

recognition of enantiomorphic solid phases as thermodynamically distinguishable entities and the absence of degrees of freedom when P and T are fixed in the 2-component system (compound and solvent).

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Multiquot algorithm for the automation of structure determination from powder

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Recently mentioned [1] inconsistency of figures of merit [2], [3] when indexing high symmetry lattices turned to a conclusion the smaller cells of lower symmetry can be applied as building units when solving a structure by direct space methods. The approach is expected to decrease time necessary for simulated annealing of one structure solution and may appear particularly useful for large organic structures. The poster illustrates the approach based of example compounds with a small pyrochlore structure. The indexing program suggest several possible solutions and the correct solution of highest symmetry among them. The repetition of the same lattice described by different cells should be considered as an indication of the correct indexing solution. From the other hand, that might be random and unfruitful indexing solution if the lattice is non-primitive but is not observed among proposed cells with its primitive representation. The extension to indexing algorithms, eliminating lower symmetry cells for the same lattice described by high-symmetry cell, and the corrected figures of merit taking into account the number of equal proposal cells might be drawn as follows:

$$M'(20) = M(20) \cdot N_{ep}$$

$$F'(20) = F(20) \cdot N_{ep}$$

where $M(20)_h$ and $F(20)_h$ are $M(20)$ and $F(20)$ for the highest symmetry cell

N_{ep} is the number of equal proposal cells

Then, one may start to search for the structural model applying a cell of lower/volume symmetry providing it may represent a building unit for the larger cell of higher symmetry or may assist to find a sublattice.

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Optimizing the input parameters for powder charge flipping

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The charge-flipping structure-solution algorithm [1,2] implemented in the program *Superflip* [3] and adapted to accommodate powder diffraction data [4] is proving to be a powerful one for solving the structures of polycrystalline materials. A variety of structures have been solved, but as the program is relatively new, the selection of the values for the input parameters has been rather arbitrary. Consequently, a systematic study of the effect of the different parameters on the structure solution was undertaken. Several aspects were investigated: (1) the effect of the input parameters themselves, (2) the effect of data resolution, and (3) the effect of including information from other sources. In parallel, criteria for identifying the better solutions were sought.

As it was not considered to be sensible to test all combinations of all parameters, a more pragmatic approach was followed. Combinations of input parameters chosen to fall into what were thought to be sensible ranges were tested. The parameters were: (1) the reflection overlap factor (κ), (2) the isotropic atomic displacement factor (B_{iso}), (3) the cycle for starting the repartitioning of overlapping reflections and the repeat interval, and (4) the threshold value for charge flipping (δ). To investigate possible differences between inorganic and organic materials, the tests were performed on the zirconium phosphate (ZrPOF-Q1) [5] and on D-ribose data [6].

The structure of ZrPOF-Q1 was originally solved using *Superflip*, but the solutions lacked locations of a few of the Zr, P and O atoms in the zirconium phosphate layers and of the quinolinium ions between the layers. Furthermore, the presence of a center of symmetry only became apparent during the refinement. With optimized input parameters, the centro-symmetric space group was recognized by *Superflip* and complete zirconium phosphate layers could be found in the electron density map. Diffuse clouds of electron density between the layers (quinolinium ions) were apparent.

The structure of D-ribose was solved using a combination of simulated annealing and difference Fourier analysis. None of the initial tests of the *Superflip* input parameters yielded a structure solution. Reasoning that starting with more realistic set of phases might help, possible models were generated using the direct-space program FOX [7]. None of these were correct, but by using the phases calculated from a flexible model to generate starting phase sets, and varying the input parameters once again, a set of optimal parameters could be found, and easily interpretable electron density maps with the correct symmetry generated.

In both cases, the optimization procedure was repeated for lower resolution data. For ZrPOF-Q1, the structure could still be solved with 1.8 Å data, and for D-ribose, with 1.18 Å data. Recognizing the best solutions from *Superflip* remains a challenge, but a cluster analysis of the solutions looks promising and is currently being investigated more carefully.

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More reliable intensity extraction from powders using texture

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The reflection overlap problem is the primary limitation in structure determination from powder diffraction data, and over the last decade, a procedure that addresses this problem experimentally by exploiting preferred orientation has been developed [1]. The intensity variation in the X-ray diffraction pattern obtained from a textured sample as a function of sample orientation (tilt χ and rotation φ) can be applied to resolve the relative intensities of reflections that overlap in a conventional powder diffraction pattern. The availability of a new version of the 1-dimensional Si-microstrip detector (Mythen II) on the Materials Science Beamline at SLS [2] and new features in the computer program *Maud* [3] have allowed both the experimental and the data analysis procedures to be redesigned. By applying these modifications, it was expected that a significant improvement in the quality of the extracted reflection intensities could be achieved.

