13

01-Instrumentation and Experimental Techniques (X-rays, Neutrons, Electrons)

be affected so much by r. 3) More data will be recorded on a film, when the Weissenberg geometry will be applied, 4)Bijvoet pairs are recorded with high accuracy on the same film as simultaneous reflections. 5) Almost all X-ray pass should be evacuated or filled with helium gas to reduce the scattering and absorption by air.

absorption by air.

The fundamental requirements for the detection of X-ray diffraction from protein crystals are: 1) high detective quantum efficiency, 2) wide dynamic range, 3) linearity of response, 4) high spatial resolution, 5) large detective area 6) uniformity of response, 7) high counting rate capability and 8) low back ground noise. A X-ray integrated-type area detector, called an imaging plate(IP) is one of the most suitable detectors for the data collection of X-ray diffraction from protein crystals. The combination with the camera, IP and SR X-ray becomes a very powerful (N. Sakabe, Nucl. Instr. and Meth., 1991, A303, 448).

becomes a very powerful(N. Sakabe, Nucl.Instr. and Meth., 1991, A303, 448).

The camera type 3 was designed for PF users at BL6A2 station. Normally, one data set around an axis is recorded on 10-30 IP sheets with consuming only one or two crystals and 5-10 sets of data are collected in 24 hours. The size of IP which we are using is 400x200mm. Strong demanding of the large size of IP, we have designed 800x400mm IP reader which will be installed this April detached room at BL18B(N. Watanabe et al, poster in this meeting).

poster in this meeting).
The application of this data collection system and future data collection system will be discussed.

MS-01.02.05 PRESENT AND FUTURE PROSPECTS FOR MACROMOLECULAR CRYSTALLOGRAPHY AT THE CORNELL HIGH ENERGY SYNCHROTRON SOURCE (CHESS). Steven E. Ealick, Section of Biochemistry, Molecular and Cell Biology, Cornell University, Ithaca, NY 14853

The Cornell High Energy Synchrotron Source (CHESS) consists of five beamlines and 11 experimental stations which receive radiation from the Cornell Electron Positron Storage Ring (CESR). Beamlines A, B and C receive radiation from circulating electrons while beamlines D and F receive positron radiation. Macromolecular crystallography is currently supported on four experimental stations. Station F-1 is a doubly focused, tuneable (6-14 keV) station that receives half the radiation from a 25-pole wiggler. This popular station is used primarily for monochromatic data collection on samples that have large unit cells, small size, weak diffraction or radiation sensitivity. Station F-1 is part of a BL-3 containment facility that can be used for X-ray diffraction studies of bio-hazardous samples. For the past ten years, station A-1 has been a doubly focused, fixed wavelength station (8.0 keV) that receives radiation from a 6-pole wiggler. Station A-1 has been used for routine

monochromatic data collection on a variety of crystallographic systems. In January of 1993, station A-1 was dismantled to make way for a new tuneable station that will be comparable to F-1. Station B-2 receives white radiation from a bending magnet and has been used for Laue protein crystallography and micro-diffraction. Station F-2 receives the other half of the radiation from the F line wiggler and is the most recent station at CHESS to be used for macromolecular crystallography. The F-2 optics consist of a double crystal monochromator with fixed offset and a vertically focusing mirror. Sagittal bending of the second monochromator crystal provides focusing in the horizontal direction. The station can be rapidly tuned over the range 6-40 keV with an energy resolution of a few eV. The station has been used for multiple wavelength anomalous diffraction (MAD) phasing experiments and general purpose diffraction on non-biological samples. The high critical energy of the F line wiggler (24 keV) makes station F-2 an ideal station for high energy diffraction studies. High energy diffraction is potentially useful for maximizing signal-to-noise, reducing radiation damage and reducing absorption effects. Future plans at CHESS include an upgrade of the A line by insertion of a 25-pole wiggler and rebuilding of a tuneable experimental station A-1. In addition, current plans call for a gradual increase of the storage ring current from 100 to 500 mA during the next two years. Finally, the long term capabilities of CHESS will be determined by the outcome of a pending B-factory proposal. If approved, the storage ring will be reconstructed as two separate rings one operating at 8 GeV and 1 A and the other operating at 4 GeV and 2 A. The corresponding CHESS laboratory will consists of eight beamlines mostly based on insertion devices.

MS-01.02.06 PROCESSING OF DATA FROM LARGE UNIT CELL VIRUS CRYSTALS. By *M. S. Smyth , J. G. Tate and D. I. Stuart. Laboratory of Molecular Biophysics, University of Oxford, U. K.

We have crystallized three picornaviruses: bovine enterovirus (BEV), a benign virus of interest as a vaccine vector; coxsackievirus A9 (CAV9), causative agent of a range of human disorders, and human rhinovirus type 2 (HRV2), a common cold virus. The crystal morphologies are suitable for X-ray analysis, and each diffracts to beyond 3Å resolution. The unit cell dimensions and space groups are summarized in the table.

