PS01.11.07 BIOCRYSTALLOGRAPHY AT THE HIGH BRILLIANCE BEAMLINE (ID2) OF THE ESRF. E. P. Mitchell, A. Åberg, J. Shaw, S. Wakatsuki, D. Spruce, L. Claustre, P. Bösecke, O. Diat, B. R. Rasmussen, ESRF, BP 220, F-38043, Grenoble Cedex, France, EMBL, 38042 Grenoble Cedex, France

The high brilliance beamline at the ESRF is one of the most intense sources of low divergence X-rays for protein crystallography. The beamline has been designed with studies on large cell proteins and small crystals in mind.

Recent and ongoing improvements to the protein crystallography end station of the ESRF high brilliance beamline now make routine data collection from crystals with very large unit cells (for example viruses and ribosomes) and very high resolution data possible. Notably data has been successfully collected from crystals of Blue Tongue Virus (largest unit cell dimension 1550Å; Stuart *et al,* Oxford, UK).

Until recently a complete 30cm MAR Research system was used, now a five circle Huber diffractometer, retaining only the MAR detector itself, has been installed, with cryogenic cooling possible and a maximum crystal to detector distance of 1 metre. The fifth circle ($\delta 2$) of the goniometer allows an angular rotation of $\pm 20°$ on the long detector arm. The fourth shorter circle ($\delta 1$) allows mounting of a scintillation counter for ease of alignment. It is envisaged that the $\delta 1$ arm could also be used to mount a small fast-scan CCD camera for the purpose of screening crystals (for example heavy metal derivatives) speedily.

New software for a rational data collection strategy using the Huber goniometer is being developed. The aim is to use the flexible Huber goniometer to its full capacity and maximise data collection efficiency. The software will use a graphical user interface (TclTk) to allow smooth use of the beamline.

PS01.11.08 A HIGH PRECISION SPECTROMETER FOR THE ABSOLUTE DETERMINATION OF X-RAY ABSORPTION EDGES AS CALIBRATION STANDARDS. J. Stümpel, P. Becker, Physikalisch Technische Bundesanstalt, Bundesallee 100, Braunschweig, Germany

The general characteristics and spectrometric features of a high resolution fourcrystal reflection x-ray monochromator with wavelength analysis installed at the HASYLAB beamline L at DESY are presented.

The monochromator is part of a spectrometer developed to calibrate x-ray absorption edge spectra in the energy range of 6 - 36 keV with a relative uncertainty $\Delta E/E$ from 10^{-5} to 10^{-6}. This requires an extremely effective suppression of harmonics and also a negligible instrumental influence in order to obtain almost intrinsic spectra. As the results show, the monochromator fulfills the requirements, including very high stability.

One essential advantage of the (+ - - +)-setting is that the monochromator itself limits the divergence of the primary radiation, which is very useful for energies above 5 keV where the natural divergence of the synchrotron radiation exceeds the width of the crystal reflection curve. This setting needs no further optics such as slits to improve the resolution, which is therefore not influenced by vertical movements of the primary radiation source.

Harmonic suppression can be achieved by detuning the channel-cut nondispersive monolithic part slightly out of its parallel-sided position and is described by the detuning angles $\delta 1,2$. For reflections at netplanes with Miller's indices all odd, the structure factor of the diamond structure leads to the forbidden harmonic with $n = 2$ in the Bragg-equation $n\lambda B = 2d \sin\theta B$. It is therefore advantageous to choose reflecting netplanes with odd indices, (e.g. (111) or (311)).

PS01.11.09 NOVEL SYSTEMATIC PURIFICATION METHOD DEVELOPMENT TO RAPIDLY OBTAIN HIGH PURITY BIOLOGICAL MOLECULES FOR CRYSTALLOGRAPHY STUDIES. Daryl J. Vanderburgh, Patrick R. Carberry, Michael Meys, PerSeptive Biosystems, Inc. 500 Old Connecticut Path, Framingham, MA 01701

Obtaining adequate quantities of high purity biological molecules that support crystal growth often becomes a frustrating bottleneck that impedes the progress of structural studies. The molecular subjects of these studies have either never been purified before, or existing published purification protocols weren't designed with the purity requirements of crystallography in mind. Crystallographers must either face the tedium of developing new purification methods themselves or get their purified material from collaborators - which can introduce its own set of bottlenecks. Perfusion Chromatography® is a breakthrough technology that allows you to perform chromatographic separations 10 to 100 times faster than with conventional media. With individual run times of 3 to 5 minutes, it now becomes practical to quickly examine all the variables that impact on a separation and to systematically hone in on the best possible purification protocol. It is not uncommon to develop a complete method yielding crystallization quality material in just a few days. Once a method is developed, the fast run times can be equally exploited to generate the necessary quantities of final material for crystallization experiments on an "asneeded" basis - offering the potential for improved experimental flexibility and productivity. Fast run times also help to ensure that purified molecules are recovered in their biologically active form. The principles of Perfusion Chromatography technology will be discussed and some practical examples of its application in structural studies will be shown. The technology has already been eagerly embraced by a number of researchers in the X-ray crystallography field.

PS01.11.10 HIGH SPEED DIGITAL X-RAY SPECTROMETER WITH TIME RESOLUTION CAPABILITY. W. K. Warburton, B. Hubbard, C. Zhou, X-ray Instrumentation Associates, 2513 Charleston Rd. STE 207, Mountain View, CA 94043-1607

Digitally based, energy dispersive x-ray spectrometer electronics have been developed which allow data to be collected in several time resolved modes to the microsecond time scale. The instrument was first developed for collecting x-ray fluorescence data using multielement detector arrays at synchrotrons but is readily adaptable to time resolved work using laboratory x-ray sources as well.

Energy dispersive x-ray fluorescence measurements underlie several powerful experimental techniques for studying materials' compositions (x-ray fluorescence analysis: XFA) and their physical and chemical structure at the atomic scale (x-ray absorption spectroscopy: XAS). Arrays of x-ray detectors are becoming popular in these applications as samples become ever more dilute. Our new instrument uses digital processing techniques to implement 4 complete sets of spectrometry electronics, each including a 1000 channel multichannel analyzer, in a single CAMAC module. Each spectrometer is capable of handling input rates of over 500,000 counts/sec with triangular peaking times down to 0.5 μs. All setup parameters are digital inputs, including gain, peaking time, pileup inspection values, and detection thresholds. This allows a convenient approach to completely automating all data collection and verification tasks.

The instrument's digital basis also allows x-ray arrival time information to be encoded, allowing the power of x-ray fluorescence measurements to be applied to time dependent phenomena as follows. The module has both a "gate" and a "sync" input, which accept standard TTL pulse or level signals. In time resolved data collection mode, the gate is pulsed each time the experiment is retriggered (e.g. electrically or by laser) and the collected x-rays are tagged with