

Copper(II) Complex

Subjects: [Chemistry, Applied](#) | [Chemistry, Inorganic & Nuclear](#) | [Chemistry, Physical](#)

Contributor: Dayane Reis , Francielli Santana

A dinuclear copper(II) complex of (1) (where *bipy* = 2,2'-bipyridine, *bzt* = benzoate and *ox* = oxalate) was synthesised and characterised by diffractometric (powder and single-crystal XRD) and thermogravimetric (TG/DTG) analyses, spectroscopic techniques (IR, Raman, electron paramagnetic resonance spectroscopy (EPR) and electronic spectroscopy), magnetic measurements and density functional theory (DFT) calculations. The analysis of the crystal structure revealed that the oxalate ligand is in bis(bidentate) coordination mode between two copper(II) centres. The other four positions of the coordination environment of the copper(II) ion are occupied by one water molecule, a bidentate *bipy* and a monodentate *bzt* ligand. An inversion centre located on the *ox* ligand generates the other half of the dinuclear complex. Intermolecular hydrogen bonds and pi-pi for the organisation of the molecules in the solid state. Molar magnetic susceptibility and field dependence magnetisation studies evidenced a weak intramolecular-ferromagnetic interaction ($J = +2.9 \text{ cm}^{-1}$) between the metal ions. The sign and magnitude of the calculated J value by density functional theory (DFT) are in agreement with the experimental data.

dinuclear copper(II)

ferromagnetic interaction

magnetic properties

noncovalent interaction

1. Introduction

Copper(II) complexes are interesting in coordination chemistry because of their vast applicability for bioinorganic purposes and synthesis of metallodrugs,^{[1][2]} catalysis^{[3][4]} and magnetism.^{[5][6]} Concerning molecular magnetism, it is known that the magnetic interaction between two or more copper(II) centres is strongly dependent on the nature of the bridging ligand that works like a magnetic exchange pathway.^{[7][8]} Among these ligands, the oxalate ion, ox^{2-} , for example, is well known for its capability to create adequate magnetic exchange pathways for ferro- and antiferromagnetic interactions in oligonuclear copper(II) compounds.^{[9][10][11]} The combination of oxalate and copper(II) ions leads to a large structural variety including different nuclearities such as mononuclear,^{[12][13]} dinuclear,^{[14][15]} trinuclear,^{[16][17]} tetranuclear^{[17][18]} and hexanuclear species^{[19][20]} and coordination polymers.^{[14][21]} This class of oxalate-bridged compounds is noteworthy in magnetic applications, as it may comprise many other transition metal ions such as Mn^{II} , $\text{Fe}^{\text{II/III}}$, Co^{II} , Ni^{II} , $\text{Cr}^{\text{II/III}}$, V^{IV} and Ru^{II} .^{[22][23][24][25]}

2. Dinuclear Copper(II) Complexes

The dinuclear copper(II) complexes, which contain only one unpaired electron per metallic ion, are the most straightforward systems to investigate the through-ligand electron exchange mechanism from experimental and theoretical perspectives.^{[26][27][28]} It is well known that the nature and strength of the magnetic interactions in

dinuclear complexes including simple inorganic and extended organic bridging ligands such as hydroxo, azide, aromatic dicarboxylate, diamine, oxalate and related derivatives are highly dependent on the nature of the chelating terminal blocking ligands, which prevent complex polymerisation.^[5] Furthermore, the variation in the spatial arrangement of terminal ligands may change the orbital overlap angle between the copper(II) and the bridging ligand, leading to different types of magnetic interactions.^{[29][30]}

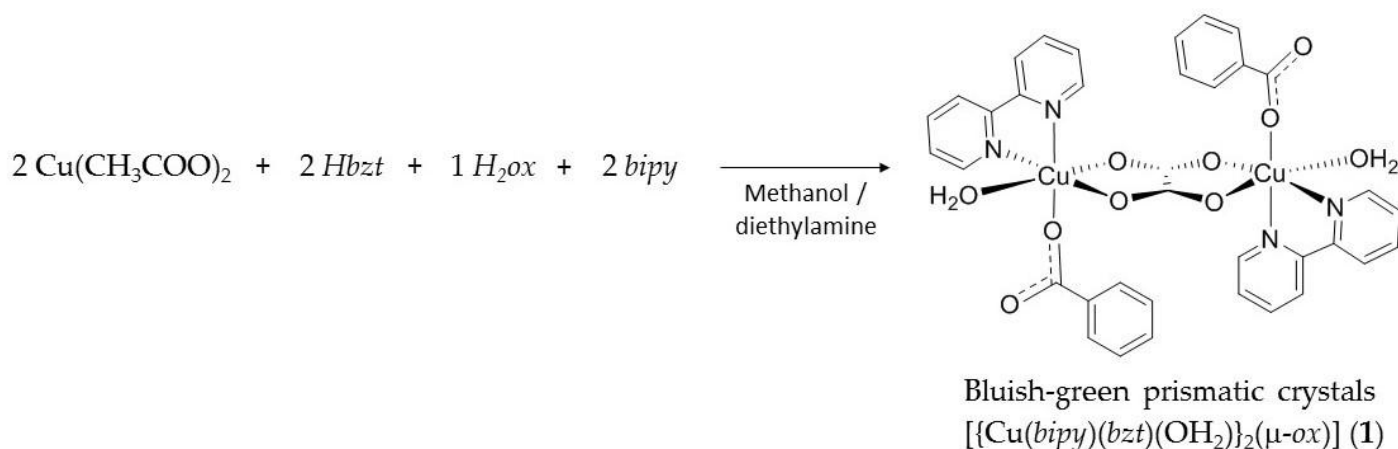
Regarding oxalate-bridged copper(II) compounds, the ferromagnetic or antiferromagnetic behaviour is determined by the overlap between the two magnetic orbitals. Poor overlap through oxalate leads to a weak antiferromagnetic interaction. On the other hand, if the overlap is zero, one could expect a ferromagnetic coupling.^[8] Steric hindrance arising from the volume of the ancillary ligands leads to different orientations of the magnetic orbitals concerning the bridge plane, hence generating a different magnitude and nature of the magnetic interaction among the copper(II) centres.^[17]

