

Kratos AXIS Ultra X-Ray Photoelectron Spectrometry

Instructions

Do not bring loose powders, or any other similar material, into CEMMA!

Prepare you sample stubs in your own lab!

Calibration (Do not load sample)

0. Never run the calibration procedure with a sample in the XPS.
1. With chamber empty run calibration (Manager, open>Windows>Real-time display>Manual control).
2. In manual control window, Stage>Calibrate>Confirm

Sample loading

0. Make sure the sample holder is clean. Use compressed air can to blow off any loose debris.
1. Mount sample on the sample holder. Make sure the sample height is less than 5 mm measured from the top of the sample holder.
2. Make sure sample is not protruding from the edge of the sample holder.
3. Load the sample holder onto the transfer rod and latch the chamber door.
4. Turn on the turbo pump (green switch, pump on floor).
5. Close the N2 tank.
6. Wait until the BOTTOM #2 gauge is $5\text{E-}7$ Torr (this may take 30 minutes).
7. Turn the exchange chamber valve (black valve) counter-clock-wise. Wait another 20-30 minutes until TOP #2 gauge $< 1\text{x}10^{-7}$ Torr.
8. In "Manual Control" window make sure Automatic Sequence is engaged on the Vacuum page.
9. Double check that both the TOP and BOTTOM #2 gauges are at $1\text{E-}7$ Torr.
10. Check the sample transfer rod light is green (light is at the far end of the transfer rod).
11. Open STC to SAC using the button on the Vacuum page.
12. Use the transfer rod knob to move the sample holder into the SAC chamber.
13. If you have not been certified to proceed, stop. Check with CEMMA staff.
14. This part is dangerous to the instrument! Transfer the sample holder onto the SAC fork mount.
15. Retract the transfer rod until the sample transfer rod light is green.
16. Close STC-SAC valve (Never leave the instrument while this valve is open).
17. Close the exchange chamber valve manually by rotating it clock-wise.
18. Do not turn off the pumps!

Data acquisition

Vision Manager – control of XPS

0. After closing STC-SAC valve and before actually starting a run, allow the SAC to pump approximately 30 min. Do not begin XPS run until SAC pressure is below $4\text{E-}8$ torr
1. Adjust the laser stop to be coincident with the black X on the TV screen.
2. Follow the further instructions on the table. Never exceed 6mA anode current and 10 kV anode voltage.

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XPS shut down

1. Turn off X-ray source (make sure it is not just in standby).
2. Move the sample holder to the “unload position”.
3. Open the exchange chamber valve and wait until both TOP and BOTTOM #2 gauges are at 1E-7 torr.
4. Open the STC-SAC valve.
5. Insert transfer rod and move into position near sample holder.
6. Be careful when removing the sample holder from the SAC sample fork. This procedure is dangerous to the microscope! Check with CEMMA staff if you are uncertain that you can safely complete this task.
7. Extract the sample holder from the SAC and move left until sample transfer rod light is green.
8. Close STC-SAC valve and the exchange chamber valve.
9. Turn off turbo pump.
10. Vent the exchange chamber (this may take several minutes).