

Kratos UHV System XPS Data Collection

Focusing on the Sample

- 1) Verify the sample holder is loaded into the SAC and the flap valve is closed. Turn on the TV screen and light in the chamber if they are not already on.
- 2) Move the stage using the autostage manual control pad or the stage controls in the “Instrument Manual Control” window of the software. Find a position on your sample that is appropriate for data collection and position it at the center of the crosshairs on the TV screen.
- 3) In the “Analyser” panel of the “Instrument Manual Control” window, select “Mode: Spectrum,” “Lens: Hybrid,” “Resolution: Pass Energy 80 and slot.” If your sample is very sensitive in XPS, reduce the pass energy to 40.
- 4) In the “Acquisition” panel of the “Instrument Manual Control” window, select “Technique: XPS,” “Type: Snapshot, B.E.” Select the appropriate reference X-ray source, typically “Al (mono).”
- 5) Open the “Energy Regions Table” in the “Acquisition” panel. Type in the region name you wish to focus on (e.g. C 1s, Si 2p, Au 4f) and press enter. The region should autocomplete, but if it doesn’t, just manually enter in the desired center eV and press enter. Use a 500 ms dwell time for focusing with “# Sweeps” set to 1. Ensure the check box under “Active” is selected.
- 6) Verify the SAC pressure is $< 5 \times 10^{-9}$ Torr. If it is not, wait for the pressure to fall below this threshold.
- 7) Verify that the X-ray source you wish to use is selected in the “X-ray Gun” panel of the “Instrument Manual Control” window. Turn on the X-ray gun by pressing the “On” button in the “X-ray Gun” panel. Typical settings for the X-ray are 10 mA emission and 15 kV anode HT.
- 8) Press “On” in the “Acquisition” panel. It will take ~20 s for the acquisition to begin, so be patient.
- 9) In the “Real-Time Display” window, the counts per second (CPS) or peak area should be displayed on the left. To switch between CPS and area, right click in the middle panel on the left and select the desired units. You can also reset the trace from this menu.
- 10) Once the “Real-Time Display” window is showing counts, move the sample up or down (try up first) using the “Stage” panel in the “Instrument Manual Control” window. Watch the sample in the camera (TV or optical imaging panel in the “Real-Time Display window.” Maximize the count rate and ensure the crosshairs on the TV are still at the center of the area you wish to analyze.
- 11) If you are only running 1 sample, turn off the acquisition by pressing “Off” in the acquisition panel and proceed to the “Data Collection” section.
- 12) If you are collecting data on more than one sample, go to the “Stage” panel of the “Instrument Manual Control” window and select the “Table” check box. Enter a name for the first position and select “Update” to copy the stage coordinates into the first line.
- 13) Move the stage to the next position and repeat steps 10-12 to enter more positions into the table. Remember to press “Update” only when you have the correct line

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selected and you wish to copy the position coordinates into the line. Save the table in your folder when done.

- 14) When all of the positions are entered, press “Off” in the “Acquisition” panel to turn off the acquisition.

