	Experiment title: Densities and structures of salty-ices VI and VII from single crystal X-ray diffraction	**Experiment number:** ES-474
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Report :

Scientific background

The potential habitability of extraterrestrial oceans in icy moons and exoplanets requires inputs of nutrients and chemical energy from exchanges with the silicate interior. Because of the presence of high-pressure (HP) water ices (VI, VII, etc.) between the liquid ocean on the surface and the silicate inner core, such interactions are generally considered to be limited in large icy moons, like Ganymede and Titan, and water rich exoplanets. However, solid- state convection could allow chemical transport through HP ice. Indeed, several remote sensing observations of icy moons in the solar system support the common presence of salts in their deep interiors such as NaCl and $MgSO_4$ suggesting chemical exchange with the silicate core. Recent experimental work demonstrated the possibility to incorporate significant amounts of salt (e.g. NaCl, LiCl, RbI) and small volatile molecule (CH_3OH) in the structure of HP ices (*e.g.* up to 2.4(8) mol% NaCl in Ice VII). The crystal structure of these ice solid solutions remains similar to pure H_2O HP ice phases, as observed by previous work using X- ray and neutron powder diffraction. Nevertheless no measurements have been made so far on $MgSO_4$ incorporation in HP ices, and incorporation/substitution sites of salt species and effects of dopant on the equation of states have not been constrained experimentally for any ice HP polymorphs.

To better constrain the chemical evolution, structure and the potential habitability of water-rich planetary bodies, it is important to know exactly how common solutes will affect HP ices properties such as chemistry, volume and density.

During beamtime ES-474 on ID-15b we measured single crystal X-Ray diffraction (SC-XRD) patterns for ice VI and ice VII crystals formed from solutions of pure H_2O, and aqueous solutions of NaCl, $MgSO_4$ and RbI up to 5 GPa.

Experimental procedure

Pure H_2O, and aqueous solutions of NaCl, $MgSO_4$ and RbI were loaded in HP diamond anvil cells (DAC) along with ruby spheres (pressure gauge). K-type and S-type thermocouple were used to monitor the temperature. The pressure and temperature were then raised and to allow selection of less than 3 crystals of ice VI or ice VII in equilibrium with the solution to obtain hydrostatic conditions. Then measurements were completed as follow:

1. Ruby fluorescence measurement for pressure and thermocouple reading for temperature
2. Microphotograph of the pressure chamber

3. Wide angle X-Ray pattern acquisition up to ±35° depending on DAC aperture to obtain a first preview of spots pattern quality and refine lattice parameters from powder diffraction analysis technique
4. Small steps X-Ray diffraction patterns with a 0.5° step to fully explore reciprocal space for single crystal structure refinement.
5. Ruby fluorescence measurement for pressure and thermocouple reading for temperature to check stability.

Preliminary results

We measured SC-XRD patterns for all solutions (H_2O; H_2O-NaCl; H_2O-$MgSO_4$; H_2O-RbI) for ice VI at several temperature steps and liquid fractions for each. Measurements in ice VII were much more challenging because of the tendency of the crystal to recrystallize rapidly and therefore making acquisition small step scans difficult (>10min). Further analysis are required to see if we were able to obtain good quality enough data. Nevertheless, primary results for ice VI show a clear volume increase tendency when ice crystalize from a salty solution, even for NaCl and $MgSO_4$, which has never been measured before (figure 1). This confirms previous measurements made at the ESRF for RbI[1]. We are also able to observe differences of volume depending on the position in the crystal, suggesting that an increasing salt incorporation results in an increase lattice volume.

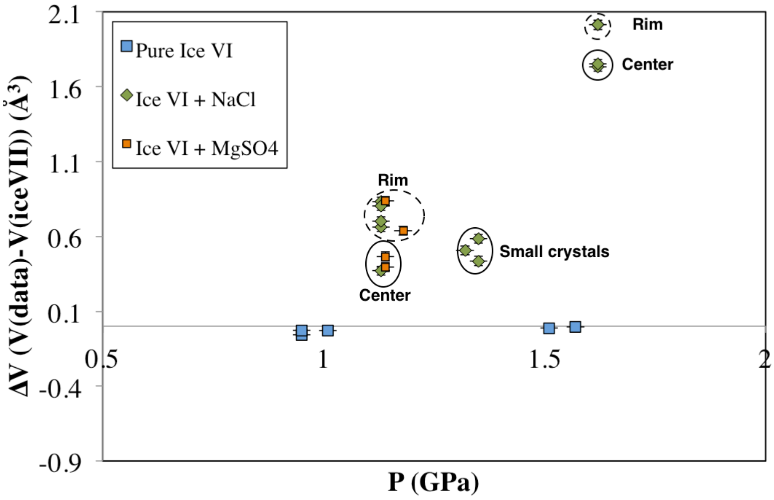

Figure 1 : Examples of volume differences of ice VI grown from different solutions for center and rims of the crystals compared with pure H_2O thermal equation of states at the same temperature[2].