# Small Angle X-ray Scattering
# GeniX Mo Low Divergence

Application Note n° AN-G10


## A b s t r a c t

*Molybdenum radiation is quite rarely used for SAXS experiment since the integrated flux level as well as the achievable resolution makes this radiation not advantageous. Nevertheless whenever samples contain high Z – high absorbing materials, or whenever a wide wave vector q_range is required on a single image a higher energy is required.*

*In this application note, experimental results on test samples are described with an innovative single shot SAXS/WAXS laboratory setup.*


*Fig. 1 : GeniX Mo Low Divergence beam delivery system installed at ICSM CEA Marcole, France.*

## One shot SAXS/WAXS of heavy atoms solution scattering

*Data courtesy of Dr Olivier Diat, ICSM - CEA Marcoule, France.*

## I n t r o d u c t i o n

The ICSM (Institute for Separation Chemistry of Marcoule) is in charge of fundamental research linked to new aspects and needs of renewal of processes for development of sustainable and environment-responsible nuclear energy. One of the main axis of research common to the different teams is the combination of nanostructure and thermodynamic aspects in the physical chemistry of complex fluids used for extraction, decontamination, precursor solution for nanomaterials or matrices for interim storage of nuclear waste. This implies the analysis of high-Z species containing samples (heavy metal and lanthanide/actinide ions, highly dense material...) and very complex and absorbing containers a shown on Fig 2.

The main X-ray based techniques used to characterize the nanostructure of colloids, polymers, and most suspensions are based on simultaneous small and wide scattering angle data collection covering atomic and supramolecular scales of studies which are fully suitable and complementary to electronic microscopy techniques. The practical geometry of SAXS/WAXS applications being transmission mode, high-Z elements-containing specimen is even further complicated due to the strong absorption of the sample. As a reason, Molybdenum radiation has to be used in order to optimize the scattering versus sample transmission.

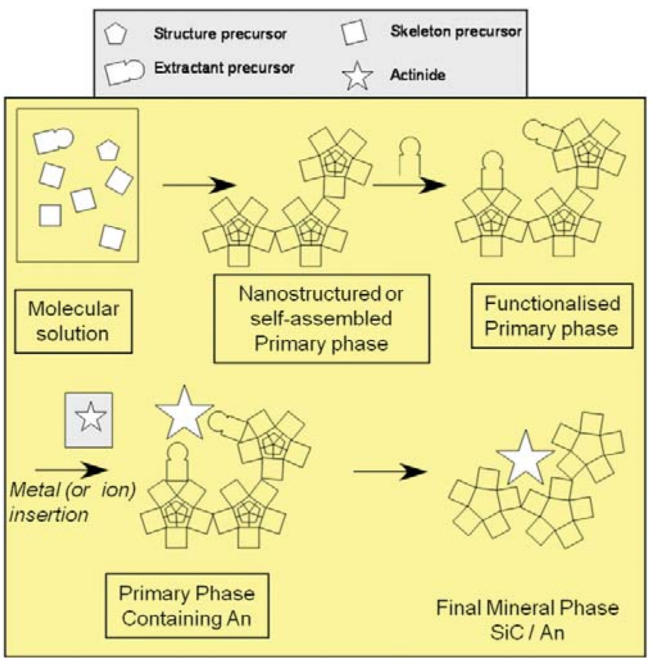


*Fig. 2 : Fuel design and multiscale controlled Actinide-containing materials (from D. Meyer, ICSM)*

