Total-Reflection XAFS of Aqueous Solution Surface

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In order to determine the solvation structures of ions segregated at the solution surface by surfactant, we are developing a new XAFS technique and this report follows the previous one, 1997B0039-NX -np.

The X-rays are introduced onto sample solution surface at very small incidence angle(θ^{inc}) which is less than the critical angle for total-reflection. The smaller the θ^{inc}, the smaller the penetration depth of X-ray into solution, and the smaller dependence of penetration depth upon X-ray energies during the energy scan for collecting spectrum data. Therefore, it was expected that the quality of data was higher at smaller θ^{inc}, of course at the expense of fewer photon available and larger noise due to the ripple of liquid surface, so we have to carefully determine the θ^{inc} for best result.

The BL01B1 station has two mirrors, one in front of the monochromator and the other after that. Normally both of them incline by the same angle so that the outgoing beam is in parallel with the incoming beam. However, for the present experiments they incline by different angles, then the beam can be introduced onto the solution surface at any desired θ^{inc}.

In search for θ^{inc} at which best quality spectra can be produced, we have measured the spectra at different θ^{inc}s for the same sample surface. The sample was Br^- ions segregated by stearyltrimethylammonium ion. The spectrum for the same solution surface shown in the previous report (1997B0039) was noisy and had irregular structures. It could be much improved by tuning the $\Delta\theta$ of the double-crystal monochromator at each energy step.

Some of the Br K-edge spectra obtained are illustrated here with the values in parentheses (θ^{inc}/mrad, vertical slit width/μm). It is now obvious that the XAFS spectra critically depend on θ^{inc} and highest quality spectra were obtained at θ^{inc} around 1 mrad.