

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

Acta Cryst. (2005). **A61**, C164

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

Acta Cryst. (2005). **A61**, C164

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

Acta Cryst. (2005). **A61**, C164

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

Acta Cryst. (2005). **A61**, C164-C165

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 zeolite 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 $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ to show how fuzzy clustering in conjunction with hierarchical agglomerative cluster analysis and principal components analysis can help to reveal the

phase relationships and detect phase mixtures in a complex multiphase system.

[1] PANalytical X'Pert HighScore Plus V2.x

Keywords: clustering, statistical analysis experimental data, phase analysis

P.03.02.1

Acta Cryst. (2005). A61, C165

Combined Experimental and Theoretical Studies of Solid State Proton Migration

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Hydrogen bonds are of great interest, due to their importance in structural, functional and dynamical properties of chemical systems, ranging from inorganic to biological chemistry [1]. The very strong hydrogen bonds are of current interest because they enable charge and energy to be transferred between molecules in the solid state.

Recent work has focused on urea-acid complexes, which contain a rich variety of short, strong hydrogen bonds in a relatively simple framework. It has been demonstrated that the combination of cutting edge computational techniques (MD/PW-DFT) with variable temperature neutron diffraction results was successful in showing the migration of the proton with increasing temperature and a plausible explanation for the effect has been presented [2].

A collaborative project with the aim of continuing this work has now begun. A series of hydrogen-bonded adducts have been prepared in a systematic manner for experimental and theoretical investigation. In particular, the effects of temperature and pressure on proton migration and disorder in these adducts are being explored to shed light on the factors that influence proton migration. In this poster presentation we will describe some of our most recent results, which include studies on periodic acid-urea and ammonium iodate.

[1] Steiner T., *Angew. Chem. Int. Ed.*, 2002, **41**, 48. [2] Morrison C. A., Siddick M. M., Camp P. J., Wilson C. C., *J. Am. Chem. Soc.*, *in press*.

Keywords: short hydrogen bonds, solid state, computational chemistry

P.03.03.1

Acta Cryst. (2005). A61, C165

EdMol: A Graphical Molecular Editor

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EdMol is a graphical molecular editor tool for the input of molecular structures in 2D or 3D. The format of output coordinates (Z-Matrix, Spherical, Fractional or Cartesian are some of the options presently implemented) can be selected by the user as input for other programs, as FullProf [1], Expo2004 [2] or StructRes, in order to aid the structure solution, mainly from powder diffraction, using global optimisation as Simulated Annealing, Genetic algorithms or any other algorithm or method as Patterson or Direct Methods.

EdMol is written in Fortran95 using the CrysFML [3] (Crystallographic Fortran Module Library) and X/Winteracter [4] libraries for Linux (x86) and Windows 9X/NT/2K/XP versions. The use of EdMol is totally free for the scientific community and it is distributed within the FullProf Suite package or as a stand-alone program in the FullProf Suite Web site[5].

[1] Rodriguez-Carvajal J., *Physica B*, 1993, **192**, 55. [2] Altomare A., Caliandro R., Camalli M., Cuocci C., da Silva I., Giacovazzo C., Moliterni A. G. G., Rizzi R., *J. Appl. Cryst.*, *submitted*. [3] Rodriguez-Carvajal J., Gonzalez-Platas J., *Compcomm Newsletter*, 2003, **1**, 90. [4] *Winteracter. The Fortran 9X GUI Toolset* <http://www.winteracter.com> [5] *FullProf Suite Web* <http://valmap.dfis.ull.es/fullprof>

Keywords: computer applications, computer software, computer graphics molecular

P.03.03.2

Acta Cryst. (2005). A61, C165

DRAWxtl 5.1 - A Multi-Platform Computer Program to Display Crystal Structures

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The computer program DRAWxtl is designed to display a crystal structure with minimal user input and maximum flexibility. It reads the description of a crystal structure, including unit-cell parameters, space group, atomic coordinates and thermal parameters, combines them with options that define the view, and outputs a geometry object that may contain polyhedra, planes, lone-pair cones, spheres or ellipsoids, bonds, and the unit-cell boundary lines.

Since the recently released version 5.0, the program presents an easy to use graphical user interface where the drawing can be previewed, and all parameters are accessible through convenient menus or direct interaction. A functionally identical command-line version is still available for easier integration into other program packages that provide their own user interface.

The most prominent new feature in version 5.1 is its ability to import electron density information from GSAS and JANA, which can be rendered as meshes and/or solid surfaces. Using a 3D cursor, local maxima in the electron density can be determined to aid in structure solution and refinement.

Both the complete source code and precompiled binaries for Linux, OSX, MS Windows and Irix are freely available from <http://lwfinger.net/drawxtl>.

Keywords: graphics, software, crystal structures

P.03.04.1

Acta Cryst. (2005). A61, C165

AFITT- Working with Good Chemistry

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AFITT is a new molecular graphics program developed by OpenEye Scientific Software for protein crystallographers. It runs on most operating systems, reads almost all data formats, easily connects to databases and will generate good chemical structures from a SMILES string. It has the most up to date rotamer library and Ramachandran plot to easily check the protein structure for outlying residues. It will create refinement dictionaries (either REFMAC or XPLORE format) for ligands and other small molecules automatically. Because AFITT uses the OEChem chemical library and OMEGA conformer generator, good chemistry is preserved even when starting from a SMILES string. Rapid docking into electron density is provided by OpenEye's Shape technology. To enhance communication with chemists, it also provides a 2D graphics window and automatic SMILES naming. Scripting is easily accomplished using Python. AFITT should be particularly useful for those involved in modeling large numbers of structures with small molecules.

Keywords: graphics, computational chemistry, drug design

P.03.04.2

Acta Cryst. (2005). A61, C165-C166

Molecular Dynamics Simulations of tetramethylketone p-tert-butyl calix[4]arene

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We describe the results of Molecular Dynamics simulations of the calixarene tetramethylketone p-tert-butyl calix[4]arene in vacuum, in pure acetonitrile and in the presence of Pb⁺² ions solvated by acetonitrile. The main goal of these calculations is to validate the parametrization of the system model using experimental thermodynamics and crystallographic data and to describe the process by which the calixarene traps one Pb⁺² ion in its hydrophilic cavity and one acetonitrile molecule in its hydrophobic one. This information