

Synthesis, Structure And Thermogravimetric Analysis Of Copper(II) Complex Derived From N^1, N^3 -Bis(1-(Pyridin-2-Yl)Ethylidene)Propane-1,3-Diamine

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Abstract:

Cu(II) complex derived from ligand, (N^1, N^3 -bis(1-(pyridin-2-yl)ethylidene)propane-1,3-diamine (L) which was synthesized in situ, is reported. The isolated Cu(II) complex was characterized by elemental analyses, IR, and UV-Vis spectroscopies, and Thermogravimetric analyze (TGA). Additionally, the structure of the complex was determined by single crystal X-ray diffraction study. The diimine ligand was coordinated to Cu(II) ion through two azomethine nitrogen atoms and two pyridine nitrogen atom. One bromide ion complete the coordination sphere yielding a distorted square pyramidal geometry. The structure of the copper(II) complex is confirmed by X-ray diffraction. The complex crystallizes in the monoclinic space group $P2_1/c$ with unit cell dimensions $a = 8.3433(3) \text{ \AA}$, $b = 13.2605(3) \text{ \AA}$, $c = 18.838(6) \text{ \AA}$, $\beta = 97.522(16)^\circ$, $V = 2066.2(6) \text{ \AA}^3$, $Z = 4$, $R_1 = 0.048$ and $wR_2 = 0.081$. The Cu(II)cation in N_4Br inner is situated in a distorted square pyramidal environment. Thermogravimetric analyze of the complex were carried out at 20–800 °C and showed that the complex were decomposed into three steps.

Keywords: 2-acetylpyridine, 1,3-diaminopropane, Copper(II) complex, spectroscopy, TGA, X-ray diffraction.

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I. Introduction

Transition metal complexes of amine ligands are important compounds in coordination chemistry because of their interest in various applications such as the preparation of bioactive compounds, catalysts, magnetic compounds and compounds with optical properties [1–9]. Transition metal coordination chemistry has developed with the use of organic compounds functionalized with amino groups. The tendency of polyamines to form chelated compounds is exploited by carrying out condensation reactions with ketoprecursors to give new organic molecules. The latter make it possible to produce coordination compounds with specific properties [10–13]. Diimine ligands and their transition metal complexes have undergone considerable development due to their ease of synthesis, the presence of several types of donor sites, the easy combination of hard and soft sites such as nitrogen, oxygen or sulfur. The stability of the ligands and complexes generated and the numerous applications in various fields, such as medical imaging, catalysis, biomimetic chemistry, design of magnetic or optical molecular materials [14–17] are important assets for the development of coordination chemistry. Several methods are used in the preparation of these coordination complexes. One can prepare and isolate the ligand first or prepare the ligand in situ and react to it with the chosen metal ion. Most of the time the complexes are prepared from the isolated ligand. In this work we have chosen the in situ method to prepare the Cu complex which is characterized by various spectroscopic methods, by thermogravimetric analysis and X-ray diffraction.

II. Materials And Physical Methods

Starting materials and Instrumentations

All chemicals and solvents were of analytical reagent grade and were used directly without further purification. The ligand N^1, N^3 -bis(1-(pyridin-2-yl)ethylidene)propane-1,3-diamine (L) was synthesized according to the literature [18]. Elemental analyses of C, H and N were recorded on a VxRio EL Instrument. Infrared spectra were obtained on a FTIR Spectrum Two of Perkin Elmer spectrometer in the 4000–400 cm^{-1} region. The UV-Visible spectra were recorded on a Perkin Elmer Lambda UV-Vis spectrophotometer. The

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molar conductance of 1×10^{-3} M in DF solutions of the metal complexes was measured at 25 °C using a WTW LF-330 conductivity meter with a WTW conductivity cell. Room temperature magnetic susceptibilities of the powdered samples were measured using a Johnson Matthey scientific magnetic susceptibility balance (Calibrant: Hg[Co(SCN)₄]).

