Standard Operating Procedures for SSX-100 (updated June 2015)

These instructions are only guidelines, this cannot be used in lieu of proper training, and cannot account for any system changes or uncommon occurrences. Users must be familiar with the system lights, sounds, smells, etc. and let common sense prevail.

- Please work with other users to use the system efficiently. Use the whiteboard above the load lock to label samples, and put a sticky note on the load lock if necessary. Due to long pumpdown times, 1—2 users can use the system each day, 3/day is ambitious and would require coordination for loading/unloading samples.
- Teamviewer is available for remote desktop and file transfer. Use only if experienced.
- Web camera also available to monitor system electronics, see manager for details.
- Restarting the computer or ESCA program often turns the filament off, Xrays and filament must be restarted. Sometimes you cannot save recipe changes, restart the program. Restarting the computer is often the safest thing to do.
- If you are analyzing samples, you must be ENABLED in Coral as yourself. If someone else is enabled, you must still enable, which will automatically disable the previous user. If your run ends in the middle of the night, just let the instrument manager know, and the time can be adjusted.

Instructions below for:

Loading pucks into the load lock
Transferring samples to the analysis chamber
Transferring samples out of the analysis chamber
Turning X rays off
Turning X rays on
Setting up samples for analysis (including PTM/automated scans)
Exporting Data
Angle resolved XPS (ARXPS)
Ion Sputtering and how to turn off the Argon gas if you need to.

Loading pucks into the Load Lock (LL) from air, or vice versa:

1) To open the load lock chamber:
    a. **Close Toggle2** to isolate turbo pump
    b. Turn off the yellow LL Ion Gauge (follow instructions posted)
    c. **Open Toggle3** to vent
        i. When LL door opens (~30 seconds) **close Toggle3**
        ii. Wearing gloves, change samples out of LL
        iii. Put large-diameter holders into first or last positions
2) To pump out load lock:
    a. Turn on power strip switch for rough pump.
    b. Open the manual rough pump valve while holding LL door closed
    c. When LL pressure gets <=1 Torr,
        i. Close the manual valve
        ii. Turn off power strip
        iii. Open Toggle 2. The Pfeiffer Turbo speed may decrease slightly and then return to 1500Hz.

iv. Turn on LL ion gauge (follow instructions posted)

Inserting Samples into Analysis Chamber(AC) from the Load Lock(LL)

1) **If there is a sample puck in the main chamber, complete steps 13-22 before step 2**
2) **The LL chamber pressure must be ≤5x 10^{-6} Torr.**
3) X rays should be on standby, if not turn x ray HV off (but leaving filament on) by unchecking the Xray Operate box **3 times** in the ESCA Control Panel.
4) Make sure the Sample stage is down (**Toggle7 Down**) and empty
5) **The stage should already be aligned to home in X,Y,Z.** Use the external stage marks to verify. Use the solid Y line as the dotted line is the best line for stage travel in the +X direction.
6) Manipulator magnet should have the flat facing down, slide forward slowly while watching your sample.
7) Pick up sample in the LL and engage by rotating magnet flat up **before** opening the gate valve.
8) Open AC gate valve (**Toggle5 up)**
9) Move manipulator magnet towards main chamber viewing through load lock window or main chamber monitor.
10) When sample is inserted into the AC holder, rotate the manipulator flat to down, and withdraw the arm leaving the sample behind.
11) Withdraw manipulator arm fully and close AC gate valve (**Toggle5 down**)
12) Raise sample stage. (**Toggle7 up)**

Transferring Samples from the Analysis Chamber(AC) into Load Lock(LL)

