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Keywords: neutron, glycosyltransferase, mechanism

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Joint Neutron/X-ray crystallogrpahic study on the mechanism of pectate lyase

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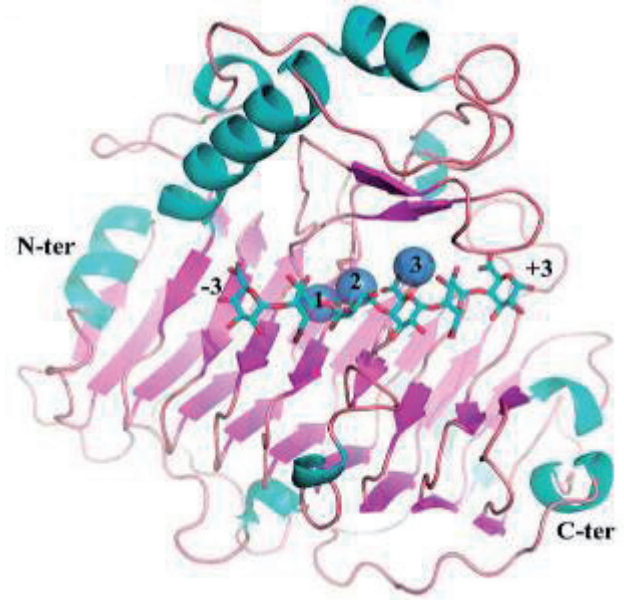
Bacterial soft-rot disease is a major problem for plants in the field and in storage. It is caused by the invasion of pathogens after cell wall damage by pectate lyases secreted by bacteria such as *Bacillus subtilis* (BsPel).

BsPel belongs to a family of lyases that cleave α-1,4-linked galacturonic acid units of the pectate component of plant cell walls via an *anti*-β-elimination reaction. A proposed catalytic mechanism [1] features a conserved arginine acting as a base as low as pH4.5. Where calcium ions are required for activity; a primary Ca^{+2} ion binds substrate and an additional 2 stabilise the intermediate.

At present the major mechanistic question is the protonation state of this active site arginine, which at physiological pH (7.0) is expected to be protonated. Therefore, proton abstraction initiating the reaction is likely to result from a local shift of pKa that has yet to be proven.

A joint Neutron and X-ray study has been carried out to study the structure of BsPel and the active site residues in particular. We have produced perdeuterated BsPel, crystallised it, and collected both Neutron and X-ray data on the same crystal sample.



Above: Cartoon representation of the parallel β-helix architecture (arrows) and 3 calcium ions (spheres).

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Keywords: neutron, X-ray, pectate-lyase

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Transthyretin amyloidosis – insights from neutron crystallography

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Human Transthyretin (TTR) is a homotetramer protein that transports thyroxine in the blood and cerebrospinal fluid. TTR is intrinsically amyloidogenic and associated with three major amyloid diseases. Familial amyloydotic polyneuropathy (FAP) manifests itself by deposition of fibrils and amorphous aggregates in the liver, whereas familial amyloidotic cardiomyopathy (FAC) afflicts the heart. Both diseases are hereditary and due to point mutations in the genome, rendering the protein more labile and thus prone to dissociation and aggregation. The third, senile systemic amyloidosis (SSA) is linked to native TRR and is the most widespread; it affects about 25% of the population over 80 years old. Early diagnosis and new therapies, including small molecule compounds stabilizing the native fold, offer the possibility of a prolonged remission of this otherwise inexorable disease. The analysis of the protonation states of the subunit interface and changes occurring upon ligand binding is therefore of great interest for an understanding of the factors that stabilise the homotetramer, prevent dissociation and ultimately amyloidosis. Perdeuterated human TTR has been overexpressed in *E. coli* in fermenters of the ILL Deuteration Laboratory and large crystals have been grown. Neutron quasi-Laue data to 2.1 Å resolution and room temperature X-ray data to 1.9 Å resolution were collected on perdeuterated TTR crystals and used for a joint X-ray/neutron structural refinement with phenix.refine. The results are being used to study protonation and hydration in native TTR with a view to follow-up studies of TTR in complex with a number of promising ligands.

Keywords: ttr, amyloidosis, neutron crystallography

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The first neutron structure analysis of protein with ibix in j-parc

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Since 2004, Ibaraki prefecture has constructed the TOF neutron

biological diffractometer (IBARAKI Biological Crystal Diffractometer: iBIX) at BL03, in Material and Life Science Facility in J-PARC. The diffractometer is designed to cover samples that have their cell edges up to around 135Å with a resolution up to 1.2Å (biological macromolecules) and to 0.7Å (organic compounds). In 2008, the basic part of the instrument of iBIX, including 14 detectors (a two dimensional detector which consists of ZnS:Ag/$^{10}B_2O_3$ scintillators with a wavelength shift fiber system, and the total solid angle of the detector system: 9%) has been completed to prepare for diffraction experiment. Since the end of December in 2008, iBIX bas been opened for users. Neutron diffraction datasets of several organic compounds of the known structure have been collected by using the iBIX and molecular structures obtained from the analysis agreed with the reported structures.

