P.01.03.2
Acta Cryst. (2005). A**61**, C140
Phase Determination using a Wide-band Parallel Synchrotron Radiation Beam
Tomoyuki Koganezawa, Takeshi Udagawa, Yukio Yoshimura, Naotake Nakamura, Hiroshi Iwasaki, *Faculty of Science and Engineering, Ritsumeikan University.* E-mail: kt770217@se.ritsumei.ac.jp

New diffraction system has been constructed at the Synchrotron Radiation Center at Ritsumeikan University, in which a wide-band parallel X-ray beam is produced by reflection from the depth-graded multilayer monochro –mator [1]. The band width is 600eV and the monochro -mator is useful in the photon energy range from 6000eV to 8000eV.

In diffraction patterns of an oscillating single crystal recorded using this system, Bragg reflections appear in an elongated form on an imaging plate and, if the absorption edge of an atom in the sample crystal is included in the band, a characteristic intensity profile is seen due to anomalous dispersion. As an application of this system, we determined the phase of the structure factor of a ferrocene derivative crystal, $C_{36}H_{32}O_7Fe$, choosing the Fe atoms as anomalous scatterers, based on a newly developed method of phase determination [2].

[1] Koganezawa T., Uno K., Iwasaki H., Nakamura N., Yoshimura Y., Shoji T., *J. Appl. Cryst*., 2004, **37**, 136-142. [2] Iwasaki H., Yurugi T., Yoshimura Y., *Acta Cryst. A*, 1999, **55**,864-870.
Keywords: synchrotron radiation, anomalous dispersion, wide-band beam

P.01.03.3
Acta Cryst. (2005). A**61**, C140
Simultaneous XRPD-MS Study on Iron Oxides Supported on Spinel-like Aluminate
Danila Ghisletti[a], Ugo Cornaro[a], Claudio Contardi[a], Domenico Sanfilippo[b], Mauro Gemmi[c], Marco Merlini[c], Gilberto Artioli[c], *[a]EniTecnologie Spa, S.Donato Milanese, Italy. [b]Snamprogetti Spa, S.Donato Milanese, Italy. [c]Dipartimento di Scienze della Terra "Ardito Desio", University of Milan, Milan, Italy*. E-mail: dghisletti@enitecnologie.eni.it

Iron oxides are suitable oxygen exchangers in redox cycles, to be employed in an innovative process of hydrogen production from natural gas [1]. Infact, these oxides, once reduced with hydrocarbons, are capable to re-oxidize by splitting [O] from water, thus producing a pure stream of H_2.

In-situ time-resolved synchrotron X-ray powder diffraction (XRPD) experiments coupled with mass spectrometry (MS) were performed on iron oxides supported on spinel-like aluminate [2], both during high temperature reduction with methane and oxidation with air.

The reactive gases were fed directly through a capillary quartz reactor containing the sample and the evolved products analyzed by an on-line connected mass spectrometer

By means of the Translating Image Plate (TIP) installed on the GILDA beamline (ESRF- Grenoble, France), the photons diffracted from the sample were collected step by step during the reaction, the translating speed determining the time resolution. The Rietveld refinement of the diffraction spectra gave the quantitative sample composition at each step of the reaction and information about the interaction of the active species with the support.

[1] EP 1134187 (19/09/2001) to Snamprogetti. [2] United States Patent Application 20040152790.
Keywords: synchrotron, in-situ time-resolved powder diffraction, iron oxides

P.01.03.4
Acta Cryst. (2005). A**61**, C140
Automation of MX Data Collection and Processing
Olof Svensson[a], Karen Ackroyd[b], Alun Ashton[c], Gleb Bourenkov[d], Steve Kinder[b], Andrew Leslie[e], Sean McSweeney[a], Colin Nave[b], Alexander Popov[f], Harry Powell[e], Darren Spruce[a], Graeme Winter[b], *[a]ESRF, BP220, 38043 Grenoble, France. [b]CLRC Daresbury Laboratory, Daresbury, Warrington WA4 4AD, UK. [c]Diamond Light Source, Rutherford Appleton Laboratory, Chilton, Didcot, Oxon OX11 0QX, UK. [d]DESY Hamburg, Notkestrasse 85, 22607 Hamburg, Germany. [e]MRC Laboratory of Molecular Biology, Hills Road, Cambridge CB2 2QH, UK. [f]EMBL Hamburg, Building 25A, DESY, Notkestrasse 85, 22603 Hamburg, Germany*. E-mail: svensson@esrf.fr

We present the DNA project, a collaboration with the aim of fully automating the collection and processing of macro-molecular X-ray crystallography data including rapid crystal screening. The DNA system takes strategic decisions about the data collection based on information provided by the data processing software and some basic parameters relating to the project which will be supplied by the user. The modular nature of the system simplifies installation on different beamlines and allows the use of different data collection and data processing software.

