# SSX-100 Standard Operating Procedures

## 1 Introduction

You must be trained and authorized to operate the SSX-100 independently. These instructions are intended to supplement that training. The instrument has some idiosyncrasies and occasional hardware or software glitches. **If you encounter an unusual circumstance and are not certain how to proceed, you must stop and contact the instrument manager.**

**FOM USAGE**: You are required to reserve time on the instrument through Facility Online Manager (FOM) at fom.ccmr.cornell.edu. Your reservation time encompasses the time between sample loading and sample retrieval from the Airlock.

1. You should Reserve time on the instrument starting when you want to load your samples into the Airlock and until you remove your samples from the XPS instrument.
2. You should Log-ON in FOM when you transfer your samples from the Airlock to the Analysis chamber.
3. You should Log-OFF in FOM only you are completely finished with using the instrument or XPS computer.

**Data Retrieval**: You cannot use any USB or other external drives on the XPS computer. Therefore, you cannot retrieve data using any external drive. You can retrieve data by using e-mail or your Cornell BOX account.

### 1.1 Important cautions

1. Any unexpected pressure rise should be treated with extreme caution, turning off the 9600-filament controller or hitting the red 'kill switch' and contacting the manager. The biggest concern is a leak at the water-cooled anode which could essentially destroy the instrument
2. The detector must be kept under vacuum. Extended exposure to moister will ruin the microchannel detector plate
3. Do not vent the load lock if an orange cable labeled 'Do not vent load lock, ion gun in use' is connected to the vent toggle. Venting the load lock in this case will also vent the ion gun and analysis chamber, and will probably result in damage to the instrument.

## 2 Procedures

### 2.1 Preparing Samples

XPS analysis is done in an ultra-high vacuum environment. The technique is extremely sensitive to surface contamination. You must take care to keep all vacuum components exceptionally clean and keep the surface of your sample pristine.

1. Wear powder free gloves while preparing samples and handling equipment that goes into the vacuum system.
2. Clean tweezers or other tools with IPA before manipulating samples or sample pucks.
3. Do not put volatile materials in the chamber without approval from the instrument manager (contact ccmr-xps@cornell.edu).
4. Leave the sample prep area clean.

### 2.2 Transferring samples to or from the load lock chamber:

1. Open the load lock chamber:
    a. Close toggle 2 to isolate the turbo pump
    b. Turn off the Hornet ion gage
        i. Press the menu button
        ii. Press ion gage
        iii. Press FP Operate
        iv. Press ON/OFF-> arrow to OFF -> enter

c. Open toggle 3 to vent the load lock.
d. When the MKS pressure gage reaches 740 Torr, close Toggle 3.

2. While wearing gloves, install or remove the sample puck. The puck should sit in the carriage on the top groove of the puck's base. The bottom grove mates with the transfer arm, which slides under the carriage.
   a. 3.5'' pucks should go in position 3.
   b. 2'' pucks should go in positions, 1,3, or 5.
   c. 1'' pucks can go in any position.
   d. 2'' inert sample transfer puck must go into position 1 (closest to the load lock door).
      i. Remove the door to the load lock and set it on a chair.
      ii. Start with the puck disconnected from the base, resting loosely in position 1.
      iii. Loosen the linear manipulator and extend the rod into the chamber.
      iv. Push the carriage into the load lock chamber and puck up the puck with the fork.
      v. Install the puck base in position 1.
      vi. Push the puck down to install it on the base. You will need to shift the puck as far to the right in the fork as possible to line the puck with the shaft.
      vii. Tighten the set screw to firmly attach the puck to the base.
      viii. Close the door by turning the carriage knob and letting the door gently follow. The trick here is that the position of the puck is now constrained by the fork, opposing the carriage and the load lock door. You need to close the load lock door while maintaining the position of the puck in the fork.
      ix. Item 5 below proves instructions on opening the inert sample puck.
3. Close the load lock:
   a. Check the o-ring on the load lock door to make sure it is seated securely.
   b. Close the load lock door.
   c. Turn on the scroll pump using the switch on the power strip.
   d. Open the black scroll pump vacuum valve, a few turns are plenty.
   e. If the MKLS vacuum gauge does not drop to 0 Torr, there is a problem. Do not proceed and contact ccmr-xps@cornell.edu.
4. When the MKS vacuum gauge reads 0 Torr.
   a. Close the scroll pump valve.
   b. Open Toggle 2 to begin pumping to high vacuum with the turbo pump.
   c. Turn off the scroll pump using the switch on the power strip.
   d. Turn on the Hornet ion gauge
      i. Press the menu button
      ii. Press ion gage
      iii. Press FP Operate
      iv. Press ON/OFF-> arrow to ON -> enter
5. If you are using the inert sample puck:
   a. While holding the linear manipulator in place, loosen the manipulator thumbscrew (pressure difference will try to force the rod down).
   b. Pull the linear manipulator up. The cap should pop free.
   c. Tighten the linear manipulator thumbscrew.
   d. Leave a note on the load lock door that the inert sample cap needs to be removed. Don't forget to remove it when you remove your sample.

