

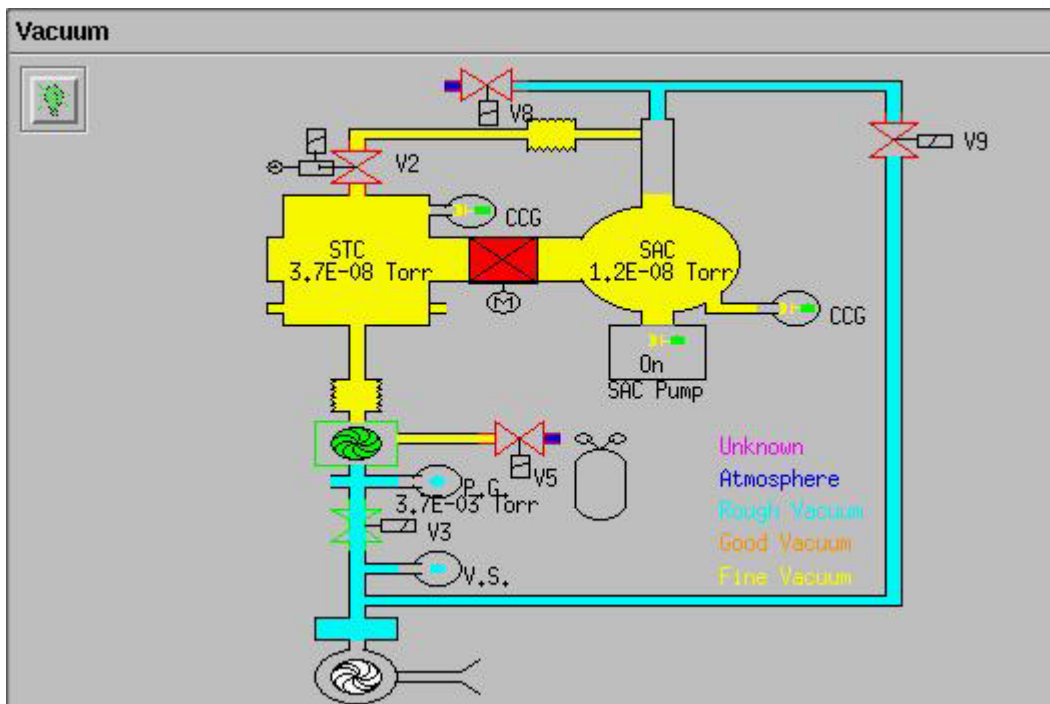
Operation Procedure for XPS1 Kratos Axis Ultra

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Startup Procedure

1. **Create or Start an XPS1 reservation in the CoreResearch@Duke system**
2. **Verify that XPS software is still open.** If it is not, follow the XPS1 Software Re-Start and Start-Up Procedure.
3. **In the Instrument Manual Control Window, go to the Vacuum section and verify that the STC pressure is $< 2 \times 10^{-6}$ and the SAC pressure is $< 3 \times 10^{-8}$.** *If vacuum levels are higher than these values, notify SMIF staff and do not proceed.*



Click [HERE](#) for a video showing the following XPS Startup steps

4. **Verify that the controller processor unit on the XPS displays “d3” and the red High Voltage light is illuminated on the Analyser Control Unit.** If this is not the case, you will need to restart the software.
5. **Turn up the SAC chamber light**
6. **Turn on the video monitor**

