ESRF	**Experiment title:** Phase Transition in an alpha-D-glucose-Sodium Chloride-Water Complex (6:3:3)	**Experiment number**: 01-02-668
Beamline: BM01-A	**Date of experiment**: from: 03.02.05 16:00 to: 05.02.05 08:00	**Date of report**: 25.05.05
Shifts: 5	**Local contact(s)**: Phil Pattison	*Received at ESRF:*

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Report:

Of the nine shifts allotted to this project, four were spent on the preceding experiment 01-02-698 in order to complete that study. The remaining five shifts were devoted in part to observing the effects of a conditioned environment on crystal stability under ambient pressure, and to studies of an expected phase transition under pressure in the range up to 7 kbar (*cf.* Experiment Report 01-02-653).

After some initial testing, a single crystal cut from a larger specimen was mounted and centred in our thermostat sample cell which was conditioned at t = 22^{o}C and relative humidity (RH) = 45%, and a data collection was started. The profiles of a set of 5 reflections were checked by ω-scans using a point detector at the beginning and at certain intervals during this work. A sudden increase in the magnitudes of both data and monitor counts by nearly a factor 2 could be traced back to an instability of the monochromator setting, and a restart was necessary after readjustment of the monochromator position. 12 hrs after the first exposure of the crystal to radiation most test reflections showed some broadening, but to varying extent, from 0 to 65% in FWHM. Compared to previous tests with the crystal kept in an unprotected and drier environment the present conditions represent an improvement. However, the optimal RH range for avoiding both dehydration and deliquescence of the crystals may be at even higher values than the 45% used in the present experiment. This will be examined in further tests.

A new crystal was mounted in the thermostat sample cell and about 2750 intensities were collected with a CCD detector and used for calculation of an orientation matrix (OM) and a set of cell parameters. The crystal was identified as a phase GE specimen. It was then transferred to a DAC,

immersed in Si oil, and recentred. Intensities were measured for calculation of an OM and cell parameters. The pressure was increased in steps to 2, 3.8 and 5.5 kbar, and OM and cell parameters were determined for each step from smaller sets of reflections. Unfortunately, the size and the quality of these data sets did not allow a definite conclusion as to possible deviation from hexagonal symmetry. Following the measurements at 5.5 kbar the pressure was reduced to ambient, the crystal was removed from the DAC, cleaned of oil and mounted again in the thermostat sample cell. Calculation of an OM and cell parameters from about 22400 reflections showed clearly that hexagonal symmetri was retained. A structure refinement suggested that a phase transition had occurred, but this finding needs to be corroborated.

The difference in structure between the two phases comes out predominantly in the weak reflections. Therefore, the weak reflections are very important for the identification of phase, and high quality data is required. This quality is difficult to obtain with the crystal placed in the DAC, and at present it seems a difficult task to observe a phase transition *in situ*.