

# Operation Procedure for Kratos Axis Ultra XPS

## Typical Experiment Sequence

1. Sign-in in the logbook
2. Start up system and verify vacuum parameters
3. Load sample(s)
4. Perform a survey scan and identify peaks of interest
5. Sputter clean sample surface (if desired)
6. Perform spectroscopy around peak(s) of interest to get additional chemical information (if desired)
7. Perform imaging to identify areas on sample of particular elemental or chemical states (if desired)
8. Perform small area or multipoint spectroscopy in areas of interest on sample (if desired)
9. Process data (perform data analysis)
10. Unload sample
11. Return system to proper state for next user
12. Sign-out in the logbook

Each of these procedures is explained in more detail on the following pages

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### **Startup Procedure (typical)**

1. **Sign in on the written logbook** and note the comments of the previous users.
2. **Verify that the SAC pressure is  $< 1 \times 10^{-8}$  and SEC pressure is  $< 5 \times 10^{-7}$**  on the vacuum control unit display. *If vacuum levels are higher than these values, notify instrument staff and do not proceed.*



3. **Turn on the computer display screen**
4. **Verify that XPS software is still open and that the controller processor unit displays “d3”**
5. **The manual control window should be displayed on the screen.** If it is not, click on the “Manual” zone in the top task bar.

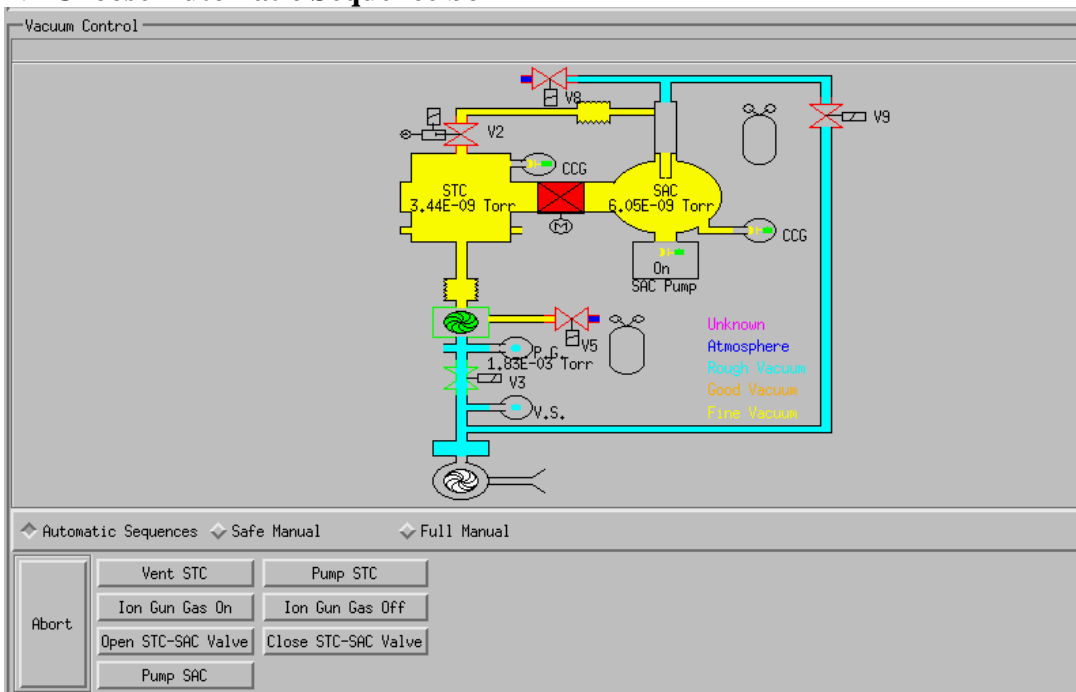
*If the software is not open, the controller processor unit will not display “d3”. You will need to re-start the software and re-calibrate the stage. The procedure for doing this is found in the section “Start up procedure for when computer or software has been shut down” at the end of this document.*

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

## 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.*

1. Scroll down to Vacuum Control section in the Manual Control Window
2. Choose Automatic Sequence box



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<sub>2</sub> gas purge pressure.
5. Completely loosen the three brass screws on the end of the STC chamber and rotate the chamber door open
6. Rotate the translation knob to move the transfer arm forward for sample loading
7. Attach the sample holder to the end of the transfer arm
8. Rotate the translation knob to move the transfer arm backward so that the sample holder does not collide with the chamber opening when closing the chamber door
9. Close the chamber door and partially tighten the three brass screws
10. Click on “Advance” button on the Vacuum Control Unit to stop the N<sub>2</sub> purge  
Click on “Pump” STC and walk over and hold STC chamber door closed while pump down initiates
11. After pump-down is initiated, tighten the three brass screws on the STC chamber door and wait for pump down to complete (typically about 15 minutes)

12. When completed, “Automatic Sequence Successfully Completed” will be displayed in the Vacuum Control Section
  - If the sequence fails to complete (Vacuum to high in STC), repeat sequence only once. If it still fails, check door tightness, decrease amount of outgassing sample
  - Report problem to Jonas, Jean or Courtney
13. Verify that the SEC pressure is  $< 5 \times 10^{-7}$

*DO NOT open the STC-SAC valve and transfer the sample if the SEC pressure is  $> 5 \times 10^{-7}$*

14. Click on “Open STC-SAC Valve” and wait to see valve open in the vacuum diagram
15. Verify that the Dual Anode x-ray source is backed out from the sample area.
16. Scroll to Manipulator section in the Manual Control Window and click on “Position” box if the position table rows are not displayed

Manipulator

Halt Manual Positions Parameters Move Sequ. External

Initialised Calibrated

Manual Control

X Axis (mm) Y Axis (mm) Z Axis (mm) Theta X (deg)

0,000 0,000 0,000 0,000

General Table

Save Positions Read Positions Goto Position Update Position Move In Sequence Optimize Z

Name	X Axis	Y Axis	Z Axis	Theta X	Select Row
					<input type="checkbox"/>
					<input type="checkbox"/>
					<input type="checkbox"/>

Insert Row Reset Row Delete Row Clear All Rows Table View

Insert Mode

Current Point Increment Point Increment Point, Use Rot. Axis

Position Increment

X Axis/R Polar Y Axis/Phi Polar Z Axis Theta X

Increment N Times No. Increments

17. If “Bar Load” and “Bar Unload” positions are not displayed in the position table rows, then perform the following:

- Click on “Read Positions”
- Go to C directory and double click on file: /C=/Stage Positions
- Choose Bar.dset

*Note: This assumes you are using a bar type sample holder. Load and unload positions for other types of sample holders can be found in the same directory.*

18. Highlight the position table row labeled “Bar Load” and click on “Go to Position”. This will move the stage to the proper position for transferring the sample holder.

19. Click on “Manual” in the Manipulator section

20. 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.

21. Use the Autostage manual controller to move the stage away from the transfer arm, releasing the sample holder from the transfer arm:

- While jiggling the translator knob, press the left button on the autostage manual controller to move the stage away from the transfer arm.
- Holding down the right button after pressing and holding the left button will speed up the stage movement

22. 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.

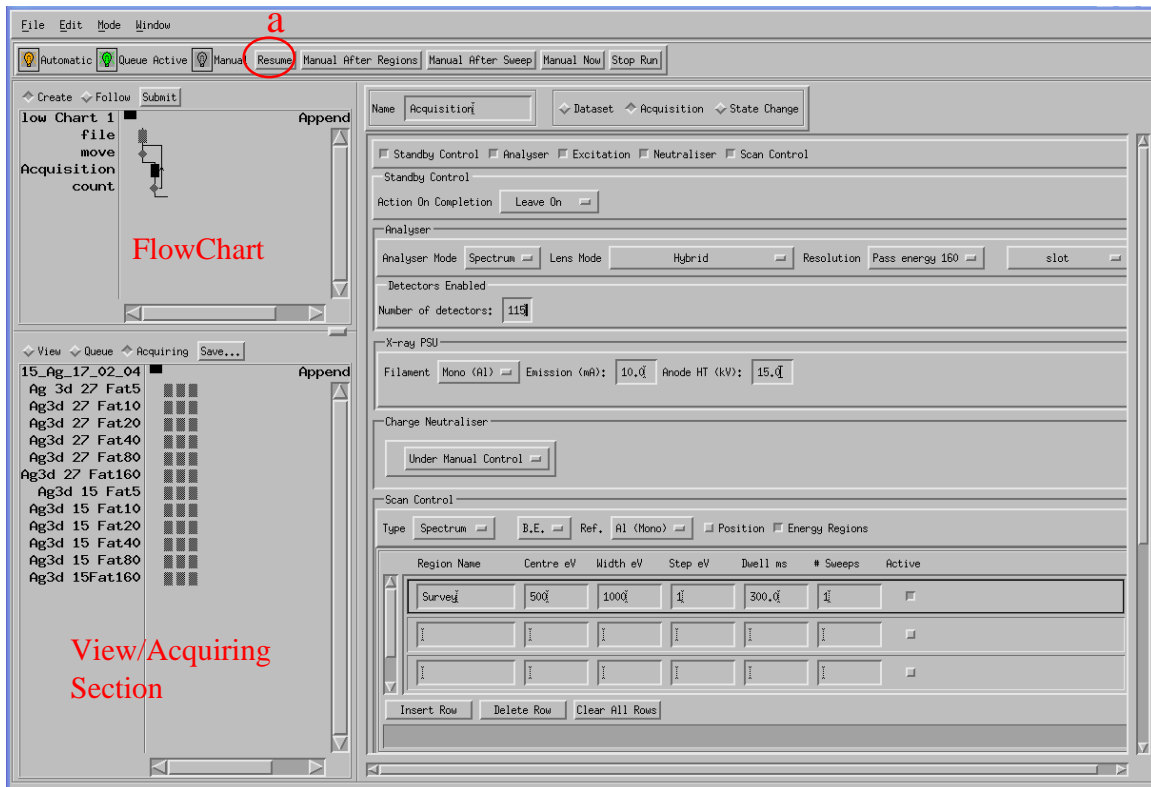
23. Scroll to the Vacuum Control Section, and click on “Close STC-SAC Valve”

24. Position sample using the Autostage Manual Controller while watching Video Monitor. Note: Magnification on monitor can be varied by adjusting zoom control on video camera

- Use Left, Right, In, Out buttons to position sample area of interest in center area of video monitor
- Use Up, Down buttons to adjust height of sample so that center region of sample is in focus on the monitor. The top and bottom 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 found in the section “Optimizing Z for Spectroscopy”. Optimizing Z for imaging is explained in the Imaging procedure section.*

## Performing a Survey Scan



1. Click on the “Manager” zone in the top task bar to open the Vision Instrument Manager work area
2. Click “resume” (a in figure above) button at the top of the window to set to Automatic mode.
3. Choose the “Dataset” button
  - a. Enter a filename for storing your data in the filename field
  - b. Enter a name for this flow chart item in the name field. (e.g., Filename)
4. Click on the middle mouse button to paste the “Filename” sequence in the flow chart section of the manager window.
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):
  - a. Name: Survey
  - b. Standby Control: Leave On (leaves X-rays on after scan completed)
  - c. Analyzer
    - i. Mode: Spectrum
    - ii. Lens Mode: Hybrid
    - iii. Resolution: Pass Energy 160
    - iv. Aperture: Slot
  - d. Detectors enabled: 115
  - e. X-Ray PSU (Power Supply Unit)
    - i. Filament: Mono

- ii. **Emission:** 10     *Never go above 20, as it will degrade source life*
    - iii. **Anode HT:** 15
  - f. **Charge Neutralizer:** Under Manual Control
  - g. **Scan Control**
    - i. **Region Name:** Type in “Survey”
    - ii. **Start eV:** 1200 (right click on header to change from Centre eV to Start eV)
    - iii. **End eV:** -5
    - iv. **Dwell ms:** 200
    - v. **# Sweeps:** 1
    - vi. **Click on “Active” box**
- 6. **Click on the middle mouse button to paste the “Survey” sequence in the flow chart**
- 7. **Click on “Submit” to start the flowchart job (Survey scan)**
- 8. **If desired, choose “Acquiring” in the view window to see the scan data being collected in the Real Time Window**
- 9. **When completed, the scan may be viewed in the Real Time Window by highlighting the item in the acquiring window and middle mouse button “pasting” 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 element to display in the element list window

### **Sputter Cleaning**

It is suggested that you acquire a spectra on peaks of interest before and after performing a sputter clean so that a comparison can be made and the effect of the clean determined. (See procedure for performing spectroscopy around peaks of interest). For example, you may want to look at the oxygen and carbon peaks before and after sputtering to verify that they have decreased sufficiently and the sample peak of interest to determine that it has increased sufficiently.

- 1. Click on the “Manual” zone in the top task bar** to open the manual control window.
- 2. Scroll to the Vacuum Control Section and choose Automatic Sequence**
- 3. Click “Ion Gas” On and look for the valves to open.** This opens the valves to allow the Argon gas in for sputtering.
- 4. Scroll to the Ion Gun Section and choose Table button.** The table contains pre-set setting for various ion sputtering conditions.
- 5. Choose the 5kV Large Spot row in the Table** (for depth profiling, choose the 5kV medium spot and a raster of 2mm should normally suffice)
- 6. Click “Restore Row”** to enter these settings. You may click on the “states” button to watch the sputtering parameter values
- 7. Choose desired Raster Size in Operation Settings**
- 8. Click on “Standby” to ramp up voltage and currents.**
- 9. After ramp-up complete, click on “Start” to start the sputter process**
- 10. Allow to run for desired time. (Typical time is 10 minutes for a sputter clean)**
- 11. Click on “Standby” after desired time has elapsed**
- 12. Perform a spectra around the peaks of interest and compare post-sputter spectra with pre-sputter spectra.**
  - a. If additional sputtering is desired, click on “Start” and sputter for additional desired time**
- 13. Click on “Off” after acceptable sputter cleaning has been achieved**
- 14. In Vacuum control section, click on “Ion Gun Gas Off” and look for valves to close**

(The manual leak valve should be set so that the pressure in the SEC is  $3-4 \times 10^{-7}$  torr, the SAC  $3-4 \times 10^{-8}$  torr, and the Ion Pump reading  $\sim 7 \times 10^{-5}$  amps on the 7kV setting. This is all with the gun on. The regulator takes a little time to respond once the line is opened, and the pressure is still somewhat dynamic early on in the process when the valves have just been opened.)



### **Performing spectroscopy around peak(s) of interest**

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

- 1. Click on the “Manager” zone in the top task bar to open the Vision Instrument Manager work area**
- 2. Click in the flow chart area of the Manager window**
- 3. As was done to create the survey scan, create an acquisition flow chart element with a descriptive name such as “Core Scan”**
- 4. Choose the “Acquisition” button and set up the parameters for the scan. It is suggested that you use the conditions suggested in the Acquisition Conditions Reference Sheet at the end of this document for a Region or Valence Band energy range scan.**
- 5. Scroll to Scan Control Section and enter in desired scan settings using one of the following methods:**
  - a. Use Element List**
    - i. Choose element list from the pull down Windows menu**
    - ii. Click on a peak of interest in the Real Time Display Window**
    - iii. Click on the associate element in the element list, and the scan settings for that element will be automatically loaded in the scan control settings. You may choose multiple elements from the element list to enter multiple lines in the scan settings**
  - b. Enter region name in manually (e.g., O 1s) and settings will automatically load. (Note – region name must match exact spelling and case of entry contained in database)**
- 6. Click on “Active” box beside each row in the Scan Control Section for which you want a scan to be made**
- 7. Click on the middle mouse button to paste the “Core Scan” sequence in the flow chart**
- 8. Click on “Submit” to start the flowchart job (Core Scan)**
- 9. If desired, choose “Acquiring” in the view window to see the scan data being collected in the Real Time Window**
- 10. When scans are completed, highlight them in the viewing window and paste into the Real Time Display window to view all scans.**

## **Imaging**

The imaging mode allows you to pick specific peak energies and make a spatial map of their locations on your sample. You will typically follow these steps to perform imaging:

1. Pick one element or chemical state energy peak value of interest (e.g., from your survey scan)
2. Optimize the sample height to bring the image map of this energy value into best resolution
3. Set up an image mapping flowchart in the manager to collect image maps of all energy peak values of interest

### ***Optimizing Sample Height***

1. Click on the “Manual” zone in the top task bar to open the manual control window.
2. Scroll to Manipulator section in the Manual Control Window and click on “Position” box if the position table rows are not displayed
3. Insert increments of Z positions in the table by inputting increment parameters in the section below the position table
4. Click on the “Manager” zone in the top task bar to open the Vision Instrument Manager work area
5. Choose the “Dataset” button
6. Enter a filename for storing your data in the filename field
7. Enter a name for this flow chart item in the name field. (e.g., Filename)
8. Click on the middle mouse button to paste the “Filename” sequence in the flow chart
9. Choose the “State Change” button and enter a name for this flow chart item in the name field (e.g., Position)
10. Choose the Sample Position button and load the position table. This will load all the rows in the position table that was created in the manual control window.
11. Click on the middle mouse button to paste the “Position” sequence in the flow chart
12. Choose the “Acquisition” button and enter a name for this flow chart item in the name field (e.g., Map)
13. Set up the parameters for general alignment imaging (see Acquisition Conditions Reference Sheet):
  - a. Analyzer
    - i. Analyzer mode: Imaging
    - ii. Lens Mode: FOV1
    - iii. Resolution: 160
    - iv. Aperture: Low Res
  - b. Scan Control
    - i. Choose Map
    - ii. Enter region name or energy value based on element or chemical state energy peak value of interest
    - iii. Dwell: 20 (or longer if a low intensity peak)
    - iv. # Sweeps: 1

14. Click on the middle mouse button to paste the “Map” sequence in the flow chart
15. Choose the “State Change” button and enter a name for the counting flow chart item in the name field (e.g., Count)
16. Choose the Counter button and enter the # of cycles. This should be the same as the number of Z increments that were entered in the position table
17. Click on the middle mouse button and paste the “Count” sequence in the flow chart
18. In the flowchart area, highlight the “Position”, “Map” and “Count” items. Then right click and choose “Loop Back”
19. Click on “Submit” to start the flowchart job. An image map will be taken and a file created for each Z height setting that was entered.
20. If desired, choose “Acquiring” in the view window to see the image data being collected in the Real Time Window
21. When run is completed, highlight the files in the viewing window and paste into the Real Time Display window to view all maps. (You may also use the functions in the Process zone to view all maps simultaneously).
22. Choose the optimum image and note which Z position produced that image
23. Click on the “Manual” zone in the top task bar to open the manual control window.
24. Scroll to Manipulator section in the Manual Control Window and highlight the row in the position table which contains the optimum Z value
25. Click on “Go to Position”. This will move the stage to the optimum Z height

**Setting up an image mapping flowchart**

1. Click on the “Manager” zone in the top task bar to open the Vision Instrument Manager work area
2. Clear all flowchart items from the Z optimization flowchart
3. Choose the “Dataset” button
4. Enter a filename for storing your data in the filename field
5. Enter a name for this flow chart item in the name field. (e.g., Filename)
6. Click on the middle mouse button to paste the “Filename” sequence in the flow chart
7. Choose the “Acquisition” button and enter a name for this flow chart item in the name field (e.g., Map)
8. Set up the parameters for elemental or chemical state imaging (see Acquisition Conditions Reference Sheet):
9. Analyzer
  - i. Analyzer mode: Imaging
  - ii. Lens Mode: Typically FOV2

*Note: FOV2 is the only field of view that is referenced to the various apertures to give the spot size shown in the Acquisition Conditions Reference Sheet.*

**iii. Resolution:** 160 for elemental imaging, 40 or 80 for chemical state imaging

**iv. Aperture:**

1. FOV2: Medium Res or High Res
2. FOV3: High Res

**10. Scan Control**

**i. Choose Map**

**ii. Enter region names or energy values based on element or chemical state energy peak values of interest** (Multiple energy values can be entered on separate rows to map multiple elements or chemical states)

**iii. Dwell:** 20 (or even much longer for a low intensity signal or at lower pass energy)

**iv. # Sweeps:** 1 (or more if desired)

**11. Click on the middle mouse button to paste the “Map” sequence in the flow chart**

**12. Click on “Submit” to start the flowchart job.** An image map will be taken and a file created for each energy value that was entered.

**13. If desired, choose “Acquiring” in the view window to see the image data being collected in the Real Time Window**

**14. When run is completed, highlight the files in the viewing window and paste into the Real Time Display window to view all maps.** (You may also use the functions in the Process zone to view all maps simultaneously or overlap maps, etc.)

## **Small Area and Multipoint Spectroscopy**

After an image map is taken, it is often desired to collect spectroscopic scans in smaller areas of interest on the sample. This procedure explains how to take spectroscopic scans in small regions that have been identified from an image map.

### **Multipoint Small Area Scans**

- 1. Click on the “Manager” zone in the top task bar to open the Vision Instrument Manager work area**
- 2. Choose the “Dataset” button**
  - a. Enter a filename for storing your data in the filename field**
  - b. Enter a name for this flow chart item in the name field. (e.g., Filename)**
- 3. Click on the middle mouse button to paste the “Filename” sequence in the flow chart**
- 4. Choose the “Acquisition” button and enter a name for this flow chart item in the name field (e.g., Small Spot)**
- 5. Set up the parameters for a small area scan.**
  - a. See the Acquisition Conditions Reference Sheet to set values for desired spot size. Note that FOV2 is the only field of view that is referenced to the various apertures to give the spot size shown in the Acquisition Conditions Reference Sheet.**
  - b. Analyzer mode should be “Spectrum”**
  - c. Enter Scan Control parameters for desired energy and spectra settings.**

*Typical parameters are:*

    - *Energy width of 15eV around the energy value of interest*
    - *Step size of 0.1*
    - *Dwell time of 200*
    - *# Sweeps = 1*
- 6. Bring up the image map and point the mouse to the area where you want the scan to be performed, and then left click on the mouse button.**
- 7. In the Analysis Position section of the Manager window, click on “Import Position”. The x,y position of the point identified in the image map will be input.**
- 8. Click on the middle mouse button to paste the “Small Spot” sequence in the flow chart**
- 9. To perform small area spectroscopy on multiple points in an image map:**
  - a. Copy and paste the “Small Spot” sequence in the flow chart. Paste as many copies as desired points**
  - b. Go into each small spot sequence, choose the position on the image map and import the position (as in Steps 6 and 7).**
- 10. Click on “Submit” to start the flowchart job**
- 11. If desired, choose “Acquiring” in the view window to see the scan data being collected in the Real Time Window**
- 12. When completed, the scan may be viewed in the Real Time Window (You may also use the functions in the Process zone to view all scans simultaneously, etc.)**

### Line Scans

1. **Set up a filename and flowchart in the Manager Window** (See steps 1-3 in the Multipoint Small Scans Procedure)
2. **Choose the “Acquisition” button and enter a name for this flow chart item in the name field (e.g., Line Scan)**
3. **Set up the parameters for a small area scan.**
  - a. **See the Acquisition Conditions Reference Sheet to set values for desired spot size.**
  - b. **Analyzer mode should be “Spectrum”**
  - c. **Enter “Line Scan” in the Scan Control Sections**
  - d. **Enter other Scan Control parameters for desired energy and spectra settings. (as in Step 5 of Multipoint Small Scans Procedure)**
4. **Bring up the image map and click and drag the mouse to form a rectangle in the area where you want the scan to be performed. The line scan will proceed from the upper left corner of the rectangle to the lower right corner.**
5. **In the Analysis Position section of the Manager window, click on “Import Position”.** The coordinates of the line identified in the image map will be input.
6. **Click on the middle mouse button to paste the “Line Scan” sequence in the flow chart**
7. **Click on “Submit” to start the flowchart job**
8. **If desired, choose “Acquiring” in the view window to see the scan data being collected in the Real Time Window**
9. **When completed, the scan may be viewed in the Real Time Window (You may also use the functions in the Process zone to view all scans simultaneously, etc.)**

### Data Processing

The data processing software allows you to change the way in which data is viewed (including overlaying of multiple spectrum scans or images), and also allows you to quantify the results (e.g., weight or atomic per cent of elements)

1. **Click on the “Process” zone in the top task bar to open the data processing work area**
2. **From the “File” pull down menu, choose “Open Dataset for Processing”**
3. **Click on the “Update” button to load recent datasets**
4. **Choose the dataset filename(s) for which you want to process the data.**
5. **See the on-line manual in the “PDF” desktop folder for instructions and explanations of the various features in the Processing software.**

## Sample Unloading

*Always wear gloves when handling samples and sample holders*

1. Click on the “Manual” zone in the top task bar to open the manual control window.
2. Scroll to Manipulator section in the Manual Control Window and click on “Position” box if the position table rows are not displayed
3. Highlight the position table row labeled “Bar Unload” and click on “Go to Position”. This will move the stage to the proper position for transferring the sample holder.
4. Click on “Manual” in the Manipulator section
5. Scroll down to Vacuum Control section in the Manual Control Window
6. Choose Automatic Sequence box
7. Click on “Open STC-SAC Valve” and wait to see valve open in the vacuum diagram
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, releasing the sample holder from the stage:
  - a. Press the right button on the autostage manual controller to move the stage toward the transfer arm.
  - b. Holding down the left button after pressing and holding the right button will speed up the stage movement
  - c. Drive stage in right direction while jiggling the translation knob until sample holder hooks onto the transfer arm, and then drive back to the left to center the manipulator and relieve tension between the sample and fork. When completely transferred, the movement of the translation knob should loosen up considerably
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.
11. Scroll to the Vacuum Control Section, and click on “Close STC-SAC Valve” and watch display for valve to close
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 N<sub>2</sub> gas purge pressure.
14. Completely loosen the three brass screws on the end of the STC chamber and rotate the chamber door open
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 so that the transfer arm does not collide with the chamber opening when closing the chamber door**
18. **Close the chamber door and partially tighten the three brass screws**
19. **Click on “Abort” to stop the N<sub>2</sub> purge**
20. **Click on “Pump” STC and walk over and hold STC chamber door closed while pump down initiates**
21. **After pump-down is initiated, tighten the three brass screws on the STC chamber door and wait for pump down to complete (typically about 15 minutes)**
22. **When completed, “Automatic Sequence Successfully Completed” will be displayed in the Vacuum Control Section**

### **Returning System to Proper State for Next User**

1. **DO NOT CLOSE ANY WINDOWS OR EXIT THE SOFTWARE**
2. **In the Manager zone window:**
  - a. **Clear all flowcharts from the flowchart section**
  - b. **In the acquisition section, right click on the mouse and choose the “Close Dataset” option**
3. **In the Process zone window:**
  - a. **From the “File” pull down menu, choose “Close all Datasets and Clear Scratch”**
4. **Click on the “Manual” zone in the top task bar to open the manual control window, and leave this window open**
5. **Turn off the computer monitor**
6. **Complete entries in the written log book**

