ESRF	**Experiment title:** *Investigation of the proton disorder in ice at low temperature in the megabar range.*	**Experiment number:** *HS501*
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Names and affiliations of applicants (* indicates experimentalists):

PRUZAN Philippe
WOLANIN Elodie
CHERVIN Jean-Claude
CANNY Bernard

Physique des Milieux Condensés
Universite P. et M. Curie
T13-E4-B77
4 place Jussieu
75252 Paris Cedex 5

Report:

In order to reach the so-called symmetric solid ice X, from ice VII, a sequence of transformations involving the proton site disorder has been proposed. In the various regions l-2-3, in Fig. 1, the proton disorder arises respectively from reorientation of the molecules, static disorder of the molecular dipole moments, and proton tunnelling. These modifications with pressure of the proton disorder alter the equation of state. This was shown for ice VII at 300 K. These investigations were performed at the ESRF : in the regions 1, 2 and 3 the volume pressure dependences are different. Thus, new lines were added to the phase diagram of ice (Fig. 1). Our present experiment concerns ice VIII. No crystallographic data exist for this phase above 25 GPa, except from our own previous preliminary measurements performed at the ESRF along isothermal paths at 220, 180 and 100 K. The main goals of the present work were the determinations of the transition line from regime 2 to regime 3 for isotherms >100 K, and the investigation of the EOS of ice at low temperature, around 50 K, in the region of ice VIII and in the regime 3.

A membrane diamond anvil cell, placed inside a cryostat, was used with rhenium gaskets. We used angular dispersive x-rays with an imaging plate system. We investigated the isothermal paths 200, 50 and 20 K (see fig. 1). The results obtained are presented in Figure 2. The isothermal paths 200, 50 and 20K were investigated in the pressure range shown in Fig. 1. At 200 K the VII-VIII transition was observed around 40 GPa. Along the lower temperature isotherms (50 and 20 K) the obtained diffraction patterns were of poor quality and did not allow to locate the transition pressures. Actually along this run, and for the three isotherms, we obtained very broad peaks, even in the region of ice VIII where the splitting of the various reflections (due to the tetragonal structure) was not resolved. Strong pressure gradients, observed along these runs, account for that feature : this was checked by the various pressure markers placed close to the center and at the edge of the sample. Typically we measured at e.g. 90 GPa and 50 K, a pressure gradient around 20 GPa. The use of a rhenium gasket may be the cause of this large pressure gradients. Rhenium is very rigid, this favours a zero strain condition along the gasket wall and consequently a large uniaxial stress component. Actually, above 40 GPa, our present data are scattered and not consistent with the previous ones, as shown in Fig. 2 where the cell volume was computed from the refinement of the various diffraction peaks. Due to these bad results it was not possible to perform further treatments of the data to locate the transitions between regions 2 and 3.

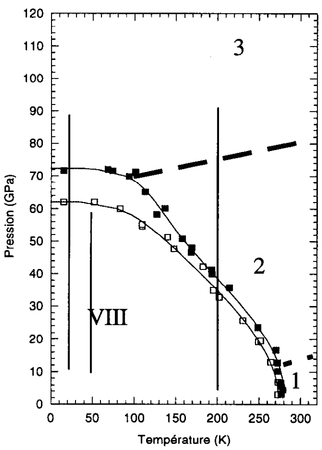

fig.1 : phase diagram of ice above 2 GPa. The vertical lines represent the investigated isothermal paths.

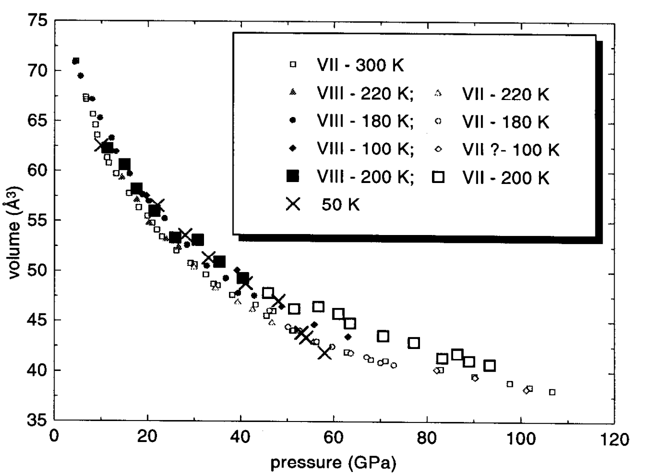

fig.2 : Equation of state of ice at various temperatures. The present results are shown at 200 and 50 K (large symbols).