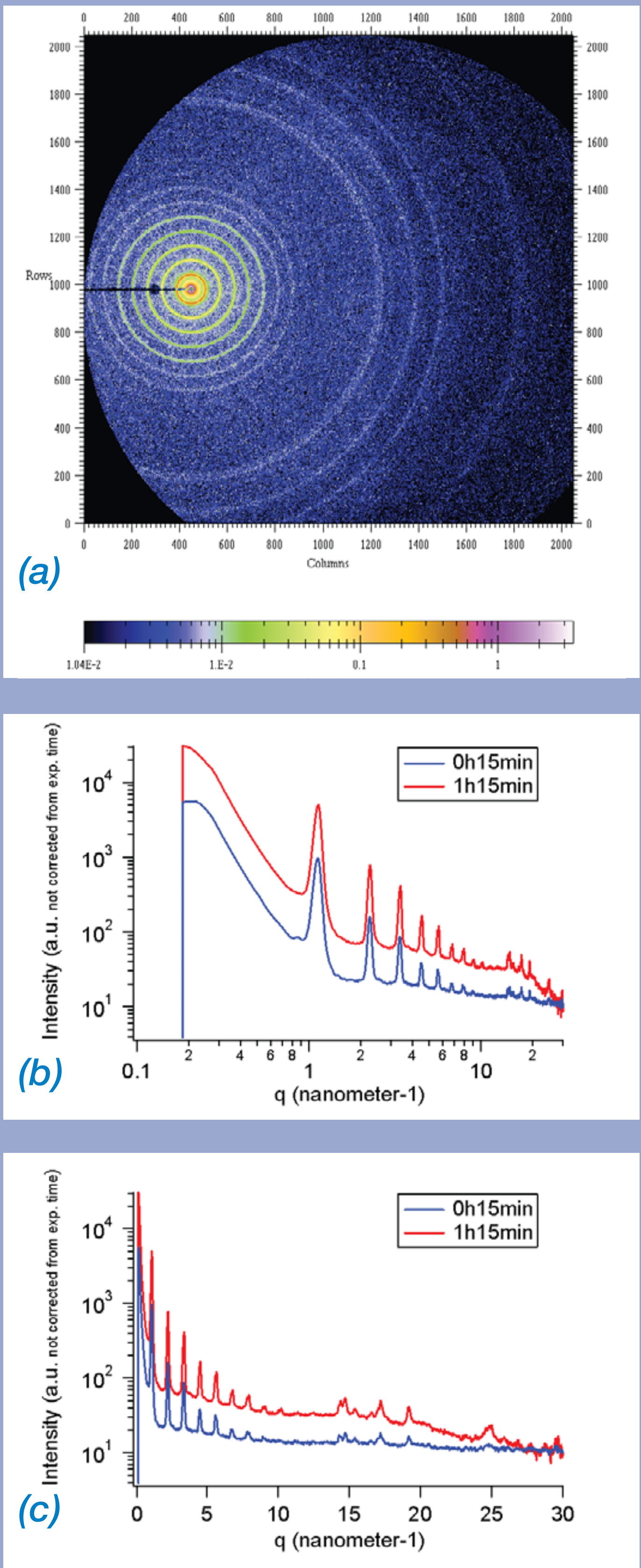


*Fig. 4 :*
*(a) SAXS/WAXS data of Silver Behenate Q_range: [0.02, 30] nm-1 and Dq: 0.017nm-1*
*(b) 2D data, and corresponding azimuthal averaging within log-log representation for emphasis on SAXS*
*(c) or log-lin representation for emphasis on WAXS.*

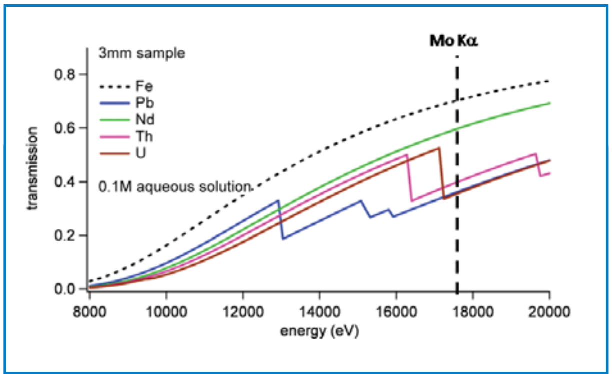


*Fig. 3 : Typical transmission values for a 3 mm thick sample composed of a suspension in water of high Z particles, with a concentration of 0.1 M.*

The high energy of 17.48keV radiation of Mo Kα helps penetrating the sample containing a high concentration of high Z elements, as such as Zr, Nb or other ionic species from the nuclear waste chain (Fig 3). Optimum thickness of the sample is obtained for a transmission of 1/e, with the transmission expressed as: $I/I0 = \exp(-\mu.t)$ and μ being the lineic absorption coefficient.

The advantage of using 3 mm capillary is to fully use the incoming beam, optimizing the used flux and further reducing the upstream parasitic scattering (sample shadow effect). Such a thickness is not an issue even for the WAXS regime since the detector is kept at 714mm. In this domain, the largest angle on the detector being about 15°, the resolution remains acceptable even with 3mm thickness sample.

