



	Experiment title: Differential anomalous X-ray scattering on complexes of uranium and extractant.	Experiment number: SC 454
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Shifts:	Local contact(s): Peter Boesecke	<i>Received at ESRF:</i>

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Report:

The time for this experiment was initially allowed in January from 27/01 to 31/01/1999, but was reported due to technical problems (high rate of rejection in the detector system even at 50kHz count rate) ; it finally took place in July (19/07-22/07/99) during single bunch mode.

The final aim of this project is to explain the kinetic delay, observed in the transfer of uranium by an extractant (DMDBTDMA) from aqueous to organic phase, by combination of anomalous and GISAXS techniques.

The anomalous SAXS results described here, constitute a preliminary step of this project. Previous SANS measurements show the structure of the organic phase as reverse micelles of complexes of extractant and uranyle nitrate in hexane ; structure of agregates (diameter of 20 Å) seems to be deeply linked to the ratio (uranium/extractant). The understanding of the uranium contribution in the scattering of the micelle requires anomalous SAXS measurements.

ASAXS data recording in q-range [0.04 - 0.8 Å⁻¹] has been carried out at two energies (17.100 and 17.237 keV) below the LIII absorption edge (17.244 keV), corresponding to (-13) and (-21) electrons.

Feasibility of ASAXS experiment on complexes of extractant and uranyle nitrate has been studied in two diluents : hexane and hexafluorobenzene ; notice that concentration of uranyle nitrate should be maintained below a limited value to assure the stability of the organic phase.

RESULTS

Anomalous effect is apparent in hexane, confirming previous calculations, as shown figure 1. However unwanted noise due to high fluorescence yield of uranium was observed due to imperfect monochromatisation and had to be subtracted of the scattering intensity ; the experiment should be improved by a choice of an additional energy far from the uranium absorption edge. Absolute scale has been obtained via calibration using lupolen as secondary standard.

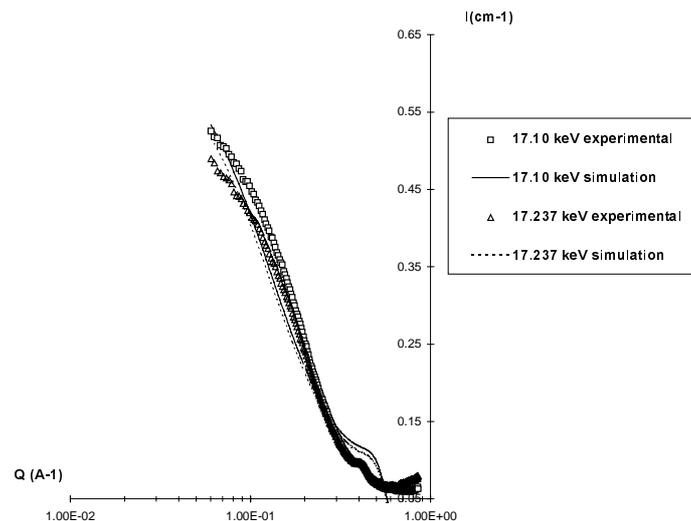


figure 1 : ASAXS spectrum / anomalous effect on micelles of DMBTDMA in hexane at uranium absorption LIII edge.

SAXS measurements in transmission mode show the influence of concentration of uranyle nitrate on the structural organization of extractant molecules (figure 3) in hexafluorobenzene : formation of complexe (DMBTDMA-($\text{UO}_2(\text{NO}_3)_2$) induces a slope of the scattering intensity in q^{-1} , characteristic of cylindrical structure of micelles.

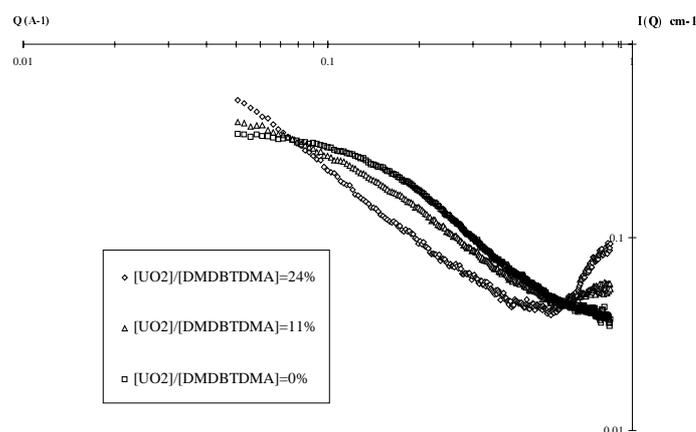


figure 3 : SAXS stectrum / scattering intensity of extractant DMBTDMA in hexafluorobenzene for variable ratios (extractant-uranyle nitrate).

On the contrary, anomalous effect could not be observed in hexafluorobenzene, (figure 2), due to the insufficient difference of contrast between the two energies : in consequence,

further experiment of GISAXS can not be conducted in hexafluorobenzene and would be replaced by a classical reflectivity one in hexane.

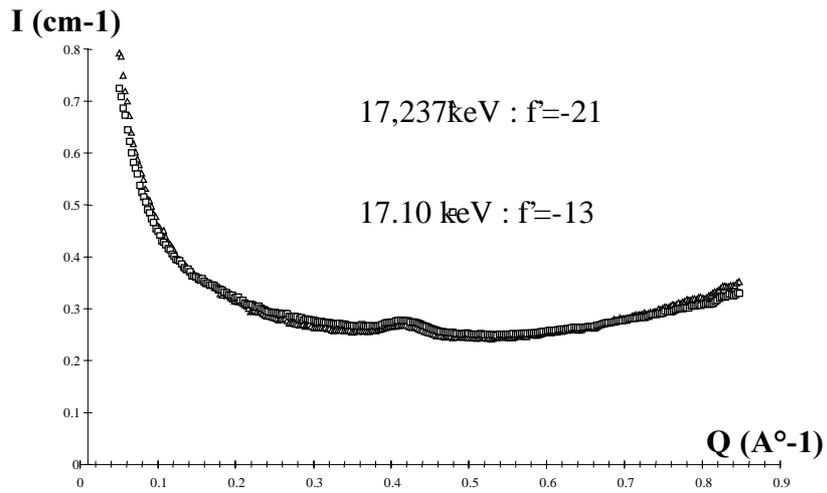


figure 2 : ASAXS spectrum / scattering intensity of extractant DMBTDMA in hexafluorobenzene at two energies below uranium edge.