Synthesis of the complex

In a 100 mL flask, 2-acetylpyridine (0.242 g; 2 mmol) was introduced and then added 1,3-diaminopropane (0.074 g; 1 mmol) previously dissolved in 5 mL of methanol was added. Two drops of glacial acetic acid were added. The mixture was refluxed for two hours, and the initially colorless solution takes on a yellow color. After cooling, CuBr₂ (0.223 g; 1 mmol) dissolved in 5 mL of methanol was added. The dark green solution was refluxed for two hours. On cooling, the solution was filtered, and the filtrate was left to evaporate slowly for two weeks. Green crystals suitable for X-ray diffraction analysis were collected. The compound is soluble in common organic solvents such as MeOH, DMSO and DMF. Yield = 50%; Melting point > 260°C. Anal. Calc for C₁₇H₂₄N₄Br₂O₂Cu: C, 37.83; H, 4.48; N, 10.38. Found: C, 37.81; H, 4.45; N, 10.35. IR ν (cm⁻¹): 3490, 1601, 1430, 490, 459, 339, 316. UV-visible (λ (nm)): 255, 309, 369, 658. Conductance Λ (S·cm²·mol⁻¹): 82 [fresh solution]; 85 [15 days after]. Magnetic moment : $\mu_{\text{eff}} = 1.8 \mu_{\text{B}}$.

Crystal structure determination

Details of the X-rays crystal structure solution and refinement are given in Table 1. Diffraction data were collected using an ENRAF NONIUS Kappa CCD diffractometer with graphite monochromatized MoK α radiation ($\lambda = 0.71073$ Å). All data were corrected for Lorentz and polarization effects. No absorption correction was applied. Complex scattering factors were taken from the program package SHELXTL [19]. The structures were solved by direct methods which revealed the position of all non-hydrogen atoms. All the structures were refined on F^2 by a full-matrix least-squares procedure using anisotropic displacement parameters for all non-hydrogen

atoms [20]. The hydrogen atoms of water molecules and NH groups were located in the Fourier difference maps and refined. Others H atoms (CH and CH₃ groups) were geometrically optimized and refined as riding models by AFIX instructions. Molecular graphics were generated using ORTEP-3 [21].

Table-1. Crystallographic data and refinement parameter for the complex.

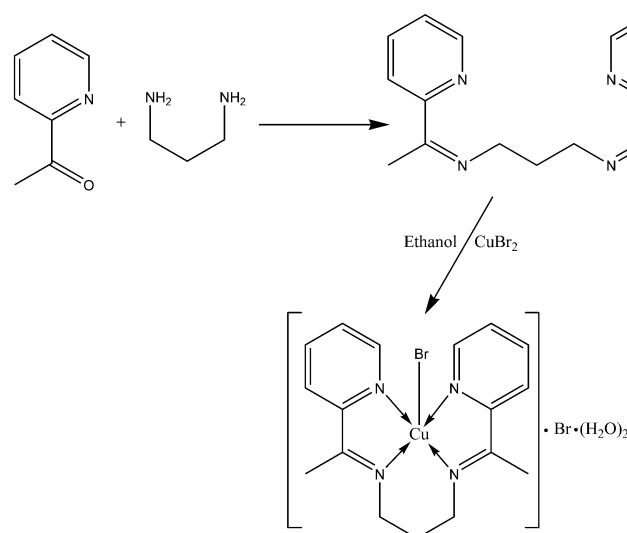
Chemical formula	C ₁₇ H ₂₀ BrCuN ₄ ·Br·2(H ₂ O)
M_r	539.76
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
Crystal size (mm)	0.19 × 0.14 × 0.12
a (Å)	8.3433 (16)
b (Å)	13.2605 (13)
c (Å)	18.838 (3)
β (°)	97.522 (16)
V (Å ³)	2066.2 (6)
Z	4
D_{calc} (g·cm ⁻³)	1.735
Mo $K\alpha$ (Å)	0.71073
T (K)	293
μ (mm ⁻¹)	4.95
Index ranges	$-12 \leq h \leq 11, -19 \leq k \leq 19, -27 \leq l \leq 25$
$F(000)$	1076
θ range (°)	3.069-31.015