Virus	a	Ъ	с	β	Sp. group
BEV		390Å		113°	P2 ₁
CAV	495Å	495Å	695Å	90°	P4,22
HRV	382Å	352Å	681Å	90°	P2,2,2,

The collection of such data necessitates the limitation of resolution to 3.5Å in the cases of the CAV9 and the HRV2. Nevertheless, we have collected sufficient data from each of the virus crystals

14 01-Instrumentation and Experimental Techniques (X-rays, Neutrons, Electrons)

to enable us to commence the structural analysis by the molecular replacement method. In addition, we have collected data from BEV and HRV2 crystals with anti-viral drugs bound (WIN compounds), an HRV2 monoclonal antibody escape mutant, and a possible HRV2-Fv antibody fragment complex. We will report the stage of data processing and discuss problems imposed by large cell dimensions.

PS-01.02.07 A NEW MACROMOLECULAR CRYSTAL-LOGRAPHY STATION ON THE BEAMLINE BL-18 AT THE PHOTON FACTORY. By N. Watanabe*1, S. Adachi²⁾, A. Nakagawa¹⁾, and N. Sakabe¹⁾, ¹⁾ Photon Factory, National Laboratory for High Energy Physics, Tsukuba, Ibaraki 305, ²⁾ The Institute of Physical and Chemical Research (RIKEN), Wako, Saitama 351-01, JAPAN.

A new experimental station (BL-18B) has been constructed on the bending-magnet beamline at the Photon Factory (PF) to extend its capability for macromolecular crystallography. The branch beamline is equipped with a 1m long fused quartz bent cylindrical mirror with 1:1 focusing, located 13.75m from the source. The surface of the focusing mirror is cylindrically polished and platinum coated with a sagittal radius of 41.3mm, and bent to a radius of ca. 4,500m. The glancing angle of the X-ray beam with the mirror is set to about 3mrad, which gives a cut-off wavelength of approximately 0.4Å. The mirror can focus the X-ray beam to about 0.4mm (vertical) \times 1.2mm (horizontal), which is consistent with the expected focus size simulated by the raytracing technique. The monochromator is a fixed-exit doublecrystal, located 23.1m from the source. The monochromator consists of two kinds of flat crystals, usually silicon (111) and germanium (111), mounted parallel on the goniometer. The two types of crystal are therefore interchangeable without opening the vacuum chamber. The monochromator θ_B range is 5° to 70°. Photon flux of the monochromatic beam is 9.6×109 Photons/sec (Si) and 2.3×1010 Photons/sec (Ge) at 1.38Å at the sample position when the PF ring is operated at 2.5GeV, 300mA and the acceptance of the first siit is 0.2mrad (vertical) and 2.0mrad (horizontal).

BL-18B has been built as an end station in order to have enough space available for installing a large camera and other instruments in the experimental hutch. The station will extend the capability at PF beyond what is provided for with the Weissenberg camera (Sakabe, N., NIM,1991, A303, 448) at the BL-6A2 station (Satow, Y. et al., Rev. Sci. Instrum. 1989, 60, 2394). In addition to increasing the experimental time available for users BL-18B, unlike BL6A2, provides a point focused white beam. This latter feature gives the station time-resolved Laue capability. Special Image Plates (IP) (400mm×400mm

and 400mm×800mm) and IP scanner are also being developed to allow more effective exposures using Weissenberg and Laue cameras at the station.

PS-01.02.08

POSITION SENSITIVE PHOTOMULTIPLIER TUBE NEUTRON DETECTOR. Clive Wilkinson, Mogens Lehmann*, Andre Gabriel, John Allibon and Francois Dauvergne, European Molecular Biology Laboratory, BP156X, 38042 Grenoble CEDEX, France.

We are presently constructing a neutron Laue diffractometer to extend the range of protein structures which can be measured to atomic resolution by neutron diffraction. Finding a two-dimensional detector with digital readout, spatial resolution of lmm, good dynamic range and a large angular coverage has become a significant part of the project.

One possible solution is to have an array of position sensitive photomultiplier tubes, each with a Li glass scintillator, around the sample. We have tested a Hamamatsu 3" square tube with a Nuclear Enterprises 902.scintillator greased onto its front surface. Light produced by the scintillator falls on the photocathode and the resulting electron shower is amplified by a chain of dynodes and subsequently detected on an anode grid at the back of the tube. The data reading system consists of delay lines which are connected to each set of anode wires, along which the electron pulses travel simultaneously. By timing the arrival of the pulses at each ends of the delay lines, the X,Y coordinates of the original neutron arrival at the scintillator can be found. The integrated intensities of reflections have been measured on a four-circle neutron diffractometer with the tube and with a 'normal' ${\rm BF}_3$ monodetector. Using a 2mm thick scintillator, the tube has been found to be more than 60% efficient at a neutron wavelength of 0.84A, and to have a resolution better than 1mm.

* also at ILL, Grenoble

PS-01.02.09 A NEUTRON-SENSITIVE TV-IMAGE INTENSIFIER SYSTEM. By H. G. Smith* and J. B. Davidson, Oak Ridge National Laboratory, Oak Ridge, TN 37830.

A neutron-sensitive TV-image-intensifier system has been in use at the ORNL HFIR reactor for a number of years. This system, though only qualitative and somewhat bulky, has been extremely useful in monochromator alignment, sample alignment in environmental containers, and in the characterization of samples -- single crystal and polycrystalline -- all in real time. With the recent development of minature CCD cameras coupled with PCs and frame grabbers and imaging processing techniques, the new systems are almost off-the-shelf items and can be readily assembled in-house. While not quite at the quantitaive stage for accurate data accumilation, processing, and analysis, the new systems are compact, easy to use, and relatively inexpensive.