ESRF	**Experiment title:** Single crystal studies on Chondrodite ($Mg_5Si_2O_8(OH)_2$) and Clinohumite ($Mg_9Si_8O_{16}(OH)_2$) at high pressure	**Experiment number**: HS 1241
Beamline: BM01A	**Date of experiment**: from: 22.11. / 06.12. 2000 to: 25.11. / 12.12. 2000	**Date of report**: 18.06.2003
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Report:

A significant portion of our planet's water may be held within the crystal structure of minerals in the mantle. Hence, the behaviour of hydrous minerals at pressure has important implications with respect to the mechanical properties of the Earth's crust and mantle. Humite minerals (e.g., chondrodite and clinohumite) are potential storage sites for water in the deep Earth. In order to elucidate the pressure-dependent behaviour of the hydrogen bonds in chondrodite and clinohumite, we aimed at the investigation of the structural evolution of the heavy atoms as a function of pressure in a first step.

Single crystals of deuterated hydroxylchondrodite ($Mg_5Si_2O_8(OD)_2$) and hydroxyl-clinohumite ($Mg_9Si_8O_{16}(OH)_2$), respectively, were loaded in an ETH diamond anvil cell together with a quartz crystal and a ruby ball for pressure determination. A 4:1 methanol:ethanol mixture served as pressure-transmitting medium. Intensity data were collected at room pressure, 3.3 and 7.4 GPa for hydroxylchondrodite and at 3.9 GPa for hydroxylclinohumite on the KUMA six-circle diffractometer using a point detector and synchrotron X-ray radiation at a wavelength of 0.7 Å (at room temperature). The diamond-

anvil cell was centred in the *x* and *z* directions by the use of an X-ray eye. Unfortunately, the *y* direction remained quite inaccurate. Intensity measurements were carried out with ω-scans (scan speed 0.05 °/s) at the position of least attenuation of the pressure cell, according to the fixed-φ technique [1]. All symmetry-allowed accessible reflections within a full sphere were collected up to θ = 40°. Three standard reflections served as intensity control. Intensity data were obtained from the scan data with the program *xd_red* [2] using a Lehmann-Larsen algorithm [3]. Intensities were corrected for Lorentz and polarisation effects and absorption of the X-ray beam by the diamond and beryllium components of the pressure cell using a modified version of *ABSORB* [4]. Averaged structure factors were obtained by averaging symmetry-equivalent reflections in the appropriate Laue symmetry following the criteria recommended by Blessing (1987) [5]. Structure refinements were carried out with *RFINE99* [6, modified by Angel 1999].

Due to instrument set-up problems, the centring of the sample crystal within the diamond-anvil cell on the diffractometer was not good enough. The reflections were not centred homogeneously, but started to leave the scan window at various diffractometer angles until they even were absent. Therefore, the data could not be refined and evaluated satisfactory.

Literature:

[1] Finger, L.W. and King, H.E. (1978) American Mineralogist 63, 337 – 342.

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[4] Burnham, C.W. (1966) American Mineralogist 51, 159 – 167.

[5] Blessing, R.H. (1987) Crystallography Reviews 1, 3 – 58.

[6] Finger, L.W. and Prince, E. (1975) United States National Bureau of Standards, NBS Technical note, 854.