

six sandwiches per unit cell stacked in rhombohedral symmetry [1]. Intercalated Cu atoms and additional M atoms are incorporated on octahedral and tetrahedral sites in the Van der Waals gaps between  $MS_2$  layers. Here we present the evolution of the complex superstructures of selected intercalated compounds at different temperatures between 14K and 400K using single-crystal X-ray diffraction data. The interest in these compounds is the phase transitions related to ordering of the intercalated atoms and charge-density wave transitions at different temperatures.

[1] B. Harbrecht, G.Z. Kreiner, *anorg. allg. Chem.* **1989**, 572, 47-54.

**Keywords:** X-ray\_diffraction, dichalcogenides, superstructure

## MS67.P04

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### Thermal studies of ammonium-cobalt-nickel phosphates, $NH_4[Co_xNi_{1-x}PO_4] \cdot H_2O$

Laura Torre-Fernández,<sup>a</sup> Camino Trobajo,<sup>b</sup> José R. García,<sup>b</sup> Santiago García-Granda,<sup>a</sup> <sup>a</sup>Physical and Analytical Chemistry Department and <sup>b</sup>Organic and Inorganic Chemistry Department, University of Oviedo - CINN, (Spain). E-mail: torrelaura@uniovi.es

Ammonium-metal(II) phosphates of general formula  $NH_4M^{II}PO_4 \cdot H_2O$  were first described in 1864 by Debray [1]. These compounds have been used as pigments for protective paint finishes on metal and as fire retardants in paints and plastics, and they can be also applied as catalyst, fertilizers and magnetic devices.

The first crystal structure for a member of this family,  $NH_4CoPO_4 \cdot H_2O$ , was determined in 1968 by Tranqui et al. [2] using powder X-ray diffraction. In 1995, using neutron powder diffraction techniques, Carling *et al.* [3] determined the crystal structures of  $ND_4M^{II}PO_4 \cdot D_2O$  ( $M^{II} = Mn, Fe, Co, Ni$ ) compounds. In 1999, Yakubovich *et al.* [4] have contributed with the first structure, for  $NH_4CoPO_4 \cdot H_2O$ , obtained by single-crystal (X-ray diffraction) data. Apparently, all these layered compounds crystallize in the orthorhombic space group  $Pmn2_1$ .

In a previous work, we introduced a new family of ammonium-cobalt-nickel phosphates,  $NH_4[Co_{1-x}Ni_xPO_4] \cdot H_2O$  ( $x = 0.00, 0.34, 0.60, 0.71, 1.00$ ) [5]. In this series, although all crystals are orthorhombic, the space group changes as a function of the composition. In this communication, we report thermal studies for all synthesized compounds within this family. These thermal studies were focused in the structural changes, showing different thermal behavior as a function of the composition.

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[1] C.R. Debray, *Academic Science* **1864**, 59, 40. [2] D. Tranqui, A. Durif, J.-C. Guitel, M.T. Verbuch-Pouchot, *Bulletin de la Societe Francaise Mineralogie et de Cristallographie* **1968**, 91, 10. [3] S.G. Carling, P. Day, D. Visser, *Inorganic Chemistry* **1995**, 3917. [4] O.V. Yakubovich, O.V. Karimova, O.V. Dimitrova, W. Massa, *Acta Crystallographica* **1999**, C55, 151. [5] C. Trobajo, J.R. García, S. García-Granda, L. Torre-Fernández, *Inorganic Chemistry* **2011**, submitted.

