

Scienta Omicron ESCA 2SR XPS Standard Operating Procedure

This standard operating procedure (SOP) is intended for general use of the ESCA2SR XPS and does not contain instructions for operation of the Ion Gun, UPS source, Dual Source, ARXPS, imaging XPS, GCIB, heating stage, or inert-gas sample puck. Initial training for this instrument only covers basic XPS and software usage and is required for any operation of the instrument. If you are interested in learning/using any of the aforementioned extra capabilities of the instrument, please contact Darrah Johnson-McDaniel at dj378@cornell.edu; separate training sessions are required for using each.

FOM USAGE: You are required to reserve time on the instrument through Facility Online Manager (FOM) at fom.ccmr.cornell.edu. Your reservation time encompasses the time between sample loading and sample retrieval from the Airlock.

1. **You should Reserve time on the instrument starting when you want to load your samples into the Airlock and until you remove your samples from the XPS instrument and replace the empty stage.**
2. **You should Log-ON in FOM when you transfer your samples from the Airlock to the Analysis chamber.**
3. **You should Log-OFF in FOM only after you have read the checklist in Section VIII and are completely finished with using the instrument or XPS computer.**

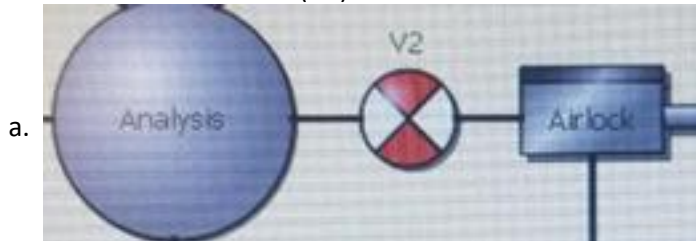
Data Retrieval: You cannot use any USB or other external drives on the XPS computer. Therefore, you cannot retrieve data using any external drive. You can retrieve data by using e-mail or your Cornell BOX account.

NOTE: There are not many safeguards on the instrument. So, be very careful when operating the Mistral Control panel or the manual controls on the instrument. This Mistral Control is the LED panel on the control tower with the schematic of the instrument. Venting the wrong pump or opening the wrong valve at incorrect times may result in damage to the instrument. Clicking on the turbo pumps opens options to turn them off, to vent, or turn them On. **THE MAIN ANALYSIS CHAMBER TURBO PUMP (T1) MUST ALWAYS STAY ON. Basically, never press T1.** **You can unselect a pump by clicking on another location on the screen.** Pictures are noted below of what the Mistral Controller should look like at each stage. Please feel free to contact the facility manager, Darrah Johnson-McDaniel at dj378@cornell.edu if you have any issues or questions.

I Loading or removing samples from the XPS Airlock

Note: The Airlock should be kept under vacuum unless loading or removing samples. To load or remove samples, the Airlock can be **Vented** to bring it to atmospheric pressure by flowing nitrogen. Please keep sample loading/removing time to under 10 minutes. If longer is required, please **Pump Down** the prep chamber to vacuum while preparing the sample stage.

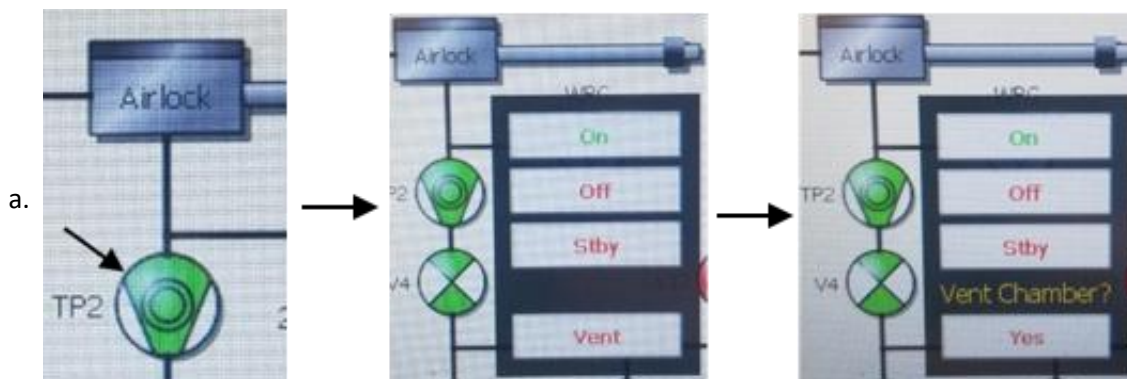
1. Make sure the Gate Valve (**V2**) between the Airlock and Analysis chamber is closed



2. **RED** means it is CLOSED and **GREEN** means it is OPEN.

