CRYOGENIC MOUNTING, ARCHIVING AND TRANSPORTATION OF BIOLOGICAL MACROMOLECULAR CRYSTALS. Sean Parke, Bernhard Rupp and Håkon Hope. 1BRRP, L-452, Lawrence Livermore National Laboratory, Livermore, CA, 94550. 2Department of Chemistry, University of California, Davis, CA, 95616.

Cryogenic data collection for macromolecular crystals is increasing in popularity as a means of improving data quality. The advantages include the postponement of radiation damage, elimination of errors arising from inter-crystal scaling and merging, reduced thermal motion, enhanced contrast in electron density maps, and a possible reduction in conformational disorder. Crystals are generally cooled after modification of the aqueous layer surrounding the surface of the crystal, either by some antifreeze (cryoprotectant) or oil. When a crystal is sufficiently robust that it can survive cooling by being placed directly in a cold gas stream, that should be the cooling method of choice. Often, however, successful crystal cooling requires that the cooling operation be as rapid as possible. A currently popular myth dictates that the fastest transition between room and cryogenic temperatures is achieved by dunking the crystal in liquid propane. In reality, dunking in liquid nitrogen provides faster cooling and is not prone to the problems of bubble formation that are known to plague larger samples, as is seen in electron microscopy. Safety concerns surrounding the use of open containers of liquid propane in close proximity to electrical equipment are also an issue with liquid nitrogen.

Tools and techniques are presented for the easy manipulation, mounting, dismounting, archiving and transportation of sensitive crystals based on immersion in liquid nitrogen. These techniques have been in routine operation in our laboratories for several years now. They are simple to learn and above all they are reproducible and safe under all circumstances. The temperature history of a crystal during the full procedure of mounting, dismounting, transfer to a storage dewar and remounting has been measured and it is shown that it never rises above the temperature of the warmest component of the whole cryogenic system, namely the cold gas stream. These procedures now form the preferred modus operandi at the Stanford Synchrotron Radiation Laboratory (SSRL).

CRYOCRYSTALLOGRAPHY OF 3-ISOPROPYLMALATE DEHYDROGENASES AT 100 AND 150K. Nobuo Tanaka, Chikahiro Nagata, Hideaki Moriyama, Tairo Ueki, 2lOT, Lawrence Livermore National Laboratory, Livermore, CA, 94550. 2Deparment of Chemistry, University of California, Davis, CA, 95616.

In the pathway of biosynthesis of leucine, 3-isopropylmalate dehydrogenase (IPMDH) catalyses the conversion from 3-isopropylmalate to 3-ketocaproate through the dehydrogenation and decarboxylation. It was found that its chimeric enzymes isolated from Thermus thermophilus, 10T, and its thermostable chimeric mutant, 2T2M6T-S82R, at 100 and at 150K, to observe the thermal behavior with the suppressed temperature factor. The diffraction data was collected from the cryo-cooled crystals on the R-AXIS IIC according to the reported method (1). Positional deviations of the domains were detected in the subunit, though the overall structures of both enzymes were similar to the structures at room temperature. Observed shrinkage of the molecules, that calculated from the atomic coordinates, was found larger in the chimera than in the 10T. The overall temperature factors were almost the same in the structures at 100K and at 150K, although those decreased very much from that of the room temperature. The 2T2M6T-S82R showed the overall temperature factor decreased monotonically with the temperature. The extrapolation of temperature factors at higher temperature indicated that the chimera vibrates more than the 10T, which may be a cause of temperature sensitivity of the chimera molecule.


SYNCHROTRON RADIATION I
Instrumentation and Techniques

HIGH ENERGY X-RAY SCATTERING VS. NEUTRON DIFFRACTION. J.R. Schneider, HASYLAB at DESY, Notkestr. 85, D-22603 Hamburg.

Diffraction experiments with synchrotron radiation of wavelength $\lambda=0.15\AA$ provide a new probe in condensed matter research because they combine the high penetration power of thermal neutrons with the extreme momentum space resolution obtained in modern X-ray diffraction experiments. After a short presentation of the diffractometers installed at the high field wiggler beamline BW5 at DORIS III and the undulator beamline at the PETRA storage ring at HASYLAB, examples will be discussed which highlight the power of combining the results of such synchrotron radiation experiments with those obtained by neutron scattering. The gain in information achieved this way is due to the different intrinsic cross sections involved and the possibility to perform high resolution inelastic neutron scattering experiments on the identical samples.

The static structure factor of amorphous materials, $S(Q)$, can be measured accurately up to very high momentum transfer both with neutrons and high energy synchrotron radiation. The partial distributions functions calculated from the combined data sets provide more structural informations than each individual measurement.

The superstructures of YBCO high $T_c$ superconductors have been studied with a high resolution triple-crystal diffractometer for high energy synchrotron radiation. The intensity gain of 3 orders of magnitude compared to neutron diffraction studies performed on the same sample allowed for novel quenching experiments. Analysis of the diffuse scattering in the vicinity of the four fold degenerate 200/020 reflections provided information on the size of the domains and the domain walls.

Different from neutron scattering, with X-rays two length scales have been observed in the critical fluctuations above the second order cubic to tetragonal phase transition in SrTiO$_3$. In order to find out if this unexpected behaviour is a bulk property or bound to the surface near region probed by X-rays, the critical scattering has been studied with 100 keV synchrotron radiation on the identical sample as used in the neutron studies. The critical scattering from the bulk is well described by a simple Lorentzian and also its temperature dependence fully agrees with the neutron data. However, a sharp component has been observed in the critical scattering in a surface near layer of 65 $\mu$m thickness.