

structure $[\text{Ga}_3\text{F}(\text{O}_3\text{PC}_2\text{H}_4\text{PO}_3)_2](\text{C}_5\text{H}_5\text{N})$ (**I**) is not currently known, and the nitrogen atom from the pyridine molecular has coordinated with metal gallium to form strong N-Ga bonds in an octahedral GaO_4FN unit, and pyridine molecules sit in the channels which are formed by building unit, connected by diphosphonate. The structure $[\text{Ga}_4\text{F}_2(\text{O}_3\text{PC}_2\text{H}_4\text{PO}_3)_3]^{2-} 2[\text{C}_5\text{H}_6\text{N}]^+$ (**II**) contains pyridine cations within the channels formed by building unit, connected by diphosphonate and has a stronger hydrogen contact between oxygen from diphosphonate and nitrogen from pyridine. The new structures (**I**) and (**II**) are good examples to present three dimensional structures with channels constructed by two novel building units connecting diphosphonate groups, and two kinds of pyridine moieties. One pyridine has linked through Ga-N bond and another is free as cation, which has been found in channels as a template. The two materials exhibit microporous frame-work materials with pyridine template within the pores.

Keywords: gallium 1,2-ethylenediphosphonate, framework, pyridine

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Mechanism of Phase Transition Caused by Water Absorption in FAPO-34

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It was made clear that the phase transition mechanism caused by the water absorption in FAPO-34 using the single-crystal X-ray (SCX) analysis and EXAFS Fourier transform spectra analysis.

FAPO-34 is known as the one of the Chabazite type zeolite. [1] The crystals of FAPO-34 including organic template in the framework (**as-made**) were obtained by hydrothermal synthesis method. To utilize as the water absorption material, the **as-made** crystals were baked in some temperature conditions to remove the organic template. The **as-made** crystals baked at high temperature (**HT**) caused phase transition, but the one baked at low temperature (**LT**) kept the structure after the water absorption.

As the results of SCX and EXAFS analysis, it was made clear that the specific water coordination to Fe on the framework causes the phase transition of **HT**. SCX analysis showed the direct coordination of the water to Al and Fe on the framework and the structural skew of the framework caused by the specific water coordination to Fe of **HT**. EXAFS analysis showed that Fe of **LT** is covered by something else other than water.

[1] Ristic A., Tusar N.N., Arcon I., Thibault-Starzyk F., Hanzel D., Czyniewska J., Kaucic V., *Micropor. Mesopor. Mater.*, 2002, **56**, 303-315.

Keywords: absorption material, phase transitions, structure analysis

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Direct Observation of Hydrogen Molecules adsorbed in a Coordination Polymer

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Hydrogen is a very important material for the development of a clean energy system. The use of physisorption in microporous coordination polymers is one of the most promising candidates for the hydrogen gas storage. The fundamental structural information of adsorbed H_2 molecules is indispensable for the rational synthetic strategy of these materials. Although the weakest X-ray scattering amplitude of hydrogen has made it difficult to determine the structure of H_2 molecules, we have succeeded in direct observation of H_2 molecules adsorbed in the nanochannels of the coordination polymer

by the *in-situ* synchrotron powder diffraction experiment of gas adsorption and the MEM/Rietveld charge density analysis [1].

The H_2 molecules were found to be adsorbed without any chemical bonding to the host framework, in the condition that they can be easily adsorbed and released. The position of H_2 molecule was displaced from the center of the nanochannel. It was located near the metal-oxygen unit near one corner of the rectangular nanochannel. The size of the H_2 molecule is suited to the size of the pocket of the cavity. The structural information of H_2 molecule at the beginning stage of gas adsorption was obtained. That gives us the guideline for the design of high performance hydrogen gas storage materials.

[1] Y. Kubota, et al., *Angew. Chem. Int. Ed.*, 2005, **44**, 290-293.

Keywords: gas storage materials, microporous coordination polymer, synchrotron powder diffraction

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Magnetic and Structural Properties of Rare Earth Hybrid Frameworks

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Aliphatic dicarboxylates (**AD**), $-\text{O}_2\text{C}(\text{CH}_2)_n\text{CO}_2-$, have shown to be excellent ligands for crystal engineering transition metal molecular magnets [1]. With the aim of extending these studies to rare earth systems we have synthesized a series of organic inorganic hybrids of Ln(III) and **AD** anions, with $1 \leq n \leq 4$, from solutions under ambient conditions. Their single crystal x ray structural analysis show the formation of inorganic polymeric arrangements of different dimensionalities. AC susceptibility, χ' , measurements performed within the temperature range $13 < T < 330\text{K}$ show noticeable changes in the $\chi'T$ curve slope at different T values. To investigate the potential structural contribution to those magnetic ordering variations, we performed single crystal x-ray analysis in the temperature ranges of interest in steps of 2K. We discuss possible links between modifications in superexchange paths connecting cations —and eventually in hydrogen bonds networks— and magnetic response.

[1] Rodríguez-Martín Y., et al., *Cryst Eng Comm*, 2002, **87**, 522.

Keywords: organic inorganic hybrid materials, framework structures, molecular magnets

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Novel Modular Coordination Frameworks

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Crystal Engineering of Coordination Frameworks has been experiencing a great growth in recent years. Despite the majority of the compounds reported in the literature could only be usually isolated in small amounts due to inherent limitations of the synthetic techniques employed, we have optimised the hydrothermal synthetic approach for the synthesis of large and phase-pure quantities of such materials [1]. N-(phosphonomethyl)iminodiacetic and etidronic acids are precursors of multidentate organic ligands which not only exhibit unusual and flexible coordination properties, but also contain a number of potentially interesting nuclei to study using, e.g., solid-state NMR techniques. We have focused our research in the *in situ* synthesis of rigid building blocks (*i.e.*, Secondary Building Units, SBUs) formed by these ligands and V^{4+} which, along with various metal centres (e.g., Co^{2+} , Mn^{2+} and Ce^{3+}) and rod-like bridging organic molecules (such as 4,4'-bipyridine and pyrazine), self-assemble into peculiar frameworks of various topologies and dimensionalities [2], [3].