

P.02.04.9*Acta Cryst.* (2005). A61, C155**In-house S-SAD and Se-SAD phasing with Cr K α 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 K α radiation compared to those for Cu K α 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 K α 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

P.02.07.1*Acta Cryst.* (2005). A61, C155**Local Structure of the Metal-oxygen Bonds in ZrO₂-CeO₂ 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 ZrO₂-CeO₂ 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). ZrO₂-15, 40, 50, 60, 65, 70, 80 and 90 mol% CeO₂ 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 CeO₂ content is varied. This transition was found to be at (85±5) mol% of CeO₂, 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

P.02.09.1*Acta Cryst.* (2005). A61, C155**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.

[1] Head-Gordon T., Hura G., *Chem. Rev.*, 2002, **102**, 2651. [2] Wernet Ph., et al., *Science*, 2004, **304**, 995. [3] Ishii H., Johnson B., Brennan S., *AIP Proc.*, 2004, **CP708**, 969.

Keywords: liquid, synchrotron X-rays, water structure

P.02.10.1*Acta Cryst.* (2005). A61, C155**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 empirical 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 empirical values obtained by x-ray diffraction method. These results showed that for the C₁₁H₁₁NO₃S crystal, in bond lengths AM1 method and in bond angles MNDO method was found to be consistent with the empirical x-ray diffraction data. By geometry optimization using PM3, AM1 and MNDO methods relevant energy values of the molecular structure were calculated.

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

P.02.10.2*Acta Cryst.* (2005). A61, C155-C156**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