

**P.11.14.2***Acta Cryst.* (2005). A61, C402**A New, Rapid 3D Tomographic Energy Dispersive Diffraction Imaging System for Materials Characterisation and Object Imaging (Rapid TEDDI)**Kern Khor, Robert Cernik, *School of Materials, Manchester, UK and Daresbury Laboratory, Warrington, UK.* E-mail: r.j.cernik@dl.ac.uk

In 1998 Hall et al [1] demonstrated that the interior features of solid objects could be very effectively and simply imaged in a non-destructive manner using synchrotron energy dispersive diffraction. They were able to demonstrate big improvements over the measurements obtained by Harding [2] who used conventional rotating anode X-ray sources. Both papers demonstrate the huge potential of (TEDDI) in materials science. Although there are many forms of tomographic imaging, virtually all rely on absorptive or spectroscopic responses of a material object to invading radiation. By contrast TEDDI is unique in using both diffraction and absorption or diffraction and spectroscopic data. A white beam from a synchrotron or laboratory X-ray source is collimated to the desired spatial resolution. The small diffracting sample volume is defined by the track of the incident and scattered beams through the sample and the angle subtended by the collimator aperture. The sample is scanned in 3 dimensions in small steps. This is, however, a very time consuming process. As a consequence we are developing array solid state detectors with corresponding collimator arrays that can reduce the time for object scanning from 14 hours to a few minutes. In this way a 3D density contrast map will be obtained with full structural and chemical information at each voxel point. This development will be explained in more detail in the context of a wide range of diverse applications.

[1] Hall C., Barnes P., et al, *Nuclear Instruments & Methods in Physics Research Section B-beam interactions with materials and atoms*, 1998, **140**, 253-257. [2] Harding G, et al, *Physics in Medicine and Biology*, 1990, **35**, 33-41.

**Keywords:** synchrotron, materials, tomography**P.11.15.1***Acta Cryst.* (2005). A61, C402**Synthesis and Structural Characterization of Novel Metal-Organic Frameworks**Hyunsoo Park<sup>a</sup>, David M. Moureau<sup>a</sup>, John B. Parise<sup>a,b</sup>, <sup>a</sup>*Department of Chemistry, SUNY Stony Brook.* <sup>b</sup>*Department of Geosciences, SUNY Stony Brook, NY, USA.* E-mail: hypark@notes.cc.sunysb.edu

Studies of structurally and chemically diverse open framework solids have been flourishing in the recent years in an attempt to increase and improve applications of such materials in areas such as catalysis, sorption and ion-exchange. Our research is aimed at finding new 3-dimensional porous frameworks, which may possess interesting physical properties. We report the hydrothermal synthesis and structural characterization of several new metal-organic hybrid frameworks constructed from various metal cations and organic ligands of different functionalities.

For example,  $Zn_2(C_2H_2N_3)_2(C_8H_4O_4) \cdot H_2O$ , synthesized using 1,2,4-triazole and 1,4-benzenedicarboxylic acid as organic linkers, possesses a structure (P4/ncc,  $a = 13.521(2) \text{ \AA}$ ,  $c = 27.221(5) \text{ \AA}$ ,  $Z = 8$ ) based on the sheets containing 8-membered rings of  $ZnON_3$  tetrahedra and triazole molecules along the (001) direction. The sheets are connected by benzenedicarboxylates through Zn – O – C links to produce channels which are occupied by water molecules. Its thermal behavior has also been characterized by thermogravimetric analysis. It is stable up to 300°C before the organic molecules start to decompose, leading to the framework collapse.

