is an extension of the widely used powder diffraction based 1D-PDF technique. It is obtained by Fourier transformation of single crystal (diffuse) scattering. Contrary to 1D-PDF it provides not only information about the length, but also about the spatial orientation of real inter-atomic vectors. The 3D-PDF method allows employing techniques that are difficult to apply in the case of powder diffraction. Complexity of 3D-PDF maps may be strongly reduced by filtering out information that is either already known or not of interest. As an example Bragg scattering from the usually well-known average structure may be eliminated before calculating the 3D-PDF what strongly enhances visibility of disorder information [1]. Further, independent disorder phenomena may be separated in reciprocal space, if they show distinct sets of diffuse scattering, and/or in PDF space, if the orientation of inter-atomic vectors allows distinguishing different kinds of disorder e.g. intra- and interlayer disorder. An instructive example for demonstrating the power of 3D-PDF analysis is diffuse scattering from quasicrystals, which certainly belongs to the most complex disorder problems. It will be shown that the 3D-PDF method allows understanding of extremely complicated diffuse scattering from decagonal Al-Cu-Co and Al-Co-Ni quasicrystals by multiple reduction of complexity in reciprocal and PDF space [2,3].

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Keywords: pair distribution function, diffuse scattering, quasicrystals

MS.77.5

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Nanostructure of silver-free photochromic glasses studied by anomalous small angle X-ray scattering

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Cuprous halide nanocrystals, embedded in a glassy matrix are of interest with respect to their photochromic behaviour. A silver-free photochromic glass was prepared using a $14.2Na_2O$-$6.0Al_2O_3$-$26.6B_2O_3$-$53.2SiO_2$ base glass doped with small amounts of Cl, Br, Cu, Cd, and Sn ions. During isothermal heat treatments at T = 600 ℃, small liquid droplets of CuX precipitate resulting in $CuCl_{0.4}Br_{0.6}$ nanocrystals after cooling. It is known, that both Cd and Sn ions have a large influence on the photochromic properties and on the sizes of the nanocrystals. But the structural arrangement of the Cd and Sn ions in- or outside the nanocrystals is not known. In order to investigate the influence of Cd and Sn on the precipitation process and to understand the growth and growth delay processes, anomalous small angle X-ray scattering (ASAXS) experiments have been performed. The X-ray energy has been tuned near below the K-absorption-edges of Cu, Br, Cd and Sn giving rise to a variation of the atomic scattering factor of the corresponding element. The result of the simultaneous fits of all curves assuming two different models for the particles will be presented. Both models, poly-disperse core-shell and diffusion zone surrounding spherical crystals, lead to the same conclusions. The crystalline core consists of the element Cu, Cl and Br. Cd and Sn are concentrated in a shell surrounding the nanocrystals. These structural models together with the measured viscoelastic behavior of the glass can explain the growth stop of the crystals after annealing of about 60 min at 600 ℃, knowing from previous SAXS studies.

Keywords: ASAXS, photochromic glasses, nanocrystals

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Can amphipols be used to crystallize membrane proteins?

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'Amphipols' are a family of specially designed amphipathic polymers that can substitute to detergents at the hydrophobic transmembrane surface of membrane proteins (1,2). Amphipol-trapped membrane proteins are soluble in aqueous solutions in the absence of detergent, in their native state, and, as a rule, much more stable than in detergent solutions (1-4). Because amphipols are very mild surfactants, they provide a favorable medium in which to fold to their native state denatured membrane proteins (5), including G protein-coupled receptors overexpressed as inclusion bodies (6). Amphipol-trapped membrane proteins can be studied by NMR (7), electron microscopy (8), and most spectroscopic and other biophysical methods (2,3). Because the protein/polymer association is irreversible, trapping with a functionalized amphipol will functionalize the protein without having to modify it chemically or genetically (9). Thus, trapping with a biotinylated amphipol makes it possible to attach the protein to a solid support for the purpose of screening for biological partners, ligands, drugs, antibodies etc. (10). Whether amphipols can be used to crystallize membrane proteins remains, however, an open question. An update will be presented of where we stand relative to this particular application and what the perspectives seem to be.

