-explanatory. Some very useful options are “Edit Endpoints”, “Recoordinate”, “Reverse Scan Direction”, and “Move Scans to Crosshairs”.
 - **Edit Endpoints:** allows the exact coordinates of the pattern to be altered in the x, y, and z-axes. This is useful to control the exact length of a line or the position of a spot.
 - **Recoordinate:** allows the pattern to be moved to a new position keeping its current relative proportions.
 - **Reverse Scan Direction:** in conjunction with the move to function is useful for checking the length of a scan before running it. Simply click reverse, then click move to and the crosshairs will follow the scan. Then click reverse and move to again and it will revert to the original position.
 - **Move Scans to Crosshairs:** must be used with caution. It is useful for moving a scan a long distance very easily, but it will move all of your patterns to the new crosshair position keeping their positions relative to each other.



5.3.6. Pattern Properties

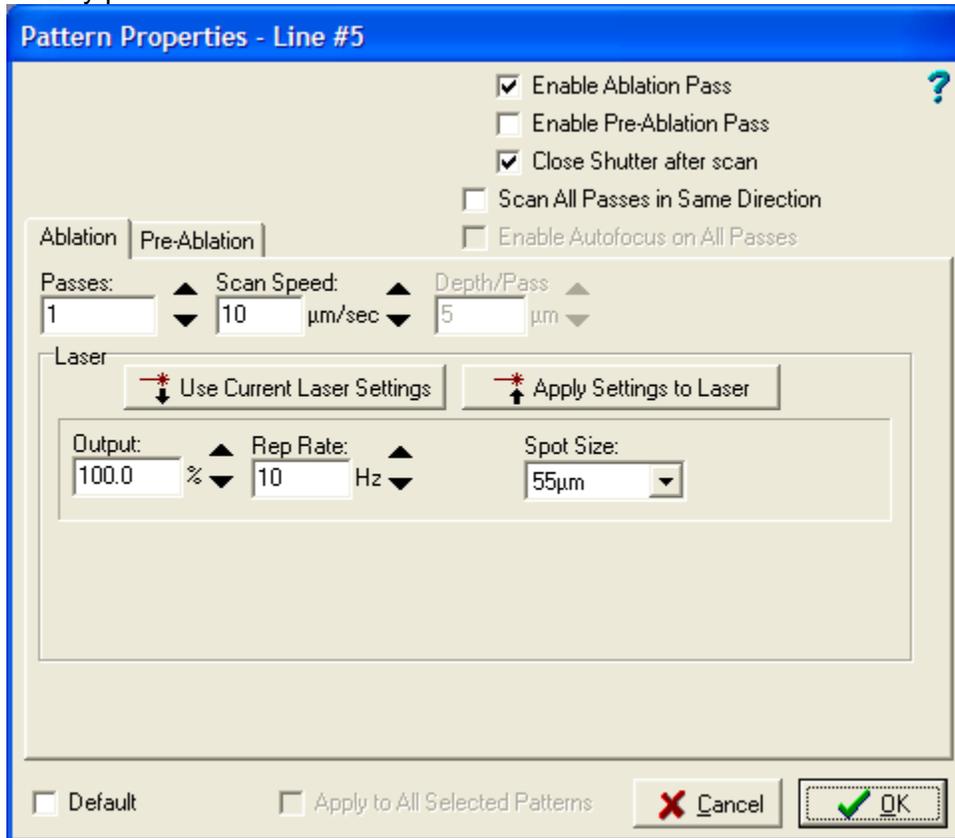
Pattern properties can be accessed via the button below the scan patterns box, or by right clicking on the pattern. This portion is very important as it deals with how the laser settings at the top of the window differ from the laser settings for a pattern.



- The properties window looks similar for each type of pattern. The settings should be chosen properly for the type of pattern you are running and for the type of material. For example, a soft sample such as a polymer might need a slow scan speed, but a lower power like 50 %. The main thing to pay attention to is that each pattern will have different properties associated. If you want them all to have the same properties, then after you make the first one, select the “Default” checkbox at the bottom of the pane and all subsequent patterns will have the same settings.
- Another easy way to change settings is by using either of the buttons in the middle. You can apply your setting from the laser to this pattern or apply pattern settings to the laser. This means the setting in the main window of the software will change or apply the change.
- A number of patterns can be altered at the same time. If multiple patterns of the same type are selected, then all their settings will change.

5.3.7. Preablation Pass

From the pattern properties pane, a preablation pass can be performed. A preablation pass is when the pattern is scanned before the actual scan with a much lower power setting to clean off the surface of any potential contaminants.



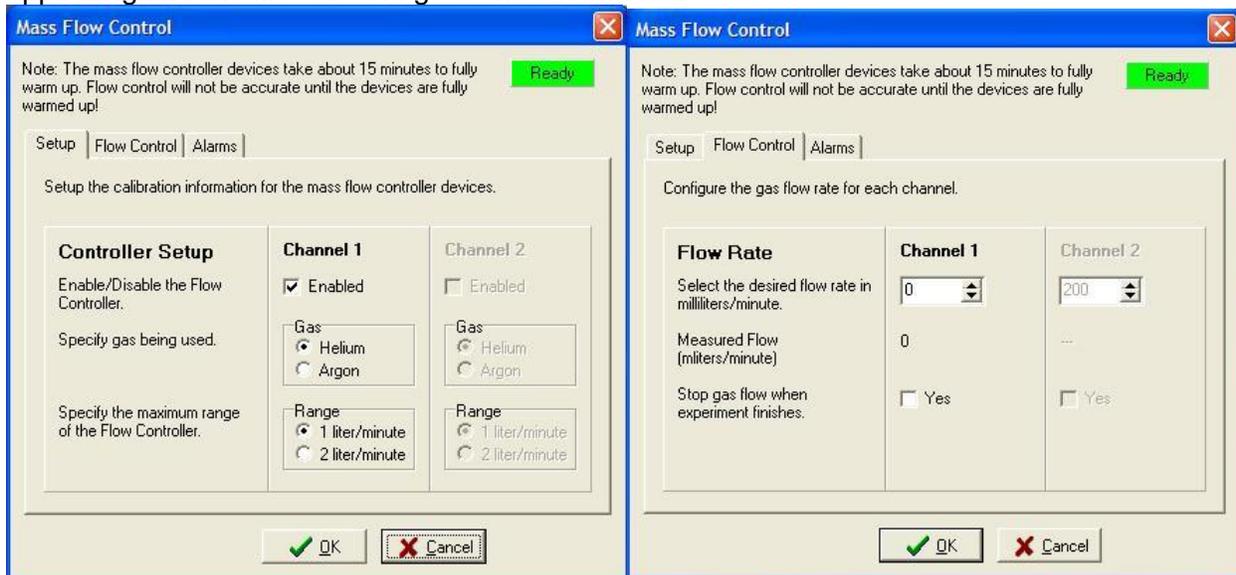
- The first thing that must be done is clicking the checkbox at the top to enable the preablation pass. Once that is done, the second tab of this box will be available, and the setting can be chosen for the preablation pass.
- The setting one should choose are variable and should be tested out. The sample should not be ablated during this process so different setting should be chosen to apply the most power to the surface without ablating the sample.

5.4. Gas Flow

The gas flow of the laser cell is a very important parameter. The flow should be kept constant for all experiments of the same type. The flow rate can be tested by ablating a standard and watching the signals across the screen to see how intense the signal is and how long it takes to go back to background. This time after ablation for the signal to go to background levels is called the washout delay. Typical flow rates are 250, 400, and 600 mL/min.

5.4.1. Changing the Flow Rate

To change the flow rate, click on the “Mass Flow” button next to the bypass, purge, and online buttons. The following window will appear. Ignore the first tab and go to the second tab.



To change the flow rate, the value desired can be typed into the box under channel 1. It is recommended to use the up arrow to change the flow rate when the plasma is lit. This way will extinguish the plasma less often. Once the desired value is attained, select OK and the box will close. If cancel is chosen, the value will revert to what it was before the dialogue was opened.

5.4.2. Bypass, Purge, and Online



The bypass, purge and online buttons are important for the analysis as well as for keeping the plasma running while changing samples.

Bypass: carries the helium around the sample chamber, but the helium gas still is flowing to the plasma torch.

Purge: flows helium through the sample cell and purges the air to the room, not to the plasma. After 30 seconds, the purge will finish, and the system will switch to online mode automatically. NOTE: There must be helium flowing in the Mass Flow window for the purge to work.

Online: sends the helium gas through the sample cell and to the plasma for analysis.

5.4.3. Helium and Igniting the Plasma

There are different methods recommended for what to do with the helium when igniting the plasma. The easiest method so far seems to be the following.

- After the software is first opened, click the bypass, purge, and online buttons to get rid of any built-up pressure that may have been stored in the system.
- Set the flow to the desired flow rate such as 250, 400, or 600 mL/min.
- Purge the cell.
- After purge, set the flow rate to 50 mL/min.
- Ignite the plasma. See [Section 4](#) on plasma ignition.
- Once the plasma is fully lit, use the up arrow in the Mass Flow window to increase the flow rate to the desired value.
- Pause every two to three seconds to ensure the plasma isn't extinguished by the increase in flow.

5.5. Analyzing a Sample with the Laser

5.5.1. Spectrum Analysis

The trick in analyzing a sample with the laser is the timing between the laser and the ICPMS. There is a delay between when the ablation starts and when the acquisition occurs that depends on the gas flow. For 250 mL/min it is about 8 seconds. Usually more time is given as a safety measure so 10 seconds is recommended. At 600 mL/min it only takes around 4 seconds for the sample to reach the ICPMS to be analyzed. Five seconds is a safer bet.

- First, make sure the laser and ICPMS are all setup. See the [Concise Operating Procedures](#) for more info and links to other sections.
- Load your method file. [Section 7](#).
- Click Method→Run. I prefer to use this option over manual sampling because you get better control over the timing of the run in spectrum mode. There isn't much difference between the two except the second window that pops up. The following window will appear:

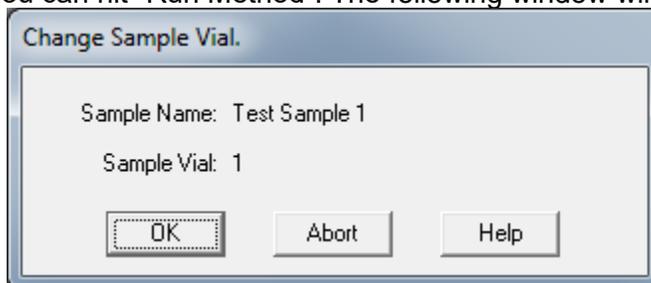
The 'Start Run' dialog box is a standard Windows-style window with a title bar. It contains several sections of controls:

- Data Batch:** A text field with 'E:\DATA\RESEARCH\AMSEC\Test.b\'. A 'Browse...' button is to its right.
- Data File Name:** A text field with '001.d'. A 'New Batch' button is to its right.
- Sample Type:** A dropdown menu showing 'Sample'.
- Cal Level:** A text field with '1'.
- Import DA Method from Existing Batch:** A checkbox that is unchecked. Below it are two radio buttons: 'DA Method Only' (selected) and 'DA method and Std Data'.
- Source Data Batch Directory:** A text field with 'E:\DATA\RESEARCH\SOFIELD\CWALL\TOP\1nofal0.batch.xml'. A 'Browse...' button is to its right.
- Disk Space:** A text field showing '343,378M bytes free on drive E:'.
- Operator Name:** A text field. To its right is a 'Vial:' label and a text field with '1'.
- Sample Name:** A text field with 'Test Sample 1'.
- Misc Info:** A text field.
- Dilution:** A text field with '1.000' and a 'Calculate...' button to its right.
- ISTD Conc:** A text field with 'Level 1' and a 'Change...' button to its right.
- Custom Std Conc:** A text field with 'Change..' and 'Remove...' buttons to its right.
- Method Sections To Run:** A section with three checkboxes: 'Pre-Run Macro/Cmd:', 'Pre-Run Monitor', and 'Post-Run Macro/Cmd:'. Each has a corresponding text field. The 'Pre-Run Monitor' checkbox has a 'Configure...' button to its right.
- Buttons:** At the bottom are 'Edit DA Method', 'Run Method', 'OK', 'Cancel', and 'Help' buttons.
- Footer:** A small text label 'Sample Name: Describe the sample name'.

Important Parts:

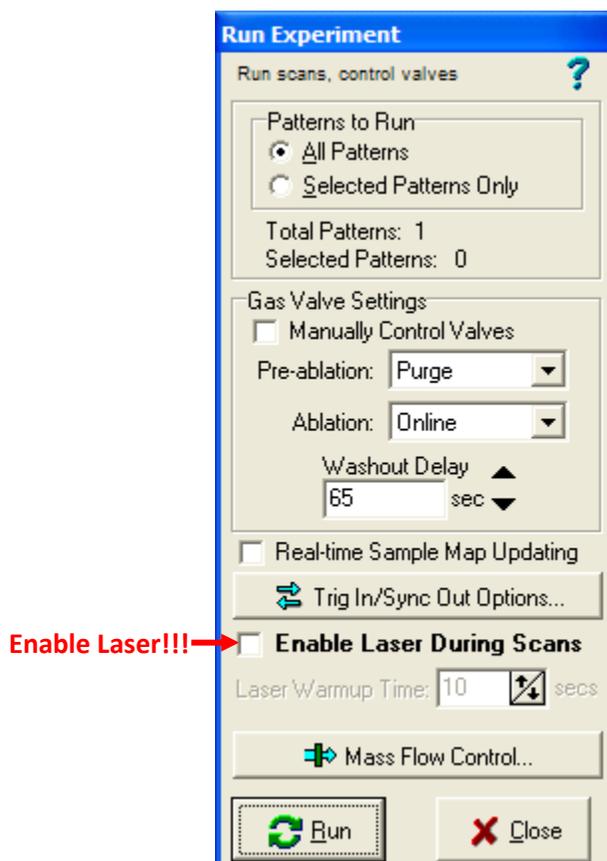
- **Data Batch:** redirect the file path to your batch folder using the browse button. [Section 9.2](#).
- **Data File Name:** do not duplicate file name. This can be complex or simple such as 001.d, 002.d, etc.
- **Sample Name:** this is where it recommended that the sample name is input.

Once you are ready, you can hit “Run Method”. The following window will appear:



Once “OK” is pressed in this window, the acquisition will commence. This is where the timing takes place. Once this box is seen, then the laser part needs to get set up.

Make sure that a pattern is already drawn. See [Section 5.3.5](#). for more information. Also, make sure that the pattern has the appropriate scan properties. Make sure it is long enough to last the duration of the acquisition. Once the pattern is drawn and setup and selected, then hit “Run Scans” in the laser software on the left above the sample map. The following window will appear:



It is important to check the box “Enable Laser During Scans” or else the laser won’t actually fire. The laser warmup time is 10 seconds. Once you hit “Run”, the laser will warm up and then fire. You need to wait until the laser fires and then count between 5 and 10 seconds for the sample to reach the plasma depending on the flow rate. Then hit OK in the ICPMS software and the analysis will commence. Then repeat for each sample. Don’t forget about the [Washout Delay!!!](#)

5.5.2. Time Resolved Analysis

Time resolved analysis is very similar to the spectrum mode, except that there is not as much need for timing the laser firing with the ICPMS acquisition. It is typical to have the background plotted before the sample is acquired so that the background can be seen as flat and subtracted out later.

- First, make sure the laser and ICPMS are all setup. See the [Concise Operating Procedures](#) for more info and links to other sections.
- Load your method file. [Section 7.](#)
- Click the data acquisition main panel button seen below left. Then click the acquire data button. The following window will appear:

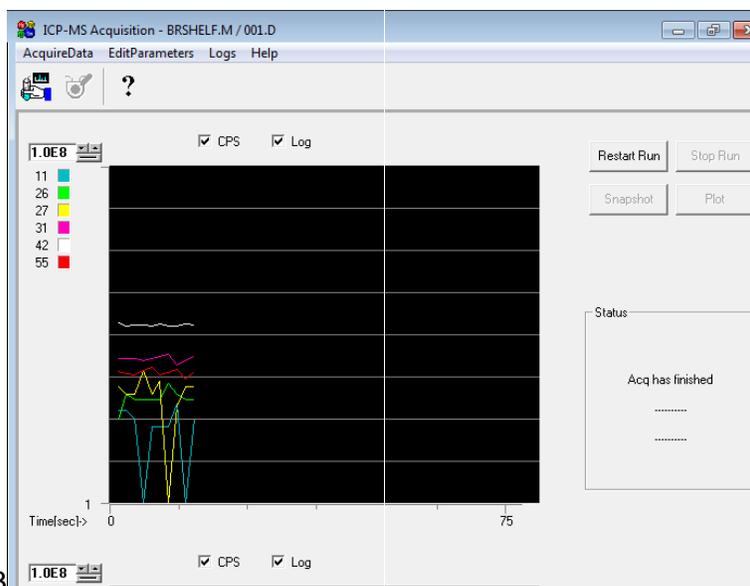
The screenshot shows the 'Acquire Data' dialog box with the following fields and options:

- Data Batch: E:\DATA\RESEARCH\AMSEC\Test2.b\
- Data File Name: 001.D
- Sample Type: Sample
- Cal Level: 1
- Import DA Method from Existing Batch:
- DA Method Only: DA method and Std Data:
- Source Data Batch: E:\DATA\RESEARCH\SOFIELD\CWALLI\1nofal0.batch.xml
- Disk Space: 343,377 MB free on drive E:
- Operator Name:
- Sample Name: Test Sample 1
- Misc. Info:
- Dilution: 1.000
- ISTD Conc: Level 1
- Custom Std Conc:
- Pre-Run Monitor:

Important Parts:

- **Data Batch:** redirect the file path to your batch folder using the browse button. [Section 9.2.](#)
- **Data File Name:** do not duplicate file name. This can be complex or simple such as 001.d, 002.d, etc.
- **Sample Name:** this is where it recommended that the sample name is input.

Once you are ready, you can hit “Acquire”.

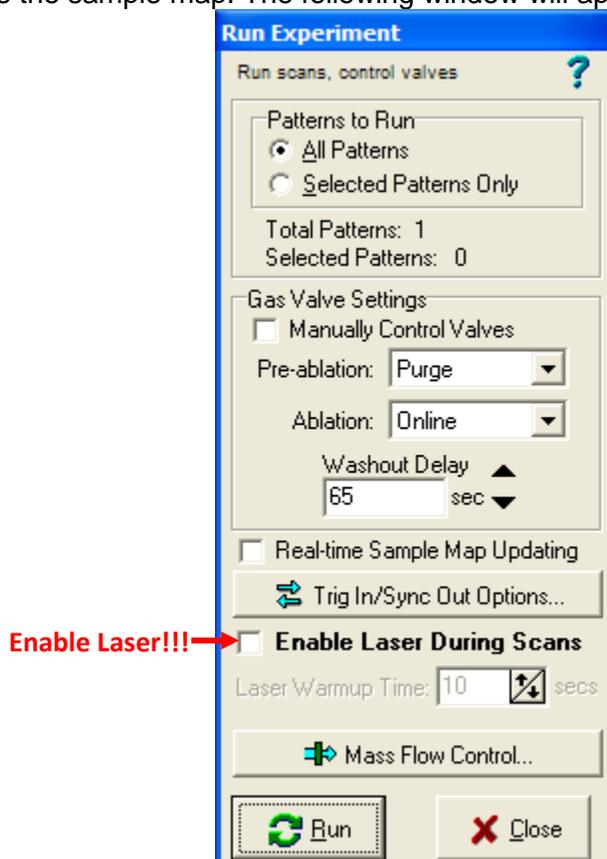


3

The acquisition will commence. You will typically go to the laser window and hit run at this point. This will usually give you about 15 seconds of background.

