[1] Turner D. R., Spencer E. C., Howard J. A. K, Tocher D. A., Steed J. W., *Chem. Commun.*, 2004, 1352. [2] Turner D. R., Light M. E., Hursthouse M. B, Steed J. W., *Chem. Commun.*, 2004, 1354. [3] Turner D. R., Smith B., Goeta A. E., Evans I. R., Tocher D. A., Howard J. A. K., Steed J. W., *Cryst. Eng. Comm.*, 2004, **6**, 633. [4] Turner D. R., Smith B., Spencer E. C., Goeta A. E., Evans I. R., Tocher D. A., Howard J. A. K., Steed J. W., *New. J. Chem.*, 2005, **29**, 90.

Keywords: anion, hydrogen bond, motif

MS31.26.5

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Molecular-Level Devices and Machines

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The chemical, bottom up approach, based on the concepts of supramolecular chemistry, can be very useful to design and construct interesting nanostructures.

By using this approach, the macroscopic concepts of a device and a machine can indeed be straightforwardly extended to the molecular level [1]. A *molecular–level* device can be defined as an assembly of a discrete number of molecular components designed to achieve a specific function. Each molecular component performs a single act, while the entire assembly performs a more complex function, which results from the cooperation of the various molecular components. A *molecular–level machine* is a particular type of molecular–level device in which the component parts can display changes in their relative positions as a result of some external stimulus.

Molecular–level devices and machines operate via electronic and/or nuclear rearrangements and, like macroscopic devices and machines, are characterized by *(i)* the kind of energy input supplied to make them work, *(ii)* the way in which their operation can be monitored, *(iii)* the possibility to repeat the operation at will (cyclic process), *(iv)* the time scale needed to complete a cycle, and *(v)* the performed function. In this lecture, we will illustrate examples of recent achievements in this field.

[1] Balzani V., Credi A., Venturi M., *Molecular Devices and Machines - A Journey in the Nano World*, Wiley-VCH, 2003.

Keywords: supramolecular chemistry, photochemistry, electrochemistry

MS32 STRUCTURE DETERMINATION FROM POWDER DIFFRACTION DATA (INORGANICS)

Chairpersons: Angela Altomare, Holger Putz

MS32.26.1

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Exploiting Preferred Orientation to Resolve the Intensities of Overlapping Reflections

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In 1999, Wessels *et al.*[1] demonstrated the practical viability of the 'texture method' for resolving reflections that overlap in a powder diffraction pattern. By collecting synchrotron data on a textured polycrystalline sample as a function of sample orientation, more information about the relative intensities of overlapping reflections could be obtained. A full texture analysis is used to establish how the crystallites are oriented in the sample, and then a single set of (single-crystal-like) reflection intensities is extracted via a joint refinement procedure using all diffraction patterns (between 5 and 1296) simultaneously. The data collection and analysis strategies for both reflection and transmission geometries have been described [2]. To develop the method further so that even more complex structures can be accessed, several possibilities are being explored. (1) A new method for preparing textured powder samples using a repetitive pressing procedure has been developed. (2) To optimize the resolution of the data, diagonal displacement of the imaging plate at the maximum sample-to-detector distance has been evaluated. (3) To improve the resolution still further, an experimental setup with a one-dimensional Si-microstrip detector, has been devised. (4) A new background and scaling procedure has been implemented in the data analysis software.

[1] Wessels T., Baerlocher Ch., McCusker L.B., *Science*, 1999, **284**, 477. [2] Baerlocher Ch., McCusker L.B., Prokic S., Wessels T., *Z. Kristallogr.*, 2004, **219**, 803.

Keywords: powder diffraction, preferred orientation, structure solution

MS32.26.2

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New Strategies for the *ab-initio* Structure Solution in EXPO2005

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The full pathway in the *ab-initio* crystal structure solution from powder data has been made more straightforward by the package EXPO2004 [1] which is able to: index the diffraction pattern; identify the most plausible space group; estimate the reflection integrated intensities; solve the crystal structure by Direct Methods, in eventual combination with Monte Carlo approach; refine the structure model by Rietveld technique.

