

processes were used to prevent environment pollution and support valuable material recovery. The main purpose of this study was to determine the crystal phases of the metal plating sludge at different temperatures to obtain the optimum thermal treatment conditions. Sample was heated at 105, 125, 200, 400, 625, 850 and 1000°C; and X-ray diffraction (XRD) examination of the powder samples was carried out using a Philips Panalytical X'pert Pro Diffractometer. The instrument was operated at 45 kV with a filament current of 40 mA and Cu K α radiation. Scanning operation covered a wide range of 2θ from 5 to 70° with a scanning speed 0.0025°/s at 0.02° steps. Crystal phase identification results of thermal treated sludge samples at different temperatures concluded that sulfate complexes with metals (Zn, Cr, Cu) transformed to oxide phases such as calcium iron phosphate (Ca₉Fe(PO₄)₇), zincochromite (ZnCr₂O₄) and tenorite (CuO) with Powder Diffraction File (PDF) cards: 00-045-0338, 01-073-1962 and 00-003-0884, respectively.

Keywords: waste treatment, crystal phases, metal oxides

P09.03.30

Acta Cryst. (2008). A64, C483

Two binuclear molecular magnet: K₂Fe₂(C₂O₄)Cl₆(H₂O)₂ and K₆Fe₆(C₂O₄)₃Cl₁₄(H₂O)₁₀

Yan Zhang¹, Bin Zhang²

¹Peking University, Department of Physics, Institute of Condensed Matter and Material Physics, Beijing, Beijing, 100871, China, ²Organic Solid Laboratory, CMS & BNLM, Institute of Chemistry, The Chinese Academy of Sciences, Beijing 100080, P. R. China, E-mail : zhang_yan@pku.edu.cn

Two oxalate-bridged binuclear iron(III) compounds K₂Fe₂(C₂O₄)Cl₆(H₂O)₂ (I) and K₆Fe₆(C₂O₄)₃Cl₁₄(H₂O)₁₀ (II) were synthesised. Their crystal structures were determined by single crystal X-ray diffraction. Both of them are composed of oxalate-bridged binuclear iron (III) anions with K⁺ as cation and Cl⁻ coordinated to iron on equator plane with oxalate group. The coordination ligands perpendicular to equator plane is different on I and II: In I, one H₂O and one Cl⁻ coordinated to Fe³⁺ at same time. But there are two kinds of binuclear anions with different coordination environments in (II): in one anion, H₂O occupy four axial positions, in another anion, one of them is replaced by Cl⁻. There are extensive hydrogen bonds in the crystal I and II. In I, it is only layered [4⁴,6²] lattice in the ac plane; in II, the hydrogen bond form a NaCl-type framework in the crystal. The cation exist in the hole between layer of II and vacant in the NaCl lattice. Their magnetic properties were measured. Both of them show anti-ferromagnetic interaction as dimer modes with J = -5.18cm⁻¹ in I and J = -1.50cm⁻¹ in II. The difference on magnetic property was determined by coordination environments and hydrogen bonds. Both of them are potential ionic conductor.

This work was supported by NSFC.20673120, MOST.2006CB601001, 2006CB932102, P. R. China

Keywords: iron compounds, oxalate-bridged, magnetic property

P09.03.31

Acta Cryst. (2008). A64, C483

Structure diversity and reversible anion exchange properties of metal complexes with tripodal ligand

Wei-Yin Sun¹, Guan-Cheng Xu¹, Taka-aki Okamura², Norikazu Ueyama²

¹Nanjing University, State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing, Jiangsu Province, 210093, China, ²Department of Macromolecular Science, Graduate School of Science, Osaka University, Toyonaka, Osaka 560-0043, Japan, E-mail : sunwy@nju.edu.cn

We focus our attention on crystal engineering and properties of metal-organic frameworks with flexible multidentate organic ligands, such as 1,3,5-tris(imidazol-1-ylmethyl)benzene (tib), 1,3,5-tris(imidazol-1-ylmethyl)-2,4,6-trimethylbenzene (titmb). Systematic study of reactions of tib and titmb with varied metal salts was carried out, and coordination architectures with zero-, one-, two- and three-dimensional structures were obtained and their properties were investigated. For example, six cadmium(II) coordination polymers, [Cd(tib)₂](BF₄)₂ (**1**), [Cd(tib)₂](NO₃)₂·2C₂H₅OH (**2**), [Cd(tib)(OAc)₂]₄·5H₂O (**3**) (OAc = acetate anion), [Cd(tib)(μ_2 -Cl)Cl]·CH₃OH (**4**), [Cd(tib)₂(H₂O)₂]Cl₂·4H₂O (**5**) and [Cd(tib)₂][Cd₂I₆] (**6**), were obtained by reactions of tib with the corresponding metal salts, respectively. Their structures were determined by X-ray crystallography. **1** and **6** have infinite 2D cationic double layered structures with different shapes. **2** possesses a 3D framework structure with two different kinds of channels. The structure of **3** is a two-fold interpenetrated 3D framework. **4** and **5**, synthesized by the same reactants in the same solvent, have different 2D network structures due to the different synthetic methods. The distinct structures of the complexes (e.g. **1**, **2** and **6**) indicate that the counteranions have significant influence on the structures of coordination polymers. **4** and **5** provide nice example that synthetic methods can affect the structures of the frameworks in this system. In addition, the results also attest that the flexible ligand tib can adopt different conformations and coordination modes to form complexes with varied structures. Furthermore, the reversible anion exchange properties of **1** and **2** were studied.

