

The X-ray single crystal structure of **2** revealed that the closest chains are arranged side by side to facilitate the N-H $\cdots\pi$ and C-H $\cdots\pi$ weak interactions, forming 2D sheets. The 2D sheets are further locked by two weaker $\pi\cdots\pi$ interactions from de pyrazole rings. The N atom of the thiocyanate is found to be intramolecular hydrogen bonded to an N-H pyrazole.

Keywords: supramolecular assemblies, polymers, cadmium

P.07.04.5

Acta Cryst. (2005). A61, C306

Connectivity Variation in Coordination Compounds of Isophthalate and Transition Metals

Luca Russo^a, William Clegg^b, ^aDepartment of Chemistry, University of Jyväskylä, Jyväskylä (FI). ^bDepartment of Chemistry, University of Newcastle, Newcastle upon Tyne (UK). E-mail: luca.russo@ncl.ac.uk

The coordination of the isophthalate ligand (IPA²⁻) with transition metal cations gives extended networks of variable architecture and dimensionality. The use of different auxiliary ligands and the variation of reaction conditions can provide control over the network dimensionality and/or the assembly of (3-n)-D networks.

Four coordination compounds, prepared by hydrothermal synthesis, are presented and compared to some related examples from the literature.

One structure of Mn(IPA)(Py)₂ is known from the literature [1]: it is made by chains assembled parallel to each other through weak non-bonded interactions. A polymorph of this structure has been prepared, in which there is less symmetry within the network and different packing of the chains.

Three examples of Co-IPA chain-structures are known from the literature [2]: in different reaction conditions one more structure of the same type has been prepared with increased metal-ligand connectivity giving a more robust 1-D network.

The structure of one Ni-IPA 1-D network is known from the literature [2]: two different coordination compounds with Ni²⁺ and IPA²⁻ have been prepared where the connectivity is progressively increased up to obtain a 2-D network.

Hydrothermal conditions were effectively used to investigate the structural variations in closely related coordination compounds structures. The detailed description of the structures, both from the point of view of the metal-ligand connectivity and of the non-bonded connectivity is meant to give some hint about the underlying structure-determining factors.

[1] Wang W., Ma., Zhang X., Chen C., Liu Q., Chen F., Liao D., Li L., *Bull. Chem. Soc. Jpn.*, 2002, **75**, 2609. [2] Bourne S.A., Mondal A., Zaworotko M.J., *Crystal Engineering*, 2001, **4**, 25.

Keywords: hydrothermal, connectivity, isophthalate

P.07.04.6

Acta Cryst. (2005). A61, C306

A New Organically Templated Vanadium Arsenate

Samroeng Krachodnok, Kenneth J. Haller, *School of Chemistry, Institute of Science, Suranaree University of Technology, Nakhon Ratchasima 30000, Thailand*. E-mail: Lipopoo@yahoo.com

Materials of open-framework and microporous structures are extensively studied because of their potential applications in catalysis, ionic conductivity, ion-exchange, and magnetic devices. Here we report a compound synthesized under hydrothermal conditions as part of a study of organically templated vanadium arsenates. V₂O₅, Na₂HAsO₄·7H₂O, H₂N(CH₂)₃NH₂ and H₂O in the molar ratio of 2:1:2.4:556 were heated at 180 deg C for 3 days to give black crystals.

The product was characterized by X-ray diffraction, SEM/EDX, FTIR, and TGA. The EDX spectrum indicates presence of vanadium and arsenic in a 3:1 ratio. The IR spectrum has a strong peak at 999 cm⁻¹ that can be assigned to the $\nu(\text{V}=\text{O})$ vibration, strong bands at 973, 800, 634 and 567 cm⁻¹ characteristic of $\nu(\text{M}-\text{O}-\text{M})$ (M = V or As) and $\nu(\text{As}-\text{O})$ [1], and bands in 1623-1383 cm⁻¹ region are due to the NH₂ and CH₂ bending of 1,3-dap. The broad band at 3438 cm⁻¹ can be attributed to O-H stretching.

[1] Beneš L., Melánová K., Zima V., Trchová M., Uhlířová E., Matějka P., *Eur. J. Inorg. Chem.*, 2000, 895-900.