The DNA system forms a major part of the fully automated sample screening pipeline being but in place at many European synchrotron radiation facilities. Version 1.0 of the software is now working successfully at both the ESRF (Grenoble, France) and SRS (Daresbury, UK) and will be transferred to beamlines of other synchrotrons in the near future. We are also working on an off-line version that will be used at laboratory sources.
Keywords: automation, automatic data collection, data processing

P.01.03.5
Acta Cryst. (2005). A**61**, C140
Still Bad Crystals, but Good Data and Results from Synchrotron Facilities
William Clegg, Ross W. Harrington, *School of Natural Sciences (Chemistry), University of Newcastle upon Tyne, NE1 7RU, U.K.*. E-mail: w.clegg@ncl.ac.uk

In many cases it is simply not possible to transform bad crystals into good ones, but a crystal structure is still required. The use of high-intensity synchrotron radiation can overcome some of the problems, particularly weak diffraction, whether this be a result of small crystal size or of structural faults such as disorder or twinning.

For real success, dedicated single-crystal facilities are needed, optimized for this application and using good quality X-ray optics, state-of-the-art diffractometer and detector systems, and usually low-temperature equipment. The "small-molecule" diffraction station at Daresbury SRS, constructed about 10 years ago, has been a spectacular success with great productivity and a high level of oversubscription by users, and a second station is now available.

More recently the facility has been used for a national crystallography service for UK chemistry research groups, providing rapid access and expert staffing. As many as 12 data sets per day have been measured from samples that are known to be beyond the capabilities of the most powerful rotating-anode area-detector chemical crystallography system in the country (perhaps in the world) and have been brought to us as a last resort. Although some samples really are beyond hope (including those that simply aren't crystalline at all), the majority have yielded their inner secrets, though the effort involved is often considerable when twinning, disorder, pseudosymmetry and other challenges have to be faced.
Keywords: synchrotron X-ray diffraction, structure determination, service crystallography

P.01.03.6
Acta Cryst. (2005). A**61**, C140-C141
Recent Developments and Diffuse Scattering Studies at Beamline F1 (Hasylab/DESY)
Carsten Paulmann[a], Ulrich Bismayer[a], [a]*Mineralogisch-Petrographisches Institut, Universität Hamburg, Germany*. E-mail: carsten.paulmann@desy.de

In order to enhance data collection possibilities at beamline F1 a new marresearch CCD-detector with an active area of 165 mm in

diameter (2048² pixels in 2x2 binning) has been installed in February 2005 to replace an older CCD-system which was in operation since 1996. To increase the quantum efficiency for energies between 20 and 30 keV the detectors are fitted with 100 μm $Gd_2O_2S_2$:Tb phosphor instead of a standard 40 μm phosphor.

A previously developed software suite [1] for the reconstruction of diffuse scattering from CCD raw data has been adopted to the new data format and successfully applied in a composition- and temperature-dependent study of precursor-induced diffuse scattering in V-diluted lead-phosphates. Compared to pure lead-orthophosphate, the system $Pb_3(P_xV_{1-x}O_4)_2$ displays a complex sequence of phase transitions. The CCD-data show an overall rhombohedral symmetry (R-3m) of the paraelastic high-temperature phase, but broad diffuse intensities are clearly visible even 40 K above T_c. These maxima are centered around symmetry-allowed Bragg-reflections of the paraelastic HT-phase and must be indexed by half integers. The diffuse scattering shows a slight anisotropic spatial distribution in reciprocal space with an elongation along a*, indicating a higher degree of disorder along $[111]_{rh}$. This effect results from small (ca. 50 Å), dynamic precursor-clusters of the monoclinic ferroelastic LT-phase in an overall paraelastic matrix.

[1] Paulmann C., et al., *Nucl. Instr. Phys. Res. A*, 2001, **467**, 1293.

Keywords: CCD detectors, synchrotron X-rays, diffuse scattering

P.01.03.7

Acta Cryst. (2005). A**61**, C141

Efficiency of Light Atoms on the Low Energy SAD Phasing

Yusuke Yamada[a], Tadashi Satoh[a], Kentaro Ihara[a], Noriyuki Igarashi[a], Naohiro Matsugaki[a], Nobuhisa Watanabe[b], Mamoru Suzuki[c], Soichi Wakatsuki[a], [a]*Struct. Biol. Res. Cent., Photon Factory, KEK, Japan.* [b]*Grad. Sch. Sci., Hokkaido Univ., Japan.* [c]*Inst. Prot. Res., Osaka Univ., Japan.* E-mail: yusuke.yamada@kek.jp

Recent advancements in the X-ray data-acquisition techniques and phasing algorithms have enabled structure determination using weak anomalous signals from light elements such as sulfur that are naturally present in most proteins. This is rapidly becoming a useful technique for high-throughput protein crystallography because it does not require preparation of heavy atom derivatives. Photon Factory is building a new insertion device beamline BL-17 with emphasis on diffraction experiments with microcrystals and low X-ray energy phasing (around 6 keV) [1]. To establish a standard experimental protocol of the low energy SAD, we carried out diffraction experiments on two lectins [2] and a small GTPase with low energy X-ray beams. In the case of the lectins, it was found that the number of potassium ions in the crystal is critical for the phasing. The crystal structure containing two potassium ions were solved successfully, but crystals with only one potassium ion were not. In the case of the small GTPase, phasing was successful despite the fact that it contains fewer sulfur atoms than the proteins whose structures have been solved by the low energy SAD so far. This is because the phosphates of GDP and the calcium ion bound to the GDP contributed significantly to the anomalous signal. This suggests that the low energy SAD is a successful method especially for nucleotide binding proteins.