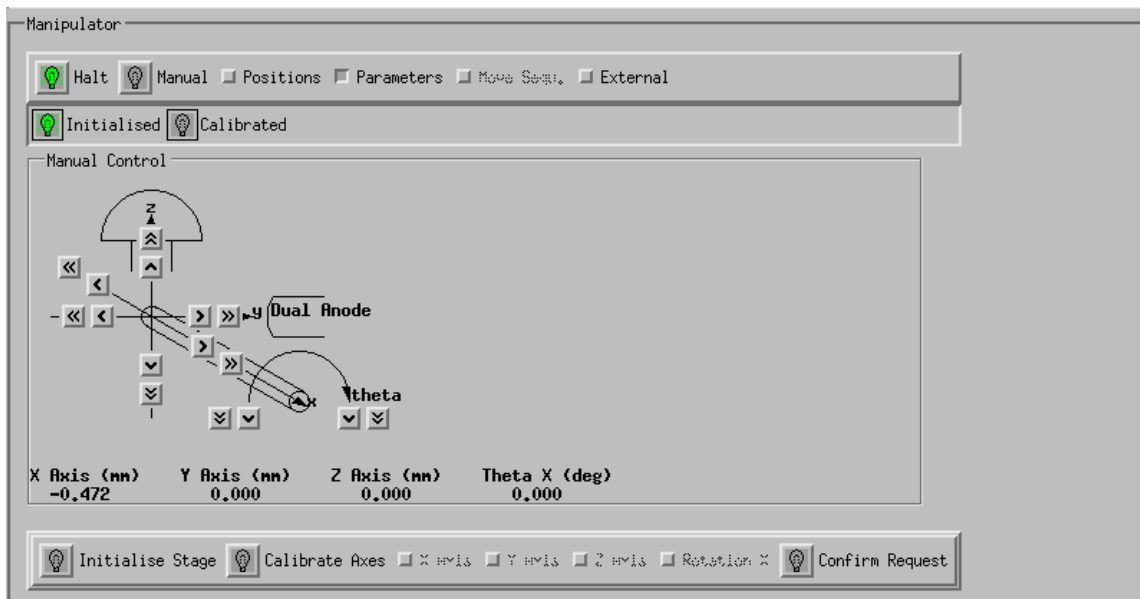


### **Optimizing Z for Spectroscopy**

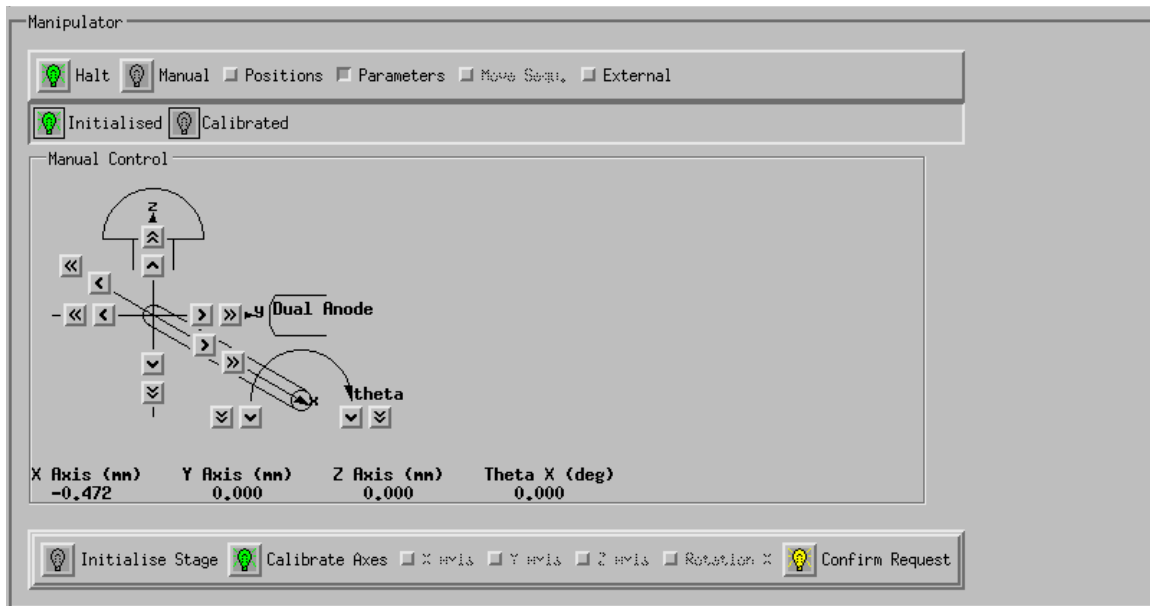
1. **From a survey scan or region scan choose an energy peak to use for the sample height optimization. Make sure this scan is pasted into the real time window**
2. **Click on the “Manual” zone in the top task bar to open the manual control window.**
3. **In the analyzer section, enter the following parameters:**
  - a.
4. **In the acquisition section, choose the type “Snapshot”**
5. **In the analyzer section, enter the following parameters:**
  - a. **Mode:** Spectrum
  - b. **Lens Mode:** Hybrid
  - c. **Resolution:** 160
  - d. **Aperture:** Slot
6. **In the acquisition control section choose type “Snapshot”**
7. **Enter the region settings as follows:**
  - a. **Open the Element List window**
  - b. **Choose element list from the pull down Windows menu**
  - c. **Click on a peak of interest in the Real Time Display Window**
  - d. **Click on the associate element in the element list, and the scan settings for that element will be automatically loaded in the acquisition region settings.**
  - e. **Modify the region settings if needed (e.g., Peak value, width eV, dwell, etc.)**
8. **In the acquisition control section, click on the “On” button**
9. **In the manipulator section, enter in the desired Auto Z parameters. A typical set of values are:**
  - a. **No. of increments:** 16
  - b. **Z increment:** 0.05mm
  - c. **Ordinate choice:** Area (This optimizes Z based on the peak area)
10. **Click on “Optimize Selected Line”**

