Procedure for General Use of the XPS

Loading a sample into the XPS:

1. Fill out the XPS log book on the computer desk. Make a note of the pressure readings of IG1 and IG2 on the valve control panel over the XPS, the time you sign in, and your sample material. Unusually high pressures (ca. IG1 > 10^{-7} or IG2>10^{-8} Torr) could indicate a problem with the instrument and should be brought to the attention of the GLA.

2. Check the control panel to make sure that all gates are closed EXCEPT gates 2 (load lock/tubo), 6 (XPS/cryo) and possibly gate 1. Gates 2 and 6 should remain open (lights on) and the XPS stage (control 7) can be up or down. Note that there is a Cryo pump (gate 0) on the load lock that is controlled by a switch attached to the left side of the XPS table.

3. Read the pressure in the back chamber above the turbo by looking at the pressure gage sitting above the regular pressure gages above the control panel for the gates. When the load lock is pumped down, the pressure will be ≈10^{-4} (the lowest the capacitance gauge reads) or 0 Torr. If the pressure is > 10^{-4} you will need pump down the load lock (see step 11-13). The actual pressure in the load lock, if it is below 10^{-5} Torr, is observable on the left ion gage above the control panel.

4. Check that no samples are in the instrument in either the analysis chamber or the load lock.

5. If gate 1 is closed, open it now.

6. Mount your sample on the XPS stub. Public use stubs should be in glass jars on the counter. 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.

7. Wipe the stub with a Kimwipe moistened with MeOH or isopropanol before use. If you must use carbon tape (only UHV carbon tape is allowed) use only a small amount and do not load more then 3 samples with carbon tape at one time.

8. Turn off IG1

9. To bring the load lock up to atmospheric pressure,
   • Close the gate valve to the load lock cryo pump, gate 0 (switch left side of XPS table).
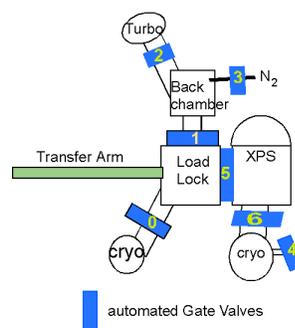
- Turn off the load lock turbo (Pfieffer) pump by pressing the "ON/OFF" button on the Pfieffer controller to the left of the valve control panel. The mechanical vacuum pump on the floor will turn off automatically.
- Close gate 2 (load lock/turbo) and wait until you hear the gate click shut.
- Open gate 3 (load lock N_2). The pressure in the load lock will increase as N_2 fills the chamber. When the pressure reads approximately 760 Torr, the door to the load lock should open. To easily remember these steps think "0,off,2,3,".

10. Place the stub onto the load lock arm. The stub has a freely rotating spindle below a short, cylindrical collar. The bottom notch of the spindle should fit snuggly into the arm. Rotate the magnet on the arm to hold the stub in place. Close the load lock door.

11. **Check that the turbo is stopped.** If it is not, wait until it is. The speed of the turbo is shown on the turbo controller. It takes about 5 minutes for the turbo pump to stop spinning.

12. Close gate 3 (load lock N_2) and open gate 2 (load lock/turbo). While holding the door closed, turn on the turbo pump by pressing the "On/Off" button on the right side of the "Load Lock Turbo" control panel(3,2,on, 0). Watch the pressure in the back chamber; it should decrease continuously to the low 10^{-3} Torr range. The turbo pump occasionally will hang at a pressure, if this persists for 30 seconds or more, turn off the turbo pump, wait for it to spin down, and restart it.

13. After a few minutes, the turbo pump should reach maximum speed (1500 Hz) and stabilize in power (0-4W).

14. When the pressure reaches $< 3 \times 10^{-5}$ Torr, you can open the gate 0 to the load lock cryo and close gate 1. At this point it is acceptable to leave the lab as the samples should remain under the cryo pump vacuum for a minimum of 30 minutes.

Loading Samples Summary
1. Check no sample in XPS
2. Open gate 1
3. close gate 0
4. turn off IG1
5. Pfeiffer off
6. close gate 2
7. open gate 3
8. load stub;
9. Wait for Pfieffer to stop
10. close gate 3
11. open gate 2
12. Pfeiffer on
13. wait for back chamber $P < 10^{-5}$ Torr
14. open gate 0
15. close gate 1
16. Turn on IG1
17. wait ½ hour till IG1 $< 10^{-7}$ Torr

Moving a sample into the XPS chamber:

1. **After 30 minutes** turn on IG1 and check the pressure in the load lock. It should read $<1\times10^{-7}$ Torr. If not wait longer.

2. Check that **switch 7** (XPS stage) is **down** (light is off).

3. Open gate 5 (to XPS chamber). This gate may take 10 s to fully openUse the transfer arm to move the stub into the analysis chamber, stop the magnet of the transfer arm at the beginning of the 'CAUTION" region on the arm. Check that the slot on the sample stage aligns with the stub. Move the sample stage using the stage joystick to align the shaft of the stub and the sample stage. Moving the joystick in
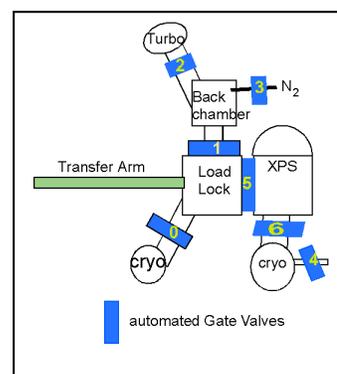
the x and y directions will move the stage in the same direction. Holding the button on the joystick knob and moving the joystick up and down will move the stage up and down. To mount the stub on the stage, the stage must be lowered from the data acquisition height. The top of the stage should be positioned slightly lower then the bottom of the top collar of the stub (see figure below). Move the transfer arm slowly when placing the stub on the stage. The stub is fully on the stage when the collar above the stub spindle completely overlaps the semicircular wear mark on the stage. Unlock the stub from the transfer arm. While watching the stub on the sample stage, withdraw the transfer arm. If the stub starts to move push it back onto the stage and try again. If the sample stage still moves call a GLA.