## 2.3 Transferring samples from load lock to analysis chamber

1. Rotate the dial on the load lock door to translate your puck into the transfer position. There are stickers with arrows to indicate the optimal alignment. Use the video monitor to identify your sample puck.
2. Ensure the analysis chamber gate value is closed (toggle 5 down).
3. Pick up the sample puck with the transfer arm:
   a. With the transfer arm magnet facing down, slide the magnet forward while watching your sample on the monitor.
   b. You should feel the transfer arm engaged with the puck. The magnet should be aligned with a score mark on the transfer arm vacuum tube.

c. Rotate the magnet so the flat side is face up.
4. Open the analysis chamber gate valve (toggle 5 up).
5. Slide the manipulator magnet towards the analysis chamber whole watching the analysis chamber monitor:
   a. Insert the puck into the analysis chamber stage. The sample holder should drop down into place and the magnet should be close to, but not touching, the 2nd score mark on the transfer arm vacuum tube.
   b. If the puck does not easily engage with the stage, don't force it. Back it off and check that the sample stage is in the home position.
6. Rotate the transfer arm magnet so the flat side is facing down.
7. Fully withdraw the transfer arm to the end of its travel, so that the sample arm is clear of the analysis chamber.
8. Close the analysis chamber gate valve (toggle 5).
9. Shift the analysis chamber stage to the measurement position (Toggle 7 up).

## 2.4 Transferring samples from Analysis chamber to load lock

1. Shift the analysis chamber stage to the transfer position (toggle 7 down).
2. Make sure the sample stage is in the home position, using the joystick:
   a. Move the x stage to line up with the marks
   b. Move the y stage to line up the marks
   c. Move the z stage to line up the arrows
3. Make sure the load lock pressure is less than 1e-5 Torr.
4. Make sure the X-ray source is on standby:
   a. If the source is on, open the ESCA control panel and click the X-ray operate box 3 times and then leave it unchecked (3 times is necessary to disable to source but leave the filament on).
5. Rotate the transfer arm magnet so the flat side is facing down.
6. Align the load lock sample carriage, using the arrow on the dial on the load lock door. Remember the appropriate transfer positions:
   a. 3.5'' pucks should go in position 3
   b. 2'' pucks should go in positions, 1,3, or 5
   c. 1'' pucks can go in any position
7. Open the analysis chamber gate valve (Toggle 5 up).
8. Use the transfer arm magnet to shift the transfer arm into the analysis chamber. You should feel it engage with the puck and see it engage on the analysis chamber monitor. The magnet should be close to the 2nd score mark on the transfer arm vacuum tube.
9. Rotate the transfer arm magnet so that the flat side is facing up.
10. Slowly withdraw the transfer arm to disengage the puck from the analysis chamber sample stage.
11. As the sample enters the load lock, watch the load lock monitor, When the puck finally touches the puck carriage and is clear of the analysis chamber gate valve, close the gate valve. That way if the carriage alignment is off and the puck pops out of the transfer arm, we don't have to vent the analysis chamber to retrieve the puck).
12. Shift the transfer arm to seat the puck in the load lock carriage.
13. Rotate the transfer arm magnet so the flat side is facing down.
14. Fully withdraw the transfer arm to the end of its travel.