No. of measured	33896
No. independent	6581
No. observed [$I > 2\sigma(I)$] reflections	3415
R_{int}	0.090
$R[F^2 > 2\sigma(F^2)]$	0.048
$wR(F^2)$	0.081
Goodness-of-fitt (Gof) on F^2	1.01
No. of reflections	6581
No. of parameters	249
No. of restraints	6
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ ($e \text{ \AA}^{-3}$)	0.80, -0.66

III. Results And Discussion

General studies

The ligand N^1, N^3 -bis(1-(pyridin-2-yl)ethylidene)propane-1,3-diamine (L) was prepared by a facile condensation of 1,3-diaminopropane and 2-acetylpyridine in ethanol (Scheme 1). The copper complex was synthesized by mixing ligand solution with copper dibromide in a molar ratio 1:1. The afforded compound is soluble in polar organic solvent such as methanol or DMSO. Elemental analyses gives result in agreement with the chemical formula obtained from X-ray diffraction study. The complex behave as 1:1 electrolyte in DMF with Λ value of $82 \text{ } \Omega^{-1}\cdot\text{cm}^2\cdot\text{mol}^{-1}$ in DMF [22]. The complex show paramagnetic properties with μ_{eff} of $1.8 \text{ } \mu_{\text{B}}$. This magnetic moment value is in accordance with one unpaired electron in d^9 configuration [23]. The IR spectrum of the complex shows a broad absorption band at 3490 cm^{-1} which is attributed to the $\nu_{\text{O-H}}$ vibration of the free water molecule. The band at 1601 cm^{-1} is due to the $\nu_{\text{C=N}}$ vibration of the azomethine groups. The low value of the $\nu_{\text{C=N}}$ vibration is indicative of the involvement of azomethine nitrogen atom in the coordination to the Cu atom. In fact, the value of the reported $\nu_{\text{C=N}}$ vibration for analogous ligands is higher than this value [24]. Coordination is confirmed with the apparition of a new band at 459 cm^{-1} due to the $\nu_{\text{Cu-N}}$ [25]. The band identified in the low frequency region at 339 cm^{-1} is attributed to the $\nu_{\text{Cu-O}}$ vibration [26]. The $\nu_{\text{Cu-Br}}$ frequency is observed at 316 cm^{-1} , which is consistent with a terminal Cu-Br band in bromo complex [27]. The electronic spectrum of the copper complex was recorded with millimolar DMF solution. The absorption band pointed at 309 nm is attributed to the ligand \rightarrow metal charge transfer. The bands at 255 nm and 369 nm absorption are due to the $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions of the ligand. This demonstrates metal coordination with the ligand. The absorption band centered at 658 nm is attributed to the $d \rightarrow d$ transitions of the Cu(II) ion in square pyramidal environment [28].



Scheme 1. Synthetic scheme for the ligand and it's Cu^{II} complex preparation.

Thermogravimetric study

Thermal decompositions of the complex has been investigated using TGA (Figure 1). The complex was heated in temperature ranging from 30 to 900 °C with a flow rate of 10.00 °C/min under N₂ atmosphere. The complex is stable up to 100 °C and then begins to decompose continuously (Fig). The mass loss of 6.3 % of around 100°C corresponds to the loss of the two water molecules (6.67 % of the molar mass). Between 187°C and 478°C, a mass loss of 29.58 % is noted which corresponds with the loss of the two bromide ions (29.64 % of the molar mass). In the range of 478°C - 635°C, the mass loss of 52.05% indicates that the organic molecule has undergone a complete degradation (51.87 % of the molar mass). Beyond this temperature, 12.07% of the mass remains which probably corresponds to the formation of CuO. This indicates that this complex may be used as single source precursor for the formation of metal oxide.

