SHORT TERM SCIENTIFIC MISSION (STSM)
SCIENTIFIC REPORT

This report is submitted for approval by the STSM applicant to the STSM coordinator

Action number: CA18222 - 48202
STSM title: Enabling photoelectron spectroscopy experiments of gas-phase biomolecular ions in a new crossed-beam apparatus at the PLEAIDES beamline of the SOLEIL synchrotron.
STSM start and end date: 23/06/2021 to 04.07.2021
Grantee name: Lucas SCHWOB

PURPOSE OF THE STSM:

The scientists of the host institute at the PLEIADES beamline are currently developing a new custom-made apparatus to perform photoelectron-photoion coincidence experiments of electrosprayed biomolecular ions. Briefly, the new PLEAIDES instrument consists of an atmospheric pressure ESI source, a double radiofrequency ion-funnel and a rectilinear quadrupole ion guide. The ions delivered by the source are further transferred and refocused into the interaction region inside the EPICEA vacuum chamber by a custom-made system of lenses and deflectors hosted inside a differentially pumped chamber, designed and built on PLEAIDES. In the interaction region, the ion beam crossed orthogonally the photon beam from the synchrotron beamline. The first beamtimes using the new instrument are planned for the end of this year but this newly built instrument requires testing and characterization. The purpose of the STSM was for me to bring my expertise in this type of experimental setup in order to help characterizing the actual design of the PLEAIDES' instrument and further identify what can and have to be improved before the beamtime. The STSM was coinciding with an in-house commissioning beamtime to further test the instrument with photons.

DESCRIPTION OF WORK CARRIED OUT DURING THE STSM

Many tests and improvements on the PLEAIDES instrument have been carried out during the STSM, including:

- Improving the ion production, transport and focalization in the interaction chamber by optimizing the DC and RF voltages on the different RF ion guides and electrostatic lenses. From the initial 150 pA achieved before the STSM, I could increase the total ion flux up to 700 pA. We used tannic acid solution at 50 uM in 50:50 water:methanol solvent with 0,1%vol formic acid for protonation.
- Optimizing the extraction of the ion in the ion time-of-flight side of the EPICEA spectrometer. The time-of-flight spectrometer was not conceived for this application and the electrodes which are part of it are not shielded for ions outsides of the spectrometer. Thus, the ions, when approaching the interaction region at the center of the spectrometer may feel the electric field created by a DC voltage applied on the TOF electrodes and be deflected. Avoiding this problem requires to either keep electrodes grounded all the time or to pulse the DC voltage from the ground to a desired value. With the help of SIMION simulation, we could establish set of suitable parameters to extract the ion beam to in TOF, considering all constraints: the number of available HV pulsers and HV power supply able to deliver high current, drift tube set to ground because of noise created on the MCP signal.
- We tested a variety of pulsing scheme in order to avoid a high number of precursor ions to reach the detector of the TOF, risking damaging the microchannel plates (MCP). This included pulsing of some electrodes in the TOF as well as pulsing the MCP. The first option was not satisfying are

precursors ions and potential fragments may not have separated enough flight times to pulse out precursors ions without affecting the fragments. The later showed very long (>200us) rise and fall time of the MCP voltage, meaning that the MCP voltage would not be turned down quickly enough to prevent the detection of the ions. This was due to the fact the resistive bridge connecting the all detector electronics is placed inside the chamber and connected to a single DC voltage input. In principle, only the front plate of the MCP should be pulsed, with other voltage always applied. This could not be modified during the STSM.

- Setting up and optimizing the pulsing of the ion beam by gating the ion at the exit of the quadrupole ion guide with the high voltage (HV) pulser. By doing so, the ions accumulate in the ion guide and intense pulse of ion (3 x the intensity of the continuous beam) is ejected to interaction region. By counting the number of ions reaching the MCP, we could determine the flight time of the ions from the ion guide to the center of the interaction region and therefore accurately trigger the extraction pulse of the TOF voltages, and define the highest repetition rate achievable with enough accumulation of ions.

DESCRIPTION OF THE MAIN RESULTS OBTAINED

The many tests performed during the STSM and, in parts described above, are results in themselves. Additionnal, we performed long runs during the night shifts to measure photoelectrons in coincidence with photofragments from the tannic acid precursors ions, used as test molecule during this STSM. These are very challenging experiments with a calculated very low signal-to-noise ratio (~10^{-4}). During the STSM, the runs were unsuccessful.
The main results may be translated in the fact that we established 1. a detailed protocol for running and troubleshoot the experiment and 2. a list of modification, which could not be done during the STSM, to make to the instrument to improve it. The main modifications discussed consist of:

- Building a grounded tube lens around the ion path from the last Einzel lens to the spectrometer in order to shield the electric field of the TOF electrodes and avoid deflection of the ions prior to entering the interaction region.
- Build a “single point” faraday cup mounted on a 3-axis translator which could be accurately placed at the center of the interaction region, in overlap with photon beam, in order to focus the ion beam at this exact point and ensure the best overlap between the ion and the photon beams.
- Decouple the electronics of the ion detector in order to be able to pulse the DC voltage of the front plate of the MCP only.

FUTURE COLLABORATIONS (if applicable)

The STSM was a very fruitful collaboration in regard of all the work which have been achieved in few days. It also revealed the complementarity and excellent ability to work together of the host and the grantee of this STSM. This work will be followed by a collaborative beamtime in December on this instrument. All the knowledge I acquired on running the instrument and the EPICEA spectrometer will be an invaluable asset for the beamtime. After this project, it is much likely that this collaboration will continue further in the future and show the first ever electron spectra of photoionized biomolecular ions, using synchrotron light and electrospray ionization source.