13) **Make sure LL chamber pressure is < 5x10^{-6} Torr**
14) **If Xrays are on, turn Xrays to Standby by unchecking the Xray Operate box 3 times in the ESCA Control Panel**
15) Sample stage down (**Toggle7 Down**)
16) **Make sure the stage is aligned properly in X,Y,Z.** Use the external stage marks. Use the solid Y line as the dotted line is the best line for stage travel in the +X direction.
17) Manipulator magnet flat down
18) **Choose which of the five load lock slots you'll be using. 3" pucks go into the first or last slots. Move transfer arm in ~2" and align with the LL slot**
19) Open AC gate valve. (**Toggle5 up)**
20) Move manipulator magnet towards main chamber. When the sample is in the AC holder, rotate the manipulator flat to up, and slowly remove sample into LL while watching the sample in the viewport. Whenever possible, close the gate valve when the sample is clear of the gate valve.
21) **Carefully** withdraw sample into LL tray
22) Rotate flat to down and pull arm completely to the end.
23) Close Main Chamber gate valve (**Toggle5 down**) if not already closed

Turning X-Rays off

1) Normally just click the **Xray Operate** box 3 times or more until unchecked. It's important to leave the filament on (the 9600 controller must show a current in the display. Otherwise the next user will experience arcing issues for 15-20 minutes)

Turning X-Rays on

1) Normal Operation (use computer, not manual mode):

A. Check the **XRayOperate** box in the ESCA Control Panel and HV will ramp up (~5 sec)
B. After the Glassman has ramped to maximum voltage, all green lights should be lit. You are ready to go. Skip part 2 below

2) **If the Filament and XRays are off** (not normal), follow the steps below. Line letters correspond to letters on the electronics rack.
   A. Turn on the Glassman power supply if not already on
   B. **Press HV ON if not already on.**
   C. Turn the 9600 Controller on 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, 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

   If the filament does not turn on, check to see if any red fault indicator lights on the panel with toggle switches are on, if not, STOP contact manager.

Aligning sample

1) Looking into the main chamber from above, and using the joystick, center your sample in the viewport
   a. Samples on solid pucks will only move in X,Y,Z
   b. Samples on rotating or tilting stages will move in X,Y,Z, theta.
2) Carefully lift the microscope onto the viewport and view your sample.
   a. Adjust height using button+Y to focus on the sample
   b. When the sample is in focus, the crosshair will indicate approximately where the xray hits the sample.
   c. 800 spot (~1x2mm) is ~4.4 divisions wide and 2.2 high at 25X magnification and 2.2 and 1.1 divs wide at 12X, respectively. 12X is normal
3) You still need to fine-tune the height to where the focus point of the analyzer and x-rays converge. Should be done to the nearest 0.1mm in Z.
   a. Open Hawk Capture if the program is not already open.
   b. Open View---ESCA Control PNL in the software.
      i. Set the center binding energy (CBE) to 533 for oxygen or (285 for carbon)
      ii. Set the spot size to 800 for largest xray spot and highest count rate
      iii. Resolution to 4 (150V pass energy) for highest count rate
      iv. Scan box should be unchecked
      v. Press START (if the resolution has changed, there is a 10-second delay)
   c. You may see an oxygen peak in the window at 532eV. If not:
      i. the sample may not be at the correct height. To raise/lower the sample using the joystick, put your finger on the Z stepper motor spindle so you know how fast the motor is turning. Maximize the count rate while watching the rate meter
      ii. the sample may be insulating/charging, press Abort and make sure the flood gun (neutralizer) toggle switch on the rack is on. Check the box for the Flood Gun and you should see the red light turn on at the rack. The black

potentiometer in the lowest setting works for most insulators, but you may need to increase the flood gun energy.
   iii. you can look for another element on the surface like Carbon (284eV)
d. When the count rate is maximized, the sample is aligned at the focusing intersection of the xrays and analyzer.