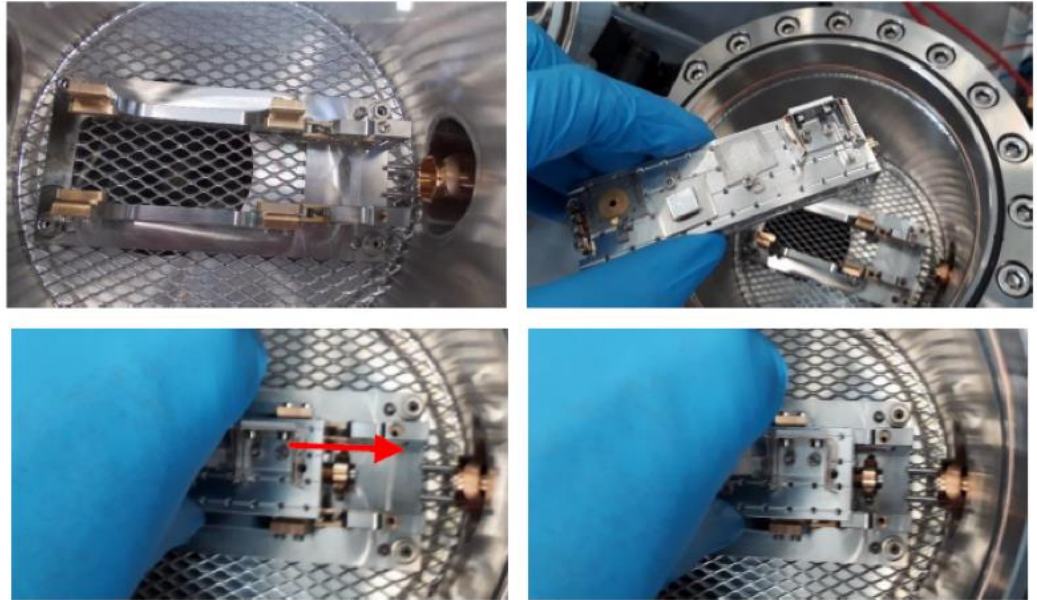


3. Press the Turbo pump connected to the Airlock (**TP2**) on the Mistral controller, select **Vent**, and then press **Yes**.



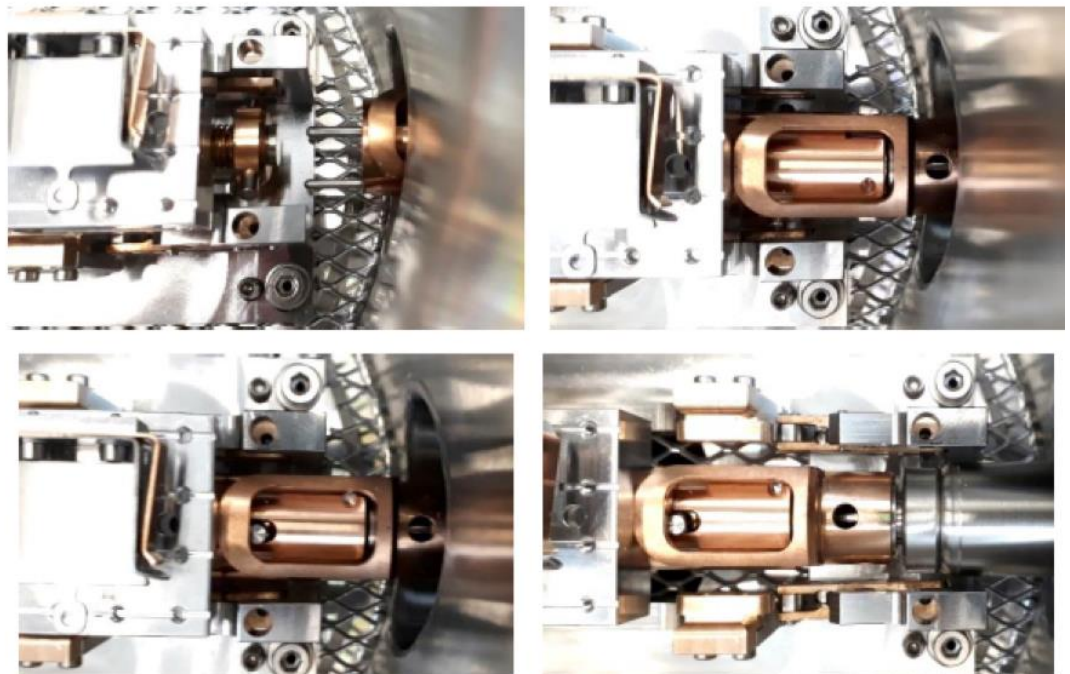
4. V4 will close, and TP2 will slowly power down and turn red. This process takes a few minutes. The chamber is flooding with nitrogen during this time and is gradually being brought up to atmospheric pressure.
5. Wait for the system to finish Venting (pressure will read "atm")
6. **Open** the lid to the Airlock
7. The mounting stage can then be removed/loaded for sample removal/loading onto the stage.
8. Keep the Airlock lid closed as much as possible.
9. Try to keep the time TP2 is in Vent mode at a minimum because Nitrogen is constantly flowing in this state.
10. If you are expecting sample mounting onto the stage to take an extended amount of time $\geq 10\text{min}$, please instead pump down the Airlock during sample preparation to prevent excess Nitrogen loss. (Perform Step#7 below, and then repeat the venting process to load your prepared Sample Stage into the Airlock).
11. What it should look like after removing and/or loading the sample stage

