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Crystal Structure Refinement of $Sr(Mg_{1/3}Nb_{2/3})O_3$
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Generally a perovskite compound undergoes the phase transition through a cation substitution originating by ordering and tilting phenomena. Barium magnesium niobate, $Ba(Mg_{1/3}Nb_{2/3})O_3$ (BMN) are typical compounds which have revealed 1:2 ordered structures with the trigonal symmetry. As the La substituted, the structure undergoes the phase transition by cubic, monoclinic[1,2]. In this paper, we present the structural changes in accordance with the cation substitution of Sr by using neutron Rietveld refinement. Neutron powder diffraction data of the sample was obtained at room temperature using high resolution powder diffraction at Korea Atomic Energy Research Institute. The structure model of SMN was used the results deduced from the HRTEM experiments, which SMN has the antiphase tilting and 1:2 ordering. From the refinement, SMN has the monoclinic structure which has 1:2 ordering and antiphase tilting. The space group was determined to be C2/m (#12) with a(Å)=9.8042(2), b(Å)=13.7954(2), c(Å)=5.6310(1), β=90.145(2)^o, V=761.60(3)Å^3. The structure of SMN is distorted by the antiphase tilting of oxygen octahedral with the a^{o}b-b- system of the $(MgNb)O_6$ polyhedra.

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Keywords: perovskite, antiphase tilting, crystal structure

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Improvement of Automated Phase Analysis by Scaling on Standard
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Low deviations in diffraction angles are prerequisite for successful application of automated phase analysis on X-ray diffraction data. However, the angular 2θ values are often biased by errors in sample position or a finite, but unknown offset in angle for the counter zero position. To overcome these systematic errors without knowing their sources in details we developed a two-step procedure, based on Si-Standard (NIST 640 C) added to the unknown sample. The first step is a Rietveld refinement focusing on the profiles of the Si-lines. As a special feature, Program SIMREF2.8 [1], [2] refines coefficients for a polynomial (3. order) to adjust the observed 2θ values to pivots, given by Si lines positions calculated from temperature-dependent Si lattice constants. As output, SIMREF2.8 creates a file with observed intensities, however, with calculated 2θ values.

This file is input for the following phase analysis, using program X'PERT Highscore, e.g.. After scaling results become much more lucid and reliable: In MgH_2+Nb scaling proved to be crucial to obtain correct results for several admixtures and additives.

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Keywords: phase analysis, Rietveld refinement, X-ray diffraction

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A Robust Bulk Solvent Correction and Anisotropic Scaling Procedure in the *CCTBX*
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Macromolecular crystals contain a large amount of disordered solvent which contributes significantly to the diffracted amplitudes at low resolution. The importance of low-resolution data has been demonstrated for many crystallographic calculations [1, 2] and hence an appropriate modelling of the bulk solvent is very important. Jiang & Brünger [1] demonstrated that a flat solvent model is the most reliable while more sophisticated models bring only marginal improvements. The combination of a bulk solvent correction and overall anisotropic scaling [3] is known to be a numerically ill behaved problem [1, 4].

In this work we describe a robust protocol for determination of bulk solvent and anisotropic scaling parameters which we have implemented in the *Computational Crystallographic Toolbox* [5]. This fully automated protocol does not require any user intervention and assures the calculation of optimal, and physically reasonable, output values for these parameters. Also we present a new maximum-likelihood target function for the determination of the flat solvent parameters and anisotropic scale matrix.

[1] Jiang J.-S., Brünger A.T., *J. Mol. Biol*., 1994, **243**, 100-115. [2] Urzhumtsev A., *CCP4 Newsl*., 2000, **38**, 38-49. [3] Sherif S., HenricksonW. A. , *Acta Cryst*., 1987, **A43**, 118-121. [4] FokineA., Urzhumtsev A., *Acta Cryst*., 2002, **D58**, 1387-1392. [5] Grosse-Kunstleve R.W., Sauter N.K., Moriarty N.W., Adams P.D., *J. Appl. Cryst.*, 2002, **35**, 126-136.
Keywords: bulk solvent, anisotropic scaling, maximum-likelihood

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Monitoring Molecular Metamorphosis Using Wide-angle Solution Scattering Data
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The structure motifs of proteins and other biological macromolecules have characteristic distributions of interatomic distances that produce features buried within the x-ray scattering pattern from that molecule in solution. We have demonstrated that wide-angle x-ray solution (WAXS) scattering contains rich details of the secondary, tertiary and quaternary structure of multiple classes of proteins. Uses to date include the observation of ligand-induced structural cdhanges and the monitoring of fold stages during chemical and radiation-induced protein denaturation. WAXS scattering patterns obtained at high flux third generation synchrotron beam lines are not only sensitive to protein conformational states, but the scattering patterns generated can be quantitatively compared to data calculated from detailed structural models derived from crystallographic data.

Our group has used the 18ID beamline at the Advanced Photon Source to study various classes of molecular transitions of proteins and nucleic acids. WAXS is shown here to be a sensitive reporter for such phenomena as radiation-induced quaternary structure breakdown, molecular crowding, folding transition states and changes in structure induced by ligand-binding. As such, WAXS has great potential as a complementary methodology to augment the structural information gleaned from static crystalline arrays.
Keywords: protein structure analysis, WAXS, macromolecular structures

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Total Scattering: the Key to the Local and Medium Range Structure of Complex Materials
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Structural characterization is mainly based on the measurement of *Bragg intensities* and yields the *average* structure of the crystalline material. However, this approach ignores any defects or local structural deviations that manifest themselves as *diffuse scattering*. It also fails in case of disordered materials, badly crystalline such as

many nano-materials, or not crystalline at all, such as glasses. In some cases crystalline and amorphous phases coexist making the traditional crystallographic structure refinement difficult or incomplete. The total scattering pattern, however, contains structural information over all length scales [1] and can be used to obtain a complete structural picture of complex materials.

