MS65.28.5
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Structural and Functional Insight into Celldefending Non-specific Nucleases
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The bacterial toxin ColE7 bears an H-N-H motif that has been identified in hundreds of prokaryotic and eukaryotic endonucleases, involved in DNA homing, restriction, repair or chromosome degradation. The crystal structures of the nuclease domain of ColE7 in complex with an 8-bp [1], 12-bp and 18-bp duplex DNA have been determined respectively by X-ray diffraction methods. In each of the structure, the H-N-H motif is bound at the minor groove primarily to DNA phosphate groups, with little interaction to ribose groups and bases. This result provides a structural basis for sugar and sequence independent DNA recognition. Structural comparison shows that several families of endonucleases bind and bend DNA in a similar way to that of the H-N-H ColE7, indicating that endonucleases containing a similar His-metal finger fold of active site possess a universal mode for protein-DNA interactions [2].

[1] Hsia K.-C., Chak K.-F., Liang P.-H., Cheng Y.-S., Ku W.-Y., Yuan H. S., *Structure*, 2004, **12**, 205-214. [2] Hsia K.-C., Li C.-L., Yuan H. S., *Curr. Opin. Struct. Biol.*, 2005, **15**, 126–134.
Keywords: DNA-protein interactions, DNA recognition, endonucleases

MS66 Topology of Crystal Structures: Nets, Knots and Surfaces

Chairpersons: Davide M. Proserpio, Jean-Guillaume Eon

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Self-dual Tilings and Interpenetrating Periodic Nets
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Some self-dual 3-periodic tilings of ordinary space are described. Pairs of nets carried by these tilings (their 1-skeletons) can interpenetrate so that all the rings of one net are catenated with rings of the other and *vice versa*. For a special class of nets their natural tilings are self-dual and these are called *naturally self-dual* nets.

It is shown that a given net can have an infinite number of distinct self-dual tilings, but (probably) at most one natural self-dual tiling.

Four vertex-transitive naturally self-dual nets are identified and it is conjectured that this list is complete. They are among the minimal nets of Bonneau *et al.* [1]. It is shown that these, and closely-related, nets account for the great majority of observed [2] instances of interpenetration in crystal structures.

Some naturally self-dual tilings and their associated nets with more than one kind of vertex are also described and their importance in crystal chemistry indicated.

[1] Bonneau C., Delgado-Friedrichs O., O'Keeffe M., Yaghi O. M., *Acta Crystallogr.*, 2004, **A60**, 517. [2] Blatov V. A., Carlucci L., Ciani G., Proserpio D. M., *Cryst. Eng. Comm.*, 2004, **6**, 377.
Keywords: tilings, periodic nets, interpenetrating nets

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Sphere Packings with Exceptional Properties
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Homogeneous sphere packings with different kinds of unusual and strange properties are discussed.

For most sphere-packing types there exists a minimal density, and the corresponding sphere packings show the highest inherent symmetry of that type. In the other cases the density decreases towards a boundary of the parameter range. So far, three exceptions are known: in one case, the minimum of density refers to parameter values very close to but not identical with those of highest inherent symmetry; two sphere-packing types exist the minimal densities of which occur not only at a single point but at a whole line of its parameter region.

Normally, the small rings of spheres within a sphere packing are not linked. Very few examples, however, have been found where such rings are catenated. In such a case, a purely graph-theoretical characterization of the type is not sufficient [1].

Some sphere packings may be intertwined in such a way that 2, 3, 4, 5 or 8 congruent or enantiomorphic copies interpenetrate each other without mutual contact. For such interpenetrating packings the contact numbers per sphere vary between 3 and 6. In a few cases, sphere packings of the same type may be fitted into each other in different ways. In addition, interpenetrations of two-periodic 6^3 and 48^2 nets of spheres have been derived. Here the nets are arranged in two or three sets of parallel nets [2, 3] with a mutual angle of 90° or 60°, respectively.