Keywords: hydrothermal synthesis, vanadium arsenates, organic template

P.07.04.7

Acta Cryst. (2005). A61, C306

Noncovalent Interactions in a Three Component Supramolecular Structure

Kittipong Chainok^a, Kenneth J. Haller^a, Herman H.-Y. Sung^b, Ian D. Williams^b, ^aSchool of Chemistry, Institute of Science, Suranaree University of Technology, Nakhon Ratchasima 30000, Thailand. ^bDepartment of Chemistry, Hong Kong University of Science and Technology, Clear Water Bay, Hong Kong, China. E-mail: kchainok10@yahoo.com

Preparation of [(ImH₂)⁺]₂[Co(H₂O)₆]²⁺[Co(TMA)₂(H₂O)₄]⁴⁻ by hydrothermal synthesis, and characterization by single crystal X-ray diffraction is reported. There are distinct cationic and anionic cobalt complex ions. The coordination environment of the cobalt atom in the cationic complex is essentially octahedral, formed by water molecules, while the coordination environment of the cobalt atom in the anionic complex, is distorted octahedral with four water molecules and two trans trianionic trimesate ligands. Two imidazolium monocations complete the structure and provide charge balance. The three moieties interact in the lattice through noncovalent attractions. The carboxylate groups are good hydrogen bond acceptors, while the solvate water molecules and imidazolium cations are good hydrogen bond donors. Thus, it is not surprising to find strong hydrogen bonds between the imidazolium N-H groups and the carboxylate oxygen atoms from the trimesate ligand. The N-H \cdots O hydrogen bonds are in the range 2.693-2.777 Å, and the O-H \cdots O hydrogen bonds are in the range 2.666-2.948 Å. The two imidazolium cations exhibit a pair of N-H $\cdots\pi$ hydrogen bonds (π - π stacking) across an inversion center, and additional π - π interactions to the trimesate ligands.

Crystal Data: *P*-1 *a* = 9.1175(9), *b* = 9.3507(9), *c* = 10.6299(11) Å, α = 79.224(2), β = 87.448(2), γ = 71.995(2)°, *V* = 845.59(15) Å³, *Z* = 1; *T* = 298(2) K; *R*₁ = 0.042, 2688 observed data.

Keywords: crystallography, noncovalent bonding, supramolecular structures

P.07.04.8

Acta Cryst. (2005). A61, C306

Preparation and Characterization of NiCl(NO)(PPh₃)₂

Ratchadaporn Puntharod, Kenneth J Haller, *School of Chemistry, Institute of Science, Suranaree University of Technology, Nakhon Ratchasima, Thailand*. E-mail: ratchada_aim@hotmail.com

Previous work [1] on the title compound reports an intractable disorder problem; the complex was prepared again following the method of Feltham [2], and purple crystals were prepared by vapour diffusion of hexane into a benzene solution. The compound crystallizes in a *C*-centered monoclinic space group, with a cell of dimensions: *a* = 17.399(3), *b* = 13.136(3), *c* = 16.954(3) Å, β = 104.74(1)°, *Z* = 4 and *T* = 296 K.

FTIR spectral absorptions attributed to aromatic $\nu(\text{C}-\text{H})$ at 3050 cm⁻¹, $\nu(\text{N}-\text{O})$ at 1716 cm⁻¹, aromatic $\nu(\text{C}-\text{C})$ at 1434 cm⁻¹, and bands for CH in- and out-of-plane bending at 1095 cm⁻¹ and 693 cm⁻¹, respectively. The supramolecular structure contains the expected [3] six-fold phenyl embrace, 6PE, chains linking nickel bis(phosphine) moieties into one-dimensional zigzag chains. However, the lattice also contains a benzene solvate. The benzene molecule is able to form considerably more C-H $\cdots\pi$ interactions than a phenyl ring and becomes the major link, interconnecting three of the 6PE chains via ten C-H $\cdots\pi$ interactions centered about the benzene of crystallization. Additional weak noncovalent interactions supplement these major interactions to form an extensive three-dimensional crystal structure.

[1] Haller K.J., Enemark J.H., *Inorg. Chem.*, 1978, **17**(12), 3552. [2] Feltham R.D., *Inorg. Chem.*, 1964, **3**(1), 116. [3] Dance I., Scudder M., *J. Chem. Soc., Chem. Comm.*, 1995, **10**, 1039.

Keywords: supramolecular structure, nitric oxide complexes, cooperative phenomena