XPS: X-ray Photoelectron Spectroscopy

Sample Mounting:

-Always use gloves and tweezers to avoid contamination of the sample surface.
-Use the spring clips to mount samples.
-Conductive carbon adhesive can also be used (but sparingly,otherwise it will effect vacuum)
-Sample should not go outside the area of the loading plate. <u>Samples must not overhang the edges.</u>
-Before loading, check sample limit gauge.

1. **Login** to the computer:

2. **Turn on Avantage** software

3. **Turn on optical view** via Main Command Toolbar (See General layout of software at the end of this manual)

4. **Use the Sample transfer control to open the loading dock**.

5. **Load sample and pump in the loading dock** (do not send to the analysis chamber right away).

   - Make sure the sample holder is centered in the middle of the sample loading dock and is steady. The edges of the sample holder should be in line with the edge of the loading dock. The holes under the sample holder should align with the pins on the carrier plate.

6. **A good vacuum** should range from E-06 to E-07 (Check Vacuum Details) Can setup experiment while waiting for vacuum. In optical view, take a snap of image if needed. Load sample into the analysis chamber when ready.

7. **Click on Experiment in the "Experiment & Sample" Tab and setup the experiment tree**. Start with Source: X-Ray Gun, then go to point/line/area, then select Spectrum, Multispectrum.

Choose where you wish to save the data and also edit the names of the experiment, points, lines, as needed on the experiment tree:

   a. **Navigate to sample by double-clicking on the platter** (upper view). Adjust lighting/illumination in the optical view to see the sample if need.

   b. Set Source as X-Ray Gun

c. **Set the height via Z.** Change the view and set the Z to have the view the same for both. Can also set “Enable auto-height” (use relative) in under the Position tab in Properties under Experiment.

d. **Select “point”, “line”, “area” or “depth profile” from menu as needed** Point analyzes one point, line analyzes a line, area, analyzes and area. Depth profile will etch the sample surface. This can be used to remove hydrocarbons off the surface of the sample to collect a clean surface using “ion beam etch”

e. **Hold down “Ctrl” and left-click on the analysis point required to create point, line, or area.**

f. **Set the size of the x-ray spot** (largest spot size is 400 for most signal)

g. **Click on the spectrum icon** (Can use Survey Spectrum for all signal or unclick to do individual selected elements.

h. **Select “multi spectrum” for data collection.**

i. **Choose required narrow scans / surveys** (Scans for high resolution, snaps for quick frame acquisition)

j. **Turn on the flood gun in the x-ray object if required** (for insulator, semiconductors, and unknown samples)

k. **Insert a Gun Shutdown** (sources) object to shut down the gun if done with an experiment tree.

l. **Check that the vacuum** is ready and then load the sample into the analysis chamber via the Sample Transfer icon (Got back to Sample tab from Experiment tab). If the experiment tree is ready, but the vacuum is not ready, set up sample loading and click on “Automatically transfer sample and run experiment after “X” minutes.

m. **Run the experiment or the experiment step**.

n. **Repeat and expand the experiment tree as needed.** Steps in the experiment tree can be disabled (shotcut: use space bar to disable or enable experiment step). “Enable auto analysis” and “Enable Auto height” are useful when samples are flat. Change experiment name otherwise, repeated locations will override data. At the bottom left corner of the software, the lit up icons show which sources are active.

o. Export data by “Reporting to Excel” or Saving data on processing view tabs and saving. Can also export data using DataSpace.

8. **UPS: Ultraviolet Photoelectron Spectroscopy:** To set up UPS, use UV as the source, go to vacuum diagnostics and vacuum details. Make sure to click on “Flush He Gas lines (3 purges).”
    - **a.** Use the work function holder instead of the normal sample holder.
    - **b.** Once the sample is pumped and in the chamber, adjust the Azimuth (A value at the both right corner of the software window using the turn icons). The A value is near the X, Y, Z, values. Usually ~76 degrees and the spring from the holder should be touching the stage. You can see the spring by adjusting the lights in the analysis chamber. Spring is roughly located to the left of the sample holder near the D slot.
    - **c.** It’s recommended to have an etching step (ie. ~10 seconds) before your UPS step to get a clean area analysis.
    - **d.** Use the sample steps as step 7 but use UV source. Use low energy (1-4eV)
    - **e.** UPS He Survey can be used (Similar to Survey Spectrum in XPS)
    - **f.** Note, cannot use elements, only UPS HE survey for work function.

9. **Raman Spectroscopy:**
    - **a.** Make sure all lights are off and do not use the flood gun.
    - **b.** In OMIC software, use “Exp. Set” and go to “bench” tab to turn on laser (~5 minutes to warm up, wait for Raman icon to light up to active)
    - **c.** Set desired parameters
    - **d.** “Col. Sample” to collect data. Bench view shows live acquisition view.
    - **e.** Add libraries to analyze and identify unknowns.
    - **f.** Turn off laser when done.

10. **Turn off software and logout.**

General Layout: