

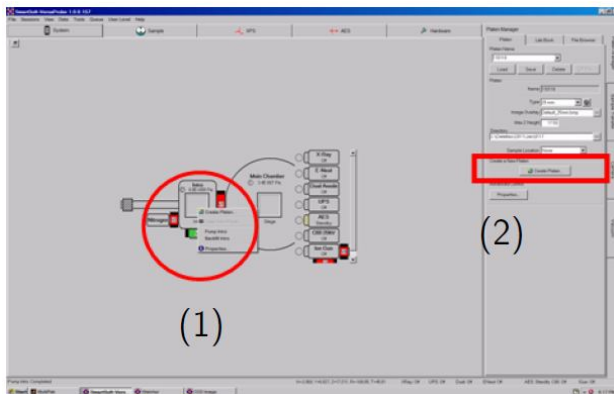
X-ray Photoelectron Spectroscopy (XPS)

Model: PHI 5000 Versa Probe-II

The Versa Probe is PHI's second generation XPS based Multi-technique system. The unique strength of the system is its ability to image and analyze features as small as 10 μ m in diameter. The following procedure should be followed while operating the system:

Standard Operating Procedure (SOP)

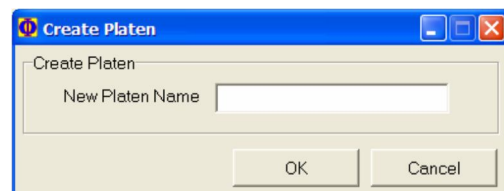
1. Confirm Neutralizer off and Argon ion gun off, and light source is off. Also confirm no platen holder is inside the main chamber or Intro chamber.
2. Prepare the sample and mount onto holder.
3. Under "System" tab, right click on Intro and do a backfill. Then right click Intro again to create new platen. Give it a name.



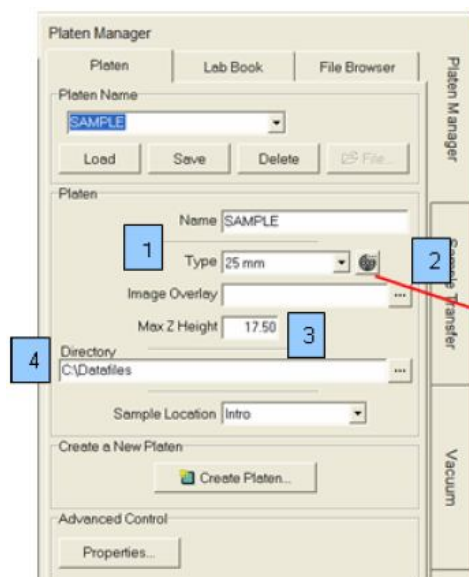
Create Platen: (1) Right click, or (2) Click on the button

Setup for "Create Platen" window:

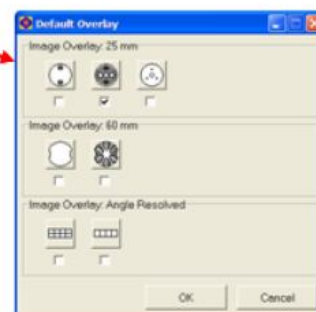
- (a) Confirm the data folder name.
- (b) Choose the platen type.
- (c) Enter the Maximum Z Height. (In the case of standard sample holder, enter the value by subtracting sample height from 20mm)
- (d) Choose the directory to save the data.



