PS01.03.10 A NEW SYNCHROTRON MAD DATA ACQUISITION METHOD: SIMULTANEOUS MULTI-WAVELENGTH ANOMALOUS DIFFRACTION (SMAD). Peter L. Lee* and Craig M. Ogata†, *Materials Science Division, Argonne National Laboratory, Argonne, Illinois 60439, †Howard Hughes Medical Institute, X4, NSLS, Brookhaven National Laboratory, Upton, NY 11973

In recent years, Multi-wavelength Anomalous Diffraction (MAD) phasing has emerged to be a powerful synchrotron technique for solving protein crystal structures. However, a straight forward MAD data collection requires a stable crystal, synchrotron source, and beamline optics. The customized beamlines for fast energy changes, the improvement of flash freezing techniques and the availability of stable long lifetime synchrotron sources around the world have made MAD data measurement a more accessible experiment. Nevertheless, switching among different wavelengths to measure anomalous data sets is still very time consuming and puts great demands on the stability and reproducibility of the monochromator and the synchrotron beam.

We are here introducing a new synchrotron MAD data acquisition method, Simultaneous Multi-wavelength Anomalous Diffraction (SMAD), which can avoid switching energies. SMAD employs a variable bandwidth curved crystal monochromator (polychromator) coupled with an energy selecting grid plate. Our SMAD experiment demonstrates the ability to measure reflections at six different wavelengths and their Bijvoet pairs at the same time. On one diffraction image, we record both the dispersive and Bijvoet information from a myoglobin crystal for MAD phasing.

This work was carried out at both the X4A and the X6A beamlines, National Synchrotron Light Source, Brookhaven National Laboratory, which are supported by the Howard Hughes Medical Institute, and the Division of Materials Sciences and Chemical Sciences of DOE respectively.

PS01.03.11 X-RAY DIFFUSE SCATTERING FROM A LYSOZYME CRYSTAL ANALYSED WITH A RIGID-BODY MODEL OF DISPLACEMENTS. J. Perez, Ph. Faure and J.-P. Benoit, LURE, CNRS-CEA-MENESR, Bat. 209D, Universite Paris-Sud, F-91405 Orsay Cedex, France

X-ray cloudy diffuse scattering from a tetragonal crystal of lysozyme has been collected at room temperature on the wiggler W32 station of LURE synchrotron and interpreted with a simple model of rigid-body displacements. Cloudy diffuse scattering is the part of the scattered intensity which arises from atomic displacements not correlated from cell to cell, and is therefore the signature of intramolecular or molecular correlations.

It is shown here that most of the pattern can be considered as due to independent rigid-body translations and rotations of the protein molecules within the crystal. The normal-mode analysis performed on a single molecule of lysozyme, which accounts only for the intramolecular correlations [Faure et al., 1994], results in a too smooth pattern, underlying the existence of displacements correlated at the molecular scale. By further performing an analysis of the temperature factors of the individual atoms, derived from the crystallographic refinement, it is possible to estimate the meansquare displacement due to the molecular rigid-body motion. The respective values are 0.1Å^{2}for rotations and 0.1Å^2 for translations.

The present diffuse scattering analysis confirms and completes the TLS hypothesis proposed in 1979 by Artymiuk et al., in the sense that it allows to differentiate between a rigid-body rotation movement and a breething movement of the proteins and to estimate the part of the total disorder due to rigid-body tranlations of the whole molecules.

References:
Artymiuk, P.J., Blake, C.C.F., Grace, D.E.P., Oatley, S.J., Phillips, D.C., Sternberg, M.J.E. (1979), Nature, 280, 563-568.
Faure, Ph., Micu, A., Perahia, D., Doucet, J., Smith, J., Benoit, J.P. (1994), Nature:Structural Biology, 1, 124-128.

Detectors & Data Processing I Macromolecular

MS01.04.01 DETECTORS AND DATA PROCESSING: OPTIMISED ANOMALOUS SCATTERING, HIGH RESOLUTION AND DYNAMICAL STUDIES. J R Helliwell, Chemistry Department, University of Manchester, M13 9PL, U.K.

The last 20 years has seen an important evolution of position sensitive detectors for crystallography data acquisition. Film densitometry, MWPC's, TV detectors, image plates and CCDs have been exploited. These devices all have strengths (true counting accuracy/sensitivity, MWPCs; wide wavelength response and high count rate, TVs/IPs/CCDs; large size, IPs) and weaknesses (wavelength range and count rate limits, MWPCs; detector noise, TVs; limited aperture, TVs/CCDs; poor duty cycle, IPs). In recent years very impressive results have been obtained with on-line IP devices, very large IP (Weissenberg) off-line devices, and on-line CCD devices. It has become possible, for e.g., in conjunction with cryoprotection against radiation damage of the protein sample, and/or intense, tunable synchrotron radiation, to readily measure multiple wavelength data sets, reach atomic resolution and record time-slicing dynamical protein crystallographic data. Examples include a brominated oligo-nucleotide MAD study on station 9.5 at Daresbury (IP), a seleno hydroxy methyl bilane synthase (HMBS) MAD study (collaboration with Dr A Haedener) at 9.5 (IP) and ESRF BL19 (CCD), a time-resolved study, also on HMBS, at ESRF BL3 and BL19 (CCD) and data collection on cryocooled concanavalin A to 0.94A (CCD compared with IP) at CHESS. In chemical crystallography examples include use of high photon energy (24 keV) and a CCD at CHESS with a nickel octahexylphthalocyanine and a temperature dependent space group transition. In neutron crystallography the use of IP's has started (e.g. neutron Laue of concanavalin A). Further evolution of detectors is important; the combination of the large aperture of IPs with the better duty cycle of CCDs might be possible with the 'pixel detector', a silicon based device with independent pixel counting chains. The ultimate diffraction measurement scheme of reflections measured only during their active range (seconds of arc rocking widths for lysozyme protein crystals have been realised using microgravity crystal growth) can yield optimal peak to background ratios. New sources beckon. Detector investment needs to be enhanced. Finally data processing at increased reflection measuring rates will be vital for full exploitation of both source and detector developments.

MS01.04.02 COMPARISON OF IMAGING PLATE AND CCD-BASED X-RAY DETECTORS FOR MACROMOLECULES. Y. Amemiya[1] and K. Ito[2], [1]Department of Applied Physics, School of Engineering, The Univ. of Tokyo, Yayoi, Bunkyo, Tokyo 113, [2]Graduate Univ. for Advanced Studies

In x-ray diffraction experiments for macromolecules with use of synchrotron radiation, imaging plate (storage phosphor) detectors[1] and CCD-based x-ray area detectors[2] are currently two of the most widely used x-ray area detectors[3]. Regarding the CCD-based detectors, they are classified into two types; one employs an image intensifier (referred as an "intensifier-coupled CCD")[4,5], and the other employs a tapered optical fiber (referred as a "fiber-coupled CCD")[6,7,8] as a device to de-magnify x-ray image onto a small format CCD. The above three types of the x-ray detectors have high detective quantum efficiency (DQE) (30 - 80 %) and wide dynamic range (4 - 5 orders of magnitude). Besides, they all are well suited to experiments with use of synchrotron radiation, because they don't suffer from count rate limitation,