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In order to elucidate the intermolecular interactions involved in complex formation of chitosan with hydrogen halides, crystal structures of chitosan complexes with HBr and HI were analyzed based on synchrotron X-ray fiber diffraction data (BL40B2, SPring-8, Hyogo, Japan). Both crystals are isomorphous and belong to the monoclinic space group $P2_1$. The unit cell constants are a = 9.299(9), b = 9.504(8), c(fiber axis) = 10.41(1) Å and beta = 106.93(8) deg, and a = 9.46(2), b = 9.79(2), c(fiber axis) = 10.33(2) Å and beta = 105.1(2) deg for HBr and HI complexes, respectively. The final packing models were obtained by the linked-atom least-squares refinement, which gave R-factors of 0.192 for HBr complex (93 observed spots) and 0.193 for HI complex (44 observed spots). The halide ions are aligned along the c-axis at intervals of about 5Å and are surrounded by four polymer chains. In an asymmetric unit, there are two halide ions. One ion accepts three hydrogen bonds from NH_3^+ groups (N2 nitrogen). The other one participates in one hydrogen bond from N2 and two hydrogen bonds from primary hydroxyl groups (O6 oxygen). In addition, the chitosan chains are linked by N-H...O and C-H...O hydrogen bonds along the b-axis direction. The crystal structure of the hydrated form of chitosan was reanalyzed using synchrotron X-ray fiber diffraction data (BL38B1, SPring-8). The chitosan chains make sheet structures parallel to the bc-plane and these sheets stack along the a-axis. Water molecules form columns between these sheets. The sites of the halide ions in the complex crystals are similar to those of the water molecules in the hydrated form. It was suggested that the columnar structure of water in the hydrated form plays an important role for the complex formation.

Keywords: complexes, fibre diffraction, synchrotron radiation

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Structural analysis of F-actin using fiber diffraction

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Actin is on of the most abundant protein and works in the eukaryotic cells. Actin has two state－monomeric G-actin and polymerized F-actin. The F-actin is a functional form in muscle and cellar transportation. Also the processes of polymerization of actin are important for cell motility. In 1990, crystal structure of actin-DNase I complex was solved by Holmes group. Since then, a lot of crystal structures of actin were solved. Actin has a nucleotide-binding cleft enclosed by two major domains. In almost all of actin crystal structure, the cleft is closed and the two domains are tilted each other in the propeller-like manner. The conformation appears to be typical G-actin conformation. In the back-to-back paper, his group also proposed the structural model for F-actin using the fiber diffractions from well-oriented sols. The monomer arrangement and orientation in the F-actin model have widely been accepted. However, to precede the next steps of actin studies, for example detailed interaction between F-actin and myosin, actin ATPase and polymerization, we need a precise model for the F-actin structure. We recorded the diffraction patterns from well-oriented F-actin sols at SPring-8. Using the diffraction pattern up to 3.3 Å resolution in the radial direction and 5.5 Å along the equator, we made a new model for F-actin structure. We put crystal structures in helical arrangement of F-actin and fitted the model to the reflection data using a combination of normal mode motions of G-actin. After that, we refined the structure using the MD refinement by FX-plor. We found a novel conformation of actin in F-actin filament. In this conformation, whole molecule is flat and the nucleotide binding cleft remains closed.

Keywords: actin, fiber diffraction, macromolecular polymer

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Computational methods in fibre diffraction

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Fibre diffraction is used in structure determination of filamentous biological polymers when conventional macromolecular crystallography cannot be applied. The technique made a critical impact in the early days of structural biology, but since then has gone largely unheralded, partially owing to the limitations of available computational tools. With the advancement of computer and synchrotron technologies and the arrival of new structural problems, creating reliable software for fibre diffraction research became both feasible and urgent. In collaboration with fibre diffraction communities in North America and Europe, we have developed and integrated a collection of such software tools. These tools include WCEN for analysis of diffraction patterns, correcting systematic effects and mapping diffraction data into reciprocal space, RAD for angular deconvolution of diffraction data from partially oriented specimens, FX-PLOR for model building and refinement against high resolution diffraction data, and FNV for analysis using Fourier-Bessel transforms and syntheses. The software collection, already successfully applied in the study of non-crystalline filamentous systems such as amyloids and viruses, together with the CCP13 software suite, covers all aspects of fibre diffraction. The work is supported by FiberNet, a Research Coordination Network for Fiber Diffraction from Biological Polymers and Assemblies established through NSF grant MCB-0234001.

