

of aromatic dicarboxylic acids and N-donors such as imidazole ligands, which resulted in compounds having 1D to 3D frameworks with interesting physicochemical properties. A large number of novel architectures assembled through the combination of 1,4-benzenedicarboxylic acid (terephthalic acid) and imidazole ligand have been reported [3][4][5][6].

In our course to obtain extended structures with mixed ligands, a mixture of $\text{Co}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$, terephthalic acid and imidazole was heated at 60°C . Pink single crystals were isolated after several days. Unfortunately, no polymeric structure was obtained but monomeric complex $[\text{Co}(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_3\text{H}_4\text{N}_2)(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}$ was isolated. It has three isomers: *fac*, *trans*, *mer* and *cis*, *mer*. The structure of the title compound is the latter, with two aqua ligands *cis* and three N-coordinated imidazole ligands meridional, relative to the $\text{Co}(\text{II})$ center.

The asymmetric unit of the neutral complex contains one Co^{II} ion, two coordination water molecules, three imidazole ligands, one fully deprotonated terephthalate ligand and one free water molecule. The $\text{Co}(\text{II})$ center is coordinated by one oxygen atom from one monodentate terephthalate ligand and two water molecules and three nitrogen atoms from three imidazole ligands, in a slightly distorted octahedral coordination geometry. The imidazole molecules act as typical monodentate ligands terminally coordinated to the metal center. Notably, only one of two carboxylates of terephthalate ligand coordinates to the central $\text{Co}(\text{II})$ atom in monodentate mode and the second one, which is deprotonated, remains free of coordination. The two carboxylate groups are respectively inclined at $18.76(15)^\circ$ to each other. In the former (O1-C1-O2) the carboxylate group is almost coplanar with the plane of its parent aromatic ring, and the other is rotated out of this plane by $17.27(20)^\circ$. The dihedral angles between imidazole co-ligands are $45.90(7)^\circ$, $61.58(6)^\circ$ and $76.56(7)^\circ$, respectively.

The geometry of the complex is constrained by one intramolecular O-H...H bond formed between one hydrogen atom of an aqua ligand, and adjacent terephthalic carbonyl oxygen atom.

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Keywords: terephthalate, cobalt, imidazole.

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Polymorphic crystallization and kinetic pathways of triacylglycerols

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The kinetic properties of crystallization and structural transformation are important, particularly for the application of polymorphic crystal systems in pharmaceutical, biomedical, food technology, and other applications.

Triacylglycerols (TAGs) are the main components of alimentary fats and oils. Fat structures and compositions determine their physical properties (e.g., rheology, morphology, and texture), where polymorphism exerts a strong influence. There are different

polymorphic forms of TAGs: the most general ones (in increasing stability) are: α form, which crystallizes in the hexagonal system, β' form (orthorhombic system) and β form (triclinic system).

In this study, several techniques, like Synchrotron Radiation X-Ray Diffraction, Thermal Analysis and Thermo-optical Microscopy, were used in order to characterize the polymorphism under the kinetic point of view of some TAGs present in edible oils and fats. Therefore, the role of some kinetic parameters (e.g. cooling and heating rates) is highlighted.

Keywords: fat, polymorphism, nucleation rate

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Conformational polymorphism and order-disorder phase transition in Enalapril-Zn complex

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Polymorphism is the capacity of a substance to exist in more than one crystalline form [1]. When different conformers of the same molecule occur in different crystal forms, the phenomenon is named conformational polymorphism. A classical example of this polymorphism is observed in ferrocene which presents one room-temperature disordered phase and two low-temperature ordered crystalline forms [2,3]. Conformational polymorphs crystals may or not interconvert through a phase transition.

Some crystals of the enalapril-Zn complex were analyzed by X-ray diffraction at room temperature (298 K) and low temperature (150 K). This analysis was performed on a Oxford GEMINI A-Ultra diffractometer with $\text{CuK}\alpha$ radiation ($\lambda = 1.54184 \text{ \AA}$). The structure was solved and refined by means of SHELXL-97 program [4]. These crystal structures showed the presence of conformational polymorphism. Significant differences in molecular conformation are essentially localized in torsion angles. These polymorphs interconverted to each other by a phase transition. Figure 1 shows the crystal structure of complex formed at low and room temperature, respectively. Order-disorder phase transition is observed in these crystals, since the crystalline structure of enalapril-Zn complex is ordered at low temperature but presents occupational disorder at room temperature.

Crystal data are summarized next.

Enalapril-Zn (room temperature): orthorhombic, C222, Z=2, $a=24.870(2)$, $b=21.002(2)$, $c=9.230(2) \text{ \AA}$, $R=0.067$, $wR=0.184$, $s=1.044$, for 2911 reflections.

Enalapril-Zn (low temperature): orthorhombic, P2₁2₁2₁, Z=4, $a=23.655(2)$, $b=20.840(2)$, $c=17.519(2) \text{ \AA}$, $R=0.037$, $wR=0.098$, $s=1.022$, for 12453 reflections.

