PS01.04.10 TIME-RESOLVED PROTEIN DATA COLLECTION SYSTEM WITH LARGE IMAGING PLATE. K.Sakabe, N.Kamiya[1], N. Watanabe[2], S. Adachi[1], K. Sasaki[3], S.Ikemizu[4], T.Higashi[5], & N. Sakabe[4], Dept. of Chem. ,Nagoya Univ., Chikusa, Nagoya, 464 Japan, [1]RIKEN, Hirosawa 2- 1, Wako, Saitama, 351 01 Japan, [2]PF, KEK, Tsukuba 305 Japan, [3]College of Medical Technology, Nagoya Univ. Higashi, Nagoya, 461, [4]Institute of Applied Biochem.,Univ. of Tsukuba, Tsukuba ,Ibaraki 305 Japan, [5]Rigaku Corporation Matsubara, Akishima, Tokyo 196 Japan

We have developed a data collection system which can fit for both time resolved Laue and LOT, and name it as time-resolved camera system. Typical nature of this camera for time-resolved Laue is that imaging plate cassette with 800x800mm ditector area can be moved quickly while rotating fast shutter is chopping the X-ray to get m-sec order of time resolution spots whose images are aligned along horizontal direction. Chopping is better than the streaks as following reasons; 1. Easy to get accurate integrate intensity data. 2. Back ground can be reduced extensively. 3 To reduce the dose of X-ray to the crystal and to reduce the crystal damage. The evaluation of this system has been done by crystal of ω-amino acid aminotransferase whose space group I222 and cell dimensions are a=137.9, b=124.7, and c=61.5A The normal Laue data collected from 42 frams with 400x800mm. The recovery is 62% within 2Å resolution. The R (I) is 8.4%. Time resolved Laue data was collected from three shots at 2mm interval. The R factor(I) between three spots is 0.07 for 3580 reflection in a frame which corresponds to 75% of single spots exposure in the same condition. Time-resolved expriment using LOT has been done by this system switching to as the Weissenberg camera In the case of tetragonal lysozyme using flow cell, independent data up to 1.8Å resolution has been collected within 15 min with two frames. Rmerge(I) is 0.045.

PS01.04.11 ROTATING ANODE/AREA DETECTOR DATA COLLECTION ON AXES>300 Å AND NEW, RAPID CALIBRATION METHOD. James C. Phillips, Siemens Energy and Automation, Inc. Analytical Instrumentation, 6300 Enterprise Lane, Madison, WI 53719-1173

Crystallographers are investigating ever larger unit cells. HI-STAR with high resolution mode and Dual HI-STAR systems were designed to meet this challenge. The Siemens HI-STAR multiwire area detector has previously been calibrated with an Fe^{55} radioactive source, as have the X100 and the X1000, earlier models. This procedure has taken several hours when the system is configured for large unit cells. However, the new calibration method, using amorphous Iron foil placed at the crystal position and generating fluorescence by irradiating it with CuKα radiation is much faster.For example, using a dual HI-STAR at 300/452 mm from the sample, on a rotating anode generator calibrations were done for both detectors simultaneously for flood field (15 minutes) and spatial (15 minutes). The 300 mm detector was in 1024x1024 pixel mode, the 452 mm detector was in 512x152 mode. An intensity comparison was made with a 2.92 year old 100μC Fe^{55} source (reduced to 46% of original activity) for the dual detector configuration above. Foil count rates were 43 times stronger for the 300 mm, 46 times for the 452 mm. With calibration by this method data was collected on a crystal with a 346 Å axis and one 400 Å. The cell parameters and the statistics of the data integrated by SAINT clearly show the power of the dual HI-STAR system for these "front-line" experiments and that calibrations are accurate as well as convenient.

PS01.04.12 AN ANALYSIS OF DATA COLLECTION STRATEGIES AND DATA REDUCTION SOFTWARE FOR IMAGE PLATE DATA. Bing Hu[1], John Rose[2] and Bi-Cheng. Wang[2], [1]Dept. of Crystallography, University of Pittsburgh, Pittsburgh, PA 15260, U.S.A., [2]Dept. of Biochemistry & Molecular Biology, University Georgia, Athens, GA 30602, U.S.A.

