



## Research Networking Programmes

Short Visit Grant  or Exchange Visit Grant

(please tick the relevant box)

### Scientific Report

The scientific report (WORD or PDF file – maximum of eight A4 pages) should be submitted online within one month of the event. It will be published on the ESF website.

**Proposal Title:** Influence of Ice Microstructure on Proton-Ordering

**Application Reference N°:** 7379

#### 1) Purpose of the visit

The influence of different preparation protocols and, hence, different ice morphologies on the proton-ordering patterns in high-pressure ices were investigated using neutron diffraction. The measurements were granted 96 hours of beamtime at the ISIS facility on the GEM instrument (experiment number RB 1600002). Rietveld refinement and PDF analysis of the data will provide an ultimate answer about the influence. It was the aim of the visit to quantify the degree of proton-ordering in these high-pressure ice phases prepared in Innsbruck by cold-loading them into the cryostat at the GEM instrument at ISIS and using powder neutron diffraction and Rietveld refinement.

#### 2) Description of the work carried out during the visit

In total 17 samples of differently prepared variants of the high-pressure ice phase were prepared in Innsbruck in their deuterated forms. These samples were shipped in a liquid nitrogen Dewar to the ISIS facility in Chilton/Didcot. On site Violeta Fuentes-Landete and Thomas Loerting were cold-loading the samples to vanadium cans and collecting neutron diffraction data, both in forward and backward scattering geometry. In the 96 hours of beamtime 17 samples were measured at 80 K to analyze the proton-order in the network of oxygen atoms. Two of the most interesting samples were in addition cycled between 10 K and 250 K in order to see the influence of temperature on the proton-ordering pattern. Furthermore, the phase-transition of the high-pressure form to the low-pressure forms cubic and (common) hexagonal ice were observed in these

measurements. In addition to the D2O-ice phases studied we made short tests about the isotope effect on the proton-ordering by also studying two H2O samples.

**3) Description of the main results obtained**

The main result is that the different preparation protocols indeed result in different patterns of proton-ordering, different lattice-spacings and presumably also in different space groups. Further PDF data analysis and careful Rietveld refinement is necessary to back-up this claim.

**4) Future collaboration with host institution (if applicable)**

The measurements will be continued in September 2015 on the SANDALS instrument, which will be employed to continue to study the isotope effect and H2O samples. As mentioned in 3) data processing will be done in collaboration with the host institution, in particular Dr. Sam Callear and Dr. Daniel T. Bowron

**5) Projected publications / articles resulting or to result from the grant (*ESF must be acknowledged in publications resulting from the grantee's work in relation with the grant*)**

Two publications will at least be published about the proton ordering in two high-pressure ice phase.

**6) Other comments (if any)**