[1] Koch E., Sowa H., *Acta Cryst*., 2004, A**60**, 239. [2] Fischer W., Koch E., *Acta Cryst*., 1976, A**32**, 225. [3] Sowa H., Koch E., *Acta Cryst.*, 2004, **A60**, 158.
Keywords: sphere packings, interpenetration, catenation

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Knotted Nets and Weavings – from 2D Hyperbolic to 3D Euclidean Patterns
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Crystalline patterns in 3D euclidean space can be constructed from tilings of 2D hyperbolic space, followed by projection onto three-periodic hyperbolic surfaces. We demonstrate the technique by projection onto the simpler three-periodic minimal surfaces. The edge arrays of finite tiles, that can be systematically enumerated using Delaney-Dress tiling theory, generate many - though not all - of the commonly encountered crystalline networks.

A variety of hyperbolic tilings can be formed using infinite tiles, whose edges are lines or trees. Projections of those examples generate more complex patterns. Packed trees result in multiple interpenetrating networks, which define "polycontinuous" space partitions. Packings of hyperbolic lines project to arrays of generalized helices, whose simplest examples are well-known rod packings. Generic examples are complex 3D weavings, whose knottedness can be captured by analyzing the homotopy of their quotient graphs (links) embedded in the relevant hyperbolic manifolds (the "minimal embeddings" of the links).
Keywords: patterns, minimal surfaces, knots and links

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The Nomenclature of Interpenetration
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The interpenetration of 1D, 2D and 3D networks (especially hydrogen bonded nets and coordination polymers) has become an important phenomenon in crystal engineering [1,2]. In such cases it is important that not only the topology of the interpenetrating networks is described, but also the topology of the interpenetration itself.

Therefore a simple descriptive nomenclature for describing the various modes of interpenetration is necessary. Interpenetration of 1D and 2D nets may be described as parallel or inclined interpenetration, depending on whether the mean directions of propagation (1D) (or mean planes (2D)) are co-linear (or parallel) or not, respectively. The overall topology of the entanglement may be of higher dimension than the individual networks, and thus descriptors of the form mD → nD, where mD is the dimensionality of the individual nets and nD is the dimensionality of the overall entanglement, can be used. It is also

possible to indicate when nets of different dimensionality interpenetrate (e.g. 1D/2D → 3D). Finally, it is equally important to examine the topology of interpenetration for 3D nets – diamondoid networks, for example, can show a number of topologically different modes of interpenetration.

[1] Batten S.R., Robson, R., *Angew. Chem. Int. Ed.*, 1998, **37**, 1460. [2] Batten S.R., *CrystEngComm*, 2001, **3**, 67.
Keywords: interpenetration, topology, networks

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Computer Analysis and Classification of Entanglements in Crystal Structures
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Computer algorithms and programs are developed for the automated analysis and classification of any type entanglements in crystal structures of any complexity and composition. The programs are implemented within a novel version of TOPOS (a program package for multipurpose crystallochemical analysis), where the procedure of recognition of entangled systems is based on the description of a crystal structure as a finite quotient graph. Several levels of the structure representation are provided: strong valence, valence, H bonded, *etc*, to find entanglements in substances of different nature. TOPOS allows one to analyze various entanglement phenomena: interpenetration, polycatenation, polythreading, and polyknotting of any dimensionality. Each entanglement is characterized by a set of topological indices (coordination sequences, Schläfli and vertex symbols). A special classification scheme is proposed and programmed for 3D interpenetration, and a database on topological types of 3D nets is embedded into TOPOS.

