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STRUCTURAL DIVERSITY OF *syn*-2-PYRIDINEALDOXIME IN Cu(II) COORDINATION POLYMER WITH BIPYRIDINE AS AUXILIARY LIGANDS

Aliona Vitiu [a*], Eduard Coropceanu [b], Paulina Bourosh [a]

[a] *Institute of Applied Physics, Moldova State University, 5, Academei str., Chisinau MD-2028, Republic of Moldova*
[b] *Institute of Chemistry, Moldova State University, 3, Academiei str., Chisinau MD-2028, Republic of Moldova*
[*] *e-mail: vitiualiona@gmail.com*

Abstract. The interaction of copper(II) nitrate, *syn*-2-pyridinealdoxime (PaoH), and 4,4′-bipyridine (bpy) in a 1:2:1 ratio (in mixture of water, methanol and *N*,*N*-dimethylformamide (dmf)) resulted in the formation of the compound $\{[Cu_4(Pao)_2(L)_2(bpy)_2](NO_3)_2{\cdot}2dmf{\cdot}4H_2O\}_n$. The coordinated ligand $(L)^{2-}$ is formed as a result of *in situ* chemical transformation of the PaoH ligand. The structure of this compound was determined by single-crystal X-ray diffraction. The compound has been found to be formed from a symmetric cationic coordination polymer, in which bridging bpy ligands link the structural units $[Cu_4(Pao)_2(L)_2]^{2+}$, nitrate anions, dmf and water as crystallization molecules. The outer-sphere components of the crystal are linked to the cation polymers by a system of hydrogen bonds. The intra- and intermolecular interactions present in the crystal were identified and quantified, and their 2D footprint diagrams were illustrated using Hirshfeld surface analysis.

Keywords: synthesis, coordination compound, copper(II), *syn*-2-pyridinealdoxime, X-ray study, Hirshfeld surface.

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Introduction

The chemistry of coordination compounds has undergone significant development in recent decades due to its theoretical and practical importance. The diversity of these compounds is enhanced by varying the metal ion or ligand employed, particularly organic ligands, which can influence the molecular design, supramolecular architecture, and properties of the resulting materials. Among heterocyclic ligands, *syn*-2-pyridinealdoxime (PaoH) is noteworthy because of its structural versatility and ability to form stable complexes with transition metals [1]. Moreover, several complexes containing this ligand exhibit interesting biological and anticancer properties [2-4]. A detailed analysis of data from the Cambridge Structural Database (CSD, The version 2025.1.1, subsets 6.00.) [1] concerning PaoH compounds with Cu(II), despite the limitations of the available structural information, indicates that the ligand is more frequently stabilized in its original form. Furthermore, the ligand exhibits various coordination modes toward the metal centre(s), leading to the formation of mono-, bi- and trinuclear complexes [5-8].

It has been observed that different Cu(II) starting salts significantly influence the formation of the metal coordination polyhedron, as well as the final supramolecular architecture of the crystal structure, owing to the effect of the inorganic anion. Anions may affect the structure of the compound either through direct coordination to the metal centres or by acting as "*templates*" for the rearrangement of the organic building blocks involved in the construction of infinite multidimensional networks. For example, the use of $CuCl_2$ resulted in the formation of the mononuclear complex [Cu(Pao)Cl(PaoH)] [5], which contains two bidentate *N*,*N*-donor ligands, monodeprotonated Pao and the neutral PaoH, together with a chloride anion. In addition, the copper atom in this complex exhibits a distorted square-pyramidal coordination geometry (4+1 type) with a distortion parameter $\tau = 0.288$. The structure of the *in situ* synthesized compound $Cu(PaoH)_2SO_4{\cdot}3H_2O$ [6] revealed that the complex also adopts a square-pyramidal geometry, stabilized by two neutral PaoH ligands coordinated in an *N*,*N*-bidentate chelating mode, while the sulphate anion acts as a counterion. Importantly, this compound catalyses the selective oxidation of veratryl alcohol (3,4-dimethoxybenzyl alcohol) to the corresponding aldehyde using molecular oxygen in aqueous solution. This process results in the deprotonation of the oxime groups of both PaoH ligands under alkaline conditions. Thus, it has been established that the conformational rigidity of pyridinaldoxime ligands favours the formation of discrete 0D compounds, whereas coordination polymers are obtained in the presence of additional bridging ligands [1]. The compounds $\{[Cu(\mu\text{-}ClO_4)(2\text{-}Pao)(2\text{-}PaoH)]\}$, $\{[Cu(oxa)(2\text{-}PaoH)]\cdot H_2O\}_n$, $\{[Cu_3(\mu_3\text{-}OH)(2\text{-}Pao)_3(fum)]\cdot 6.5H_2O\}_n$ [9], and $\{[Cu_3(\mu_3\text{-}OH)(2\text{-}Pao)_3(bdc)]\cdot 6H_2O\}_n$ [10] form coordination polymers in which each copper centre is interconnected through inorganic or organic anions acting as bridging ligands, while the pyridine-*n*-oxime ligands coordinate in bi- or tridentate modes to generate metallocyclic units. However, other coordination modes and structural forms involving this ligand have also been reported in the CSD [11-14].

