

acids is an important challenge. The structure-forming units in the crystals of amino acids are similar to those in the biopolymers: robust head-to-tail chains of molecules linked by hydrogen bonds are preserved even on polymorphic transformations. Packing of the head-to-tail chains can mimic polypeptide structures: helical structures, β -sheets, amyloids. Knowledge about the temperature and pressure-induced changes in these crystals can help to understand better the contributions to the dynamic properties of proteins related to the intrinsic motions of structural fragments. Besides, crystalline amino acids are important materials with non-linear optical and piezoelectric properties. The structures and properties of the chiral and racemic crystals of amino acids are remarkably different. In the present contribution we illustrate this using several selected examples (L- and DL-serine, L- and DL-cysteine, L- and DL-alanine, L- and DL-phenylalanine, L- and DL-phenylglycine) studied at variable temperatures (the range 3-450 K) and variable pressures (up to 1-10 GPa, depending on the compound and the technique) by single-crystal and powder X-ray diffraction, IR-, Raman, inelastic neutron scattering spectroscopy, and calorimetry. The differences in the structural response to variations in temperature and pressure (different values of compressibility and thermal expansion, different strain anisotropy, different stability with respect to phase transitions induced by cooling, heating or increasing pressure). The study was supported by Integration projects #49 and #110 of SB RAS, a grant from RFBR (05-03-32468) and grants from BRHE (NO-008-XI and RUX0-008-NO-06).

Keywords: crystalline amino acids, structure-properties relations, head-to-tail chains

MS.14.3

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Chiroptical properties of *N*-benzoylglycine crystals

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Despite the fact that *N*-benzoylglycine is an achiral molecule, chiral crystal is spontaneously formed. The molecules are helically arranged through the hydrogen bond chains in the crystal. We measured the optical rotatory dispersion by High Accuracy Universal Polarimeter (HAUP). The magnitudes of optical rotation along the helical axes were larger than those perpendicular to the helical axes.

Keywords: chiroptical properties, *N*-benzoylglycine crystals, chiral crystal

MS.14.4

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A solution to the problem why chiral hydrophobic amino acids form crystals with $Z' = 2$

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Chiral amino acids that do not have functional groups in their side chains (hydrophobic amino acids) regularly form crystals with two molecules in the asymmetric unit (with different side-chain conformations as shown for L-valine). In contrast, racemates of

the same compounds always form crystals with $Z' = 1$. What is the origin of the difference between enantiomeric and racemic crystals? No attempts have apparently been made to answer this fundamental question concerning the essential building blocks of life. The present investigation shows that by using a combination of crystallographic, statistical and theoretical methods it is indeed possible to explain the observed behaviour, and furthermore tell why these amino acids do not undergo racemic separation upon crystallization, but rather choose to form racemates in the solid phase.



Keywords: *ab-initio* calculations, amino acids, crystal packing

MS.14.5

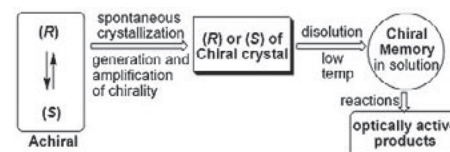
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Control of chirality by spontaneous crystallization and absolute asymmetric synthesis in fluid media

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Asymmetric generation influenced by a chiral crystalline environment is considered an attractive methodology for obtaining optically active compounds from achiral compounds. In recent years, the solid-state photochemical reaction using chiral crystals in a variety of new systems has progressed to such an extent that it can now be regarded as an important branch of organic chemistry. Furthermore, a new methodology using molecular chirality in crystal as a source of chiral memory in solution was explored. The chirality can be effectively transferred to optically active products by asymmetric reactions involving a nucleophilic carbonyl addition and intermolecular photochemical reaction. Now we have found that achiral coumarin-3-carboxamide crystallized in a chiral fashion, and the molecular chirality could be transferred to products by intermolecular 2+2 photocycloaddition reactions, cyclopropanation, and the reaction with carbenes in fluid media with high enantiomeric excesses.