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Keywords: membrane proteins, detergents, amphipols

MS.78.2

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Crystallisation of the calcium pump of skeletal muscle sarcoplasmic reticulum

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Ca^{2+}-ATPase of skeletal muscle sarcoplasmic reticulum (SERCA1a) is an integral membrane protein of 110K and the best characterised member of the P-type (or E1/E2-type) ion translocating ATPases. It transports 2 Ca^{2+} and counter-transports 2~3 H^+ per ATP hydrolysed. SERCA1a consists of 10 transmembrane helices, 3 cytoplasmic domains (A, actuator; N, nucleotide binding; P, phosphorylation) and small lumenal loops [1]. We have determined the crystal structures of this enzyme in 8 different states, in which the ATPase shows drastically different domain arrangements [1]. All the crystals are of type I (i.e. stacks of membraneous crystals) and required phospholipids, which form bilayers in the crystals. The crystals diffracted to fairly high resolution (better than 2.5 Å resolution for most of them) at BL41XU, SPring-8. As we use the dialysis method for crystallisation, we can accurately control important parameters, such as protein : lipid : detergent ratio. In this presentation, I will briefly describe our experience in crystallisation of SERCA1a.

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Keywords: membrane proteins, crystallisation methods, ATPases

MS.78.3

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Crystallization of visual pigments and archaeal rhodopsins

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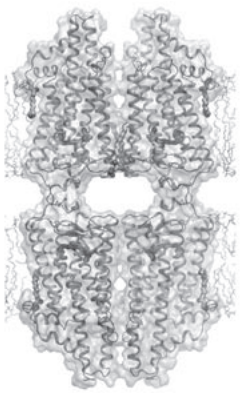
For structural investigation of bacteriorhodpsin, we previously developed the membrane fusion method, by which the trimeric bacteriorhodopsin-lipid complex was crystallized [1]. Recently, we applied this method to prepare 3D crystals of archaerhodopsin-2 without destroying the trimeric structure [2]. These crystals provided information as to the physiological roles of native lipids including bacterioruberine. For structural investigations of visual pigments, we developed a crystallization method by which the protein-lipid interactions can be maintained. Our recent study of squid rhodopsin showed that native lipids mediate the intra-membrane dimerization and that the N terminal polypeptide contributes to the inter-membrane dimerization; i.e., squid rhodopsin is able to form a tetrameric structure [3]. It is suggested that such tetramers are arranged in the apposed microvillar membranes so that the absorption dipole moments of all the retinal chromophores are aligned in parallel with the microvillar axis.



Tetramer of squid rhodopsin

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Keywords: squid rhodopsin, archaerhodopsin, bacteriorhodopsin

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X-ray structure of human gap junction channel

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Inercellular signaling is one of the most essential properties exhibited in multicellular organisms. Gap junction is a structure to allow direct intercellular communication. Here, we have determined the crystal structure of human gap junction channel at 3.5Å. The crystal belonging to a monoclinic space group of *C*2 has six molecules in an asymmetric unit. The initial phases were obtained at 8.0Å resolution by the single isomorphous replacement method combined with the molecular replacement method with a hypothetical structure consisting of four helices. Phase extension was performed up to 3.5Å resolution by six-fold non-crystallographic symmetry averaging and multicrystal averaging. Anormalous dispersion signals from selenium atoms of a seleno-methionines derivative crystal and from sulfur atoms of the native crystal uniquely located amino acids and disulfide bonds in the electron density map. Consequently the trans-membrane parts a typical four－helix bundle, which was quite different from that previously proposed. The two extracellular loops in each monomer that interact with the opposing units have three intramolecular disulfide bonds. The first loop makes the wall of the channel pore and the second loop, which extends to overlay the first loop, makes subtype specific interaction. From our structure , the molecular basis of the specific junctional interaction is revealed.

Keywords: membrane protein X-ray crystal structure determina, cell adhesion, cell communication

MS.78.5

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Diffraction-capable microfluidic crystallization chips for screening and structure determination

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We have designed and fabricated prototype microfluidic crystallization devices from which high-quality diffraction data can be collected without handling of individual crystals. We have also developed tools for straightforward handling and collection of diffraction data. These include modified synchrotron pins for collection of oscillation data from chip sections under cryogenic conditions and a X-Y stage for rapid diffraction scanning that has been integrated with the beamline control system at BL 8.3.1 at the Advanced Light Source. The prototype chips have been designed to minimize background scatter during the X-ray diffraction experiment. The prototype screening chip tests the sample against 96 crystallization reagents at two mixing ratios. The sample chamber for each of these 192 experiments holds ~ 9 nl sample. In addition to the screening chip, we have also developed a prototype chip for the growth of larger crystals that screens 24 reagents at two mixing ratios. The sample chamber for each of these 48 experiments holds ~ 90 nl sample. Using these diffraction-capable chips, we have been able to solve the structure of a seleno-methionine substituted sample using both SAD and 2-wavelength MAD methods. Data will also be presented showing the use of diffraction-scanning data,