## 2.5 Turning the X-rays off

1. When turning off the x-ray source, it is important to leave the filament on. Otherwise the next user will experience arching for 15-20 minutes when they turn on the source.
2. In the ESCA control panel, click the Xray Operate box 3 times until unchecked.

## 2.6 Turning X-rays on

1. Computer operation (Standard):
   a. In the ESCA control panel, check the Xray operate box in the ESCA control panel 3 times
   b. After the Glassman has ramped to maximum voltage, all green lights should be lit. You should be ready to go.
2. Manual Operation (Unusual):

a. The steps in this section are only required in the unusual circumstance that the filament and x-rays are off. Line letters are correspondingly labeled on the electrons rack.
b. Press "HV ON", if not already on.
c. Turn on the 9600 Controller, if not already on.
d. Press START FILAMENT if the green FIL ON light is not on. When the green FIL ON and 2KV ON lights are on, and the filament is in STANDBY mode.
e. Press OPERATE. The green 10KV ON light will light after the Glassman voltage ramps
f. Selecting a spot size other than OFF will turn on the green XRAY ON light.
g. Press COMPUTER on the 9600 to enable remote commands.

3. If the filament does not turn on, do not proceed. Contact ccmr-xps@cornell.edu.

## 2.7 Aligning Samples

1. Looking into the main chamber from above using the small viewport, use the joystick to center your sample in the illumination spot from the microscope:
   a. Samples on solid pucks move in the X, Y, and Z direction.
   b. Samples on rotating or stilting stages will move in the X,Y,Z, and θ.
2. Adjust the height, using buttons 3 and 6 on the joystick, to bring the sample into focus in the microscope:
   a. When the sample is in focus, the crosshair will indicate approximately where the X-ray hits the sample.
   b. 800 μm source size (~1x2 mm spot at the sample) is ~4.4 divisions wide and 2.2 high at 25x magnification and ~2.2 high and ~1.1 divs wide at 12x).
3. You still need to fine-tune the height of the sample to the point where the focus of the analyzer and the x-rays converge:
   a. Open the ESCA control panel.
   b. Set he center binding energy (CBE) to 533 for oxygen or 285 for carbon.
   c. Set the spot size to 800 for the largest X-ray spot and the highest count rate.
   d. Set resolution to 4 (150 V pass energy) for highest count rate.
   e. Scan box should be unchecked.
4. Press START (if the resolution has changed, there is a 10 second delay).
5. You may see an oxygen peak in the window at 532 eV. If not:
   a. Your sample may be insulating. Press ABORT and enable the flood gun neutralizer in the ESCA control panel. You may even need to adjust the flood gun energy using the potentiometers on the 8711 Charge Neutralizer panel, but this is usually not necessary.
   b. Your sample does not contain oxygen. Se the energy in the ESCA to look for another element like carbon.
   c. The sample may not be at the correct height. Raise or lower the sample using the joystick buttons 3 and 6 to optimize the count rate on the 8713 Rate Meter.

## 2.8 Preparing Measurements

Measurements are defined by preparing sequences of scans, which the Hawk acquisition program calls "recipes". You can define an individual recipe and then use it to measure the current sample location, or program the instrument to apply one or more recipes to one or more positions on the sample(s)

### 2.8.1 Preparing individual recipes

1. Click on a recipe and then go to "File/Save Recipe As". Save it in the "Sample Project" category. This will be your new template. Do not use names longer than 29 characters, or the data will not be saved.
2. Modify the individual scan paraments in the recipe based on your observation scans
   a. Add, delete, or move scans using the buttons on the right of the recipe window
   b. You can press TEST REGION and the highlighted line will be sent to the ESCA control panel and begin scanning. In the ESCA control panel, you can press update region to send the parameters back to the highlighted line in the recipe.
   c. Enter the transition information in the box under the window width. We recommend the convention "C 1s" for high-res scans, "HS C 1s" for high-sen scans, and "survey" for survey scans.
3. Use the ESCA control panel to perform "Observation" scans to determine parameters like the lower/central binding energy (LBE/CBE), window width, number of scans, and if the flood gun is required.