Keywords: chiral space groups, asymmetric synthesis, organic synthesis

MS.15.1

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Small beams can play big roles in macromolecular crystallography

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Progress in many biologically important projects is hindered by the inability to grow large homogeneous crystals of macromolecules and their complexes. Useful data can be collected from 10-mm or smaller crystals when background is reduced by matching the beam size to the size of the sample crystal. An inexpensive apparatus was implemented for delivering high-flux-density, user-selectable, 5-mm and 10-mm beams to the sample. The device is based on overfilling an aperture placed 30 mm upstream of the sample. The aperture is held within a set of nested tubes that act as downstream and upstream scatter guards. The mini-beam apparatus was integrated in the user program on the 23-ID dual canted undulator beamlines of GM/CA CAT at the APS, and its benefits for both large and small crystals were demonstrated (Sanishvili et al., 2008). The advantages of small beams for small crystals are undisputed; quantitative results will be presented. Small beams also offer many advantages for large, inhomogeneous samples. For example, small beams may reduce refined crystal mosaicities; they can dramatically improve diffraction quality when large beams lead to smeared and/or irregularly shaped spots. Operational simplicity and interchangeability with larger beams allows small beams to be used as probes to identify optimal regions of a crystal for data collection with a larger beam.

Sanishvili, R., Nagarajan, V., Yoder, D., Becker, M., Xu, S., Corcoran, S., Akey, D. L., Smith, J. L., and Fischetti, R. F. (2008). *Acta Crystallogr D* 64, 425-35.

Keywords: micro-crystals, micro-beam, radiation damage

MS.15.2

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Recent developments and success on ID23-2, at the ESRF

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The ESRF ID23-2 beam line is a microfocus beam line fully dedicated for studying macromolecular microcrystals. It is a fixed wavelength beam line using a single bounce Si[111] monochromator; the beam is focused down to 7.5 μm horizontally by 5 μm vertically (FWHM) by Pt-coated silicon mirrors in a Kirkpatrick-Baez (KB) geometry. The experimental setup is composed of a MD2m diffractometer (from MAATEL, under an EMBL patent), a SC3 sample changer [1] and a MarMOSAIC 225 CCD detector. The main challenge for ID23-2 was to provide to the MX user community a beam line with a beam size smaller than 10 μm in diameter while keeping the same "easy-to-use" environment and reliability as the other ESRF MX beam lines (ID14, ID23-1, ID29, BM14) [2]. The beam line has been open to the user community since mid-November 2005. The first year of user operation has demonstrated that the design was basically sound and that the beamline can be used by inexperienced users. Instrumentation developments, data collection strategies and some user results will be presented and discussed.

[1] Cipriani et al. *Acta Cryst.* (2006) D62, 1251-1259.

[2] http://www.esrf.fr/UsersAndScience/Experiments/MX/About_our_beamlines

Keywords: microcrystals, synchrotron X-ray instrumentation, synchrotron structural biology research

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Microbeam studies of insect virus polyhedra, infectious protein crystals containing virus particles

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Cypovirus and baculovirus are two unrelated types of insect virus that both produce very unusual infective particles - stable micron sized protein crystals called polyhedra that can remain infective in soil for years. Polyhedra form inside infected larval cells and consist of a body centred cubic lattice of 28kD viral polyhedrin protein molecules. How the growing crystals selectively incorporate virus particles from the complex intracellular 'soup' is intriguing. Cypovirus polyhedra form in the cytoplasm and baculovirus polyhedra form in the nucleus. The amino acid sequences of the corresponding polyhedrin molecules have no evident homology. Despite these differences the unit cells of the two polyhedra have nearly identical 103Å cell dimensions. Polyhedra are unusually stable and easily obtained protein crystals and may in future provide an interesting platform for protein engineering. We are interested in these possibilities and in understanding the structural biology of these unique viral structures. Using samples provided mainly by Hajime Mori at the Kyoto Institute of Technology, we have been collecting micro-beam X-ray diffraction data from viral polyhedra since 2004 in collaboration with Clemens Schulze-Briese at the Swiss Light Source. The ~2Å resolution atomic structure of cypovirus was obtained in 2006 using MIR methods. The talk will summarise the project.

Keywords: cypovirus, polyhedrin, micro-crystallography

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A new beamline to achieve protein micro-crystallography at SPring-8

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BL32XU, a new undulator beamline at SPring-8 for protein micro-crystallography, is being under construction and will be operated for the National Project from 2010 in Japan. Recently, users' demands for high-quality data collection from protein micro-crystals are increasing as target proteins get large and difficult to be crystallized. Achieving the objective requires very high signal-to-noise ratio of diffraction spots from a sample crystal. Spatially brilliant and small beam, of the order of a few or several micrometers, is proved to achieve the objective at several synchrotron radiation facilities. We designed a micro-focus beamline, BL32XU at SPring-8, which provides dense and micro-sized X-rays. A hybrid in-vacuum undulator developed at SPring-8 will be equipped. X-rays