We have tried to collect the first TOF neutron diffraction dataset of a protein crystal by using iBIX in order to estimate the performance and characteristics of iBIX. The selected crystal for the purpose is ribonuclease A(RNase A) soaked in heavy water. The crystal volume was 4.7 mm^3. The cell parameters were a=30.4Å, b=38.6Å, c=53.4Å, b=105.8º in a monoclinic form, respectively. Measurement conditions are as follow: the accelerator beam power: 120kW, the pulse repetition: 25Hz, the range of wavelengths: 1.5~4.5Å (the 1st frame), 4.2~7.5Å (the 2nd frame), the number of measurement settings: 100 settings (1st frame: 67settings, 2nd frame: 43 settings), the exposure time: 5 hours/setting (the 1st frame), 1 hour/setting (the 2nd frame), the total amount of measurement time for full dataset: 17 days.

The data reduction (to extract a HKLF list from raw data) was carried out by using a new data processing software "STARGazer" which we have developed for TOF neutron diffraction data. The data reduction for almost all of the both frame data was finished and consequently HKLF list was obtained. The completeness of Bragg reflections is 88.8% of 1.7Å resolution. The structure refinement was carried out with this intensity dataset. We have succeeded in obtaining the reasonable structure after the structure refinement by comparing with the already-reported structure [1]. After accelerator power will become 1MW in J-PARC and the total number of detector for iBIX will become 30, the full dataset of standard sample RNase A which is 1mm^3 in volume can be collected in about 3 days.

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Keywords: TOF neutron diffractometer, protein crystallograpy, RNase A

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Crystallographic autostereograms

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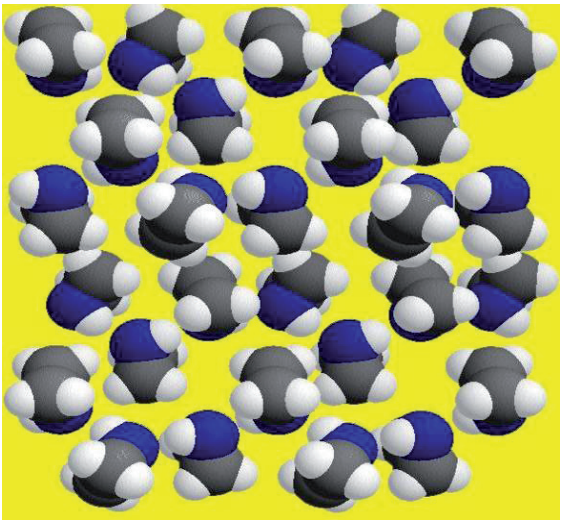
Crystallographic studies are inherently connected with the drawings of crystal structures on publications pages or on computer screens. Various methods have been worked out for facilitating the 3-dimensional arrangements of atoms in 2-dimensional drawings. The most commonly used were stereoscopic pairs, which were prepared after computers could be used for drawing crystal structures [1]. The in-depth viewing of stereopairs required stereoscopes, although it was pointed out (cit.) "that most readers – maybe after a little practice – should be able to achieve stereopsis without mechanical aid" [2]. Recently stereopairs are seldom found in scientific publications, owing to commonly used computers allowing crystal structures to be easily processed and viewed in any style and at any chosen direction. This possibility somewhat degraded the value of clear presentations of crystal structures in crystallographic publications.

Alternative methods for perceiving 3-dimensional crystal structures were also presented. One is based on the concept of autostereograms, or single-image stereograms [3]. Autostereograms can be used not only for scientific publications, but also for artistic presentations of crystal structures.



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Keywords: stereographic, crystal structure, illustration

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Incorporation of crystallographic data use into undergraduate chemistry education

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Nearly every chemistry course in an undergraduate chemistry major's curriculum contains a significant number of topics that can be illustrated or enhanced through use of crystallographic information. This presentation will describe specific examples of the use of crystallographic information in a wide range of chemistry courses. At the General Chemistry level, students learn the basic VSEPR geometries and are faced with need to visualize molecules in three-dimensions. Manipulation, *in silico*, of examples whose coordinates are derived from crystal structures is perhaps the most obvious use. Students are also faced with consideration of evaluating the relationships between bond lengths and bond enthalpies and direct investigation of actual molecular structures is of value. In Organic Chemistry, students can examine specific molecular structures to better understand the 3-D nature of molecules. (Unlike a typical drawing on paper, benzophenone is not flat.) In Physical and Analytical Chemistry, the potential for using large sets of data to evaluate statistical populations such as the average aromatic CC bond length is best accomplished by mining crystallographic data. Biochemists can clearly examine the structures of amino acids to evaluate their zwitterionic nature. In Advanced Inorganic courses, examining molecular structure is particularly relevant. For example, the hexachlorotellurate ion has seven electron domains but has an octahedral VSEPR geometry. Main group VSEPR structure can be nicely juxtaposed with the d-block complexes with structures consistent with the Kepert model. The presentation will