



This guide obtains just brief information. In case you need detailed instructions, read KRATOS "Getting Started Guide" manual. It is recommended to read the **Safety Instruction and Troubleshooting** manual before your work.

## **KRATOS-XPS**

## **Obtaining XPS spectra**

- Mount your samples on an appropriate bar (sample holder). Samples must not overlap the bar (maximum sample height is 7 mm). When a sticking tape is needed, use a copper one (conductive) or a Kapton one (insulated) – both are located in the flow box next to Kratos spectrometer.
- 2. Vent the Flexi-lock chamber by clicking on the **Vent** button in the **Sample Loader** window and loosen the Flexi-lock swing bolt to prevent over pressuring.
- 3. When the chamber is vented (it takes ~3 min), open the door, turn the latch clockwise, pull

magazine out and put the bar on a free slot (prefer slot 2). Ensure that the bar is laying on the pin properly (check it by gentle shaking). <u>Align</u> the bar in the slot by pushing it towards the Flexi-lock (as shown in the figure).



Slowly push the magazine fully in and turn the latch on the magazine anti-clockwise to secure it.

- 4. Close the Flexi-lock door, move the swing bolt into position and <u>loosely</u> secure it. Press the **Pump** button in the **Sample Loader** window and fill the **Magazine Contents** dialogue which pops up. It is very important to specify exactly, which slot is free and which is occupied (photo of the bars is shown overleaf). After pressing **OK**, <u>push</u> the door on the hinge side, until you hear start of pumping (sound of opening valve). Check in the **Status window** whether the Flexi-lock pressure starts to decrease. If not, release the swing bolt and push the Flexi-lock door again now uniformly. If the pressure does not start to decrease in 5 mins, restart the ESCApe software, press again the **Pump** button, confirm the dialogue and push the door.
- 5. If the bar is in the magazine slot 1 or 2, you can take an image of the bar when the message Pumping Flexi-lock completed appears. If the bar is in the magazine slot 3, you have to wait to pass pressure conditions mentioned in paragraph 7. To take an image press the Move to camera position button (the Magazine Section in the Sample Loader window), then in the Camera Image section click on the Start loader camera button to see a live image of your bar. To save the image, click on the Stop loader camera [button.]
- 6. Define analysis locations on your bar. Open the Analysis Location window and select your Sample Holder to view the desired image. To create a new analysis location, click on the Select multiply items button, if the dialogue. In the list of locations, unselect the checkbox if you do not need charge neutralisation (if needed, leave the checkbox selected). Default charge neutraliser parameters are fine for most samples, however, the filament current can be changed by the user. Do not set the current higher than 0.52 A. Set an experiment where all the measured data from the specified location will be saved to. A new experiment can be created in the Data organizer window.





- 7. If pressure in the Flexi-lock is in the -7 Torr order (or lower) and the pressure difference between the Flexi-lock and the SAC is less than 2.5 orders of magnitude, you can transfer the bar to the SAC by clicking on the Transfer the sample holder to the stage button (Sample Loader window). Wait for the message Transfer holder to stage completed.
- 8. Set correct *z* positions (heights) for your analysis locations. This can be done either by focussing a microscope image on the sample surface or by the *AutoZ* method ("*D*:\*ESCAPE"*). For the former, in the **Analysis location** window, select your location in the list and in the **Positioning** section click on the button **Move to platen position**.
  Click on the **Microscope** button, set the microscope zoom to 12 (max. zoom in) and focus the microscope image by changing the *z* position (do not go lower than -7 mm). Press the **Update position** button is the new position.
- 9. Before running XPS analysis, be aware that to prevent damaging of the detector, it is important to keep maximum intensity below 1 000 000 counts/sec all the time.
- 10. Open the Acquisition method window. For start, open method "safe wide spectrum.acquisition" located in "D:\ESCAPE\" folder. The "safe wide spectrum" method ensures collecting of wide (survey) spectra with reduced intensity (emission 5 mA, resolution 80) which cannot go over 1 000 000 cps for any sample. If the max. intensity is below 200 000 counts/sec, emission can be increased to maximum allowed value of 15 mA. High resolution spectra of peaks are usually obtained using emission 15 mA and resolution 20.
- 11. To start data acquisition click on the **Submit location for acquisition** blue arrow button in the location list. The experiment will be added to the queue in the **Analysis** window. Select the **Auto** radio button and press **Start** (The software will perform consequent experiments automatically).
- 12. When finished, move the bar to the Flexi-lock by clicking on the **Transfer the sample holder from the stage** button to a free magazine slot. Then vent the Flexi-lock, remove the bar and pump the Flexi-lock. Check Flexi-lock pressure before leaving, it should be in the -6 Torr order (or lower).



Plain dual height Plain + stub end entry Plain + stub side entry Heat and Cool Azimuthal