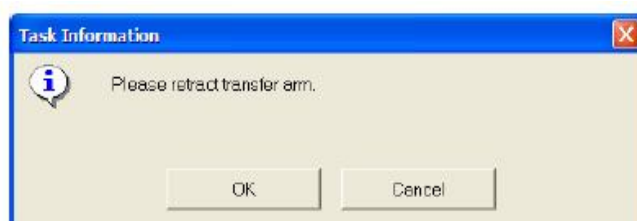
Enter a name for data folder



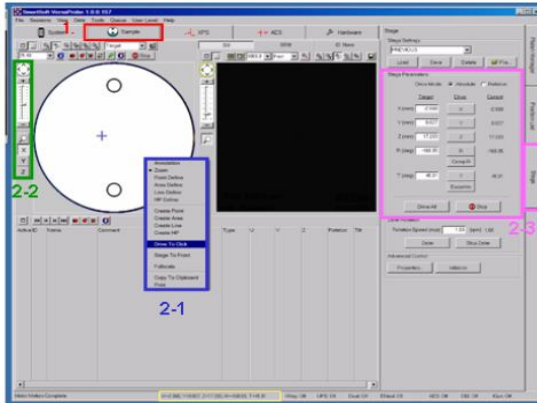
Platen Manager



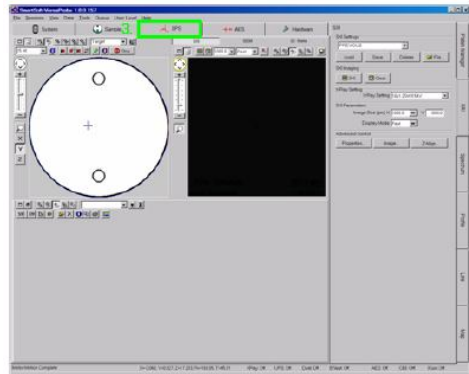
4. When Intro is up-to-air ($1.0E+5\text{Pa}$), open the Intro cover and put your sample it. (Make sure it is correctly put on)
5. Drag the created platen to main analysis chamber. (Turn on Light source now).
 - This action will make software automatically take Intro sample photo (> Close the Intro cover) > pump down > wait for good vacuum > once vacuum is ok, move stage to transfer position > then proceed for introducing the sample into main analysis chamber.
 - The following message windows will appear. Read them carefully and follow them systematically.



6. After sample transfer, in Smart Soft-VP lower-right status bar, right click E-Neut & Ion gun to put them to "Neutralization Auto"
 - Notice that prior Ion gun operation, V4 & V8 will automatically be opened (V4 = Ion gun differential pumping, V8 = Argon gas in).
 - Light source can now be switched off.
7. Go to next tab "Sample". Navigate to your interested sample by operating the taken sample photo.(Various ways possible such as right click > create point, Area, Line or HP (for blanket layer), or right click > move to mouse click)
8. Move to your desired sample #1. Run Z-align.
 - After Z-align, the optimum Z will be calibrated automatically. Update your current position for the new Z.
 - If all your samples are of similar height, you can go ahead and do a "Set All" to same Z for all interested samples.



Three ways to move the Stage



Z height alignment

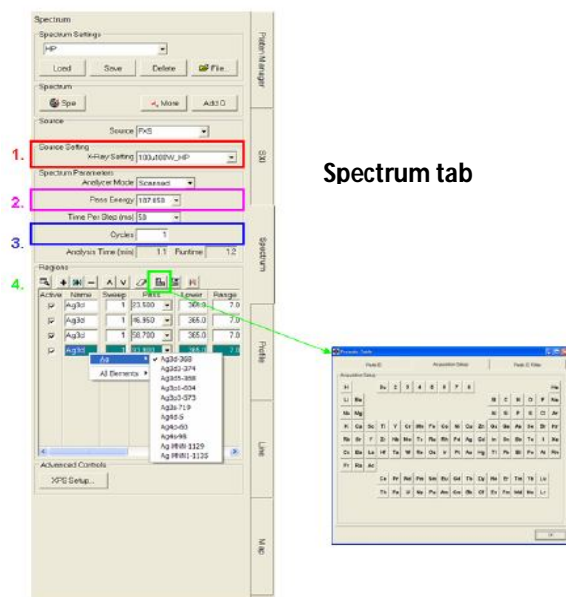
9. If feature is small, use SXI function to accurately locate your analysis area. ***Remember to do Auto Z before SXI.**
10. The added area could either be a point, an area, a line or a HP (High-Power) area.
11. To start taking spectrum for samples, operate between the “Sample” tab and “XPS” tab. In “Sample” tab, check a tick for the area you want to analyze. In “XPS” tab, set the desired analysis condition you want.
12. Before starting the analysis, choose the X-ray settings you want to use for analysis.
13. Typical Analysis:
 - Define a **survey [wide] scan** the first time (usually wide scan has 1100eV to 0eV binding energy)
 - Identify the elements from the survey scan by [ID] button (sometimes need users’ fine judgement)
 - If desired, define **narrow scan** on each element that appears in the survey scan (for making accurate Quantification & Chemical State information).

