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