

# Kratos AXIS Ultra DLD X-ray Photoelectron Spectrometer

## Instructions

**Note:** Enter your complete name, CMRF user code, and date into the record book. You must have previously filled out a project information sheet.

**Note:** Vision Manager and Instrument Manual Control control the instrument, Vision Processing is used for converting data files.

### 1. About XPS:

- XPS measures the top 10 nm of samples. You only need 110 um for sample (tiny piece).
- Samples can be set up the night before with vacuum if you want to use XPS in the morning (wouldn't be charged for vacuum overnight).
- A survey scan scans all elements and only takes 2-3 minutes.
- For trace elements, a core level scan can have 2000 ms dwell time for a high resolution (publication quality) or 1000 ms for non-publication data. One element takes 10 to 20 minutes to scan.

### 2. Sample prep:

- With gloves on, scrape samples off with a razor blade and clean the holder with isopropanol.
- With clean tweezers, load samples onto the sample holder—samples may be loaded directly onto carbon tape, cemented on with carbon/silver paint or on metal (Si, In, etc.) mounted on the tape. (Samples go on top of sample holder, which is a thinner metal strip than the bottom. The end with 2 posts between the top and bottom will go into the XPS first). Keep samples toward the center of the sample holder.
- Press samples onto the mounting medium with a clean spatula. Tip the sample holder to the side and tap the side of the sample holder with mounted sample(s) on clean surface to remove all loose sample material.

### 3. Loading the sample(s) and setting up vacuum:

**Note:** If the Instrument Manual Control window is not open, then in Vision Manager go to “Window” and select “Manual Window.”

**Note:** If Vision Manager is not open, then consult the restarting instructions or contact a staff member.

- Ensure that the STC-SAC valve is closed (Red).
- Loosen (by partially unscrewing) the knobs on the door (by the sample exchange probe).
- Vent the STC (in “Vacuum” in Instrument Manual Control).
- Turn on the flow of N<sub>2</sub> Gas on the bottle and flow valve (on the pipe).
- Hold the probe (support the weight) and completely unscrew knobs.
- When the door seal opens a little (enough to open the door) turn off the N<sub>2</sub> Gas at both the bottle and the flow valve.

**Note:** The door seal gasket will probably fall out—Do not let it fall on the ground—Catch the gasket when you open the door.

- Open the door all the way and turn the probe knob clockwise until the claw end of the sample exchange rod is visible.
- Put the sample holder on the claw.
- Turn the probe knob counterclockwise until the light at the end of the probe with the cord is green.
- Reseat the door seal gasket. Be sure it does not come out when trying to close the door—if the seal gets pinched in the door, the STC will not pump down
- Close the door and screw the door knobs back in (Do not overtighten).
- In Instrument Manual Control, go to “vacuum” and pump STC.
- Support the probe until the vacuum has sealed the sample exchange port.

**Note:** The STC on the “Vacuum” schematic will go from pink to blue to cyan to orange - orange is good and indicates the pump is working. If this does not happen, lift the probe to support it and retighten the screws. Do not leave until you are sure the pump is working. When the STC turns yellow, you will start to see vacuum values.

**Note:** In the meantime, in “X-ray” (In Instrument Manual Control) click “standby” to warm up the source so that this is completed during vacuuming. This vacuum step takes 40 minutes to 1 hour or longer depending on the type and number of samples and the amount of sample outgassing. You want to see “5x10e-7” or better in the STC. (in the meantime, follow part 4 for setting up data collection).

#### 4. Setting up data collection:

**Note:** Data may be collected point by point manually in instrument manual control or by setting up a flowchart of points in Vision Manger. Following are the instructions for using Vision Manager for analysis.

- Make a folder with the user name in the data folder on the desktop.

**Note:** In Vision Manger, there are three function buttons near the top of the Vision Manager window that will be used to set up a flowchart: “Dataset,” “Acquisition” and “State Change.” A typical run starts with a “Dataset,” followed by one or more “Acquisitions” and ending with a “State Change” or a change to a new sample position. See below:

- “Dataset” is the file where subsequent scans are saved. A new “Dataset” must begin every series of analysis or all of the scans will be saved to one file.
- “Acquisition” is where the user can set up a scan of energies. A full energy range scan is called a “Survey” scan. A scan focused on a specific element orbital energy is called a “Region” or “Core Level” scan.
- “State Change” is where the user can change analysis type or conditions, such as sample position.

