

### MS30-P38 Synthesis and X-ray structural study of two new polymorphs coordination complexes based on imidazol derivative

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Polymorphism is very common among pharmaceutical substances. As the polymorphs possess different internal organization within the solid, they often show different melting points, solubilities, chemical reactivity or stability. These can appreciably influence pharmaceutical properties such as dissolution rate and bioavailability. It is therefore important to evaluate the polymorphism in early stages of new formulation studies.

We report here the synthesis, crystallographic study and hydrogen bond interactions of two new polymorph coordination complexes based on imidazol derivative and Cobalt.

Polymorph (I) is triclinic with space group P-1 and cell parameters  $a=7.0882(8)$  Å,  $b=11.9322(12)$  Å,  $c=14.1882(15)$  Å,  $\alpha=71.417(5)^\circ$ ,  $\beta=86.927(5)^\circ$ ,  $\gamma=83.489(5)^\circ$ ,  $V=1129.9(2)$  Å<sup>3</sup>, and  $Z=2$ . Polymorph (II) is monoclinic with space group P 21/n and cell parameters  $a=7.5554(2)$  Å,  $b=13.0466(5)$  Å,  $c=23.3958(9)$  Å,  $\beta=94.405(2)^\circ$ ,  $V=2299.36(14)$  Å<sup>3</sup>, and  $Z=4$ .

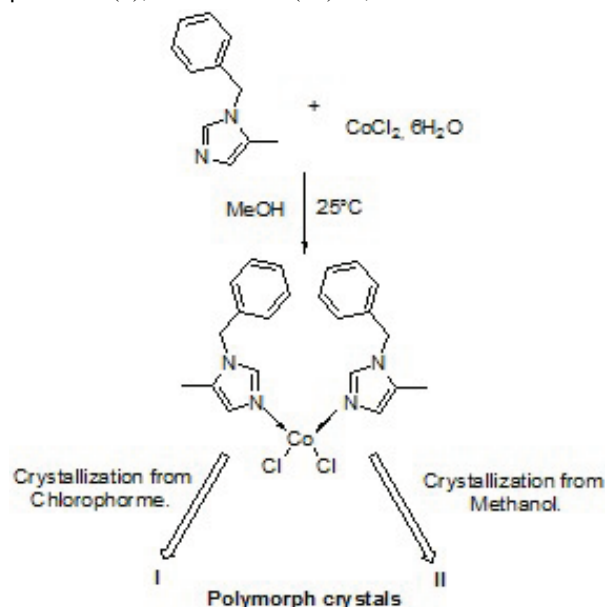


Figure 1. Scheme

**Keywords:** Polymorphism, single crystal, hydrogen bond, imidazol derivatives

### MS30-P39 The role of methanol molecule in supramolecular assembling of $[\text{MoO}_2\text{L}(\text{CH}_3\text{OH})] \cdot \text{CH}_3\text{OH}$ and $[\text{MoO}_2\text{L}(\text{CH}_3\text{OH})]$ Schiff base type complexes

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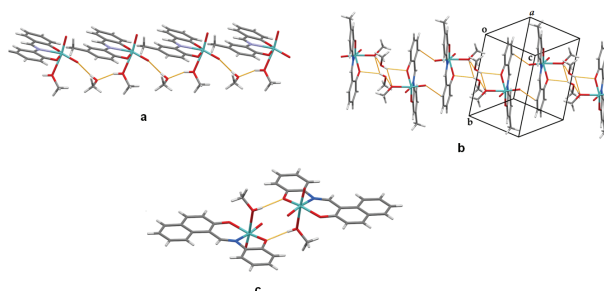
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Investigations of Schiff base molybdenum(VI) complexes have been stimulated by discovery of molybdenum in a number of redox enzymes and their efficiency as catalysts both in heterogeneous and homogenous reactions. Reactions of tridentate O,N,O Schiff base ligands  $\text{H}_2\text{L}$  with  $[\text{MoO}_2(\text{acac})_2]$  afforded a series of distorted octahedral *cis*- $\text{MoO}_2^{2+}$  complexes of the  $[\text{MoO}_2\text{L}(\text{CH}_3\text{OH})] \cdot \text{CH}_3\text{OH}$  (**1-4**) and  $[\text{MoO}_2\text{L}(\text{CH}_3\text{OH})_3]$  (**5**) type. Coordinated  $\text{CH}_3\text{OH}$  molecule, positioned *trans* to oxido oxygen atom, could be readily removed from first coordination sphere of molybdenum(VI) cation, yielding a  $[\text{MoO}_2(\text{L})]$  intermediate responsible for catalytic activation of substrates during Lewis acid catalyzed transformations.

