### FA4-MS34-P07

Supramolecular Architecture of Novel Hydroxamato Complexes of Os(II) and Ru(II). <u>Attila C. Bényei<sup>a</sup></u>, Gábor Paragh<sup>a</sup>, Norbert Balázs<sup>b</sup>, Attila Godó<sup>b</sup>, Zsigmond T. Nagy<sup>a</sup> and Péter Buglyó<sup>b</sup>, <sup>a</sup>University of Debrecen, Department of Chemistry, Laboratory for Xray Diffraction, Debrecen, Hungary, <sup>b</sup>University of Debrecen, Department of Inorganic and Analytical Chemsitry, Debrecen, Hungary E-mail: abenyei@delfin.unideb.hu

Derivatives of hydroxamic acid gained significant importance as hard donors because of their biological relevance especially as inhibitors of deacetylases or antimicrobial activity. This feature is connected to their strong metal binding capabilities making them enzyme inhibitors or constituents of siderophores as well as their similarity to carboxylic acids. Some of us have shown very recently that hydroxamato complexes of *soft* platinum group metals could be prepared strarting from the respective half-sandwich, organometallic pcymene-containing precursor see bellow. [1] Supramolecular analysis of ten different complexes of Os(II) or Ru(II) revealed that careful balance of mono- or bidentate hydroxamate coordination, steric hindrance of the R substituents, stability of coordination of auxiliary pyridine ligand,  $\pi$ - $\pi$  stacking of the phenyl rings as well as van der Waals and Coulombic interactions give as a result the final mono- or dinuclear complex having one or more molecules in the asymmetric unit. These compounds have significant potential in homogeneous catalytic reactions as well.



Acknowledgement. BP thanks the members of the EU COST Action D39 for motivating discussions. This work was supported by the Hungarian Scientific Research Fund (OTKA K76142). [1] Buglyó, P., Farkas, E: Novel Half-sandwich Ru(II)-Hydroxamate Complexes: Synthesis, Characterization and Solution Study in Aqueous Solution, Dalton, 2009, 8063.

Keywords: hydroxamic acid, platinum group complexes, supramolecular structures

## FA4-MS34-P08

Crystal structure of Na<sub>3</sub>MoCl<sub>6</sub> – a new 3-1-6 phase of molybdenum(III). <u>Martin Beran</u><sup>a</sup>, Gerd Meyer<sup>a</sup>,

<sup>a</sup>Department für Chemie, Universität zu Köln, Germany E-mail: <u>mberan@uni-koeln.de</u> During attempts to secure new coordination compounds with the anionic cluster complex,  $[\{Mo_6\}Cl^a_8Cl^a_6]^{2^*}$ , we have, except for normal solvents like water or volatile organic solvents, also explored ionic liquids and molten salts as solvents for  $MoCl_2 = [\{Mo_6\}Cl^i_{8/1}Cl^a_{-4/2}]$ .

The reaction of MoCl<sub>2</sub> in an excess of the flux AlCl<sub>3</sub>/NaCl (55:45 mol%) in a silica ampoule at 350°C yielded dark red, dodecahedrally shaped, air-sensitive single crystals of Na<sub>3</sub>MoCl<sub>6</sub>. It crystallizes with the Na<sub>3</sub>CrCl<sub>6</sub>-type of structure [1], trigonal, space group  $P3; ^{-}/c$  (no. 163), a = 687.1(1), c = 1225.3(2) pm, Z = 2.



Fig. 1. Two views of the crystal structure of  $Na_3MoCl_6$ : [1-10] projection (top) and projection of a  $Cl_6MoNa$  double layer onto (001) (bottom).

In Na<sub>3</sub>MoCl<sub>6</sub>, hexagonal-closest packed layers of chloride ions are stacked in the [001] direction of the hexagonal unit cell, see Fig. 1. Two thirds of the octahedral interstices are filled between these layers with, alternatively, two sodium ions and sodium and molybdenum(III) ions, respectively. The structure is considerably different from that of K<sub>3</sub>MoCl<sub>6</sub> [2] where the potassium ions are involved in the closest packed layers together with chloride ions and Mo(III) occupies octahedral voids between the K<sub>3</sub>Cl<sub>6</sub> double layers, see also [3]. Mo—Cl distances are 245.2 pm in Na<sub>3</sub>MoCl<sub>6</sub> and compare very well with the respective distances in K<sub>3</sub>MoCl<sub>6</sub>, 244.7 pm on the average.

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#### Keywords: Molybdenum, Flux, Crystal Structure

#### FA4-MS34-P09

Silver(I) isobutyrate, Ag(i-OBu), with dimers connected to layers, not chains. <u>Thomas Bierke</u>, Gerd Meyer Department für Chemie, Universität zu Köln, Germany

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Only a few silver(I) monocarboxylates have been characterized, of which the acetate Ag(OAc) [1] and the trifluoroacetate Ag(OTfa) are important examples [2]. Outstanding structural features are carboxylate bridged silver(I) dimers which are further bridged to chains. We have obtained several new Ag(I) carboxylates of which silver(I) isobutyrate, Ag(i-OBu), is especially interesting because the  $[Ag(i-OBu)]_2$  dimers are connected to layers.