**Note:** In Vision Manager, a flowchart from a previous run can be loaded and modified for current acquisition or a new flowchart can be created. To load a flowchart from a previous run, select “File,” “Load Run” and navigate to a previous run. To create a new flowchart, follow the instructions which follow.

**Note:** In Vision Manager, type file names without spaces and names that are fewer than 30 characters in length. If you need a space, use an underscore (\_).

**Note:** In Vision Manager, the first “Dataset” and “Acquisition” do not need a starting sample position in the flow chart (you will move to the first sample position manually in Manual Control before starting the run).

-Select the “Dataset” button in the top center of the screen. Type a name for the dataset in the “Name” field. In the “Filename” field, browse to and select your folder in the data folder. In the “Selection” box, type the name for the dataset at the end of the filename (eg. /C=/data/yourfoldername/**datasetname**). To add the “Dataset” to the flowchart, move the mouse cursor to the flowchart section of Vision Manager in the upper left and click with the mouse scroll wheel.

-Select the “Acquisition” button in the top center of the screen.

For a “**Survey**” or wide energy range scan:

-Name the acquisition “Survey” or another name to indicate a wide energy range scan.

**Note:** Be sure to press enter after any manual typed changes. If you see a vertical black bar next to a field that has been manually altered, then you need to press enter to save the changes.

In “Standby Control”:

-Under “Action on Completion” select “Leave On.” If this acquisition is the final acquisition in the flow chart, then refer to the instructions below for the last sample.

-In “Analyser”:

-Under “Mode” select “Spectrum.”

-Under “Lens” select “Hybrid.”

-Under “Resolution” select “Pass energy 160.”

-Choose the “Slot” for the aperture.

In “Excitation”:

-Select “Xray Gun.”

-Under “Source” select “Mono (Al).”

-Under “Emission (mA)” enter 10 mA.

-Under “Anode HT (kV)” enter 15 kV.

In “Neutraliser”:

-Choose “On for Acquisition” for non-metal samples, or choose “Under Manual Control” for metal samples.

In “Scan Control”:

-Under “Technique” select “XPS.”

-Under “Type” select “Spectrum.”

-Choose “B.E.” for binding energy.

-Under “Ref.” (reference) select “Al (Mono).”

-Select the box for the “Energy Regions Table.” In the table:

-Under “Region Name,” type “survey” and press enter to populate the table.

-Set the “Start eV” to 1200 eV. If “Centre eV” is displayed, right click on “Centre eV” and select “Start eV” to change.

-Set “End eV” to -5 eV.

-Set “Step eV” to 1 eV.

-Set “Dwell ms” to 200 ms.

-Set “# Sweeps” to 2 sweeps.

To add the "Aquisition" to the flowchart, move the mouse cursor to the upper left, flowchart section of Vision Manager and click with the mouse scroll wheel.

For a "Region," "Core Level," or narrow energy range scan:

-Name the acquisition "Region" or another name to indicate the narrow energy range scan.

In "Standby Control":

-Under "Action on Completion" select "Leave On." If this acquisition is the final acquisition in the flow chart, then refer to the instructions below for the last sample.

-In "Analyser":

-Under "Mode" select "Spectrum."

-Under "Lens" select "Hybrid."

-Under "Resolution" select "Pass energy 20."

-Choose the "Slot" for the aperture.

In "Excitation":

-Select "Xray Gun."

-Under "Source" select "Mono (Al)."

-Under "Emission (mA)" enter 10 mA.

-Under "Anode HT (kV)" enter 15 kV.

In "Neutraliser":

-Choose "On for Acquisition."

In "Scan Control":

-Under "Technique" select "XPS."

-Under "Type" select "Spectrum."

-Choose "B.E." for binding energy.

-Under "Ref." (reference) select "Al (Mono)."

