ICSM / LDD : Small angle x-ray scattering (SAXS bench built by Xenocs)

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A suitable and non-conventional collimation (SAXS) set-up for specific studies on colloidal dispersion, complex fluids, thin films and absorbing mesoporous materials using a hard x-ray beam (17.5 keV). This is a volume and non-invasive scattering technique for mesoscopic structural characterization as well as elementary forces determination as a function of thermodynamical parameters.

In the case of isotropic structure, the scattering is independent of the azymuthal angle; 2D scattering can be transform in 1D scattering curve I(q), q the transfer momentum, being proportional to the scattering angle but normalized by the x-ray wavelength.

To do this and using fit-2D software to average "mar2300" format 2D-scattering file: i) determine the center of the 2D-spectrum using the silver behenate scattering spectrum for which you have concentric scattering rings.

ii) Determine the right sample to detector distance, still using the silver behenate scattering and knowing the q-value of the first order scattering peak.

iii) Apply a mask on the 2D data to remove from the data averaging the "bad" pixels from the detector (surrounding parts, beamstop shadow and detector artifacts).

Once, all scattering parameter are entered in FIT2D (center, D and mask) you can apply "integrate" with the option "geometrical correction" and save using "output" in "chi" format (2 columns text file with q (in nm⁻¹) and I (in counts)

Now , the normalisation step to get absolute values of scattering intensity in cm-1. K is obtained from high density PE sample knowing the absolute intensity at 0.035 $Å^{-1}$:4.9 cm⁻¹

 $\frac{1}{e} \left(\frac{I_{sample}}{t_{acq} Tr_s} - \frac{I_{cv}}{t_{acq} Tr_{cv}} \right)$

0 (Å⁻¹)

-+- water 2 mm (th3b)

Things to remember:

Q- covering range: 2.10⁻² – 2.5 Å⁻¹ Before to run an experiment: safety doors closed and main shutter opened (upper light shutter ON) He gas open and indicate it on the safety doors! Don(t forget to close the circuit at the end of the experiment!

Main SPEC command in "SAXS" unix window: "sc" or "so", secondary shutter closed and opened respectively.

"umv motor_name abs_value" or "umvr motor_name abs_value", to move a motor (if you type the command and "enter" then SPEC will display the various parameters that you have to add.

"ascan motor_name abs_start abs_finish interval ct_time" or "dscan", to move step by step a motor while counting with the photodiode! Check first that the photodiode is in place in the beamaxis by typing "camin".

"startacq acq_time filename" : acq_time in second (<4200) and file_name starting with the date and user initiales like: 100522_OD_blabla

You can write your serie of command in a macro in a nedit window that you can copy/paste in SAXS window.

All the samples, with their thicknesses, acq-time, and transmission values should be noted in the logbook!!