- Make sure that a pattern is already drawn before hitting acquire. See [Section 5.3.5](#) for more information. Also, make sure that the pattern has the appropriate scan properties. Make sure it is long enough to last the duration of the acquisition.

- Once the pattern is drawn and setup and selected, then hit “Run Scans” in the laser software on the left above the sample map. The following window will appear:



It is important to check the box “Enable Laser During Scans” or else the laser won’t actually fire. The laser warmup time is 10 seconds. Once you hit “Run”, the laser will warm up and then fire. Once the laser fires it takes between 5 and 10 seconds for the sample to reach the plasma depending on the flow rate. Time your acquire button press with your laser firing to give adequate background signal and enough of your required acquisition. This is where the method setup is very important.

The acquisition will complete and save. Repeat for each sample. Don’t forget about the [Washout Delay!!!](#)

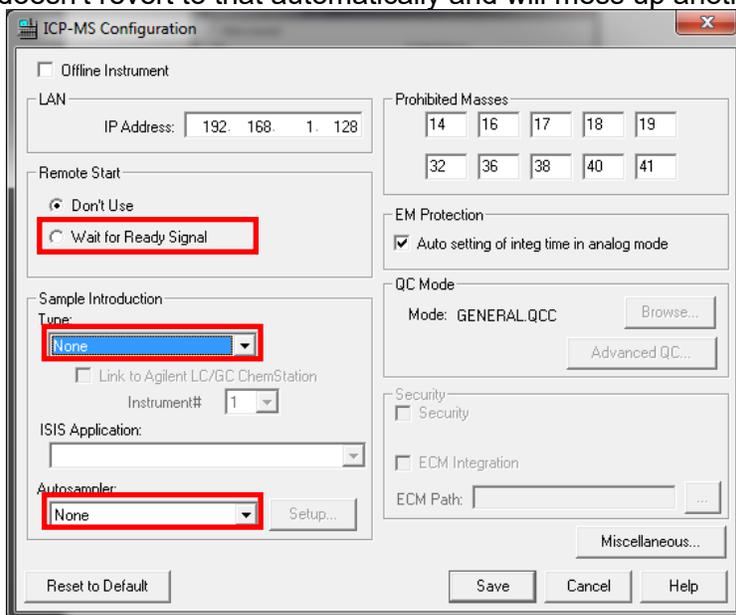
5.5.3. Washout Delay

Between runs with the laser, the lines will need to be purged. This normally is achieved by waiting between runs. Usually somewhere in the realm of 60 – 120 seconds is adequate for the lines to be cleared of the previous sample. If you notice your backgrounds climbing, you may need to increase your washout delay.

5.6. Analyzing a Sample with a Sequence

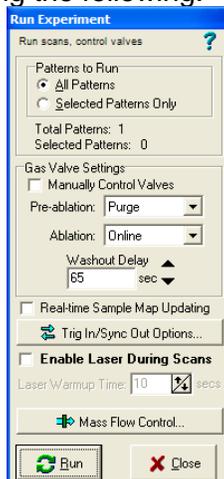
An advanced method of using the laser in combination with the ICPMS is available by which repetitive analyses can be performed. This method utilizes the ability of the laser and ICPMS to “talk” to each other using a trigger signal. The laser triggers the ICPMS, but the ICPMS does not send any input back to the laser so precise timing is required.

The first step is to change the Remote Start option in the configuration window to “Wait for Ready Signal”. We ask users who use this option to remember to change it back to “Don’t Wait” after analysis as it doesn’t revert to that automatically and will mess up another user’s run.

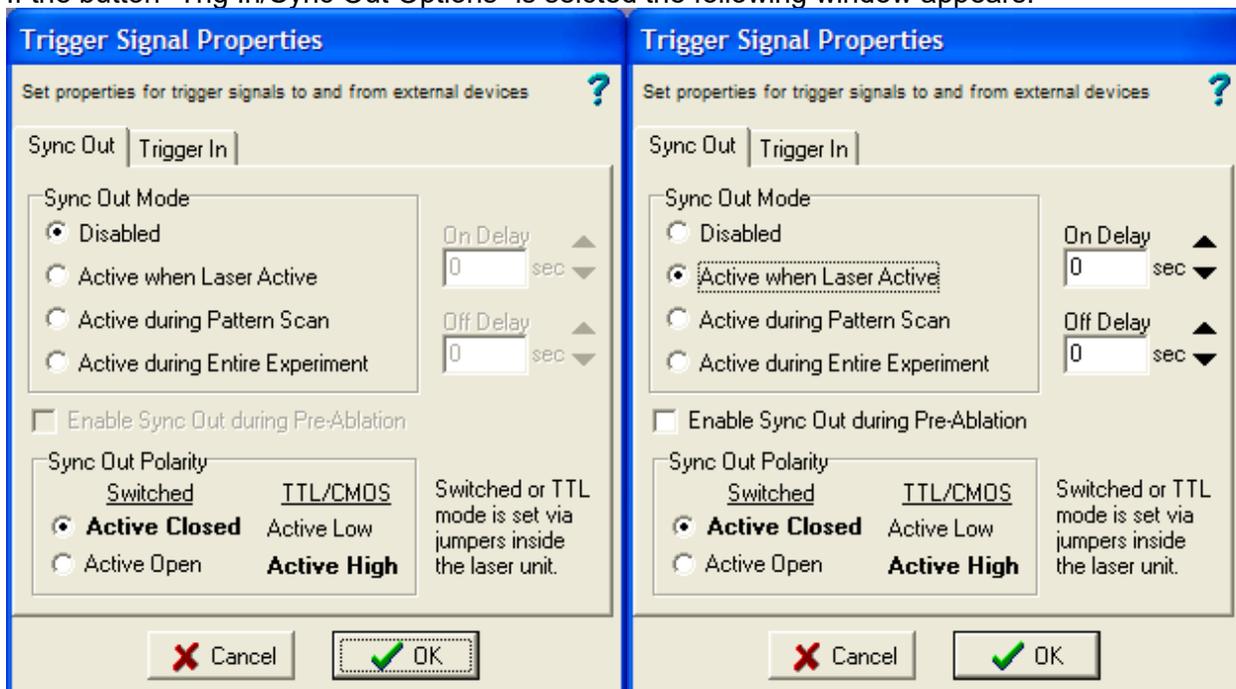


The timing part comes when setting up your scan patterns in the laser software. The scan must be long enough that the acquisition can complete, but not so long that it goes far past the acquisition. Your method file needs to have the proper timing as well. Then the washout delay needs to be adequate but also take into account the fact that the ICPMS software will take a few seconds to reset after each run and it will wait for the trigger signal that you decide to send it.

The following relates to how the trigger signal works. When you select “Run Scans” in the laser software window, you are used to seeing the following:



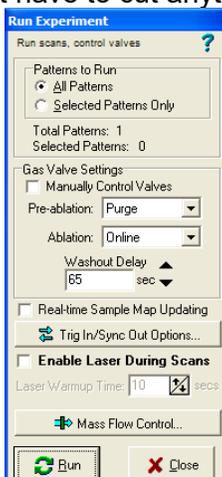
If the button “Trig In/Sync Out Options” is selected the following window appears:



Change the Sync Out mode to Active when laser active or active during pattern scan. The first option is the easiest to deal with. They are self explanatory. When it says active when laser active, then the trigger signal will be sent when the laser turns on.

You may also use the on delay in case you want the laser warmup to be ignored or let the sample make it to the plasma before collecting data. The off delay is not used since the laser only tells the ICPMS when to start a collection and not when to stop.

We do not use trigger in because we can't. The ICPMS doesn't talk back to the laser. Going back to the run window will show the last parameter to time and that is the washout delay. Typically, longer is better and you won't have to cut anything too close in your timing.



After this part is all setup and timed, you can then setup your sequence file and continue with the acquisition.

You must have a method created before setting up or running a sequence file. For information on this, see [Section 7. Creating/Editing a Method](#). You must also have ignited the plasma and selected your tune file. See [Section 1. Concise Operating Procedures for Liquids](#) for more information.

- Set up your sequence file as described in [Section 8. Sequence Files](#).
- Once your sequence file is set up and saved, you are ready to begin a sequence.
- From the Sequence dropdown, select Run.
- The Start Sequence Dialogue will appear.

Start Sequence default.s Last Modified: Thu Apr 24 17:15:02 2008

Overwrite Existing Data Files

Print Sequence Log at Sequence Termination

Sequence Comment:

Operator Name:

Data Batch Directory:

Import DA Method from Existing Batch

DA Method Only DA method and Std Data

Source Data Batch Directory

Pre-Seq Macro/Cmd:

Post-Seq Macro/Cmd:

Sequence will pause if a data file already exists

- Unless you are confident that you want to, do not check the Overwrite Existing Data Files box. This will overwrite any file with the same name.
- Next, use the Browse button to locate your batch file. For more information on this, see [Section 11.2. Creating a Batch File](#).
- Click Run Sequence and the process will begin.

If the Data Analysis window is not already open, it will open, and the data will appear there. For more information see [Section 11.2. on Creating a Batch File](#).

Note: always stick around for a couple runs to make sure the timing works properly. Also, you can test your timing out on a piece of glass or with the laser disabled during the scan.

6. ICPMS Software Overview

6.1. Before Starting the Software

- Before the ICP-MS software can be opened, the configuration must be set properly based on which mode, Liquids or Laser Ablation, the instrument will be run in.
- First, click on the Configuration icon on the desktop. This must be done with the ICP-MS Top window closed along with any other masshunter windows.



- Once the window opens, ensure that it looks as seen below for either Liquids (Top) or Laser Ablation (Bottom), then click save and proceed to open the ICP-MS Top software:

The ICP-MS Configuration window is shown for the 'Liquids' mode. The 'Sample Introduction Type' dropdown is set to 'Peristaltic Pump' and the 'Autosampler' dropdown is set to 'AS-500'. Both dropdowns are highlighted with red boxes. Other settings include IP Address: 192.168.1.128, Remote Start: Don't Use, and QC Mode: GENERAL.QCC.

Liquids

The ICP-MS Configuration window is shown for the 'Laser Ablation' mode. The 'Sample Introduction Type' dropdown is set to 'None' and the 'Autosampler' dropdown is set to 'None'. Both dropdowns are highlighted with red boxes. Other settings include IP Address: 192.168.1.128, Remote Start: Don't Use, and QC Mode: GENERAL.QCC.

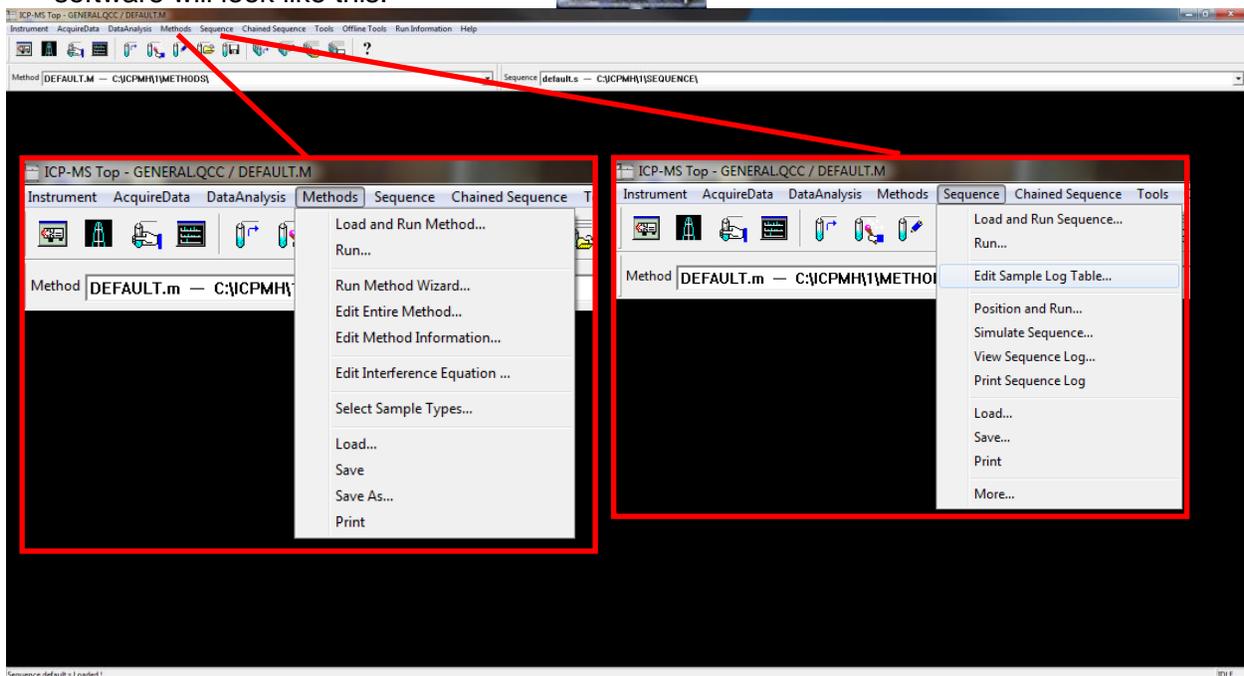
Laser Ablation

6.2. Layout

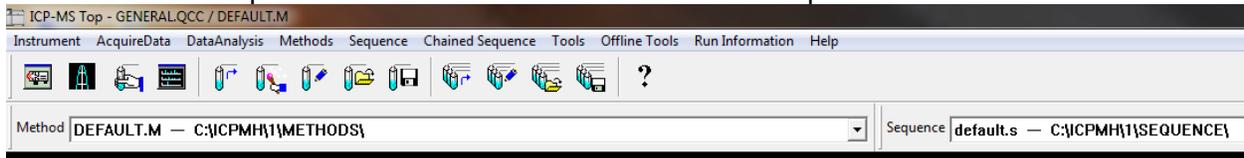
- Upon opening the software by clicking



on the desktop, the general layout for the software will look like this:



- Most of the important menus and information are in the top left:



From left to right across the menu bar:

- 1) **Instrument:** enter the instrument control and tune panel
- 2) **Acquire data:** perform a measurement manually
- 3) **Data analysis:** opens the Masshunter online data analysis window for data handling
- 4) **Methods:** load, save, edit, and run a method
- 5) **Sequence:** load, save, edit, and run a sequence
- 6) The rest are not typically used

From left to right across the icon bar:

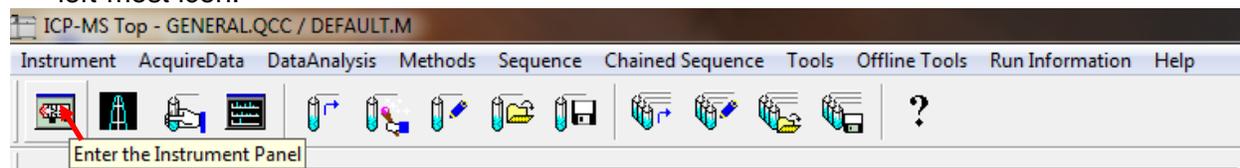
- 1) Instrument Control
- 2) Tune Panel
- 3) Acquire Data
- 4) The rest are not typically used

From left to right below the icon bar:

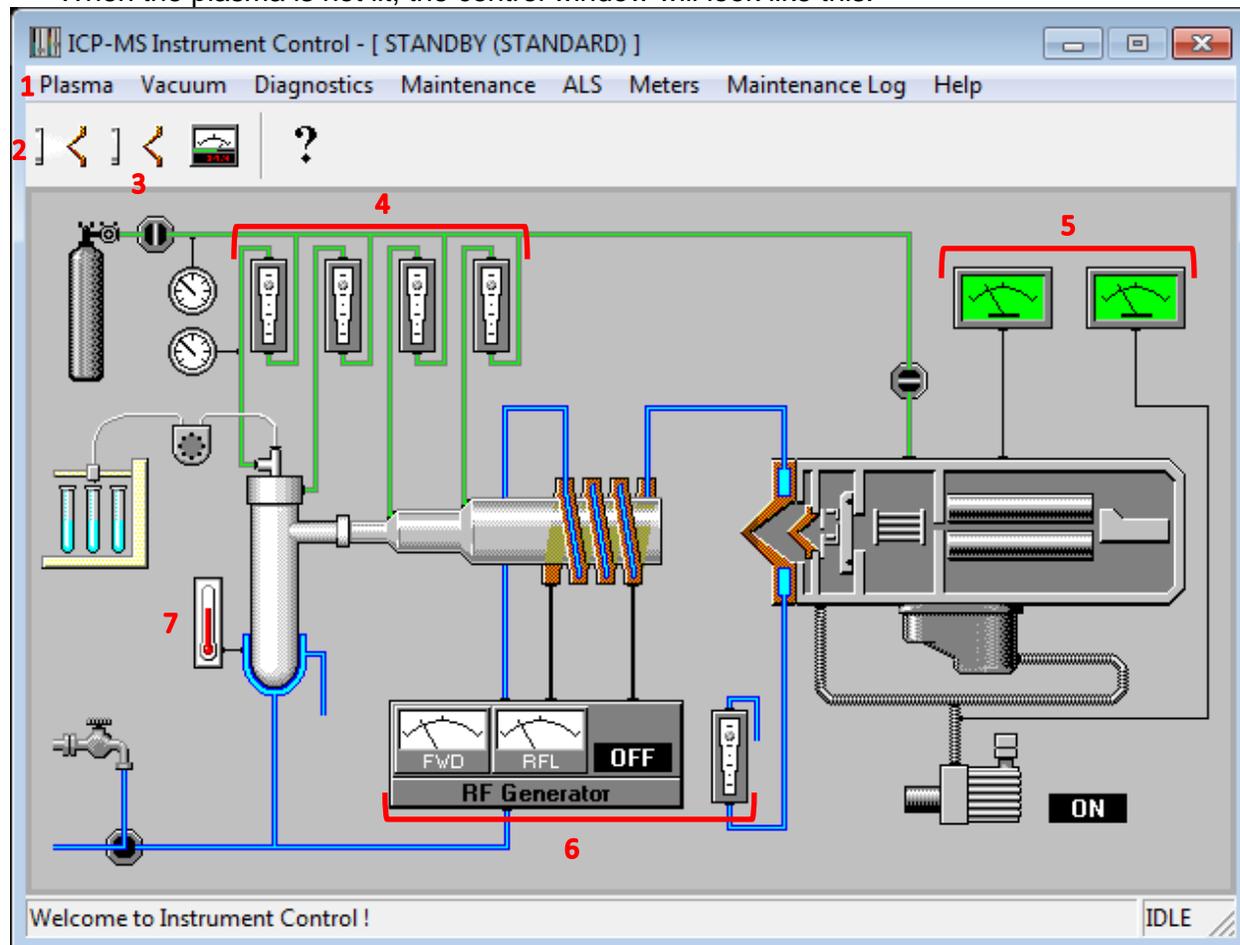
- 1) **Method:** Displays the currently selected method. The most recently used 4 are in the dropdown.
- 2) **Sequence:** Displays the currently selected sequence file. (LA users don't typically use a sequence file, it can be used for repetitive scans)

6.3. Instrument Control

- To enter instrument control, go through the instrument menu on the menu bar or click the left-most icon:



- When the plasma is not lit, the control window will look like this:



