

Ambient Pressure Gas-Flow Total Electron-Yield Cell for *Operando* XAFS Studies of Liquid Jets with Soft and Tender X-rays

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There is increasing interest in studying speciation of low-Z elements in solutions and other liquid phases by XAFS in the soft (< 1 keV) and tender (1-5 keV) X-ray ranges. Use of free liquid jets is a versatile way to perform such measurements. Continuous replenishing of the sample minimizes the effects of beam damage. Another advantage of jets over flow cells is windowless operation, avoiding complications arising from contact between liquid and window materials. Here we highlight another benefit of this approach, namely the ability to collect XAFS data by ambient pressure total electron-yield (TEY) detection, which is not an option when the liquid sample is confined under an X-ray window.

We have developed and commissioned a versatile liquid jet cell that permits recycling of the sample and incorporates a gas-flow TEY detector for XAS measurements, which is a metal collector biased at +60 V DC using a battery box. Key to the construction of the detector is to minimize condensation of evaporated liquid from the jet on the electrical feedthrough of the TEY detector. This was achieved using a continuous He stream preventing condensation in the feedthrough section. We demonstrate the performance of the cell by monitoring the carbonation of Ca(OH)₂ in solution *operando* at the Ca K-edge.

We demonstrate that gas-flow TEY detection is one of the most convenient methods for recording high quality XAFS spectra of materials with a high concentration of the X-ray absorbing element of interest.¹ It is particularly useful for photon energies below 5 keV, where transmission detection is often not possible. Gas-flow TEY can be used with electrically insulating samples, as emitted energetic electrons (particularly Auger electrons) create positive and negative charges in the gas phase by pair formation, which neutralize charge buildup on the sample.² TEY detection is also much less prone to self-absorption distortions of the spectra than fluorescence-yield measurements and for samples with high concentrations of X-ray absorbers the signal-to-noise quality can be comparable to that of transmission measurements.

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