（ ※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。 ）

CROSS TOKAI **Experimental Report** J-PARC	承認日 Date of Approval 2015/4/23 承認者 Approver 服部 高典 提出日 Date of Report 2015/4/23
課題番号 Project No. 2014A0048 実験課題名 Title of experiment Search for LiBr·6D_2O ice VII 実験責任者名 Name of principal investigator Stefan KLOTZ 所属 Affiliation IMPMC, University of Paris, France	装置責任者 Name of Instrument scientist Hattori/Sano 装置名 Name of Instrument/（BL No.） PLANET 実施日 Date of Experiment 16.–21.6. 2014

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

LiBr·6D_2O and LiCl·6D_2O aqueous solutions

2. 実験方法及び結果 （実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

The aim of the experiment was to investigate the recrystallisation of glassy LiBr aqueous solution with a concentration close to the eutectic point, i.e. R=5.5–6 in LiBrRD2O, under pressure of 4–6 GPa when heated to room temperature, similar to our previous observations in LiCl–D2O system [1]. Solutions with R=5.6 (LiBr5.6D_2O) were loaded into the MITO high–P/low–T cell using WC anvils and polished TiZr SMS gaskets. A few small pieces of lead were added as pressure gauge. After applying an initial load of 2 tn to seal the gasket, the cell was cooled as fast as possible (5–8 K/min) to 100 K and the sample was checked by neutron diffraction at this temperature. From 3 samples, one turned out to be crystalline, the two others were amorphous (glassy). One of the two glassy (good) samples (run 5) was then compressed at 110 K to 4 GPa and warmed. During the warmup to 300 K the pressure was adjusted to reach approximately 5.2 GPa at 300 K where the sample started to transform, see Fig. 1. The transformation at this temperature carried on for approximately 1 hour. The sample was then collected to 293 K and data were, collected at this temperature. The other glassy sample (run 3) was compressed at 110 K to lower pressures, 3.1 GPA, and heated with

2. 実験方法及び結果（つづき） Experimental method and results (continued)

adjusted to reach 4.5 GPa at 274 K. At 283 K and 4.5 GPa, crystallization occurred, again with a slight pressure drop, over a timescale of typically an hour. Diffraction patterns were taken at this temperature and pressure over 3 hours where a slow transformation of the sample was observed. After 12 hours of no beam the sample was checked again at 293 K and found to be fully transformed to a crystalline solid with diffraction patterns very similar to what has been found of the LiCl–system at approx. 3 GPa (unpublished). We conclude that the stability limit in LiBr5.6D2O ice VII is shifted to higher pressures compared to the LiCl–system, to approximately 4.5 GPa.

The data of the first samples were analyzed by Rietveld methods, based on an ice VII structure with incorporated Li and Br, given the evidence for the LiCl–system. A marked difference is the absence of the (111) reflection at approx. 2.0 A which testifies a much strong disorder in the H–positions. The refinements can indeed reproduce this, as well as a unit cell volume which is 18% (!) larger compared to pure ice VII at this pressure.

Overall, these experiments were a great success and details will be submitted for publication this year!

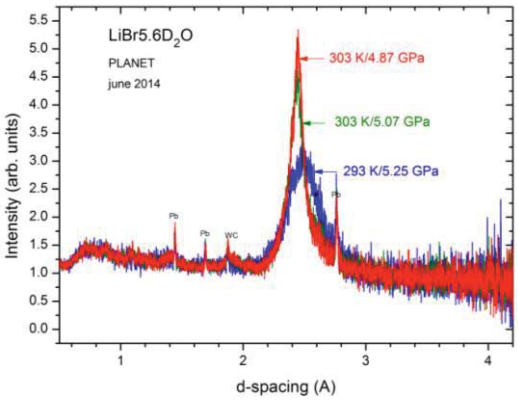

Figure 1: Diffraction patterns during recrystallisation of the glass (blue) into a strongly disordered “salty” ice VII (red).

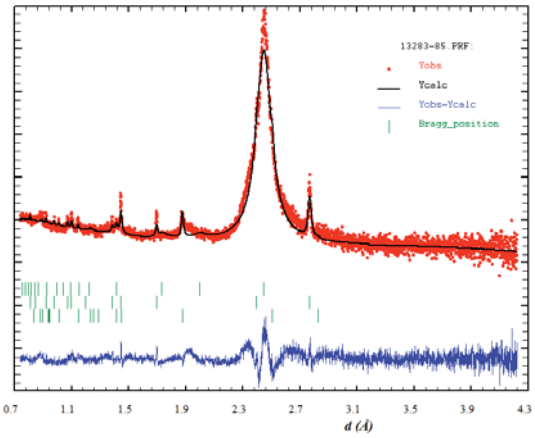

Figure 2: Rietveld refinement of diffraction pattern to “salty” ice VII (including Pb and WC).

[1] S. Klotz, L. Bove, Th. Strässle, A.M. Saitta, Th. Hansen, Nature Materials 8, 405 (2009)