a.



12. **Close** the lid on the Airlock.
13. You now need to lock the manipulator onto the sample stage. Using the sample manipulator, twist and lock onto the sample stage.

a.



14. Be sure that the stage is locked onto the manipulator. Test this by gently pushing the manipulator forward and make sure the sample If it is not done correctly, the stage can fall off and into the analysis chamber during the following steps. (This is a big deal and will put the instrument down for a few days)
15. Now that the sample is locked in place, we can pump down the chamber. Press **TP2** again on the Mistral controller and Press **On**. This will slowly pump down the chamber to UHV
16. You must wait for UHV pressures ($<7 \times 10^{-7}$ mbar) to be reached in the airlock before continuing.

II Transferring samples from Airlock to analysis chamber

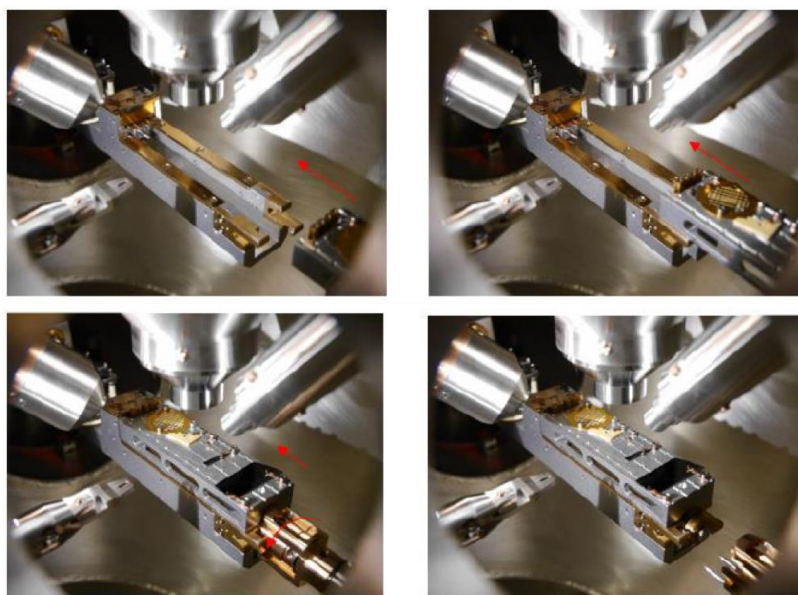
1. You MUST wait for the Airlock pressure to read at least 7×10^{-7} mBar, or lower.
2. The stage holder in the analysis chamber should already be at the Sample Transfer position.

1. **Open** the gate valve (V2) between the Airlock and Analysis chamber

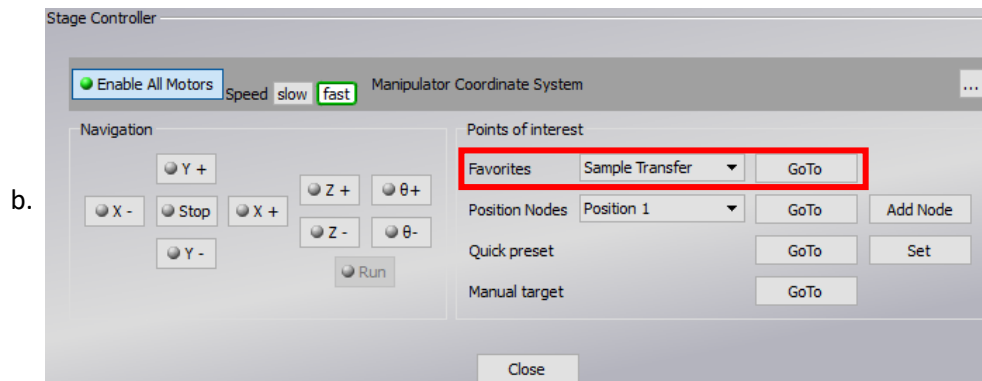
- a. Press V2, changing it from **Closed** (red) to **Open** (green)



2. Using the sample manipulator, move the sample from the Airlock to the analysis chamber mount
 - a. Carefully push the sample onto the mounting base and lock it into place by rotating the manipulator so the pins connect with the grooves in the mount.