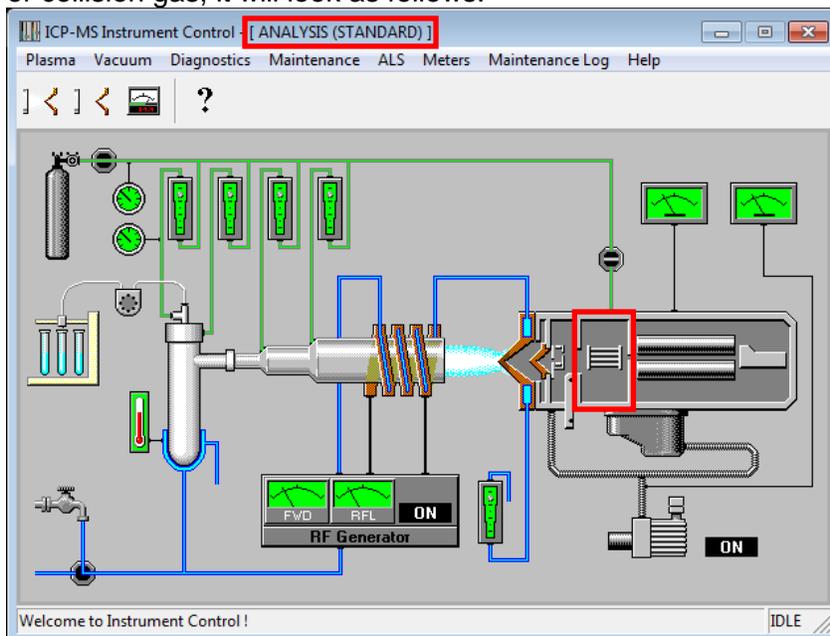
From the instrument control window, we have the following:

- 1) The plasma menu where you can ignite or shut off the plasma
- 2) The plasma ignite icon
- 3) The plasma shut-off icon
- 4) The gas flow meters
- 5) The analyzer (left) and backing (right) pressure gauges
- 6) The RF generator meters (left) and the water flow meter (right)
- 7) The spray chamber temperature gauge

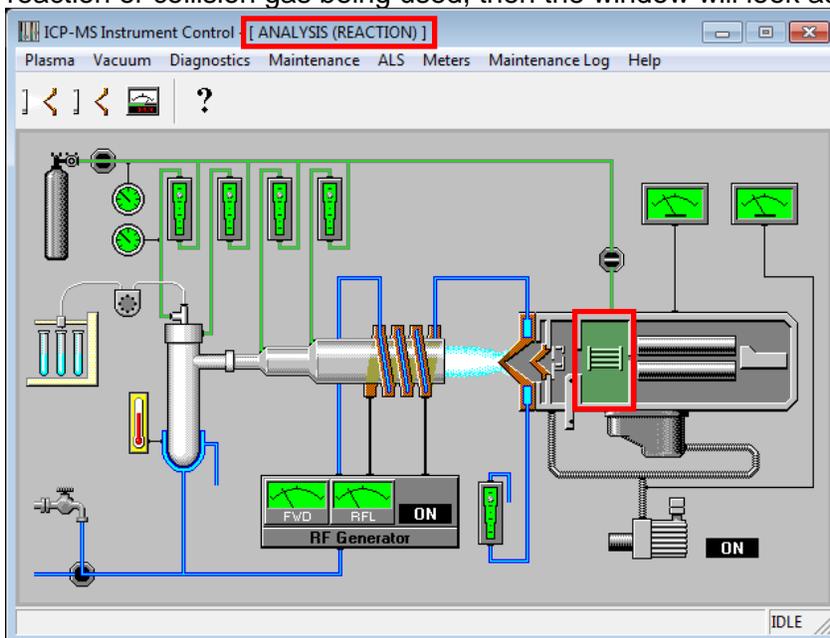
Notes:

- to view a meter value, click and hold the meter icon on the diagram.
- the title bar says that the instrument is currently in standby mode. This will indicate the current status of the instrument.
- to light the plasma, see [Section 4. Igniting the Plasma.](#)

- When the plasma is lit, the instrument control window will look one of two ways. If there is **no** reaction or collision gas, it will look as follows:



- If there is a reaction or collision gas being used, then the window will look as follows:

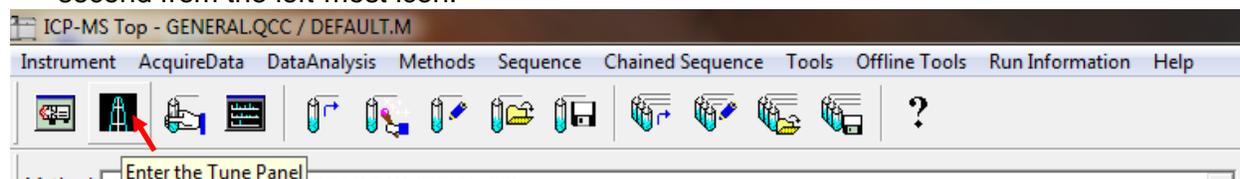


Notes:

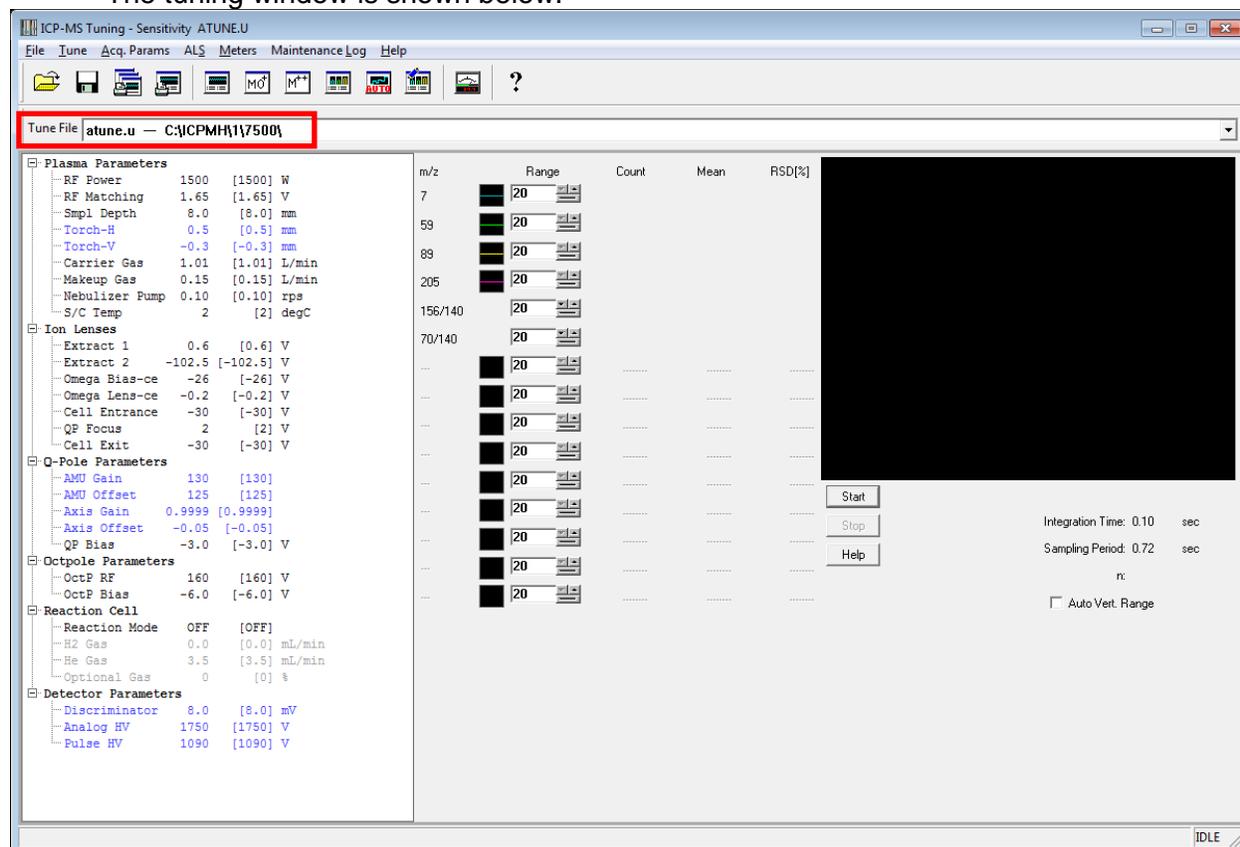
- On both windows, the title bar says it is in analysis mode. You must wait for it to say this during ignition before you will be able to access the tuning panel. You can also access the tuning panel while in standby mode. It is recommended to select a tune file before igniting the plasma.
- The instrument status may show it is standard or reaction mode upon ignition regardless of what mode the user intends to run in. This is due to the mode the last user ran the instrument in. As long as the correct tuning file is chosen before or after ignition, the instrument will operate in the correct mode. See [Section 10.1. Selecting a Tune File](#) for more information.

6.4. Tuning Panel

- To enter the tune panel, go through the instrument menu on the menu bar or click the second from the left-most icon:



- The tuning window is shown below:



The menu bar of the tune window has the following options the user can access:

- 1) **File**-load and save a desired tune file
 - 2) **Tune**-perform autotune and P/A Factor tuning.
 - 3) **Acq. Params**-change which masses are being monitored in a sensitivity tune
 - 4) **ALS**-control the autosampler (liquids)
 - 5) The rest are not typically used.
- The Tune File box below the menu icons shows the current tune parameter file.
 - The white area on the left displays the current instrument parameters.
 - The area to the right of the tune parameters is the sensitivity tuning section.

We generally do not allow student users to tune the instrument. This is for informational purposes only.

Notes: - The autune.u file is loaded by default. This file is the one that the instrument reads to run an experiment. The file parameters are equivalent to the parameters the user had loaded when the tune panel was closed last.

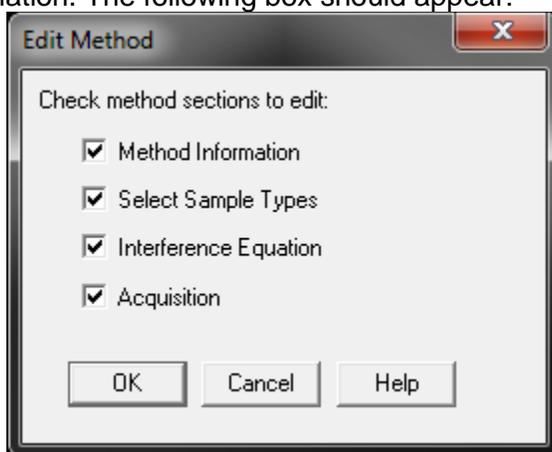
7. Creating/Editing a Method

The creation of the method file is perhaps the most important part of the analysis using ICP-MS after proper sample preparation. The method file is what tells the mass spectrometer what to measure, and it will only measure what is in the method file. In the case of sparsely available or tough to prepare samples, an error in the method could mean disaster if an isotope wasn't measured that needed to be. Take care when making a method and always double check everything!

- To edit a method, you must choose a method similar to yours to edit or start with the DEFAULT.M method. To do this, select "load" from the method dropdown menu.

Note: It is not recommended that you edit someone else's method without their permission as it is easy to overwrite their configuration.

- Start by selecting "edit entire method" from the method dropdown menu. See [Software Layout](#) for more information. The following box should appear:



- We will go through all four of the options to see what the dialogue looks like. In the future, if you are editing your own method, you can select only the options you would like to edit.
- Select OK to move on to Method Information

7.1. Method Information

Method Information

Method Comments:
This is an example comment

Save Copy of Method With Data
 Export AIA format for Agilent LC/GC
 Export Agilent LC/MSD raw data
 Export Spectrum to TSV File
 Tabulate Chart to CSV
 CPS Data Raw Data

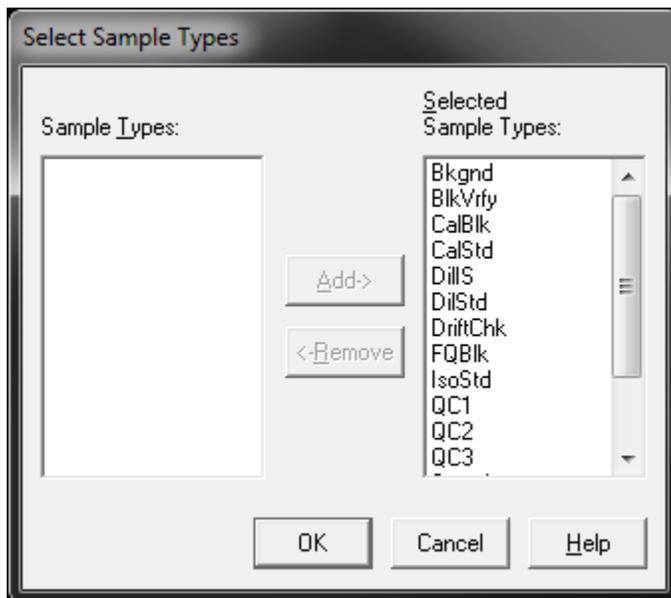
Method Sections To Run:

Pre-Run Cmd/Macro:
 Pre-Run Monitor
 Post-Run Cmd/Macro:

- The comment section of the Method Information dialogue can be read when you are loading a method and is useful for ensuring you are using the correct method.
- The two check boxes selected are optional.
 - By saving a copy of the method with the data, you will have a snapshot of your method file in case you change it in the future and don't remember what configuration was used while collecting past data.
 - The option to tabulate chart to CSV is only for users running in time resolved mode, also called time resolved analysis. This will automatically tabulate all your data in either CPS (counts per second) or in the raw format. If you are unsure of the difference contact the lab manager for more information.

Note: There have been some issues with checking the "Save Copy of Method With Data" checkbox when sampling with a sequence file. The user receives "sequence error or user terminated" error. This indicates that you should uncheck this option and try again.

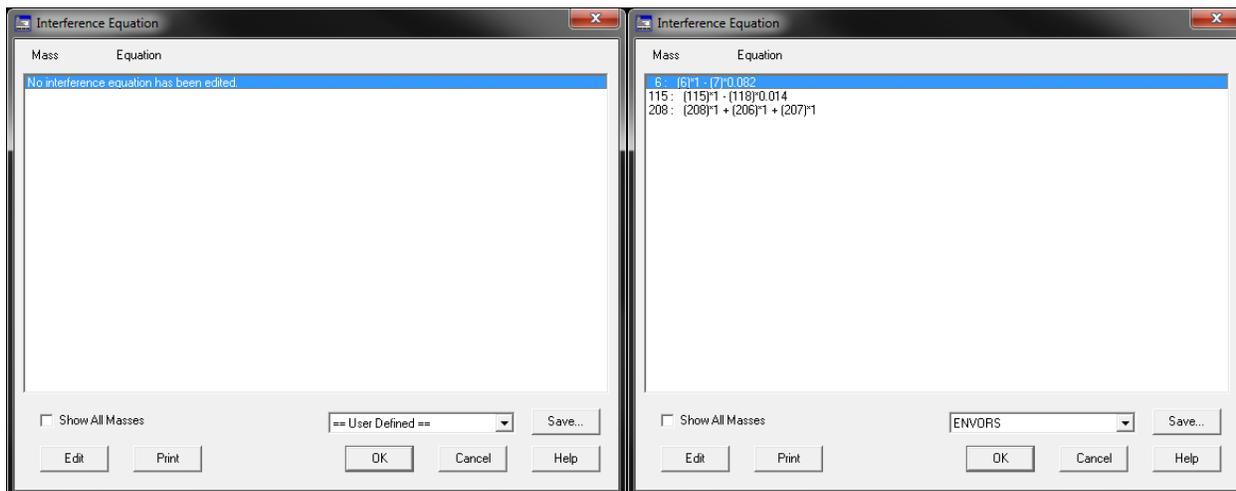
7.2. Select Sample Types



- This dialogue allows the user to select which types of samples they will be running on the instrument. Later in the document, you will see a couple different spots where the sample type must be selected. If you remove a sample type, you will not be able to define a sample as this type.
- The Select Sample Types dialogue is generally ignored. The sample type is set by the user and, therefore, having all the sample types available is not detrimental to using the instrument. This means that it is easier to have them all selected than to worry about which ones you need.

7.3. Interference Equations

Selecting interference equations is an advanced technique that is not typically used except for specific circumstances. This technique allows the user to correct for an interference that may be seen and is used for difficult isotopes such as arsenic, mercury, or lead. It requires extensive knowledge of the sample matrix and the interferences associated with the difficult isotopes of interest.



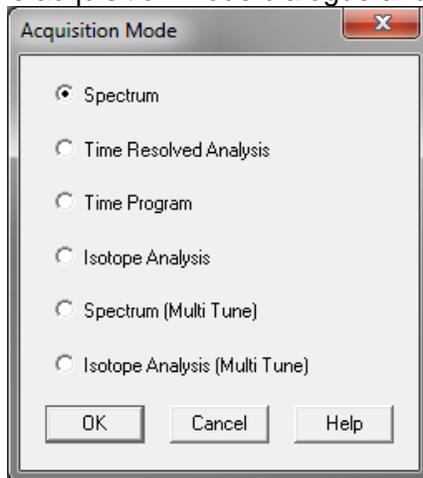
- The left side is the default interference equation dialogue. There is no equation selected in this frame. A user can use the edit function to define their own equations and save it as a preselect, or you can select from a premade one using the dropdown box.
- The box on the right shows the preselect of ENVORS. The first line is an internal standard correction for a sample high in lithium. The second is an internal standard correction for a sample high in tin. Indium (115) is used as an internal standard and indium has an overlap with tin (118) and this equation is correcting for this. The third line is a simple way to add up all the isotopes of lead to simplify the analysis.
- As you can see, the equations used are specific to each user and their samples. You should consult an experienced ICP-MS user, or the AMSEC lab manager for more help with this topic.

7.4. Acquisition

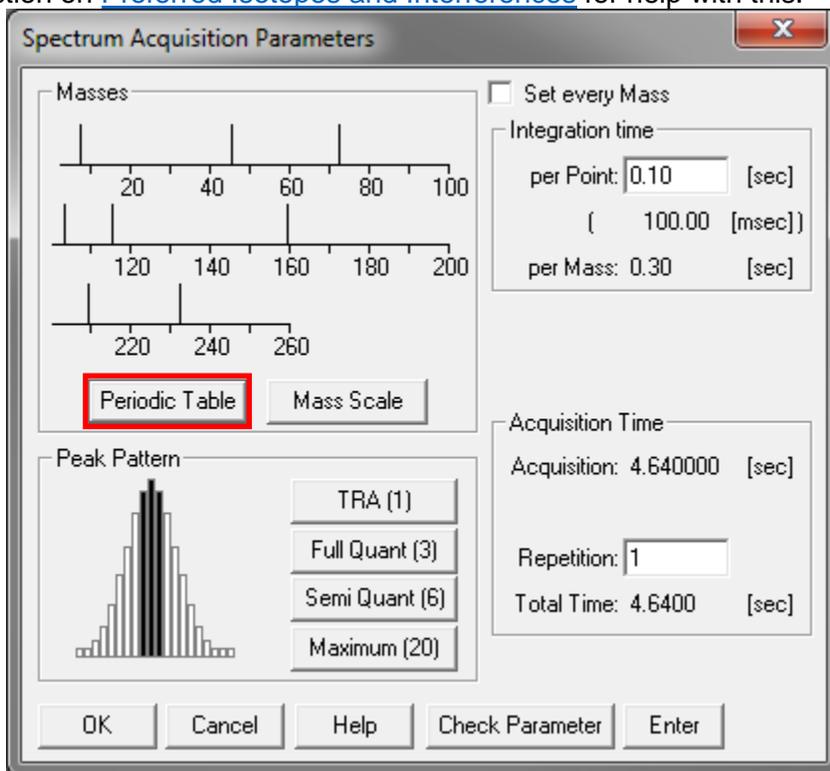
The acquisition portion of the method creation is the most important. This is where the user selects the mode that they will run in: spectrum, time resolved, or spectrum multi tune. The following windows appear after finishing with the interference equations or if acquisition is the only box checked when editing the method. We will cover each section separately.