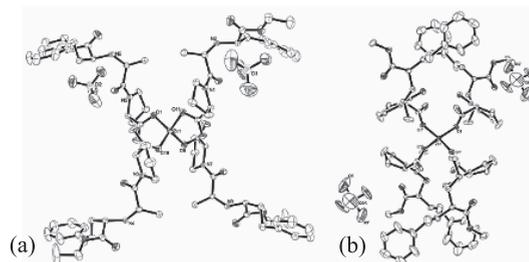


Figure 1: Crystal structure of the enalapril-Zn complex (a) low temperature and (b) room temperature. Symmetry codes: (i) $-x, -y, z-1$; (ii) $x, -y, -z$; (iii) $-x, -y, z$; (iv) $-x, y, -z$; (v) $-x+1/2, -y+1/2, z$; (vi) $x-1/2, y-1/2, z-1$.

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Keywords: polymorphism, phase, transition

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A thallium intermediate compound trapped in a crystal and its SCSC evolution

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The identification and isolation of intermediate compounds are of importance in chemistry for understanding reaction mechanisms, and because of their special reactivities and sometimes for their special bonding or structural characteristics. However, the isolation of these species is in general rare due to their exceptional chemical reactivity and/or lower thermodynamic stability in relation to other species.

We present the identification of a CoTl compound (**1**) which is an intermediate in the formation of a heterometallic CoTl polymer with bridging acetates (**2**). Compound **1**, which is also a polymer, has been trapped as a minor species in crystals of the CoTl coordination polymer (**2**) and evolves to (**2**) when heated.

The crystal structures of the compounds and their evolution when heated have been studied using the same single crystal for all measurements. The Tl atoms are bonded to oxygen from the carboxylate groups in both polymers. The carboxylate groups are also bonded to the cobalt centers, thus acting as bridges between the Tl and the Co. However, both metals (Co and Tl) are ligated by the same oxygen atom [O(1)] in compound **1**, Fig. 1, while in compound **2**, the metals are bonded to different oxygen atoms of the carboxylate group, [O(1), O(2)], Fig. 2.

When heated, the Tl atom in compound **1** moves away from O(1) [from 2.798(3) Å to 3.044(3) Å] and gets closer to O(2) [from 3.348(3) Å to 2.735(3) Å], Fig. 3, thereby changing the nature of the bridge acetate between Co and Tl.

There are few heterometallic compounds of Co and Tl and in the only carboxylate derivative to have been characterized structurally, the carboxylate group bridges only Co atoms[1]. The thallium atom in the intermediate compound (**1**) is three coordinated, with a nearly planar geometry around the thallium center, quite distinct from the usual pyramidal geometry found for three-coordinate Tl(I).

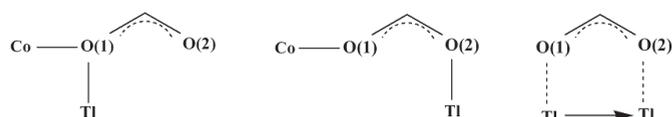


Fig. 1

Fig. 2

Fig. 3

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Keywords: thallium, intermediate, transformation

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Di- and Tetranuclear Zinc(II)-complexes of a new ditopic-hydrazone ligand

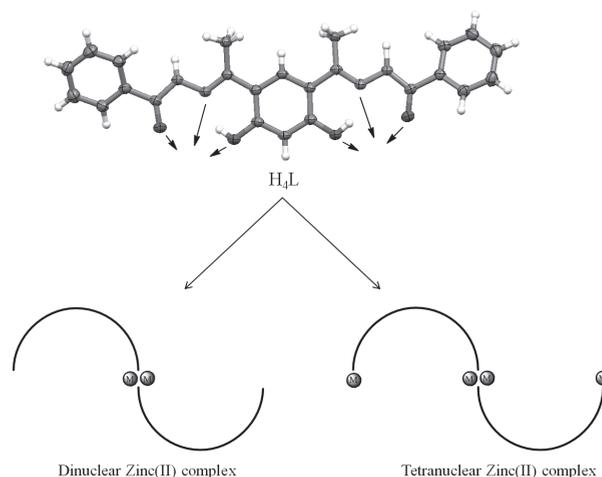
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Coordination chemistry of polytopic-hydrazone ligands is an interesting area of research [1]. These kind of organic compounds are useful to coordinate more than one metal center into $[n \times n]$ grid complexes [1, 2] or polymeric structures [3]. Moreover, magnetic and/or optical and structural properties are in continuous investigation [4].

Herein, we report the synthesis and structural characterization of a new ditopic hydrazone ligand, **H₄L**, a dinuclear zinc(II)-complex **1** ($[\text{Zn}(\text{H}_2\text{L})(\text{dmsO})_2]$) and a tetranuclear one, **2**, ($[\text{Zn}_2(\text{L})(\text{dmsO})_3]_2 \cdot 2\text{dmsO}$) (Scheme 1). Both compounds were obtained by reaction of equimolar amounts of zinc(II) acetate and **H₄L** using conventional and solvothermal mediated reactions, respectively. Formation of an hydrolyzed-ligand-zinc-complex was also obtained ($[\text{Zn}(\text{HL}^*)(\text{H}_2\text{O})_2](\text{dmf})$, **3**) when an excess of zinc(II) nitrate was used.

Ligand **H₄L** contains two ONO-binding pockets capable of coordinate one or two metal ions. This structural versatility has been shown in complexes **1** and **2**. Free and coordinated pockets co-exist together in the dinuclear complex **1** and all the pockets are occupied in complex **2**.

In addition, oxygen atom of the hydroxyl group bridges two metal centers forming a Zn_2O_2 diamond core.



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Keywords: hydrazone, ditopic ligand, polymetallic complex.