The choice of Molybdenum radiation implies more stringent requirements for X-ray optics. Indeed, the molybdenum target has a lower emission than Copper. As a reason, the flux output from the source is low and the downstream components (mostly optic and collimation) quality is very critical and have to be optimized in order to achieve an acceptable system performance.

## Experiment

For this type of experiments the Institute for Separation Chemistry of Marcoule recently installed a GeniX Mo Low Divergence combined with a collimation system equipped with motorized anti-scattering vacuum slits and a large (345 mm) MARresearch Image plate detector placed at 714 mm from sample on a motorized X-Y stage (vertical + lateral directions).

### Optical settings:

The beam delivery system Genix is composed of a microfocused source (50W) and a FOX2D collimating single reflection optic. For SAXS requirements, where low divergence and high flux are required in Molydbenum radiation, the single reflection design of Xenocs FOX2D optics is a clear advantage. Indeed, comparison can be performed with Montel type or graphite doubly curved optics. In the first case, the double reflection multilayer optics (Montel or KB design) suffers from lower reflectivity and smaller capture angle. In the second case, in spite of the large capture angle of graphite material, its low reflectivity and large intrinsic mosaicity makes it useless for real SAXS experiment since this material is not able to reach a low level of divergence. The FOX2D is the only X-ray optic combining both the efficiency (captured solid angle x reflectivity) and the low divergence suitable for SAXS.

**Collimation settings:**

The strategy used in the collimation is highly innovative. In the scheme installed in Marcoule the collimation is achieved based upon two pairs of scatter free slits, defining the beam without sacrifice on the flux.

The high brightness beam delivered by Genix and the anti-scattering slits enable a total flux at sample position of 3 E6 ph/s/mm² and a q-range between 2.10-2 and 3 Å-1 with an off-center detection. The combination of Mo radiation with a large Image Plate detector allows simultaneous SAXS and WAXS within a reasonable q_resolution of Δq=0.017nm-1. The generous WAXS area offers reinforced statistical quality of isotropic samples and anisotropic sample studies.

In order to demonstrate the performance of this one shot SAXS/WAXS capability a data set was acquired with a test sample of Silver Behenate. Test results are shown in Fig. 4. The comparison of the two different exposure times of 15 and 75min shows that already after 15min the pattern is fully exploitable on the full SAXS and WAXS ranges. This single shot SAXS WAXS setup avoids to repeat the experiment twice or complex multiple detectors operation.

To demonstrate the ability to measure SAXS-WAXS on highly absorbing material, data from high-Z lamellar phase containing 0.1M Neodyme, 50% surfactant and related extracting agent in a 3mm capillary is shown on Fig. 5. This model systems is interesting to be studied since it mimicks multivalent ions extraction systems used in sustainable and environment-responsible nuclear energy.

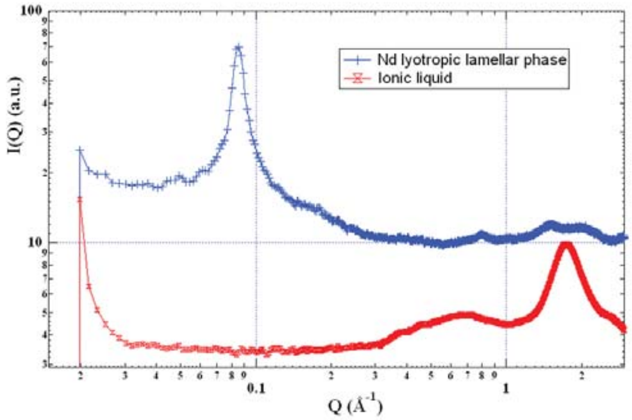


*Fig. 4 : Raw intensity of 0.1M Nd and 50% surfactant agent in an extracting agent (blue curve) . Extracting solvent is here in red.*

# Conclusion

Taking benefit of the high efficiency of our Single reflection optical design and of our scatterless slits collimator, Xenocs proposes a high performing set-up for SAXS/WAXS experiments with Molybdenum radiation. Advantage of this system is to enable the analysis of high absorbing sample without compromise on the flux level on the sample.

This type of set up can also be used to achieve full data set over a wide q wave_vector range [2.10-2 and 3 Å$^{-1}$] in a single image without any artifact usually encountered at the junction of the SAXS and WAXS domain (~1 Å$^{-1}$).

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