accumulation is very time-consuming when using full matrix least-square for larger problems.

The sparsity of the normal matrix has long been recognized, and Jelsch[1] has explained the origin of the sparsity in macro-molecular structure refinement where the resolution reaches atomic and subatomic levels. This analysis of the normal matrix revealed a rapid diminution of the cross terms between "distant atoms".

Jelsch's findings can be used to post-rationalize a development in CRYSTALS, where anticipated sparsity was used to optimize the accumulation of a true sparse full matrix. This matrix has almost the full rate of convergence of a traditional full matrix, but for a trial structure with 1,700 parameters the time per cycle was cut by a factor of 12.

The poster describes the implementation of this strategy for building the sparse full normal matrix in CRYSTALS. This has been achieved without compromising any of the existing features, and has the potential to be extended and automated for situations involving pseudo-symmetry.

[1] Jelsch C., *Acta Cryst.*, 2001, **A57**, 558.

**Keywords: least-squares refinement, algorithms, optimization**

**P.03.01.6**

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**Whole Powder Pattern Fitting Methods Focused on Nanocrystalline Materials**

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The methods of diffraction data analysis from polycrystalline materials have reached maturity for samples at the micrometer size scale. Whole Powder Pattern Fitting (WPPF) techniques have become more and more efficient to meet the complex task of structural and microstructural sample characterization. However, when the particle size reduces to few nanometers, special size-related features occur and specifically tuned methods of analysis are needed to extract relevant information, like size, strain and structure concentrations, from powder diffraction patterns. The atoms on the surface may play the major role in very small nanoparticles (NPs), determining important strain contributions and size-related lattice parameters. In the most complex cases of some noble metal, non-crystallographic structures (icosahedron and decahedron) can occur.

WPPF methods will be presented to deal with powder diffraction data of randomly oriented nanocrystals.

Two different approaches will be described:

1) a dedicated approach dealing with noble metal fcc NPs, making use of the Debye function to calculate the diffracted intensity and based on a full-Newton least-squares techniques [1]; 2) a based-shaped convolution method dealing with spherical NPs with lognormal size distribution [2]. Applications will be shown in both cases.

[1] Cervellino A., Giannini C., Guagliardi A., *J. Appl. Cryst.*, 2003, **36**, 1148.
[2] Cervellino A, Giannini C., Guagliardi A., Ladisa M., *Phys. Rev B*, **2005**, *submitted*.

**Keywords: powder diffraction analysis, methods development, nanoparticles**

**P.03.01.7**

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**Automatic Element Assignment and Model Completion for Small-Molecule Structures**

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From reflection file to fully assigned and validated structure – from dream to reality. We will present new software which needs nothing more than the reflection file, the unit cell dimensions and some information about the crystal symmetry. From this starting point, a variety of tools are employed to solve the structure and then correctly assign the atom types, including hydrogen positions. In most routine cases, a correctly assigned and finished structure, complete with IUCr cif-check report and ready-to-submit ".cif" files will result without the need for intervention.

The crystallographic core of this software is provided by G. M. Sheldrick. XS, XD and XL have earned the trust of many crystallographers during decades of wide-spread use in the community. These new tools build on this proven crystallographic basis and are designed to work with the Bruker axs "Apex" software suite. We will present in detail their effectiveness, limitations and detailed roles in the process of fully automatic structure solution and refinement. Issues arising from twinning and disorder as well as incomplete or missing formula and their impact on the system will be discussed.

**Keywords: small molecules, structure determination, automated software**

**P.03.01.8**

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**SXD2001 - a Program for Treating Data from TOF Neutron Single-crystal Diffraction**

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In May 2001, the upgrade of the detector array of the time-of-flight Laue single-crystal diffractometer SXD at ISIS has been completed and the detectors cover a solid-angle of 2π steradians. To meet the needs for data processing a new IDL based program, SXD2001, has been developed.

SXD2001 incorporates various visualisations for raw data and provides a complete route from indexing to intensity extraction and export to structure refinement programs in user friendly graphical interface. File formats suitable for GSAS, FULLPROF, SHELX and JANA2000 are supported. In addition, reciprocal space volumes can be displayed and arbitrarily sliced. Planar cuts or complete volumes can be exported.

Recent examples of science will be shown, including magnetic structure solution and diffuse scattering analysis.

**Keywords: data analysis, Laue diffraction, neutron diffraction**

**P.03.01.9**

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**Phase Mixture Detection by Fuzzy Clustering of X-ray Powder Diffraction Data**

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For application areas ranging from pharmaceutical research (polymorph screening, high-throughput screening) through classical non-ambient experiments to zeolithe research it is necessary to measure a large number of X-ray powder diffraction patterns. The goal is to create a dense grid of measurements that not only offer a reliable overview, but also ensure that information that may be important is not missed

Nowadays this is easy to achieve using modern, fast X-ray diffraction equipment. However before analysis, the massive amount of datasets needs to be reduced. Here full pattern cluster analysis is nowadays seen as a very valuable approach.

However, cluster analysis is not only a data reduction tool, it can also be used to discover hidden patterns in data as well as exposing phase relationships in series of scans of complex mixtures.

In order to be able to deal with phase mixtures without prior knowledge of the possible constituents we have added fuzzy clustering to our other clustering methods in our latest software package [1].

We will use the data from a high temperature phase decomposition experiment on $CuSO_4 \cdot 5H_2O$ to show how fuzzy clustering in conjunction with hierarchical agglomerative cluster analysis and principal components analysis can help to reveal the