MAD PHASING OF HIGHLY MEROHEDRALLY TWINNED CRYSTALS OF INTERLEUKIN 1-β

M.G. Rudolph[1] M.S. Kelker[2] P.A. Jennings[3] I.A. Wilson[2]
[1]Department of Molecular Structural Biology, Institute For Microbiology And Genetics, Georg-August University, 37077 GOttingen, Germany [2]Department of Molecular Biology And The Skaggs Institute For Chemical Biology, The Scripps Research Institute, La Jolla CA 92037, USA [3]Department of Chemistry, University of California San Diego, La Jolla CA 92307, USA

The crystal structure of a double mutant of interleukin 1-β (F42W/W120F), a cytokine secreted by macrophages, was determined by multi-wavelength anomalous dispersion (MAD) using data from highly twinned, selenomethionine-modified crystals. The space group is $P4_3$ with unit cell dimensions of a = b = 53.9 Å and c = 77.4 Å. Self-rotation function analysis, cumulative intensity distributions, analysis of second moments, and reduced $|E^2-1|$ values revealed the presence of merohedral twinning in crystals of both the native (twinning fraction α = 0.36) and SeMet (α = 0.4) forms. The structure was determined to 1.8 Å using the untreated, twinned intensity data (final overall figure of merit FOM = 0.53). The initial electron density maps were of sufficient quality for partial automated chain tracing. The structure was refined to 1.54 Å against twinned native data using SHELXL97. The results show that sufficient anomalous signal may still be extracted from twinned data in favorable cases for structure determination by MAD.

Keywords: MAD, TWINNING, INTERLEUKIN 1-β

HOW CRITICAL ARE ABSORPTION CORRECTIONS FOR PHASING BRAGG REFLECTIONS WITH THE MAD METHOD AT WAVELENGTHS BETWEEN 3 AND 6 Å?

H.B. Stuhrmann[1,4] P. Carpentier[1] P. Boesecke[2] J.-M. Bois[3] M.-L. Chesne[1] R. Kahn[1] S. Kozielski-Stuhrmann[2] J. Vicat[1]
[1]Institut de Biologie Structurale Jean-Pierre Ebel CEA-CNRS-UJF (IBS), Grenoble Grenoble, France [2]European Synchrotron Radiation Facility (ESRF), Grenoble, France [3]European Molecular Biology Laboratory (EMBL), Grenoble, France [4]GKSS Forschungszentrum, Geesthacht, Germany

Recent experiments carried out at the beamline ID1 of ESRF have shown that a cold helium atmosphere provides optimal conditions for protein crystallography at wavelengths up to 6 Å. This observation opens the way to the use of anomalous dispersion at the K-edge of light elements, like phosphorus, sulfur, chlorine and calcium at ID1. First results of anomalous diffraction from a uranium derivative of lysozyme show a variation of the intensity that is dominated by the absorption from uranium at its MV edge. This is rather different from the observation near the sulfur K edge in trypsin where the average variation of the structure factor in the near edge region is comparable to that due to absorption. The change of the penetration depth near an absorption edge is largest when the size of the protein crystal is close to the penetration depth of the radiation. For a protein crystal of 0.1 mm size this is the case at a wavelength λ = 3 Å. At longer wavelengths the change of absorption due to anomalous dispersion decreases, as the diffracted intensity preferentially emerges from the surface of the crystal. This is of practical importance for absorption corrections at longer X-ray wavelengths, as these becoming smaller are less likely to degrade phasing.
Recent X-ray diffraction experiments with lysozyme doped with uranyl ions at wavelengths near the uranium MV edge at 3.5 Å and earlier X-ray diffraction experiments from trypsin at wavelengths near the sulfur K edge at 5.0 Å confirm the rule.

Keywords: **MAD SOFT X-RAYS ABSORPTION CORRECTION**

POWDER QUANTITATIVE ANALYSIS WITHOUT RIETVELD USING SNAP-1D

G. Barr C. Gilmore J. Paisley
University of Glasgow Department of Chemistry Room A5-22a Joseph Black Building, University of Glasgow GLASGOW SCOTLAND G12 8QQ UK

SNAP-1D is a user-friendly PC-based program for both quantitative and non-quantitative pattern matching. SNAP utilizes the full diffraction profiles of patterns, unlike other programs which usually represent patterns as a list of d-spacings and corresponding peak intensities, thus discarding a great deal of useful information. Powder patterns can be imported from a variety of file formats into a proprietary SNAP database. Patterns can be processed to remove background artefacts and to reduce noise using wavelets. An automatic peak-picking algorithm may also be employed.
The program then performs two main functions: (1) The pattern-matching module answers the question: To what patterns in an existing database is a new unknown pattern most similar? User-controlled combinations of both non-parametric and parametric statistical tests are utilised to answer this. Matching results can be displayed in numerical or graphical form. This process can be used for comparison of unknown patterns, or in sample quality control, for example. (2) In quantitative mode, given a mixture pattern known to consist of a combination of other patterns already in the program database, SNAP-1D can produce a list of suggested component phases and their relative weight fractions in the mixture. This is done using a variation of a generalised inverse linear least squares technique that is much more straightforward to use than Rietveld refinement-based analysis systems. Identified components can be examined in a variety of different ways. The program can also be used with non-powder one-dimensional data types, for example solid state IR or ROA spectra.

Keywords: POWDER DIFFRACTION RIETVELD REFINEMENT COMPUTER SOFTWARE

WinLALS AND PreLALS: LINKED-ATOM LEAST-SQUARES PROGRAM AND VISUAL WORKBENCH FOR HELICAL POLYMERS

K Okada[1] K Noguchi[2] K Okuyama[2] S Arnott[3]
[1]Ricoh R & D Center, Yokohama, Japan [2]Tokyo University of Agriculture And Technology, Tokyo, Japan [3]Imperial College School of Medicine, Imperial College, London, England

The fiber diffraction patterns from helical polymers are very different from those from small molecules. In their structure analyses, the definitions of the internal coordinates and the refinement parameters (bond distances, bond angles and torsion angles) are also different. WinLALS program was reconstructed from the LALS2000 program which was firstly developed in 1966, then mainly updated by Chandrasekaran (2000). That is, (1) all mathematical expressions were reviewed, (2) WinLALS program coded with Fortran 90 can be run on MS-Windows PCs, (3) the structure factor calculation by one of Trigonometric or Bessel functions are selectable by user, (4) all input/output assignments of LALS2000 version were amended, (5) all array dimensions are expanded to the enough size from normal use, (6) all the output parameters at each cycle with SC cycle control code were included in the update file, and (7) several bags were removed and some miscellaneous items were amended. Four computational results of WinLALS program were completely coincident within the range of with those obtained by LALS2000 program. PreLALS workbench was designed to help WinLALS users to analyze molecular and crystal structures of fibrous polymers. Its main features are (1) menus for data preparation with graphical user interface, (2) viewer for molecular and crystal structures at various stages of model building and structure analysis, and (3) execution of WinLALS program.

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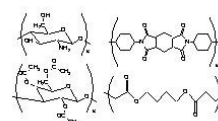
Keywords: **HELICAL POLYMERS LEAST-SQUARES GRAPHICAL INTERFACE**