•How to choose **Pass energies and step size:**

- **Survey:** Pass Energy 117.4 or 187.85eV. Step size 0.8eV to 1.6eV/step
- **Narrow (Good resolution):** PE 58.7eV. Step size 0.125 to 0.25eV/step
- **Narrow (Superior resolution):** PE 11.75 or lower. Step size 0.05 or lower per each step.

•**Sweep & Cycle**

- Sweep only to individual created elements or regions
- Cycle to all created regions
- For low intensity peak, make more sweeps.



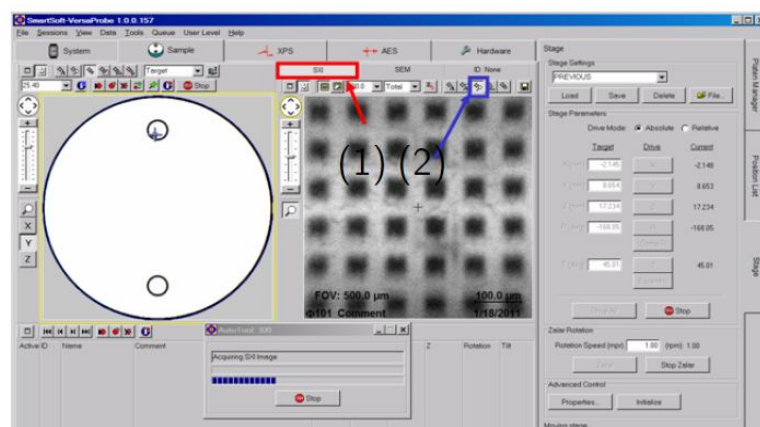
Spectrum tab

14. If one wants to go on for another analysis point/area, please go back to step #11.
15. Complete the analysis.
16. At lower-bottom right of Smart Soft-Versa probe, right-click and set I-Gun and E-Neut from “Neutralization Auto” back to “Off”. Right click again and set E-Neut & Ion gun back to state “OFF”.
17. Drag the sample from Main analysis chamber back to Intro chamber.
 - This will do all the Intro pumping > Move stage to exchange position.> V1 opens > User should move fork in > Click software pop-up message to move stage for releasing sample > User should move fork out > V1 closes automatically > DONE.
18. Right-click Intro to backfill Intro. After taking out sample, if you have another sample then go back to step #3. Otherwise, put back on the Intro cover and right-click Intro to **pump back down again so as to keep system in vacuum.**
19. Go to “system” tab > “Platen Manager” > “Platen” tab > at “Sample Location” choose “None.”
20. Make sure again to switch off the Light source.
21. Finally confirm the following:
 - V3, V7 open. All other valves closed.
 - Light source switched off.
 - E-Neut state: Off
 - Ion-gun state: Off
 - X-ray state: Off
 - UV light state: Off

Scanning X-ray Imaging (SXI)

If analysis area is narrow, a few tens to a few hundred micro meters, it is convenient to use SXI after coarse positioning using CCD image.

1. Set the analysis point through CCD image



Click to choose the new centre of the analysis area

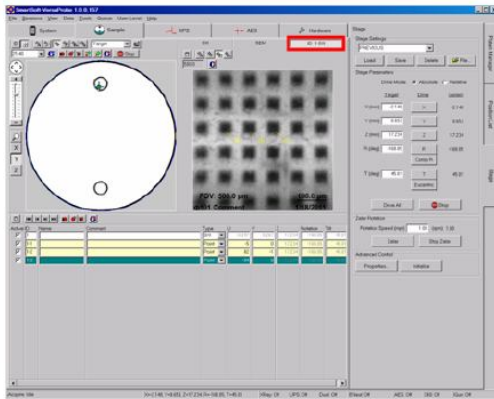
- Use “SXI” mode, click on the “Zoom” icon to choose.
Click on the point to become centre of SXI image.
- Click on the “Save SXI image” button.
- Click on the “Stop” button.
- Click ID button.