3. If your sample is heavy (e.g., using the inert-gas puck), the manipulator arm may droop too low. You will just need to lower the mounting base more in the $-Z$ direction before you can push your sample on top of it.
3. If the sample holder does not smoothly slide on to the sample stage in the analysis chamber, follow the steps in **Section III** to connect to the stage controller and then:
 - a. Select Sample Transfer from the Stage Controller Favorites dropdown menu and then click the GoTo button.



- c. This will move the mounting base in the analysis chamber to the correct position for transferring the stage.
4. Fully retract the mounting arm back to its original position.
5. **Close** the gate valve (**V2**) between the airlock and analysis chamber.

III Operating Matrix and stage controller software

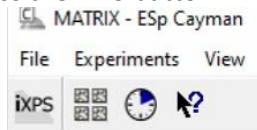
1. If not already open, Open Matrix and the Camera software



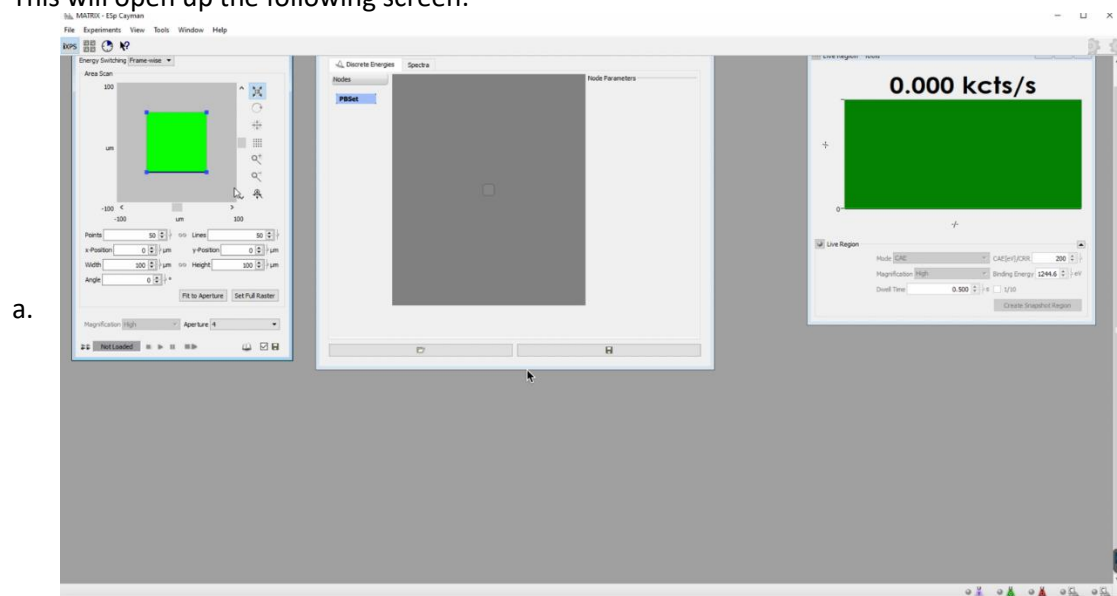
- b. Press the Cayman (C) software option in the Matrix pop-up window



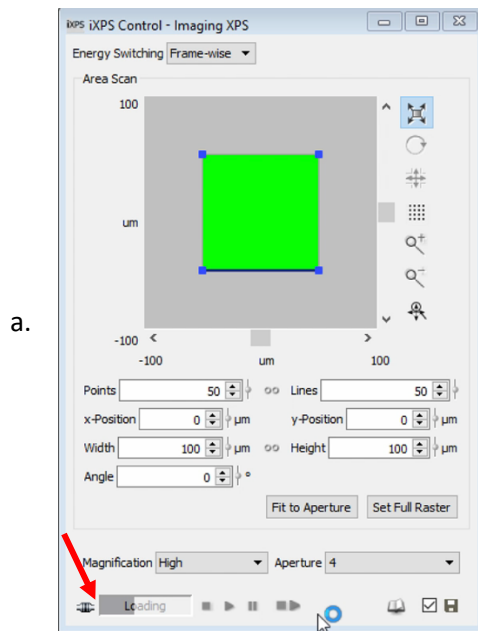
2. Press the iXPS button.