a. To determine if the flood gun is necessary:
    i. Uncheck the “scan” box
    ii. Set the energy to 285 for carbon or another known element if necessary
    iii. Use resolution 4
    iv. Press start
    v. If you see the peak drift to higher binding energy over the course or one or more such measurements, the sample is charging and requires the flood gun. If you don’t see a peak at all, try running the above steps with the flood gun on, your sample may be so strongly insulating that the peak has shifted out of the detector window.
    vi. It is ok for the peak to appear at lower than expected binding energy, this is common with the flood gun, it just shouldn’t drift over time.

4. For **survey scans**:
    a. Use resolution 4
    b. Any spot size (generally 800)
    c. Set the LBE to 0, window width to 1100
    d. Step size 1 eV
    e. Time/step 100
    f. Scan box should be checked
    g. Press start
5. For **high-resolution scans**:
    a. Use resolution 2
    b. Scan box checked
    c. Set **CBE** and window for peak of interest
    d. Step size 0.065 eV (default)
    e. Press start
    f. CBE, window size, may need to be adjusted. For good signal to noise, determine the number of scans necessary to obtain 2,000 counts above the background (very rough rule of thumb)
6. For **high-sensitivity scans**:
    a. Use resolution 4
    b. Scan box checked
    c. Set **LBE** and window for peak of interest (NB: this scan type is defined by the **lower binding energy**, not the central binding energy.)
    d. Press start
    e. LBE, window size, may need to be adjusted. For good signal to noise, determine the number of scans necessary to obtain 2,000 counts above the background (very rough rule of thumb)
7. If you are running an individual recipe, the last scan in the recipe should be the following:
    a. Unscanned 85 eV, type in “off” for the spot size
    b. Neutralizer “off”, if you are using the flood gun
8. Set the Project and Experiment names:
    a. Do not use names longer than 29 characters, or your data will not be saved.
    b. For the project name, we recommend the convention netid_YEAR-Month-Day, using the date samples were submitted.
    c. For the experiment name, we recommend the convention YEAR-month-day_AR_FG, where the date indicates the date the measurement was executed, AR indicates as received, (IC10s would indicated a 10s ion cleaning before the measurement) and _FG is only included if the flood gun is used.
    d. Press “Run” to run the recipe.

### 2.8.2 Applying individual recipes to a series of points or samples

1. In the Sample Project recipe section, open the ‘Default’ recipe, which is a PTM (multipoint) scan (if it does not exist, create one). Click on the pencil icon and the position windows will open. Click the “Recipe radio button so the recipes show. Click the ‘Update’ or “Auto Add’ buttons depending on if you want to update an existing line with the current X,Y,Z,R positions or add a new line. When you press the ‘get position’ button on the screen or the joystick, the current positions will be entered.

2. Add a final, extra line. Your last scan recipe should be GLASSMANOFF. This will turn off the flood gun and the Glassman to the lowest power. If your samples charge, you can add lines for turning the neutralizer on/off. You can just use the GlassmanOFF recipe to turn the neutralizer off, the next recipe will turn the Glassman on.
3. You should always go through all the positions from 1-N to see that the stage moves correctly. Clicking on the row number will prompt a move to that row positions. (Be sure to run positions in the proper order to look for problems).
4. Make sure you have the PMT recipe loaded (as opposed to the individual recipe) when you press 'RUN', otherwise only the recipe showing will run the current position. :POS01, :POS02, etc will be added onto the experiment name. The experiment name in total should not be more than 30 characters or it will not be saved in the database.
5. You can adjust and save visible recipes while the analysis is running and these changes will work for later positions that use the same recipe.