7.4.1. Spectrum

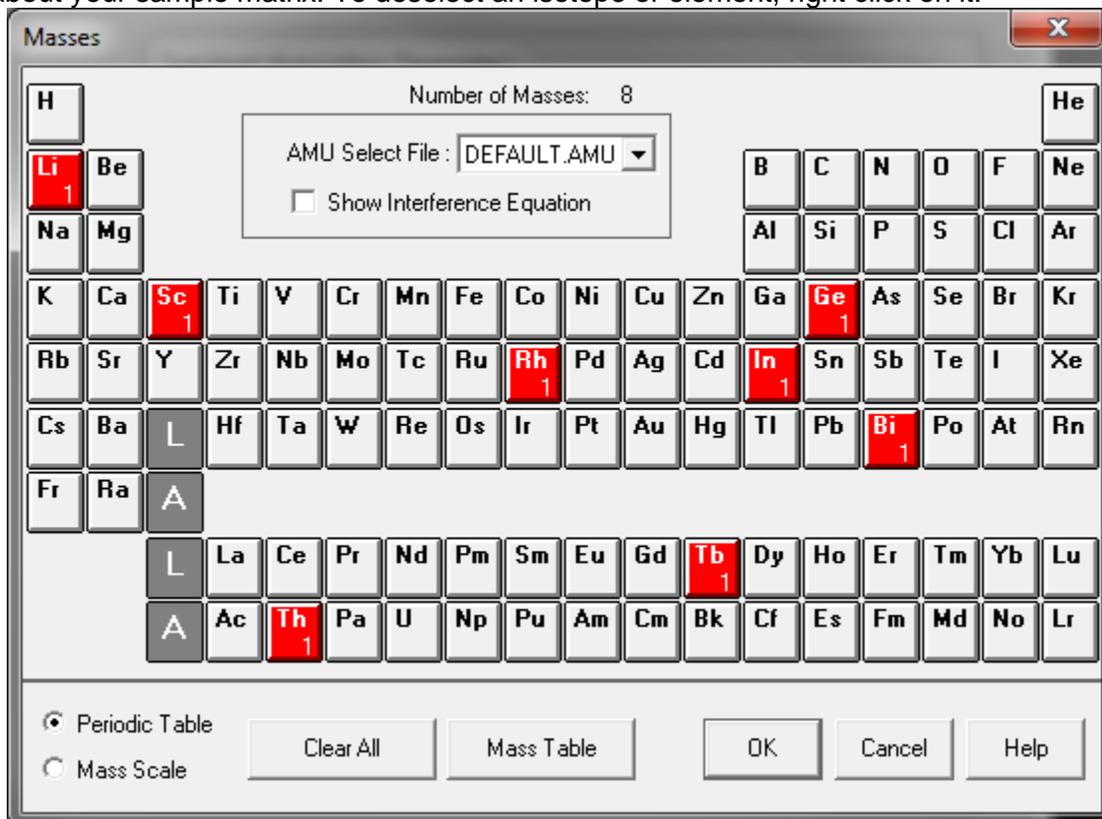
- The spectrum mode takes a single mass spectrum based on which masses are selected. Simply select spectrum in the acquisition mode dialog and press OK.



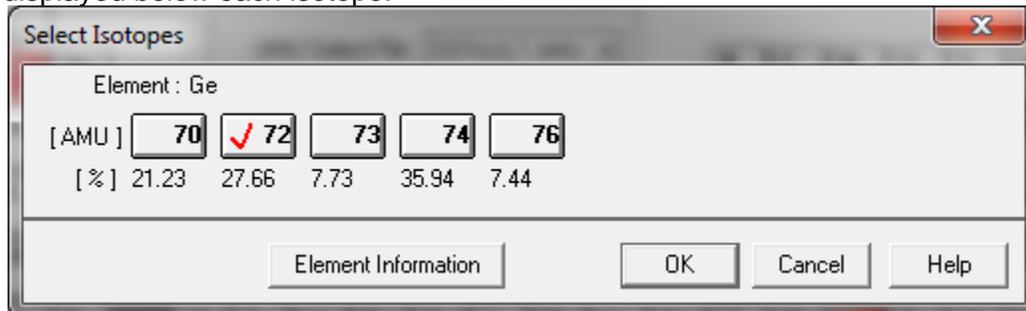
- The acquisition parameters box is displayed next. The first step is to click on the periodic table and decide which elements and which isotopes of those elements you want to analyze. See the section on [Preferred Isotopes and Interferences](#) for help with this.



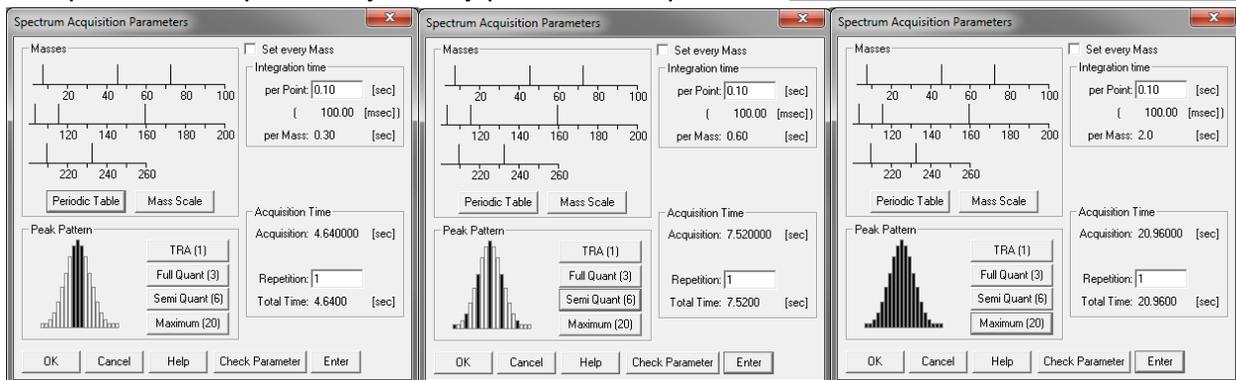
- You have the option of choosing the periodic table or the mass scale to select isotopes. The mass scale is less intuitive so we will only discuss the periodic table since they do the same thing.
- If you left click on an element, the preferred isotope will be selected. This is not always the isotope that you want to analyze. Which one you choose is based on information you have about your sample matrix. To deselect an isotope or element, right click on it.



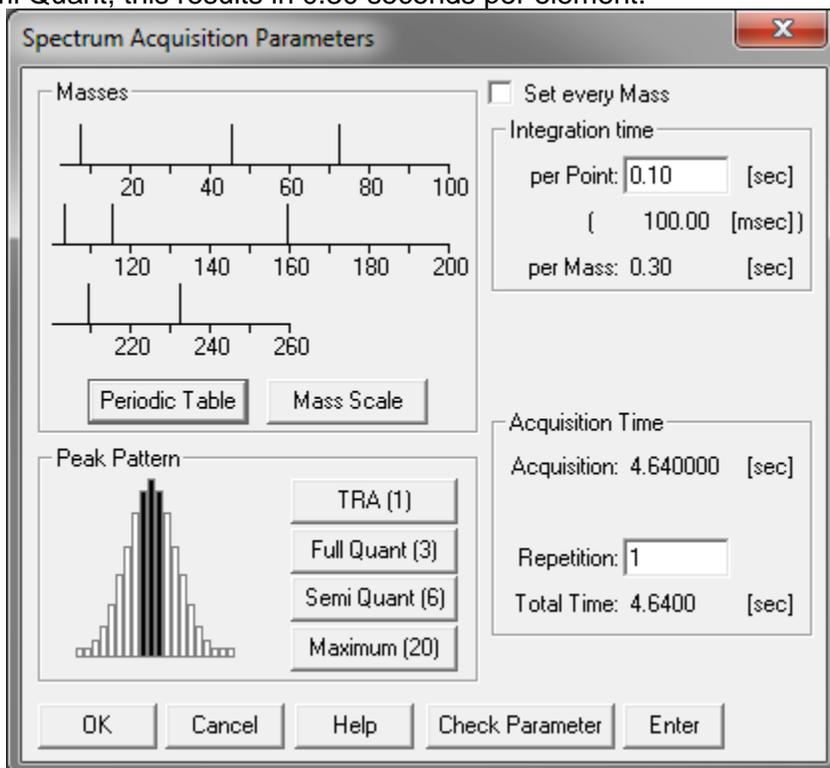
- If you want to select a specific isotope, simply double click on the element of interest. All the possible isotopes are shown. The sample below is for germanium. The natural abundances are displayed below each isotope.



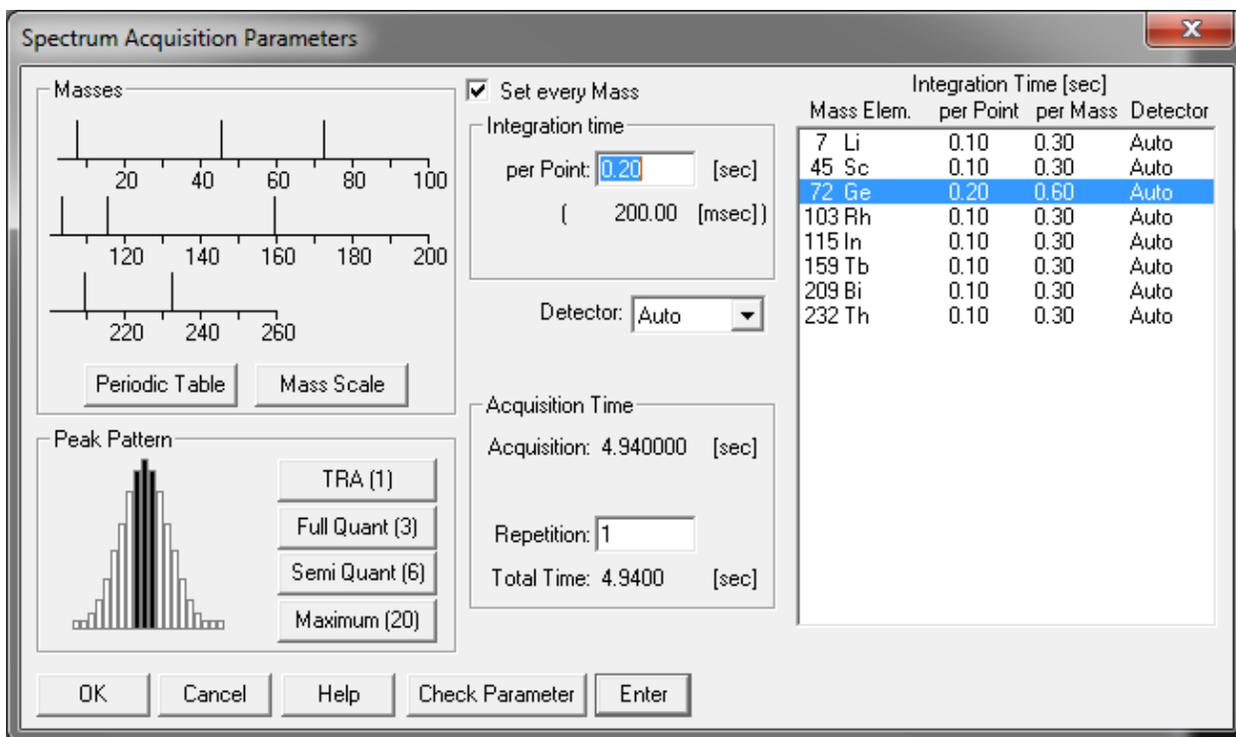
- The next step is to choose what peak pattern you want to analyze. For spectrum mode you will only want to choose from Full Quant (left), Semi Quant (center) and Maximum (right). There are advantages and disadvantages to each.
- Full Quant is the most commonly used due to a good sampling with little spectral overlap and a short sampling time. Semi Quant takes twice as long as Full Quant and is exactly what it sounds like, semi quantitative. If you are looking for quantitative data, do not choose this. The maximum takes much longer and, since it samples the entire peak, is prone to spectral overlap from any nearby peaks in the spectrum. **Semi Quant is NOT recommended.**



- After selecting the peak pattern, you must set the integration time for the elements. The minimum time is 10 msec. Typically, 100 msec is chosen as shown below. Since we have chosen Semi Quant, this results in 0.30 seconds per element.



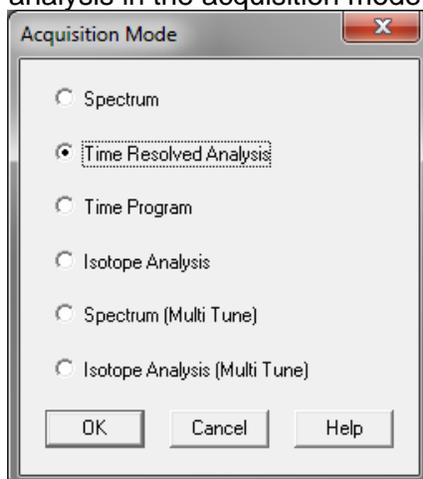
- Under acquisition time, we must now choose how many replicates we want to perform. One acquisition will take us 4.64 sec with 8 masses chosen. You will want 3-5 repetitions to get a data set appropriate for statistical analysis, although some users do more. Five repetitions are recommended.



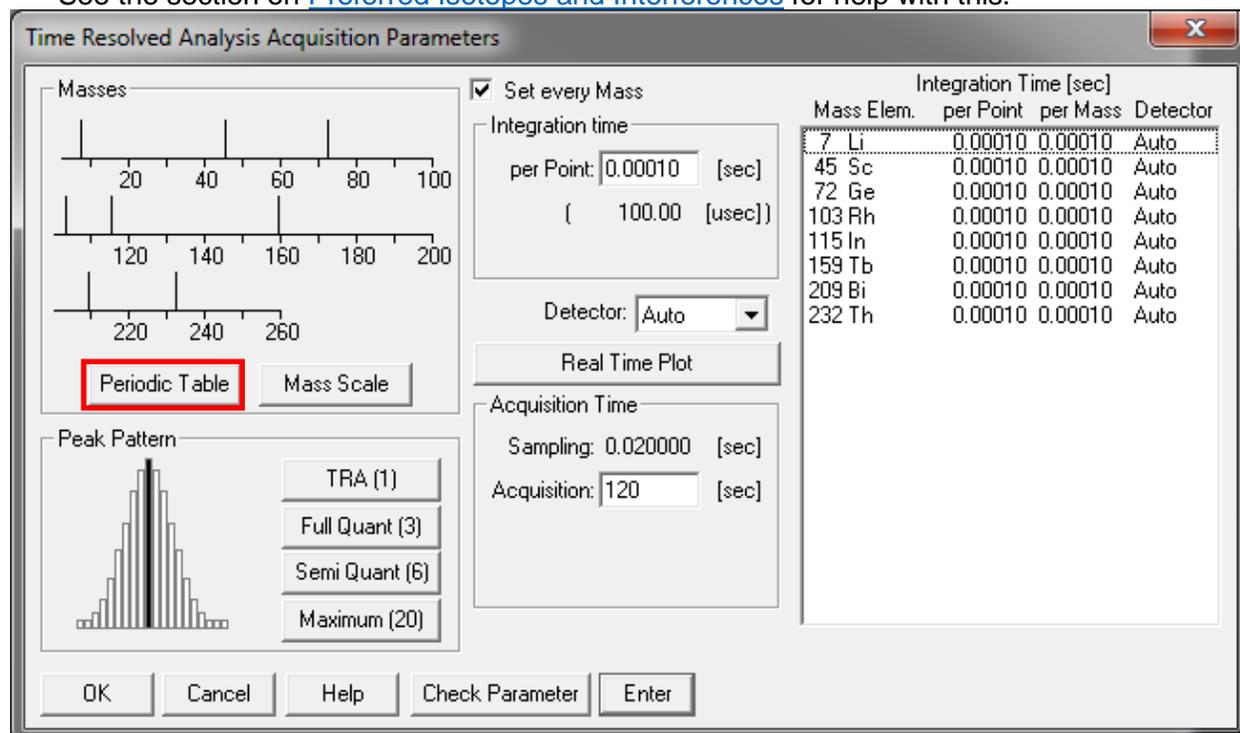
- By checking the “Set every Mass” box, the dialogue will extend to the right. This allows you to set individual masses with different integration times if necessary. This may be useful for difficult masses that have either low or noisy counts.
- **For liquid ICPMS using the internal standards you must always acquire ^7Li ^{45}Sc ^{72}Ge ^{103}Rh ^{115}In ^{159}Tb ^{209}Bi and ^{232}Th at 0.10 sec integration. If you forget to select these in the periodic table, your data will not be quantifiable.**
- Click OK. If performing liquid analyses go to the section [Peristaltic Pump Program](#), if performing solid analyses, go to the section [Saving a Method](#).

7.4.2. Time Resolved Analysis

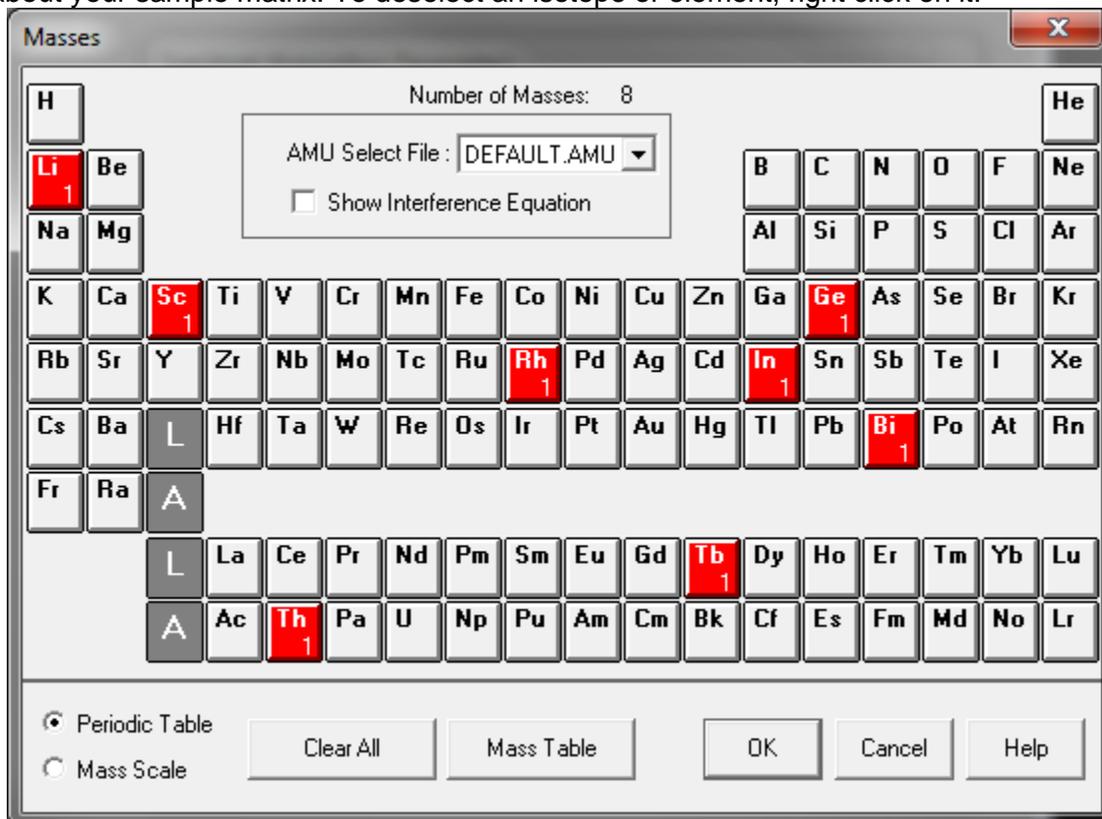
- The Time Resolved Analysis (TRA) monitors selected masses as a function of time. You can set which masses you want to view on the screen (up to 12) and which masses you want to collect data for overall (any amount).
- The spectrum mode takes a single mass spectrum based on which masses are selected. Simply select time resolved analysis in the acquisition mode dialogue and press OK.



