Development of a liquid-filled cell for HiSOR BL06

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The development of soft X-ray-transmissive thin-film windows has brought significant advancements to soft X-ray absorption spectroscopy (XAS) of liquids. Currently, several methods are employed: SPring-8 BL17SU and NanoTerasu BL07 utilize solution flow cells to measure fluorescence yield through a single window. In contrast, UVSOR BL03U uses a transmission-type cell with two windows sandwiching a thin liquid layer to measure light transmittance. While solution flow cells allow for easy sample exchange under atmospheric pressure, they require large sample volumes for pumping and suffer from spectral distortion due to the saturation effect. Conversely, while transmission-type cells provide true spectral shapes following the Beer-Lambert law, controlling the gas pressure to adjust the liquid film thickness remains a challenge.

To address these issues, we designed a liquid cell for HiSOR BL06 that enables easy installation with minimal sample volume while allowing for the correction of spectral distortion. First, we adopted a sealed-cell structure. By eliminating pump-driven flow, we removed concerns regarding liquid leakage and cleaning during sample exchange, thereby minimizing the required sample volume. To facilitate rapid sample replacement, the area surrounding the cell is housed in a low-vacuum chamber, allowing for easy vacuum purging and evacuation during measurements. Furthermore, to introduce soft X-rays propagating through high vacuum, the end of the high-vacuum chamber was designed with a nozzle shape, using a thin film at the tip to partition the high-vacuum and low-vacuum sections.

The system is designed for fluorescence yield (FY) measurements using photodiodes (PDs). By installing two PDs, we increased the signal intensity and enabled the correction of the saturation effect by varying their installation angles. We have previously demonstrated that by formulating the fluorescence intensity measured by a PD, the correct absorption spectrum can be retrieved from experimental spectra using concentration-dependent data [1]. This method allows us to determine parameters representing the degree of spectral distortion—governed by experimental conditions (such as the PD angle and solid angle) and the solvent—thereby correcting the observed spectra.

However, concentration-based correction has limitations: the molecular state may change with concentration, and preparing multiple samples is costly. Therefore, we have attempted a new approach: performing measurements at different fluorescence detection angles to enable spectral shape correction using a single sample in a single experimental setup.

Figure 1 shows O K-edge XA spectra of acetic acid in hexane solution normalized by heights of the first resonance peak (a) and the spectra after correction for saturation effects (b) [1]. The width of the first resonance peak around 532.2 eV is all the same after saturation correction, indicating that the correction is successful.

Figure 2 shows schematic diagram of the BL06 terminal equipment (left) and a photo of the low-vacuum chamber (right).

Figure 3 shows the cross-section of soft X-ray emission nozzle and photodiode mount installed in the low vacuum chamber. By setting the detection angles to 40° and 80°, two types of absorption spectra are acquired, and correction is performed using these two data.

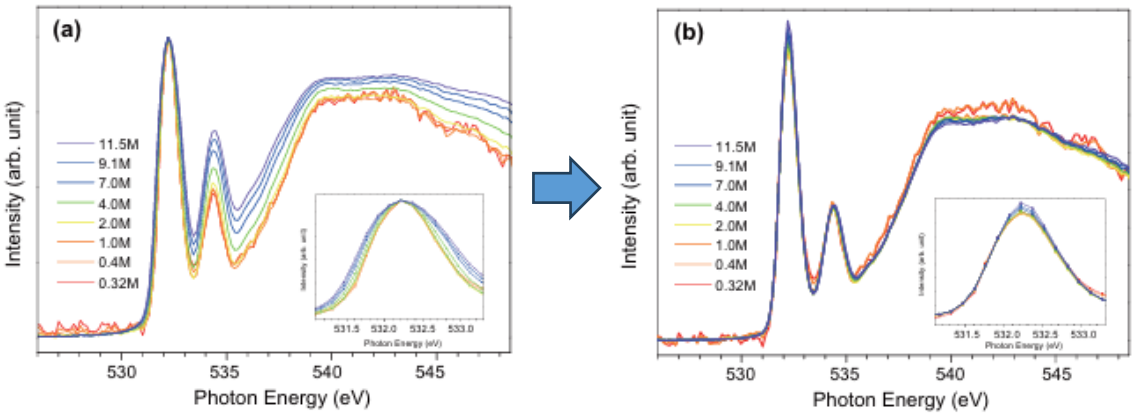

FIGURE 1. (a) O K-edge XA spectra of acetic acid in hexane solution normalized by heights of the first resonance peak. (b) The spectra after correction for saturation effects. [1]

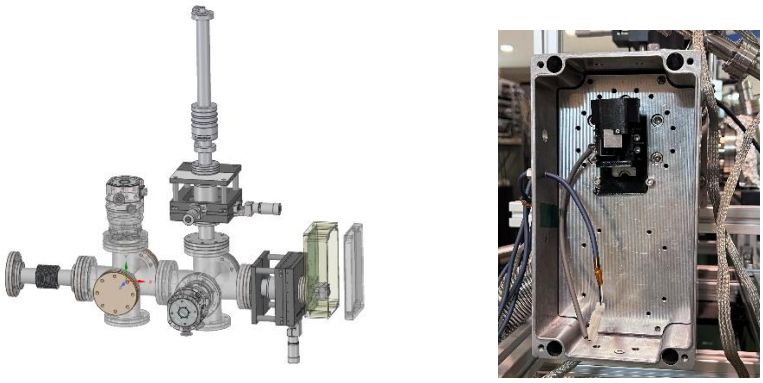

FIGURE 2. Schematic diagram of the BL06 terminal equipment (left) and a photo of the low-vacuum chamber (right)

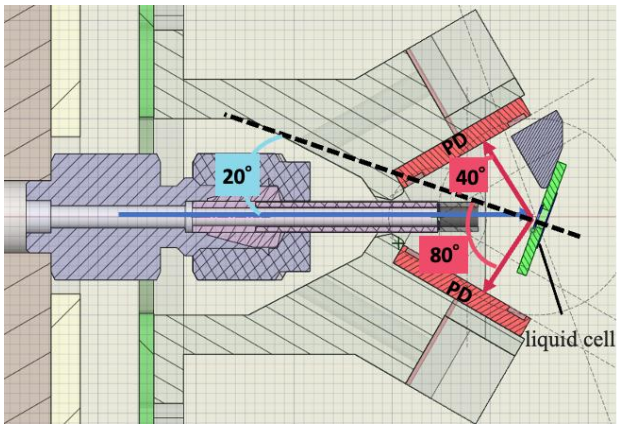

FIGURE 3. Cross-section of soft X-ray emission nozzle and photodiode mount

REFERENCES

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