-Select the box for the "Energy Regions Table." In the table:

-Below the table select "Clear All Rows."

- Under "Region Name," type a region of interest (e.g. C 1s, O 1s, Cl 2p, etc.) and press enter to populate the table.

**Note:** The neutralizer will shift the binding energy peak to a lower value, so manually subtract 2 eV from all end eV values. If "Centre eV" is displayed, right click on "Centre eV" and select "Start eV" to change.

-Set "Dwell ms" to 1000 ms.

-Set "# Sweeps" to 1 sweeps.

To add the "Aquisition" to the flowchart, move the mouse cursor to the upper left, flowchart section of Vision Manager and click with the mouse scroll wheel.

-Select the "State Change" in the top center of the screen.

-Select "Position Change" from the button options under the "State Change" button. Enter a name for the position (usually a sample name) and press enter.

To add the "Position Change" to the flowchart, move the mouse cursor to the upper left, flowchart section of Vision Manager and click with the mouse scroll wheel. An actual sample position will be added to the sample position table later.

**Note:** The first position change will be to the location of your second analysis point, as you start analysis already positioned on your first sample.

Additional “Datasets,” “Acquisitions,” and “Position Changes” can be added to the flow chart for each additional sample. You can manually create “Datasets,” “Acquisitions,” and “Position Changes” following the above instructions. Alternatively, you may select one in the flowchart, edit it in the central window section of Vision Manager for your next sample and then paste it into the flowchart.

For the last sample in the flowchart, in the last “Acquisition” and in “Standby Control,” under “Action on Completion” select “go to standby.”

In Vision Manager:

-Select “File” and “Save the Run.” In the “Filename” field, browse to and select your folder in the data folder. In the “Selection” box, type the name for the dataset followed by “flowchart” at the end of the filename (eg. /C=/data/yourfoldername/**datasetname/flowchart**). This saves the flowchart schedule so that it can be loaded again and modified for additional analysis.

#### 5. Transferring the Samples for Analysis

- In Instrument Manual Control, scroll to the “Stage” window.
- At the bottom of the Positions table select “Clear all Rows.”
- Select “Load Table” and select “Load.dset” under “Files” and click “Ok” (this will load the preset load position of the analysis claw in the SAC)
- Click on the load position xyz coordinates line in the table to highlight the row and click “Go To” above the table to drive the SAC analysis claw into the load position.
- When pressure is less than  $5e-7$  torr (ideally) in the STC, open the STC-SAC valve. Be sure the light on the end of the transfer probe is green.
- Turn on the light source (KL1500)
- Insert the sample into the SAC by rotating the knob on the sample exchange probe clockwise. Watch the sample holder through the port and make sure it loads onto the sample analysis claw.
- Take the sample exchange probe hook off the sample holder by moving it “left” (moving it towards you. To go faster, while holding “left,” also press “right”) using the remote control (the Autostage Manual Controller) or the stage controls in Manual Control.
- Turn the sample exchange probe knob to take the probe out.
- Close the STC-SAC valve in “Vacuum” in Instrument Manual Control.

#### 6. Setting up sample positions:

- Turn on the TV monitor. The small “x” on the screen is the location of analysis.
- Adjust the camera field of view to the widest field of view to locate the samples. Adjust the camera using the adjustment ring in the slot near where the camera is attached to the SAC.
- Move the sample holder using the remote control until the first sample is centered.
- Decrease the camera field of view to the smallest field of view.
- Adjust the X, Y, and Z position of the sample to get the sample in focus. The sample should be in focus from the NW corner, through the center, to the SE corner of the image. The NE and SW corners should be slightly out of focus.

**Note:** The camera image (on the TV) is also viewable through the software. In Vision Manager, at the top, select “Window” and select “Real Time Display” from the dropdown options. Move the “Real Time Display” window to the right computer monitor and maximize it (make it use the whole

screen). In Instrument Manual control, turn on (click the button at the top of the screen) and scroll to the "Optical Image" section. Select "View" and the image should appear in the "Real Time Display" window in the lower left.

**Note:** The stage can be moved using the X, Y, and Z controls in the "Stage" section in Instrument Manual Control, instead of using the remote control.