Save SXI image button



Stop button

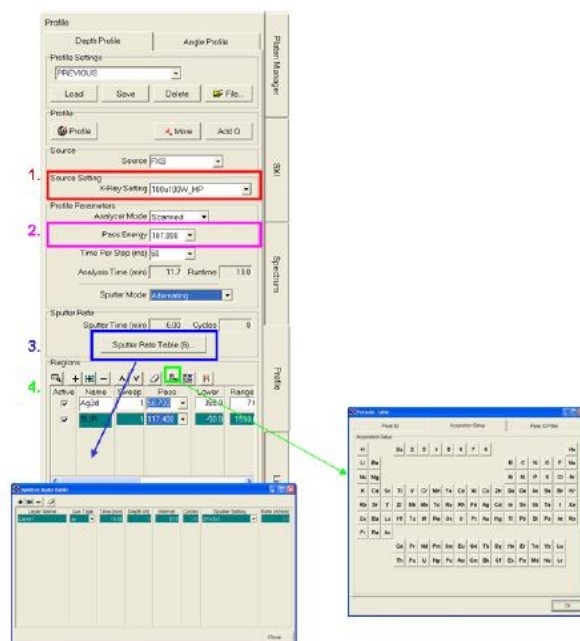


Click on the point to be measured at SXI, then set the required conditions.

Depth Profile

Qualitative and quantitative analysis in depth direction, using Ar ion sputtering, is available.

- Choose X-ray condition that meet analysis area from “X-Ray Settings”
- Choose “Pass Energy” that meets analysis purpose.
- Set the following at “Sputter rate Table”:
 - Sputter setting for sputtering conditions
 - Interval for sputtering time for each cycle
 - Time for total sputtering
- Choose the element to analyze from the periodic table by clicking on the “Periodic table” button icon. “Su” stands for survey.

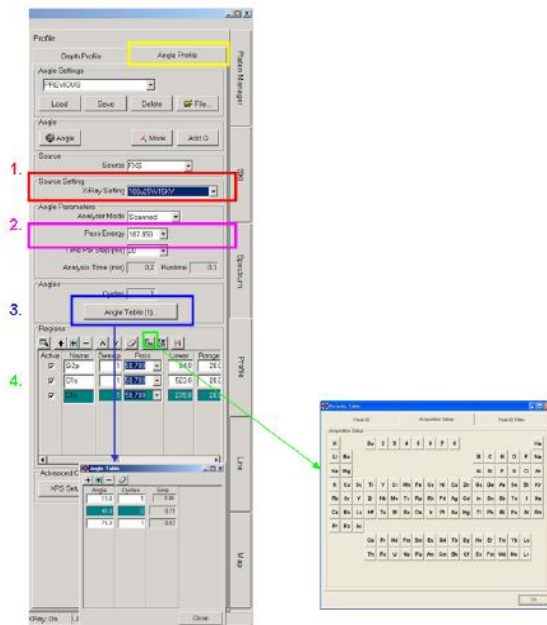


Depth Profile tab

Angle Profile

Non-destructive depth profiling that is taking advantage of escape depth and sample tilt, is available.

1. Choose X-ray condition that meet analysis area from “X-Ray Settings”
2. Choose “Pass Energy” that meets analysis purpose.
3. Set the following at “Angle Table”:
 - Angle for the sample
 - Cycles for the number of measurement accumulation
 - Sputter setting for sputtering conditions
 - Interval for sputtering time for each cycle
 - Time for total sputtering time.
4. Choose the element to analyze from the periodic table by clicking on the “Periodic table” button icon.
“Su” stands for survey.

