P / kbar

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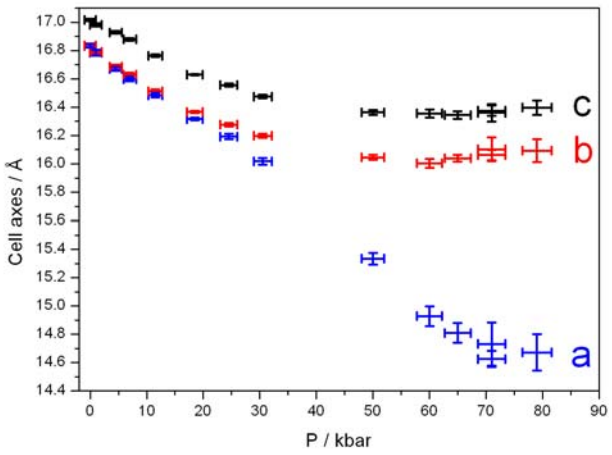
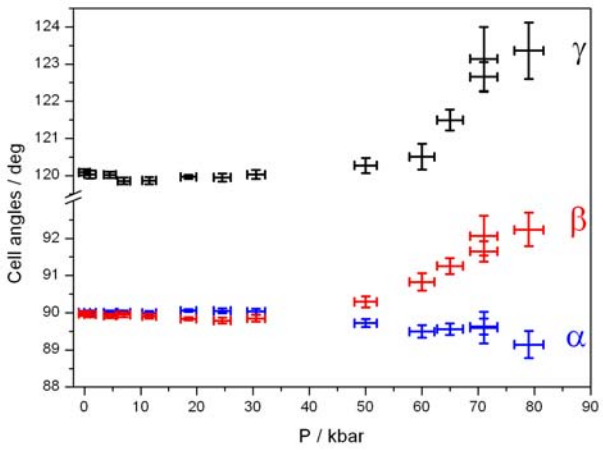
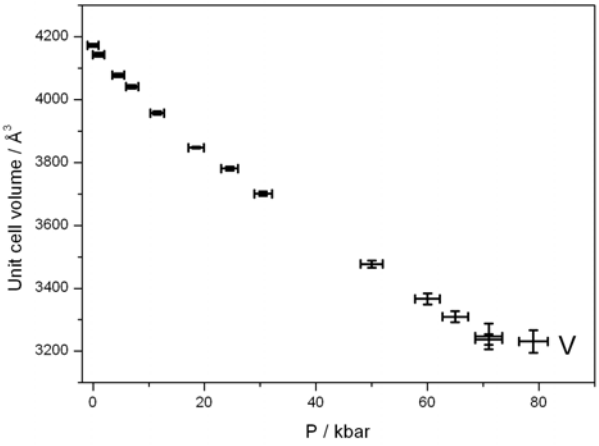
Report:

Crystals for high pressure studies are limited in size by the small opening in the gasket of the diamond-anvil cell (DAC). On the other hand, a fairly large crystal was needed in the present case for identification of phase since the difference in structure between the two phases of the glucose complex resides almost exclusively in the weak reflections. The first part of the work consisted mainly in establishing a crystal size in compliance with both requirements, and observing the behaviour of these crystals under high pressure. We found that crystals with cross sections ~ 35 x 50 μm and ~ 50 x 85 μm were too small. ω-scans for determining the FWHM-values for two reflections from the latter crystal showed a certain broadening after a period of 8 hrs, indicating the onset of radiation damage. The wavelength in all experiments was 0.71 Å.

A crystal of size ~ 125 x 90 x 65 μm was mounted on a glass pin, centred and identified as a phase CA specimen from measurement of a set of phase-sensitive reflections. It was then transferred to the DAC, deposited with Si oil as the pressure medium, and the pressure was increased in 13 steps from ambient to 90 kbar, which is well into the quasi-hydrostatic regime. At each step intensity data were collected with a CCD detector and an orientation matrix (OM) and cell parameters were calculated. For all pressures, except at the final p = 90 kbar, the crystal diffracted well enough to provide adequate data for calculating an OM and a set of cell parameters. After the maximum pressure had been reached, it was reduced stepwise to 60, 42 and 16 kbar. However, diffraction remained very poor, the diffuse intensities could not be indexed, indicating irreversible changes of the crystal at p = 90 kbar. The development of unit-cell axes, angles and volume are shown in a set of plots on p. 2. Error bars in unit-cell parameters (vertical) and in pressure correspond to 3 σ from the least-squares refinement and 1 kbar + 2% of the nominal value, respectively.







The plots show that a significant departure from hexagonal lattice symmetry takes place from about 18.5 kbar. A closer look at the refined cell parameters (not reproduced here) reveals that this departure appears to start already at about 7 kbar. The decrease of the unit-cell volume V with increasing p is roughly linear, as expected, however, with one slope in the range up to about 18.5 kbar, and a less steep slope in the range 18.5 to 70 kbar. Loss of crystal water(s) may be involved in the change of rate in linear decrease.

Following the high-pressure experiment, another crystal was mounted for data collection. A total of about 133 000 reflections were collected with a CCD. Analyses of the data show that the quality is degrading with time, the effect becomes noticeable already after about 5 hrs exposure. During this time 19 658 reflections were collected yielding 8 575 unique reflections after merging. The observed deterioration confirms previous observations on radiation damage of this hydrated complex, and suggests that the crystals must be kept in an environment with control of temperature and relative humidity during data collection. We have developed a sample cell for this purpose, and have used it successfully in diffraction studies of crystals of Rochelle salt.