New strategies have been recently introduced in EXPO2004 in order to enhance its power, leading to EXPO2005. Among them the most relevant are: a) an improved algorithm for space group determination; b) a new definition of the background contribution; c) efficient methods for estimating the integrated intensities *via* a systematic procedure based on coding theory and/or Patterson inversion technique; d) an effective figure of merit able to identify the most plausible phases set; e) a powerful global optimization approach to be applied in case of organic structures; f) a more robust structure refinement procedure.

The EXPO2005 features and applications will be described.

[1] Altomare A., Caliandro R., Camalli M., Cuocci C., Giacovazzo C., Moliterni A.G.G., Rizzi R., *J. Appl. Cyst*, 2004, **37**, 1025-1028.

Keywords: ab-initio structure determination, powder software, computational crystallography

MS32.26.3

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Are Well Known Phase Diagrams Really Well Known ?

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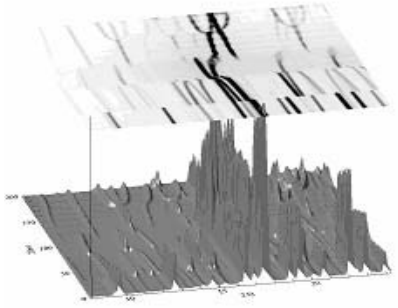

Fig. 1: Temperature dependent powder patterns of RbC_2O_4 [1].

Phase diagrams, which show the preferred physical states of matter at different temperatures and/or pressure, are available for many common substances near ambient conditions. The number of previously unidenti-fied polymorphic phases increases con-siderably even for "well known" com-pounds (see Fig. 1) if the technique of high-throughput *in-situ* synchrotron powder diffraction in combination with fast 2D-detectors is applied. The main problem is related to the enormous amount of data which need to be processed efficiently. Techniques to solve part of this problem [2] are presented during the talk.

[1] Dinnebier R.E., Vensky S., Hanson J., Jansen M., *Chem. Eur. J.*,2005, **11**, 1119. [2] Hinrichsen B., Dinnebier R.E., Jansen M., 2005, *in preparation.*
Keywords: in-situ powder diffraction, phase transitions, phase diagram

MS32.26.4
Acta Cryst. (2005). A**61**, C46
Structure Solution of Thermal Decomposition Compounds using Laboratory X-rays
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Powder X-ray diffraction at non ambient conditions is developing tremendously, thanks to the rapid progresses in diffraction techniques, methods and software. The present study focuses on the structure solution of inorganic powdered compounds resulting from thermal transformations, using the Bragg-Brentano optics with a conventional X-ray source. Some features related to *in situ* powder data collection are discussed. They include the sample surface displacement, which generates errors on peak positions for pattern indexing, the thermal stability of the products upon heating and the problem of line overlap. Indeed, the latter may arise from diffraction line broadening generated by the crystallite fragmentation during the thermal transformation. This is a major limiting factor for solving the crystal structure, since it strongly affects the structure solution with the direct methods and global optimisation approaches. The influence of the microstructure on the structure solution of the decomposition compound γ-$Zn_2P_2O_7$ is illustrated by a study from simulated patterns.

Representative examples of *ab initio* structure determination of thermal decomposition products will be described, such as those obtained by dehydration reactions of open-framework oxalate and phenylphosphonate materials, and by degradation of nitrate and squarate compounds.
Keywords: structure determination, thermal decomposition, powder diffraction

