Experiment Report

Proposal ID and Title: **ES-1757 "Selected D2O and H2O ices and phase transitions among them with Hirshfeld Atom Refinement"**

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The main goal of our experiment was to obtain data of low-temperature ice phases such as ice II, ice III and ice V, suitable for further investigation with use of Hirshfeld Atom Refinement (HAR). HAR is inevitable when we want to improve ice structures in such details as exact determination of hydrigen positions or their atomic displacement parameters. It is especially challenging in the case of disordered ice structures.

Because we wanted to controll not only the temperature but also the pressure of measured sample we used diamond anvill cell mounted in cryostat. Cryostat was filled in with helium. Thanks to this fact the lowest temperature which we have obtaind during our measurements was 40 K. Diamond anvill cell allowed us to change the pressure. In this way it was possible to travel across the phase diagram in two dimensions. We assumed that it would be possible to obtain particular ice phase either by changing the temperature or by changing the pressure. Due to the fact that within our scope of interest where first of all low-teperature phases of ice we worked within the pressure range up to 0.6 GPa to avoid ice VI.

During our beamtime we have loaded the diamond anvil cell twice. First time with deuterated D_2O and second time with H_2O. It is well known that there are subtle differencies between D_2O and H_2O phase diagrams, that is why we wanted to cover both types of data.

Diamond anvill cell filled with D_2O was investigated within temperature range from room temperature to 67 K, and within the pressure range from 0.15 GPa to 0.6 GPa. Theoretically these ranges should cover area of existence of three ice phases: ice II, ice III and ice V. However, we have managed to crystallize form of ice V only, which was stable even in zones of phase diagram devoted for ice II or ice III.

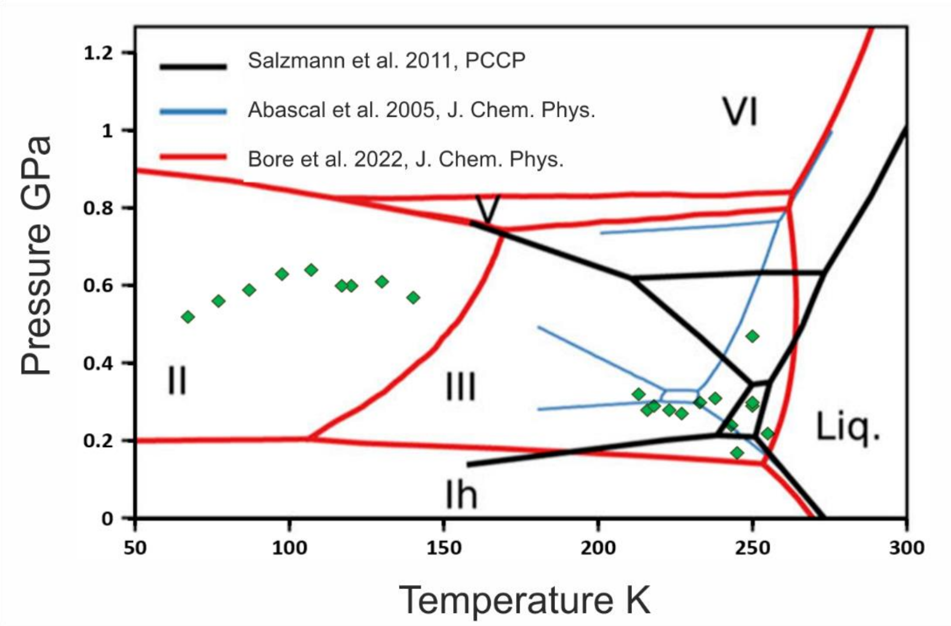

Figure 1. Comparison of data collected in this measurement (green diamonds) with areas on ice phase diagram which belongs to different phases (on the basis of different scientific papers).

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On figure 1 we present where on the phase diagram of ice were measured our data. As we see, the particular values of temperature and pressure suggest that we should obtain mainly ice II and ice III. Instead of this we were measuring ice V all the time.

Also the second loading with H_2O gave us results for ice V only. This time temperature range was between RT and 40 K and pressure range between 0.25 GPa and 0.35 GPa.

The next step of our research will be HAR calculations to determine exact positions of hydrogen atoms and their ADPs. We have already done sumilar calculations for ice VI and ice VII as a result of our previous measurements. However, structure of ice V is relatively more demanding than ice VI or ice VII from point of view of HAR. There are 28 molecules in the unit cell, most of the atoms are placed at general positions and hydrogen atoms are mostly disordered. That is why this structure is more challenging and HAR calculations will be much more time consuming.

References:

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