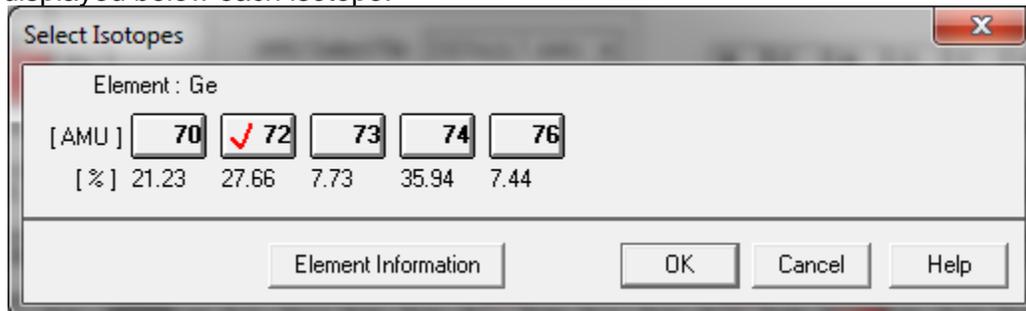
- The acquisition parameters box is displayed next. The first step is to click on the periodic table and decide which elements and which isotopes of those elements you want to analyze. See the section on [Preferred Isotopes and Interferences](#) for help with this.



- You have the option of choosing the periodic table or the mass scale to select isotopes. The mass scale is less intuitive so we will only discuss the periodic table since they do the same thing.
- If you left click on an element, the preferred isotope will be selected. This is not always the isotope that you want to analyze. Which one you choose is based on information you have about your sample matrix. To deselect an isotope or element, right click on it.



- If you want to select a specific isotope, simply double click on the element of interest. All the possible isotopes are shown. The sample below is for germanium. The natural abundances are displayed below each isotope.



- For the TRA you must make sure to select TRA in the Peak Pattern section of the parameter dialogue box.
- After selecting the peak pattern, you must set the integration time for the elements. The minimum time is 100 μ sec. Typically, 1 msec is chosen as shown below. You can see the current sampling time in the acquisition time section.
- It is important to keep the integration time as small as possible unless you have an element of low signal, in which case you can click on it and set that mass specifically as seen below for germanium. If germanium was giving low and noisy counts, you can increase the integration time to help with this. This is specific to each user's sample matrix.

Time Resolved Analysis Acquisition Parameters

Set every Mass

Integration time
per Point: [sec]
([msec])

Detector:

Real Time Plot

Acquisition Time
Sampling: [sec]
Acquisition: [sec]

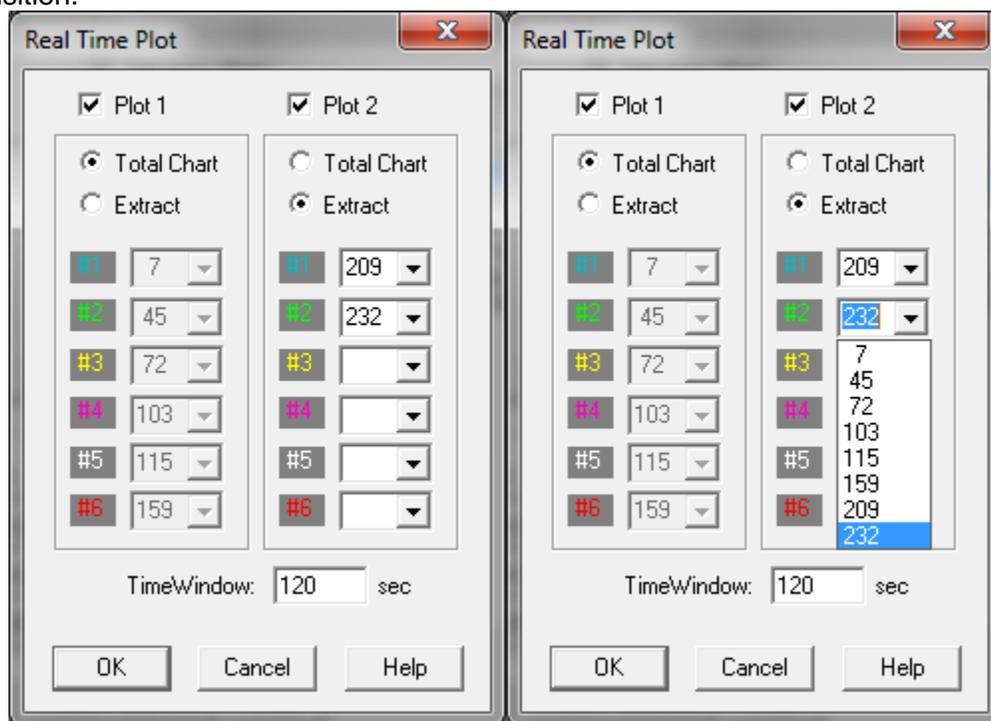
Mass Elem.	per Point	per Mass	Detector
7 Li	0.00010	0.00010	Auto
45 Sc	0.00010	0.00010	Auto
72 Ge	0.0010	0.0010	Auto
103 Rh	0.00010	0.00010	Auto
115 In	0.00010	0.00010	Auto
159 Tb	0.00010	0.00010	Auto
209 Bi	0.00010	0.00010	Auto
232 Th	0.00010	0.00010	Auto

Peak Pattern

OK Cancel Help Check Parameter Enter

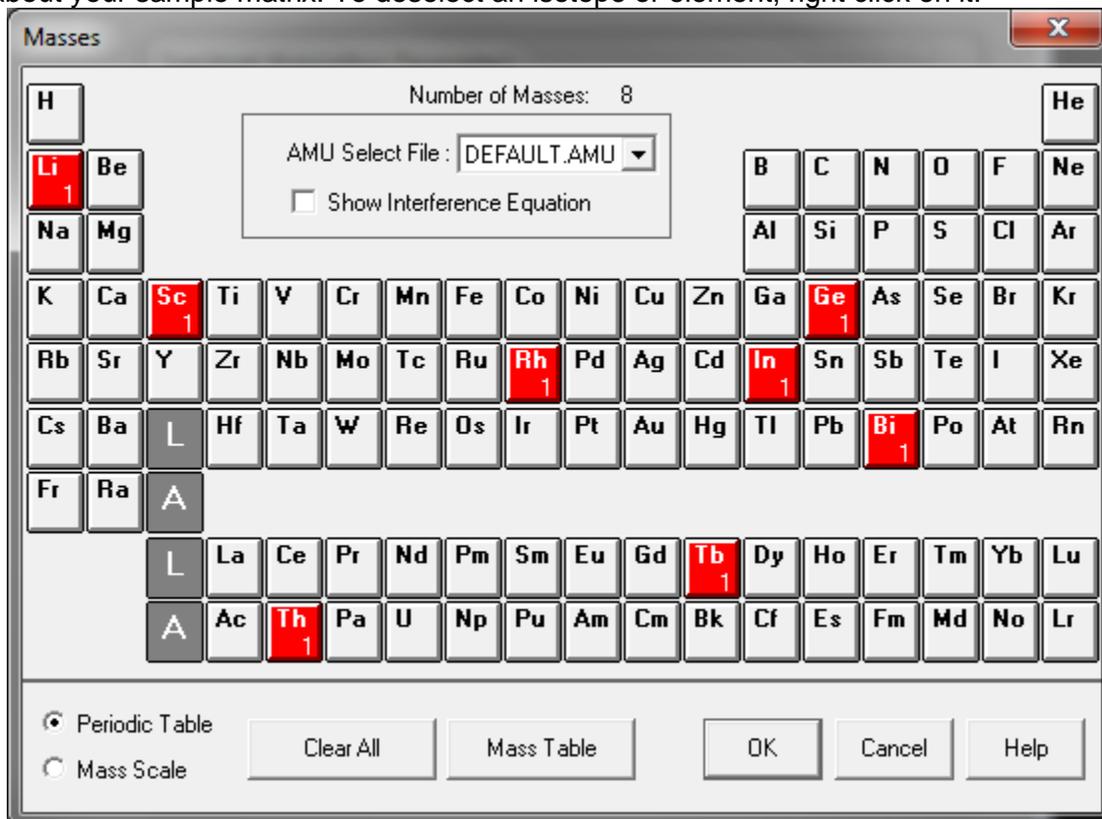
- Under acquisition time, we must now choose how long we want to acquire data. This is specific to each user's applications. Two minutes has been set as an example.

- We will now click on Real Time Plot to choose what we are monitoring on the screen during acquisition.

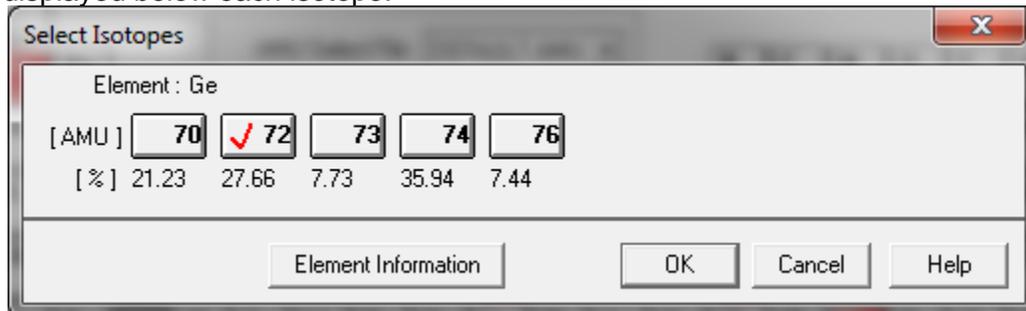


- You have two plots to view during a run. You can either view all elements summed together in one line. Or you can select six elements per plot by checking the “Extract” option and choosing from the drop down (right screenshot) which masses will be monitored.
- Typically, users select one plot with lighter masses on it and the other with heavier masses. **Regardless of which masses are selected to display, all masses selected in the periodic table will be collected in the data file.**
- Once finished, click OK on this window and the Acquisition Parameters window.
- If performing liquid analyses go to the section [Peristaltic Pump Program](#), if performing solid analyses, go to the section [Saving a Method](#).

- You have the option of choosing the periodic table or the mass scale to select isotopes. The mass scale is less intuitive so we will only discuss the periodic table since they do the same thing.
- If you left click on an element, the preferred isotope will be selected. This is not always the isotope that you want to analyze. Which one you choose is based on information you have about your sample matrix. To deselect an isotope or element, right click on it.



- If you want to select a specific isotope, simply double click on the element of interest. All the possible isotopes are shown. The sample below is for germanium. The natural abundances are displayed below each isotope.



- The next step is to choose what peak pattern you want to analyze. **Semi Quant is recommended.** For possibly using other modes talk to your research advisor or the lab manager for help.
- Select the tune files you want to use for each step using the drop-down menu
- After selecting the peak pattern, you must set the integration time for the elements. The minimum time is 10 msec. Typically, 100 msec is chosen as shown below.

Spectrum (Multi Tune) Acquisition Parameters

Masses:

Integration time: **per Point: --- [sec]**

Detector: Auto

Peak Pattern:

Acquisition Time:
 Repetition: 5
 Total Time: 160 [sec]

Mass Elem.	Detector	Step 1		Step 2		Step 3	
		per Point	per Mass	per Point	per Mass	per Point	per Mass
7 Li	Auto	0.10	0.30	0.10	0.30	0.10	0.30
45 Sc	Auto	0.10	0.30	0.10	0.30	0.10	0.30
72 Ge	Auto	0.10	0.30	0.10	0.30	0.10	0.30
103 Rh	Auto	0.10	0.30	0.10	0.30	0.10	0.30
115 In	Auto	0.10	0.30	0.10	0.30	0.10	0.30
159 Tb	Auto	0.10	0.30	0.10	0.30	0.10	0.30
209 Bi	Auto	0.10	0.30	0.10	0.30	0.10	0.30
232 Th	Auto	0.10	0.30	0.10	0.30	0.10	0.30

Enter

Return to First Tune Step

- The important thing here is knowing which elements you want to analyze in which mode, or you can do all of them in all modes. This can be risky since it will use a lot of sample. To only select certain elements for certain modes, you will want to click on that element in that specific step and alter the integration time.
- To remove an element from a step, click on the element and change the integration time to "--" and press enter. Be sure you are entering the integration time where the red box is located on the above image and to the left.
- It is important for each step to select a suitable stabilization time. This is the amount of time that the instrument is allowed to equilibrate at a new tune mode before acquiring data. A recommended time is 30 seconds for each. Shorter times may be selected after some experimentation.

- Under acquisition time, we must now choose how many replicates we want to perform. You will want 3-5 repetitions to get a data set appropriate for statistical analysis, although some users do more. Five repetitions are recommended. Be sure to pay attention to the total amount of time required for each sample. **This will be in addition to rinse times for liquid users.**

Spectrum (Multi Tune) Acquisition Parameters

Masses: 20, 40, 60, 80, 100, 120, 140, 160, 180, 200, 220, 240, 260

Integration time: per Point: --- [sec], (--- [sec])

Detector: Auto

Peak Pattern: TRA (1), Full Quant (3), Semi Quant (6), Maximum (20)

Acquisition Time: Repetition: 5, Total Time: 160 [sec]

Tune File: Step 1: nogas.u, Step 2: he.u, Step 3: h2.u

Stabilization Time: Step 1: 30 [sec], Step 2: 30 [sec], Step 3: 30 [sec]

Mass Elem.	Detector	Integ Time[sec]		Integ Time[sec]		Integ Time[sec]	
		per Point	per Mass	per Point	per Mass	per Point	per Mass
7 Li	Auto	0.10	0.30	0.10	0.30	0.10	0.30
45 Sc	Auto	0.10	0.30	0.10	0.30	0.10	0.30
72 Ge	Auto	0.10	0.30	0.10	0.30	0.10	0.30
103 Rh	Auto	0.10	0.30	0.10	0.30	0.10	0.30
115 In	Auto	0.10	0.30	0.10	0.30	0.10	0.30
159 Tb	Auto	0.10	0.30	0.10	0.30	0.10	0.30
209 Bi	Auto	0.10	0.30	0.10	0.30	0.10	0.30
232 Th	Auto	0.10	0.30	0.10	0.30	0.10	0.30

Enter

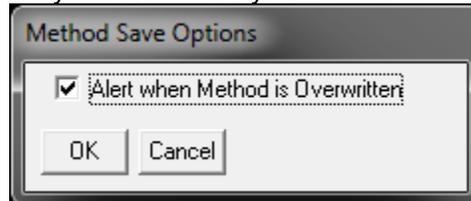
Return to First Tune Step

OK Cancel Help Check Parameter

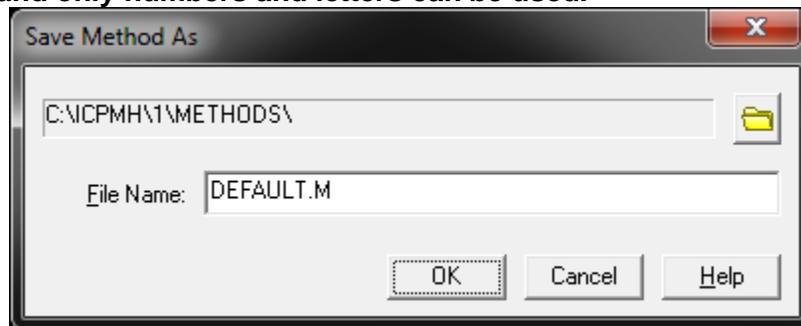
- For liquid ICPMS using the internal standards you must always acquire ${}^7\text{Li}$ ${}^{45}\text{Sc}$ ${}^{72}\text{Ge}$ ${}^{103}\text{Rh}$ ${}^{115}\text{In}$ ${}^{159}\text{Tb}$ ${}^{209}\text{Bi}$ and ${}^{232}\text{Th}$ at 0.10 sec integration. If you forget to select these in the periodic table, your data will not be quantifiable. Make sure to have them acquired in all tune modes where a mass depends on that internal standard.
- Click OK. If performing liquid analyses go to the section [Peristaltic Pump Program](#), if performing solid analyses, go to Section [9.6. Saving a Method](#).

7.5. Saving a Method

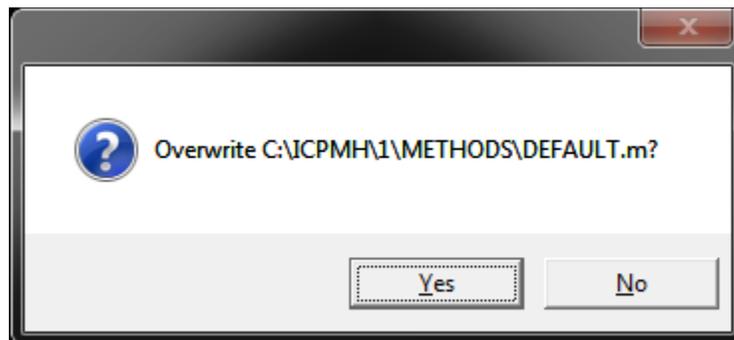
- Once you have completed any method edits you should arrive at the following dialogue.



- It is always recommended to check this box to ensure you don't accidentally overwrite something you didn't want to.
- Following the previous box is the Save Method As box. You should give your method a characteristic name such as JohnS.M or JSWater.M. **The name must never exceed 8 characters and only numbers and letters can be used.**



- There is no reason to change the directory in most cases. If you were editing your own method, simply click OK without changing the name. If you were editing another method to make your own or making a method based on another of yours, enter the new method name and click OK.



- Once you click OK in the save method box, you may be prompted with this dialogue. If you are confident you want to overwrite, click yes, if not, click no and rename the method or cancel the save.
- You have now created or edited a method successfully!

8. Sequence Files: Creating/Editing for Automated Sampling

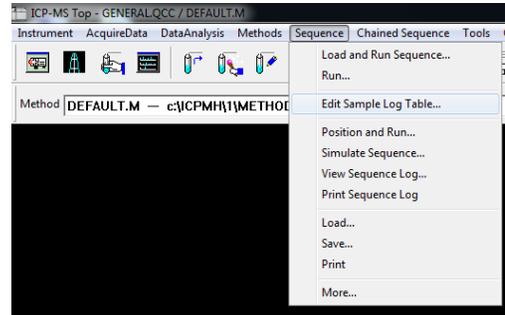
NOTE: For laser ablation users, the sequence file won't be used unless automated laser sampling will be performed. This can only be done on routine and repetitive analyses. Ask the lab manager for more info on this technique.

The large difference between a sequence file and a method file is what they are communicating with. The method file tells the mass spec what isotopes to measure and the sequence file helps the laser with timing what sample is running to make sure everything lines up with ablation and data acquisition.

- To create a sequence, you must start with the DEFAULT.S sequence. To edit one you already created, simply load it using sequence → load and follow the same procedures listed below.

Note: It is not recommended that you edit someone else's sequence without their permission as it is easy to overwrite their configuration.