Although a variety of oxalate (ox^{2-})-bridged copper(II) complexes can be found in the literature, just a few contain more than one bulky ligand besides the oxalate bridge. A detailed search carried out in the Cambridge Structural Database (CSD)^[31] for oxalate-bridged copper(II) complexes with ox^{2-} in bis-bidentate coordination mode displayed a total of 423 results, of which 86 correspond to dinuclear complexes that possess two additional ligands besides ox^{2-} . Out of 86 structures, 66 contain pentacoordinate metal ions bound to only one type of organic ligand such as pyridine,^[1] 2,2'-bipyridine,^{[11][32]} phenanthroline,^{[33][34]} ethylenediamine,^[35] pyrazole^{[36][37]} and imidazole^[38] and their derivatives, the coordination sphere being completed by simple inorganic ions such as nitrate,^[39] chloride,^[33] hydroxide^[40] and perchlorate,^[1] or by solvent molecules such as water,^[6] methanol,^[34] dimethylformamide,^[41] tetrahydrofuran^[42] or acetonitrile.^[43] In the 20 remaining structures, all of which have been reported more recently, the metal ions are hexacoordinate and the chemical composition of the complexes, regarding the presence of at least one organic ligand, solvent molecules and simple inorganic ions, is similar to that observed for the pentacoordinate species.^{[8][11][14][15][44][45]} Exceptions are found when tetradentate ligands direct hexa-coordination, decreasing the need for other molecular entities to complete the coordination sphere of the copper(II) ion.^{[46][47]} Therefore, such systems show high structural and electronic versatility, which can be modulated by an appropriate choice of ligands other than the ox^{2-} bridge.

Herein the synthesis of a new heteroleptic oxalate-bridged copper(II) dinuclear complex $[\{Cu(bipy)(bzt)(OH_2)\}_2(\mu-ox)]$ (**1**) (where *bipy* = 2,2'-bipyridine, *bzt* = benzoate and *ox* = oxalate) is reported, in which the coordination environment of the metal ion is described as distorted octahedral with the unusual coordination of two relatively bulky ligands, a bidentate 2,2'-bipyridine and a monodentate benzoate, in addition to one water molecule. Structural correlations on how the orientation of the magnetic orbitals of the Cu^{II} ions is affected by the crystal field in **1** and the nature of magnetic interactions were investigated. We have also carried out theoretical calculations on the electronic structure and the electron paramagnetic resonance spectroscopy (EPR) spectra to bring light into its magnetic properties.

The reaction between copper(II) acetate monohydrate, benzoic acid, 2,2'-bipyridine and oxalic acid dihydrate in methanol gave the bluish-green prismatic shape crystals of $[\{Cu(bipy)(bzt)(OH_2)\}_2(\mu-ox)]$ (**1**) in high yield, ca 90%,

from a reproducible synthetic methodology (Scheme 1). Powder X-ray diffraction (PXRD) analysis revealed good correspondence between the simulated and experimental diffraction patterns.



Scheme 1. Synthetic methodology carried out between $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$, Hbzt , $\text{H}_2\text{ox} \cdot 2\text{H}_2\text{O}$ and bipy to produce complex **1**. Where bipy = 2,2'-bipyridine, bzt = benzoate and ox = oxalate.

3. Conclusions

A new dinuclear copper(II) complex was synthesised and characterised. The crystal structure showed that $[\{\text{Cu}(\text{bipy})(\text{bzt})(\text{OH}_2)\}_2(\mu\text{-ox})]$ (**1**) is centrosymmetric and contains two rather bulky ligands, benzoate and 2,2'-bipyridine, while the majority of the complexes of this class contain just one bulky ligand. The magnetic interaction predicted by EPR was confirmed by magnetic measurements and indicated ferromagnetic coupling between the metal centres. The determined J value was supported by DFT calculations. Therefore, the present study highlights the importance of the oxalate bridge as a magnetic-exchange pathway, and how bulky ancillary ligands can affect the magnetic response.

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