Instructions for XPS Operation and Data Acquisition

Please go through the instructions carefully. These notes are to supplement the instructions you received during training. These notes are for typical operations. It is recommended to make your own notes for your measurements. Feel free to contact the staff in case of any difficulties with operations. If you have any suggestions/questions, contact staff.

Preparing a sample: Wear gloves! Never directly touch sample, holder or anything that will go into the UHV chamber except with instruments. Use appropriate sample holder. Note down your sample corresponding to a marker/reference position. If not there, mark the position using scratch mark. DON’T USE INK MARKERS.

Caution: With the multiple sample holders it is extremely hard to access the extreme positions.

Loading a sample:

1. Auto-valve control panel: XPS from “standby” to “on”
2. Break the sample introduction chamber by pressing “backfill intro” vacuum.
3. Remove the upper plastic cover from the introduction chamber.
4. Hold the sample holder from the upper tracks using the large curved tweezers. Use the lower track on the sample holder to mount them on the Fork of the introduction arm. Make sure that the sample is held nice and firm
5. Put the plastic cover back on the introduction chamber.
6. Hit “pump intro” and wait until all 5 air→hivac LED’s are lit. It is better to wait for few more minutes (2-3) after all LED’s are lit. It helps in improving the vacuum of the introduction chamber.

Caution: If you have reasons to believe that your sample may degas, leave your sample in the introduction chamber for long time. You may also keep your sample in the introduction chamber over night, if the equipment is not being used or may try to dry your sample in your lab.

7. Go to the computer screen and select the software PC-Access. It takes a while for the software to come up. So please have patience. After the Ion Gun Control menu and Neutralizer Control has appeared on screen, window describing the Printer Properties would come up. Click OK on that screen

8. Select the menu for the Sample Setup and in that select Stage Control. In the menu select “Dock Stage”. By doing so we bring our sample stage to a pre-selected value of x, y, z and angle so that the when we can simply introduce the long arm and grab our sample holder using the fork. At this position if we try to introduce the sample the sample holder will collide with the stage. So select “Unload stage” option on the software menu. This brings down the stage where it is safe to bring the sample.
9. Now hit the button “Intro sample” on the Auto-valve control. This opens up the valve for the introduction of the sample into the UHV chamber.

10. Check ion gauge to ensure that vacuum is in low \(10^{-8}\) range, if not slide sample in toward the chamber and then extract it. This will cause the gate valve to the chamber to close. Hit “pump intro” and wait another 5-10 minutes and repeat step 9.

11. Move the sample arm all the way in assuming pressure is good (~5x10^{-8}).

\[\text{Caution: To prevent the possibility of collision, watch the sample holder while you move the arm in.}\]

12. On the software menu select “Load sample”. This brings the stage up and grabs the sample.

\[\text{Caution: Watch the sample holder while the stage is moving up. If the stage is missing sample holder or there is lot of turbulence, notify the staff.}\]

13. Pull the arm all the way out. You will hear the noise of valve between the introduction and UHV chamber closing down.

\[\text{Caution: If the valve doesn’t closes, press “intro-sample” again (so that it is lit) and move arm in and out.}\]

14. On the screen select “done”. This makes you exit out of sample stage control.

15. On the Auto-valve control, make sure to turn the “diff pump ion gun” on. You will need to switch the valve control on the back of the instrument to manual, then press V4 to open the valve for differential pumping. Immediately after opening V4 you should hear the sound of the pneumatic valve opening. After opening the valve flip the switch back to automatic.

16. Make sure that the vacuum reading on the Ion gauge is in low nines (~2-3 x 10^{-9} Torr).

\[\text{Caution: If you find that the vacuum is either in high nines (6-7 x 10^{-9}) then your sample may be degassing. It’s a good idea to take your sample out of the main chamber and leave it in introduction chamber for a while (may be overnight).}\]

**Setting up the sample for measurement**

1. Under the menu Sample setup, select “Sample Setup”. You will have two options. You may either select (a) single point or (b) multiple points on a sample surface for analysis. Enter (a) prefix (the file name for your analysis) (b) file # (1; this no. keeps increasing as you keep acquiring the data for a particular sample/file name ) (c) comment (any comment you may want to give about your sample)

2. Change x(mm) and y using arrow keys until sample is centered (watch target/black mark at the center of the Video screen)

3. Zoom in completely
4. Select a feature on your film as displayed on Video screen and try to focus on that feature by varying the z height using up and down arrow keys.

5. You may try to select the area of your interest again by moving in x or y direction.

6. Exit the sample setup.

**Setting up the Ion gun control**

**IMPORTANT: Make sure that the “DIFF PUMP ION GUN” on Auto valve is set to on**

1. On the Ion gun control menu, to the right of your screen, select the radio button for Standby.

2. In the View menu on the Ion gun control, select “View Extractor Pressure”. A window showing the extractor pressure will come up. We need to regulate the extractor pressure at 15 mPa for neutralization and sputtering. This will be done by the Thermo-valve control to your right hand side. However, for this to work the starting pressure needs to be close to 40 mPa and then it will be regulated at 15 mPa.

3. Under the common hardware menu select, ion gun setup. In the ion gun setup menu select “pressure display on”.

4. Ensure the bleeder valve from the argon source to the main chamber is closed, then open the green knob on the ion gun one full turn and close right away! When you open up the valve one full turn, you should feel it moving loose.