## Start up procedure for when computer or software has been shut down

1. **Turn on computer** (You will not need to log into Windows)
2. **Double click on the “Zones” icon on the desktop and click OK on the error message that appears.** The Zones taskbar should appear at the top of the screen.
3. **Double click on the “Shortcut to Manager” icon on the desktop** to open the manager software
4. **Click on the Window menu and:**
  - a. **Open manual window**
  - b. **Open real time display**
5. **Left click on the Zones button in the top task bar and choose “Control”**
6. **Drag the manual window into the manual zone**
7. **Drag the real time display window into the manual zone and into the manager zone**
8. **Calibrate the stage as follows:**
  - a. **Go to the manipulator section in the manual window**
  - b. **Click on the “parameters” box**



c. Click on “calibrate axes”



- d. Verify that the dual anode gun located on the back of the SAC chamber is all the way out (away from the sample stage), and verify that nothing is sticking out from the sample stage
  - e. Click on “confirm request”. The stage will go through a series of motions to reach the stage limits and calibrate the stage position
  - f. When completed, the calibrated light will illuminate green
9. Click on the position box in the manipulator section of the manual window
  10. Click on “Read Positions”
  11. Go to C directory and double click on file: /C=/Stage Positions
  12. Choose Bar.dset

*Note: This assumes you are using a bar type sample holder. Load and unload positions for other types of sample holders can be found in the same directory.*

## Acquisition Conditions Reference Sheet

Note that these parameters should only be considered as a reasonable starting point. Acquisition parameters may be optimized dependent on the instrument or overall experimental goal.

I. Charge Neutralizer Conditions – exact conditions may vary slightly between instruments.

Charge balance (2.6 – 3.2 V)

Filament current ( 1.6 – 2.1 A)

Filament Bias (0.9 – 1.3 V)

II. Spectroscopy (Based on use of **monochromatic** x-ray source)

III.

A. Large area (300um x 700um) – slot

Energy Range	Magnification	Pass Energy eV	Step Size eV	Time (min's)
Survey	<b>hybrid</b>	160	1	1 - 3
Region	<b>hybrid</b>	10 or 20	0.1	1 - 10
Valence Band	<b>hybrid</b>	20 or 40	0.2	5 - 20

B. Small area – small area spectroscopy must be referenced from medium magnification images for following spot sizes

** Spot size (Physical size and name in software)	Magnification	* Pass Energy	Step Size eV	Time (min's)
110 um (2mm Physical)	<b>FOV2</b>	10 – 40	0.1	1 - 4
55 um (1mm Physical)	<b>FOV2</b>	10 – 40	0.1	1– 10
27 um (0.4mm Physical)	<b>FOV2</b>	20 – 80	0.1	3 – 15
15 um (0.15mm Physical)	<b>FOV2</b>	20 – 80	0.1 – 0.2	5 – 30

\* use 160 eV for surveys

\*\* a predetermined iris setting must be used for each spot size

IV. Imaging (parallel)

A. Elemental

Goal	Magnification	PE (eV)	Acq. Time (min's)
General Alignment	<b>FOV1</b>	160	1
Small area spec. Position referencing	<b>FOV2</b>	160	1 – 5
Highest Lateral Res.	<b>FOV3</b>	160	2 – 8

C. Chemical State and generating spectra from images

Goal	Magnification	PE (eV)	Acq. Time (min's)
General Alignment	<b>FOV1</b>	40 or 80	1
Small area spectroscopy. Position referencing	<b>FOV2</b>	40 or 80	1 - 10
Highest Later Res.	<b>FOV3</b>	40 or 80	3 - 15