Keywords: crystal engineering, metal-organic complexes, supramolecular assembly

P09.03.32

Acta Cryst. (2008). A64, C483-484

Lanthanoid, transition metal and heterobimetallic complexes with polynitrile and derivative ligands

Anthony S. R. Chesman, Stuart R. Batten, Glen B. Deacon, David R. Turner

Monash University, School of Chemistry, Wellington Road, Clayton, Victoria, 3800, Australia, E-mail: anthony.chesman@sci.monash.edu.au

Anionic polynitrile ligands such as dicyanonitrosomethanide (dcnm) have been shown to readily undergo transition metal promoted nucleophilic addition, providing a convenient pathway to simultaneous *in situ* ligand and complex synthesis. These derivative ligands have targeted coordination modes allowing the formation of 3d/4f heterobimetallic species. Solvothermal synthesis allows for the formation of networks, not accessible through bench top methods, which display ferromagnetic coupling.¹ Lanthanoid complexes of dcnm display a unique η^2 nitroso bonding,² the symmetry of which can be affected by co-ligands, such as crown ethers and 1,10-phenanthroline, by introducing steric crowding into systems. This bonding mode is also observed in the water addition product, carbamoylcyanonitrosomethanide (ccnm), where extensive intermolecular hydrogen bonding influences the degree of asymmetry in the nitroso bonding. As the ccnm ligand retains its anionic character the requirement of a counter cation allows for tuneable solubility properties and has been shown to dramatically effect of the topology of resulting networks, despite the absence of any obvious

templating effects. The ccm ligand has also been shown to be conducive in the formation of transition metal and lanthanoid clusters which are of interest in the area of single molecule magnets.

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2. Anthony S. R. Chesman, David R. Turner, Ekaterina I. Izgorodina, Stuart R. Batten and Glen B. Deacon, *Dalton Trans.*, 2007, 1371-1373.

Keywords: lanthanide coordination, bimetallics, magnetic materials

P09.03.33

Acta Cryst. (2008). A64, C484

Synthesis and characterization of mixed metal ($\text{UO}_2^{2+}/\text{TM}^{2+}$) inorganic/organic framework materials

Karah E Knope¹, Christopher L Cahill^{1,2}

¹The George Washington University, Chemistry, 725 21st Street, N.W., Washington, DC, 20052, USA, ²Geophysical Laboratory, Carnegie Institution of Washington, Washington, DC, 20015, USA, E-mail : kknop@gwu.edu

Inorganic materials containing UO_2^{2+} comprise a structurally diverse catalog of compounds wherein layered architectures are most commonly observed. The dimensionality of these structures is typically governed by the terminal nature of the uranyl oxo atoms and the propensity for structure propagation to occur almost exclusively in two dimensions. As a result, three-dimensional structures are not only less frequently observed but also a challenge to obtain synthetically. Within purely inorganic systems, researchers have used templates or, alternatively, incorporated main group or transition metal cations to achieve higher dimensional frameworks. These strategies, in addition to flexible organic linkers, have also been applied to hybrid inorganic-organic systems. Presented here is a series of novel, three-dimensional, mixed metal uranium(VI)/transition metal(II) inorganic-organic hybrid materials. A representative example includes the framework structure of $(\text{UO}_2)_2(\text{O}_3\text{PCH}_2\text{CO}_2)_2\text{Cu}(\text{H}_2\text{O})_3$, monoclinic, $P2_1/c$, $a = 7.6358(3)$, $b = 19.2962(3)$, $c = 11.2401(8)$, $\beta = 100.4990(10)$. This compound exhibits coordination of copper through the nominally terminal uranyl oxygen atoms and illustrates how the addition of transition metal cations can influence the dimensionality, overall topology and luminescent behavior of hexavalent uranium containing architectures.

Keywords: framework structures, uranium(VI), hydrothermal synthesis

P09.03.34

Acta Cryst. (2008). A64, C484

A nine-coordinated Zr^{IV} complex obtained from a novel supramolecular proton transfer compound

Hossein Aghabozorg¹, Mohammad Ghadermazi², Shabnam Sheshmani³

¹Tarbiat Moallem University, Chemistry, Faculty of Chemistry, Tarbiat Moallem University, 49 Mofateh Ave., 15614, Tehran, Iran, Tehran, Iran, 15614, Iran, ²Department of Chemistry, Kurdistan University, Sanandaj, Iran, ³Department of Chemistry, Islamic Azad University, Shahr-e Rey Branch, Tehran, Iran, E-mail: haghazorg@yahoo.com