5. Turn the bleeder valve (valve opposite to the Ar cylinder) very slowly so that extractor P=35-40mPa. Watch the Torr gauge on screen increase. Once the pressure starts to increase, turn the knob slowly. Torr gauge on screen is more sensitive than Extractor Pressure window. *If your sample is not degassing and your starting pressure, for the main chamber, after the introduction of sample was 2-3 x 10⁻⁶ Torr, then you should have a pressure ~ 1-2 x 10⁻⁸ Torr.*

--- Caution: Vacuum in the main chamber should be better than 8 x 10⁻⁸ Torr

6. Turn the **Thermovalve** (placed on the right side of computer) “on” and set it to “limit”. Flipping the thermovalve to Limit drives the extractor pressure to ~0.1 mPa. Wait for 2-3 minutes and flip the thermovalve button to Set point. This drives the extractor pressure to 15 mPa and will control at the set pressure (15 mPa). Wait for the Extractor Pressure to stabilize at 15 mPa.

7. On the PC-Access Menu turn the pressure display off and Exit from the men.

8. Set the Ion gun control radio button to Neutralize and wait for it to turn “green”.

**Electron Neutralizer**
1. Turn the **electron neutralizer** (on your left on equipment, brown color) all the way in.
2. On the **electron neutralizer** window set the **state** radio button to “on” and wait for it to turn **green**.

*Caution: While taking the neutralizer arm out, NEVER BRING IT PAST THE METALLIC RING PLACED ON THE ARM, OTHERWISE THE NEUTRALIZER ARM WILL BE DAMAGED. Set its **State to standby** when not using.*

**Setting up the Survey Scan**

1. On the PC-Access Menu, select **Acquire Setup**, and then select **Setup Survey**
2. Select either "**New**" or "**Previous**" by clicking the right mouse button on the appropriate selection.
3. Set the **lower limit (b)** to 10 eV and **range (c)** to 1000 eV (*These are the typical values you may select different values for your own analysis*)
4. Set the **resolution (d)** to **Survey** (*you may also select Utility or HRES*).
5. Set the **Acquisition time (e)** to **1 or 2 min**.
6. **X-ray anode (ff)** is typically set to **Al monochromated 2 mm filament**, but you may select others for your analysis.
7. Set the option “**Create Multipak file clone**” to **YES** to get file for analyzing the data with Multipak.
8. Hit **Acquire** to start acquiring the data.

**Peak Identification**

1. Select **Next Bank** on the **extreme right** on PC-Access Menu. Next Bank changes the menu options
2. Select the Menu **AC Peak ID** and then select either **Peak ID or Peak ID setup**. Peak ID labels the Peak on the data curve.

**Aligning the sample**

The sample now needs to be aligned for the correct Z height to maximize the counts.

1. Select **Next Bank twice** and go to the **ESCA/ISS** menu and select **Setup Align**.
2. You need to pick up any of the elemental peak from your data. In the element name (a) enter the peak from the element family e.g. (C1 or Au2)

3. Hit “Refresh Acquire”.

4. After the elemental spectrum appears on the screen, hit “Stage Control”.

5. Select the \((d)\) Z. Using the up and down arrow keys, move the sample up and down the Z height to maximize the counts. You may use other parameters also for your measurement. Use Coarse Adjust and Fine Adjust in the menu to select the proper Z height.

6. After you have aligned the sample, select “Previous Menu” and abort/stop the acquisition and exit from this Menu.

**Acquiring the data**

You need to acquire your survey data gain after aligning the sample.

Survey Scan
1. Under the “Acquire” menu option and select “Acquire Survey”.

2. Hit next bank. Identify the peaks using AC Peak ID.

3. Under the menu Output control, select Print Graphics.

4. You may also find out the atomic concentration of elements by selecting the option “Peak ID AC Table” under AC Peak ID Menu. Print it out using the “Print all” option under the “output control”

Multi Scan
1. Under the “Acquire Setup” menu option and select “Setup Mult”.

2. Select “New” or “Previous” by clicking the right mouse button.

3. Type in the elemental peak in “element name”, “resolution” (Survey/utility/ HRES) and the number of “sweeps” for the peak.

4. To add other region of energy range (adding other elemental peaks) hit “Add region” and fill in the information

5. Hit Next Menu and fill in/select the information for Acquisition time, X-ray anode and Multipak clone

6. Hit “Acquire” to acquire the data.
Removing the sample from the chamber

1. Set the radio button for “Neutralizer Control” to standby.
2. Set the radio button for the “Ion gun control” to off.
3. Wait for few minutes after the extractor pressure to falls to ~0.0 mPa.
4. Pull the Neutralizer arm out, not past the metallic ring on the arm.
5. Select the “Stage Control” under the “Sample Setup” Menu.
6. Hit Dock Stage.
7. Make sure that the pressure in the main chamber ~ 2-3x 10^-9 Torr.
8. On the Auto Valve Control hit “Pump Intro” and wait for few minutes.
9. Then hit Intro Sample on the Auto Valve Control and push the forked arm in to grab the sample.
10. On the PC-Access Menu select “Unload stage” and pull the forked arm with sample all the way out. Make sure that the valve is closes on pulling the arm out.
11. Hit “Back fill intro” and remove your sample. Put back the cover and hit “pump intro”
12. Wait for all the LEDs on Air-HiVac display to turn on. Set the Auto valve control to standby.

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