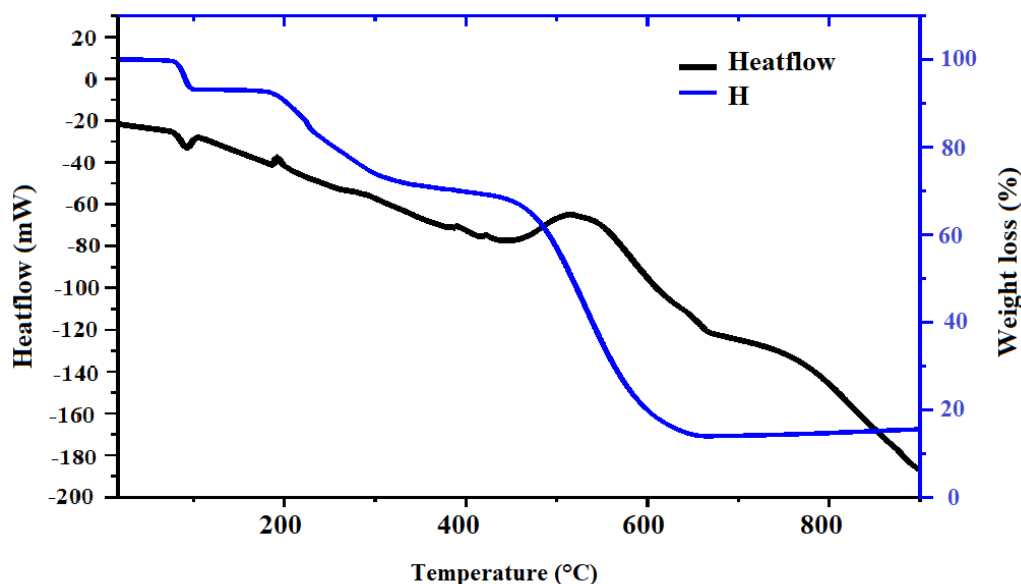


Figure 1. TGA curve of the complex.

Crystal structure

The analysis shows that the compound crystallizes in a monoclinic system with a space group P2₁/c. The lattice parameters are $a = 8.3433 \text{ \AA}$, $b = 13.2605 \text{ \AA}$, $c = 18.838 \text{ \AA}$ and $\beta = 97.552(16)^\circ$. Selected bonds distances and angles are reported in Table 2. The molecular structure of the compound with atomic labelling scheme is shown in Figure 2.

The crystal structure reveals that the asymmetric unit consists of a Cu²⁺ ion, an organic ligand *L*, two bromide ions and two water molecules. The ligand *L* is in its neutral form and coordinates to the Cu²⁺ ion in tetradentate fashion through two azomethine nitrogen atoms and two pyridine nitrogen atoms. One bromide ion is coordinated to the metal ion while another bromide ion and two water molecules remain uncoordinated. Therefore, the Cu²⁺ ion is pentacoordinated and its environment can be described as a trigonal bipyramid or a square pyramid. To resolve this ambiguity on the geometry of the complex, the Addison parameter [29] (τ) is used: $\tau = (\beta - \alpha) / 60$ where α and β are the two largest angles around the Cu²⁺ ion. A value of $\tau = 0$ corresponds to a perfect square pyramid geometry and a value of $\tau = 1$ indicates a perfect trigonal pyramid geometry. In this study, the value of $\tau = 0.372$ indicates a highly distorted square pyramid geometry around Cu²⁺. The base of the pyramid is formed by the atoms belonging to the ligand N1, N2, N3, N4 and the apical position is occupied by Br1. Indeed, the values of the *cisoid* angles formed by the atoms located on the base of the pyramid and which are in the interval [8.31(11)°-100.76(11)°] are different from the ideal value of 90°. The values of the *transoid* angles [N1—Cu1—N3 = 171.37(10)° and N2—Cu—N4 = 148.70(10)°] are far from the ideal value of 180°. The sum of the angles subtended by the atoms which are on the basal plane [355.82°] is different from the ideal value of 360°. All these differences observed with the expected values for a perfect geometry indicate a strong deformation of the geometry around the Cu²⁺. After coordination to the metal ion two five membered rings [CuNCCN] with bite angle values of 80.31 (11)° and 80.88 (12)° respectively, and one six membered ring [CuNCCCN] with bite angle value of 93.87 (1)°, were formed. The Cu1 atom is situated 0.337 Å out of the plane defined by the atoms in the basal plane. The Cu—N_{pyridine} bond lengths [Cu1—N1 = 2.004 (3) Å and Cu1—N4 = 2.032 (3) Å] are longer than the Cu—N_{imine} bond lengths [Cu1—N2 = 1.993 (3) Å and Cu1—N3 = 1.974 (3) Å]. This phenomenon is observed in an analogous complex {*N,N*-Dimethyl-*N'*-[1-(2-pyridyl)ethylidene]propane-1,3-diamine}bis(thiocyanato- \square *N*)copper(II) [30]. The length of the axial bond Cu1-