In designing a strategy for macromolecular data collection, one of the most often asked questions is "What scan range should I use?". The answer may vary depending on the facilities used for data collection and the programs used for data processing. Intituitively, narrow oscillation data slices (0.1-0.25°) should give a better signal-to-noise ratio than large oscillation data slices (say, 0.5-2.0°). However most image plate data is collected using a large oscillation data slice due in part to the slow readout time of the commerical detectors, the deacy of diffraction quality with time and the initial lack of data reduction software for narrow oscillation data slices. In addition, there appears to be no reported systematic study on the relation of scan range to data quality for image plate data which prompted us to do a systematic study on this subject. Since data quality may also be affected by the techniques (e.g. 2D versus 3D profile fitting) used in the data reduction, we have included data quality versus data reduction software used as part of our study.

The data used in this study was a 2.6Å set of 0.25° oscillation data collected at room temperature using a Mar Research 30 cm image plate scanner on crystals of the Neurophysin-hydrin I complex (space group $P4_12_12$, a=b=68.7Å and c=113.64Å). The 0.25° images were combined to form the 0.5°, 0.75°, 1.00°, 1.25° and 1.5° used in the analysis. Each data set was then processed using X-GEN, XDS, MOSFILM and DENZO. The results of our analysis will be presented.

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Synchrotron Radiation III - Applications Time Resolved Micro-crystal High Energy

MS01.05.01 STUDIES OF THE SYNTHESIS, AND TRANSFORMATION OF MATERIALS USING IN SITU TIME-RESOLVED POWDER DIFFRACTION. J. C. Hanson, J. Aruajo, P. Norby, Brookhaven Nat. Lab., USA; A. N. Christensen, Aarhus Univ., Denmark; K. Ståhl, DTU, Lyngby, Denmark; G. Artioli, Univ. of Milan, Italy; A. Gualtieri, Univ. of Modena, Italy.

Time-resolved synchrotron powder diffraction data will be presented that have been collected in situ with position sensitive detectors in order to study the kinetics of hydrothermal syntheses[1] and phase changes[2].

The hydrothermal syntheses of Co and Mg substituted alumino phosphates have been found to form many different MAPO frameworks depending on the template, pH and hydrothermal conditions.

The phase transformations of KNO_3 in the range 303 to 533K have been shown to be dependent on the thermal history of the sample.

We have also found that the in-situ time-resolved data can be used for Rietveld profile refinements[3]. For example, the study of laumontite[4] offers great insight on the fine details of water molecules-cation-framework oxygen atoms interaction during the thermally driven release of the water molecules from the zeolitic channels. A molecular movie showing the steps in the dehydration of laumontite is available on the WWW at http://www.chemistry.bnl.gov/x7b/x7bhome.html. In stilbite, the site distribution of the water molecules is more complex, but the study clearly shows the temperature-dependent shift of the cations towards the framework oxygens, finally leading to a first-order phase

transition with change of space group symmetry, and the systematic rearrangement of T-O-T bonds in the high-temperature phase. To our knowledge, this is the first direct experimental evidence of a zeolite framework disruption during dehydration caused by increased cation coordination to framework oxygens.

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MS01.05.02 THE EFFECT OF VARIOUS NUCLEATING AGENTS UPON THE CRYSTALLISATION OF CORDIERITE GLASS CERAMIC. S. M. Clark, G. N. Greaves, M. Oversluzien[1], G. Sankar[2], J. M. Thomas[2]. CCLRC, Daresbury Laboratory, Warrington, WA4 4AD, UK, [1]Netherlands Organisation for Scientific Research, The Hague, The Netherlands, [2]Royal Institution of Great Britain, 21 Albermarle Street, London, W1X 4BS, UK

Cordierite glass ceramics are of considerable relevance to the electronics packaging industry due to their high dielectric constant and their low coefficient of thermal expansion. Interest has also been shown in their optical properties for use in tunable lasers and solar concentrators. They are presently being produced in bulk quantities for use as catalyst supports in catalytic converters for car exhaust systems.

Cordierite glass ceramics can be made by heating a glass of the appropriate stoichiometry ($Mg_2Al_4Si_5O_{18}$) above the glass transition temperature. An intermediate crystalline phase known as μ-cordierite is first formed as the final product alpha cordierite crystallises.

We have studied this reaction using time resolved powder diffraction, EXAFS and small angle scattering as well as conventional static measurements. The results of a detailed kinetic analysis will be presented together with a study of the effect of adding a number of nucleating agents to the reaction mixture.