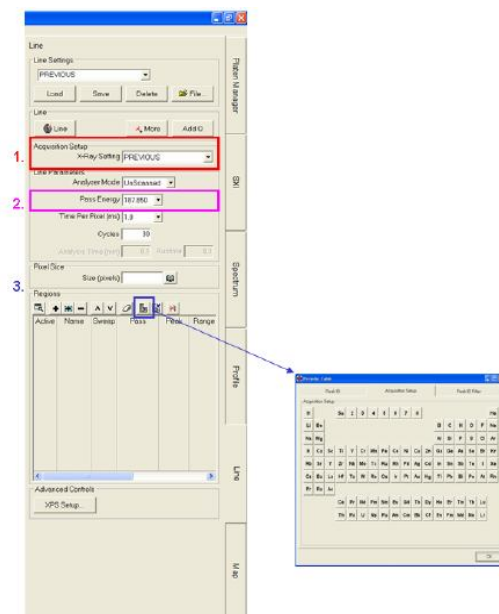


Angle Profile tab

Line

Execute line analysis with scanning X-ray.

1. Choose X-ray condition that meet analysis area from “X-Ray Settings” (The smaller X-ray beam diameter, the higher special resolution analysis.)
2. Choose “Pass Energy” that meets analysis purpose. For normal element map 117.4eV and for chemical state mapping 58.7eV or 93.9eV is recommended.
3. Choose the element to analyze from the periodic table by clicking on the “Periodic table” button icon.
“Su” stands for survey which would not be used in line analysis.

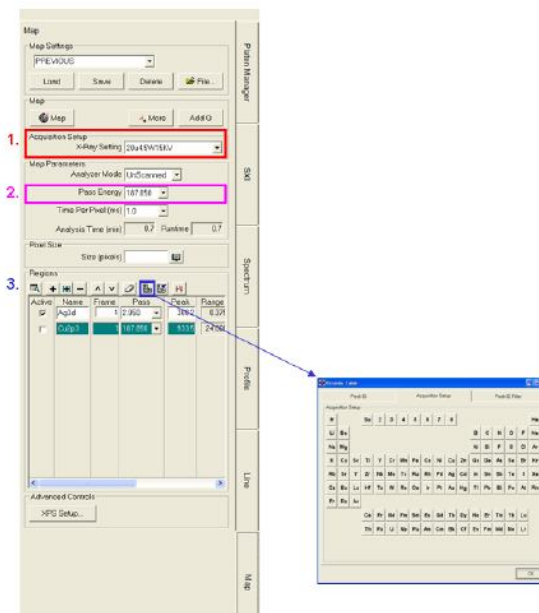


Line tab

Map

Execute area analysis with scanning X-ray.

1. Choose X-ray condition that meet analysis area from “X-Ray Settings” (The smaller X-ray beam diameter, the higher special resolution analysis.)
2. Choose “Pass Energy that meets analysis purpose. For normal element map 117.4eV and for chemical state mapping 58.7eV or 93.9eV is recommended.
3. Choose the element to analyze from the periodic table by click on the “Periodic table” button icon.
“Su” stands for survey which would not be used in area analysis.

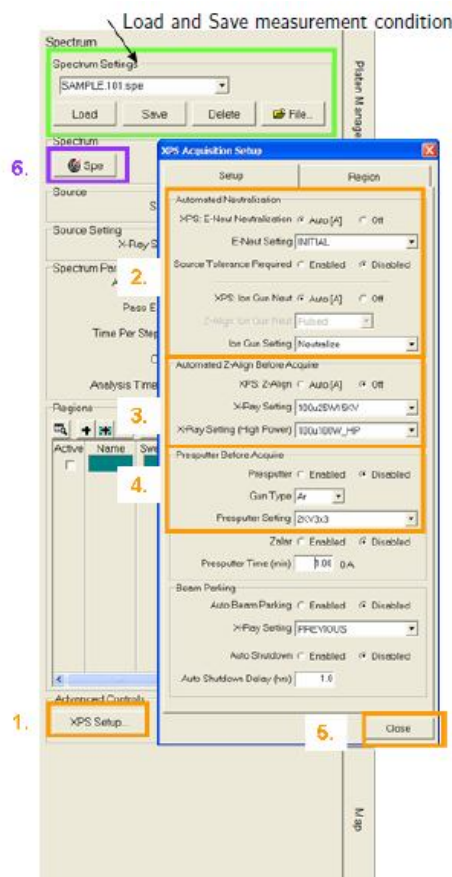


Map tab

Common Settings

1. Click on the “XPS Setup ...” button to show XPS Acquisition Setup window, then choose Setup tab.
2. Check whether “Automated Neutralization” is Auto, if the sample is an insulator
3. Set “Z-align” Auto if execute Auto-Z before the analysis.
4. Enable “Presuptter” if sputtering the sample before analysis is necessary.
5. Click on the “Close” button to close window
6. Click on the “Spe” (or another type of analysis) button to start analysis.