The supramolecular architecture of complexes is dominated by two types of hydrogen bonds (HB):  $\text{O}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$ . The supramolecular motif shaped by  $\text{O}-\text{H}\cdots\text{O}$  HB in **2**, **3** and **4** differs in comparison with that formed by the same type of HB in **1** and **5**.

The infinite chains in **2**, **3** and **4** are shaped via the  $\text{O}-\text{H}\cdots\text{O}$  HB which include the coordinated  $\text{CH}_3\text{OH}$  oxygen atom,  $\text{CH}_3\text{OH}$  solvent molecule and the oxido oxygen atom of the complex molecule (Fig. 1a). On the contrary, the supramolecular dimers in **1** are formed by two molecules of solvent  $\text{CH}_3\text{OH}$  and two complex molecules via the phenolate oxygen atom and oxygen atom from coordinated  $\text{CH}_3\text{OH}$  (Fig. 1b). Formed dimers are condensed with complex molecules via  $\text{C}-\text{H}\cdots\text{O}$  intermolecular hydrogen bond which form centrosymmetric puckered 15-membered ring. (Figure 1b). Complex **5** does not contain solvent molecule of crystallization and the main supramolecular synthon formed via  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bond is centrosymmetric dimer between the  $-\text{OH}$  group of coordinated methanol and the oxygen donor atom of five-membered chelate ring of another complex molecule (Fig. 1c).

The determination of supramolecular role of  $\text{CH}_3\text{OH}$  molecules is essential in the context of complexes solubility and their catalytic activity in the conditions of epoxidation.



**Figure 1.** Supramolecular motifs in complexes: 3 (a), 1 (b) and 5 (c).

**Keywords:** molybdenum(VI) complexes, Schiff bases, supramolecular assembling

## MS30-P40 Novel complexes of copper(II) with tridentate hydrazone ligands

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New complexes with different structures and properties were obtained by the reaction of *tetrakis*( $\mu$ -acetato)diaquadicopper(II) with appropriate hydrazone ligand.

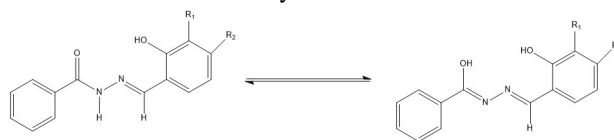
Hydrazones represent the most common type of tridentate ONO donors. They are stabilized by ketamine-enolimine tautomerization as shown by scheme 1.

Hydrazone ligands  $H_2L^1$ ,  $H_2L^2$  and  $H_2L^3$ , used in this investigation, were prepared by condensation of benzohydrazide with different aldehydes: salicylaldehyde ( $R_1 = H$ ,  $R_2 = H$ ), 3-metoxysalicylaldehyde ( $R_1 = OCH_3$ ,  $R_2 = H$ ) and 4-metoxysalicylaldehyde ( $R_1 = H$ ,  $R_2 = OCH_3$ ), respectively.

Reactions of *tetrakis*( $\mu$ -acetato)diaquadicopper(II) with  $H_2L^1$ ,  $H_2L^2$  or  $H_2L^3$  in methanol resulted in formation of polymers of the general formula  $[Cu(L)]_n$  while in the case of reaction with  $H_2L^2$  a cuban type tetranuclear cluster  $[Cu(L)]_4$  is also obtained. Addition of pyridine to the reaction mixtures of  $[Cu(OAc)_2(H_2O)]_2$  and  $H_2L^1$ ,  $H_2L^2$  and  $H_2L^3$  resulted in formation of dimeric complexes  $[Cu(L)(py)]_2$  with pyridine molecules coordinated to copper atoms. Mononuclear complex  $[Cu(L^3)(D)]$  was isolated when in reaction mixture 1,10-phenantroline was added. These complexes are unstable and upon standing at room temperature lose pyridine molecules yielding  $[Cu(L)]_2$ . After exposure of  $[Cu(L)]_2$  to pyridine vapours, it is possible to recover the starting complex. This is accompanied with colour change from pale green to original dark green.

Preliminary magnetochemical measurements of prepared dinuclear and tetranuclear complexes indicate antiferromagnetic arrangement of spins at low temperatures.

All isolated complexes were identified by elemental and thermogravimetric analysis, IR spectroscopy and powder X-ray diffraction method. In case of  $[Cu(L)(py)]_2$  ( $L=L^1$ ,  $L^2$  or  $L^3$ ),  $[Cu(L^2)]_4$  and  $[Cu(L^3)(D)]$ , single crystal X-ray diffraction was additionally used.



**Figure 1.** Scheme 1. Ketamine-enolimine tautomerization of hydrazones

**Keywords:** hydrazones, dimeric complexes, cuban type clusters