Data Collection – Instrument Manual Control Window

- 1) Create a dataset. To do this, open the “Data” folder on the desktop and navigate to your folder. Create a file for the data to be saved in and close the window.
- 2) To make this dataset active, select the “Acquiring” radio button in the main “Vision Instrument Manager” window. If a dataset is already open, it will be displayed as a file path in the top of this region of the window. If the file path is not all visible, drag the resizing tab at the bottom of the window to the right. Right click in the region of the window that is on the right of the file path and select “Open Dataset...” Navigate to your folder and open the folder you wish to save data in.
- 3) Ensure the parameters you selected for focusing on your sample are still present in the “Analyzer” and “Acquisition” panels of the “Instrument Manual Control” window. For survey scans, use “Resolution: Pass Energy 40 or 80 slot” and for high-resolution scans, use “Resolution: Pass Energy 10 slot.” Also verify that the X-ray is still on, and if it is not, simply press “On” in the “X-ray Gun” panel.
- 4) In the “Acquisition” panel of the “Instrument Manual Control” window, select “Type: Spectrum” and type the name of the region you wish to collect into the “Region Name” box and press enter. The line should autocomplete, but any of the entries can be manually changed. Remember to press enter when you change any of the region parameters. Change the number of sweeps to an appropriate number and press enter.
- 5) You continue to enter as many regions you wish to collect in the manual window. See the “Recommended Scan Parameters” section below if you need guidance.
- 6) Make sure the “Active” box is checked after all of the regions you want to collect, and not checked for any regions you wish to skip.
- 7) Press the “On” button in the “Acquisition” tab. Verify that the data collection starts in ~20 s in the “Real-Time Display” window. Once data collection has started, press the “Off” button in the “Acquisition” panel. This will turn off the data acquisition when the instrument has completed all of the regions that are active in the “Acquisition” panel.
- 8) When your scans are complete, make sure to set the X-ray to standby by pressing “Standby” in the “X-ray Gun” panel. There is no way to do this automatically using the “Instrument Manual Control” window.

Data Collection – Automatic Flow Chart

- 1) Create a dataset. To do this, open the “Data” folder on the desktop and navigate to your folder. Create a file for the data to be saved in and close the window.
- 2) In the “Vision Instrument Manager” window, select the “Create” radio button. Click “File” then “New run” to clear the open flow chart.

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- 3) If you are only running one position, select the “Dataset” radio button in the “Vision Instrument Manager” window and click “Browse...” Navigate to your dataset and type in a filename. Click “OK” and then use the middle mouse button to paste the dataset into the flow chart. Then skip to step 8.
- 4) If you are running multiple positions, select the “State Change” radio button and then the “Sample Position” radio button in the “Vision Instrument Manager” window.
- 5) Click “Load Position Table” to copy your position table from the “Instrument Manual Control” window into the sample position table. You can also select positions individually and copy them as desired by using the “Load Position” button.
- 6) Select the first position and click “Select Dataset...” Navigate to the folder you wish to save data in and type in a name for the position. Click “OK.” Repeat this step for all of your positions.
- 7) When finished assembling the position table, use the middle mouse button to paste the position table into the top of the flow chart.
- 8) Select the “Acquisition” radio button and select the scan parameters you intend to use. Follow the guide under the “Recommended Scan Parameters” section below. When each acquisition step is ready to be added to the flow chart, use the middle mouse button to paste it into the end. Remember to deselect the pasted step to avoid changing it while you are making the next step. If you paste something out of order, you can select the step, right click, and click “cut.” Then click on the step adjacent to where you want to paste the step, right click, and select “paste before” or “paste after.”
- 9) If you are using a position table and you have finished adding all of your acquisition steps, select the “State Change” and “Counter” radio buttons. Type in one less than the number of positions you are collecting ($n - 1$) and use the middle mouse button to paste the counter step to the end of your flow chart. Select the steps including the position table and all acquisition steps, right click, and select “loop back.”
- 10) Make sure that the last step will set the X-ray gun to standby. That is, under the “Acquisition” radio button in the “Standby Control” panel, the option “Goto Standby” should be selected in the last step. If you need to create a step after your looping section (for position tables), see the “Recommended Scan Parameters” section below for the parameters you should use.
- 11) Check all of your steps. The dataset or position table steps should be complete and the acquisition steps should all have the correct regions entered and the “Active” box should be checked. Make sure the pass energy is correct for each step, and the correct X-ray source with the correct power is selected.
- 12) Make sure the X-ray is on. If it is off, click the “On” button in the “X-ray Gun” panel of the “Instrument Manual Control” window and wait for it to stabilize. Make sure you have the same X-ray source selected in both the manual window and your flow chart.
- 13) Once you are sure the flow chart is assembled correctly, click the “Submit” button to send the flow chart into the queue. Then click “Resume” to start automatic data collection.

