Total Reflection XAFS of Aqueous Solution Surface

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The structure of liquid surface is not well known in spite of its importance as a playground of surfactants. It also is used as the place to arrange self-assembly monolayer for producing Langmuir-Blodgett multilayers. Since most of surface sensitive analytical techniques require high vacuum condition, it is quite difficult to perform surface analysis on the air/liquid interface. It has long been desired to develop a method to determine surface concentration of elements.

The XAFS analysis should give us the information not only of the elemental surface concentration but also of the local structure around ions at the liquid surface, if the technique can be modified to acquire surface sensitivity.

The present method works under the total-reflection condition, thus the X rays do not penetrate inside the bulk solution. In order to monitor the absorbance of X-rays at the surface, total-conversion-electron yield or helium ion yield method is used.

The critical angle is around 2.5mrad for the total-reflection at 10keV photon energy at the air/water interface. Then the incidence angle to aqueous solution surface should be around 1mrad to give constant detection depth over the EXAFS region. There are serious problems to attain such a low incidence angle for XAFS study. It requires high precision and stability of the mirror system. The solution surface must be ripple free, thus it must be placed on a perfect vibration isolator. The vertical width of X-ray beam usable becomes very small, typically 50 to 100μm, then the photon flux becomes small.

The present experiment was undertaken to check the performance of the mirror system for BL01B1. It has two mirrors, one in front of the double crystal monochromator and the other after the monochromator. Each mirror can be driven independently, thus the direction of X-ray beam can be tilted by very small angle against the horizontal plane by adjusting the second mirror. The second mirror has another functionality, i.e. it can be bent to focus the beam vertically.

For the first trial, the surface of stearyltrimetylammonium bromide solution was studied. Since the cation is highly surface active, although its concentration was only 0.1mM, the signal from Br^- was clearly observed as shown in the figure. There are quite large noise structures. The cause for the peculiar shape of the spectrum must be studied and a higher beam current for SR is awaited to progress the present study further.