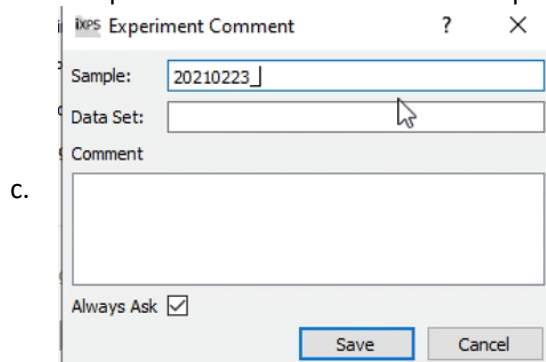
3. This will open up the following screen:



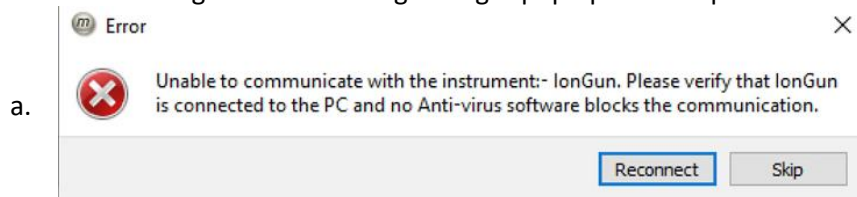
4. Click on the connection symbol to connect to the control tower-



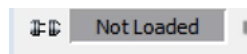
- b. A experiment comment window will open- save the sample with the days date



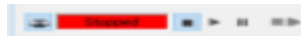
5. An error message about the ion gun might pop up- click skip



- b. A closed connection will look like this:



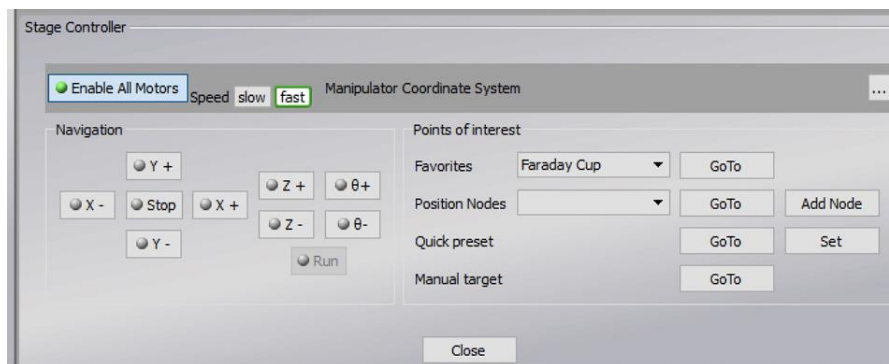
- c. When connected, it should look like this:



6. Open the stage movement controller in the bottom right you can move in the X, Y, and Z directions, as well as rotating the sample stage. This will be useful for setting up your experiment, described in the following section (IV)



b.



IV Setting up for Experiment in Matrix

1. Using the stage controller, move in the X and Y direction and bring one of your samples to the cross-hairs. You can use the camera for this purpose. Cross-hairs indicate the position where data will be collected.
2. You will need to re-position your sample along the Z-axis in step 4. The goal of this will be to bring your x-ray spot and analysis spot into focus, by maximizing counts
3. Turn on the mono AlK α high power X-ray source

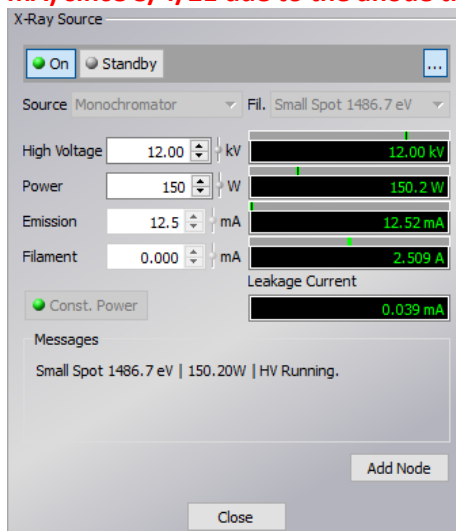
a.



