

Procedure for General Use of the XPS

Saturday, April 10, 2004

Loading a sample into the XPS:

1. Fill out the XPS log book on the computer desk. Make a note of the pressure readings on the control panel over the XPS and the time you sign in.
2. Read and note in the log book the following: Pressure and temperature of the Neslab circulator in the back corner of the room. The pressure, temperature and flow rate of the D & W circulator under the glove box. The flow rate of the overhead flow meter that is covered with Al foil. The pressure of the cryo pump.
3. Check that the X-ray High Voltage is on (Glassman High Voltage Power supply: meter function 12 KV; Spot size: remote). **If the high voltage ps is not on:** off the Glassman HV PS and the 8702 X-ray controller. Wait about 30 seconds. Turn on the 8702 X-ray controller and check if the interlock ready light is on. If not call the GLA. If the interlock light is on push the preheat button. It should light up. Wait until the read light comes on about 2 minutes. Turn on the Glassman HV ps. The HV should go to about 10 KV.
4. Mount your sample on the XPS stub. Public use stubs should be in a sealed box next to the sink. If you are a regular user, you should purchase your own stub to avoid contamination. Stubs should be handled only while wearing gloves, and should be stored in a clean, sealed container. Please do not remove stubs from the XPS room. Samples should not overlap the edges of the stub by more than a few mm.
5. Check the control panel to make sure that **all gates are closed EXCEPT Gates 2 (load lock/tubo) and 6 (XPS/cryo) are closed. Gates 2 and 6 should remain open.** Check the pressures in the small cross and XPS chamber. Unusually high pressures (ca. 10^{-8}) could indicate a problem with the instrument and should be brought to the attention of the GLA.
6. Read the pressure in the load lock. This is done by pushing the soft button labeled "IG2/CG2 SELECT" on the control panel. The panel will now indicate the pressure inside the load lock. When the load lock is pumped down, the pressure will be 10^{-4} (torr, the lowest the capacitance gauge reads).
7. **Close Gate 2** (load lock/tubo). Turn off the load lock turbo pump. Press the yellow "ON/OFF" button on the left side of the control panel labeled "Load Lock Turbo Pump" to **turn off the load lock turbo. Open Gate 3** (load lock/N₂ inlet gate indicated by a gas cylinder symbol on the control panel). The pressure in the load lock will increase as N₂ fills the chamber. When the pressure reads approximately 760 torr, open the door to the load lock. When

the load lock is coming up to atmosphere, the pressure in the small cross tends to rise to 10^{-8} or even 10^{-7} . This is normal.

8. Place the stub onto the load lock arm. The stub has a freely rotating spindle below a short, cylindrical collar. The top notch of the spindle should fit snugly into the arm. Only use one of the three slots facing sideways. The forward looking slot is unusable. Close the load lock door.
9. While holding the door closed, close Gate 3 (load lock/ N_2 inlet gate), and **open Gate 2** (load lock/turbo gate). This should establish a vacuum strong enough to hold the door closed. Turn on the turbo pump by pressing the yellow button under the “Load Lock Turbo Pump” sign on the control panel. The pressure inside the load lock will decrease rapidly to 10^{-4} . Once it reaches this point, **wait 30 minutes** to allow the load lock to pump down before moving the stub.

Moving a sample into the XPS chamber:

1. **After 30 minutes** have elapsed, **open Gate 1** (load lock/small cross).
2. Manually **open Gate 8** (small cross/LEED).
3. Using the controller with a speed knob and a forward-backward switch move the motorized transfer-arm stored behind Gate 8 into the small cross. Position the sample on the load lock arm in the small cross just in front of the motorized arm. Maneuver the prong of the motorized transfer-arm slowly forward so that the prong on the transfer arm is around the stub spindle. Move the transfer arm very slowly onto the stub; **do not bend the load lock arm**. Lock the stub into the prong of the transfer arm by rotating the transfer arm base 180° . Very slowly pull the stub off the load lock arm by moving the transfer arm back away from the XPS chamber, keeping a close eye that the stub is securely locked. If the stub begins to slip as if it was not securely locked, push the stub back on the load lock arm and try again to lock the stub on the transfer arm. Once the stub is securely on the transfer arm, move the load lock arm back into the load lock.
4. **Close Gate 1** (load lock/small cross). (Note that Gate 5 (small cross/XPS) to the analysis chamber and Gate 1 to the load lock cannot be open at the same time. Opening Gate 5 before withdrawing the load lock arm from the small cross will cause Gate 1 to close on the load lock arm, bending it.)
5. Log into the XPS computer to access the motion control system. You may obtain the user ID and password from the GLA. Open the program ESCA200 on the desktop.
6. . Check that **Gate (switch) 7** (XPS stage) is **closed**. This will lower the XPS stage into the position that will accept the stub. (If it is open close it) Lower the XPS stage with the joystick till it is fully down.

7. **Open Gate 5** (small cross/XPS). Use the transfer arm to move the stub onto the stage in the XPS chamber. If the slot on the sample stage does not line up with the stub, move the sample stage using the stage joystick. Moving the joystick in the x and y directions will move the stage in the same direction. Pressing and holding the button on the joystick knob and moving the joystick up and down will move the stage up and down. To mount the stub on the stage, the stage must be fully down. When placing the stub on the stage, proceed very slowly. The stub is fully on the stage when the collar above the stub spindle completely overlaps the semicircular wear mark on the stage. When the stub is settled onto the stage, unlock the clamp of the cross-arm by turning the transfer arm base 180°.
8. **Move the transfer arm back behind Gate 8.**
9. **Close Gate 5** (small cross/XPS).
10. **Close Gate 8**(small cross/LEED).
11. **Open Gate 7**(XPS stage) . This lifts the stage into the appropriate position to collect data.

Collecting XP spectra:

1. Turn on the microscope light power supply located on top of the box that contains the switches for the gate values and the pressure readings that is above the instrument.
2. Focus the microscope on the sample by moving the sample stage along the x, y, z and rotational axes with the joystick. The x, y, and z axes are moved as described in 7 above. The stub is rotated by twisting the joystick knob in the direction you wish to rotate. The focus should be done with the microscope set at 50x.
3. Open the program on the desktop called ESCA2000, and then open your personal database.
4. To collect a survey scan of the sample, make sure the “recipes” button is selected in the upper left-hand corner of the screen. Select the project and then recipe file that contains the appropriate scan parameters. Recipe parameters are shown in the right-hand dialog box. Along the far right-hand side of the box are buttons that add, delete, and move regions within a recipe. Following are brief descriptions of some common region parameters that are used often:
 - a. Survey scan from 0-1000 BeV
 - i. Function: Survey
 - ii. LBE (low binding energy): 0
 - iii. Window width: 1000

- iv. Spot size: 800
- v. Scans/Time: 2
- vi. Res #: 4
- vii. eV/step: 1

b. Detailed scan of a particular peak: method 1

- i. Function: UnScanned
- ii. CBE (center binding energy): 101.5 (for si 2p)
- iii. Window width: 8
- iv. Spot size: 800
- v. Scans/Time: 10:00
- vi. Res #: 2

Note: although you are asked to set a window width, the actual data collection window is determined by the detector width, which is set by the resolution number. The detector widths are posted on the glass window next to the computer.

c. Detailed scan of a particular peak: method 2

- i. Functions: ResHi
- ii. CBE: 101.5
- iii. Window width: 8
- iv. Spot size: 800
- v. Scans/Time: 20
- vi. Res #: 2
- vii. eV/step: 0.065

Note: in this function, setting the window width does determine the width of the data collected.

