KRATOS–XPS
Obtaining XPS spectra

1. 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.

2. 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. Put the bar on a free slot of the magazine. Ensure that the bar is laying on the pin properly (check it by gentle shaking). Align the bar in the slot by pushing it towards 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.
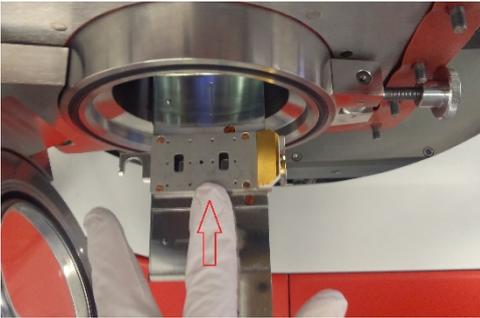
3. Close the Flexi-lock door, move the swing bolt into position and loosely secure it. Press the **Pump** button in the **Sample Loader** window and fill the **Magazine Contents** dialogue which pops up. It is very important to specify exactly, which slot is free and which is occupied (photo of the bars is shown overleaf). After pressing **OK**, push the doors on the hinge side, until you hear the Flexi-lock backing valve opening. Check in the **Status window** whether the Flexi-lock pressure starts to decrease. If not, try to press again the Flexi-lock door – now uniformly (maybe the swing bolt needs to be released). Wait for the message **Pumping Flexi-lock completed**. (This message is usually shown before the system really starts to pump).

4. Once the message **Pumping Flexi-lock completed** appears, you can take an image of your bar. If the bar is in the magazine slot 1 or 2, you can do that immediately after the message appears. However, if the bar is in the magazine slot 3, the gate valve must be opened to transfer the bar to the camera position. The gate valve is allowed to be opened only if the pressure difference between the Flexi-lock and the SAC (sample analysis chamber) is low enough (max. 2.5 orders of magnitude). 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.

5. 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, then click on the desired location in the image, press the **Add location** button and fill 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.

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6. If the pressure difference between the Flexi-lock and the SAC is less than 2.5 orders of magnitude and pressure in the Flexi-lock is in the -7 Torr order (or lower), 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**.

7. 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. For the former, in the **Analysis location** window, select your location in the list and 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).

8. Press the **Update position** button to save the new position. The latter method for setting *z* position AutoZ can be loaded in the **Acquisition method** window from "*C:\ESCAPE*" folder and submitted for the acquisition according to point 9.

9. Open the **Acquisition method** window. Set **Mode** to Spectroscopy, **Type** XPS-monochromatic, **Tuning** Spectroscopy, **Emission current** (standard 15 mA), **Collimation mode** Slot, **Lens mode** Hybrid, **Resolution** (for wide spectrum 80, for peak 20). Then specify regions to measure. For a wide spectra type *wide*, for a certain peak type name of the line (for example *Si 2p*). ***Attention**: To prevent damaging of the detector keep the output signal bellow 1 000 000 counts/sec all the time. For unknown samples, take the first wide spectrum with emission 5 mA and resolution 80. (use method "C:\ESCAPE\safe wide spectrum.acquisition"). If the max. intensity is bellow 200 000 counts/sec, emission can be increased to 15 mA.*

10. To start data acquisition click on the **Submit location for acquisition** blue arrow button on the desired location 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).

11. 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).
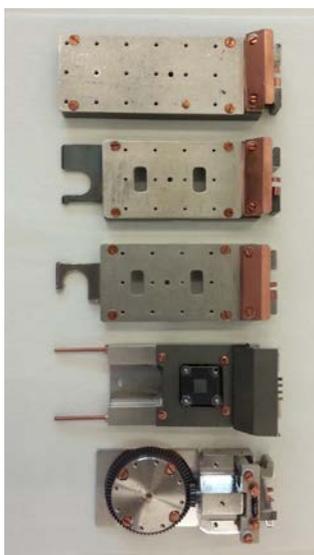
Plain dual height

Plain + stub end entry

Plain + stub side entry

Heat and Cool

Azimuthal