PROTEIN AND VIRUS CRYSTALLOGRAPHY AT HIGH HYDROSTATIC PRESSURE BEYOND 2 Kbar

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On beamline ID30 at the ESRF, the combination of a diamond anvil cell, ultra-short wavelength X-rays from undulators (0.3305 Å) and a large imaging plate has allowed the extension of the field of high-pressure macromolecular crystallography both for the accessible pressure range, increased by one order of magnitude with respect to previous studies, and data quality. Results obtained on two tetragonal hen egg-white lysozyme crystals at 7.0 Kbar demonstrate that high pressure data can meet usual standards (resolution 1.6 Å, R_{merge} 0.057, multiplicity 7.2, completeness 0.93). These data were used for structure refinement.

The cowpea mosaic virus (CPMV) particle is the first example of crystallized macromolecular assembly studied at high pressure. Single oscillation images of a cubic crystal of CPMV at 1 bar, 1.1, 2.0 and 3.3 kbar gave evidence that pressure induces a phase transition in the initial P23 disordered and poorly diffracting crystal. At 3.3 Kbar, a highly ordered I23 crystal was obtained which diffracts at 2.6 Å resolution with high signal-to-noise ratio.

The possibility of obtaining accurate structural information under high pressure opens a wealth of possibilities such as exploration of sub-states, study of interactions between macromolecules and between subunits, and detection of the onset of pressure-induced denaturation. Furthermore, high pressure might become a standard tool to improve order in macromolecular crystals, either by favoring a more ordered packing or by restricting amplitudes of atomic motions in regions which are disordered at atmospheric pressure.

Keywords: HIGH PRESSURE PROTEINS VIRUSES

METAL ORDERING OF 2ZnS-$CuInS_2$ (ZCIS) SOLID SOLUTION SERIES STUDIED BY NEUTRON DIFFRACTION

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Physical and chemical properties of materials are often controlled by substitutions and non-stoichiometries. Thus solid solution series especially including phase transitions are strongly recommended for reflecting dependencies of chemical composition and element ordering as well as stability parameters as temperature and pressure. We are dealing with structure-property-correlations of a photovoltaic material, the semiconducting solid solution system 2ZnS-$CuInS_2$ (ZCIS), which shows a phase transition from the cubic sphalerite type to the tetragonal chalcopyrite type structure. The electronic band gap decreases exponentially from the ZnS-value to reach the gap value of the end member $CuInS_2$ even in the phase transition region. This behavior is suggested to correlate with different metal contents and ordering. Conventional and synchrotron radiation methods failed due to the electronic similarity of Zn and Cu, also EXAFS studies were not significant. Since neutron radiation helps to overcome these problems neutron powder diffraction experiments have to be used firstly done on those materials by us.

We used the high resolution powder diffractometer at BENSC (Hahn-Meitner-Institut Berlin, Germany). Rietveld refinements were done, in the tetragonal region with different metal ordering models. The refined lattice parameters and sulfur positions are independent from these models, but metal site occupancy and displacement factors vary. The R-values are used as a first significance check, enforced by simulation calculations. Near the $CuInS_2$-rich region the model with statistic distribution of Zn on Cu- and In-site is favorite. With decreasing tetragonality the distribution models lose significance and the anti-site occupation of Cu and In is increasing. In the cubic region the displacement factor of the statistically occupied metal site remains constant in the ZnS-rich region and is increasing towards the phase transition. The first with its strong decrease of the optical gap may be due to doping properties, whereas the other regions are due to substitution effects.

Keywords: STRUCTURE, SOLID SOLUTION, NEUTRON DIFFRACTION

NEUTRON AND X-RAY STRUCTURE ANALYSES OF POLY(PYRIDOBISIMIDAZOLE) PIPD

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Neutron structure analysis of poly(pyridobisimidazole) PIPD was carried out at 10, 100, 200, 295 K on the c-projection. It was found that PIPD assumes the statistical structure between two molecules of the pyridine groups with different orientations. By the constrained least-squares refinements, R-factors reduced to 15.6, 13.0, 16.5. and 10.5 % for 12 data at 10, 100, 200, 295 K, respectively. Three-dimensional structure was clarified by X-ray data (R = 16.5 % for 17 reflections). The OH bonds extend to outside of the molecule and form the intermolecular H-bonds. The molecule is virtually planar differing from PBO and PBZT, which may be attributed to the intramolecular H-bond NH···O(H).

Keywords: NEUTRON, X-RAY, POLY(PYRIDOBISIMIDAZOLE)

CRYSTAL STRUCTURE OF 3-MERCAPTO-4-N-(3', 4', 5'-TRIMETHOXY BEZYLIDENE) 5-PHENYL, 1, 2, 4-TRIAZOLE ($C_{18}H_{18}N_4O_3S$)

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Derivatives of triazoles are of clinical importance. They exhibits antibacterial, antifungal and anti- inflammatory activities. 3',4',5'-trimethoxy phenyl moiety is present in drug like trimethiprim. The title compound synthesised by the reaction of 4-amino-mercapto-5-phenyl triazole with corresponding aldehyde has been screened for its antimicrobial activity and it can exist in other tautomeric thione form also.

The molecule on the whole is non-planar. The phenyl ring makes a dihedral angle of 40° with triazole ring. 3',4',5'-trimethoxyphenyl moiety is anti to triazole ring about the azomethine linkage. The molecular packing is achieved through N-H···O and a few weak intra and inter molecular hydrogen bonds along with a weak S···O interaction.

The structure analysis of the compound has been done by X-ray methods. 3-D intensity data were collected on Rigaku four circle Diffractometer using Mo-Kα-radiation. The structure was solved by direct methods and refined by full-matrix least-squares method using SHELXL-97 program.

Keywords: TRIAZOLE, MERCAPTO TRIAZOLE, 3',4',5'-TRIMETHOXYBENZYLIDENE