

MS01.03.06 SYNCHROTRON RADIATION FOR HIGH RESOLUTION DATA COLLECTION Z. Dauter, EMBL c/o DESY, Notkestrasse 85, 22603 Hamburg, Germany

Recent advances in the macromolecular data collection technology has made very high resolution data collection on macromolecular crystals much more tractable. Several points have contributed to this.

The construction of very bright beam lines at different synchrotron sites based on wigglers or undulators provides X-radiation of unprecedented intensity. This is required to obtain a meaningful signal from protein crystals giving thousands of inherently weak reflections.

Macromolecular crystals are susceptible to radiation damage, especially when exposed to strong synchrotron beam. Shock freezing of the sample alleviates this problem and allows to collection of data to maximum resolution from a single crystal.

The possibility of using fast and accurate 2-dimensional detectors, such as imaging plates or CCD's, plays in practice a very important role. The number of reflections increases with the cube of the resolution so does the contrast in intensity between the low and high resolution reflections. This requires the use of fast detectors with high dynamic range and spatial resolution. IP and CCD fulfill this condition.

Equally important is the availability of data processing programs, which are fast, efficient and easy to use. Significant progress in this field has taken place in the last few years. It is possible to obtain the complete data set, perhaps consisting of several hundreds of thousands of merged reflections, just minutes after finishing the synchrotron session.

Advances in macromolecular data collection do not make the human contribution to this process redundant. Progress in macromolecular crystallography and especially in computing in the recent years means that the data collection plays even more important role, since it is much easier to repeat all the other stages, if necessary. It pays off to collect the data as optimally as possible, since it makes all later steps of the structure analysis considerably easier.

MS01.03.07 OPTIMISING SYNCHROTRON FACILITIES FOR DATA COLLECTION IN PROTEIN CRYSTALLOGRAPHY. Colin Nave, CLRC Daresbury Laboratory, Warrington WA4 4AD, UK

A method is presented for specifying the optimum x-ray source, optics and detectors for protein crystallographic data collection. There is intense effort in obtaining facilities matched to the large volume demand and most exacting requirements in protein crystallography. Simply asking for the highest flux or highest brightness is misleading. A better definition of the requirements would be to have the maximum flux in to the phase space volume required by the specimen. A proper evaluation of the requirements should therefore start from the characteristics of the range of specimens which are to be studied.

A graphical representation is derived to illustrate how various sources and optics are matched to parameters such as crystal size and unit cell dimensions. The perfection of the crystal should also be considered, the ultimate (and impractical) extreme being to match the incident beam to the diffraction characteristics of each diffraction spot using Du Mond (or similar) diagrams. Recent investigations have found that some protein crystals have a very low mosaicity. Less attention has been devoted to the divergence of beams diffracted from protein crystals. Simple measurements of mosaicities and diffracted beam divergences have therefore been made from crystals of various degrees of perfection. It was found that the procedure of converting crystal rocking widths to arcs in the detector plane does not apply. However, a simple 3 parameter

model can be used to describe crystal perfection. The procedures described have consequences for the design of beamlines and detectors for protein crystallography. Typical issues are whether there is a limit to the source emittance required or the size of an area detector system. The aim is to stimulate discussion of these issues and encourage further similar measurement from a much wider range of specimens, including those with the higher mosaicities typically obtained at cryo-temperature.

PS01.03.08 A PROTEIN CRYSTALLOGRAPHY STATION FOR MULTIWAVELENGTH ANOMALOUS DISPERSION AT THE SRS. E. M. H. Duke*, S. M. McSweeney†, S. H. Kinder*, S. G. Buffey*, M. W. Bailey*, R. C. Kehoe*, P. W. Atkinson*, C. Nave*, *Daresbury Laboratory, Warrington. WA4 4AD. U.K., †EMBL, Grenoble, France

Protein Crystallography Station 9.5 at the SRS Daresbury has been equipped with many features to enable ease of use for both the novice and experienced user. Data collection is executed through a graphical user interface which controls the detector, goniostat and monochromator. In addition the ability to automatically optimize the beam and an automated procedure for crystal alignment along an axis are available. A two-theta arm and an harmonic rejection mirror allow data to be collected to higher resolution at wavelengths above 1.5Å.

The station itself is designed for Multiwavelength Anomalous Dispersion and beamline instrumentation includes a toroidal mirror at 18m, a rapidly tunable Si 111 channel-cut monochromator at 30m and an harmonic rejection mirror at 31m. Data are collected using a CAD4 3-circle goniostat and a 30cm MAR image plate.

Examples of data collected on the station will be shown.

PS01.03.09 CONSTRUCTION OF THE BIO-CRYSTALLOGRAPHY (MIROAS) BEAMLINE AT THE SPRING-8. N. Kamiya, Y. Kawano, T. Uruga, H. Kimura, T. Ishikawa, and H. I. Kitamura, Jaeri-Riken SPring-8 Project Team, The Institute of Physical and Chemical Research (Riken), Hirosawa 2-1, Wako 35101, Japan

The Bio-Crystallography beamline¹ under construction at the SPring-8 is aimed at routine analyses in macromolecular crystallography by the multiple isomorphous replacement (MIR) technique with optimized anomalous scattering (OAS). The beamline also focuses on data collections for very large macromolecules such as ribosomal particles and viruses and for small crystals less than 100 µm. The light source is an invacuum undulator of 3.2 cm magnetic periodicity, which emits highly brilliant X-rays in an energy range of 9 - 38 keV. The highest power and power density of the undulator are 5 kW and 300 kW/mrad², respectively. To handle the tremendous power density a rotated-inclined double-crystal monochromator will be used. The first crystal will be chilled by a pin-post water cooling technique. To focus the high-energy X-rays up to 38 keV, two super mirrors (OSMIC) will be installed to get quasi-isotropic and small beam profile of about 100 µm at focal position. This beamline will be characterized by uses of high-energy X-rays up to 38 keV and X-rays (9 - 18 keV) with excellent energy resolution (2 x 10⁻⁴) at one beamline. The former is useful for ideal data collections at high resolution without absorption effects, and the latter is preferred for the OAS data collections of the heavy atom derivatives utilized in the MIR technique. For recording the diffraction patterns, imaging plates (IPs, Fuji Film) of large active area (400 x 500 mm²) will be used as the X-ray detector in the experimental station. To read out the IPs within one minute, a new readout mechanism is under development by using a lineshaped laser beam and a charge-coupled device.

1) N. Kamiya et al., Rev. Sci. Instrum. 66, 1703 (1995).