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Protein Powder Diffraction: why Bother?
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This discussion of powder protein powder diffraction looks at what is currently achieved with small molecule powder data, and low resolution single crystal protein crystallography and asks what techniques from these areas can be translated into the world of protein powders:

1. Qualitative PXRD: Pattern matching - can we classify and match patterns using the full pattern profile and not just the peaks as in the SNAP-1D [1] and PolySNAP [2] computer software, and can this information be used as an aid to crystallization?

2. Quantitative PXRD: Can we identify components in powders in a quantitative mode using full powder profiles as used in the SNAP-1D/PolySNAP software?

3. Unit Cells with protein PXRD: Can we index poor quality patterns? Can using the full profile help? What about brute force methods using grid computing techniques?

4. Single crystal low resolution protein diffraction can give the molecular envelope; can this be achieved with powder data?

All these issues will be discussed with examples where possible.

[1] Gilmore C.J., Barr G., Paisley J., *J. Appl. Cryst*.,2004, **37**, 231-242. [2] Barr G., Dong W., Gilmore C.J., *J. Appl. Cryst*, 2004, **37**, 243-252. [3] Von Dreele R.B., *Acta Cryst*.,2005, **D61**, 22-32. [4] Gilmore C.J., Wright J., Fitch A., *Transactions of the Amer. Cryst. Assocn*.,2002, **37**, 113-123.
Keywords: protein powder diffraction, pattern matching, envelopes

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Development of Powder Diffraction Methods for Macromolecular Crystallography
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Modern developments of the powder diffraction technique have allowed the investigation of systems with large unit cells like proteins [1]. Powder diffraction measurements can give a range of complementary information beyond that which can be obtained from a single crystal. For example, the peak shapes depend on the microstructure of the material, accurate unit cell parameters can easily be determined, and the sample generally survives under more varied or extreme conditions. In the present work, we aim in establishing the full potential of powder diffraction technique in the research of macromolecular systems. Specific examples that will be presented refer to: **(a)** structural modifications of the hen and turkey egg-white lysozymes (HEWL & TEWL) with crystallisation conditions and temperature [2-3]. **(b)** in-situ observation of crystal growth of HEWL **(c)** the development of a successful cryoprotection protocol for powder diffraction, with an almost complete suppression of radiation damage in pancreatic porcine elastase & **(d)** heavy atom derivatives of HEWL and elastase. A key point of the current work is to carefully assess various instrumental configurations and experimental strategies for the recording of protein powder data, either with high-resolution scanning instruments or with area detectors.

[1] Von Dreele R. B.. *Acta Cryst*., 2005, D**61**, 22-32. [2] a) Margiolaki I., et al., *Acta Cryst*., 2005, D**61**, *in press*; b) see also: *ESRF Scientific Highlights*, 2004, 24. [3] Basso S. et al., *in preparation.*
Keywords: proteins, powder diffraction, synchrotron radiation

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Protein Measurements on a Laboratory Powder Diffractometer
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Han egg white lysozyme has been dissolved into 0.1 M sodium acetate-acetic buffer at pH 4.8 to a final concentration of 60mg/ml. This has been mixed with a second solution of 8% sodium chloride in the same 0.1 M puffer and kept at 22^{o}C. Tetragonal crystals of various sizes smaller than 150 μm were obtained after 46 hours. These crystals were removed from the mother-liquor using a pipette, and placed into a 0,5mm glass capillary. The crystals were manually compacted and the remaining mother-liquor has been removed.

The sample was measured on an X'Pert PRO Multi-Purpose Diffractometer (MPD), configured in transmission geometry with a focusing mirror on the incident beam side and an X'Celerator detector on the diffracted beam side. The sample was measured at room temperature with a scan range of 1-45^{o} (2Θ), a step size of 0.004^{o}.

The raw data were indexed by the DICVOL program, integrated into the HighScore Plus software suite, giving a tetragonal cell with parameters: a= 79.09Å, c=37.94Å. Le Bail and Pawley fit , as well as structural refinement showed good agreement and results will be discussed.

Keywords: proteins, powder X-ray diffraction, X-ray optics

MS40 COMPUTATIONAL CRYSTALLOGRAPHY APPLIED TO EXTREME CONDITIONS

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Structural Paths for the High-Pressure Phase Transitions of AgI
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First-principles calculations with Density-Functional-Theory (DFT) Hamiltonian and localized basis set (CRYSTAL code [1]) were performed on AgI, showing that it transforms from the cubic zinc blende to the tetragonal anti-litharge structure at 1.2 GPa, and then to cubic rocksalt at 1.6 GPa, in agreement with experiment [2]. For both reconstructive phase transitions, a monoclinic *Pm* pathway was considered and tested by computing the enthalpy profile (with full structural optimization) vs. the order parameter, according to a previously presented method [3]. On the basis of the activation enthalpies obtained, a bifurcated three-step kinetic mechanism is proposed. One step relates the anti-litharge structure to a metastable orthorhombic *Bmm2* phase which appears along the transformation path. Then two alternative steps follow, transforming the intermediate phase into either zinc blende or rocksalt. The enthalpy curve along the *Pm* pathway shows two bottle-neck states bracketing the metastable phase, with a predicted maximum activation enthalpy of 0.088 eV. The mechanism is characterized by changes of the Ag coordination number from 4 (zinc blende and anti-litharge) to 5 (*Bmm2* phase) to 6 (rocksalt), which account for the dependence of the unit-cell volume on the order parameter.

[1] Saunders V.R., et al., *CRYSTAL03: User's manual*, 2003, University of Torino, Italy, and CLRC Daresbury Laboratory, UK. [2] Keen D.A., Hull S., *J. Phys.: Condens. Matter*, 1993, **5**, 23. [3] Catti M., *Phys. Rev. Lett.*, 2001, **87**, 035504.
Keywords: high-pressure phase transformations, ab initio calculations, phase transition kinetics

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The Fifth Element in the Periodic Table, Boron: do we know the Ground State Structure?
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Boron exhibits the most complex structure of all elemental solids, with more than 300 atoms per unit cell arranged in interconnecting