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Cu(II) complexes with dicarboxylic acids and 3-aminopyridine

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Over the past decade an enormous attention has been paid to the construction of coordination polymers due to their intriguing structural diversity and potential function. In this aspect, the anions of dicarboxylic acids constitute one of the most commonly employed classes of organic ligands. The flexibility and conformational freedom of aliphatic dicarboxylate ligands give rise to a variety of interesting structural motifs. Additionally, when combining with aromatic N-donor ligands, the π - π stacking interactions between the aromatic ligands play an important role in the design of polymeric architectures. The structures of Cu(II) complexes with the oxalate, first from the series of anions of aliphatic dicarboxylic acids ($\text{HOOC}-(\text{CH}_2)_n-\text{COOH}$, $n=0,1,2,\dots$) and aminopyridines with different position of the amino group with respect to the pyridine nitrogen atom have already been reported [1]. We extend the research on the complexes with higher dicarboxylates from malonate with 3C atoms to suberate with 8C atoms. The structures of complexes containing 3-aminopyridine will be presented and compared in terms of the known related structurally characterised compounds.

[1] Castillo O., Luque A., Roman P., Lloret F. and Julve M., *Inorg. Chem.*, 2001, 40, 5526-5535.

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Structural Characterisation of Two Molybdenum(V) Thiosemicarbazonato Complexes and Mo-containing Intermediates Derived from $\text{X}_2[\text{MoCl}_5]$ ($\text{X} = \text{NH}_4$ or PyH)

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Thiosemicarbazones and their transition metal complexes are interesting compounds due to antibacterial, antiviral and antitumor activities observed for some of them [1]. By the reaction of $(\text{NH}_4)_2\text{MoOCl}_5$ and the corresponding thiosemicarbazones in acetonitrile and in argon atmosphere we prepared single crystals of two molybdenum(V) thiosemicarbazonato complexes: $[\text{MoOCl}_2\{\text{SalTSC-4-Ph}\}]$ (**1**; SalTSC-4-Ph = salicylaldehyde-4-phenylthiosemicarbazonato) and $[\text{MoOCl}_2\{\text{NaphtTSC-4-Ph}\}] \cdot \text{CH}_3\text{CN}$ (**2**; NaphtTSC-4-Ph = naphthaldehyde-4-phenylthiosemicarbazonato). The by-product of both synthetic reactions was ammonium chloride. In both **1** and **2** the Mo^{V} coordination is a distorted octahedron with the chloro ligands occupying mutually *trans* positions and with the meridionally bonded deprotonated tridentate (*N, O, S*) thiosemicarbazone ligand. Although crystallised in different space groups (**1** in *Pbca*, **2** in *P2₁/n*), in both crystal structures molecules of the complex are connected in infinite chains by forming $\text{N-H}\cdots\text{Cl}$ hydrogen bonds. Also, in both structures there are similar $\pi\cdots\pi$ interactions between the aromatic moieties.

We also prepared crystals of $\text{NH}_4[\text{MoOCl}_4(\text{CH}_3\text{CN})]$ (**3**) from the acetonitrile solution of $(\text{NH}_4)_2\text{MoOCl}_5$. This suggests $[\text{MoOCl}_4(\text{CH}_3\text{CN})]^-$ to be the reaction intermediate in the synthesis of Mo(V) thiosemicarbazonato complexes. In **3** Mo^{V} is in distorted octahedral environment with the acetonitrile molecule being *trans* to the oxo group. It crystallises in the space group *Cmcm* with both ammonium cation and $[\text{MoOCl}_4(\text{CH}_3\text{CN})]^-$ lying at the intersection of two mirror planes. The failure of the synthesis of **1** and **2** from the analogous pyridinium salt, $(\text{PyH})_2[\text{MoOCl}_5]$ (*Py* = pyridine), could be explained by the non-existence of $[\text{MoOCl}_4(\text{CH}_3\text{CN})]^-$ anions in its acetonitrile solution. Namely, unstable crystals of $(\text{PyH})_2[\text{MoOCl}_5] \cdot \text{CH}_2\text{Cl}_2$ (**4**) belonging to the space group *Pnma* were obtained from acetonitrile and dichloromethane (1:1) solution of $(\text{PyH})_2[\text{MoOCl}_5]$. In **4** two pyridinium cations are hydrogen bonded to the chlorine atom *trans* to the oxo group in $[\text{MoOCl}_5]^{2-}$.

[1] Belicchi Ferrari M., Bisceglie F., Pelosi G., Tarasconi P., Albertini R., Dall'Aglio P. P., Pinelli S., Bergamo A., Sava G., *J. Inorg. Biochem.*, 2004, 98, 301.