**Keywords:** materials structure and characterization, porous materials, X-ray crystallography**P.11.15.2***Acta Cryst.* (2005). A61, C402**Spin-Crossover and Nanoporosity: Magnetic, Structural and Guest-Exchange Studies**Natasha F. Sciortino, Cameron J. Kepert, *Department of Chemistry,**University of Sydney.* E-mail: sciort\_n@chem.usyd.edu.au

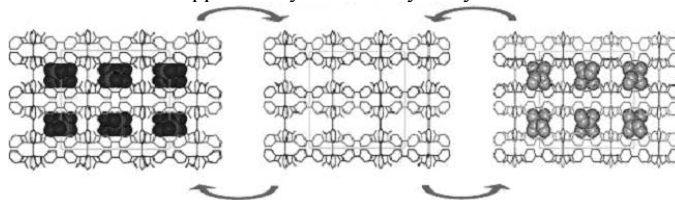
Spin-crossover and permanent nanoporosity describe two highly desirable and traditionally independent properties concerning the design, synthesis and characterisation of functional coordination framework materials. By integrating these properties, we are developing a new series of *smart* materials capable of a physical response to their environment. Such capabilities include guest-induced electronic, magnetic and optical switching with applications towards chemical sensors, molecular switches and data storage materials.

The new network  $[Fe(dps)_2Ag(CN)_2ClO_4] \cdot 3(C_2H_6O)$  (**1**) ( $dps = 4,4'$ -dipyridylsulfide) is the first three-dimensional framework to display a synergy of spin-crossover and nanoporosity (~46% void volume). Variable-temperature single-crystal X-ray diffraction (SCXRD) techniques were employed over the range 90 K–300 K to elucidate the structural consequences of a reversible low-spin to high-spin transition. Modified *in-situ* SCXRD techniques were used to observe the implications of guest-exchange on structural integrity and spin-crossover functionality.

Several unique properties of **1** have been demonstrated: 1) complete spin transition, incurring a 7.7% crystal volume change; 2) robustness to desorption at 375 K, with complete retention of structural integrity; and 3) sensitivity of spin-crossover to the presence of guest species. Additional guest-exchange studies also establish **1** to be a highly nanoporous framework that exhibits rapid, reversible and complete sorption/desorption of a range of small molecules.

**Keywords:** spin-crossover, nanoporosity, *in situ* SCXRD**P.11.15.3***Acta Cryst.* (2005). A61, C402**In situ Control of Guest Exchange in Single Crystal X-ray Diffraction**Joseph J. Bevitt, Cameron J. Kepert, *School of Chemistry, The University of Sydney.* E-mail: bevitt\_j@chem.usyd.edu.au

By variation of template, with novel *in-situ* variable temperature Single Crystal X-ray Diffraction techniques we demonstrate the reversible nature of guest uptake by a coordination polymer, confirming their analogy with zeolites. Our work also demonstrates the subtle changes imposed on the host framework due to various guests (including 1-propanol, carbon dioxide, dinitrogen and argon) and their occupancies, quantifying the extent of host-guest interactions. Host distortions include asymmetric layer shifts of up to 0.7 Å, framework 'scissoring', ligand torsion angle changes, changes in nitrate binding and host symmetry. We present our techniques and demonstrate their applicability to the study of dynamic structures.



**Figure:** Reversible exchange of guests into the evacuated framework is achieved through repeated heating and cooling of sample under guest atmospheres.

**Keywords:** guest exchange, *in-situ* experiments, gas sorption**P.11.15.4***Acta Cryst.* (2005). A61, C402-C403**Two new Inorganic-organic Hybrids with Framework Gallium 1,2-ethylenediphosphonates Containing Pyridine**Zhanhui Yuan<sup>a</sup>, William Clegg<sup>a</sup>, Martin Attfield<sup>b</sup>, <sup>a</sup>*School of Natural Sciences - Chemistry, University of Newcastle upon Tyne, UK.* <sup>b</sup>*Centre for Microporous Materials, University of Manchester, UK.* E-mail: zhanhui.yuan@ncl.ac.uk

This paper reports the synthesis of two new open framework gallium 1,2-ethylenediphosphonate materials which contain open zeolite like structure linked to form a completely novel type of open framework architecture with interconnecting channels. The new materials are templated by pyridine. As far as we know, the new