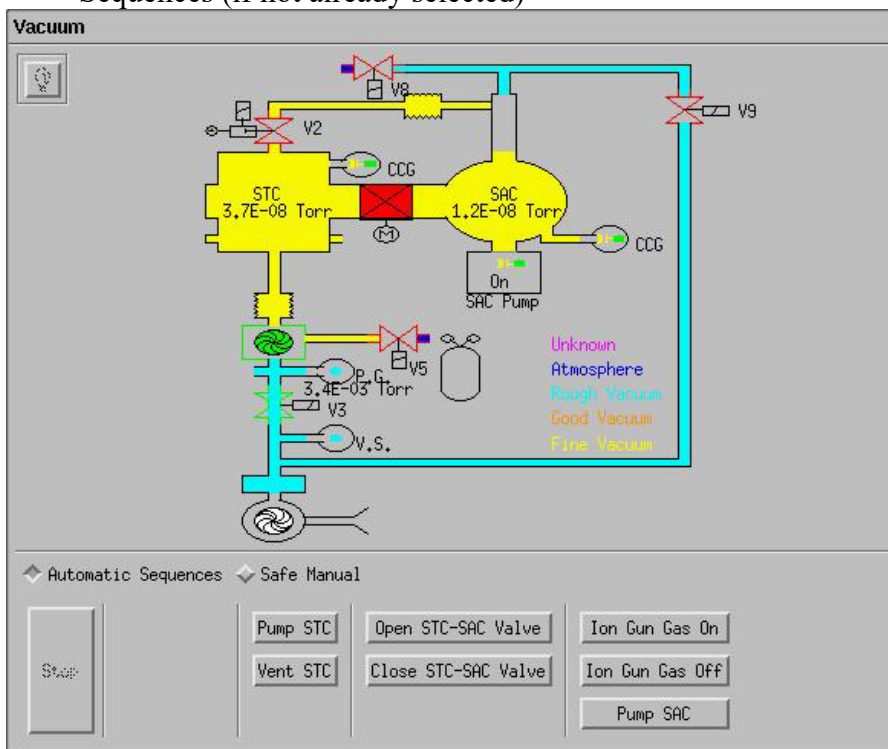
Never close any XPS program windows, as this will shut down the program and require you to re-start the software

Sample Loading

*Always wear gloves when handling samples and sample holders
Failure to do so will compromise the chamber vacuum
and produce bad measurement results.*

Click [HERE](#) for a video on loading the samples onto the bar sample holder

1. Load your sample(s) onto the bar sample holder
2. Go to the Vacuum section in the Manual Control Window and select Automatic Sequences (if not already selected)



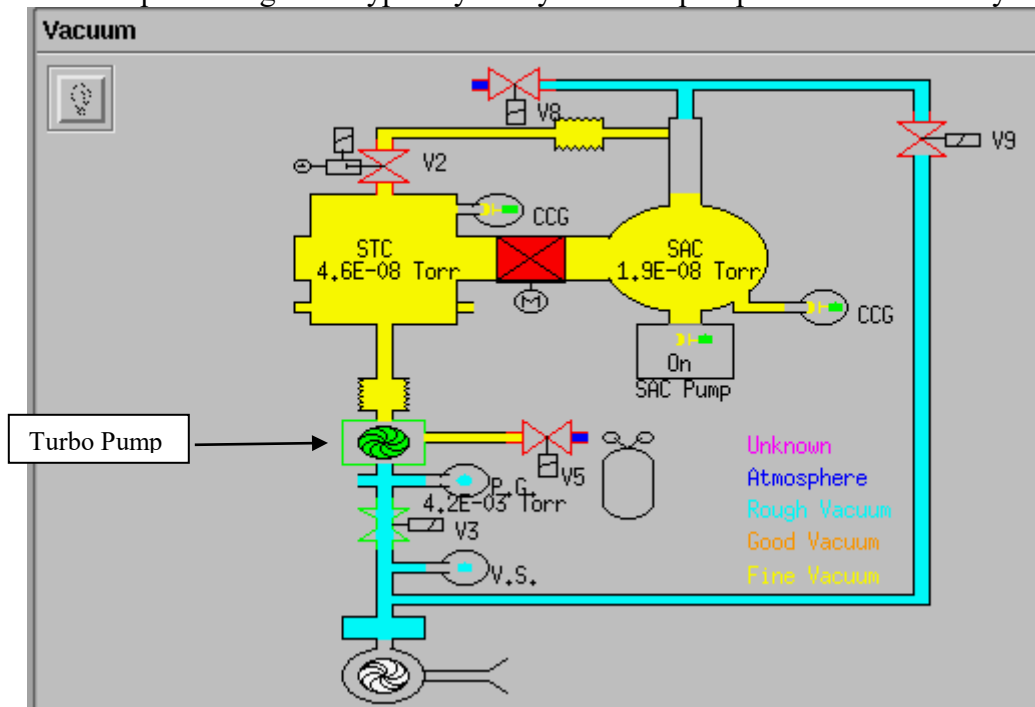
Click [HERE](#) for STC venting video

3. Click on “Vent STC”. This will vent the Sample Transfer Chamber to allow sample loading.
4. Partially loosen the three brass screws on the end of the STC chamber and wait for chamber to completely vent. When the chamber is completely vented, the seal behind the screws will open slightly due to the N_2 gas purge pressure.

Click [HERE](#) for a video on loading the samples and pumping down the STC chamber

5. Completely loosen the three brass screws on the end of the STC chamber and rotate the chamber door open
6. Ensure that the rubber O-ring on the door is clean and is securely in place in the circular groove of the STC chamber door

7. **Rotate the translation knob to move the transfer arm forward for sample loading**
8. **Attach the sample holder to the end of the transfer arm.** Make sure the top of the bar holder is placed under the upper plate of the transfer arm. It should not be placed on top of this plate.
9. **Rotate the translation knob to move the transfer arm backward until the green light at the end of the transfer arm is illuminated.**
10. **Close the chamber door and fully tighten the three brass screws**
11. **Press the “Advance” button on the Vacuum Control Unit of the XPS to stop the vent cycle and the N₂ purge**
12. **Click on “Pump STC” in the vacuum control section of the software**
13. **a. Ensure that the Turbo Pump comes on before leaving** (the pump icon will turn yellow and then green in the vacuum control section). This will occur when the P.G. gauge below the turbo pump reaches less than 1.0 E-01 Torr and should take about 3-4 minutes, when the sequence reaches about 14%.
 - b. **Ensure that the STC gauge stays on before leaving.** The STC gauge will turn on when the sequence reaches about 80% (10 minutes or so). If the vacuum reading is above 1.0E-05 torr, the gauge will turn off and the vacuum cycle will be aborted (turbo pump will turn off). If this happens, just click “Pump STC” again. Typically the system will pump down on the 2nd try.



- c. **Wait for the pump down automatic cycle to complete and for the STC pressure to reach less than 5×10^{-7}** (This typically takes about 60 minutes for dry samples)

DO NOT attempt to open the STC-SAC valve and transfer the sample if the STC pressure is greater than 5×10^{-7}

Click [HERE](#) for a video on opening the STC-SAC valve and setting the stage position

- d. Click on “Open STC-SAC Valve” and wait until the automatic sequence is completed.
- e. Scroll up to the Stage section in the Manual Control Window



- Select the “Table” box if the position table rows are not displayed
- If “Bar Load” and “Bar Unload” positions are not displayed in the position table rows, then enter the positions as follows:
 - a. Click on “Load Table”
 - b. Go to the C directory and double click on file: /C=/Stage Positions
 - c. Choose Bar.dset file and click OK
- f. Click on the position table row labeled “Bar Load” to highlight that row and then click on “Go to”. This will move the stage to the proper position for transferring the sample holder. Wait for the Halt light to turn green (stage movement stopped).

Click [HERE](#) for a video on transferring the samples to the SAC stage

- g. **While looking through the viewing window, rotate the translation knob to move the sample holder into the SAC chamber until it interlinks with the stage and stops.**
- h. **Use the Autostage manual controller to move the stage away from the transfer arm, releasing the sample holder from the transfer arm:**
 - a. While slightly wiggling the translator knob back and forth, press the left button on the autostage manual controller to move the stage away from the transfer arm.
 - b. Hold down the right button after pressing and holding the left button to speed up the stage movement
- i. **When the stage and sample holder have cleared the transfer arm, rotate the translation knob to move the transfer arm completely out of the SAC chamber – until the green light at the end of the transfer arm barrel is illuminated.**
- j. **Scroll to the Vacuum Section, and click on “Close STC-SAC Valve”.**

Click [HERE](#) for a video on positioning a sample for measurement

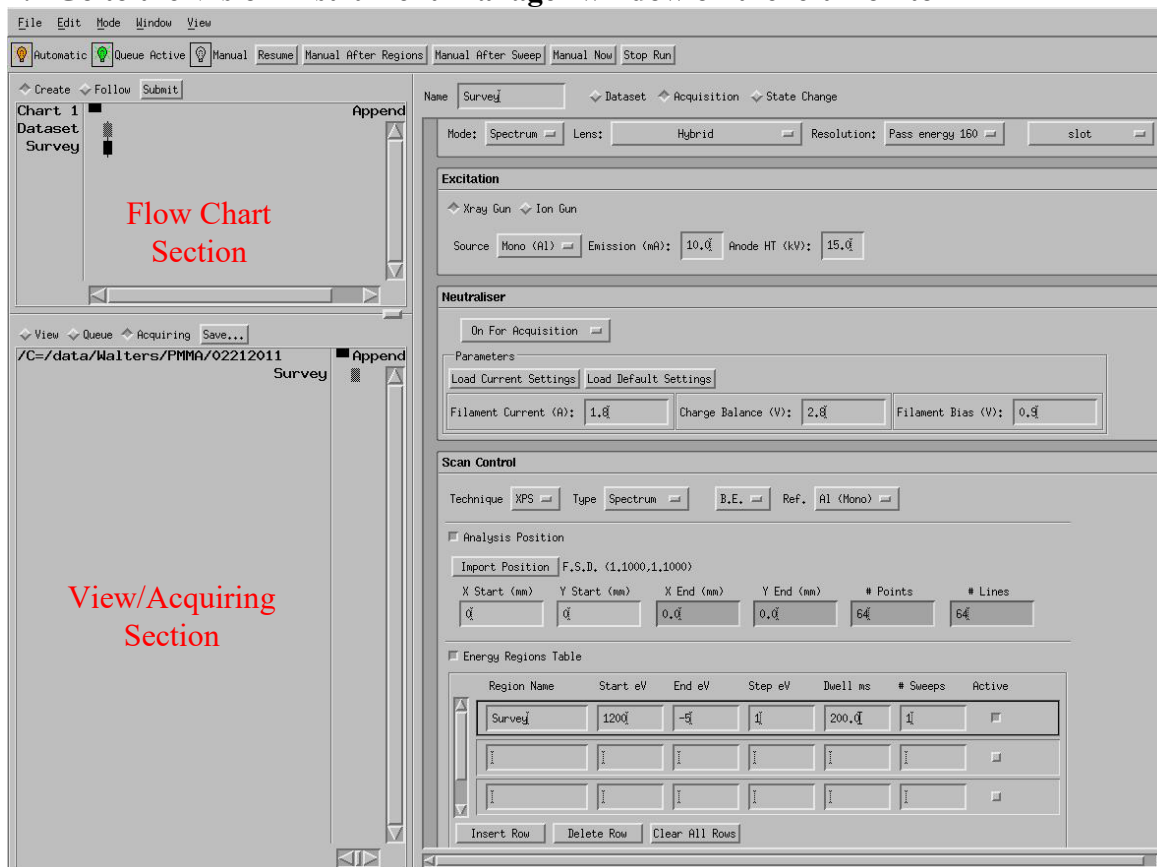
- k. **Position sample using the Autostage Manual Controller while watching the Video Monitor.**
 - a. Use the Left, Right, In, and Out buttons to position the sample area of interest in the center area of video monitor
 - b. Go to maximum magnification by adjusting the zoom wheel on the video camera. Use the Up and Down buttons to adjust the height of the sample so that the center region of the sample is in focus on the monitor. The corner regions on the monitor should be slightly out of focus.

Note: For the majority of spectroscopy acquisitions, this accuracy of setting the sample height will be sufficient to achieve an acceptable number of counts. If further Z height optimization is needed, follow the procedure “Optimizing Z for Spectroscopy”.

Performing a Survey Scan

Click [HERE](#) for a video on creating a flow chart for a survey scan

1. Go to the Vision Instrument Manager window on the left monitor



2. Click “Resume” button at the top of the window to set XPS to Automatic mode.

3. Choose the “Dataset” button

- Click on “browse” to choose a directory for storing data. Please use your personal user directory. *Note: Directories must be created outside of the Kratos software*

- Enter a filename for storing your data in the filename field

Notes:

- Always press return after making an entry
- Filenames and directories should not contain any spaces

4. Move the cursor to the flow chart section and click on the middle mouse button to paste the “Dataset” sequence in the flow chart section

5. Choose the “Acquisition” button and set up the parameters for the survey scan. The following are typical values (See also Acquisition Conditions Reference Sheet):

- Name: Survey
- Standby Control: Leave On (leaves X-rays on after scan completed)

- c. **Analyzer**
 - i. **Mode:** Spectrum
 - ii. **Lens:** Hybrid
 - iii. **Resolution:** Pass Energy 160
 - iv. **Aperture:** Slot
 - d. **Excitation**
 - i. **X-ray gun selected**
 - ii. **Source:** Mono (Al)
 - iii. **Emission:** 10.0
 - iv. **Anode HT:** 15.0
 - e. **Neutralizer:**
 - i. **For conducting samples:** “Under Manual Control” (charge neutralizer off)
 - ii. **For insulating samples:** “On for Acquisition” (click on Load default settings)
 - f. **Scan Control**
 - i. **Technique:** XPS
 - ii. **Type:** Spectrum
 - iii. **B.E.** (Binding energy)
 - iv. **Ref:** Al (mono)
 - v. **Select Energy Regions Table and click on “Clear all Rows” at the bottom of this table**
 - 1. **Region Name:** Type in “Survey”
 - 2. **Start eV:** 1200 (right click on header to change from Centre eV to Start eV if needed)
 - 3. **End eV:** -5
 - 4. **Step eV:** 1
 - 5. **Dwell ms:** 200
 - 6. **# Sweeps:** 1
 - 7. **Verify “Active” box is selected**
6. **Click on the middle mouse button to paste the “Survey” sequence in the flow chart**

Click [HERE](#) for a video on ramping up the X-Ray gun

7. **For the first sample:** Go to the Instrument Manual Control window and scroll to the Xray Gun section. Manually ramp up the X-Ray voltage and current as follows:
- a. Set Anode HT to 6kV and Emission to 1mA. Always enter the voltage before the current, and remember to hit enter after typing in the numbers
 - b. Click the “Standby” light bulb and wait for light bulb to turn green
 - c. Click the “On” light bulb and wait for light bulb to turn green
 - i. Emission current should read 1mA and Anode HT should read 6kV
 - d. Set Anode HT to 10kV and Emission to 5mA
 - i. Emission current should read 5mA and Anode HT should read 10kV
 - e. Set Anode HT to 15kV and Emission to 10mA
 - i. Emission current should read 10mA and Anode HT should read 15kV

Click [HERE](#) for a video on starting a measurement

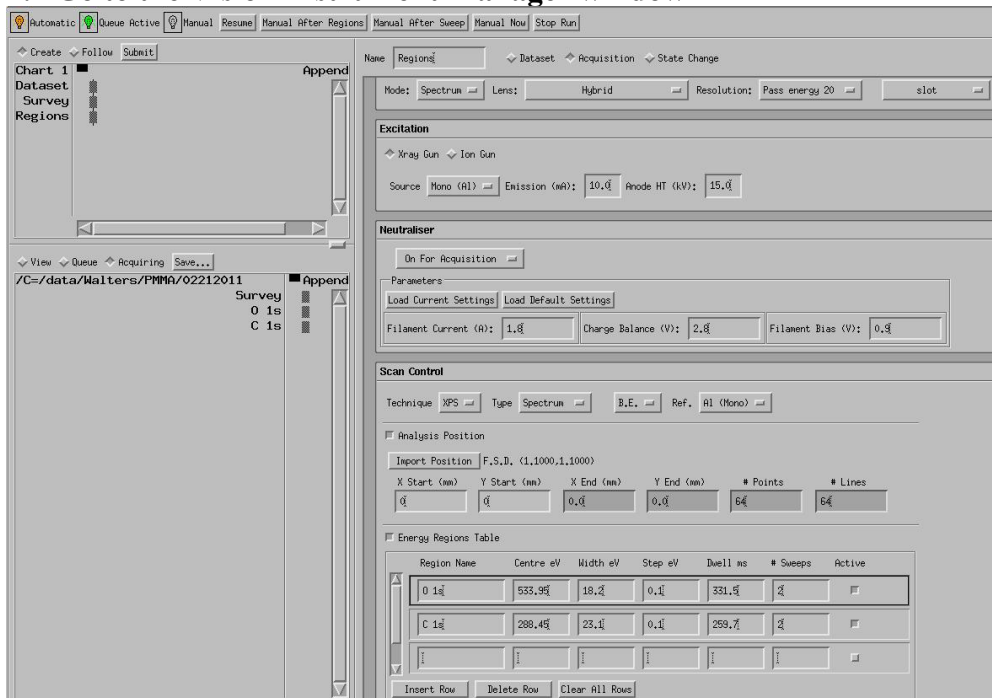
- 8. Go back to the Vision Instrument Manager window and Click on “Submit” to start the flowchart job (Survey scan)**
- 9. If not already selected, select “Acquiring” in the view window to see the scan data being collected in the Real Time Window**
- 10. When completed, the scan may be viewed in the Real Time Window by highlighting the item in the acquiring window and using the middle mouse button to “paste” the item into the right-hand section of the real-time display window.**
 - a.** Using mouse control menus you may zoom in on regions of the scan, change the scan label, etc.
 - b.** Choose element list from Windows pull down menu to open up an element list for identifying peaks
 - i.** Clicking on peaks of interest in the Real Time Window will cause associated elements to display in the element list window

Performing a Region Scan - high resolution scans around peak(s) of interest

This procedure assumes that you have already set up and performed a survey scan as described previously.

Click [HERE](#) for a video on adding region scans to the flowchart

1. Go to the Vision Instrument Manager window



2. Choose the “Acquisition” button and set up the parameters for the region scan. Use the same settings as for the survey scan, with the following changes:
 - a. Name: Regions
 - b. Analyzer
 - Resolution: Pass Energy 20 (See Acquisition Conditions Reference Sheet)
 - c. Scan Control
 - Select Energy Regions Table and click on “Clear all Rows” at the bottom of this table
 - Type in region name (e.g., O 1s) and press enter and settings will automatically load. (Note – region name must match exact spelling and case of entry contained in database). You may adjust these settings (scan width, time, number of sweeps, etc) as desired.
 - If desired, enter more regions in different rows of the table
 - Click on the “Active” box beside each row in the table for which you want a scan to be made
3. Click on the middle mouse button to paste the “Regions” sequence in the flow chart
4. Click on “Submit” to start the flowchart job
5. When the scans are completed, you can highlight them in the viewing window and paste them into the Real Time Display window to view all scans.