The aim of this study was to synthesize a Cu(II) compound containing the PaoH ligand and bipyridine, since bridging ligands of the bipyridine family are frequently involved in the formation of coordination polymers with various dimensionalities, and to determine and analyse its molecular and crystal structures. The Cu(II) ion was selected because of its d^9 electronic configuration, which favours square-planar or square-pyramidal geometries, as well as its pronounced structural diversity arising from the Jahn–Teller effect.

The purpose of synthesising this compound was to obtain coordination polymer based on rigid ligands, which create favourable conditions for producing porous shells with adsorbing properties. Due to its chelating coordination with the metal ion, PaoH forms a stable fragment, and the specific geometry of the PaoH molecule usually generates complexes in which these ligands coordinate in a *cis*-position relative to each other. This allows the development of molecular designs with coordinating ligands in the free positions of the central ion to be diversified. In this paper, was reported a novel compound with the formula $\{[Cu_4(Pao)_2(L)_2(bpy)_2](NO_3)_2\cdot 2dmf\cdot 4H_2O\}_n$, obtained from the reaction of copper(II) nitrate, 4,4′-bipyridine (bpy) and *syn*-2-pyridinealdoxime (PaoH). The $(L)^{2-}$ is generated through the *in situ* chemical transformation of the PaoH ligand. This is the first reported example in which two distinct forms of the ligand PaoH are stabilized within the same compound [1]. Consequently, the formation of coordinate bonds between the metal ion and the donor atoms of these ligands results in new properties that differ from those of the uncoordinated ligand. The peculiarities of the obtained structure can open up new perspectives regarding the electron donor atom competition capacity, coordination polyhedron specificity, and the stability of the formed compounds.

Experimental

All reagents and solvents were obtained from commercial sources (Sigma-Aldrich) and were used without further purification.

Synthesis of $\{[Cu_4(Pao)_2(L)_2(bpy)_2](NO_3)_2\cdot 2dmf\cdot 4H_2O\}_n$ (1)

A quantity of $Cu(NO_3)_2\cdot 3H_2O$ (0.024 g, 1 mmol), *syn*-2-pyridinealdoxime (0.024 g, 1 mmol), and 4,4′-bipyridine (0.018 g, 1 mmol) were dissolved in a mixture of 2 mL of water, 4 mL of methanol 0.15 mL and *N,N*-dimethylformamide. After stirring at room temperature (25°C), the resulting blue solution was filtered and placed in a covered container. Slow evaporation of this solution led to the formation of blue, single crystals, which were separated by filtration and dried at room temperature. Yield: ~42%. Anal. calc. for $C_{50}H_{56}Cu_4N_{16}O_{18}$ %: C 42.19; H 3.97; N 15.75, Found, %: C 41.87; H 3.83; N 15.62. In the IR spectrum of compound **1** the bands $\nu(C{=}N)_{oxime}$= 1652 cm^{-1}, $\nu(N\text{–}O)_{oxime}$= 1060 cm^{-1} indicate the presence of the oxime group. The bands in the regions ~1608, 1411, 814 cm^{-1} can be assigned to the pyridine ring. The presence of the pyridine ring in both pyridine-oxime and bipyridine ligands complicates the possibility of distinctly and precisely identifying the belonging of its characteristic bands. The compound is stable in air, readily soluble in ethanol, dimethylformamide, dimethylsulfoxide, water, methanol, and insoluble in acetone, acetonitrile.