Br1 is 2.6584 (5) Å, is the longer than the Cu1-Br1 distance value of 2.5447 (7) Å in the analogous complex with the same ligand [18]. The Cu1-Br1 distance is consistent with the length of a Cu—Br_{terminal} bond [31, 32]. This axial bond elongation is due to the Jahn Teller effect which is characteristic of copper complexes [31, 33, 34]. The N3—C11 [1.278 (4) Å] and N2—C6 [1.279 (4) Å] bond distances indicate that these correspond to a double bond. The N3—C10 [1.462 (4) Å] and N2—C8 [1.460 (4) Å] bond distances are in the normal range for single C—N bonds [35]. In the crystal, intramolecular and intermolecular hydrogen bonds are simultaneously present. The crystal packing of compound is stabilized by two intramolecular O_(water)—H···Br and two intermolecular O_(water)—H···Br hydrogen bonds which form layers parallel to *b* axis. Additional C—H···O [C10—H10A···O2] hydrogen bonds consolidate the structure. Intermolecular hydrogen bonds, O1_(water)—H1A···Br2ⁱ (i: -x+2, -y+1, -z) and O2_(water)—H2B···Br1ⁱⁱ (-x+2, y+1/2, -z+1/2) lead to the formation of layers parallel to the *b* axis (Figure 3, Table 3). Additional C4—H4···O1ⁱⁱⁱ (iii: -x+1, -y+1, -z), C16—H16···O2^{iv} (iv: x, -y+1/2, z-1/2) and C14—H14···Br1^v (v: x+1, y, z), C1—H1···Br1^{vi} (vi: -x+1, -y, -z), C12—H12C···Br2^{vii} (vii: -x+2, y-1/2, -z+1/2) and C10—H10B···Br2^{viii} connect the layers and consolidate the structure into a three-dimensional network (Figure 4).

