ISIS Experimental Report

Rutherford Appleton Laboratory

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Title of Experiment:	The high-pressure phase behaviour of epsomite	**Local Contact:**	M. G. Tucker
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Introduction

Epsomite ($MgSO_4.7H_2O$) is an important evaporite mineral on the Earth, and probably on Mars too, as well as being a candidate rock-forming mineral inside the icy moons of the outer solar system (see Fortes 2005). In order to be able to model the interiors of these icy moons, we are carrying out a program of investigation of epsomite and its related hydrates at high pressure (RB14491, 15133, Fortes *et al*., 2006). Previously, we have studied deuterated epsomite to 5.5 kbar in a gas pressure cell, and observed no phase changes. Our aim in this experiment was to investigate the possible high-pressure polymorphism reported by earlier workers (Bridgman 1948a,b: Livshits *et al*., 1963).

Experimental method

Large single crystals (ca. 5mm) of deuterated epsomite, grown for our previous work (RB14491) were extracted from their mother liquor, dried and powdered under air. The powder was loaded into TiZr gaskets of the encapsulated type (Marshall and Francis, 2002), with 4:1 MeOD/EtOD as the pressure transmitting fluid, and mounted between the anvils of a Paris-Edinburgh pressure cell. In two separate loadings (one at 200 K, and one at room temperature) the load on the cell was increased in 3 tons increments from 7 tons to 49 tons, with data being collected for two hours at each point. At 49 tons, 200 K, the sample was warmed to 280 K in 20 K steps. Pressure on the sample was determined using a Pb foil calibrant and the equation of state parameterised in Fortes (2004).

Results

Along the 200 K isotherm, we observed no phase transformations up to the highest load (pressure), 49 tons (5.25 GPa). The two hour data collection time was adequate for determination of the unit cell of epsomite using GSAS. The refined unit cell volume as a function of pressure was fitted with a 3rd order Birch-Murnaghan equation of state, yielding zero-pressure volume, V_0 = 965(2) Å^3, zero-pressure bulk modulus, K_0 = 25(1) GPa, and the first pressure derivative of the bulk modulus, K' = 2.0(3). This is in excellent agreement with the values determined from the gas cell work (Fortes *et al*., 2006). Moreover, the observed cross-over of the *a*- and *b*-axes, not seen in lower-pressure experimental work, had been predicted by *ab initio* calculations.

At room temperature, we observed a series of polymorphic phase transitions upon compression, broadly in agreement with existing piston-cylinder observations (Fig. 1). Epsomite appears to undergo a very sluggish transition (or series of transitions) below 1.5 GPa; the diffraction patterns from the first and second hours of data collection at 10 and 13 tons are different. However, at 16 tons (1.47 GPa) the diffraction pattern has ceased to change, and remains stable up to 28 tons (2.81 GPa). This phase is probably the phase IV reported by Livshits *et al.* (1963) and the phase seen from 1.5 – 2.5 GPa by Bridgman (1948b). It is apparent from the 10 ton and 13 ton data that at least one, and possibly two, intermediate phases exist, which would correspond to phases II and III of Livshits *et al*., (1963).

The diffraction patterns from 28 – 46 tons (no data were collected at 49 tons due to beam problems) exhibit a marked amorphous hump with some residual Bragg peaks. We interpret this as dehydration of the crystal with a lower hydrate (hexahydrate?) sitting in a viscous liquid or glassy brine.

Further details of this work, and the planetary implications are reported in the proceedings of the 37th Lunar and Planetary Science Conference (Mar. 13-17 2006).

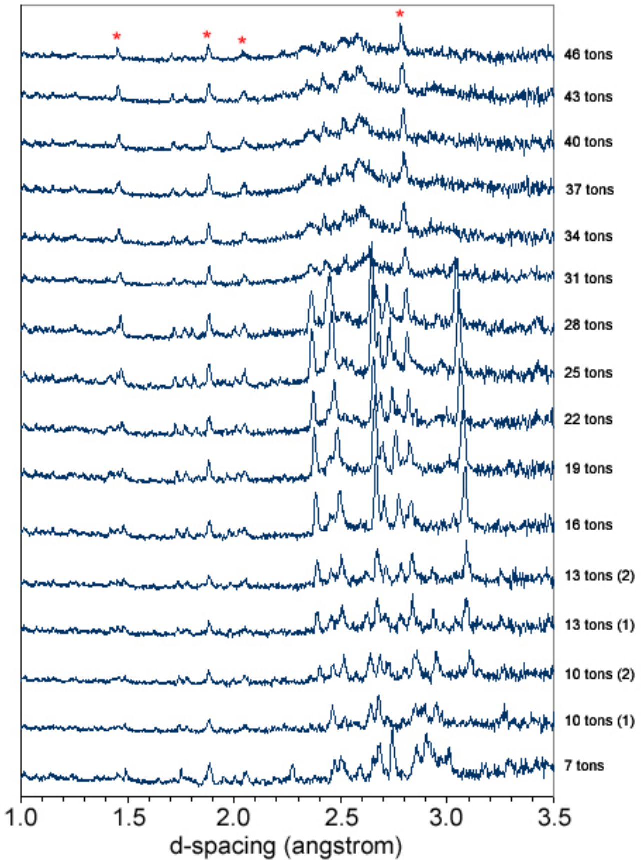

Figure 1: Evolution of the diffraction pattern as a function of applied load at room temperature. Asterisks mark scattering from the Pb pressure calibrant and from WC and Ni in the anvils.

References

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