You can save and load analysis condition to Setting files. Use appropriate buttons.



Common Settings

End Analysis

Analysis is automatically terminated and data is saved to file. There is no special operation required.

To change termination schedule, following are available. This is in common among all analysis types.

- Abort analysis
 - Click on the “Stop” in “Auto Tool” window.
 - Click on the “Abort” in “Acquisition Status” window.
- End analysis with current step
 - Click on the “Stop” in “Auto Tool” window.
 - Click on the “Stop” in “Acquisition Status” window.



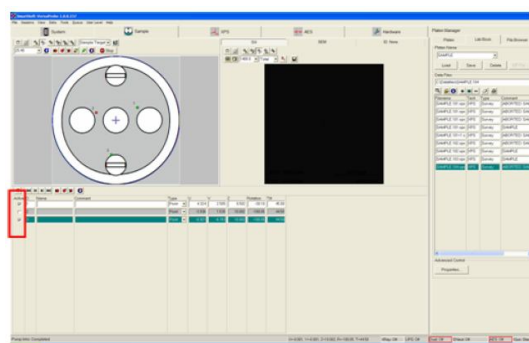
End Analysis

- Extend analysis
 - Click on “More.”
 - Enter the new cycle number in “Cycles”, which is a sum of original and increments, then click on the “Spe” (or another analysis under way) button.

Queue

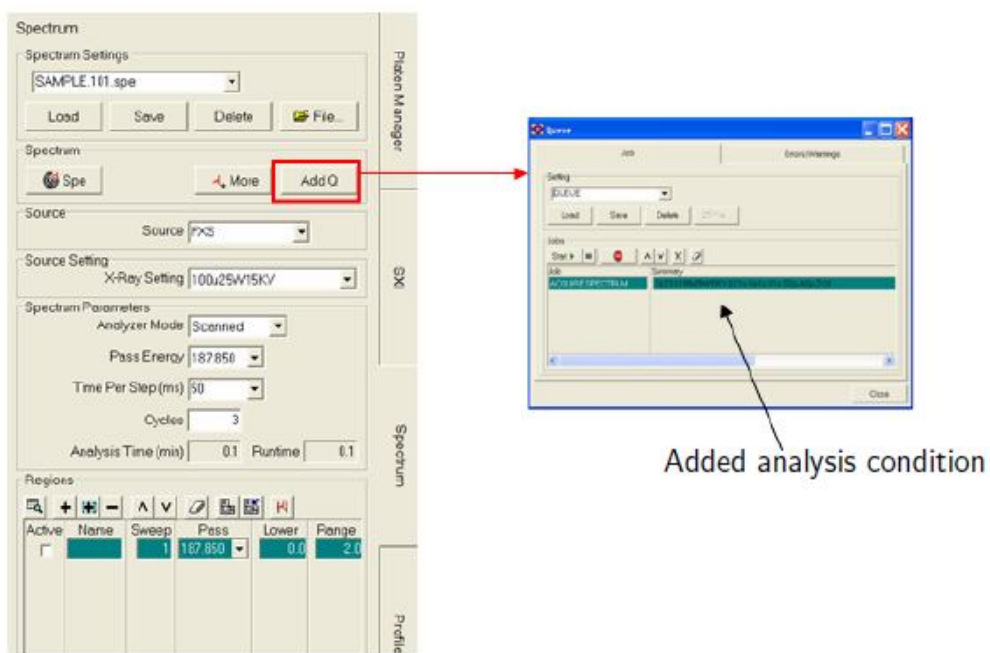
“Queue” enables to execute different analysis at different points.

1. Click on the “System” tab and confirm that the sample to measure is on the stage.
2. Click on the “Sample” tab and mark check in the check box of “ID” of desired analysis.
3. Click on the “XPS” tab to set conditions.
4. Click on the “Add Queue” button. The result can be seen in ‘Queue’ window.
5. Repeat the above steps.
6. Click on the start button in “Queue” window.