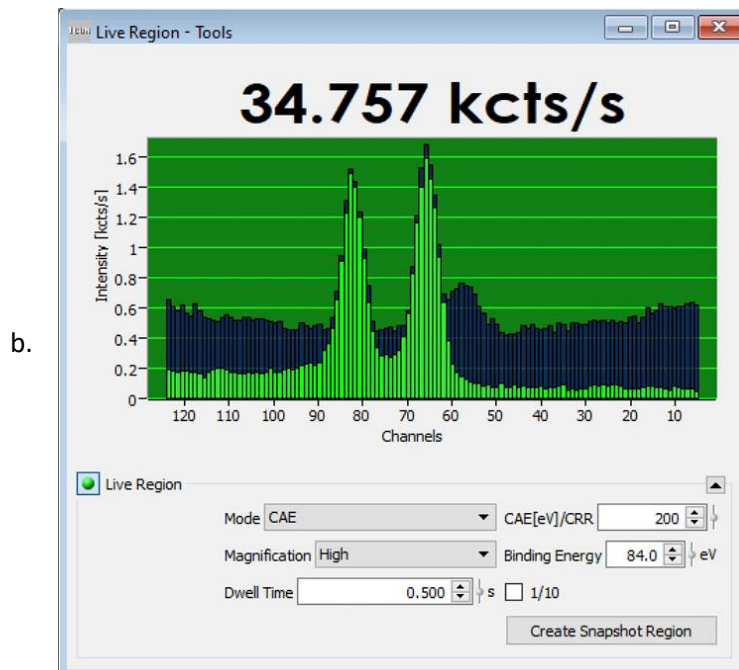
- b. Normal operation power is 300W (15kV and 20mA) but operating at 150W is also typical.

Note: we've switched to using the small spot and operating at 150 W (12 kV and 12.5 mA) since 8/4/21 due to the anode tripping at higher voltages.

c.



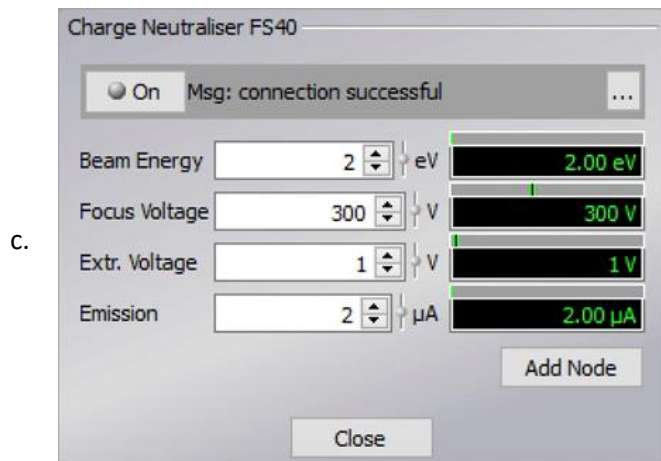
- d. Do not increase the power output above 300W unless discussed with facility manager first
4. The X-rays will ramp up slowly until the power reads 300 W. Press the *Live* button in the Live window to see your live counts in the detector.
 - a. Enter an appropriate binding energy of an element in your sample with a large relative abundance into the Binding Energy box and press enter.



- c. Adjust the pass energy to 200eV.
- d. Using the counts# in the top of the Live window adjust your sample in the Z-direction by clicking the +Z (up) or -Z (down) buttons. Maximize your counts. This will put your sample in focus.
5. **Flood Gun (for non-conducting samples):** If your sample is non-conductive you will observe a shift in the peak BE (or, your peak may not be there at all) to a higher value. If so, you will need to turn on the Flood Gun via the button shown here:

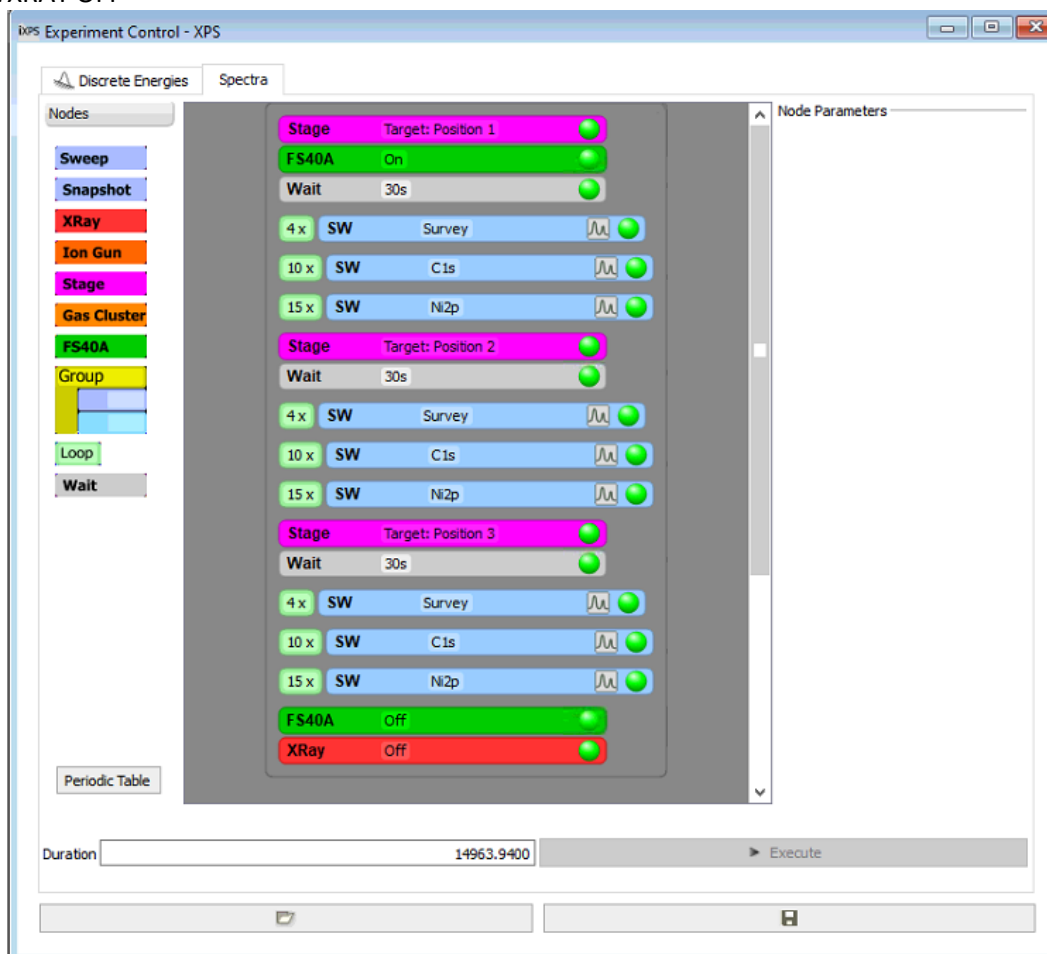


- b. For the Flood gun, you should check the live view carbon's binding energy at 284.8 eV. The peak should be centered in the in the middle of the window at channel 64, if it is not turn on the flood gun, start at low energies and low emission voltages (~1eV and 1 uA) and increase both of the flood gun until the peak is stable and close to the middle of the window. Note that as you increase the voltage (especially > 5eV), the peak will start to broaden. You might have to go as high as 20 eV.



- d. After turning on the flood gun, you may need to re-adjust your Z-height again.

6. Drag and drop experiment nodes, loops, sweeps, etc. into your experiment control window. The typical experiment may follow the following layout, from top to bottom:
 - a. Target- Position 1
 - b. FS40A Flood Gun ON (if needed)
 - c. Wait
 - d. Loop # - Sweep- Survey
 - e. Loop # - Sweep- High Res
 - f. Loop # - Sweep- High Sen
 - g. Target- Position 2
 - h. Wait
 - i. Loop # - Sweep- Survey
 - j. Loop # - Sweep- High Res
 - k. ...
 - l. FS40A OFF (if needed)
 - m. XRAY OFF



7. Click on the open folder icon, and select the blank template to remove the previous user's experiment.
8. To set up a high resolution or high-sensitivity core scan:
 - a. Drag in a sweep
 - b. Right click and select configure with periodic table
 - c. Select the element of interest and press enter
 - d. Check your scan parameters
9. The follow parameters are typically used for survey/high res/high sensitivity sweeps:
 - a. Survey: Pass energy: 200eV, Step size: 1eV, Dwell time 100ms
 - b. High Res: Pass energy: 50eV, Step size: 0.05eV, Dwell time: 200ms

- c. High sensitivity: Pass energy: should be exactly the same as you used for the survey scan, Step size: 0.2eV, Dwell time: 200ms
10. After setting up your experiment, press **Execute**. This will run your experiment from the top of the Experiment Control window to the bottom and will only run the highlighted green items.
 - a. While the experiment is running, you can view active scans by clicking on the spectrum button on each sweep.
11. After your experiment is complete, either have the script turn **OFF** any X-ray sources or Flood Gun, or manually turn them off by pressing their respective ON button so that it is greyed-out.