Keywords: fibre diffraction, macromolecular structure determination, computational method

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Application of neutron imaging plate system to crystal structure analysis of deuterated polymers

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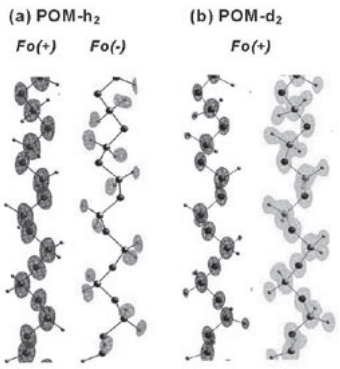
The 2-dimensional neutron imaging plate system BIX-3, develped for the structural biology, has been succesfully applied to the crystal structure analysis of various kinds of synthetic polymers, and the extraction of hydrogen atom positions has been made with high accuracy. The polymers investigated here were polyethylene, polyoxymethylene, isotactic polypropylene, poly(ethylene oxide),

and so on. For example, in the case of polyoxymethylene, synchrotron X-ray diffraction gave us the observed reflections more than 700, from which the details of the chain conformation and chain packing mode were derived accurately. The 2-dimensional neutron diffraction data collected for both of H- and D-polyoxymethylene samples allowed us to extract the H and D atomic positions exactly as shown in Figure. The similar results were obtained also for the other polymers listed above.



Keywords: deuterated polymers, neutron diffraction data, hydrogen atoms

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Invarioms for the DNA

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The application of the invariom database [1] of pseudoatom scattering factors that are transferable from one molecule to another (invarioms) in the refinement of single crystal X-ray diffraction data has so far been limited to amino acids / oligopeptide molecules and to a number of organic compounds. Nucleotide bases and the macromolecules they are part of form another class of compounds that are ideally suited for a database approach. An advantage of a scattering-factor database built by purely theoretical methodology is that the database can be extended to any chemical environment with ease. However, scattering-factor (invariom) assignment based on experimentally determined geometries is sometimes not unambiguous. This problem does occur more often for nucleotide bases, where the distinction of bond orders from the bond distance is more difficult. We present a new algorithm that circumvents these difficulties and have extended the invariom database also to nucleotide bases. Charge-density quality single-crystal X-ray diffraction data on thymidine [2], adenosine and the watson-crick base pair 1-methyl-adenine-1-methyl-thymine provide real-life examples to validate these database entries. To highlight their applicability to larger systems, initial refinement results of X-ray data of a DNA-porphyrin complex [3] are also presented. Diffraction data to 0.86 Å resolution were retrieved from the Protein Data Bank (1EM0).

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Keywords: charge density studies, nucleic acid complexes, databases

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Charge density and X-ray absorption studies on heterobimetallic phosphido-bridged Mo and W complexes

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The heterobimetallic phosphido-bridged complexes, $CpW(CO)_2(\mu\text{-}PPh_2)Mo(CO)_5$ (1) with W-Mo distance 3.1723(4)Å and $CpW(CO)_3(\mu\text{-}PPh_2)Mo(CO)_5$ (2) with W-Mo distance 4.510(4)Å have been reported with special chemical reaction properties because of the interaction between W and Mo: complex 2 can be converted into 1 after irradiation with UV or heating at reflux temperature. In order to correlate how the chemical bond of Mo-W affect the chemical reaction in complex 1 and 2, the accurate single crystal diffraction data of both complexes at 100 K are collected and the experimental electron density in terms of multipole model are derived to investigate the electron density distribution and chemical bonds. All chemical bonds will be characterized based on atoms in molecule theory, and classified by the location of the bond critical point (BCP) and its associated topological properties. The bonding characters will be complemented with x-ray absorption spectroscopy of Mo K-edge. All experimental observations will be compared with the density functional theory calculation. A reasonable explanation will be given in relation with the different chemical reaction properties between 1 and 2.

Keywords: charge denstiy, X-ray absorption spectroscopy, density functional theory

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Generalized library of experimental multipolar atoms

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A library of average multipole populations describing the electron density of common chemical groups is being built. The library values are obtained from several small peptide or organic compound crystal structures refined against available ultra-high-resolution X-ray diffraction data [1]. The atom types are defined on the basis of their chemical environment and local symmetry. New local coordinate axes systems have been defined in special cases. The introduction of optimal constrains and restrains allows for reduction of the number of multipolar parameters. As a consequence more meaningful results and stable refinements are gained. We will present the latest advancements of the crystallographic software MoPro suite [2] for the estimation of protein-ligand electrostatic interaction energy. Aldose reductase (hAR) is an enzyme involved in diabetes complications and the inhibition of the protein is a therapeutic way to treat them. The electrostatics interactions of the Fidarestat inhibitor with the enzyme active site have been characterized using the library electron density modelling. This information is useful to understand the affinity and specificity of Fidarestat with hAR compared to other