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NEW INDEXING AND DATA PROCESSING METHODS FOR THE ANALYSIS OF TWINNED CRYSTALS

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The Bruker Nonius software packages now include a number of powerful software tools for use with the κCCD and/or SMART CCD systems. The analysis and indexing of diffraction patterns from non-merohedrally twinned specimens may be carried out with numerical algorithms (Dirax or Gemini) or with visual interactive approaches (RLATT). These programs facilitate the analysis of complicated diffraction patterns to yield individual orientation matrices for multiple twin components.
Two different approaches have been utilized to process the intensity data for twinned specimens. Both of these methods are capable of simultaneously integrating reflection data for multiple twin components. The Dirax and EvalCCD programs used with κCCD systems, utilize a fundamental parameters approach, in which the user describes physical models, which predict spot shapes and impact position on the detector using X-ray tracing techniques. For mosaicity, the standard models allow you to describe the effects of isotropic mosaicity and anisotropic mosaicity (rotational). By defining a vector that describes the direction of the anisotropic mosaicity, reflection boundaries can be modeled resulting in better data than through conventional methods. Visual feedback of the reflection shape predicted by the model is obtained by overlaying it with an accurate map of reflection planes showing all scattering registered.
The Gemini and SAINT programs used with SMART CCD systems are based upon a narrow-frame data collection and integration strategy. SAINT uses an empirical profile-fitting approach to twin integration. It also estimates intensities of individual components in overlapping spots for XS structure solution.

Keywords: TWINNING, CCD DETECTORS, PROBLEM STRUCTURES

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X-RAYS AND NEUTRONS FOR THE INVESTIGATION OF CEMENT

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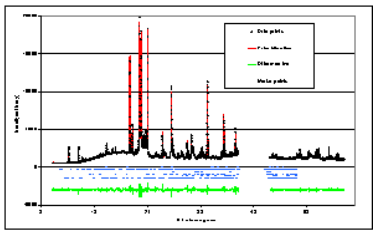
In recent years Rietveld refinement has been used more frequently to investigate the quantitative composition of cement and cement clinker. Most commonly x-ray or synchrotron x-ray radiation is used to obtain powder diffraction data, but recently there has been an increase in the use of neutron diffraction methods to investigate cement and clinker components. A study comparing results of synchrotron, see figure, and neutron analysis of cement and clinker samples will be presented. Advantages of each source for the investigation of cement materials will also be discussed.



Keywords: **CEMENT RIETVELD POWDER DIFFRACTION**

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LOCATING NON-CRYSTALLOGRAPHIC SYMMETRY ELEMENTS: THE PROGRAM FIND2FOLDS

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Averaging is a powerful method to improve phases. To start the averaging process, one needs to know the relation between the molecules whose density is to be averaged. If a large number of heavy-atom sites have been located, one can often recognize multiple copies of a constellation of sites associated with a molecule and its partners in the asymmetric unit. With only two heavy-atom sites per molecule, the problem becomes more difficult because the relation between any two molecules cannot be determined uniquely. We have tackled a case with a single Hg site per monomer in a dimeric protein.
We first calculated trial operators that would interchange the two Hg sites via a rotation of 180° about a non-crystallographic two-fold axis of symmetry lying in the plane midway between the sites. Among these trial operators was one consistent with a peak observed in the self-rotation function. Applying the averaging procedure with 2.8 Å data and SAD phases from the Hg atoms yielded an interpretable map. Somewhat surprisingly, the anomalous signal from two Hg atoms was enough to phase a structure of 1500 residues.
The program written to calculate the trial averaging operators is available upon request. It takes as input the coordinates of two heavy-atom sites and produces as output [1] a set of trial averaging operators and [2] a set of drawing instructions which can be used to display the location of each trial non-crystallographic symmetry axis within the program O.

Keywords: **AVERAGING, SAD, PHASING**

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EXTENDING THE POWER OF THE DIRECT METHODS MODULUS SUM FUNCTION

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The successful application of the direct-methods modulus sum function (=MSF) [1] has been shown in a series of 7 subsequent papers. Recently [2], it has been shown, that maximization of MSF(P)≤(E(H)) E(H,P)>H in terms of the collectivity P of phases of the large reflections is equivalent to minimizing the residual R(P)≤w(H)[E(H)- E(H,P)]^2>H wherein the E(H) are the modules of the normalized structure factors and the average extends over all H reflections (large and weak). As a preliminary step towards the extension of the MSF to larger structures, the influence of the following control parameters of the MSF has been carefully analyzed: i. The E cut-off value for large reflections. Since the E(H,P) are expressed in terms of the phases and the modules of the large E's, the cut-off value affects their accuracy. Best values lie between 1.25-1.50. ii. The ratio of weak to large reflections in H. The normal value used is one although the effectiveness of the MSF is not very sensitive to large deviations from this value. iii. The weighting function used in R(P). w(H) is selected in such a way that more weight is given to the more reliable E(H,P). For the study of the control parameters, structures with up to 500 atoms in the asymmetrical unit were used. All results have been incorporated in the XLENS direct-methods program [1].
References
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Keywords: **DIRECT METHODS SUM FUNCTION PHASE DETERMINATION**