4. Move the transfer arm back to the load lock chamber.

5. Close gate 5 (to XPS chamber).

6. Raise switch 7. This lifts the stage into the appropriate position to collect data.

Collecting XP spectra:

1. The computer should be on, if not the computer is located behind the XPS detector, log in using the ID and password posted on the monitor. If the computer is restarted you will need to reengage the joystick controls. To do this start the "ESCA Capture" program and go to View=>Motion Control Pnl. Click on the button labeled "Joystick", it should change from red to green.

2. Turn on the desk lamp.

3. Focus the microscope on the sample by moving the sample stage along the x, y, z and rotational axes with the joystick. The focus should be done with the microscope set at 50x. The magnification setting is indicated by the black dot on the side of the microscope, and not the number facing forward.

4. Note: take care not to raise the stage excessively, as this can cause the stub to become dislodged and fall off the stage. The back part of the bellows (to the right of the ESCA chamber) should be about even with the front. There are limits to how far forward and back the stage can be moved. To get a sample in view it sometimes helps to rotate the stage.

5. Open the program on the desktop called ESCA Capture, and then open your personal database.

6. Make sure that the 9600 (on the electronics rack) has the button for computer is lite (push of off).

7. In the ESCA Capture program go to View and choose ESCA control panel. Make sure X-ray Gun Operate is checked.

8. 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. 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.

c. Detailed scan of a the Si 2p peak: method 2
   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. This is not as good as the ResHi mode since it depends upon the linearity of the detector for good data; however, it is somewhat faster.

d. Normally it is best that the resolution and spot size go together as shown in the table below

Spot Size	Resolution
800	4
500	3
300	2
100	1
L1 (100x400)	1
L2 (250x1000)	2
L3 (500x1200)	3

9. **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.**

10. Save your recipe.

11. In the lower right-hand region of the screen, name your project and experiment. You can add a description of your experiment. Avoid using parenthesis, colons, semicolons, dashes, or underscores in your file names.

12. Make sure X-ray Gun Operate is checked in the ESCA control panel.

13. In the main program window choose the button labeled "Run" at the top of the screen.

14. 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. The data is saved automatically and will only be removed if you save over it; we recommend that you periodically backup your database/protocol to a thumb drive. Data may be analyzed using a variety of techniques given in the data reduction program. See the blue binder or a GLA for more information.

15. Export data to an Excel file. This must be done in the ESCA Analysis program. Select which experiment to export, and 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.

16. When you are done you can uncheck the X-Ray Gun Operate in the ESCA control panel.

Unloading the sample:

1. Lower switch 7 to lower the stage.

2. Check that the load lock is at low pressure $P < 10^{-7}$ Torr.

3. Open gate 5 and place the stub on the transfer arm. Move the stage as needed to align the stub with the transfer arm. The bottom of the transfer arm fork should be positioned slightly higher then the top of the bottom collar of the stub (see figure below). Turn the magnet 180° to lock the stub on the transfer arm.

4. Retract the transfer arm into the load lock and close gate 5 (to XPS chamber).

5. Open gate 1 (load lock/back chamber), turn off IG1,

6. Turn off turbo, close gate 2, open gate 3; (1, off, 2, 3).

7. Close gate 0 (load lock cryo pump).

8. Remove stub

9. Wait for the turbo has stopped spinning,

10. Close gate 3, open gate 2, turn turbo on (3, 2, on).

11. Sign out in the logbook and note any problems or observations while using the instrument.

12. When the turbo reaches full speed and the pressure is $< 10^{-5}$ Torr open gate 0 and close gate 1.

13. When you leave, the load lock should be under stable vacuum with the turbo pump at max speed,

    a. gates 2, 6 & 0 should be open and

    b. all others (1, 3 4, 5) should be closed, and IG1 and IG2 should be reading normal low pressures (mid 10^{-9} Torr or lower).

Send questions to

David Gleason-Rohrer: gleason@caltech.edu or in emergencies by cell phone (619)-757-8138

Joseph Beardslee josephab@caltech.edu or office

Judy Lattimer jlatt@caltech.edu or office x2789

To use the glove box connected to the XPS please contact a   GLA.

automated Gate Valves

top collar

Bottom collar
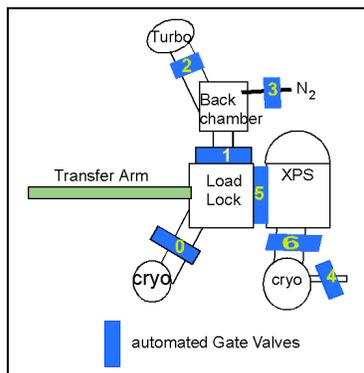
Unloading Sample Summary

1. check IG1 $< 10^{-7}$ Torr
2. open gate 5
3. lower stage
4. move sample from XPS stage to arm
5. move sample to load lock
6. close gate 5
7. open gate 1
8. close gate 0
9. turn off IG1
10. turn off Pfeiffer
11. close gate 2
12. open gate 3
13. remove sample
14. wait for Pfeiffer to stop
15. close gate 3
16. open gate 2
17. start Pfeiffer.
18. Wait for P $< 10^{-5}$ Torr
19. Open gate 0
20. Close gate 1