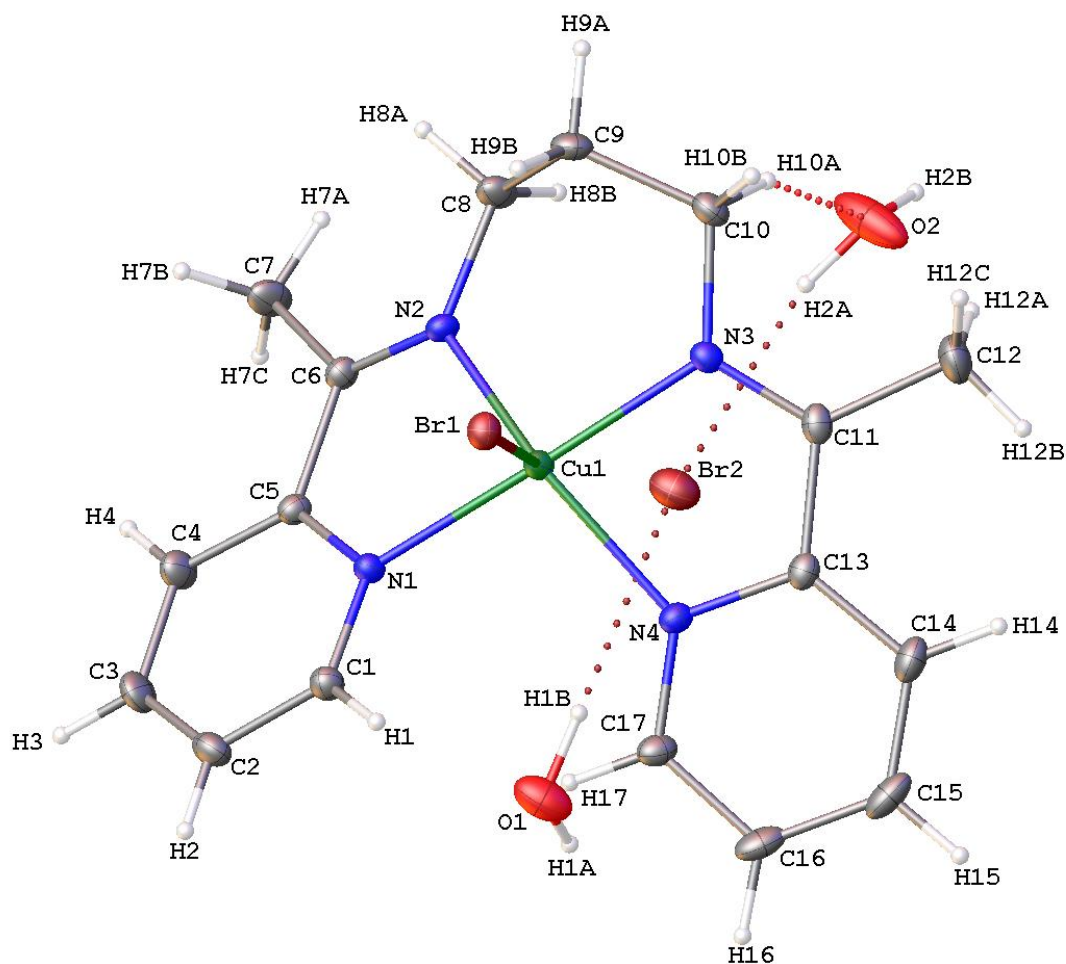


Figure 2: ORTEP plot (30% probability ellipsoids) showing the structure of compound.

Table-2. Selected bond distances [Å] and angles [deg] for the complex

Br1—Cu1	2.6584 (5)	N3—C10	1.462 (4)
Cu1—N3	1.974 (3)	N1—C1	1.322 (4)
Cu1—N2	1.993 (3)	N2—C6	1.279 (4)
Cu1—N1	2.004 (3)	N2—C8	1.460 (4)
Cu1—N4	2.032 (3)		
N3—Cu1—N2	93.87 (11)	N1—Cu1—N4	100.76 (11)
N3—Cu1—N1	171.37 (10)	N3—Cu1—Br1	92.50 (7)

N2—Cu1—N1	80.31 (11)	N2—Cu1—Br1	110.90 (7)
N3—Cu1—N4	80.88 (12)	N1—Cu1—Br1	95.53 (7)
N2—Cu1—N4	148.70 (10)	N4—Cu1—Br1	100.18 (7)

