

**PS19.00.01 HIGH TEMPERATURE FURNACE ON AN IMAGING PLATE SCANNER SYSTEM.** A. Baumgarte, J. Schreuer, M. A. Estermann and W. Steurer, Laboratory of Crystallography, ETH Zentrum, CH-8092 Zurich, Switzerland

A compact furnace operating up to 1500K under helium atmosphere for an imaging plate scanner system (marresearch) was developed to record large amounts of reciprocal space at elevated temperatures [1]. The complex scattering phenomena connected with the phase transitions of decagonal quasicrystals and corresponding approximant phases in the Al-Co-Ni system will be presented.

The heating chamber consists of a watercooled cylindrical tube with radiation windows of Kapton foil, a rotatable feedthrough to connect the spindle axis with the goniometer and a heating element of two winded Kanthal wires above and below the sample, so that up to  $2\Theta=37^\circ$  no restriction for data recording results. The heating element, including radiation shields and an internal collimation system, can be rotated and translated along the phi-axis which allows for *in situ* observation of the crystal by the integrated CCD-camera of the scanner system.

For the high temperature heat measurement we developed a new kind of sample holder. The crystal is clamped between 4-6 polycrystalline aluminium oxide fibres of 10 microns in diameter.

The practicability of the furnace is demonstrated with five different Al-Co-Ni crystals with decaprismatic morphology which undergo several phase transitions between ambient temperature and 1500K. Diffuse scattering and different types of satellite reflections were observed.

[1] J. Schreuer, A. Baumgarte, M. A. Estermann, W. Steurer and H. Reifler. *Journal of Applied Crystallography* (1996), in press.

**PS19.00.02 GROWING CRYSTALS FROM LIQUIDS AND GASES.** Alexander J. Blake, Department of Chemistry, The University of Nottingham, University Park, Nottingham NG7 2RD, UK and Norbert W. Mitzel, Simon Parsons and David W.H. Rankin, Department of Chemistry, The University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, UK

A survey of the structural databases reveals a basic discontinuity in known crystal structures: those with melting points below ambient temperatures, which are often compounds of great interest, are very poorly represented. The reason for this is obvious and practical-obtaining single crystals of compounds which are liquid or gaseous at room temperature can be orders of magnitude more difficult and labourintensive than for compounds which are crystalline solids under the same conditions.

Despite these problems, we have succeeded with a wide range of materials, both organic and inorganic, including semiconductor precursors, 1:1 adducts and compounds of fluorine, germanium, indium, phosphorus, sulphur and silicon. Although a few of these compounds could be handled using standard techniques oil drop techniques (Kotke & Stalke, 1993) or adaptations of these, the low melting points and reactivity of most of them demand *in situ* growth from samples pre-sealed into Pyrex capillary tubes.

Recent results on some silicon and germanium compounds will be reported.

Reference: Kotke, T. & Stalke, D.(1993). *J. Appl. Cryst.* 26, 615-619.

**PS19.00.03 LOW TEMPERATURE SINGLE CRYSTAL DIFFRACTION AT THE SUNY X3 BEAMLINE AT THE NATIONAL SYNCHROTRON LIGHT SOURCE.** Alex Darovsky, Robert Bolotovskiy, Vladimir Kezerashvili and Philip Coppens. SUNY X3 beamline at the National Synchrotron Light Source, Brookhaven National Laboratory, Upton, NY 11973 and Chemistry Department, State University of New York at Buffalo, Buffalo, NY 14260, USA

We have routinely collected large low-temperature imaging plate data sets at the X3A1 station at the SUNY X3 beamline at NSLS, using a Displex® cryostat equipped with a special antiscatter device to minimize background (1). Sample temperatures down to ~17K can be reached. Diffraction data are processed with the "seed-skewness" method of peak integration (2). The performance of the "seed-skewness" method was evaluated by a numerical simulation technique. The equipment was used for electron density studies of the transition metal complexes  $\text{Na}_2[\text{Fe}(\text{CN})_5\text{NO} \cdot 2\text{H}_2\text{O}]$  and  $\text{Cr}(\text{CN})_6\text{Cr}(\text{NH}_3)_3$  and for low-temperature structural studies. In favorable cases reflections up to  $2.0\text{\AA}^{-1}$  have been measured. The optimal data collection strategy as well as the detailed experimental arrangement will be discussed, and comparisons with a gas-flow system will be made.

1. A. Darovsky, R. Bolotovskiy and P. Coppens, *J. Appl. Cryst.* 27, 1039 (1994).
2. R. Bolotovskiy, M. A. White, A. Darovsky and P. Coppens, *J. Appl. Cryst.* 28, 86 (1995).

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**PS19.00.04 LOW TEMPERATURE X-RAY CRYSTALLOGRAPHY OF MAGNETS IN LOW MAGNETIC FIELDS.** V.A.Finkel, National Science Center, Kharkov Institute of Physics & Technology, Kharkov, UA 310108, Ukraine

The aim of this paper is the demonstration of the possibility of XRD investigation of some structural aspects of magnetic phase transitions and domain structure rearrangements in the temperature range of 80 - 300 K in a low magnetic field up to 1000 Oe. The model objects of the investigation were single crystals of the rare earth metals (REM) Dysprosium and Terbium. We have measured the lattice parameters and diffraction line intensities as functions of a magnitude and a direction of an external magnetic field *H* at a constant temperature or as functions of a temperature at a constant field. We have obtained some new results concerning the magnetic phase H-T diagrams and the evolution of the REM domain structure in the ferromagnetic state in a magnetic field in a wide temperature range. In particular, we discovered that the type of H-T diagrams of REM essentially depends on the vector *H* orientation concerning crystallographic axes. We determined the presence of regions of the magneto heterogeneous state "helical anti ferromagnetism-collinear ferromagnetism", the magnetic vortex state, and the magnetic fan state on the H-T diagrams of REM. Also we ascertained that at a temperature reduction and a magnetic field rise the main types of domain walls in a ferromagnetic state of Dy and Tb are 120 deg walls.