ESRF	**Experiment title:** Solvation cage structures of aqueous iodide	**Experiment number**: ch-6318
Beamline: ID09	**Date of experiment**: from: 01/03/2022 to: 07/03/2022	**Date of report**: 09/05/2022
Shifts: 18	**Local contact(s)**: Matteo Levantino	*Received at ESRF:*

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Report:

The time-resolved X-ray solution scattering (TR-XSS) experiment took place at ID09. Sodium iodide solvated in water has been studied at four different concentrations (20 mM, 30 mM, 100 mM and 300 mM). Additional test measurements have been performed on 100 mM and 300 mM sodium bromide solutions. The sample was excited with the picosecond laser setup provided by ID09 in two different setups. Firstly, the TOPAS system delivered 250 nm wavelength laser light with 20 uJ on a 180x130 um spot. Secondly, the second harmonic of the Ti:Sapphire laser provided 400 nm laser light at up to 150 uJ. The X-ray energy was set to 18 keV (pink beam) with a pulse length of 100 ps provided by a high speed chopper (⅞ + 1 (196+4 mA) filling mode). As detector, we used the Rayonix MX170-HS in WAXS geometry (53 mm sample-detector distance). The sample was deliverd by a 300 µm liquid flat jet and recycled for continuous use with a total amount of 100-200 ml.

During the experiment scattering has been observed up to Q=10 $Å^{-1}$. Difference scattering curves have been calculated in parallel to the measurements with the waxscollect macro, where the off images (without laser excitation) have been taken at -3ns. The studied range of time-delays between laser pump and X-ray probe was from 100 ps up to 1us. All iodide samples showed strong difference scattering signals and dynamic behaviour on the 100s of picoseconds time scale and on the nanosecond time scale for both 250 nm and 400 nm excitation. For the bromide sample only weak difference scattering signals could be observed with 250 nm excitation. A detailed analysis in ongoing.
Power titration on pure water samples showed direct 2-photon absortion of water at 250 nm and confirmed the absence of direct 3-photon of water at 400 nm for <75 uJ with a 160x140 um spot size.

The experimental time of 18 shifts enabled the study of 6 samples (4x NaI and 2x NaBr), where 3 parameters have been varied: the concentration, the laser setup (250 nm and 400 nm wavelentgh) and the laser power. Measured were a fine time delay series around time delays t=0 and longer delay times between 700 ps and

1 us. Each series contains 100-150 images per delay point with 3 seconds exposure time. For structural refinement, 300 images were taken at delay time t=200 ps and t=1 us.

The aim of the experiment has been achieved. We did not encounter any difficulties during the experiment. The originally proposed experiment has been altered to shift from 267 nm laser excitation (3rd harmonic of the Ti:Sapphire laser) to the usage of the TOPAS system providing 250 nm laser light. The change has been requested to maximize the absorption of iodide and bromide into the first CTTS (charge transfer to solvent) band. The decision was made based on results from absorption measurments available only after the proposal deadline.