[1] Igarashi N., et al., *IUCr2005*, Florence. [2] Satoh T., et al., *IUCr2005*, Florence.

Keywords: synchrotron radiation crystallography, low energy SAD phasing, macromolecular crystallography

P.01.04.1

Acta Cryst. (2005). A**61**, C141

Testing the Compact Light Source: A Miniature Synchrotron for the Home Lab

Ronald D. Ruth, Jeffrey Rifkin, Roderick Loewen *Lyncean Technologies, Inc., Palo Alto, CA.* E-mail: Ronald_Ruth@lynceantech.com

During the past 30 years, synchrotron light sources have become the x-ray probe of choice for physicists, chemists, biologists and research physicians. With their high-quality, intense x-ray beams, these national research facilities have spawned a large number of new techniques and technologies spanning a broad array of applications. Perhaps the most dramatic examples of this impact come from the detailed 3-dimensional studies of protein structure using powerful crystallographic techniques such as multiple-wavelength anomalous dispersion (MAD). Recent research at Stanford University and Lyncean Technologies, Inc. has led to a new x-ray source, the Compact Light Source (CLS), which will significantly broaden this impact. The CLS is a tunable, homelab x-ray source with up to three beamlines that can be used like the x-ray beamlines at the synchrotrons--but it is about 200 times smaller than a synchrotron light source. The compact size is achieved by using a laser undulator and a miniature storage ring. The photon flux on a sample will be comparable to the flux of highly productive synchrotron beamlines. In this presentation I will introduce the Compact Light Source and show how it will bring the quality, tunability and flux of a synchrotron beam line into an x-ray scientist's local laboratory. At Lyncean Technologies, Inc. we are constructing a prototype of this source with SBIR funding from the NIGMS Protein Structure Initiative. I will report on our recent experiments and long-term outlook for the CLS.

Keywords: synchrotron, X-ray source, protein structure

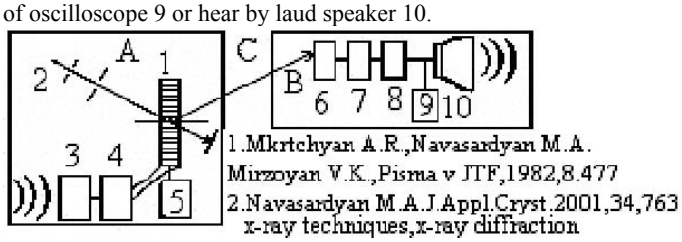
P.01.04.2

Acta Cryst. (2005). A**61**, C141

On Forced Reflection and Transmission of Speech, Using X-rays

Navasardyan Marut, *X-Ray Bunch Laboratory LTD,Yerevan, Armenia.* E-mail: x-ray@web.am

Under external influences (temperature gradient, US oscillations and so on) the intensity of the diffracted x-ray beams can be greatly increased (up to ten times or more) and at the same time the transmitted beam can be entirely reflected in the direction of diffraction (in the Laue case). This phenomenon was named by the authors of co called "controllable complete reflection" or "forced reflection" [1,2]. Three settings of the double-crystal spectrometer will be presented for this case. In the case of controllable variation of diffracted x-ray beams, it can be obtained quick increase of the intensify of diffracted x-ray beam using modulated US oscillations. This way it becomes possible to transmit and receive audio information in particular speech by means of an x-ray beam. A scheme for a practical device for transmission and reception of audio information by x-ray beams is presented in Fig. The device operates in the following manner. Single crystal modulator 1 using x-ray beam 2 and goniometer 5 is setup in the Bragg condition for one of reflecting atomic planes of sample. Modulated electromagnetic oscillations from generators 3 and 4 are sent to the crystal modulator on which modulated x-ray beams "C" is exited. We can obtain a beam with changeable counting rate of electrical impulses in detector6, which becomes electric vibrations (speech) after passing through integrating circuit 7 and amplifier 8 . This oscillations can be seen on the screen of oscilloscope 9 or hear by laud speaker 10.



Keywords: X-ray techniques, X-ray diffraction, charge densities

P.01.04.3

Acta Cryst. (2005). A**61**, C141-C142

High Brilliance X-ray Laboratory System for Microdiffraction Studies

Oleg.V. Mikhin, Victor S. Ozerov, Alexey V. Priladyshev, *Institute for Roentgen Optics, Moscow, 1st Volokolamski pr.10, Russia.* E-mail: mov@iroptic.ru

An X-ray laboratory system is described optimized for diffraction studies of protein crystals and crystals under high pressure. The system is also suitable for phase contrast analysis, small-angle scattering and other investigations.

The system is based on a micro-focus X-ray source of 50W