Table-3. Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1A...Br2 ⁱ	0.90(2)	2.49(2)	3.388(3)	176(4)
O1—H1B...Br2	0.91(2)	2.47(3)	3.370(3)	166(3)
O2—H2A...Br2	0.92(3)	2.41(3)	3.310(4)	165(5)
O2—H2B...Br1 ⁱⁱ	0.89(3)	2.48(3)	3.349(3)	165(5)
C4—H4...O1 ⁱⁱⁱ	0.93	2.55	3.478(5)	172.9
C16—H16...O2 ^{iv}	0.93	2.53	3.403(6)	155.6
C14—H14...Br1 ^v	0.93	2.77	3.625(4)	153.1
C1—H1...Br1 ^{vi}	0.93	3.01	3.739(3)	135.9
C12—H12C...Br2 ^{vii}	0.96	2.99	3.949(4)	173.3
C10—H10A...O2	0.97	2.56	3.437(5)	151.1
C10—H10B...Br2 ^{vii}	0.97	3.13	4.077(3)	164.5

Symmetry codes: (i) $-x+2, -y+1, -z$; (ii) $-x+2, y+1/2, -z+1/2$; (iii) $-x+1, -y+1, -z$; (iv) $x, -y+1/2, z-1/2$; (v) $x+1, y, z$; (vi) $-x+1, -y, -z$; (vii) $-x+2, y-1/2, -z+1/2$.

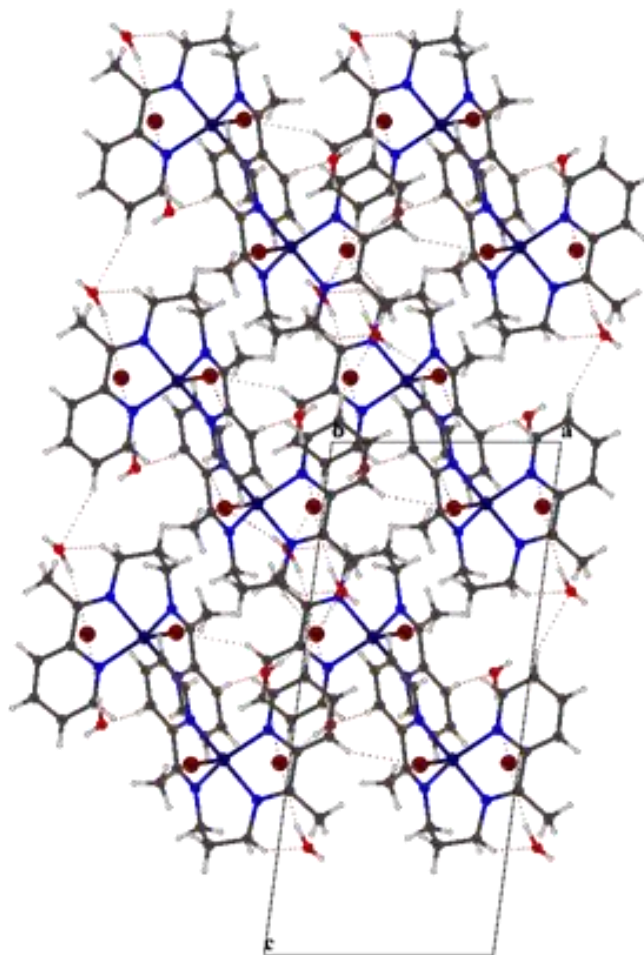


Figure 3: Layers of the trinuclear units parallel to the *b* axis.

