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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.

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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