

# ISIS Experimental Report

## Rutherford Appleton Laboratory

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Title of Experiment: The effect of pressure on the structure of meridianiite ( $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$ )

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Instrument: PEARL

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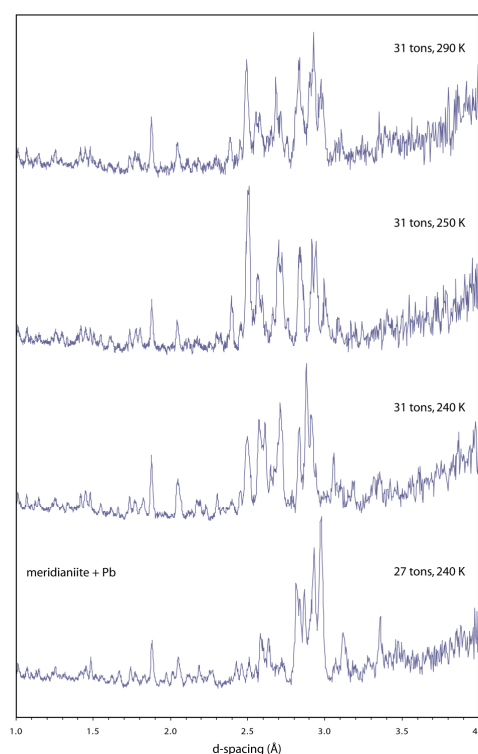
Date of Experiment: 11-14/09/2008

**Introduction:** The title substance is a relatively poorly characterised material. However, it has been the subject of greater scrutiny in the past few years since it has been suggested that it may be an important mineral on Mars [1], and also that it may be a major rock-forming mineral in the outer solar system [2]. It was recently identified in terrestrial frozen brine ponds [3], and named meridianiite (for Meridiani Planum on Mars). Peterson and Wang [4] solved the structure from single-crystal X-ray diffraction data. We have carried out a range of experimental and computational studies of meridianiite, including a neutron powder diffraction study over the temperature range 4.2 – 250 K on HRPD [5] and a single-crystal study of the protonated phase on SXD (see RB820008). However, we have no information concerning the phase behaviour or elasticity of meridianiite under the conditions appropriate to the interiors of icy moons (0 – 5 GPa, 100 – 300 K). The aim of this work was to carry out an initial exploration in the relevant P-T regime using the Paris-Edinburgh cell, making observations that will form the foundations for future detailed study, and for comparison with the phase behaviour of other hydrates in the  $\text{MgSO}_4 - \text{H}_2\text{O}$  system (see RB520039).

**Sample preparation:** Meridianiite was formed by rapid quenching with  $\text{L-N}_2$  of a stoichiometric (supersaturated) solution of  $\text{MgSO}_4$  in  $\text{D}_2\text{O}$ . Slugs of fine polycrystalline material formed in this way were broken up and ground to a powder in the UCL Earth Sciences cold rooms. Since the ISIS cold-rooms could not be reduced in temperature below  $+6^\circ\text{C}$  (meridianiite decomposes at  $+2^\circ\text{C}$ ), we spent 9 hours attempting to load the specimen at room temperature, using dry-ice to cool the anvils of the PE press (with an argon-filled glove bag to avoid icing). Despite considerable effort, this method failed (the sample decomposed), and we instead brought a pair of anvils back to UCL and loaded the specimen (at  $-15^\circ\text{C}$ ) in the Earth Sciences cold room. Due to uncertainty regarding the behaviour of the specimen in MeOD/EtOD, dry powder was loaded into the gaskets, and this resulted in a relatively poor transmission of pressure under modest loads; the sample did not begin to take up the load until it was increased to  $> 20$  tons.

**Results:** Despite the very low symmetry (space group  $P\bar{1}$ ), diffraction patterns obtained at 177 K under a load of 7 tons exhibiting strong Bragg reflections close to the instrumental resolution, suitable for unit-cell refinement. Increasing the load at 200 K resulted in considerable line broadening, and so the specimen was warmed to 240 K, whereupon the Bragg peaks became sharp once more. We determined that with load increments of 3 tons we could follow the evolution of the unit-cell parameters without difficulty. The bulk modulus found in this initial study by fitting an equation of state to the refined unit-cell parameters in the range 0 – 7 kbar is in excellent agreement with that found in our gas-cell study on HRPD (RB810006) and with *ab initio* calculations. Under a load of 31

tons ( $\sim 10$  kbar from the Pb pressure marker), the diffraction pattern of meridianiite changed substantially from that observed at 27 tons (Fig. 1). We could not ascertain positively the identity of the new phase or phase mixture; several peaks could be attributed to ice VI, suggesting that meridianiite has undergone a dehydration reaction to ice plus a lower hydrate (possibly, but not necessarily, epsomite). Warming from 240 K to 250 K resulted in a further change in the diffraction pattern. No further changes were seen until the temperature was increased to 290 K, where a further apparent phase change was detected. As there is no obvious agreement between the observed diffraction data and that of high-pressure epsomite powder patterns observed by us previously on PEARL/HiPr it is possible that these patterns represent new polymorphs of meridianiite, or some phase with a hydration number between that of meridianiite (11) and epsomite (7).



**Fig. 1.** Changes in the diffraction pattern of meridianiite indicative of a phase change on loading from 27 to 31 tons at 240 K, and subsequent warming up to room temperature under a load of 31 tons.

#### References:

- [1] Chou & Seal (2007): *J. Geophys. Res. Planets.* **112**, article E11004.
- [2] Kargel (1991): *Icarus* **94**(2), 369-390.
- [3] Peterson *et al.*, (2007): *American Min.* **92**(10), 1756-1759.
- [4] Peterson & Wang (2006): *Geology* **34**(11), 957-960.
- [5] Fortes *et al.*, (2008): *Phys. Chem. Min.* **35**(4), 207-221.