5. The last region in every scan must have the “spot size” set to “Off.” This is the only way to turn off the X-ray source.
6. In the lower right-hand region of the screen, name your project and experiment. You may add a description of your experiment if desired. Avoid using parenthesis, colons, semicolons, dashes, or underscores in your file names.
7. Choose the button labeled “Run” at the top of the screen.
8. At the end of the scan, the file may be opened by selecting the “Experiment” button in the upper left-hand corner of the screen. Data may be analyzed using a variety of techniques given in the data reduction program. See the GLA for more information.
9. Export data to an Excel file. Select which experiment to export, then choose Export from the File menu. Select “EXCEL” in the dialog box. After several seconds an Excel file will appear with every region of your scan in a different sheet of the workbook. Save this to a disk. You are responsible for exporting

and saving your own data; the MMRC and the XPS GLA are not responsible for lost data.

Unloading the sample:

1. **Close Gate 7**(XPS stage) to lower the stage.
2. **Open Gates 5** (small cross/XPS) and **8** (small cross/LEED) and use the transfer arm to move the sample into the small cross. Note that the stub will probably not line up with the transfer arm slot, and the stage will have to be moved using the joystick.
3. Move the sample into the small cross with the transfer arm. **Close Gate 5** (small cross/XPS).
4. **Open Gate 1** (load lock/small cross) (remember that Gates 1 and 5 (small cross/XPS) cannot be open at the same time) and move the sample to the load lock arm (use only one of the three the sideways pointing slots. The forward slot is nonfunctional). Unlock the transfer arm by turning the base of the arm 180°. **Retract the transfer arm behind Gate 8** (small cross/LEED), being careful to leave the stub securely on the load lock arm. Using the load lock arm, move the sample into the load lock.
5. **Close Gates 1** (load lock/small cross) and **8** (small cross/LEED).
6. **Close Gate 2** (load lock/tubo), closing off the turbo pump to the load lock.
7. **Turn off the load lock turbo** pump by pressing the yellow button marked “Load Lock Turbo Pump.”
8. **Open Gate 3** (load lock/N₂ inlet) (indicated by a gas cylinder symbol on the control panel). The pressure in the load lock will increase as N₂ fills the chamber. When the pressure reads approximately 760 torr, open the door to the load lock. **Remove stub.**
9. **Close load lock door.** Hold load lock door shut and **open Gate 2** (load lock/turbo). The vacuum should hold the door closed. **Turn on load lock turbo pump** by pressing the yellow button marked “Load Lock Turbo Pump.”
10. **Log off** the computer, sign out in the logbook and note any problems or observations while using the instrument. All problems must be noted in the book, otherwise other users may have problems. The only unforgivable mistake is not making note of any problems.

Loading a sample into the glove box from the UHV:

1. Place the sample on the load lock arm. This can be done either internally in the small cross or externally in the load lock.
2. **Open Gate 9** (small cross/glove box) manually.

3. Move the sample to the locking arm in the glove box load lock. Lock the sample in place by twisting the locking arm clockwise until it clicks.
4. Pull the load lock arm back into the load lock, and **close Gates 9** (small cross/glove box) and **1** (load lock/small cross).
5. **Turn off the glove box load lock turbo pump** by pressing the yellow button on the control panel labeled “EC Turbo @ glove box.” This is located at the bottom right side of the XPS table. The pressure in the lock is displayed on the free-standing control panel to the left of the XPS. The pressure is an average of the middle and bottom numbers displayed near the bottom of the panel.
6. When the pressure in the lock is about 760 Torr (this can take several minutes) **open glove box load lock door** from inside the glove box. Use the long metal prong stored inside the glove box to grab the sample. Walk around to the lock arm and unlock the prong by turning the arm counter-clockwise. **Move the sample into the glove box**, and **close the glove box lock door** inside the glove box.
7. **Turn on the glove box load lock turbo pump** by pushing the yellow button on the control panel labeled “EC Turbo @ glove box.”

Loading a sample into the UHV from the glove box:

1. **Make sure all gates are closed EXCEPT Gates 2** (load lock/tubo) and **6** (XPS/cryo).
2. Follow, in reverse, the procedure for loading a sample into the glove box from the UHV.
3. Wait for the pressure in the glove box load lock to pump down to 0, and then pump-down for an additional 30 minutes before moving the sample.
4. Sign into the logbook.

Updated by Lauren Webb, 8 August 2003

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