

01.1-2 AN OPTIMIZED DIFFRACTOMETER FOR NEW APPLICATIONS IN PROTEIN CRYSTALLOGRAPHY USING SYNCHROTRON RADIATION. By H.D. Bartunik*, K.S. Bartels*, J. Allibon+, *EMBL c/o DESY, Hamburg, FRG; +Inst. Laue-Langevin, Grenoble, France.

A double-focussing instrument (X31) with 1:1 imaging geometry has been set up for protein data collection using synchrotron radiation (SR) at DORIS/HASYLAB. The X-ray optics include a scanning double monochromator with a bandwidth between $\Delta\lambda/\lambda = 5 \cdot 10^{-5}$ and $1 \cdot 10^{-3}$ within the wavelength range $0.65 < \lambda < 2.5 \text{ \AA}$. X31 is equipped with a modified Huber 4-circle diffractometer with a second, long 2 θ -arm. As detectors, a single counter, a linear gas detector and an area detector (MWPC) are available for data collection for structures with cell dimensions up to $\sim 200 \text{ \AA}$. In addition, X31 includes facilities for automatic rotation data collection on photographic film for cell dimensions up to $\sim 1000 \text{ \AA}$.

X31 is optimized for a wide range of applications in protein crystallography. The combination of a scanning monochromator (with the option of hardware driven wavelength scans) and low-noise electronic detectors provides outstanding possibilities for anomalous diffraction experiments. Crystal cooling equipment allows high-resolution studies of intramolecular dynamics and enzyme kinetics within the temperature range $100 \text{ K} < T < 330 \text{ K}$. Time-resolved crystallography is feasible on a 100 μsec time scale (with a LD - development of time-resolved data collection with a MWPC is in progress), and on a 10 nsec time scale (with a plastic scintillator). A new collimator with adjustable apertures $\approx 10 \mu\text{m}$ and alignment under remote control helps to maximize the signal-to-noise ratio for data collection from microcrystals.

All types of data collection on X31 and the alignment of the whole instrument are carried out under full computer control by a PDP 11/24 via CAMAC. Automatic data acquisition is based on monitoring of the incident SR beam in order to compensate for fluctuations.

The diffractometer software is a modified version of the program "LSD" developed at the ILL, Grenoble. A new monitor controlled hardware-driven scan facility with read-out into a self-incrementing CAMAC memory has been developed which reduces dead-time during scans to a minimum.

The performance of the instrument is described on the basis of experimental results from different types of application in protein crystallography.

01.1-3 PROTEIN DATA COLLECTION WITH AREA DETECTORS USING SYNCHROTRON RADIATION. By K.S. Bartels*, H.D. Bartunik*, C. Boulin+, A. Gabriel**, J. Hendrix*, W. Prieske+, *EMBL, c/o DESY, Hamburg, FRG; **EMBL, c/o ILL, Grenoble, France; +EMBL, Heidelberg, FRG; ++Univ. Hamburg, Inst. Angew. Chemie, Hamburg, FRG.

Data acquisition and evaluation software for protein data collection with area detectors (AD) using synchrotron radiation (SR) has been developed. The rapid AD data acquisition is based on the use of a self-incrementing CAMAC memory with the option of fast dump onto disk. Refinement and evaluation of crystallographic diffraction patterns is made on-line or off-line on a VAX 11/750.

In first test applications, data were collected with two different AD systems, a multiwire proportional chamber (MWPC) and a TV camera detector, and the data processed with the new software.

Anomalous diffraction data have been collected from met-Myoglobin near the Fe K-edge using a MWPC at DORIS/HASYLAB. The AD has been mounted on the double-focussing instrument X31 which is equipped with a diffractometer and a scanning double monochromator. The MWPC has an active area of $100 \times 100 \text{ mm}^2$ and a wire spacing of 1mm. The spatial resolution (with Argon filling at 1.2 bar) is 2.5mm at FWO.1M. A rapid delay-line readout system allows data rates in excess of 200kHz.

Further, data have been collected from Catalase at a wavelength of 1.5 \AA on the double-focussing instrument X11 at DORIS using a TV camera system set up from commercial components including a 80mm Westinghouse camera (EBSICON) with integrated image intensifier and a ZnS(Ag) scintillator screen. The spatial resolution has been determined to 0.8mm at FWO.1M. An exposure time of 40msec was sufficient to record a diffraction pattern to 3 \AA resolution. Data were recorded on video tape. The digitized data were evaluated with the same programs as the MWPC data.

The performance of the detectors is discussed on the basis of the results of the data evaluation. The high efficiency of the TV detector leads to a reduction of effects of radiation damage in SR applications; such a system is already useful in qualitative applications, e.g. for alignment of crystals of high-molecular weight structures. The low intrinsic noise of the MWPC improves significantly the experimental conditions for high-accuracy data collection for anomalous phasing. Further application of the MWPC is in progress.