For the synthesis of Ag(i-OBu) 0.5 mmol silver carbonate are dissolved in in 25 ml isobutyric acid. After five weeks of isothermic evaporation cuboid shaped single crystals had formed. Ag(i-OBu) crystallizes in the monoclinic space group P2<sub>1</sub>/c (no. 14) with a = 1333.7(3), b = 835.9(1), c = 1005.5(2) pm,  $\beta = 98.74(2)^{\circ}$ , Z = 4.



Fig 1.  $[Ag(i-OBu)]_2$  dimers with two isobutyrate anions completing the coordination sphere of each Ag(I) ion as part of the crystal structure of silver(I) isobutyrate

Within the [Ag(i-OBu)]<sub>2</sub> dimers, the Ag-Ag distance is 281.4(4) pm, only slightly larger than in [AgOAc]<sub>2</sub> (279.4(4) pm) [1] and considerably shorter than in [Ag(OTfa)]<sub>2</sub> (296.7(3) pm) [2]. In Ag(i-OBu), the coordination sphere of Ag(I) is completed by two additional oxygen atoms of isobutyrate anions belonging to neighbouring dimers such that the isobutyrate ligands have two different bridging functions. Of course, the Ag-O distances are shorter within the dimers, as short as 219 pm, and much longer between the dimers, 243-259 pm. The shortest Ag-Ag distance between dimers is 331.3(5) pm, maybe not within the range of argentophilic attractions but still rather short. These short Ag-Ag distances and the 2+2 coodination by oxygen atoms lead to a twodimensional arrangement, such that the present Ag(i-OBu) must be considered as the first Ag(I) mono carboxylate with a layered structure.

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Keywords: Silver dimers, Isobutyrate, Crystal structure

## FA4-MS34-P10

Characterization and Antimicrobial Activities of Cobalt-Pyrazine-2,3-dicarboxylate Complexes. Orhan Büyükgüngör<sup>a</sup>, Okan Zafer Yeşilel<sup>b</sup>, Aylin

tyrate, Crystal structure the complex Ni(*cyclan*)N of microcrystalline power

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Novel cobalt-pyrazine-2,3-dicarboxylate complexes with 1,10phenanthroline (phen), [Co(pzdca)(phen)2][2H2O (1), N,N,N',N'-tetramethylethylenediamine (tmen), (H2tmen)-[Co(pzdca)2(tmen)]·9H2O (2) and 2,2-dimethyl-propane-1,3diamine (dmpen), [Co(CO3)(dmpen)2](pzdca)0.5·H2O (3) have been synthesized and characterized by elemental and thermal analyses, spectroscopic (IR and UV-Vis.) and X-ray diffraction techniques. In 1 and 2, pyrazine-2,3-dicarboxylate ligand coordinated to the Co(II) ions through one nitrogen atoms of pyrazine ring and oxygen atoms of carboxylate group as a bidentate ligand and distorted octahedral geometries of 1 and 2 are completed by phen and tmen ligands, respectively. In 2, the tmen molecules exhibit chemically different functions; it coordinated to the Co(II) ion as a bidentate ligand and in the other form it protonated and acts as counter ion. In complex 3, Co(III) ion is coordinated by four nitrogen atoms of dmpen and two oxygen atoms of CO3 ligand and the pzdca behaves as a counter ion. Furthermore, structures of 1 and 2 contain extensive hydrogen bonding between crystal water molecules to form infinite 2D water layers and 1D water chains, respectively. In vitro antimicrobial activities of new complexes were tested against selected wild type and clinical microorganisms by MIC. Complexes exhibited antimicrobial activity at high concentrations against the bacteria, fungi and clinical isolate tested.

# Keywords: Pyrazine complexes, Cobalt complexes, Antimicrobial activities

# FA4-MS34-P11

Crystallogpraphic study of the system Ni(NO<sub>3</sub>)<sub>2</sub> – cyclam - [Ni(CN)<sub>4</sub>]<sup>2</sup>- Juraj Černák<sup>a</sup>, Monika Stolárová<sup>a</sup>, Milagros Tomás<sup>b</sup>, Larry R. Falvello<sup>b</sup>, <sup>a</sup>Institute of Chemistry, Department of Inorganic Chemistry, P. J. Šafárik University in Košice, Slovakia, <sup>b</sup>University of Zaragoza-C.S.I.C.,Department of Inorganic Chemistry and I.C.M.A., Zaragoza, Spain E-mail: juraj.cernak@upjs.sk

Within our broader study on Ni(II) (S = 1) complexes as lowdimensional magnetics [1] we have studied the system  $Ni(NO_3)_2 - cyclam - [Ni(CN)_4]^2$  (cyclam = 1,4,8,11-tetraazacyclotetradecane). The ligand cyclam was chosen as it usually blocks four equatorial coordination sites around the central atom and thus promotes, in the presence of suitable bridging units (in our case the  $[Ni(CN)_4]^{2-}$  anion) the formation of one-dimensional structures. By direct reaction the complex  $Ni(cyclam)Ni(CN)_4(1)$  was prepared in the form of microcrystalline powder. On the other hand, the use of diffusion techniques led to a mixture of single crystals: yellow [Ni(cyclam)(NO<sub>3</sub>)<sub>2</sub>] (2) which has already been described [2], pale and two novel complexes, violet  $Ni(cyclam)Ni(CN)_4 \cdot 2H_2O$  (3) and yellow [Ni(cyclam)(CN)]NO3·H2O (4). Both 3 and 4 exhibit the expected onedimensional structures. In the structure of **3** there are present two crystallographically independent chains [-Ni(cyclam)-NC-