To test this modified method, two samples of a zirconium phosphate framework material (ZrPOF-pyr) with a known crystal structure [4] were measured: (1) a textured sample using the revised experimental setup and data collection strategy, and (2) an untextured sample in a 0.5mm capillary. Both datasets were collected at the SLS using the Mythen II detector. For the analysis, reflections up to a minimum d -spacing of 1.0 Å were considered, and a reflection separation factor of 0.5*FWHM was used to define the overlap groups.

Comparison of the structure factors extracted from conventionally measured powder diffraction data with those derived from a textured sample indeed shows the latter to be significantly better. The agreement between the amplitudes obtained with a LeBail extraction using *Maud* for the untextured data and those calculated from the refined crystal structure [4] can be expressed as an R value of 0.453. For overlapping reflections (80.2% of the reflections) an R value of 0.547 was obtained. Amplitudes derived in a joint extraction using 32 powder patterns collected on a textured sample yielded R values of 0.380 and 0.408 for all and overlapping reflections, respectively. This improvement was obtained even though the peaks in these patterns are broader (presumably a result of multiple measurements of this particular sample) and have 91.9% overlap.

The quality of the extraction could be further improved by combining the amplitudes derived from the textured sample with those derived from the sharper untextured one. This combination results in R values of 0.326 for all and 0.385 for overlapping reflections. This obvious improvement in the estimation of the structure factor amplitudes should allow some crystal structures that were previously inaccessible to be solved. If the remaining difficulties with the estimation of the background and the description of the peak shapes can also be solved, the method would become even more powerful.

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Patterson-function direct methods for structure solution from powder data

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A new type of direct methods (DM) called Patterson-function DM

are presented that directly explore the Patterson instead of the modulus function. Since they work with the experimental intensities, they are particularly well-suited for handling powder diffraction data. These methods are based on the maximization of the sum function $S_p = \sum_H (I_H - \langle I \rangle) G_H(\Phi)$ in terms of the phases of the structure factors. The quantity accessible from the experiment is I_H , the equidistributed multiplet intensity of reflection H , and $\langle I \rangle$ is the average intensity taken over all non-systematically absent reflections. $G_H(\Phi)$ is the calculated structure-factor amplitude of the squared structure that includes the positivity and the atomicity of the density function in its definition. The S_p sum function can be optimized with the Patterson-function tangent formula (TF) using a variant of the S-FFT algorithm [1]. It is important that overlapped reflections also participate in the phase refinement, so that not only the resolved reflections but the whole pattern contribute decisively to the refinement. The increase in effective data resolution minimizes Fourier series termination effects and improves the accuracy of $G(\Phi)$. The Patterson-function TF has been applied to synchrotron powder data of various organic compounds. In all cases the molecules were easily identified in the respective Fourier maps. By way of illustration the method was applied to synchrotron powder data of a dimer formed by 30 symmetry-independent non-H atoms [2]. However, the application field of the method is not restricted to organic compounds. Here some recent results obtained with coordination and inorganic compounds will be discussed. Since single-crystal data may be regarded as overlap-free powder data, it is clear that Patterson-function DM can cope with powder and single crystal data.

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PDXL Structure Analysis Wizard

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The integrated X-ray powder diffraction software suite PDXL has been developed as a comprehensive software package for the analysis of X-Ray powder diffraction (XPD) data. PDXL allows the user to perform many types of analysis using a single platform, making it possible to obtain a diverse array of analysis results from XPD patterns [1]. Ab-initio crystal structure analysis based on XPD data can be done using PDXL Structure Analysis Package [2].

Recently Rigaku Corporation has included the easy-to-use Structure Analysis Wizard into PDXL software suite. The PDXL Structure Analysis Wizard is a user interface type that presents a user with a sequence of dialog boxes that lead the user through a series of well-defined steps in ab-initio crystal structure analysis. PDXL software suite including the Structure Analysis Wizard makes it possible to easily perform the task of ab-initio crystal structure analysis even for those who are not specialists in the field of X-ray diffraction. The PDXL Structure Analysis Wizard is working together with other PDXL software suite features like indexing programs (DICVOL, ITO, N-TREOR), software tools for space group determination and a easy-to-use Rietveld analysis package (used to perform pattern decomposition and refining the initial crystal structure model).

Several initial crystal structure model construction routines can be used within PDXL Structure Analysis Wizard. PDXL Structure