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

time since the gate pulse. This allows the number of counts within a designated energy window to be binned as a function of time since the trigger. Time resolution to the μs level is possible this way.

The module can also count the number of times the sync input toggles following each gate pulse and tag x-rays with this information as well. This allows the x-rays to be binned by this information as well, allowing spectra to be collected synchronously with a modulating sample state (phase locked mode) at rates up to a few 100 kHz. We present an example of EXAFS from a high-T superconductor cycling between its normal and SC states.

PS01.11.11 A CONTAINER FOR THE TRANSPORT OF MOUNTED CRYSTALS. Marc Whitlow, Berlex Biosciences, 15049 San Pablo Avenue, Richmond, CA 94804

An airtight crystal shipping tube has been produced by modifying a 15 ml pressure reaction tube to hold a mounted crystal. A crystal mounted on a specimen pin is held in place in the crystal shipping tube by a set screw. The crystal shipping tubes are then inserted in holes prepared for them in two foam rubber blocks. The foam rubber blocks fit into a Styrofoam box, and the whole package is shipped by overnight courier. Both the foam rubber packing and the fact that the specimen pin is firmly held in place minimize the mechanical stresses that occur during transport. Furthermore, the airtight container eliminates any pressure changes that may occur. In addition, the mounted crystals are visible through the glass shipping tube. A number of macromolecular data sets have been successfully collected from crystals shipped in this way.

PS01.11.12 ELECTRON DIFFRACTOMETRY AND CRYSTAL STRUCTURE OF BRUCITE. A. P. Zhukhlistov[1], A. S. Avilov[2], G. Ferraris[3], B. B. Zvyagin[1], V. P. Plotnikov[1], [1]Inst. Ore Mineralogy RAS, [2]Inst. Crystallography RAS, Moscow, Russia, [3]University of Torino, Italy

The principles of direct measurements of the electron diffraction (ED) intensities developed in the State Optical Institute (St.Petersburg) and Institute of Crystallography RAS (Moscow) and a commercial EMR-102 ED-camera have been adapted for analysis of two-dimensional intensity distributions, in particular those of the most most informative oblique-texture patterns. With stationary detector, the ED-pattern is moved within electromagnetic fields with variable steps and along chosen directions. Special instrumental and software improvements provide corrections of different effects (e.g., incident beam instability) and increase precision, resolution, linearity and dynamic range of the measurements.

A set of intensities collected on brucite, $Mg(OH)_2$, showed that the described system of electron diffractometry is the most reliable ever produced for this purpose and opens new prospects in the field of structural crystallography by electron diffraction. In fact, it provides reliable Volt-values of the crystalline electrostatic potential field, whereas the structural features of brucite have been revealed with a precision comparable with that of X-ray diffraction for O and Mg atoms, and of neutron diffraction for the H atom.

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PS01.11.13 X-RAY DIFFRACTION AT NON-AMBIENT TEMPERATURE CONDITIONS. B. Koppelhuber-Bitschnau, F. A. Mautner, Institute of Physical and Theoretical Chemistry, Technical University Graz, 8010 Graz, Austria, and P. Doppler, Anton Paar GmbH, Graz, Austria

Several Cameras for X-ray Diffraction at low and high Temperature range were developed, most features and benefits of the following four Temperature Attachments are presented.

With the HTK 16 High-Temperature Camera investigations in the temperature range from room temperature to 1850 K can be carried out either under vacuum, air or inert gas. The HTK 16 can easily be fit to most available goniometers. Integrated alignment slit allows precise positioning even at high temperatures, the heating filament is optimized for minimum temperature gradient, the linear compensation of the heating filament elongation guarantees for sample position stabilization.

The TTK 450 Low-Temperature Camera can be operated with most of the available goniometers, both horizontal and vertical ones. It permits temperature studies by X-Ray methods at temperatures from 80 to 700 K either under vacuum, air or inert gas.

The XRK X-Ray Reactor Chamber mounted on a goniometer permits studies of solid state and solid state-gas reactions at temperatures from room temperature to approx. 1250 K. The experiments may be carried out either in reduced, inert or oxidizing atmospheres at pressure from approx 1 mbar to 10 bar. No temperature gradients within surface and whole bulk of samples, no condensation of reaction gases because housing thermostatable up to 400 K.

The He-TTK Low Temperature Attachment equipped with a closed cycle Helium cryostat permits temperature studies to be made by X-ray methods at temperatures from approx. 12 to 700 K. The He-TTK can be operated with most of the vertical goniometers available, and is suitable for powders, sheets, single crystals and thin films.

PS01.11.14 THE ANALYSIS OF MACROMOLECULES UNDER AQUEOUS AND NON-AQUEOUS CONDITIONS DETERMINING THE PARTICLE SIZE DISTRIBUTION AND MOLECULAR WEIGHT DISTRIBUTION USING LOW AND RIGHT ANGLE LASER LIGHT SCATTERING AND PHOTON CORRELATION SPECTROSCOPY (PCS) DETECTION WITH SIZE EXCLUSION CHROMATOGRAPHY. Trevor Havard and Peter Wallace.Precision Detectors Inc., 160 Old Farm Road, Amherst, MA 01002 Tel 413 256 0516.

The objective of this paper is the separation and analysis of the particle size and molecular weight of macromolecules like Poly-Carbohydrates and Proteins, carried out under aqueous conditions, and macromolecules like polystyrene, star branched polystyrene and dendrimers. The paper will describe a **new instrument** that enables the molecular weight and particle size distribution to be obtained simultaneously. The paper will also describe how an instrument of this type can be used for the first time in flow mode to determine the molecular weight distribution as well as the hydrodynamic radius of the macromolecule eluting from an SEC system. This system is especially useful in determining the amount of aggregation that occurs while trying to dissolve certain macromolecular systems like carbohydrates and proteins into solution under different conditions where pH, temperature may be varied. The results show the effective use of Photon Correlation Spectroscopy (PCS) to determine aggregation, branching, and size, independent of the usual chromatographic integration and baseline user selection. The use of Photon Correlation Spectroscopy as a technique for characterizing particles is well documented. However, until now there has never been an attempt to make these important measurements in a flowing SEC system.