Sample Unloading

Always wear gloves when handling samples and sample holders

Click [HERE](#) for sample unloading video – Part 1

1. **Go to the Instrument Manual Control window.**
2. **Turn off X-rays by clicking on the “Off” button in the Xray Gun section**
3. **Turn off the charge neutralizer by clicking on the “Off” button in the Neutraliser section (if applicable)**
4. **Scroll to the Stage section in the Manual Control Window and select the “Table” box if the position table rows are not displayed.** If needed, load the “Bar Load” and “Bar Unload” positions as described in the sample loading procedure.
5. **Click on the position table row labeled “Bar Unload” to highlight that row and click on “Go to”.** This will move the stage to the proper position for transferring the sample holder. **Wait for the Halt light to turn green** (stage movement stopped).
6. **Scroll down to the Vacuum section** and select Automatic Sequences if not already selected
7. **Click on “Open STC-SAC Valve”** and wait until the automatic sequence is completed.

Click [HERE](#) for sample unloading video – Part 2

8. **While looking through the viewing window, rotate the translation knob to move the sample holder into the SAC chamber until it interlinks with the stage and stops.**
9. **Use the Autostage manual controller to move the stage toward the transfer arm, connecting the sample holder to the transfer arm:**
 - a. While slightly wiggling the transfer arm back and forth, press the right button on the autostage manual controller to move the stage toward the transfer arm.
 - b. Hold down the left button after pressing and holding the right button to speed up the stage movement
 - c. Drive stage in the right direction until the sample holder completely hooks onto the transfer arm.
10. **When the sample holder has transferred, rotate the translation knob to move the transfer arm completely out of the SAC chamber – until the green light at the end of the transfer arm barrel is illuminated.**

Click [HERE](#) for video on closing the STC-SAC valve and venting

11. **Scroll to the Vacuum Section, and click on “Close STC-SAC Valve”** and wait until the automatic sequence is completed.
12. **Click on “Vent STC”.** This will vent the Sample Transfer Chamber to allow sample loading.
13. **Partially loosen the three brass screws on the end of the STC chamber and wait for chamber to completely vent.** When the chamber is completely vented, the seal behind the screws will open slightly due to the N₂ gas purge pressure.
14. **Completely loosen the three brass screws on the end of the STC chamber and rotate the chamber door open**

Click [HERE](#) for video on removing the sample bar holder and pumping down STC

15. **Rotate the translation knob to move the transfer arm forward for sample unloading**
16. **Remove the sample holder from the end of the transfer arm**
17. **Rotate the translation knob to move the transfer arm backward until the green light at the end of the transfer arm is illuminated.**
18. **Ensure that the rubber O-ring on the door is clean and is securely in place in the circular groove of the STC chamber door**
19. **Close the chamber door and fully tighten the three brass screws**
20. **Press the “Advance” button on the Vacuum Control Unit of the XPS to stop the vent cycle and the N₂ purge**
21. **Click on “Pump STC” in the vacuum control section of the software**
22. **Unload your sample from the sample holder and return the sample holder to the blue Kratos box.**
23. **Ensure that the Turbo Pump comes on before leaving** (the pump icon will turn yellow and then green in the vacuum control section). This will occur when the P.G. gauge below the turbo pump reaches less than 1 E-01 Torr and should take about 3-4 minutes

Returning System to Proper State for Next User

Click [HERE](#) for XPS shut down video

1. **DO NOT CLOSE ANY WINDOWS OR EXIT THE SOFTWARE**
2. **In the Vision Instrument Manager window:**
 - a. **Save the flowchart you created if desired by going to the File pull down menu and selecting “Save Run”.**
 - i. **Navigate to your personal data directory**
 - ii. **Enter a filename to save the run and click OK. (Suggestion – use the word Run in the filename to easily distinguish between a Run file and a Data file).**
 - b. **Clear all flowchart elements from the flowchart section** by highlighting the boxes, right-clicking, and choosing “cut”
 - c. **In the acquisition section, right click and choose the “Close Dataset” option**
3. **In the Instrument Manual Control Window:**
 - a. **If ion sputtering was performed, verify that the Ion Gun is off, and the ion gas is off. (See Sputter cleaning instructions)**
 - b. **Scroll down to the vacuum control section and leave this window visible**
4. **Turn down the SAC chamber light (do not turn off).**
5. **Turn off the video monitor**
6. **Stop the XPS1 reservation in the CoreResearch@Duke system**