## 2.9 ARXPS (Angle-resolved XPS with in-situ tilt stage)

1. Inert sample holder into system with bulky end to the left and aligned correctly
2. Open Motion Control Panel. Three to one ratio for rotation to tilt: 165-degree rotation = 55-degree tilt.
3. Cations:
    a. Entering large angular changes (> 180) in the Motion Control Panel may initiate the tilt in the opposite direction and cause the problem below.
    b. Exceeding the allowable tilt axis range will cause the entire puck to rotate which may result in a collision of the puck with the flood gun or other parts of the analysis chamber.
4. Using the reflection of light from the sample, rotate/tilt the stage to maximize the reflection.
5. Zero the R-axis in the motion control panel.
6. Maximize count-rate on the flat sample by adjusting X.
7. Tilt the stage to 195 degrees (make sure to go in the negative direction, i.e. down to 195. degrees like you are starting at 350) and re-zero the R-axis.
8. Maximize count-rate on tilted sample by adjusting Y. Spot should now be centered on the axis of rotation.

## 2.10 Ion Sputtering

1. Prepare the 1401A Ion Gun controller:
    a. Black emission knob to Standby
    b. Beam OFF
    c. Turn on the Ion Gun controller power, you will see the gas pressure displayed on the far-left module.
2. Prepare the sputtering gas supply:
    a. Open the 'To Vac Manifold' toggle to pump out excess Argon from the line (located behind right cabinet door).
    b. Wait until the pressure drops below e-4 Torr on the Hornet pressure gauge.
    c. Close the 'To Vac Manifold' toggle.
    d. Open the 'To Ion Gun' toggle, which introduces gas to the gun and opens the differential pumping valve.
    e. Set the gas leak valve to ~115. The pressure slowly increases to ~6 mTorr in the ion source, as measured on the ion gun controller. Wait for the pressure to stabilize and then increase the gas leak valve to ~116 and wait until the target 7-8 mTorr is reached. Try not to exceed the desired flow rate or the leak valve must be turned all the way down to <60 and then back up.
        i. Gas flow to the ion gun may slowly decrease over several hours, lowering beam current. Increase gas pressure to compensate.
3. You need to ensure that the ion beam is aligned with the x-ray beam. Turn the ion gun raster off and perform a short exposure to locate the center position. The X and Y potentiometers on the ion gun controller are used to adjust the position (x is y, y is x).
4. For manual cleaning, set Energy, Emission, and Raster. Typical time to clean adventitious carbon from a sample surface is ~ 10 seconds @ 4kV. Flip Beam On/Off toggle on for desired time and then off.
5. For automated depth profiling:

a. Set up the depth profile scan in the ESCA control panel. The dwell time during sputtering will be the last used setting. If you need 100 ms, e.g., go to the ESCA control panel and start a scan using the 100 ms dwell time.
b. Make sure the ion gun check box is unchecked/OFF in the ESCA control panel.
c. Set Remote/Local to REMOTE on the ion gun controller.
d. Beam on/off to ON.
e. Emission current knob from Standby to 10 mA.
f. Check raster ON (normal size is 2x4 mm).
g. Set Ion Energy, typically 4000 eV.
h. Normally Condenser A for small spot, Condenser C for large spot/current.
i. When the Depth Profiling run starts, it will scan with 0 seconds of etching, then it will begin the first etch.
j. Note: pressing ABORT in Esca may flip the remote toggle.
k. Set the # of scans or click Abort on the computer to end. Either should turn off the ion gun if on.

6. When finished:
    a. Turn the leak value to <60
    b. Set Emission to Standby
    c. Beam on/off to OFF
    d. Remote/Local to LOCAL
    e. Ion Gun controller power OFF
    f. Close 'To Ion Gun' toggle (Extremely important!)
    g. Remove orange cord from interlock panel

## 2.11 Exporting Data

1. Exporting individual files to Vamas:
    a. Click on the Experiment button, find your project name and experiment.
    b. Go to File, Export. Select Vamas and save into the UserData folder.

## 2.12 Wrapping up

1. Make sure the load lock is under vacuum and being pumped by the turbo pump.
2. Clean up after yourself. The sample prep area should be clean and organized. Don't leave garbage, used tape, used gloves, etc. lying around.
3. You must take all of your samples with you when you leave. The CCMR does not dispose of samples.