4) Perform some observation scans to determine # of scans needed.
   a. **Survey scan** (must use res4 to calculate atomic%'s):
      i. Check the Scan box
      ii. CBE to 600, any spot size, Res 4, Window size 1200
      iii. Step size 1eV
      iv. Time/Step default is 100
      v. Press START. You may need to press START again if the scan range is 600-1800eV. Identify elements and number of scans you will need. Typically the largest peak should end up around 10k to 20k counts for good signal to noise, but it will depend on your goals or what peaks you are interested in.
   b. **High resolution scans**, if needed:
      i. Check the Scan box
      ii. CBE to your peak of interest
      iii. Resolution to 2 (50V pass energy, higher resolution, lower count rate)
      iv. Window size 20 or higher depending on your peak(s)
      v. Step Size 0.065eV
      vi. Press Start. Identify the CBE and window size you will need as well as number of scans. For good signal to noise, shoot for at least 2k or more counts above the background, however this is not always possible.
      vii. If in doubt do more scans. It's always easier to do more scans now than it is to have to redo scans later!
   c. **High Sensitivity scans**. These are basically small survey scans of >50eV to look for weak peaks or cases where concentrations are pushing the 0.1 to 1 atomic% level. Step sizes of 0.4 to 1 eV are typical.

5) As you determine CBE's, window sizes and scans, you can enter them in to the recipe section
   a. There are already recipes in the Sample Project category that you can open and adjust.
   b. You can create more lines, delete lines and move them up/down using tabs on the right.
   c. Each recipe line has premade tabs for Survey scans, Hi Res, Hi Sen, turning flood gun on/off, etc.
   d. On a highlighted line, you can press TEST REGION to send to the Esca Control Panel and begin scanning. You can also press UPDATE REGION to send any parameters back to the highlighted recipe line.
   e. LBE appears for the high sensitivity tab and means Low Binding Energy of the scan window. CBE is center binding energy
   f. You should enter in the transition in the window below the WindowWidth box. This will save you some time later in Casa. Could be 'C 1s', 'Cu 2p', 'survey', 'Au 4f', etc.
   g. Dwell time ranges from 10, 25,50,75,100,125, 150,175, 200, 225, 250. Running 1 scan at dwell of 250ms takes slightly less time than 5 scans at 50ms because of the way the scans are run.
   h. You can save the recipe in the Sample Project category.
   i. If you are running just one recipe, you should end with the following scans
      i. Unscanned 85eV, type in 'off' in for the spot size

ii. Neutralizer, 'off' if you are using the flood gun
iii. If you are running a multi-point or multi-recipe, end with an additional position set to the GLASSMAN OFF recipe.

6) Set the Project and Experiment names
    a. Project name can be the date in the format '3aug09' or '31oct09'
    b. Experiment name should be descriptive of the whole sample set
    c. Description box can be used for your reference but I don't believe it shows up in Casa
    d. If the project name (MP recipes add 4-5 characters) is longer than 35 characters, no data will be saved (thanks to Jon Petrie for finding this out)

7) Press **Run** to run the whole recipe. **One** to run one line, **End** to run from highlighted line to end
8) If running a multi-point recipe:
    a. In the Sample Project recipe section, there should be a 'Default' recipe which is a PTM (multipoint) scan. If not, just create one. Click on the pencil icon and the position windows will open. Click the 'Recipe' button so the recipes show. Click the 'Update' or 'Auto Add' buttons depending on if you want to update a line with the current X,Y,Z,R positions or add a line with those positions. When you press the 'Get Position' button on the screen or the joystick, the current positions should be entered.
    b. Add a final, extra line. Your last scan recipe should be GLASSMANOFF. This will turn off the flood gun and the Glassman to 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.
    c. You should always go through all the positions from 1—N to see that the stage moves correctly. It's possible that going in reverse order is fine, but ascending order is not. Clicking on the row number will prompt a move to that row position.
    d. Make sure the PTM recipe is showing in the window when you press 'RUN', otherwise only the recipe showing will run. :POS01, :POS02 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.
    e. You can adjust and save visible recipes while analysis is running and these changes will work for later positions that use the same recipe.
    f. Use the Batch Export program to convert all positions to individual vamas files. You can use the Open and Merge command in CasaXPS.