In this research, a novel supramolecular proton transfer compound was synthesized from the reaction between pyridine-2,6-dicarboxylic acid, $\text{C}_7\text{H}_5\text{NO}_4$, and propane-1,3-diamine, $\text{C}_3\text{H}_{10}\text{N}_2$. According to the crystal structure, the resulting compound formulated as $2(\text{C}_3\text{H}_{12}\text{N}_2)^{2+}(\text{C}_7\text{H}_3\text{NO}_4)^{2-}(\text{C}_7\text{H}_{4.5}\text{NO}_4)^{1.5-}(\text{C}_7\text{H}_{3.5}\text{NO}_4)^{0.5-}(\text{C}_7\text{H}_5\text{NO}_4) \cdot 5\text{H}_2\text{O}$. This compound crystallizes in the triclinic system and contains two molecules per unit cell. The unit cell dimensions are $a=7.8355(4)\text{\AA}$, $b=13.5296(6)\text{\AA}$, $c=20.8930(6)\text{\AA}$, $\alpha=106.558(1)^\circ$, $\beta=90.603(1)^\circ$, and $\gamma=106.662(1)^\circ$. The final R value was 0.0427 for 9245 reflections. In anionic fragment, one of fourth hydrogen atoms of hydroxy groups is disordered. In cationic fragment, two carbon and nitrogen atoms, are disordered on two position with occupancies 0.88/0.12. Anion molecules form two independent hydrogen bonded zigzag-type chains. The hydrogen bonds as type $\text{O}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{O}$, and $\text{N}-\text{H}\cdots\text{N}$ play a role in the construction of supramolecular framework. Interactions between cationic and anionic fragments consist of ion-pairing, hydrogen bonds and $\pi-\pi$ stacking. The reaction of the proton transfer compound with ZrCl_4 was carried out. This complex formulated as $(\text{C}_3\text{H}_{12}\text{N}_2)[\text{Zr}(\text{C}_7\text{H}_3\text{NO}_4)_3] \cdot 4\text{H}_2\text{O}$. The Zr^{IV} complex crystallizes in the space group $P2_1/c$ of the monoclinic system with four molecules per unit cell. The unit cell dimensions are $a=10.0246(6)\text{\AA}$, $b=17.1883(10)\text{\AA}$, $c=17.8643(10)\text{\AA}$, and $\beta=97.2970(10)^\circ$. The metal ion is ninecoordinated by three pyridine-2,6-dicarboxylate as tridentate ligand. The Zr^{IV} atom is located in the center of a distorted tricapped trigonal prism arrangement. The crystal-packing diagram indicates the layered structure for this complex. This complex arises from ion pairing and the extensive hydrogen bonding.

Keywords: zirconium, supramolecular compounds, crystal structures

P09.03.35

Acta Cryst. (2008). A64, C484-485

Binuclear Sn(VI) complex obtained from benzene-1,3-diaminium bis(hydrogen pyridine-2,6-carboxylate)

Jafar Attar Gharamaleki¹, Shabnam Sheshmani², Zahra Hasanpour³, Mohammad Ghadermazi⁴, Hossein Aghabozorg⁵, Guido Kickelbick⁶

¹Tarbiat Moallem University, Chemistry, Faculty of Chemistry, Tarbiat Moallem University, 49 Mofateh Ave., 15614, Tehran, Iran, Tehran, Iran, 15614, Iran, ²Department of Chemistry, Islamic Azad University, Shahr-e Rey Branch, Tehran, Iran, ³Department of Chemistry, Islamic Azad University, Shahr-e Rey Branch, Tehran, Iran, ⁴Department of Chemistry, Faculty of Science, Kurdistan University, Sanandaj, Iran, ⁵Faculty of Chemistry, Tarbiat Moallem University, Tehran, Iran, ⁶Institute of Materials Chemistry, Vienna University of Technology, Wien, Austria, E-mail: attar_jafar@yahoo.com

In recent years, our research group have studied and worked on several bi- or trivalent metal complexes with several proton transfer ion pairs containing pyridine-2,6-dicarboxylic acid, pydcH_2 . In resulting complexes, some fragments of ion pairs coordinate to metal ions and some other fragments act as counter ions in the structure. Here we wish to report the molecular structure of the Sn(VI) complex, which obtained from a novel proton transfer compound $(\text{bdaH}_2)(\text{pydcH})_2 \cdot 2\text{H}_2\text{O}$. The proton transfer compound $(\text{bdaH}_2)(\text{pydcH})_2 \cdot 2\text{H}_2\text{O}$, has been prepared from the reaction between benzene-1,3-diamine, bda, and pyridine-2,6-dicarboxylic acid, pydcH_2 . The reaction of this adduct with $\text{Sn}(\text{CH}_3)_2\text{Cl}_2$ gives complex with stoichiometry $[\text{Sn}(\text{pydc})(\text{OC}_2\text{H}_5)(\text{CH}_3)_2(\mu\text{-O})]_2$. This complex was characterized by IR, NMR spectroscopy and X-ray crystallography. This complex crystallizes in the monoclinic system, space group $P2_1/c$, with four molecules per unit cell. The unit cell dimensions are $a=11.5924(14)\text{\AA}$, $b=13.2674(17)\text{\AA}$, $c=7.7566(10)\text{\AA}$