

<b>Proposal:</b>	5-11-404	<b>Council:</b>	10/2012	
<b>Title:</b>	Determination of the hydrogen positions in the sulphate mineral Bloedite			
<b>This proposal is resubmission of:</b>	5-11-398			
<b>Research Area:</b>	Materials			
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<b>Samples:</b>	NaMg(SO <sub>4</sub> ) <sub>2</sub> . 4H <sub>2</sub> O			
<b>Instrument</b>	<b>Req. Days</b>	<b>All. Days</b>	<b>From</b>	<b>To</b>
D19	6	3	03/06/2013	06/06/2013
<b>Abstract:</b>				
<p>High-pressure synchrotron X-ray experiments on the mineral Bloedite have shown that dehydration does not occur under pressure, and that rather surprisingly, there is no phase transition up to 14GPa. This mineral may be widely distributed in the crust of icy satellites, such as the satellites of Jupiter. We wish to determine accurately the H-atom positions, and whether they are ordered or disordered, in this intriguing mineral, at room temperature and at lower temperatures, and for this we ask for 6 days on D19.</p>				

Our neutron diffraction study was carried out at D19 in ILL. The aim was to locate the positions of the H atoms in the structure and the possible evolution of the O-H bond with temperature.

The sample was a fragment measuring 5.5x3x2mm, cut from a large single crystal. It was mounted on a vanadium pin with “Kwikfill” two-component glue and measured at room temperature.

Subsequently a vanadium can was mounted around the sample and the temperature was decreased under vacuum to 20K with a rate of 3K/min. After data collection the temperature was raised to 120K with a rate of 3K/min and new data were collected. Data collection was stopped when more than 90% coverage was reached. During acquisition the  $-1\ 6\ 1$  reflection was used to control the status of the crystal.

Typical measuring times were 5s/ frame for RT and 3s/frame for LT. The crystal faces were photographed and indexed in order to apply absorption correction. For the low temperature measurements, data occurring from path lengths through the vanadium can were discarded.

Data were integrated with the software Retreat 2012 and structure was refined with Shelx, starting from the X-Ray model from Hawthort et al. 1985.

The neutron data are summarized in Table 1.

Temperature	RT	20 K	120K
Wavelength, Å	1.17 Å	1.17Å	1.17 Å
Resolution, thmax	57.86	57.73	57.73
No. reflections measured	2382	3301	3304
No. unique reflections	1174	1402	1426
Completeness	76.7%	91.4%	92.7%
R <sub>sigma</sub>	2.64%	2.50%	2.64%

Table 1. Summary of the neutron data.