pressure, and it will be demonstrated that the guest-atom chains in the composite Rb-IV structure represent a realisation of the classic monatomic linear chain model. In modulated Te-III, a pronounced phonon anomaly is observed that will be discussed in the context of Fermi-surface nesting, Kohn anomalies and charge-density waves.
This work was performed in collaboration with M. I. McMahon, L. F. Lundegaard, S. R. Evans (University of Edinburgh) and A. Bossak, M. Krisch (ESRF, Grenoble).

Keywords: incommensurate crystals, lattice dynamics, inelastic X-ray scattering

MS.56.5

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Phase transformations in silane － Hydrogen-dominant material

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The metallization of hydrogen directly would require pressure of 400 GPa, out of the reach of present experimental techniques. The dense group IVa hydrides attract considerable attention because hydrogen in these compounds is chemically precompressed and a metallic state is expected to be achievable at experimentally accessible pressures(1). We report the transformation of insulating molecular silane to a metal at 50 GPa, becoming superconducting at a transition temperature of Tc = 17 K at 96 and 120 GPa(2). The metallic phase has a hexagonal close-packed structure with a high density of atomic hydrogen, creating a three-dimensional conducting network. These experimental findings support the idea of modeling metallic hydrogen with hydrogen-rich alloy. The metallic $P6_3$ phase is apparently stable in the 50 to 110 GPa range. However it partly transforms into a transparent insulating phase at pressures ≥120 GPa. Concurrent with the onset of transparency, a pronounced Raman signal appears. Two distinct phases coexist to the highest experimental pressure of 192 GPa. We determined the transparent phase structure to be a $I4_1/a$ structure. The structure and its lattice parameters are in exact agreement with the $I4_1/a$ phase predicted to be thermodynamically stable in the pressure range 50 to 250 GPa(3). The observed transformations in stoichiometric silane with pressure are unusual. Molecular SiH_4 at a pressure of ≈50 GPa does not transform to the predicted thermodynamically stable $I4_1/a$ phase(3) but instead collapses to a significantly denser $P6_3$ phase.
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Keywords: metallic hydrogen, high pressure, hydride

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Structure of drug-target proteins determined by both X-ray and neutron diffraction

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Crystallography enables us to obtain accurate atomic positions within proteins. High resolution X-ray crystallography provides information for most of the atoms comprising a protein, with the exception of hydrogens. Neutron diffraction data can provide information of the location of hydrogen atoms to the structural information determined by X-ray crystallography. Here, we show the recent result of the structural determination of drug-target proteins, porcine pancreatic elastase (PPE) and human immunodeficiency virus type-1 protease (HIV-PR) by both X-ray and neutron diffraction. The structure of porcine pancreatic elastase with its potent inhibitor (FR13080) was determined to 0.94Å resolution by X-ray diffraction and 1.75 Å resolution by neutron diffraction. It was found that there are two characteristic hydrogen bonding interactions in which hydrogen atoms were confirmed. One is located between a catalytic aspartate and histidine, another is involved in the inhibitor recognition site. The structure of HIV-PR with its potent inhibitor (KNI-272) was also determined to 0.93 Å resolution by X-ray diffraction and 2.3 Å resolution by neutron diffraction. The ionization state of the catalytic residues were clarified to show that Asp125 is protonated and Asp25 is deprotonated. The ionization state and the location of hydrogen atoms of the catalytic residue in HIV-PR were firstly determined by neutron diffraction. Furthermore, collaborative use of both X-ray and neutron to identify the location of ambiguous hydrogen atoms will be shown.

Keywords: neutron diffraction, protein, structure

MS.57.2

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Neutron crystallographic analysis of deuterated and selectively CH_3-protonated deuterated rubredoxin

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Neutron crystallography is used to locate hydrogen atoms in biological materials and can distinguish between negatively scattering hydrogen and positively scattering deuterium substituted positions in isomorphous neutron structures. Recently, Hauptman and Lang (2003) have shown that neutron diffraction data can be used to solve macromolecular structures by direct methods and that solution is aided by the presence of negatively scattering hydrogen atoms in the structure. Selective labeling protocols allow the design and production of group or residue specific H/D-labeled macromolecular structures in which the ratio of hydrogen to deuterium atoms can be precisely controlled. We have applied labeling protocols to selectively introduce protonated methyl groups into deuterated rubredoxin from Pyrococcus furiosus (PfRd). Perdeuterated and selectively CH_3-protonated, deuterated rubredoxin were crystallized. High quality neutron data sets extending to 1.75 Å resolution were collected on the new LADI-III instrument at the Institut Laue-Langevin. Of special importance, the 1.75Å data from the perdeuterated crystal required just 14 hours of beam time, a record which heralds a new era in neutron protein crystallography. We will present the production, crystallization, and neutron analysis of the perdeuterated and selectively CH_3-protonated PfRd.
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Keywords: neutron macromolecule crystallography, specific labeling, proton