An extension of the classical kinetic equations governing series reactions will also be presented.

MS01.05.03 HIGH RESOLUTION SINGLE CRYSTAL DIFFRACTION USING SYNCHROTRON RADIATION H. Graafsma, O. Svensson, A. Kvick, European Synchrotron Radiation Facility, BP 220 38043 Grenoble, France

The advantages of synhrotron radiation for high resolution single crystal diffraction, e.g. for electron density distributions,are well known [1]. The high energies reduce systematic errors such as absorption and primary extinction. The same is achieved by using small samples, possible due to the high flux at the sample. This high flux also permits the measurement of higher order data, as well as weak reflections [2]. An other advantage beside accuracy is the high speed. The combination of 2D-detectors and the high flux at the sample allows to record a full data set within hours. The results of two measurements at 56 keV (0.22 Å), performed at the materials science beamline of the ESRF will be presented. The first is the determination of the electron density of Magnesium Formate Dihydrate, using a Princeton Instruments slow scan CCD coupled to an Image Intensifier. The second is an electron density study of Amonium Dihydrogen Phosphate (ADP) using the SIEMENS SMART system. Both measurements gave an R-int of the order of 3%, with data extending to sin θ/λ=1.4. The first data set was measured in 2 hr, using an oscillation per frame of 4 degrees. The data was integrated both with DENZO and the Seedskewness package. The second data set was obtained in 9 hours using an oscillation of 0.05 degrees per frame, and integrated with the SIEMENS SAINT program using 3-dimensional profile fitting.

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MS01.05.04 STRUCTURE DETERMINATION OF MICROMETER AND SUBMICROMETER SINGLE CRYSTALS WITH SYNCHROTRON RADIATION. Neder, R. B., Burghammer, M., Grasl, T., Schulz, H., Institut für Kristallographie und Mineralogie, Universität München, Theresienstr.41, 80333 München, Germany

We have mounted individual submicrometer sized single crystals of kaolinite to thin glass fibers. We developed a novel micromanipulator for usage within a scanning electron microscope. This micromanipulator uses a combination of stepper motor controlled microtranslation units with piezo drives and is capable of nanometer resolution. The glass fiber supports are pulled from massive glass rods to a diameter of 0.5 μm.

The sample volume is estimated at < 0.1 μm^3. Diffraction experiments are carried out at the microfocus beamline, ESRF, under vacuum conditions. The combination of a vacuum chamber and submicrometer sized sample-supports effectively reduces the experimental background.

As another example we present the results of the determination of hydrogen positions from a hydrous barium oxalate. Despite the presence of a heavy metal, the positions, as well as, thermal parameters of the hydrogen could be refined.

A third example is optically anomalous topaz. An individual microcrystal was prepared from within an optical zone. Single crystal diffraction experiments at the ESRF were carried out on a crystal of 2 μm^3 volume.

MS01.05.05 APPLICATION OF MICRO-BEAM TO MINERALS IN A THIN SECTION OF METEORITE AND STRUCTURE REFINEMENT. K. Ohsumi[1], M. Uchida[2], K. Hagiya[3], M. Miyamoto[4] and M. Ohmasa[3]. [1]Photon Factory, National Laboratory for High Energy Physics, Japan; [2]Synchrotron Radi. Sci., Graduate School for Advanced Studies, Japan; [3]Department of Life Science, Himeji Institute of Technology, Japan; [4]Mineralogical Insti.,Graduate School of Science, Univ. of Tokyo, Japan.

Polychromatic SR microbeam with diameter of 1.6 μm and with divergence of 40 μrad. was produced by a micro-pinhole technique for structure refinement using Laue method. This size of micro-area on the sample is comparable to those examined by optical microscope, scanning electron microscope, electron prove micro analyzer (EPMA), Raman spectroscopy and so on. The X-ray diffraction method with this micro-pinhole provides crystallographic information from the exact same micro-area as analyzed by various methods mentioned above. The micro-pinhole was installed in the Laue camera which was developed at the beam line 4B of the Photon Factory, KEK[1]). One of the application of this method is given below.

In crystallographic studies of meteorite which must be affected by shock as presumed from its origin, the microbeam is indispensable to search micro-area that is good enough for structure