Here we present different applications of this technique including data taken on the new high resolution neutron powder diffractometer NPDF located at the Lujan Neutron Scattering Center at Los Alamos National Laboratory. This instrument is design for total scattering studies using the Pair Distribution Function (PDF) approach. We hope to attract many new users to use total scattering as a tool to fully characterize their materials structurally.

[1] Proffen Th., Billinge S.J.L., Egami T., Louca D., *Z. Krist.*, 2003, **218**, 132-143.

Keywords: pair distribution function, powder diffraction, disorder

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Structure Determination from a Quantum Mechanical Formulation in Momentum Space

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The proposed method aims at the crystal structure determination by using the theoretical background of quantum mechanics. This is achieved through the quantum mechanical formulation in momentum space by means of the Fourier Transform (FT) of the usual Schrödinger equation in direct space. The key step is the identification of the FT of the potential function V(r) with the expression $E(H)/H^2$ where E(H) is the normalized structure factor. An algorithm has been developed for practical implementation of this new method for direct phasing of x-ray data [1]. In this algorithm a new criterion, based on the crystallographic symmetry, has been introduced. The idea consists of testing the phase calculation, extension and refinement, by deliberately sacrificing the space group symmetry information and using its gradual re-appearance as a criterion of correctness [2].

New theoretical developments relevant to the convergence of this algorithm in different cases have been formulated. An upgraded algorithm for macromolecules has also been developed and tested in phase extension and refinement with promising results.

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Keywords: algorithm, phase problem, quantum mechanics

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Molecular Replacement via Normal Mode Analysis and Homology Modelling on the Web

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Molecular replacement (MR) is the method of choice for X-ray crystallography structure determination when structural homologues are available in the Protein Data Bank (PDB). However, the success rate of MR decreases sharply when the sequence similarity between template and target proteins drops below 35% identical residues. Another reason for MR failure are conformational differences between target and template, induced for example by ligand binding or different crystallogenic conditions. It has been found that screening for MR solutions with a large number of different homology models or models that are perturbed in the direction of one or two low frequency normal modes may still produce a suitable solution where the original template failed [1-3]. Here we present the web tools *elNémo* [2] and *CaspR*, [3] that implement such strategies in an automated manner. *elNémo* is accessible at http://igs-server.cnrs-mrs.fr/elnemo/, *CaspR* at http://igs-server.cnrs-mrs.fr/Caspr/.

[1] Suhre K., Sanejouand Y.H., *Acta Cryst. D*, 2004, **60**, 796-799. [2] Suhre K., Sanejouand Y.H., *Nucleic Acids Research*, 2004, **32**, W610-W614. [3] Claude J.B., Suhre K., Notredame C., Claverie J.M., Abergel C., *Nucleic Acids Research*, 2004, **32**, W606-W609.

Keywords: crystallography, phasing, template perturbation

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HUNTER: A Package of small Tools to Manipulate FOX

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The HUNTER package consists of a number of small programs for handling the .xml-input files of the simulated annealing program FOX [1]. By use of simple scripts HUNTER allows to set up genetic algorithm techniques [2] for crystal structure determination. In this context the simulated annealing serves as Lamarckian [3] component of the evolution process. The package can also be used to explore possible neighboring minima to a current local minimum of the structure cost function. This is accomplished by sophisticated application of reorientations of randomly chosen molecules or building groups.

The individual tools currently available are: MATE, which produces a new file from two parent files by choosing randomly position, orientation and content of each molecule (or atom) from one of the parent files. ORIENTATION locates the main axes of a random molecule and applies a randomly chosen reorientation of twofold symmetry. DEFORMATION applies a rotation to a randomly chosen group of atoms using a user-defined list of groups and rotation axes. FITNESS extracts the cost function from the files written by FOX and stores it into the .xml-file, using either the current cost minimum or an exponentially extrapolated value. SELECTION sorts structure files by their fitness. Additional programs will complement the package.

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Monte Carlo Search with Many CPUs: Application to 6 dim. Molecular Replacement

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Monte Carlo search is the simple method that the solution is searched by iterating the many trials for given random parameters. By randomness, every trial is assured that the searching region is different from that of the rest. And, all the trials are completely independent, that is, it is not necessary to wait the result of the other trials at all. With this method, we are able to flexibly use the whole power of many CPUs without losing its efficiency.

It is able to use a lot of computers by a modern internet technology. If it is a calculation to which the Monte Carlo search method can apply, it is possible to achieve it comparatively easily even by an enormous calculation.

We applied this method to search the six dimensional parameters at once for rotation and translation of the molecular replacement method. An initial model was obtained for the unknown protein molecular structure: SHPS-1. It was hard to find solutions by traditional way, because the peaks of the correct solutions for rotation functions are low as about two sigma level of random noises.

Space group of the crystal is P622 and the size of the cell is long as about 100 angstrom. There are two molecules in asymmetric unit. To find the correct solutions, it took about 10 days by using at most 30 various kinds of CPUs "non-exclusively".