- Start by selecting “edit sample log table” from the sequence dropdown menu. The following box should appear:



SMPL

	Method	Type	Vial	Data File	Sample	Comment	Dil/Lvl	ISTD Conc	Action on Failure	Skip	LC/GC Vial	Result
1	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	1203	1001	Throwaway		1.000	Level 1				
2	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	1204	1002	Throwaway		1.000	Level 1				
3	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	1101	1003	Rinse blank		1.000	Level 1				
4	C:\ICPMH\1\METHODS\DEFAULT.M	CalBk	1101	001	0		Level 1	Level 1				
5	C:\ICPMH\1\METHODS\DEFAULT.M	CalStd	1102	002	1		Level 2	Level 1				
6	C:\ICPMH\1\METHODS\DEFAULT.M	CalStd	1103	003	2		Level 3	Level 1				
7	C:\ICPMH\1\METHODS\DEFAULT.M	CalStd	1104	004	3		Level 4	Level 1				
8	C:\ICPMH\1\METHODS\DEFAULT.M	CalStd	1105	005	4		Level 5	Level 1				
9	C:\ICPMH\1\METHODS\DEFAULT.M	CalStd	1106	006	5		Level 6	Level 1				
10	C:\ICPMH\1\METHODS\DEFAULT.M	CalStd	1107	007	6		Level 7	Level 1				
11	C:\ICPMH\1\METHODS\DEFAULT.M	CalStd	1201	008	7		Level 8	Level 1				
12	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2101	009	Sample 1		1.000	Level 1				
13	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2102	010	Sample 2		1.000	Level 1				
14	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2103	011	Sample 3		1.000	Level 1				
15	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2104	012	Sample 4		1.000	Level 1				
16	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2105	013	Sample 5		1.000	Level 1				
17	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2106	014	Sample 6		1.000	Level 1				
18	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2107	015	Sample 7		1.000	Level 1				
19	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2108	016	Sample 8		1.000	Level 1				
20	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2109	017	Sample 9		1.000	Level 1				
21	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2110	018	Sample 10		1.000	Level 1				
22	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2111	019	Sample 11		1.000	Level 1				
23	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2112	020	Sample 12		1.000	Level 1				
24	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2201	021	Sample 13		1.000	Level 1				
25	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2202	022	Sample 14		1.000	Level 1				
26	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2203	023	Sample 15		1.000	Level 1				
27	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2204	024	Sample 16		1.000	Level 1				
28	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2205	025	Sample 17		1.000	Level 1				
29	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2206	026	Sample 18		1.000	Level 1				
30	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2207	027	Sample 19		1.000	Level 1				
31	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2208	028	Sample 20		1.000	Level 1				
32	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2209	029	Sample 21		1.000	Level 1				
33	C:\ICPMH\1\METHODS\DEFAULT.M	Sample	2210	030	Sample 22		1.000	Level 1				
34		Keyword		StandBy								
35												
36												
37												
38												
39												
40												
41												
42												

Method: C:\ICPMH\1\METHODS\DEFAULT.M

Import DA Method from Existing Batch
 DA Method Only DA method and Std Data
 Source Data Batch Directory: E:\DATA\RESEARCH\SOP\FIELD\CWA\CWAgNP2_batch.xml [Browse]
 [Print] [OK] [Cancel] [Help]

- The important features of this window are as follows:
 - 1) Method column
 - 2) Type column
 - 3) Vial column
 - 4) Data file column
 - 5) Sample column
 - 6) Dil/Lvl column
 - 7) ISTD Conc column
- The next pages will discuss the importance and functions of each column type.

8.1. Method Column

Edit Sample Log Table - DEFAULT.s

SMPL

	Method	Type	Vial	Data File	Sample	Comment	Dil/Lvl	ISTD Conc
1	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	1203	1001	Throwaway		1.000	Level 1
2	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	1204	1002	Throwaway		1.000	Level 1
3	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	1101	1003	Rinse blank		1.000	Level 1
4	C:\NCPMH\1\METHODS\DEFAULT.M	CalBlk	1101	001	0		Level 1	Level 1
5	C:\NCPMH\1\METHODS\DEFAULT.M	CalStd	1102	002	1		Level 2	Level 1
6	C:\NCPMH\1\METHODS\DEFAULT.M	CalStd	1103	003	2		Level 3	Level 1
7	C:\NCPMH\1\METHODS\DEFAULT.M	CalStd	1104	004	3		Level 4	Level 1
8	C:\NCPMH\1\METHODS\DEFAULT.M	CalStd	1105	005	4		Level 5	Level 1
9	C:\NCPMH\1\METHODS\DEFAULT.M	CalStd	1106	006	5		Level 6	Level 1
10	C:\NCPMH\1\METHODS\DEFAULT.M	CalStd	1107	007	6		Level 7	Level 1
11	C:\NCPMH\1\METHODS\DEFAULT.M	CalStd	1201	008	7		Level 8	Level 1
12	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2101	009	Sample 1		1.000	Level 1
13	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2102	010	Sample 2		1.000	Level 1
14	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2103	011	Sample 3		1.000	Level 1
15	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2104	012	Sample 4		1.000	Level 1
16	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2105	013	Sample 5		1.000	Level 1
17	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2106	014	Sample 6		1.000	Level 1
18	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2107	015	Sample 7		1.000	Level 1
19	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2108	016	Sample 8		1.000	Level 1
20	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2109	017	Sample 9		1.000	Level 1
21	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2110	018	Sample 10		1.000	Level 1
22	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2111	019	Sample 11		1.000	Level 1
23	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2112	020	Sample 12		1.000	Level 1
24	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2201	021	Sample 13		1.000	Level 1
25	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2202	022	Sample 14		1.000	Level 1
26	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2203	023	Sample 15		1.000	Level 1
27	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2204	024	Sample 16		1.000	Level 1
28	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2205	025	Sample 17		1.000	Level 1
29	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2206	026	Sample 18		1.000	Level 1
30	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2207	027	Sample 19		1.000	Level 1
31	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2208	028	Sample 20		1.000	Level 1
32	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2209	029	Sample 21		1.000	Level 1
33	C:\NCPMH\1\METHODS\DEFAULT.M	Sample	2210	030	Sample 22		1.000	Level 1
34		Keyword		Standard				

- The method column tells the instrument which method to run the sample with. This is regardless of which method you have loaded already so you must make sure to put your method in here. To learn how to make or edit a method, see [Section 7](#).
- Start by double clicking the first cell under method. A dialogue will appear where you can select your method.
- Proceed to copy and paste this cell into all method cells where a sample or calibration standard will be present. (This may be done after the following steps.)
- Different method files can be ran for different samples.

8.2. Type Column

- The type column represents the type of samples that is going to be run through the instrument. Sample will need to be selected for each type for laser analysis.
- To choose a type, simply click on the cell and a dropdown box will appear where you can find and select said option. Once selected, the cell can be copied to other cells.
- The type column is also used to define the keyword option. This can be seen in a later screenshot.

8.3. Vial Column

- The vial column is used for liquids. For laser ablation, the vial column can be filled with a zero in each row. This can be copied and pasted once a single row has been completed.

8.4. Data File Column

- The data file column is used in conjunction with the type column with keyword selected. There are a large number of options that can be chosen, but we only use the pause and standby functions since the others aren't useful for our purposes.
- NOTE: If using a keyword, you must not put anything in any other columns besides type and data file or else the system will get confused.

For Non-Keyword Rows:

- The data file column must be filled with nonrepeating values for the sake of saving the data. You can make the sample name column have anything you want in it for naming, but the data file column must only contain nonrepeating numbers. It is recommended to use a system such as 001, 002, 003, etc.

8.5. Sample Column

- The sample column is for your use to label the sample names. This can be longer names with numbers and basic characters such as a dash or underscore.

Edit Sample Log Table - DEFAULT.S

SMPL

	Method	Type	Vial	Data File	Sample	Comment	Dil/Lvl	ISTD Conc
1	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	1203	1001	Throwaway		1.000	Level 1
2	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	1204	1002	Throwaway		1.000	Level 1
3	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	1101	1003	Rinse blank		1.000	Level 1
4	C:\ICPMHX1\METHODS\DEFAULT.M	CalBlk	1101	001	0		Level 1	Level 1
5	C:\ICPMHX1\METHODS\DEFAULT.M	CalStd	1102	002	1		Level 2	Level 1
6	C:\ICPMHX1\METHODS\DEFAULT.M	CalStd	1103	003	2		Level 3	Level 1
7	C:\ICPMHX1\METHODS\DEFAULT.M	CalStd	1104	004	3		Level 4	Level 1
8	C:\ICPMHX1\METHODS\DEFAULT.M	CalStd	1105	005	4		Level 5	Level 1
9	C:\ICPMHX1\METHODS\DEFAULT.M	CalStd	1106	006	5		Level 6	Level 1
10	C:\ICPMHX1\METHODS\DEFAULT.M	CalStd	1107	007	6		Level 7	Level 1
11	C:\ICPMHX1\METHODS\DEFAULT.M	CalStd	1201	008	7		Level 8	Level 1
12	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2101	009	Sample 1		1.000	Level 1
13	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2102	010	Sample 2		1.000	Level 1
14	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2103	011	Sample 3		1.000	Level 1
15	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2104	012	Sample 4		1.000	Level 1
16	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2105	013	Sample 5		1.000	Level 1
17	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2106	014	Sample 6		1.000	Level 1
18	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2107	015	Sample 7		1.000	Level 1
19	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2108	016	Sample 8		1.000	Level 1
20	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2109	017	Sample 9		1.000	Level 1
21	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2110	018	Sample 10		1.000	Level 1
22	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2111	019	Sample 11		1.000	Level 1
23	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2112	020	Sample 12		1.000	Level 1
24	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2201	021	Sample 13		1.000	Level 1
25	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2202	022	Sample 14		1.000	Level 1
26	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2203	023	Sample 15		1.000	Level 1
27	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2204	024	Sample 16		1.000	Level 1
28	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2205	025	Sample 17		1.000	Level 1
29	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2206	026	Sample 18		1.000	Level 1
30	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2207	027	Sample 19		1.000	Level 1
31	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2208	028	Sample 20		1.000	Level 1
32	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2209	029	Sample 21		1.000	Level 1
33	C:\ICPMHX1\METHODS\DEFAULT.M	Sample	2210	030	Sample 22		1.000	Level 1
34		Keyword		Standard				

8.6. Dil/Lvl Column

- You must make sure that this is always 1.000 for laser ablation.

8.7. ISTD Conc Column

- The ISTD Conc column must always say level one. It is easiest to copy and paste this value to subsequent cells.

8.8. Helpful Hints

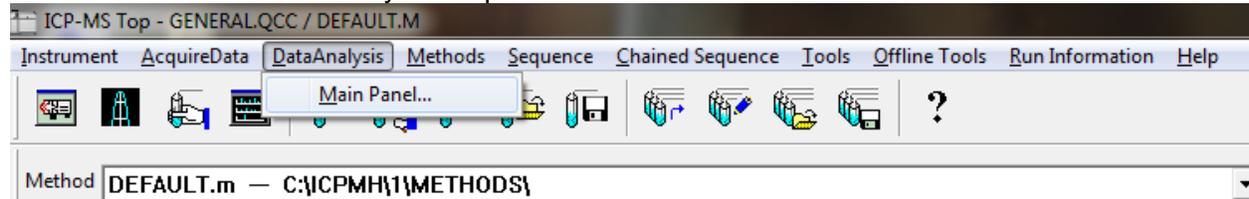
- To insert or delete entire lines, simply click on the row and then right click. You can easily insert a copied line this way.
- To learn about running a sequence, see [Section 5.6. Analyzing a Sample with a Sequence](#).

9. Data Analysis Window

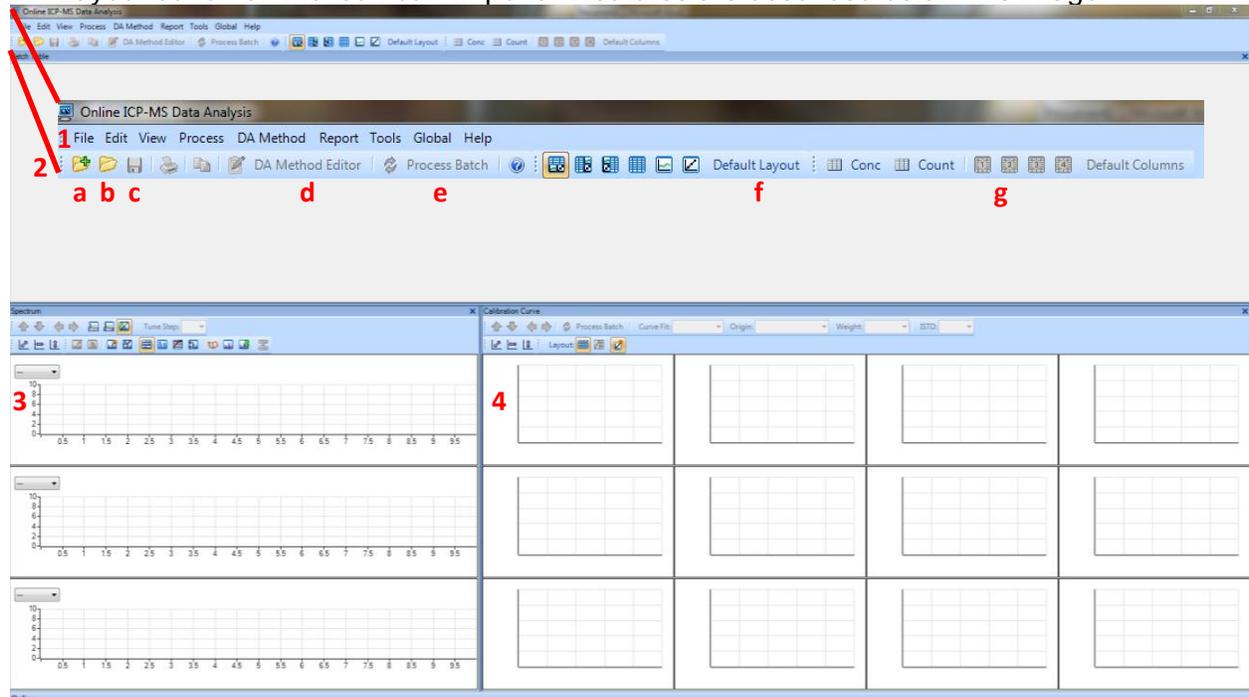
This section describes the data analysis window. This window communicates with the ICP-MS Top software and displays the data collected from the current run. There are a few slight bugs with this software. It is helpful to see [Section 13. Troubleshooting and Error Help](#).

9.1. Software Overview

- The data analysis window can be accessed from the ICP-MS Top window by selecting Main Panel from the Data Analysis dropdown menu.



- The window that opens should look like the screenshot below. If it doesn't, click the Default Layout button on the icon bar. Important features are described below this image.



Important features of the software window include:

- File Menu: save/create/load/export batches and data
- Icon Bar:
 - Create a new batch file
 - Open an existing batch file
 - Save the current batch file
 - DA Method Editor
 - Process Batch
 - Layout bar: choose default layout to return to this layout
 - Column bar: choose between display modes-concentration, count, user defined (1-4 presets) and default columns (default is in conc mode).
- Data display: displays currently selected data or ISTD stability plot.
- Calibration Curve Window: Shows calibration curves (liquids only).

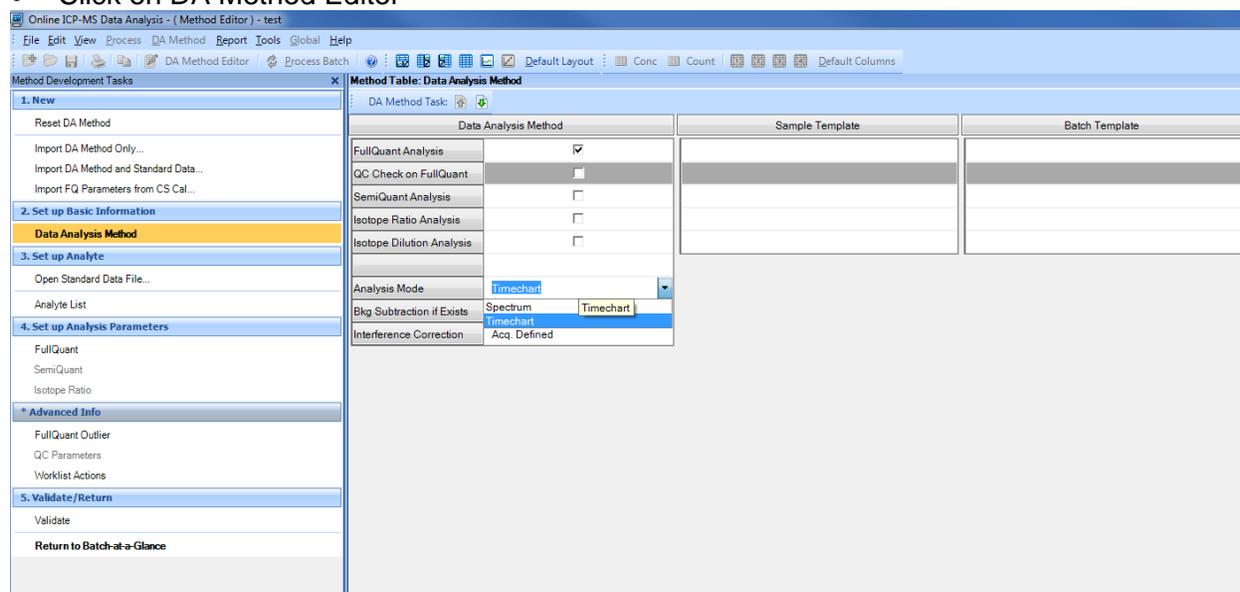
9.2. Batch Files

What is a batch file? This is a folder that is read like a file. Yes, you can even open the folder in a file browser and view the contents. This batch file will contain all the data for your standards and samples as well as your calibration curve data. If you back up this file, you will have everything you need to access the data should the file become corrupt. You should save it on the E-drive under the correct folders.

9.2.1. Creating a Batch File (Time Resolved Analysis)

For time resolved analysis with laser ablation, the Data Analysis window has less of an important function and acts mainly as an organizational tool. It also allows you to come back at any time to export data if you have lost or forgotten your files.

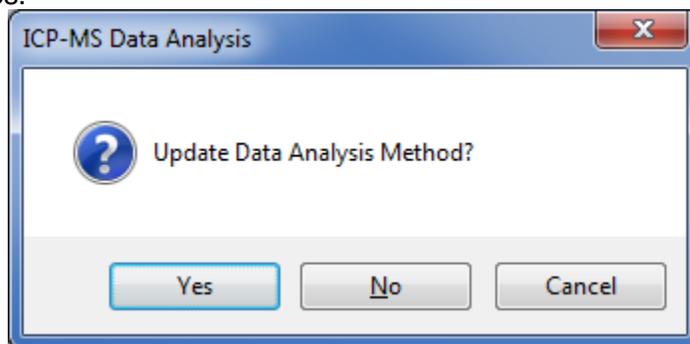
- Create a new batch folder and save it in your folder (E:\Data\Research or WWU Classes)
- Click on DA Method Editor



- Change the analysis mode from Spectrum to Timechart. Then click on Analyte List on the left-hand side.

The analyst list tells the software what elements you are collecting data for. It will only show data for elements in this list. To populate the list, click Load List From Acquisition Method. Select the method you are using to collect this data and click open. The list should now be populated.

- Next, click on **Return to Batch-at-a-Glance**. A dialogue will appear asking to update the DA Method. Click Yes.