X-ray structure determination

The experimental data for **1** were obtained at room temperature using an Xcalibur E diffractometer with a graphite monochromator and Mo-K*α* radiation. The unit cell parameters were determined and the experimental data were processed using the CrysAlis program from Oxford Diffraction Ltd. [15]. The compounds structures were determined by direct methods and refined using the anisotropic full-matrix least-squares method for non-hydrogen atoms in the SHELX software packages [16,17]. The positions of the hydrogen atoms were obtained from difference Fourier syntheses or were calculated geometrically and all were refined isotropically in the rigid body model. Full crystallographic details are given in Table 1. Crystallographic data for new structures reported herein were deposited with the Cambridge Crystallographic Data Centre and allocated the deposition numbers CCDC 2526943.These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

Table 1

Crystallographic data and structure refinement details for compound 1.

Parameters	*Value*
Empirical formula	$C_{50}H_{56}Cu_4N_{16}O_{18}$
Formula weight	1423.26
Crystal system	triclinic
Space group	$P\bar{1}$
Z	1
a(Å)	8.3476(4)
b(Å)	12.5828(6)
c(Å)	14.4414(10)
α(deg)	96.868(5)
β(deg)	103.836(5)
γ(deg)	97.502(4)
V (Å^3)	1442.23(15)
D_c (g/cm $^{-3}$)	1.639
Crystal size (mm^3)	0.15 x 0.10 x 0.03
Reflections collected/unique	8616/5086 [R(int)= 0.0415]
Parameters	399
GOF on F^2	1.005
R_1, wR_2 [I>2σ(I)]	0.0594, 0.1246
R_1, wR_2 (all data)	0.1008, 0.1430

Results and discussion

Crystal structure analysis

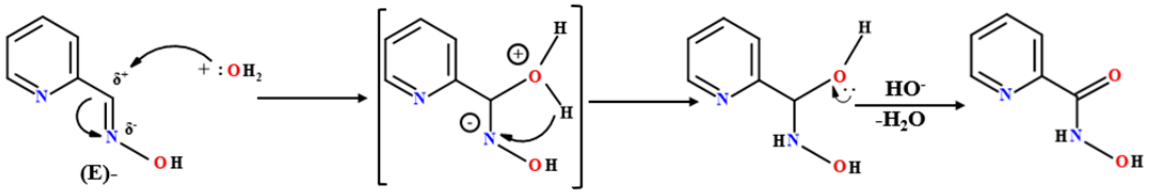
The definitive structure of new compound of Cu(II) **1** was determined using single crystal X-ray diffraction. This compound crystallizes in the triclinic space group $P\bar{1}$ (Table 1). In the asymmetric unit of the unit cell, two metal atoms located in general positions are identified, together with deprotonated (Pao)$^-$, one ligand derived from PaoH and one bridging bpy ligand. The outer coordination sphere is constituted by one nitrate anion $(NO_3)^-$ and one dmf molecule, in addition to two crystallisation water molecules. Therefore, an ionic compound $\{[Cu_4(Pao)_2(L)_2(bpy)_2](NO_3)_2\cdot 2dmf\cdot 4H_2O\}_n$ **(1)** was formed in the crystal, in which the cation is a one-dimensional coordination polymer $[Cu_4(Pao)_2(L)_2(bpy)_2]^{2+}{}_n$. Structural studies have demonstrated that proligands PaoH and derived from PaoH – H_2L, which are involved in its deprotonated forms in the formation of the centrosymmetric core $[Cu_4(Pao)_2(L)_2]^{2+}$ of the coordination polymer. This H_2L ligand, underwent *in situ* chemical transformations as a result of the reaction (Scheme 1).

The H_2L ligand can also be formed in the autoxidation process with the formation of C=O and the elimination of hydrogen atoms. This modification is possible due to the nitroso Diels-Alder reaction, which occurs *via* a concerted [4+2] cycloaddition mechanism. This reaction, by oxidizing oximes, establishes a direct bond between the oxime and the nitroso groups [12].