MS32.26.5
Acta Cryst. (2005). A**61**, C46
Structure Solution of Single-Element Molecules from Pair Distribution Function
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Recent developments of synchrotron x-ray and neutron instruments and acquisition techniques allowed fast and precise measurements of experimental Pair Distribution Functions (PDFs) from molecules, crystals and disordered materials. However, it is usually complicated to extract the structure information from PDF data, and the data processing typically involves a tedious testing of a series of structure models. Therefore it is desirable to find a better way how to analyze PDF data. For single-atom molecules the PDF curves can be converted to a table of inter-atomic distances, which transforms the PDF curve-fitting to a molecular conformation problem. We have developed several algorithms on reconstruction of single atom molecules and tested them with artificial and experimental distance data.
Keywords: ab-initio structure determination, pair distribution function, molecular structure

MS33 SOFT CONDENSED ORGANIC-BIOLOGICAL MATERIALS UNDER PRESSURE

Chairpersons: Roger Fourme, Wilson Poon

MS33.26.1
Acta Cryst. (2005). A**61**, C46
Exploring the Configurational Landscape of Biomolecular Systems under Extreme Conditions
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Lipid bilayers, which provide valuable model systems for biomembranes, display a variety of polymorphic phases, depending on their molecular structure and environmental conditions, such as pH, ionic strength, temperature and pressure. By using spectroscopic and diffraction techniques, the temperature and pressure dependent structure and phase behaviour of simple lipid bilayers as well as binary and ternary (raft) lipid mixtures have been studied. Neutron small-angle scattering, two-photon excited fluorescence microscopy, and FT-IR spectroscopy were used to study also the lateral organization of phase-separated lipid membranes and the influence of peptide incorporation. Moreover, applying the pressure-jump relaxation technique in combination with time-resolved spectroscopic and diffraction techniques, the kinetics of various lipid phase transformations was investigated. The technique was also be applied to study other biomolecular structural transformations, such as protein folding. We present data on the pressure-induced un/refolding of various proteins. A thermodynamic approach is used for determining the stability of proteins as a function of both temperature and pressure and express it as a three-dimensional free energy surface. Morover, the effect of various chaotropic and kosmotropic cosolvents on the temperature- and pressure-dependent structure and stability of proteins is discussed. Finally, recent advances in using pressure for studying misfolding, aggregation and fibril formation (amyloidogenesis) of proteins (e.g., insulin, PrP) will be discussed.
Keywords: high pressure, membranes, proteins

MS33.26.2
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Probing two Heads Configuration of Heavy Meromyosin by High-pressure SAXS Technique
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We are studying multiple conformations of myosin, by employing the high-pressure X-ray scattering (HP-SAXS). High hydrostatic pressures would shift the equilibrium between conformations. The heavy meromyosin (HMM), a chymotryptic product of myosin, is known to have two heads with one long tail, and HP-SAXS is highly sensitive to the two head orientations. We have carefully optimized the solvent condition for minimal deterioration of HMM both due to aging and radiation damage. The experiments were done at BL45XU-SAXS (SPring-8, Harima) using a compact high-pressure cell [1].

Under 0.1-200 MPa, no structural change was observed that points to that the asymmetric configuration of two heads was rather stable [2]. Above 250 MPa, HP-SAXS pattern of HMM irreversibly changed. At room temperature the change is kinetically controlled, while at -12 °C under 200 MPa HMM structure was equilibrated. The pressure treated samples were all reversible in terms of actin binding and intrinsic Trp fluorescence. We will report on the solution structure of HMM based on HP-SAXS under the low temperature and high- pressure condition.

[1] Nishikawa Y., Fujisawa T., Inoko Y., Moritoki M., *Nucl Instrum Meth A*, 2001, **467**, 1384. [2] Harris S.P., Heller W.T., Greaser M.L., Moss R.L., Trewhella J., *J Biol Chem.*, 2003, **278**, 6034.
Keywords: high-pressure research, SAXS and SANS synchrotron, proteins muscle

MS33.26.3
Acta Cryst. (2005). A**61**, C46-C47
SAXS Investigations of Conformation and Stability of Eye Lens Proteins under Pressure
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We have combined small angle X-ray scattering (SAXS) and a high-pressure cell to study the effect of pressure, temperature and pH,