V Transferring Samples from Analysis Chamber back to Airlock

1. After your experiment is complete, make sure all sources or guns are OFF.
2. The Airlock should be in the e-7 mbar range or lower.
3. In the MATRIX software, move the sample holder to the **Sample Transfer** position.
4. **Open V2**
5. Move the manipulator into the analysis chamber
6. Slowly introduce the two guiding fork pins into the bores at the sample carrier. Look at the orientation of the grooves and make sure it lines up. Move the manipulator forward until it is in contact with the sample carrier.
7. Rotate the manipulator to unlock the pins from the mount. Turn further until the pins are locked (vertical) into the manipulator arm.
8. Retract the manipulator arm from the analysis chamber, transferring your sample into the airlock.
9. Be sure your sample moves to the back position of the carrier in the airlock, over the guiderails. It will snap into this latch position.
10. Release the sample carrier by turning the manipulator arm, leaving the pins horizontal.
11. Now that the sample is separated from the manipulator arm, move the arm the rest of the way back along its track.
12. **Close V2**
13. Refer to SECTION I for venting the airlock to retrieve your samples and then return the sample carrier to the airlock.
14. After completing SECTION I, be sure the airlock is now pumping down to UHV.

VI Exporting Data using Vernissage

1. On the desktop, open Vernissage
2. File->Open-> C:\Data\Scienta Omicron\MATRIX\default448
 - a. Folders are arranged by date. Open the folder in which your data is stored.
3. Highlight all of your spectra you wish to export. Looped scans will be connected by white arrows (picture). These must all be highlighted and will be exported as 1 summed scan.
4. CasaXPS can display the Y-Axis as CPS (counts per second), so your average intensity won't change with looped/summed scans.
5. Go to File->Export Selected->VAMAS format. Select your folder and make a new folder with the date of the scan. This will export each unique scan as its own CasaXPS file.

VII Analyzing Data using CasaXPS

1. To open these files in Casa XPS:
2. Open CasaXPS
3. File->Open and Merge->highlight all of your CasaXPS files you which to open ->Open
4. To sort by sample ID, press the red EDIT button on the menu bar
5. Open the scan by double-clicking on the data block and then select the spectrum
6. Quantification of the survey spectrum:
 - a. Click on the Library icon (Lib), or press F10. Click on the Periodic Table tab.
 - b. Select elements, verify transitions that appear with data
 - c. When all peaks are identified, click Create Regions
 - d. To zoom in, draw a box around a peak, and click inside the box. Use these icons to shift the data. To zoom back out, click on the reset icon.
 - e. Important: verify that regions are correct. If a doublet region (Cr 2p, e.g.) only encloses a singlet, click F7 or the Quantify icon. When the Regions tab is selected, adjust the bars around the regions to obtain a proper background that intersects the noise at a reasonable point.
 - f. Backgrounds are typically Shirley for metals and Linear for polymers.
 - g. Some regions may need to be added manually.
 - i Zoom in on a peak
 - ii F7, or select the Regions tab, click Create.
 - iii When the Regions tab is selected, adjust the bars around the regions to obtain a proper background that intersects the noise at a reasonable point. Backgrounds are typically Shirley for metals and Linear for polymers
 - iv Make sure region name is correct, type in '# + Element + transition' to change a region and the RSF value. The '#' command utilizes information in the library.
7. Adding high-sensitivity data to the survey scan quantification
 - a. Manually add a region to the high-sensitivity
 - b. Double click on the survey spectrum
 - c. Select the tile annotation icon (pencil icon or press F9)
 - d. Select the quantification tab -> apply
 - e. Select the annotation history and delete the first annotation
 - f. Click on the sample ID to select the entire row
 - g. The HS percentage should now be added to the survey scan.

VIII Troubleshooting

1. If the X-ray source trips and shuts off, do the following to clear the error:
 - a. Close Matrix.
 - b. Power cycle the 558-power supply (located in the rack next to the ESCA).
 - c. Then re-open Matrix and turn on the X-rays.

Quick Overview for Experienced Users

1. Vent airlock and retrieve sample carrier
2. Load samples onto carrier and then place back into airlock
3. Close lid and pump down airlock
4. Once pumped down to UHV, open gate valve and transfer the stage into Analysis chamber.
5. Retract arm and re-close gate valve
6. Log into FOM. Open matrix/stage/camera software and connect.
7. Set up experiment.
8. Start source (and FG if needed) to set all target positions(X,Y, Z, and θ) coordinates
9. Add sweeps as needed under each target position
10. Add FG/X-ray ON or OFF where needed.
11. Run experiment
12. After experiment is complete, make sure all sources and guns are OFF
13. Move stage back to "Sample Transfer Position"
14. Transfer stage from analysis chamber back to the airlock, through the gate valve
15. Close gate valve
16. Vent airlock, then remove stage, remove samples from stage, replace stage into airlock, close airlock lid, and then re-pump down air lock
17. Collect all of your data as needed from computer (e-mail or BOX only, no external drives allowed). Disconnect Matrix software, and then Log off of FOM