A CSD study [1] revealed an example of a Na(I) dimer in which two H_2L ligands are monodentately coordinated *via* a carbonyl oxygen atom to each metal atom. Two similar, monodeprotonated ligands are bidentately coordinated *via* donor O,O atoms to one metal atom and one of the O atoms bridges to the second metal atom [13]. In this case, the nitroso-Diels–Alder reaction was studied systematically with a focus on the transformation of the H_2L ligand. This occurred following an *in situ* PaoH oxidation reaction influenced by anion $(NO_3)^-$ in a water and methanol environment, with the Cu(II) atom acting as a catalyst. The trinuclear molecular complex of Cu(II) with H_2L with the formula $[Cu_3(L)_2(py)_5(NO_3)_2]$ was obtained by recrystallization of $[Cu_5(L)_4](NO_3)_2$. In this complex, two doubly deprotonated $(L)^{2-}$ are coordinated as tetradentate ligands in a bidentate-chelate manner through *N,N* atoms to one metal atom and through *O,O* to the other [14].

Analysis of the crystal structure of **1** revealed that both crystallographically independent copper atoms Cu(1) and Cu(2) into an infinite linear cationic chain adopt a square-pyramidal coordination geometry, defined by a set of N_3O_2 donor atoms. These atoms originate from different ligands: (Pao)$^-$, (L)$^{2-}$ and bpy (Figure 1*(a)*). In this centrosymmetric core, (Pao)$^-$ is coordinated as a bidentate-chelate ligand to one metal atom and monodentate to the other, through the N(3),N(4) donor atoms to Cu(2), and through the O(3) to Cu(1) atom.

$$Cu(NO_3)_2\cdot 3H_2O + PaoH + bpy \xrightarrow[MeOH]{H_2O,\ dmf} \{[Cu_4(Pao)_2(L)_2(bpy)_2](NO_3)_2\cdot 2dmf\cdot 4H_2O\}_n \quad \mathbf{1D}$$



Scheme 1. Synthesis reaction and formation mechanism of the H_2L ligand.

At the same time, the pentadentate ligand $(L)^{2-}$ is coordinated differently, linking three metal atoms – through N(1),N(2) to Cu(1), through O(1),O(2) to Cu(2)* and through O(2) to Cu(1)*. The coordination polyhedron of the metal atoms is completed by the N(5) and N(6) nitrogen atoms by bpy ligands that are coordinated in an *exo*-bidentate manner to two metal atoms.

The Cu(1)–N and Cu(2)–N distances are in the interval 1.957(5) – 2.292(4) Å; the Cu(1)–O and Cu(2)–O distances are in the interval 1.913(3) – 1.951(4) Å (Table 2).

Four bridging bpy ligands link the centrosymmetric $[Cu_4(Pao)_2(L)_2]$ core of the coordination polymers into a structure that extends into a one-dimensional ribbon with 24-atom of metalomacrocycles (Figure 1*(b)*). The Cu···Cu distance in the polymer chain arranged along *a* axis is 11.650 Å.

The anions $(NO_3)^-$, crystallization molecules of water and dmf are located in the crystal cavities between the cationic polymers. These are stabilised by C–H ···O, O(W)–H ···O(5) and O(W)–H ···O(W) hydrogen bonds (Table 3).

Table 2

Bond lengths (Å) and Angles (°) in coordination Cu(II) environment in 1.

