

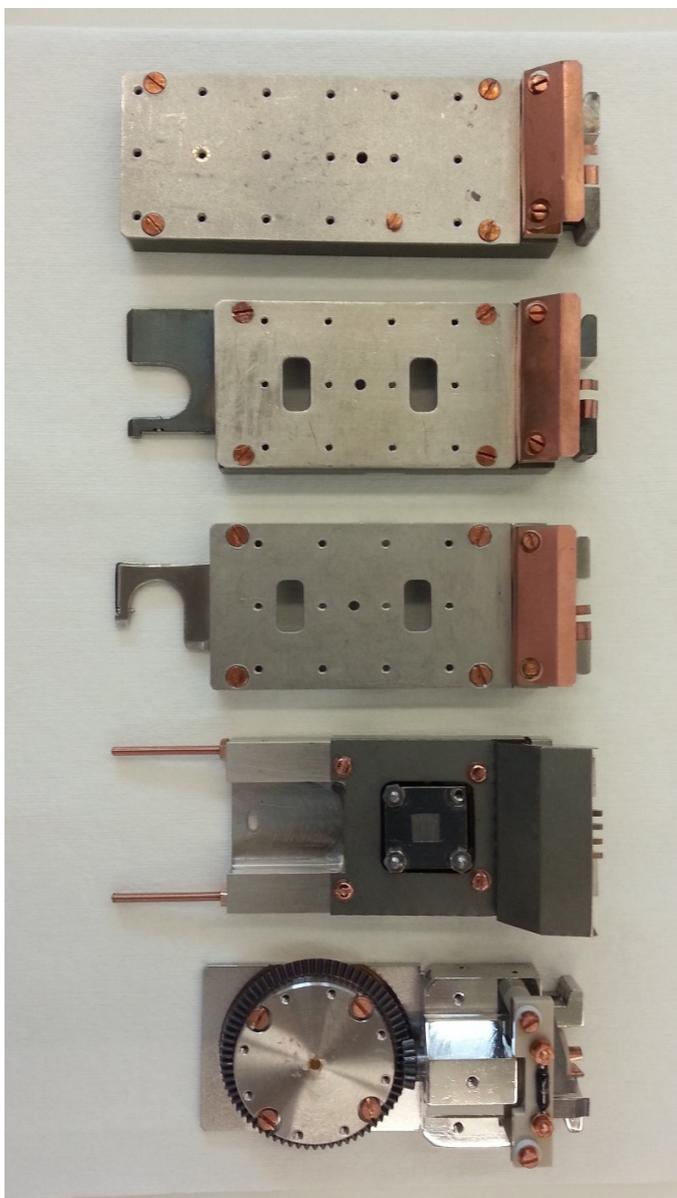
Obtaining XPS spectra (v 1.3)



1. Vent the Flexi-lock chamber by clicking on the **Vent** button in the **Sample Loader** window and loose 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 copper one (conductive) or kapton one (insulated) – both are in the flowbox 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). 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** window which appears. It is very important to specify exactly, which slot is free and which is occupied (photo of the bars are shown overleaf). After pressing **OK**, push the doors on the hinge side, until you hear opening of Flexi-lock backing valve. Check in the **Status window** whether the Flexi-lock pressure starts to go down. 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 pump).
4. When message **Pumping Flexi-lock completed** is shown, 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. If the bar is in the magazine slot 3, the gate valve must be opened to transfer the bar to the camera position. This can only happen if the pressure difference between the Flexi-lock and the SAC (sample analysis chamber) is low enough (2 orders). To take 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. Create analysis locations on your bar. Open the **Analysis Location** window and select your **Sample Holder** to view the desired image. To create new analysis location, click on the **Select multiply items** button, then click on the desired location in the image, press the **Add location to list** button and fill the dialogue. In the location list unselect the check box if you do not need charge neutralisation (if needed, leave the check box selected). Default charge neutraliser parameters are fine for most of samples, however it can be changed by a user. Do not set the filament current over 0.50 A. Set an experiment in which all measured data from the location will be saved. A new experiment can be created in the **Data organizer** window.
6. If the pressure difference between the Flexi-lock and the SAC is about 2 orders and pressure in flexi is in the -7 order Torr, 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 AutoZ method. For the former, select your location in the list, click on the **Positioning** reveal arrow and the button **Set Position to move** in the **Analysis location**. Click on the **Microscope** button, set 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 to save the new position. The latter method for setting z position AutoZ can be loaded in **Acquisition method** window from "**C:|ESCAPE**" folder and submit it for the acquisition according to the point 9.
8. 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 output signal below 1 000 000 counts/sec all the time.** For unknown samples, take first wide spectrum with emission 5 mA and resolution 80. If the max. intensity is below 200 000 counts/sec, emission can be increased to 15 mA.

- 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 then be added to queue in the **Analysis** window. Select the **Auto** radio button and press **Start** (software will perform further experiment automatically).
- When finished, move the bar to the Flexi-lock by clicking on the **Transfer the sample holder from stage** button to a free magazine slot. Then vent the Flexi-lock, remove the bar and **pump the Flexi-lock.**



Plain dual height

Plain + stub end entry

Plain + stub side entry

Heat and Cool

Azimuthal

Please report mistakes and other hints josef.polcak@ceitec.vutbr.cz