Exporting Data (Save all data to the MyDocuments\UserData folder)

1) Exporting individual data files to Vamas:
    a. If your data is visible on the screen, go to File, Export. Select Vamas and save into the UserData folder
    b. If your data is not visible, click on the Experiment button, find your project name and experiment, then do the above steps.
    c. You may also save data into excel, but CasaXPS utilizes Vamas format best
2) Exporting multiple files to Vamas (will export with ProjectName_ExperimentName_POS). Note, there is a ~130 position limit which Vamas Batch Export can see. If your database grows larger than 130 files, you will need to create a new database. You can still export data using #1 above:
    a. Open the Batch Export Utility on the desktop.

b. Select the database
c. Select the projects or individual files to move to the right window.
d. Press Convert once. There is no confirmation of the conversion, check the destination folder.
e. You can use CasaXPS to merge the vamas files into one

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

1) Open Motion Control Panel. Three to one ratio for rotation to tilt, 165-degrees rotation = 55 degrees tilt. Cautions:
   a. Entering large angular changes (>180) in the Motion Control Panel may initiate the tilt in the OPPOSITE direction and the problem below.
   b. Exceeding the tilt axis will cause rotation of the entire tilt stage and may hit the flood gun or other parts inside the chamber
2) Insert sample holder into system with bulky end to the left and aligned correctly.
3) Turn on x-rays.
4) Using the reflection of light from the sample, rotate/tilt the stage to maximize the reflection.
   a. Zero the R-axis in the motion control panel
   b. Maximize count-rate on the flat sample by adjusting Z
   c. Tilt sample to zero-angle (195-degrees) and re-zero the R-axis
   d. Maximize count-rate on tilted sample by adjusting Y
   e. Spot should now be centered on the axis of rotation

Ion Sputtering

- Attach the orange cord to the control panel, to avoid venting of the load lock while the "to ion gun" toggle is open which can inadvertently vent the main chamber.
- Gas flow to the ion gun may slowly decrease over several hours, lowering beam curren. Increase gas pressure to compensate.

1) Before turning on the 1401A controller, set:
   a. Black emission knob fully CCW to 'Standby'
   b. Beam to OFF
   c. Turn on Ion Gun controller power so you can see the gas pressure display at far left.
2) To introduce sputtering gas into the source:
   a. Open the 'To Vac Manifold' toggle to pump out excess Argon from the line.
      i. Wait until pressure drops below 10^{-4} Torr
      ii. Close the 'To Vac Manifold' toggle.
   b. Open the 'To Ion Gun' toggle which introduces gas to the gun and opens the differential pumping valve.
   c. Set gas leak valve to last setting used (~115) and increase slowly to obtain ~ 7 to 8 mTorr in the ion source. Wait >1 minute for pressure to stabilize with each change. Try not to exceed desired flow rate or the leak valve must be turned down at least one full turn, and then back up.
3) 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, then off.
4) For automated depth profiling:

a. **Set up the depth profile scan in ESCA.** The dwell time during sputtering will be last used setting. If you need 100ms, e.g., go to the ESCA control panel and start a scan using the 100ms dwell time.
b. **Make sure the ion gun check box is unchecked/OFF in the ESCA control panel.**
c. **Set Remote/Local to REMOTE**
d. **Beam on/off to ON**
e. **Emission current knob from Standby to 10mA**
f. **Check Raster ON (normal size is 2x4mm)**
g. **Set Ion Energy, typically 4000eV**
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. 2 full cycles means scan, etch, scan.**
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 ion gun if on.**

5) When finished:
   a. Turn gas flow rate down to <70 (valve behind left cabinet door)
   b. Set Emission to STANDBY
   c. Beam on/off to OFF
   d. Remote/Local to LOCAL
   e. 9600 Controller power OFF
   f. Close 'To Ion Gun' toggle (EXTREMELY IMPORTANT, valve behind right cabinet door)
   g. remove orange cord from interlock panel