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In-house S-SAD and Se-SAD phasing with Cr Kα Radiation

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With the recent advances in X-ray technology, chromium radiation (2.29 Å) has become viable for in-house data collection and phasing. Chromium radiation appears to be well suited for measuring anomalous signals from weak anomalous scatterers such as sulfur, selenium, and calcium. In particular, the anomalous scattering strengths of sulphur (f''=1.14 e) and selenium (f''=2.28 e), the most common intrinsic and derivatized anomalous scatterers, are doubled when using Cr Kα radiation compared to those for Cu Kα radiation (1.54 Å, S: f''=0.56 e, Se: f''=1.14 e). Due to the difficulties in utilizing the K absorption edge of sulphur at 5.02 Å, limited access to X-ray beamlines and related transportation issues, it has become evident that Cr Kα radiation is very good choice for collecting sulphur anomalous data and a may be an alternative to a synchrotron beamline for collecting selenium anomalous data. This report shows examples of S-SAD and Se-SAD phasing from data collected on an in-house Cr source. To break the phase ambiguity inherent with SAD, the direct-methods procedure implemented in the program OASIS-2004 was employed. Phase discrimination using the product of the Sim and Cochran distributions leads to better initial phases and, consequently, to better electron-density maps.

Keywords: Cr radiation, S-SAD phasing, Se-SAD phasing

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Local Structure of the Metal-oxygen Bonds in ZrO_2-CeO_2 Nanopowders

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Zirconia-ceria solid solutions are being widely employed in current automotive three-way catalysts as oxygen storage promoters due to their excellent catalytic properties. These properties have been proposed to be related to the local structure of the metal-oxygen bonds in these materials.

In this work, the local structure around Zr and Ce atoms in compositionally homogeneous, nanocrystalline ZrO_2-CeO_2 powders was investigated by the technique named extended X-ray absorption fine structure (EXAFS). These experiments were carried out at the D04B XAS-1 beamline of LNLS (Brazilian Light Facility). ZrO_2-15, 40, 50, 60, 65, 70, 80 and 90 mol% CeO_2 nanopowders were synthesized by a pH-controlled nitrate-glycine gel-combustion process. While Ce-O coordination sphere has a cubic symmetry in the whole composition range, the Zr-O coordination shows a tetragonal-to-cubic symmetry change as the CeO_2 content is varied. This transition was found to be at (85±5) mol% of CeO_2, in agreement with a previous synchrotron XRD study. For compositions with a tetragonal crystal structure, a 5+2 model was found for the Zr-O bond, while the coordination number was 5 for the cubic solid solutions. The coordination number of Ce was close to 8 for all the compositions. Finally, we also demonstrated that the local structure determined by EXAFS agrees well with the long-range structure deduced by XRD.

Keywords: EXAFS, zirconia-ceria, nanocrystalline materials

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X-ray Scattering Measurements of Liquid Water

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Despite decades of research using both x-rays and neutrons there still exist disagreements concerning the radial distribution function of liquid water at room temperature. These disagreements largely arise due to the challenges of extracting the elastic scattering from the significant inelastic background as well as debate about the correct independent scattering factor[1]. Recent research using spectroscopic techniques[2], in combination with modeling, suggest that liquid water contains significant numbers of broken hydrogen bonds and is better described as rings of strongly H-bonded chains rather than the tetrahedral structure observed in ice-Ih.

Using the increased brightness of the new SPEAR3 synchrotron source we have performed measurements of liquid water both at 23 C and at 5 C. These measurements take advantage of a second recent development, a variable-focus diffracted beam analyzer which provides energy dispersion of the scattered x-rays onto a position-sensitive detector[3]. We have also developed a horizontal water jet with a sub-millimeter diameter that eliminates scattering from a container and precludes radiation-induced bubble formation. The high brightness of the source has also allowed us to measure scattering from water out to 19.7 Å^{-1}, which simplifies the normalization of the data and increases the r-space resolution.

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Keywords: liquid, synchrotron X-rays, water structure

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Investigating the Molecular Formation Properties of 4-(2-Oxobenzothiazolin-3-YL) Butanoic Acid Using PM3, AM1, MNDO Methods

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In this study in order to the geometry optimization of the 4-(2-oxobenzothiazolin-3-yl) butanoic acid crystal, which is used for forming analgesic and antienflamatuar medicine and of which crystal structure was determined using x-ray diffraction method, PM3 (Parametric Model 3), AM1 (Austin Model 1) and MNDO (Modified Neglect of Diatomic Overlap) semi emprical molecular orbital methods found in the HyperChem program were used. By the geometry optimization geometric parameters of the molecules having the minimum energy were found. These values which were theoretically obtained were compared with the emprical values obtained by x-ray diffraction method. These results showed that for the $C_{11}H_{11}NO_3S$ crystal, in bond lengths AM1 method and in bond angles MNDO method was found to be consistent with the emprical x-ray diffraction data. By geometry optimization using PM3, AM1 and MNDO methods relevant energy values of the molecular structure were calculated.