These methods and software were applied to the analysis of 3D interpenetrated motifs in the crystal structures of inorganic, organic, and organometallic compounds through the whole ICSD and CSD. More than 500 examples of interpenetration were found and classified, many of them were discovered for the first time. Some unusual crystallographic features of 3D interpenetration are discussed.
Keywords: topology, entanglement, computer analysis

MS67 NON-AMBIENT POWDER DIFFRACTION AND KINETIC STUDIES
Chairpersons: Ivana Radosavljevic Evans, Roland Boese

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Simultaneous Local and Long Range Structure Determination: Application to *in-situ* Studies
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The ability to accurately determine structural disorder, both static and dynamic, requires the application of techniques that directly probe local atomic structure. Recently, the combined application of high energy X-rays, >70 keV, and area detectors have enabled the efficient collection of data suitable for PDF analysis in the time range from seconds to minutes - a three orders of magnitude reduction in measurement time from experiments using point detectors.[1] This has opened up the possibility of in-situ, time resolved experiments as complete data sets can be collected rapidly.

The potential of in-situ studies that probe both local and long-range structure has recently been demonstrated in a study of the phase transition of aluminum trifluoride, which used the combined approach of PDF analysis, Rietveld refinement, and molecular dynamics simulations.[2] The study, which used a sample environment with accurate temperature control and low background, showed clear deviations between the instantaneous local atomic structure and the long-range time averaged structure, as probed by the PDF method and Rietveld refinement, respectively.

[1] Chupas P.J., Qiu X, Lee P.L., Hanson J.C., Grey C.P., Billinge S.J.L., *J. Applied Cryst*., 2003, **36**, 1342. [2] Chupas P.J., Chaudhuri S., et al., *J. Am. Chem. Soc.*, 2004, **126**, 4757.
Keywords: pair distribution function, powder diffraction under nonambient conditions, high energy X-rays

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Qualitative and Quantitative Applications of Non-ambient X-ray Diffractometry
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Variable temperature powder X-ray diffractometry (XRD) is a technique wherein XRD patterns are obtained while a sample is subjected to a controlled temperature program. It is an excellent complement to other thermoanalytical techniques such as differential scanning calorimetry and thermogravimetric analysis. This technique has been used to detect a metastable anhydrous phase formed during dehydration of theophylline monohydrate.

Aminophylline monohydrate transformed to theophylline, either directly or through aminophylline anhydrate as an intermediate. Since XRD permitted simultaneous quantification of the reactant, intermediate and product phases, it was possible to study the effects of temperature, water vapor pressure and processing on the kinetics of this complex reaction.

Finally, low temperature XRD enabled the physical characterization of solutes in frozen aqueous solutions. By attaching a vacuum pump to the low temperature stage of the diffractometer, it was possible to carry out the entire freeze-drying process *in situ*, in the sample chamber of the XRD. This enabled real time monitoring of phase transitions during all the stages of the freeze-drying process. Several pharmaceutical excipients including mannitol, trehalose, glycine, sodium chloride and also excipient mixtures were investigated by this technique.
Keywords: XRD, low temperature, reaction kinetics

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In-situ High Temperature Microstructural Transformations of Oxide Epitaxial Thin Films
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Real physical properties of thin films are greatly influenced by their microstructural characteristics. The elaboration processes of oxide thin films with appropriate microstructures often require post deposition thermal treatments. One of the key points on the way to the introduction of oxide thin films as functional materials into electronic or optoelectronic devices is therefore an accurate control of the structural and microstructural evolution during those thermal treatments. In collaboration with the INEL company, we have recently build a specific laboratory X-ray diffraction (XRD) set-up allowing the collection of diffraction patterns between room temperature and 1500 K. The incidence angle of beam impinging the sample is adjusted with an ω-rotation with a precision of 0.001°. A specific procedure allowing an automatic compensation of the samples dilatation has been developed allowing a positioning precision of a few µm. A rotation around the normal to the sample surface allows to determine the in-plane orientation evolution through ω-scan measurements. The diffracted beams are collected using a curved position sensitive detector so that reciprocal space maps can be recorded in situ in a few minutes only.

Zirconia thin films deposited using sol-gel process onto sapphire substrates have been used as test samples. Reciprocal space maps have been successfully recorded up to 1400 K. The corresponding microstructure evolutions will be presented at the conference.
Keywords: X-ray diffraction, thin films, oxide