

Modern developments of the powder diffraction technique have allowed the investigation of systems with large unit cells such as proteins [1]. Protein powder specimens consist of a large number of randomly oriented diffracting micro-crystals. These micro-crystals are usually formed rapidly by batch crystallization. Frequently, the resolution and quality of the data are limited mainly by rapid deterioration of the protein crystal structure during exposure to the intense synchrotron X-ray beam. In a typical single crystal diffraction experiment radiation damage can be minimized by collecting diffraction data under cryocooled conditions (typically 100K) which requires the addition of a cryoprotecting agent to the protein sample to prevent freezing of the mother liquor. In this study, we succeeded in obtaining various cryocooled samples of human insulin at 100K avoiding ice formation. Powder diffraction data were collected at both room temperature and under cryocooled conditions (ID31, ESRF, Grenoble, France). As expected both the cryoprotectant and the sample container have a remarkable impact on the data quality. Significant variation of the lattice parameters and peak widths with the type and concentration of cryoprotecting agent has already been observed and will be presented for the case of insulin. Preliminary data interpretation correlating these changes with the structural and microstructural characteristics of the systems under study will be shown.

[1] Margiolaki, I. & Wight, J. P. *Acta Cryst.*, **2008**. A64, 169-180

**Keywords:** proteins; powder diffraction; cryocooling

#### FA5-MS01-P12

**Two Quinoline Zirconium Phosphate Structures Solved Using Powder Charge Flipping.** Dubravka Sisak<sup>a</sup>, Christian Baerlocher<sup>a</sup>, Lynne McCusker<sup>a</sup>, Lei Liu<sup>b</sup>, Jinxiang Dong<sup>b</sup>. <sup>a</sup>*ETH Zurich, Laboratory of crystallography, Zurich, Switzerland.* <sup>b</sup>*Taiyuan University of Technology, Taiyuan, Shanxi, China.*  
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Two novel zirconium phosphate compounds with the chemical formulae  $[(C_9H_8N)_4(H_2O)_4][Zr_8P_{12}O_{40}(OH)_8F_8]$  (ZrPOF-Q1) and  $[(C_9H_8N)_2][Zr_2P_2O_4(OH)_2F_4]$  (ZrPOF-Q2) were synthesized hydrothermally in the HF-ZrO<sub>2</sub>-P<sub>2</sub>O<sub>5</sub>-quinoline-H<sub>2</sub>O system. Because they could only be obtained in polycrystalline form, their structures had to be analyzed using powder diffraction techniques. Both diffraction patterns (ZrPOF-Q1 : synchrotron, SNBL at ESRF; ZrPOF-Q2 : laboratory, Stoe) could be indexed with triclinic unit cells, but the dimensions and volumes are significantly different (ZrPOF-Q1: ;  $a = 10.7567\text{\AA}$ ,  $b = 13.8503\text{\AA}$ ,  $c = 14.8994\text{\AA}$ ,  $\alpha = 109.6^\circ$ ,  $\beta = 101.1^\circ$ ,  $\gamma = 100.5^\circ$ ;  $V = 1979\text{\AA}^3$ , and ZrPOF-Q2: ;  $a = 7.7058\text{\AA}$ ,  $b = 12.3546\text{\AA}$ ,  $c = 6.5851\text{\AA}$ ,  $\alpha = 97.0^\circ$ ,  $\beta = 89.7^\circ$ ,  $\gamma = 101.9^\circ$ ;  $V = 610\text{\AA}^3$ ). In both cases, reflection intensities were extracted from the powder diffraction pattern using the Le Bail method and then used as input to the powder charge-flipping (pCF) algorithm [1] in the program Superflip [2]. In this implementation, the original single-crystal charge-flipping algorithm of Oszlányi and Sütő [3,4] has been modified to include a second type

of perturbation of the electron density map. This is based on a histogram matching algorithm and is performed prior to a repartitioning of the intensities of reflections that overlap in the powder pattern. The histogram used simply reflects the chemical composition per unit cell. The electron density maps obtained from Superflip showed localized electron densities in the form of tetrahedra and octahedra, which were interpreted as PO<sub>4</sub> and Zr(O,F)<sub>6</sub> groups. Some residual, less well-localized electron density, assumed to be due to the quinoline species, was also observed, but was not used in constructing the initial model for Rietveld refinement. Instead, the positions of the quinoline C and N atoms were derived from a series of difference Fourier maps. The structure of ZrPOF-Q1 (60 non-H atoms, 3909 of 4522 reflections overlapping,  $R_F = 0.053$ ,  $R_{wp} = 0.144$ ) consists of zirconium phosphate layers with quinolinium ions and water molecules in between. The layers are unusual in that they have isolated ZrO<sub>2</sub>F<sub>4</sub> octahedra anchored on their surfaces that protrude into the interlayer space. The ZrPOF-Q2 structure (19 non-H atoms, 928 of 1265 reflections overlapping,  $R_F = 0.059$ ,  $R_{wp} = 0.162$ ) consists of zirconium phosphate chains with an unusual Zr:P ratio of 1.0, interspersed with quinolinium ions. The charge-flipping algorithm allowed a straightforward structure solution in both cases, although the degree of reflection overlap is considerable. The effect of the various charge-flipping parameters on the structure solution will be presented.

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#### FA5-MS01-P13

**High Throughput Phase Diagram Mapping of Urate Oxidase via Powder Diffraction.** Ines Collings<sup>a</sup>, Sotonye Dagogo<sup>a</sup>, Yves Watier<sup>a</sup>, Irene Margiolaki<sup>a</sup>, Andrew N. Fitch<sup>a</sup>, Jonathan P. Wright<sup>a</sup>, Marion Giffard<sup>b</sup>, Françoise Bonnetet<sup>b</sup>, Richard Kahn<sup>c</sup>. <sup>a</sup>*European Synchrotron Radiation Facility, Grenoble, France.* <sup>b</sup>*Centre Interdisciplinaire de Nanoscience de Marseille, France.* <sup>c</sup>*Institut de Biologie Structurale, Grenoble, France.*  
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Modern developments of the powder diffraction technique have allowed the investigation of systems with large unit cells such as proteins [1]. Polycrystalline protein precipitates are frequently obtained under a variety of crystallization conditions and thus powder methods can be employed for structural characterization of small proteins when single crystals are unavailable. The recombinant urate oxidase from *Aspergillus flavus* (Uox) is a protein used to reduce toxic uric acid accumulation and also for the treatment of hyperuricaemia which occurs during chemotherapy. In this study, we investigate the effects of pH, salt and polyethylene