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### Solvent-dependent 2D-coordination networks of CuI and bridging triazolopyrimidines

Miguel Quirós,<sup>a</sup> Ana B. Caballero,<sup>a,b</sup> Antonio Rodríguez-Diéguez,<sup>a</sup> Jana Vieth,<sup>b</sup> Christoph Janiak,<sup>b,c</sup> Juan M. Salas,<sup>a</sup> <sup>a</sup>Department of Inorganic Chemistry, Universidad de Granada, Granada (Spain). <sup>b</sup>Institute of Inorganic and Analytical Chemistry, Universität Freiburg, Freiburg (Germany). <sup>c</sup>Institute of Inorganic and Structural Chemistry, Universität Düsseldorf, Düsseldorf (Germany). E-mail: mquiros@ugr.es

Copper(I) iodide has been used as the inorganic component in the building of novel coordination polymers with diverse structural motifs. These motifs could be further connected by *N*-heterocyclic linkers to form more complex  $[Cu_nI_n]$ -based frameworks. Preparation of such compounds has been reported to be affected by many factors such as CuI-to-ligand ratio, flexibility and rigidity of the ligands, solvents and temperature [1,2].

In this communication, we describe the syntheses and the crystal structures of two new 2D-polymers, both obtained from the reaction between CuI and the multidentate ligand 1,2,4-triazolo[1,5-a]pyrimidine (tp):  $[Cu_2(\mu\text{-tp})(CH_3CN)(\mu\text{-I})(\mu_3\text{-I})_n]$  (**1**) and  $[Cu_2(\mu\text{-tp})(\mu_3\text{-I})_2]_n$  (**2**). Structural differences between both compounds seem to be due to a solvent influence, being acetonitrile for **1** and water for **2**.

In compound **1**, the triazolopyrimidine derivative acts as bridging ligand via N1 and N3 between chair-like  $[Cu_4I_4]$  units, resulting Cu...Cu distances from 3.04 to 6.02 Å. Acetonitrile molecules are also coordinated to metal centres. Strong stacking forces involving triazole and pyrimidine rings of tp ligands contribute to the stabilization of the structure. In compound **2**, tp moieties are placed at both sides of a  $[Cu_nI_n]$  half-folded layer, displaying also a bridging mode but, in this case, via its atoms N3 and N4 (see Figure 1). In both structures, Cu(I) ions show slightly distorted tetrahedral coordination geometries.

These are the first examples of Cu(I) compounds based on the nucleobase-analog ligand tp.

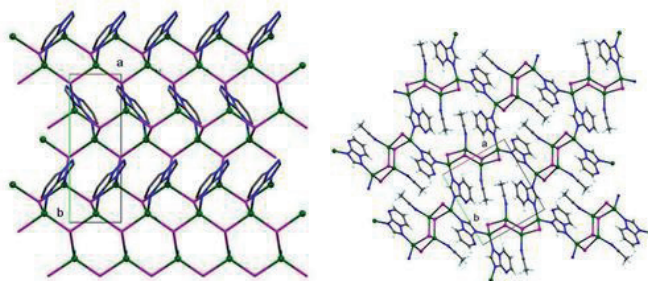


Figure 1. View of the 2D-networks of  $[Cu_2(\mu\text{tp})(CH_3)(\mu I)(\mu_3\text{-I})_n]$  (**1**) (on the left) and  $[Cu_2(\mu\text{tp})(\mu_3\text{-I})_2]_n$  (**2**) (on the right).

[1] P.M. Graham, R.D. Pike, M. Sabat, R.D. Bailey, W.T. Pennington, *Inorg. Chem.* **2000**, 39, 5121-5132. [2] C. Näther, I. Jess, *Eur. J. Inorg. Chem.* **2004**, 2868-2876.

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### Novel perovskite-like compound with crystallographic shear structure in the Pb-Ba-Fe-Sn-O system

Oleg E. Korneychik,<sup>a</sup> Marina G. Rozova,<sup>b</sup> Artem M. Abakumov,<sup>b</sup> Maria M. Batuk,<sup>a</sup> Konstantin V. Pokholok,<sup>a</sup> Dmitry S. Filimonov,<sup>a</sup> Evgeny V. Antipov. <sup>a</sup>Department of Chemistry, Moscow State University, Moscow. <sup>b</sup>Electron Microscopy for Materials Research (EMAT), University of Antwerp, Antwerp, Belgium. E-mail: kocmobaxtep@yandex.ru