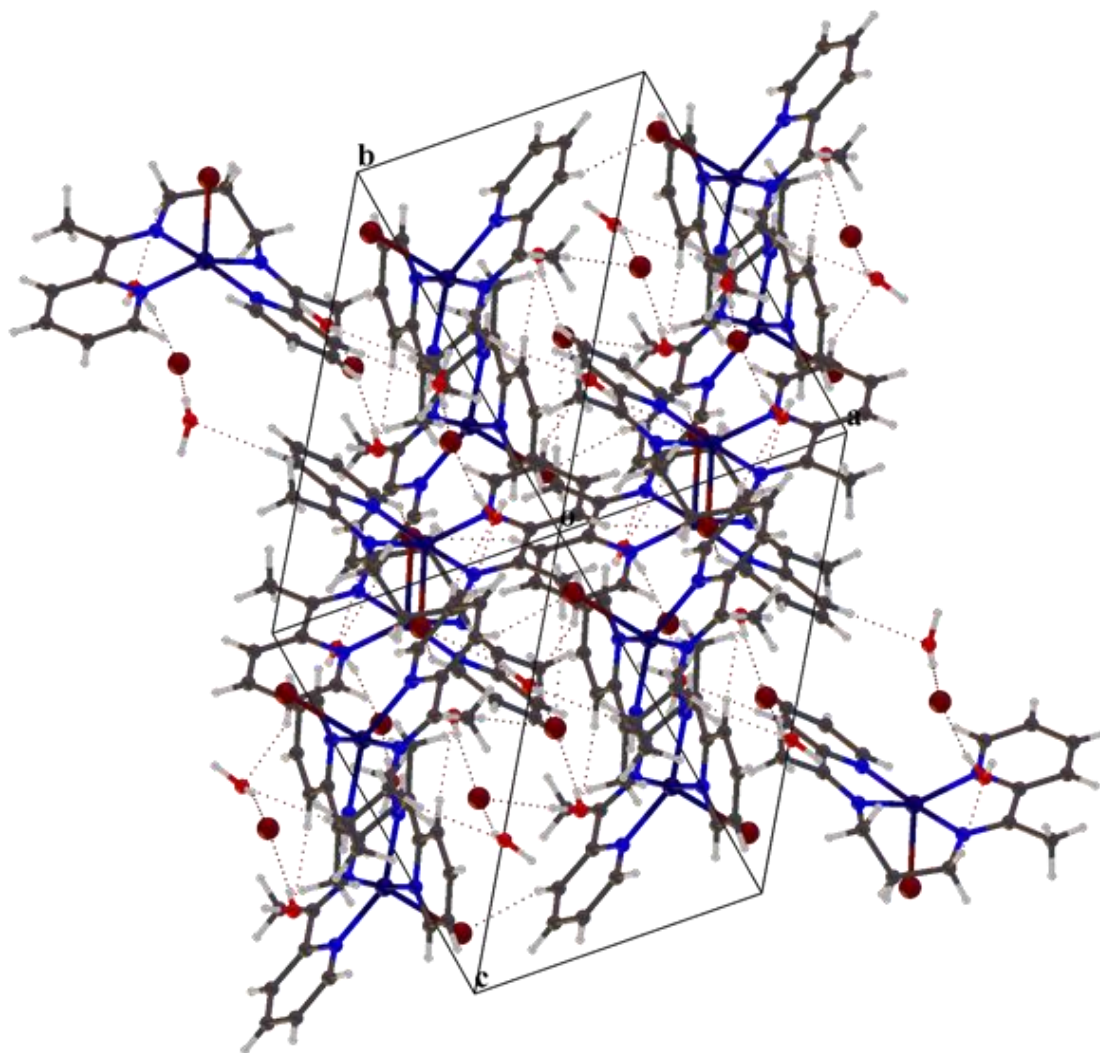


Figure 4: Crystal packing of the title compound

IV. Conclusion

The copper complex synthesized in this work is stable in air and insensitive to humidity and was recrystallized from ethanol without alteration. It was isolated in the form of crystals suitable for characterization by X-ray structure analysis. The spectroscopic and crystallographic techniques used allowed the complete characterization of the complex. The copper atom is located in an environment that is best described as a deformed square-based pyramid. TGA allowed the study of the behavior of the complex when increasing the temperature. A three-step decomposition of the complex is observed, and the residue correspond to the formation of CuO above 635 °C.

V. Supporting information

CCDC-1844619 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via <https://www.ccdc.cam.ac.uk/structures/>, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

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