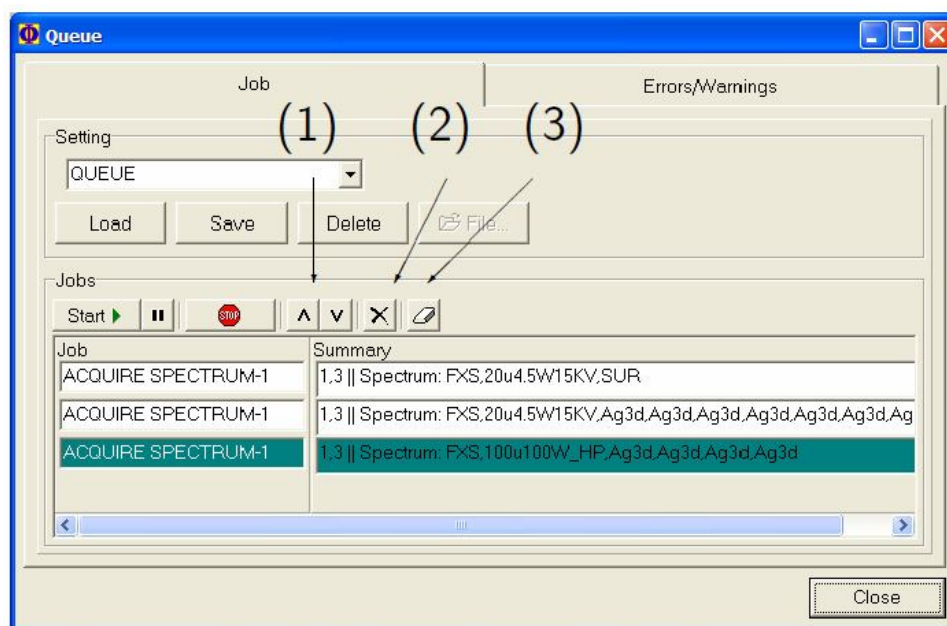


Sample tab

To add new task after you start analysis, click on the pause button. To save or load Queue task, use “Settings” in Queue window.



Add queue



Evacuation: (1) Sort (2) Selective Delete (3) Delete All

Ultraviolet Photoelectron Spectroscopy (UPS)

If ultraviolet photons (rather than X-rays) are used, the technique is called Ultraviolet Photoelectron Spectroscopy (UPS). Other than the photon source, instrumentation is identical to that of XPS.

The optional Ultraviolet photon source provides low energy photons for valence band and Fermi edge measurements. Helium gas can be used to generate single and doubly ionized UV photon sources.

- **Operating the UPS (Start-up):**

(a) First make sure about the following:

- a. UPS pump station is operating, no problem (Green button light is on for opening pumping valve).
- b. UPS rough pump is operating, no problem.
- c. VUV controller is in "Standby".
- d. Helium leak valve is fully closed.
- e. Rough pump manual valve is fully closed.
- f. Turbo pump manual valve is fully open (if it is closed, simply open it now).

(b) Switch the VUV control to "Operate"

- Set the pot for Voltage to 1000V
- Set the pot for emission current to 100mA

(c) Slowly open the Helium leak-valve and monitor the Main vacuum pressure until main chamber is 2 to 5×10^{-6} Pa.

(d) Now slowly open the Rough pump manual valve. You will see Main vacuum drops again. At this time, open the Helium leak valve more while keep opening the Rough pump manual valve to keep in 2 to 5×10^{-6} Pa in main vacuum.

(e) After Rough pump manual valve fully opens, continue to open up the Helium leak valve until 8×10^{-2} mBar on the VUV display.

(f) Hit the ignition button on the VUV controller. You will see message on bottom-left of VUV display says "Ignition active". Once the UV light is up, it will change to "READY".