MS.57.3

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Neutron macromolecular crystallography using the Laue diffractometer LADI-III

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At the Institut Laue-Langevin a new neutron Laue diffractometer (LADI-III) has been fully operational since March 2007. LADI-III is dedicated to neutron macromolecular crystallography at high-resolution (1.5 Å - 2.5 Å) and is used to study key hydrogen atoms and water structure in macromolecular structures. An improved detector design and readout system has been incorporated so that a miniaturized reading head located inside the drum scans the image-plate. From comparisons of neutron detection efficiency (DQE) with the original LADI-I instrument, the transferal of the image-plates and readout system internally provides a 2- to 3-fold gain in neutron detection, allowing data collection to higher resolution (~1.5 Å), using shorter exposure times and smaller crystal volumes. The improved neutron detector efficiency of LADI-III combined with the use of perdeuterated biological samples has enabled neutron macromolecular crystallography to become more accessible to the structural biology community, extending the size and complexity of systems that can be studied (~150 Å on cell edge) while lowering the sample volumes required (~0.1mm^3). Current projects aim to address questions concerning enzymatic mechanism, ligand-binding interactions, solvent effects, structure dynamics and their implications. Examples of recent project highlights from LADI-III will be presented.

Keywords: neutron macromolecular crystallography, perdeuteration, Laue

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Seeing hydrogens: X-ray limitations and possibilities at 0.9 Å and synergy with neutron diffraction

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Protons and proton transfer pathways play a critical role in many enzyme mechanisms. Direct information on proton (hydrogen) positions may be obtained from neutron crystallographic data. Despite advances in neutron sources and detectors, the application of neutron diffraction has remained limited to large crystals with small unit cell dimensions. This has resulted in general in a paucity of information about the proton delivery mechanisms that are central to our understanding of many enzyme mechanisms. In our attempt to understand the underlying mechanism of proton transfer, we have adopted the approach of obtaining X-ray data to subatomic (<1 Å) resolution. Electron density maps from structures determined to such resolution can reveal the positions of crucial hydrogens within the active sites and on proton pathways. Details of hydrogen location in green nitrite reductase from *Achromobacter cycloclastes* (0.90 Å) [1], human superoxide dismutase (0.88 Å) [2] and cytochrome c' (CYTcp) from *Alcaligenes xylosoxidans* will be presented. The last of these, CYTcp is able to discriminate between CO and NO by binding them on opposite faces of the heme. Recently, we have determined the X-ray crystal structure of oxidised and reduced CYTcp to 0.9 Å resolution and a detailed analysis of the structures is in progress. Crystals of CYTcp have been grown to a size of 3000x500x500 microns and neutron diffraction studies are in progress. These data will allow us to explore the synergy between the two approaches for this important system.

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Keywords: neutron crystallography, high-resolution X-ray crystallography, metalloproteins

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Locating hydrogen atoms in enzymes: A neutron structure of D-xylose isomerase with bound D-xylulose

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The transfer of hydrogen is important in many enzyme reaction mechanisms, and yet hydrogen is difficult to visualize in proteins using X-ray crystallography because of its low X-ray scattering power. We have exploited the recently developed time-of-flight neutron Laue technique to determine the location of hydrogen atoms during the reaction of an enzyme of economic importance, D-xylose isomerase (XI). Neutron diffraction studies of this deuterated enzyme with bound perdeuterated substrate show, unexpectedly, that the terminal O5 hydroxyl group of D-xylulose, the product, is not protonated but is H-bonded to doubly protonated His54 in a sugar hydroxyl-histidine-aspartate triad such as is found in trypsin. In addition, the metal ion-bound water molecule (which may be important in the enzyme mechanism) that was found in neutron diffraction studies of the native enzyme, is deprotonated in this XI-xylulose structure and exists as a hydroxyl group. Furthermore, lysine 289, which has only two protons on its terminal nitrogen atom (NZ) in the native enzyme neutron structure, has three in the XI-xylulose complex. This indicates that there is a positive charge on this amino group. These findings lead to further insight into the mode of action of this enzyme. Support from the National Institutes of Health (GM071939, CA06927, CA10925), the Office of Biological and Environmental Research of the Department of Energy, and Los Alamos National Laboratory (20070131ER and 20080001DR) is gratefully acknowledged.

Keywords: neutron structure analysis, enzyme structure function, isomerases