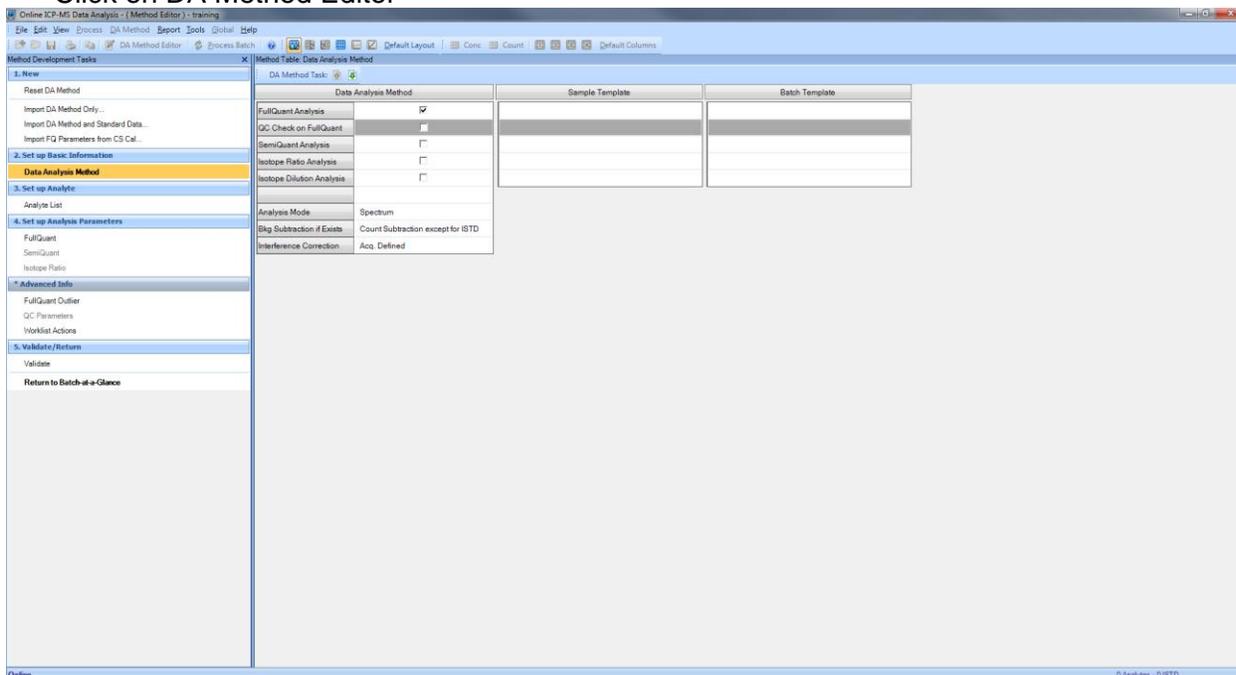


- You have now created a batch file to collect data with. Be sure to save the file before collecting data as ICP-MS Top won't collect with an unsaved batch file.

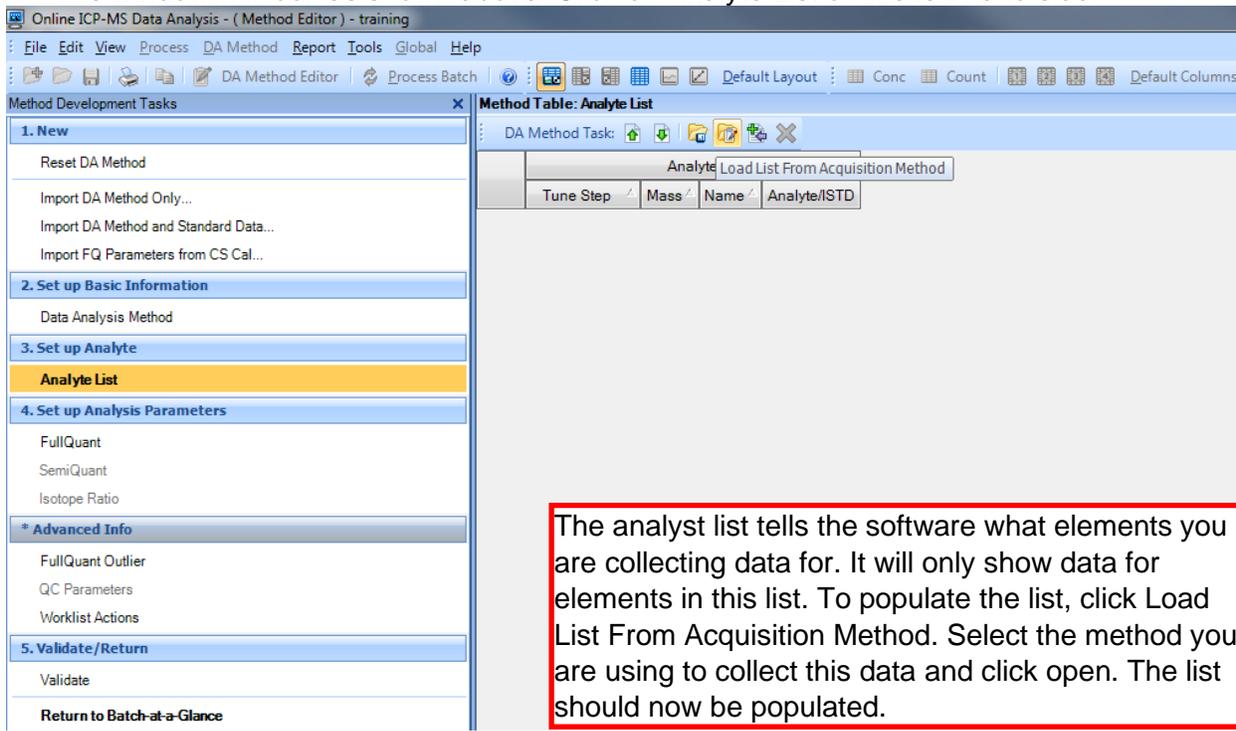
9.4.2. Creating a Batch File (Spectrum)

For spectrum analysis of laser ablation samples, the Data Analysis window has less of an important function and acts mainly as an organizational tool. It also allows you to come back at any time to export data if you have lost or forgotten your files.

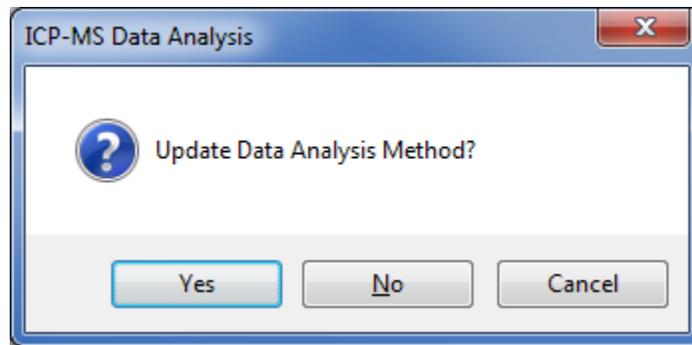
- Create a new batch folder and save it in your folder (E:\Data\Research or WWU Classes)
- Click on DA Method Editor



- The window will look as shown above. Click on Analyte List on the left-hand side.



- Next, click on **Return to Batch-at-a-Glance**. A dialogue will appear asking to update the DA Method. Click Yes.



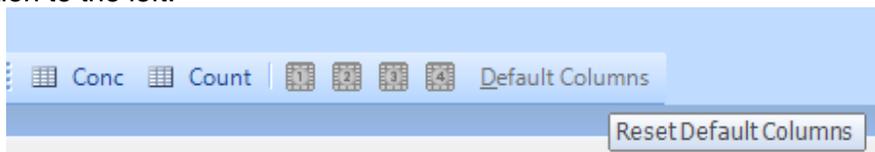
- You have now created a batch file to collect data with. Be sure to save the file before collecting data as ICP-MS Top won't collect with an unsaved batch file.

9.5. Exporting and Viewing Data

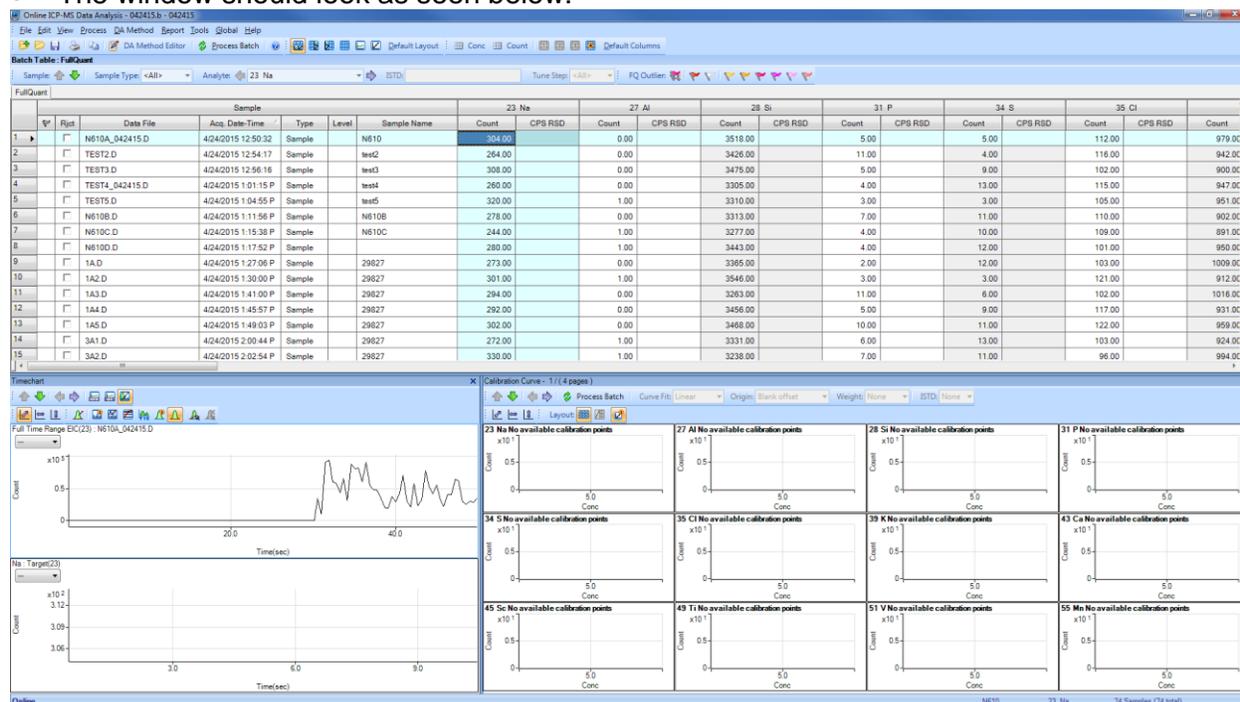
This may not be necessary if you chose the option to tabulate chart to CSV in your method information. If you did this, simply go to your batch folder in a file browser and search “.csv” and all of your files will show up.

9.5.1. Time Resolved Analysis

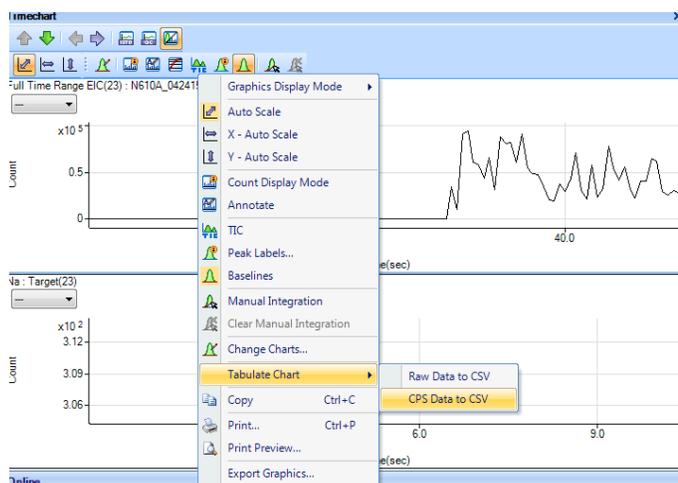
- To properly view data in time resolve analysis mode. Select Default Columns followed by the Count option to the left.



- The window should look as seen below:



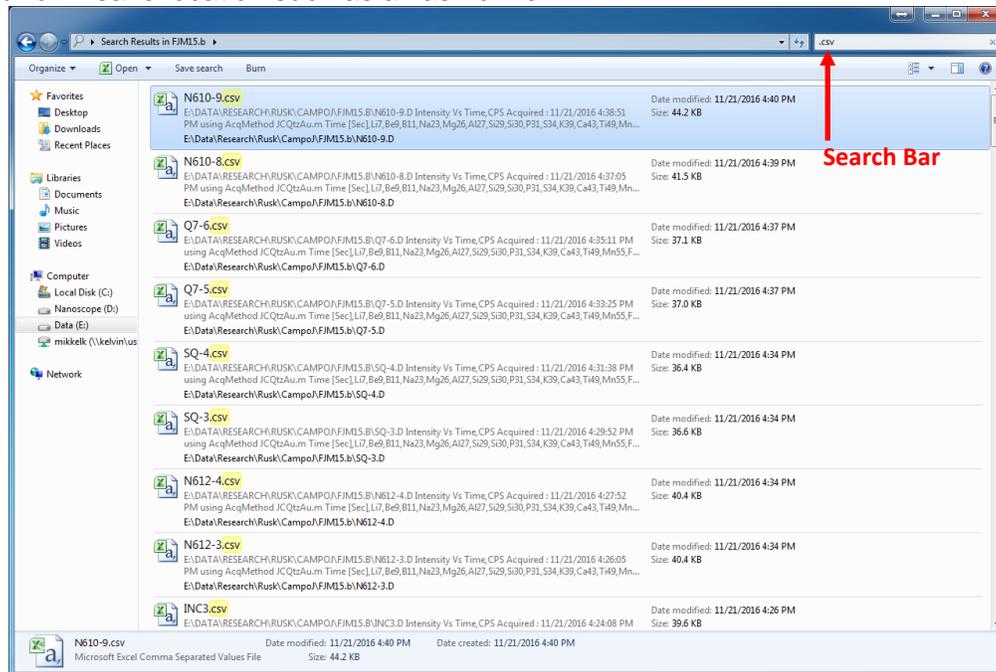
- The data shows only the first data point in the table. The actual time plot is shown on the bottom left. This window can be expanded, and you can even close the bottom right window which has no function for this analysis mode. If this isn't present, press default layout.
- To export data, highlight an entire sample by clicking on the row number. Then right click on the time chart in the bottom left pane. Choose Tabulate Chart → CPS Data to CSV.



- Once selected, an excel sheet will open with the tabulated data. A file path is shown at the top which shows where this data file is saved.

The screenshot shows an Excel spreadsheet with the following columns: Time [Sec], Na23, Al27, Si28, P31, S34, Cl35, K39, Ca43, So45, Ti49, V51, Mn55, Fe56, Zn66, Ga71, As75, Sr88, Y89, Zr90, Ba137, La139, Ce140, Pr141, Nd146, Sm147, Eu153, Gd157, Tl199. The data rows contain numerical values for each parameter across multiple samples.

- You must select each sample individually and tabulate all samples that you run. This can be done as you go or all at the end if you did not choose the option to tabulate chart to CSV in the method info.
- You can use save as to save each individual file to a location of your choosing. The other option is to extract all these files from the file browser. Simply open a file browser and navigate to your batch folder. Then, open your batch folder and use the search bar in the top right to search “.csv”. All the CSVs in that folder will show up and you can copy paste them to your own save location such as a flash drive.



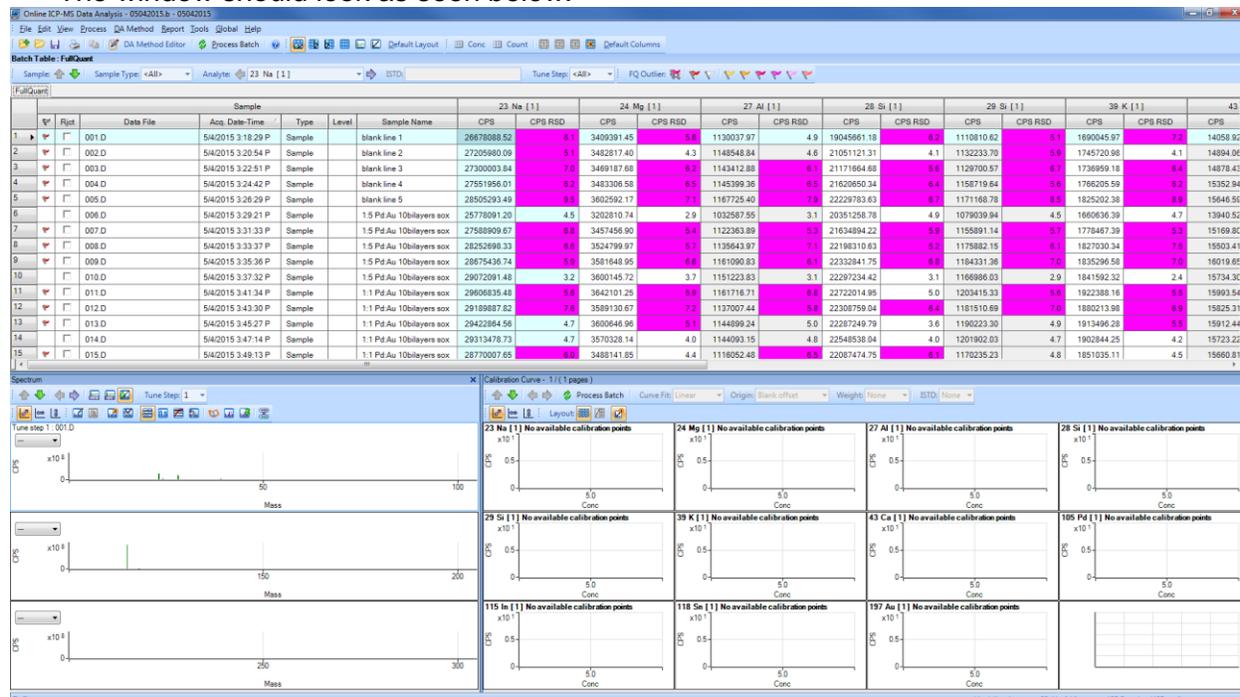
9.5.2. Spectrum Analysis

It may help to read the section on [Exporting Data for Liquids](#). This might show other useful options you may need.

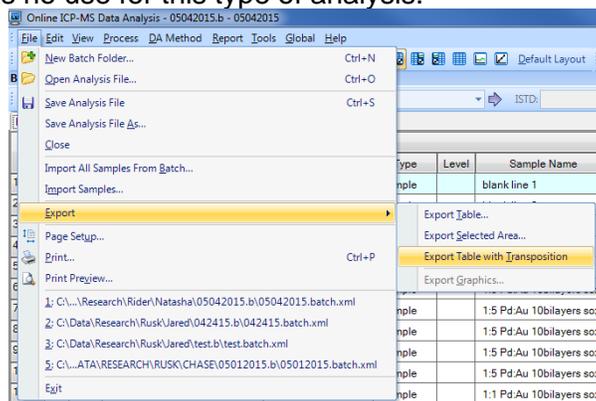
- To properly view data in spectrum mode. Select Default Columns followed by the Count option to the left.



- The window should look as seen below:



- The data is shown for each element and the actual spectrum is in the bottom left pane. The bottom right pane has no use for this type of analysis.



