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***Lafire*: a Software for Automatic Protein Structure Refinement**

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Refining an initial protein model to its final structure is usually composed of rounds of refinement performed by programs such as CNS and REFMAC, and manual model modification that includes linking and extending fragments, and fitting the ill matched residues of model by using the computer graphics program such as O. The manual model modification requires expertise of crystallography to recognize structural conformation based on electron density, and it is a time consuming process.

For the purpose of reducing the time and manual intervention of refinement, we developed a software named LAFIRE (Local-correlation-coefficient-based Automatic FItting for REfinement) to automate the whole refinement process. Four function modules are designed: building the missing parts in the current model, fitting the model to the electron density map, monitor program and an interface for combining the first two modules and the refinement programs CNS and REFMAC5.

The LAFIRE is already in the state that builds the whole model from fragments by iterative approach, and performs structural refinement process without manual intervention in a few hours or days. LAFIRE is also available on http://altair.sci.hokudai.ac.jp/g6/Research/Lafire_English.html. The overview of LAFIRE, methods of building and fitting in LAFIRE, and refinement applications will be give in this presentation.

Keywords: automatic refinement, software, Lafire

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Powder3D: Towards Automatic Image Plate Analysis

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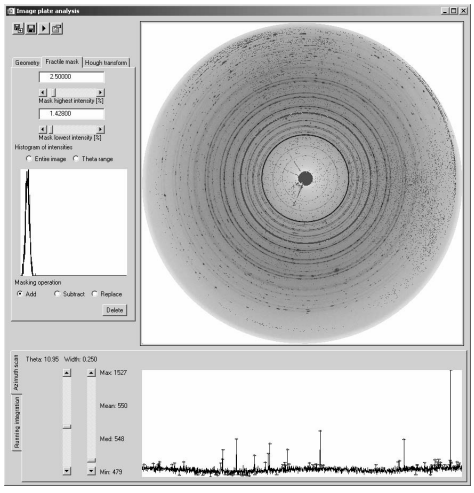

Large area detectors produce vast amounts of data automatically, however, filtering data by means of masking is a manual task, [1]. Alternative processing methods are required.

Powder3D has facilitated the data reduction and analysis of large numbers of powder diffraction patterns, [2]. A new module utilizing hough transforms and fractile statistics for automated image plate analysis and filtering is presented.

[1] Hammersley A.P., Svensson S.O., Hanfland M., Fitch A.N., Hausermann D., *High Pressure Research*, 1996, **14**, 235. [2] Hinrichsen B., Dinnebier R.E., Jansen M., 2005, *to be published*.

Keywords: powder diffraction software, image plates, time-resolved diffraction

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HipHop. A Novel Refinement Method for Protein Structures

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A novel refinement method called HipHop refinement is described. Although HipHop refinement seems to be similar to simulated annealing refinement it is based on a different philosophy - namely that it is in principle impossible to determine a complete structure at a limited resolution. Thus, the limited resolution and the inaccuracy of the underlying X-ray data cause not only errors in the refined structural parameters but principal structural errors in the single model, which is usually used to explain the data.

HipHop refinement is performed in several steps. In the first step, a proper number of pseudo waters corresponding to maxima in the difference Fourier map is added to the model (HIP step, $\mathbf{H}_2\mathbf{O}$ **inp**ut). In the next step, the model is refined and waters not fulfilling density, shape or position criteria are removed from the model (steps HOP, $\mathbf{H}_2\mathbf{O}$ **outp**ut). The process is in fact jumping between local minima – HipHop. During HipHop cycles not only the water arrangement but also side/main chain orientation is changed. The best presentation of HipHop refinement is a multi conformer PDB file.

The method was tested on several different protein structures with excellent results [1,2] The programs are available on http://www.img.cas.cz/hiphop/.

[1] Ondráček J., Weiss M.S., Mesters J.R., *Acta Cryst*., manuscript *in preparation*. [2] Ondráček J., Weiss M.S., Brynda J., Fiala J., Jursík F., Řezáčová P., Jenner L.B., Sedláček J., *Acta Crys.*, *submitted for publication.*

Keywords: protein refinement methods, protein water analysis, protein disorder

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Deconvolution of X-ray Diffraction Profile by Using the Regularization Technique

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The deconvolution of the instrumental function in X-Ray diffraction profile analysis is a basic step in order to obtain reliable results on the microstructure (crystallite size, lattice microstrain, *etc*.) and is a typical example of ill-posed inverse problems. The implementation of an eigen function method with different regularization techniques is investigated and a simple regularization algorithm is proposed.

A simulation of an instrumental-broadened profile superimposed with random noise and background signals is used to investigate the reliability and efficiency of the proposed technique. For the simulation an experimentally defined instrument function based on an accurate mathematical model for Cu emission profile, the geometry of the diffractometer and the physical properties of the specimen are used. The parameters for this instrumental function are obtained by least squares fitting of experimental data sets resulting from the reference materials LaB_6 and Al_2O_3.

Compared to established algorithms, the proposed route is faster and more reliable in terms of stability, especially in the case of large experimental noise. The evaluation of experimental diffraction data of nanocrystalline gold with respect to grain size and microstrain and the comparison with standard evaluation technique is done.

[1] Kurashov V.N., Kurashov A.V., Piskarev V.L., *Proc. SPIE*, 1997, **3317**, 36. [2] Ida T., Toraya H., *J. Appl. Cryst.*, 2002, **35**, 58. [3] Sanchez-Bajo F., Cumbrera F.L., *J. Appl. Cryst.*, 2000, **33**, 259.

Keywords: powder diffraction, profile analysis, regularization

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Faster Least-Squares Refinement of Larger Molecules using the Program CRYSTALS

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Least-squares is both powerful and the most widely used method of structure refinement for small molecules. However normal matrix

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

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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

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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

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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