Recommended Scan Parameters

XPS Survey – Instrument Manual Control

- 1) In the “Analyser” panel, select:
 - a. Mode: Spectrum
 - b. Lens: Hybrid
 - c. Resolution: Pass Energy 40 eV or 80 eV – use lower pass energy for samples with strong XPS signals
 - d. Slot aperture
- 2) In the “Acquisition” panel, select:
 - a. Technique: XPS
 - b. Type: Spectrum and B.E.
 - c. Ref.: Al (mono) or select the alternative source you are using
 - d. Energy Regions Table:
 - i. Region Name: Wide or Survey, press enter
 - ii. Center eV: 597.5 eV
 - iii. Width: 1205 eV
 - iv. Step eV: 1 eV
 - v. Dwell: 100 ms
 - vi. # Sweeps: 1 – 5

XPS High-Resolution – Instrument Manual Control

- 1) In the “Analyser” panel, select:
 - a. Mode: Spectrum
 - b. Lens: Hybrid
 - c. Resolution: Pass Energy 10
 - d. Slot aperture
- 2) In the “Acquisition” panel, select:
 - a. Technique: XPS
 - b. Type: Spectrum and B.E.
 - c. Ref. Al (mono) or select the alternative source you are using
 - d. Energy Regions Table:
 - i. Region Name: type name (e.g. C 1s, Si 2p, Au 4f) and press enter
 - ii. Center eV: determine this from XPS databases (C 1s ~287 eV)
 - iii. Width: determine from XPS database, typically 12 – 16 eV
 - iv. Step eV: 0.025 eV
 - v. Dwell: 100 ms, can be adjusted higher as desired
 - vi. # Sweeps: start with 10 – 50

XPS Survey – Flow Chart

- 1) Select the “Acquisition” radio button in the “Vision Instrument Manager” window

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- 2) In the “Standby Control” panel, select “Leave On” unless this is your last step, in which case, select “Goto Standby.”
- 3) In the “Analyser” panel, select:
 - a. Mode: Spectrum
 - b. Lens: Hybrid
 - c. Resolution: Pass Energy 40 eV or 80 eV – use lower pass energy for samples with strong XPS signals
 - d. Slot aperture
- 4) In the “Excitation” panel, the “X-ray Gun” radio button should be selected and the correct X-ray source should be active. Typical X-ray gun parameters are 10 mA emission and 15 kV anode HT.
- 5) In the “Neutraliser” panel, the option “under manual control” should be selected. Only change this box if you have been trained on use of the neutralizer.
- 6) In the “Scan Control” panel, select:
 - a. Technique: XPS
 - b. Type: Spectrum and B.E.
 - c. Ref.: Al (mono) or select the alternative source you are using
 - d. Energy Regions Table:
 - i. Region Name: Wide or Survey, press enter
 - ii. Center eV: 597.5 eV
 - iii. Width: 1205 eV
 - iv. Step eV: 1 eV
 - v. Dwell: 100 ms
 - vi. # Sweeps: 1 – 5

XPS High-Resolution – Flow Chart

- 1) Select the “Acquisition” radio button in the “Vision Instrument Manager” window
- 2) In the “Standby Control” panel, select “Leave On” unless this is your last step, in which case, select “Goto Standby.”
- 3) In the “Analyser” panel, select:
 - a. Mode: Spectrum
 - b. Lens: Hybrid
 - c. Resolution: Pass Energy 10 eV
 - d. Slot aperture
- 4) In the “Excitation” panel, the “X-ray Gun” radio button should be selected and the correct X-ray source should be active. Typical X-ray gun parameters are 10 mA emission and 15 kV anode HT.
- 5) In the “Neutraliser” panel, the option “under manual control” should be selected. Only change this box if you have been trained on use of the neutralizer.
- 6) In the “Scan Control” panel, select:
 - a. Technique: XPS
 - b. Type: Spectrum and B.E.
 - c. Ref.: Al (mono) or select the alternative source you are using
 - d. Energy Regions Table:
 - i. Region Name: type name (e.g. C 1s, Si 2p, Au 4f) and press enter