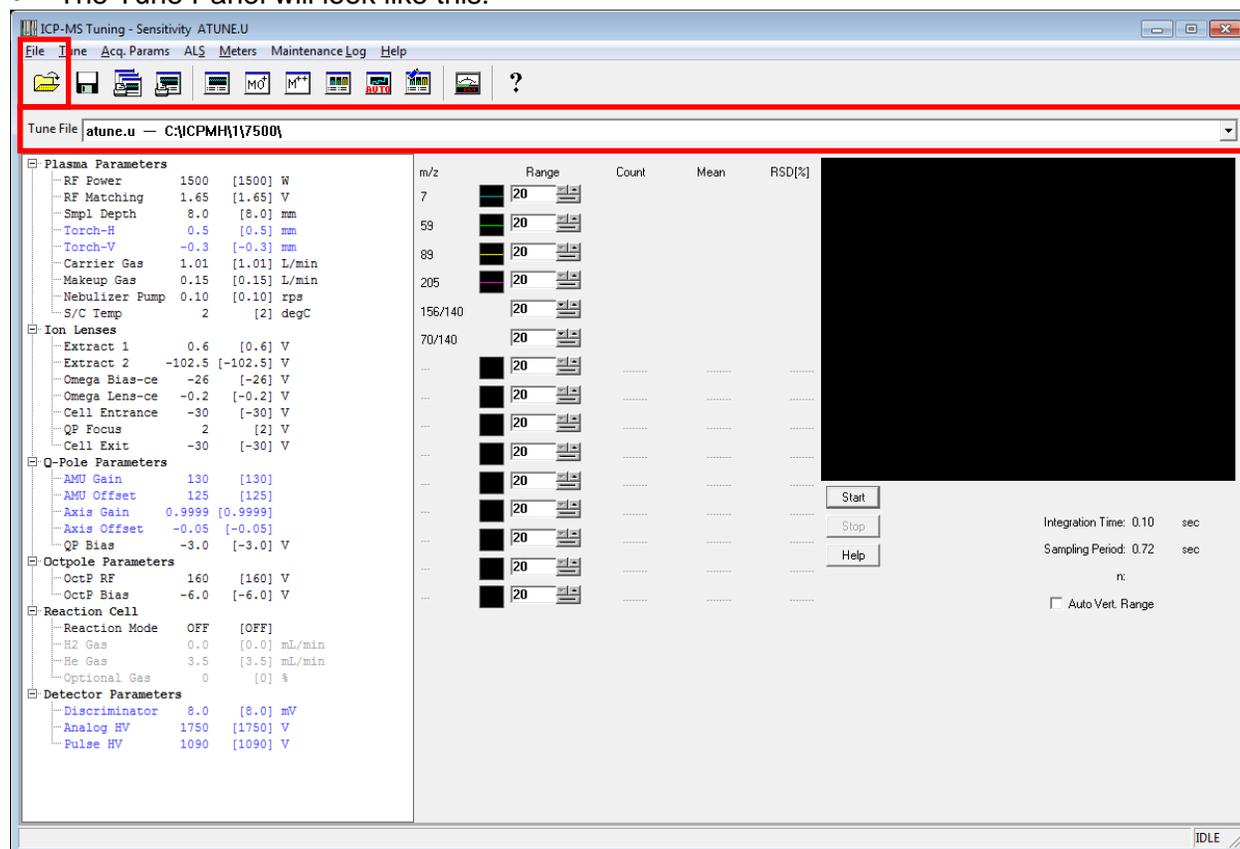
- To export, right click on the data table heading or click the File menu. Go to export and select which option you would like. Most users prefer Export Table. You can also export only selected samples and elements with Export Selected Area.
- Try out as many combinations as you want. They are all separate excel files so there is no downside to experimenting.

10. Tuning the Instrument

We do not typically allow students to tune the instrument. This is for various reasons and mainly maintains the integrity of the data you are collecting and allows for the tracking of the tune values over time to monitor changes in instrument parameters. The only exception to this is that when switching from laser to liquids mode you must do a [P/A Factor tune](#).

10.1. Selecting a Tune File

- Before running a sample, you must select the appropriate tune file for the mode you are running in. To do this, open the Tune Panel. See [Section 6. General Software Overview](#) for more information.
- The Tune Panel will look like this:



- The parameters that are displayed when you open the tune panel are the ones that were used last and it will always say atune.u. When you select a tune file and close the window, atune.u is populated with those tune values and is used by the instrument. Do not be alarmed if you had loaded a tune file and when the window reopened it said atune.u.
- To load the tune file you need, check if it is in the dropdown box or click File → Load Tune File or click the open folder. Select the proper tune file. If you are doing laser ablation, choose LATUNE.U for standard mode or select whatever tune file you need to. For liquids, select either nogas.u, he.u, or h2.u depending on what mode you are running in. For standard mode without the ORS, choose nogas.u. For helium or hydrogen mode with the ORS, select he.u or h2.u, respectively.
- For liquids, you will want to rinse the probe before starting any runs or any tuning. To do this, simply choose rinse from the ALS drop down menu. After about 30 seconds and ensuring that the rinse water has made it through the pump, choose ALS→Home.

11. Preferred Isotopes and Interferences

This section will describe a few simple explanations and examples of what isotopes are preferred and some common interferences. The periodic table on the next page is a good starting point for anyone to use but does not apply to everyone's samples or matrices. You will need to use your best judgment when choosing what isotopes to choose and how to avoid interferences. If you routinely have problems with interferences, or you aren't sure, talk to your research advisor or the lab manager for more information. Interference equations may be useful in this case.

Things to consider when choosing isotopes:

- What things will add up to the same mass as my sample? Is there a lot of this?
 - This can help you narrow down if there will even be an issue. For example, if I am worried about MgO interfering with my phosphorous 30, but there are negligible amounts of magnesium in my sample, then I can probably ignore this and use the helium ORS mode to completely remove this.
- What isotopes have overlap with other elements? Will there be a lot of this overlapping element?
 - If I am trying to study ^{118}In , but there also exists a ^{118}Sn , then I don't have to worry if there is no tin in my sample, but if there is, I may have to consider other options.
- Do I need to use helium or hydrogen in the ORS? Will this completely remove the interference, or will I still have to worry about it?
 - If the interference masses are heavily prevalent, then you cannot remove them completely. Most of the time it is easier to choose a different isotope. If this is not possible, talk to your research advisor or the lab manager for help.
- What other isotopes can I run for a selected element? Do I have many options and what are the natural abundances of these? If the abundance is extremely small, it will not be a good choice.
 - An example of this is iron. ^{56}Fe has a major interference with ArO. As those elements are very prevalent, we cannot remove this interference. We can look at the other isotopes of iron. ^{58}Fe is only 0.282% abundant and therefore is a poor choice when compared to ^{57}Fe which is 2.119% abundant. This means we should choose to analyze ^{57}Fe over ^{56}Fe for interference reasons and ^{56}Fe over ^{58}Fe for abundance reasons.

This type of questioning and reasoning should be applied to every choice of isotope you make. This can often be a lengthy process, and this is why method development is crucial to getting good results and understanding what is happening inside the instrument.

A good resource for looking at isotopes of each element with a lot of information can be found at PTable.com and then select the isotope tab. [Link](#) provided.

Acquisition Isotope Recommendations

1 H																	2 He																																																							
7 Li nogas —	6 Be nogas —	<p>Internal Standards and Masses:</p> <table border="0"> <tr> <td>Li</td><td>6</td> <td>Typical analytical mass (AMU)</td><td>66</td><td>68</td><td>Typical secondary analytical mass</td> </tr> <tr> <td>Sc</td><td>45</td> <td></td><td>Zn</td><td></td><td></td> </tr> <tr> <td>Ge</td><td>74 or 72</td> <td></td><td>He</td><td></td><td></td> </tr> <tr> <td>Rh</td><td>103</td> <td>Typical tune/ORS mode</td><td>nogas</td><td></td><td></td> </tr> <tr> <td>In</td><td>115</td> <td>Secondary tune/ORS mode</td><td>SO₂, SO₂H</td><td></td><td></td> </tr> <tr> <td>Tb</td><td>159</td> <td>Most common interferences</td><td></td><td></td><td></td> </tr> <tr> <td>Lu</td><td>175</td> <td></td><td></td><td></td><td></td> </tr> <tr> <td>Bi</td><td>209</td> <td></td><td></td><td></td><td></td> </tr> </table>																Li	6	Typical analytical mass (AMU)	66	68	Typical secondary analytical mass	Sc	45		Zn			Ge	74 or 72		He			Rh	103	Typical tune/ORS mode	nogas			In	115	Secondary tune/ORS mode	SO ₂ , SO ₂ H			Tb	159	Most common interferences				Lu	175					Bi	209					11 B nogas —	10 C	12 N	14 O	16 F	19 Ne	20 Ne
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Tb	159	Most common interferences																																																																						
Lu	175																																																																							
Bi	209																																																																							
23 Na nogas He	25 Mg nogas He C ₂ , C ₂ H																	27 Al He nogas BO, CNH	28 Si H ₂ He (HEHe) N ₂ , CO, COH	29 P He — NOH	34 S He (HEHe) — O ₂ H, O ₂	35 Cl He — O ₂ H, SH	40 Ar																																																	
39 K He H ₂ ArH	44 Ca He H ₂ (40) CO ₂ , SiO, Ar	40/43 Sc nogas He CO ₂ H, SiO	45 Ti He nogas PO, SO, Ca	47 V He nogas ClO	49/48 Cr He nogas ArC, ClOH, ArO	51 Mn He nogas KO	52 Fe He H ₂ ArO, Ni (56), ArOH	53/50 Co He nogas MgCl, CaOH	55 Ni He nogas ArO, CaO	56 Cu He nogas ArNa, PO, TiO, SO ₂ H	57/58 Zn He nogas SO ₂ , SO ₂ H	59 Ga He nogas Ba ⁺ , ArP	60 Ge He nogas Cl ₂ , ArS, KCl	62 As He nogas ArCl	63 Se He H ₂ Ar ₂ , BrH(80)	65 Br He nogas Ar ₂ H, ArK	66 Kr																																																							
85 Rb nogas He Sr(87)	87 Sr nogas He Kr(86)	86 Y nogas He	88 Zr nogas —	89 Nb nogas —	90 Mo nogas — BrO, Ru(98)	91 Tc	93 Ru nogas — Pd(102)	95 Rh nogas —	98 Pd He nogas KO	101 Ag nogas — Cd(106)	102 Cd nogas He MoO	103 In nogas He Sn(115)	105 Sn nogas —	106 Sb nogas —	107 Te nogas — Xe(128)	109 I nogas —	112 Xe																																																							
133 Cs nogas —	137 Ba nogas — La(138)	138 La nogas —	178 Hf nogas —	177 Ta nogas — HoO	181 W nogas —	182 Re nogas — Os(187)	183 Os nogas —	185 Ir nogas — LuO(191)	187 Pt nogas —	191 Au nogas —	193 Hg nogas —	194 Tl nogas —	195 Pb nogas —	196 Bi nogas —	200 Po	202 At	203 Rn																																																							
87 Fr	88 Ra	89 Ac	<table border="1"> <tr> <td>140 Ce nogas —</td> <td>142 Pr nogas —</td> <td>141 Nd nogas —</td> <td>143 Pm</td> <td>144 Sm nogas —</td> <td>145 Eu nogas —</td> <td>146 Gd nogas —</td> <td>147 Tb nogas —</td> <td>148 Dy nogas —</td> <td>149 Ho nogas —</td> <td>150 Er nogas —</td> <td>151 Tm nogas — TbO</td> <td>152 Yb nogas —</td> <td>153 Lu nogas —</td> </tr> <tr> <td>232 Th nogas —</td> <td>231 Pa</td> <td>238 U nogas —</td> <td>237 Np nogas —</td> <td>244 Pu nogas —</td> <td>242 Am</td> <td>243 Cm</td> <td>247 Bk</td> <td>247 Cf</td> <td>251 Es</td> <td>252 Fm</td> <td>257 Md</td> <td>258 No</td> <td>259 Lr</td> </tr> </table>															140 Ce nogas —	142 Pr nogas —	141 Nd nogas —	143 Pm	144 Sm nogas —	145 Eu nogas —	146 Gd nogas —	147 Tb nogas —	148 Dy nogas —	149 Ho nogas —	150 Er nogas —	151 Tm nogas — TbO	152 Yb nogas —	153 Lu nogas —	232 Th nogas —	231 Pa	238 U nogas —	237 Np nogas —	244 Pu nogas —	242 Am	243 Cm	247 Bk	247 Cf	251 Es	252 Fm	257 Md	258 No	259 Lr																											
140 Ce nogas —	142 Pr nogas —	141 Nd nogas —	143 Pm	144 Sm nogas —	145 Eu nogas —	146 Gd nogas —	147 Tb nogas —	148 Dy nogas —	149 Ho nogas —	150 Er nogas —	151 Tm nogas — TbO	152 Yb nogas —	153 Lu nogas —																																																											
232 Th nogas —	231 Pa	238 U nogas —	237 Np nogas —	244 Pu nogas —	242 Am	243 Cm	247 Bk	247 Cf	251 Es	252 Fm	257 Md	258 No	259 Lr																																																											

Tune modes and isotopes can be application specific and may not conform to the recommended setting described on this sheet. This is a typical example for common water samples. (Higher mass elements typically only have interferences from oxides of other high mass elements.)

12. Shutting Down the Instrument

12.1. Shutting Down with Liquids

To shut down the instrument follow these steps. Most of them are reverse of the setup procedure.

1. Put the ISTD tubing into the 2% Nitric bottle
2. Release the black peripump clamps and relieve the tension on the three tubes by unhooking them.
3. Send the autosampler probe home from the instrument control or tune panel. ALS → Home
4. Turn off the autosampler and release the peripump tube clamp on the back.
5. Close all the rinse bottles.
6. Shut off the plasma if not already done from the instrument control window.
Select Plasma → Plasma off
7. Turn off the argon.
8. Turn off the water chiller. Wait at least 10 minutes after turning off the plasma to do this.
(ES 128B)
9. Remove your samples and clean up any mess.
10. Notify the lab manager if anything needs to be repaired or replaced.

12.2. Shutting Down with Laser Ablation

To shut down the instrument follow the following steps. Most of them are reverse of the setup procedure.

1. Shut off the plasma from the instrument control window.
Select Plasma → Plasma off
2. Remove your sample from the laser ablation accessory.
3. Replace sample holder in laser ablation accessory.
4. Turn the He flow off in the laser ablation software.
5. Close laser ablation helium tank.
6. Shut off the argon tank.
7. Turn off key on laser ablation power supply.
8. Turn off the water chiller. Wait at least 10 minutes after turning off the plasma to do this.
(ES 128B)

13. Troubleshooting and Error Help

If you have an error or something goes wrong. Try to find a solution in the following pages or do some basic troubleshooting to determine the cause. If you can't find anything helpful and resolve the issue by yourself, contact the lab manager and they will assist with the problem.

13.1. Plasma Ignition

If the plasma won't light or shuts off, check the following:

Is the Argon tank on and is there 100 psi output shown on the top gauge of the regulator?

- If not, open the tank(s) and use the knob on the regulator to increase the pressure to 100 psi if necessary.
- Check that the valve on the far left of the regulator is open.
- Check that the liquid argon tank has sufficient internal pressure. See the section on [Liquid Argon Tank Operation](#) for more information.

Is the Argon tank empty?

- If the tank valve is open and the required pressure cannot be obtained, the tank may be empty.
- Let the lab manager know so they can order a new one.
- Change the regulator from the liquid tank to the compressed argon tank located nearby. It has a sticker on it that says argon and should have a cap on the tank as well. These tanks are normally red.
- If you have trouble with this the lab manager can help. If they are not around, try to find another person who knows how to change a regulator.

Is the water chiller on?

- Typically, if the water chiller isn't on, you will get an error specifying insufficient cooling water flow.
- Simply go into the adjoining room (ES 128B) and turn on the chiller.

Is the helium flow too high? (Laser Ablation only)

- If the helium flow is too high to start, the plasma may not light.
- Turn the flow to 50 mL/min and try to light again. After the plasma is fully lit, increase the flow slowly (by using the up arrow on the mass flow control panel) to the desired set point. You may have to turn the flow to 0 mL/min.

Did you hook up the drain tubing correctly?

- If the drain tubing wasn't hooked up correctly, the plasma chamber may be flooded.
- If this is the case, contact the lab manager as this will require cleaning and the instrument may be offline for a couple days.

If the plasma will not light after following these troubleshooting steps, try to write down the error produced when it shuts off or doesn't light and inform the lab manager.

13.2. Pumping Issues (Liquids Only)

If there are issues with your solution pumping. Try the following steps.

Are there any kinks in any of the lines?

- Check for any kinks or turns in the line that are too sharp and try to straighten them out.

Is the peristaltic pump hooked up correctly?

- The direction of the tubes hookup and the proper hookup are important. See [Section 5.1.1 Peristaltic Pump Setup](#) for more information.

Did any of the tubing come undone?

- Occasionally the tubing comes undone and the solution will be pumped out onto the tray. Check if this is the case and then look at the next question before proceeding.
- Using gloves, hook up the tubing as it should be and push the disconnected tubing back together.

Is the nebulizer clogged?

- Look at the nebulizer where the tube goes in and look for anything that looks like a clog. Sometimes they are small and not very noticeable.
- If you find a clog, report it to the lab manager. The instrument is not usable until this is resolved.

13.3. Issues with Batch Files

Some users experience issues with batch files saving and communicating with the ICP-MS Top software. Some common issues and resolutions are listed below. For more information see [Section 9. Data Analysis Window](#) for more help with your respective analysis type.

Is the data not showing up after an acquisition finishes?

- Check to see that your DA Method is set up correctly. See [Section 9. Data Analysis Window](#) for more help.
- Is the proper display type selected? Choose from the different options on the right side of the toolbar to get either counts or concentrations to show up. It helps to select "Default Columns" and then count or conc mode.

Is the batch file not communicating with the ICP-MS Top software?

- Most of the time, this is due to a batch file name being too long and containing special characters. Limit the total character count to 8 and only use letters and numbers. Most of the time it is suggested to use the current date. (i.e. 05142015.b)
- If you made a complicated batch file name and tried to run a sample, you may have to restart the computer before you will be able to use the new, shorter named batch file to collect data. The ICP-MS will stay running while the computer restarts.

13.4. Data not Displaying at all in the Data Analysis Window

The DA Method editor can be a finicky portion of the DA window. If this is not set up correctly, your data may not show up at all, but fear not! The data is in the file, it just isn't displaying due to a configuration error. See [Section 9. Data Analysis Window](#) for more help with this.

Is your data analysis type set correctly for your analysis method?

- If you are running a time resolved analysis you need to change to timechart in the DA Method Editor under Data Analysis Method.
- If you are running a spectrum mode, you must ensure this is selected in the DA Method Editor under Data Analysis Method.

Is the Analyte List correctly populated?

- Check that you have imported your analyte list from your method file. In the DA Method Editor, select Analyte List and at the top choose Load List from Acquisition Method. Select your method.
- (Liquids) Once the analyte list is loaded, be sure to set the internal standards. For a list and instructions on this, see [Section 9.2.1 Creating a Batch File](#).

13.5. Issues with Noisy Data and High Backgrounds

Occasionally, the instrument will output noisy data and the background signals will be very high and unstable or drop out suddenly. Try the following to remedy this. If this doesn't work, contact the lab manager. The other possibility for noisy signals is sample contamination or dirty tubing.

Does the background signal drop out for no reason?

- Try restarting the plasma to see if there is a torch grounding issue. If this does not work, try once more.
- See if the gas flows are consistent. Go to the instrument control panel and monitor the gas flows using the meters.

Is there a high background?

- (Liquids) Try rinsing the tubing with a 5% nitric solution to try to clear out the lines. If this doesn't work, the lines may need to be changed or your blank is too dirty.
- (Laser Ablation) Try letting the instrument warm up for longer with the helium flowing at the rate at which you wish to capture. Watch for the signals plateau with no slope such that the background is constant.
- (Laser Ablation) Allow more time between sample runs as there may be residual sample in the line that needs to be purged between acquisitions.