

MAXIMIZING MACROMOLECULE CRYSTAL SIZE FOR NEUTRON DIFFRACTION EXPERIMENTSR. Kephart¹, R. A. Judge¹, R. Leardi², D.A.A. Myles³, E.H. Snell⁴, M.J. van der Woerd⁴

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A challenge in neutron diffraction experiments is growing large (greater than 1 cubic millimeter) macromolecule crystals. In taking up this challenge we have used statistical experiment design techniques to quickly identify crystallization conditions under which the largest crystals grow. These techniques provide the maximum information for minimal experimental effort, allowing optimal screening of crystallization variables in a simple experimental matrix, using the minimum amount of sample. Analysis of the results quickly tells the investigator what conditions are the most important for the crystallization. These can then be used to maximize the crystallization results in terms of reducing crystal numbers and providing large crystals of suitable habit. We have used these techniques to grow large crystals of glucose isomerase. Glucose isomerase is an industrial enzyme used extensively in the food industry for the conversion of glucose to fructose. The aim of this study is the elucidation of the enzymatic mechanism at the molecular level. The accurate determination of hydrogen positions, which is critical for this, is a requirement that neutron diffraction is uniquely suited for. Preliminary neutron diffraction experiments with these crystals conducted at the Institute Laue-Langevin (Grenoble, France) reveal diffraction to beyond 2.5 Å. Macromolecular crystal growth is a process involving many parameters, and statistical experimental design is naturally suited to this field. These techniques are sample independent and provide an experimental strategy to maximize crystal volume and habit for neutron diffraction studies.

Keywords: NEUTRON DIFFRACTION MACROMOLECULE CRYSTAL SIZE**X-RAY RESONANCE MULTI-BEAM DIFFRACTION: EXPERIMENTS AND THE PERTURBATIVE BETHE APPROXIMATION**Y.P. Stetsko^{1,2}, G.Y. Lin¹, Y.S. Huang¹, C.H. Chao¹, Y.R. Lee¹, and S.-L. Chang^{1,2}

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X-ray diffraction/scattering for atoms in excited states or under resonance conditions has currently been used to probe atomic and electronic structures of matter. In the case of multiple diffraction, at atomic absorption edges the effects of anomalous dispersion on multi-wave x-ray interference in crystals are interesting and important topics to investigate. In this paper, highly phase-sensitive profiles of the diffraction intensity ratios of two inversion-symmetry-related multiple diffractions near/at absorption edges (about 30 eV), exhibiting strong asymmetric characteristics, compared with those far from the edges, are experimentally and theoretically observed. The changing of asymmetry of these ratios results from the anomalous dispersion corrections in the structure factors due to the significant change of the resonance phase at the absorption edge. The excellent agreement between the experimental and theoretical results is obtained. The proposed resonance perturbation Bethe (RPB) approach allows for the determination of the phase shifts of x-ray reflection under resonance conditions as a function of photon energy. This provides a highly sensitive way for experimental investigation of the spectral distribution of reflection phase shift due to the resonance.

Keywords: RESONANCE, MULTI-BEAM, DIFFRACTION.**A CRYSTAL STRUCTURE FOR THE SOUZALITE/GORMANITE SERIES FROM SYNCHROTRON POWDER DIFFRACTION DATA**A. Le Bail¹, P. W. Stephens², F. Hubert³

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In absence of suitable single crystal due to polysynthetic twinning, the crystal structure of a specimen in the souzalite/gormanite series (Fe,Mg)₃(Al,Fe)₄(PO₄)₄(OH)₆(2H₂O) is determined *ab initio* from synchrotron powder diffraction data. The crystals belong to space group P-1, the cell is different from previously reported [1-3] with a = 7.2217(2), b = 11.7812(3), c = 5.1172(1) Å; α = 90.160(2), β = 109.942(1), γ = 81.324(2)°; V = 404.03(2) Å³; Z = 1. Direct and Patterson methods failed, so that the solution was obtained by using the ESPOIR program [4], applying Monte Carlo moves to a random starting model. The structure [5] consists of infinite chains of alternating [FeO₆] and [AlO₆] octahedra sharing faces and/or edges, interconnected by corners with trimers of corner-sharing [AlO₆] octahedra, forming octahedral layers linked by [PO₄] groups. Large similarities exist with the structures of dufenite [2] and burangaite [6], as predicted in 1970 by P.B. Moore.

[1] W.T. Pecora & J.J. Fahey, *Am. Mineral.* **34** (1949) 83-93.[2] P. B. Moore, *Am. Mineral.* **55** (1970) 135-169.[3] B.D. Sturman, J.A. Mandarino, M.E. Mrose & P.J. Dunn, *Can Mineral.* **19** (1981) 381-387.[4] A. Le Bail, ESPOIR program, <http://www.cristal.org/sdpd/espoir/>[5] A. Le Bail, P.W. Stephens & F. Hubert, *J. Eur. Mineral.*, submitted.[6] J.B. Selway, M.A. Cooper & F.C. Hawthorne, *Can. Mineral.* **35** (1997) 1515-1522.**Keywords: SOUZALITE, CRYSTAL STRUCTURE, POWDER DIFFRACTION****PHASE DETERMINATION FROM OVERLAPPED THREE-BEAM DIFFRACTION PROFILES OF MACROMOLECULAR CRYSTALS BY X-RAY STEREOSCOPIC MULTI-BEAM IMAGING**S.-L. Chang^{1,2}, C. H. Chao^{1,2}, C. Y. Hung¹, Y. S. Huang¹, C. H. Ching¹, Y. R. Lee¹, S. C. Lai¹, Y. P. Stetsko^{1,3}, and H. Yuan⁴

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X-ray multiple diffraction has been proved to be able to provide phase information for macromolecular crystals on qualitative and quantitative bases. However, overlapping of multi-beam diffraction profiles is unavoidable in multiple diffraction experiments for macromolecular crystals. In this paper, we present a technique of extracting phase information from overlapped profiles by using a strong primary reflection in the stereoscopic multi-beam imaging method. A profile of two overlapped three-beam diffractions can still provide the phase information of the dominant triplet structure invariant. A detailed analysis of multiple diffraction data collected by the stereoscopic multi-beam imaging method from a tetragonal lysozyme crystal, together with calculations based on the dynamical theory are reported.

Keywords: PHASE, MULTI-BEAM, DIFFRACTION