(g) If UV light cannot light up, keep the "Ignition active", and slowly increase the Helium leak valve. You can go as high as 6×10^{-1} mBar on the gauge on VUV display. (At the time, Main chamber vacuum can be in the 10^{-5} Pa range. Do not go over 7×10^{-5} Pa for the Main chamber vacuum)

(h) Once UV light-up, adjust (close) slowly the Helium leak valve to get below pressure:

a. **He-I** > about 1.5 to 5×10^{-2} mBar

b. **He-II** > about 7 to 8×10^{-3} mBar

c. Note: To operate He-II, one probably wants to keep He-I range type of pressure with UV fire-up and keep for >2+ hours before attempting for He-II.

***It can be difficult to get He-II because it means we want to ignite the source with LESS Helium gas inlet. So stabilizing He-I for longer time before going to He-II will be helpful.

UPS operation always requires sample to Stage Tilt to be 90-degree.

- **Shutdown of UV light**

(a) If He-II is used, first thing to do is to increase the He leak valve to get back into the He-I range. Sit for He-I UV on for 30 minutes before shutdown.

(b) For shutdown, adjust down the voltage to 150 to turn UV off.

(c) Next try to close the Helium leak valve & Rough pump manual valves at the same time, until both of them are fully closed. (Monitor Main pressure, it should slowly go down. If you close Rough pump manual valve too quickly, you will see Main vacuum jump up).

***Ideally, you would eventually fully close the Rough pump manual valve just a little before the Helium leak valve is fully closed. (This action is for the purpose of keeping the Helium flow as “From gas line to Rough pump” and not the reverse).

(d) Turn the VUV controller back to standby.

(e) If the UV source is not going to be used for LONG time, then continue as below:

- a. Wait 1-2 hours after step
- b. Fully close the Turbo pump manual
- c. Switch off the Turbo pump station.

Analysis Concepts

High X-ray Power → High sensitivity but poor spatial resolution (beam size is bad)

Low X-ray Power → Low sensitivity but good spatial resolution (beam size is good)

High Pass energy → High sensitivity but poor energy resolution (peak is FAT)

Low Pass energy → Low sensitivity but good energy resolution (peak is THIN)

Big Step size → Acquire time is short but peak shape (resolution) is bad

Small Step size → Acquire time is long but peak shape (resolution) is good

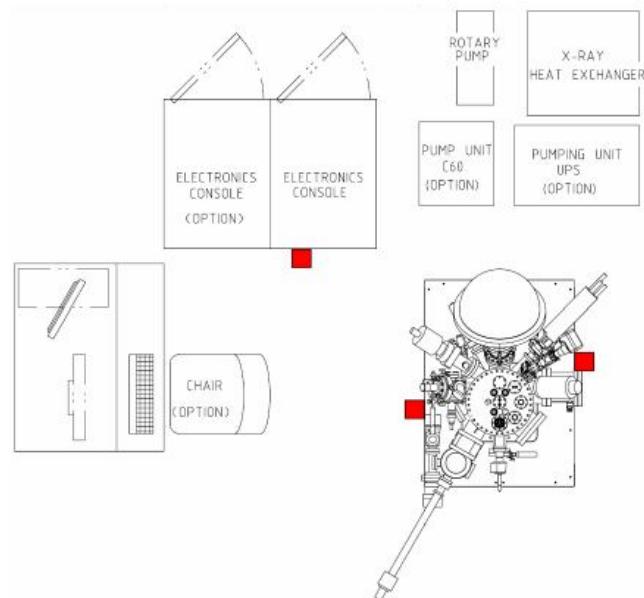
High sensitivity → Signal-to-Noise is better so total acquire time is shorter

Low sensitivity → Signal-to-Noise is worst so total acquire time is longer

Sensitivity = Intensity / counts / signals

System Power and Emergency Off

Typically, the Versa Probe system is on **24 hours** a day and not powered on and off daily. Circuit breakers are on the front panel of the 50-010 System Power Distribution. The individual circuit breakers within allow major segments of the console to be turned off for servicing purposes.



Emergency off (EMO) button locations

In the event of a hazardous occurrence or emergency, the system can be shut off, using the **Emergency Off buttons** located on the front and back panels of the vacuum console, and front panel of the electronics console. Pushing one of the Emergency Off button will disconnect all electrical power to the system. Do not use the Emergency Off button as an on and off switch, but only as a true emergency button.

Please contact SO / LIC in case of any emergency.