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- ii. Center eV: determine this from XPS databases (C 1s ~287 eV)
- iii. Width: determine from XPS database, typically 12 – 16 eV
- iv. Step eV: 0.025 eV
- v. Dwell: 100 ms, can be adjusted higher as desired
- vi. # Sweeps: start with 10 – 50

XPS Standby – Flow Chart

- 1) Select the “Acquisition” radio button in the “Vision Instrument Manager” window
- 2) In the “Standby Control” panel, select “Goto Standby”
- 3) In the “Scan Control” panel, select:
 - a. Technique: XPS
 - b. Type: Spectrum and B.E.
 - c. Ref.: Al (mono) or select the alternative source you are using
 - d. Energy Regions Table:
 - i. Region Name: type C 1s or any other region
 - ii. Center eV: 287 eV or any number 1 – 1000
 - iii. Width: 1 eV
 - iv. Step eV: 1 eV
 - v. Dwell: 100 ms
 - vi. # Sweeps: 1

Angle-Resolved Measurements

- 1) Angle-resolved measurements can be performed. In the “Stage” panel of the “Instrument Manual Control” window, check the “Increment” box.
- 2) Make sure you are focused on your sample, enter a name for the first position in the “Table” and press “Update” to copy the position coordinates into the table.
- 3) Under “Increment,” select “Position Using Axis of Rotation” from the dropdown menu.
- 4) In the box with “#” next to it, enter the number of different angles you wish to wish to collect.
- 5) In the box labeled “Theta X” enter the increment by which you want to change the angle. Use a negative number to rotate the sample towards the Al mono source, or a positive angle to rotate it towards the Mg source.
- 6) If you want to collect 3 angles that are 25 degrees apart using the Al mono source, the numbers you would use are “#: 3” and “Theta X: -25” to collect at 0, 25, and 50 degrees.
- 7) Click “Apply” and the table should autocomplete with the desired positions and angles.
- 8) Check that the instrument can navigate to each position safely by using the “Go to” button. Make sure it isn’t going to hit the magnetic lens below the sample or the Mg X-ray gun (copper tube) at the back of the chamber.

Exporting Data to .vms or .txt formats

- 1) Open Vision Processing on the desktop
- 2) Click “File” then “Open Dataset for Processing...”
- 3) Navigate to your folder and select the dataset you wish to open. If you are opening multiple datasets, select them and click “Apply” to open them individually. When finished, click “OK.”
- 4) Resize the “Vision Processing” window so you can see most of your datasets. Select the regions of the first dataset you wish to export and click “Options” then “Browser Actions...”
- 5) Click the “Describe” arrow at the top right of the “Browser Actions” window and select the “Vamas File” radio button. Then click “Apply. Navigate to the folder you wish to export the data to and give the exported file a name, usually the same as the .dset file. The click “OK”
- 6) Export as many files as you want to the .vms format this way. When finished, close the “Vision Processing” window.
- 7) To export to .txt, use CasaXPS. Open the CasaXPS program on the desktop. Click “File” then “Open...” and find your folder with the data you wish to export. Then click “Open” and your data should appear.
- 8) Fit the regions of your data as desired using CasaXPS.
- 9) Highlight the region on the right you wish to export. You can export multiple regions into the same file by selecting multiple regions with shift + click or ctrl + click, but this will put all of the data into a single .txt file and there won't be a clear delineation between different regions in the exported file. It is usually best to export each region individually.
- 10) Make sure you last clicked in the right half of the screen and the option “Save Tab ASCII” is active. Then click this button (top row, button 10 from the left), give your file a name, and click “Save.”