-In "Instrument Manual Control," scroll to the "Analyzer" section.

-In "Analyser":

-Under "Mode" select "Spectrum."

-Under "Lens" select "Hybrid."

-Under "Resolution" select "Pass energy 160."

-Choose the "Slot" for the aperture.

-Scroll to the "Acquisition" section.

-Under "Type" select "Snap Shot."

-Below the "Energy Regions Table," select "Clear all Rows."

-Enter an element present in your sample that you want to analyze, e.g. C 1s (click "enter" to populate the table, be sure there is a space between C and 1). Check with the binding energy tables on the wall to make sure the window is centered on the peak.

-In Vision Manager, at the top, select "Window" and select "Real Time Display" from the dropdown options. Move the "Real Time Display" window to the right computer monitor and maximize it (make it use the whole screen). Right click on the window in the middle of the left side of the "Real Time Display" window, and select "Trace Counts" (cts/sec).

-In Instrument Manual Control:

-Scroll to the "Neutraliser" section.

-Turn the neutraliser "On."

-Scroll to the "X-ray Gun" section.

-Turn on the X-ray source (should hear a click and water - if you don't hear these, go to standby and try to turn back on). If there is trouble turning the X-ray source on, use set the filament to 6 mA and anode to 10 kV; after it is stable, change the values to the normal 10 mA and 15 kV, respectively

-Scroll to the "Acquisition" section.

-Turn on the acquisition. After a few seconds, snapshot acquisition should appear in the "Real Time Display" window.

**Note:** If a scan does not start to appear in the "Real Time Display" window after 1 minute, you may need to activate the real time display by selecting the "Acquiring" button in the left center of the Vision Manager window. If acquisition stays off after activating the "RTD" window, you can click "Restart" in the "Acquisition" section of Instrument Manual Control which will reset the acquisition. If you see "x"s in the element table, delete that row and start again, as rows with "x"s cannot be edited.

-Move the sample (X, Y, and Z) to maximize the signal intensity (watch the numbers in the NW corner of the spectra display and the values in the count trace in the "Real Time Display"). If the sample is well focused, counts can reach 200k or 300k. In "Snapshot" mode, look for 1k to 2k counts per second (cps).

- When the signal is maximized:
- Turn off "Acquisition."
- Set the X-ray source to "Standby."
- Turn off the "Neutraliser."

-In the "Stage" section:

- Below the sample "Positions" table, select "Clear All Rows."
- Select the row for the sample position (Click on the row so that it is highlighted by a black rectangle). Click "Add" to add the sample position in the next available row or click "Update" to change the information in the selected (highlighted) row.

-Follow the instructions above to navigate to another sample, maximize the signal intensity, and add the sample position until all sample positions are listed in Instrument Manual Control in the "Stage" section.

-Select the first sample position in the "Stage" section in Instrument Manual Control and click "Go To." This moves the stage to the first sample for the start of analysis.

-Select the second sample position in the "Stage" section in Instrument Manual Control (it should be highlighted with a black rectangle).

-In Vision Manager:

-Select the first "Position Change" in the flowchart (this should represent moving from the first, starting sample location to the second sample location, prior to analysis of the second sample).

-With the second sample position highlighted in Manual Control, click "Load Position" in the Vision Manager "Position Change" window.

-Highlight the next sample position in Instrument Manual Control and then update the next "Position Change" in the Vision Manager flowchart. Do this for all positions in the flowchart.

-Resave your flowchart.

7. Running the scans:

-Turn off the TV monitor and light source (KL1500, inside the SAC) and the light in the room for the rest of the run.

-Click "Submit," and then "Resume" at the top of the Vision Manager window and monitor the X-ray source in Instrument Manual Control to make sure it turns on for the first scan. If there are any problems with the X-ray source not working, click "Standby" for the X-ray source in Manual Control, click "Stop Run" in Vision Manager. Next, in Vision Manager click "Resume" to start the run again. Stay long enough to ensure that the instrument goes from "Survey" to "Region" scans automatically.