Keywords: semi emprical molecular orbital methods, PM3 AM1 MNDO, oxobenzothiazolin butanoic acid

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Crystal Structure Prediction of Nitrobenzene Derivatives

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The main methods of crystal structure prediction base on searching crystal structures corresponding to global minimum in the lattice energy. An intermolecular force field is required to calculate intermolecular energy, which consists of coulombic, polarisation, dispersion and repulsion terms [1]. The lattice energy is calculated as a sum of interactions using the atom-atom potential approach [2].

In this work prediction of crystal structures for selected single-substituted nitrobenzene derivatives was carried out using Monte

Carlo simulated annealing [3, 4]. The prediction has been performed for nitrobenzenes with a following second substituent: hydroxyl, amino, nitro or methyl group or chlor, brom, iod atom. The calculations have been carried out for a standard choice of space groups. The *Polymorph Predictor*, module of *Cerius*^{2} program was used [5].

The predicted structures are compared with our experimental results or with crystal structures retrieved from CSD [6]. The polymorph structures are analysed in terms of molecular interactions that influence nucleation, crystallisation and stability of polymorphs.

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Keywords: crystal structure prediction, nitrobenzene derivatives, polymorphs

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Structural Modeling of Sterol Carrier Protein-2 from Plants

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Sterol carrier protein-2 (SCP-2) is a small, cytoplasmic protein that was originally described as a cholesterol transfer protein. Later it has been shown that SCP-2 binds a variety of lipids but its actual biological function remains unclear. SCP-2-like proteins have been found in various organisms from vertebrates to bacteria, and recently also in plants. In order to characterize SCP-2 from the plants *Arabidopsis thaliana* (AtSCP-2) and *Euphorbia lagascae* (ElSCP-2) we have built structural models of the two proteins in apo and ligand-bound conformation [1] based on the known crystal structures of rabbit SCP-2 [2], the SCP-2 like domain of human D-bifunctional enzyme [3] and the yellow fever mosquito SCP-2 [4]. Although the sequence identity between AtSCP-2 and ElSCP-2 is high (67.5%), they preferably bind different lipids. We have examined the ligand-binding cavities of the AtSCP-2 and ElSCP-2 structural models in apo and ligand-bound conformations in order to find out structural properties, which would explain the differences in ligand binding.

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Keywords: protein modelling, protein-lipid complexes, protein structure comparison

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Powder Diffraction and Crystal Structure Prediction: A Two-Way Relationship?

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The most common complementary use of theoretical and experimental methods is structural rationalization from crystal structure prediction and X-ray powder diffraction techniques[1]. This aids both the rationalization of crystal structures generated in a prediction, and the characterization of solids from powder data that precludes indexing or structure solution.

Powder data from the prediction is often compared visually or purely on a fingerprinting basis with the experimental, and there are only a few cases of organic materials in which the predicted structures have been used as a starting point for Rietveld refinement[1,2]. One possible reason for this is that even though the variation in lattice parameters between the experimental and calculated structures is relatively small, the difference in the respective patterns often makes automated quantitative comparison difficult and attempts at refinement unsuccessful. As prediction calculations search for the energetically optimal packing at 0 K, use of low temperature powder data would enable a more meaningful comparison of the two profiles.

We will present our results from the study of several organic materials at low temperatures and their subsequent comparison to the predicted structures using a number of quantitative guides.

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Keywords: structure prediction, powder diffraction, low-temperature structures

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Can a Computational Search Predict Complications in Single Crystal Growth?

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To study the variation in possible crystal packing of structures with respect to the relative positions of functional groups, five dichloronitrobenzenes have been studied both experimentally and computationally. A manual polymorph screen has been carried out for each compound using a variety of solvent methods and sublimation to grow crystals.

The experimental search found considerable difficulty in growing crystals suitable for single crystal X-ray diffraction with many exhibiting multiple domains and plate-like morphologies. Redeterminations have been carried out at low temperature but have not shown a marked improvement on the published refinements.

The computational searches found the known structures as the global minimum in a few cases. For each compound, though, there were many hypothetical structures within a small energy range of that minimum with many of these being variants on the experimentally observed sheet structures.

The predicted low energy structures illustrate variations in the sheet packing which could be indicative of, for example, slippage between the layers or disorder in the stacking. A possible link between this phenomenon and the problems associated with crystal growth and structure determination will be discussed.

Figure 1. Two variations on the stacking of sheets related by slippage along c.

Keywords: prediction, crystal growth, organic compounds

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Binding Pocket Shape Analysis for Protein Function Prediction

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We present a novel method for the comparison of protein binding pockets and ligands. An increasing number of protein structures are being determined for which no biochemical characterisation is available. The analysis of protein structure and function assignment is becoming an unexpected challenge and major bottleneck towards the goal of well-annotated genomes. As shape plays a crucial role in biomolecular recognition and function, shape techniques are likely to be of prime importance for understanding protein structure-function relationships.

A highly efficient shape comparison technique based on a real spherical harmonics expansion is presented for protein function prediction from structure. Our approach identifies the active site by a geometrical surface analysis method combined with an evolutionary trace technique. The binding pocket is then placed into a standard frame of reference using a heuristic that employs the first three moments of the spatial extent of the shape to find the orientation. The method uses the coefficients of a real spherical harmonics expansion to describe the shape of a protein's binding pocket. Shape similarity is computed as the Euclidean distance in coefficient space and is