**Note:** If the run shifts to manual and does not continue from "Survey" to "Region" scans automatically, either manually click "Resume" to continue the scans between each transition or check the calibration of the iris and aperture. To check the calibration of the iris and aperture, in Instrument Manual Control open the iris and aperture windows at the top of the window and then scroll down to the open iris and aperture windows. Both should have a green light bulb indicating that they are calibrated. If not, then click the lightbulb next to "Initialise," then the lightbulb next to "Confirm." Then click the lightbulb next to "Calibrate," then the lightbulb next to "Confirm." This procedure will work to calibrate both the iris and the aperture. With the iris and aperture calibrated the run should continue without stopping between scans.

#### 8. Exporting data files:

- Open "Vision Processing" on the desktop.
- In "Vision Processing," go to "File" and "Open Dataset for Processing."
- Navigate to your user folder and select the dataset to process under "File."
- Select data files for processing (ie. Survey scan, Region scan).
- In "Options," select "Browser Actions."
- Click the button next to "Describe."
- Select "Vamas file" and click "apply." You will be prompted to save the Vamas files. Navigate to your folder and write in a file name in selection (eg. /C=/data/yourfoldername/**datasetname**). Click "Ok." In your data folder, you will have the .vms file(s). Save it/them to a USB (Eventually we will save to a network drive). CasaXPS will open .vms files for data interpretation.

**Note:** Record your analysis time (XPS, UPS, ion gun, electron imaging) in the log book.

#### 9. Removing sample:

**Note:** If the Instrument Manual Control window is not open, then in Vision Manager go to "Window" and select "Manual Window."

**Note:** If Vision Manager is not open, then consult the restarting instructions or contact a staff member.

-In Instrument Manual Control, scroll down to "vacuum." If the sample is in the SAC, open the STC-SAC valve (will be green if open).

**Note:** The STC-SAC valve will not open if the sample transfer probe is moved and has a red light (must be green).

- Turn on the light source (KL1500)
- In Instrument Manual Control scroll the "Stage" window.
- At the bottom of the Positions table select "Clear all Rows."
- Select "Load Table" and select "Unload.dset" under "Files" and click "Ok" (this will load the preset unload position of the analysis claw in the SAC)
- Click on the unload position xyz coordinates line in the table to highlight the row and click "Go To" above the table to drive the SAC analysis claw into the unload position.
- Turn the knob on the sample exchange probe clockwise and align the claw with the post on the sample holder.
- Use the black controller (the Autostage Manual Controller) and press "right" (to go faster, while holding "right," also press "left"). Jiggle the sample transfer probe knob to hook the sample holder.
- Pull the sample exchange probe out using the knob until you see a green light at the end of the probe.
- Close the STC-SAC valve (Instrument Manual Control in "Vacuum").
- Ensure that the STC-SAC valve is closed (Red).
- Loosen (by partially unscrewing) the knobs on the door (by the sample exchange probe).
- Vent the STC (in "Vacuum" in Instrument Manual Control).
- Turn on the flow of N<sub>2</sub> Gas on the bottle and flow valve (on the pipe).
- Hold the probe (support the weight) and completely unscrew knobs.
- When the door seal opens a little (enough to open the door) turn off the N<sub>2</sub> Gas at both the bottle and the flow valve.



**Note:** The door seal gasket will probably fall out—Do not let it fall on the ground—Catch the gasket when you open the door.

- Open the door all the way and turn the probe knob clockwise until the claw end of the sample transfer probe is visible.
- Remove the sample holder on the claw at the end of the sample transfer probe.
- Turn the probe knob counterclockwise until the light at the end of the sample transfer probe is green.
- Reseat the door seal gasket. Be sure it does not come out when trying to close the door—if the seal gets pinched in the door, the STC will not pump down
- Close the door and screw the door knobs back in (Do not overtighten).
- In Instrument Manual Control, go to “vacuum” and pump STC.
- Support the probe until the vacuum has sealed the sample exchange port.

**Note:** The STC on the “Vacuum” schematic will go from pink to blue to cyan to orange - orange is good and indicates the pump is working. If this does not happen, lift the probe to support it and retighten the screws. **Do not leave** until you are sure the pump is working. When the STC turns yellow, you will start to see vacuum values.