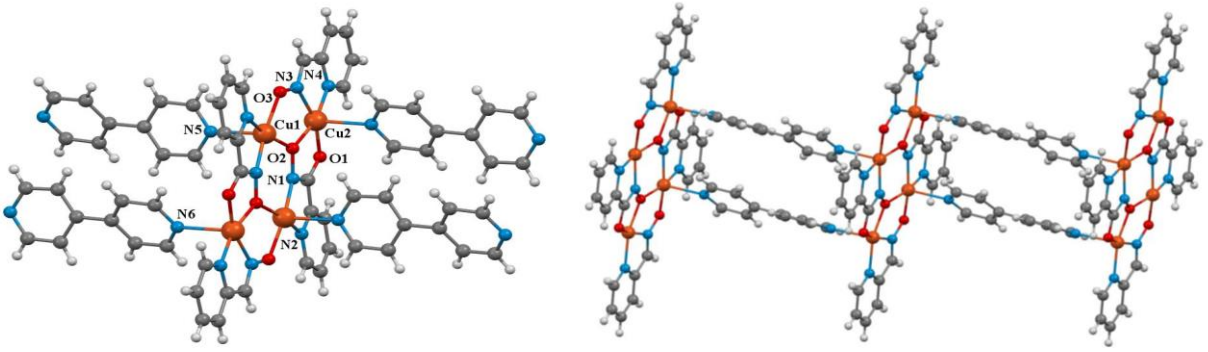
Bonds	*d, Å*	*Bonds*	*d, Å*
Cu(1)–O(2)	1.929(3)	Cu(2)–O(1)	1.937(4)
Cu(1)–O(3)	1.951(4)	Cu(2)–O(2)	1.913(3)
Cu(1)–N(1)$^{\#1}$	1.966(4)	Cu(2)–N(3)	1.957(5)
Cu(1)–N(2)$^{\#1}$	2.018(4)	Cu(2)–N(4)	1.975(4)
Cu(1)–N(5)	2.278(4)	Cu(2)–N(6)$^{\#2}$	2.292(4)
Angles	*ω, °*	*Angles*	*ω, °*
O(2)Cu(1)O(3)	88.87(15)	O(1)Cu(2)O(2)	83.88(15)
O(2)Cu(1)N(1)$^{\#1}$	99.64(15)	O(1)Cu(2)N(3)	163.49(16)
O(2)Cu(1)N(2)$^{\#1}$	165.86(16)	O(1)Cu(2)N(4)	104.65(18)
O(2)Cu(1)N(5)	98.61(15)	O(1)Cu(2)N(6)$^{\#2}$	95.91(17)
O(3)Cu(1)N(1)$^{\#1}$	98.61(15)	O(2)Cu(2)N(3)	86.71(17)
O(3)Cu(1)N(2)$^{\#1}$	87.82(16)	O(2)Cu(2)N(4)	165.11(17)
O(3)Cu(1)N(5)	97.37(16)	O(2)Cu(2)N(6)$^{\#2}$	96.90(15)
N(1)$^{\#1}$Cu(1)N(2)$^{\#1}$	80.32(18)	N(3)Cu(2)N(4)	81.97(19)
N(1)$^{\#1}$Cu(1)N(5)	96.00(17)	N(3)Cu(2)N(6)$^{\#2}$	98.68(18)
N(2)$^{\#1}$Cu(1)N(5)	95.45(16)	N(4)Cu(2)N(6)$^{\#2}$	94.35(17)

$^{\#1}$ *x, -y+2, -z+1/2* $^{\#2}$ *x, y+1, z*

Table 3

Hydrogen bond distances (Å) and angles (°) in 1.

D–H···A	*d(H···A)*	*d(D···A)*	*∠(DHA)*	*Symmetry transformations for acceptor A*
C(3)–H(3) ···O(3)	2.42	2.918(6)	113	*-x,-y+2,-z+1*
C(4)–H(4)···O(4)	2.59	3.386(9)	144	*x,y+1,z+1*
C(16)–H(16)···O(7)	2.50	3.403(9)	164	*-x+1,-y+1,-z+1*
C(22)–H(22)···O(7)	2.63	3.491(9)	155	*-x+1,-y+1,-z+1*
O(1W)–H(1W1)···O(5)	2.07	2.875(9)	177	*x-1,y,z+1*
O(1W)–H(2W1)···O(2W)	2.00	2.884(9)	174	*x,y,z+1*
O(2W)–H(1W2)···O(6)	2.09	3.037(10)	176	*x,y,z*
O(2W)–H(2W2)···O(1W)	2.10	2.936(9)	179	*-x+1,-y+1,-z+1*



(a) ***(b)***

Figure 1. The centrosymmetric core of the coordination polymer cation of 1 *(a)*; Fragment of 1D chain of $[Cu_4(Pao)_2(L)_2(bpy)_2]^{2+}{}_n$ *(b)*.

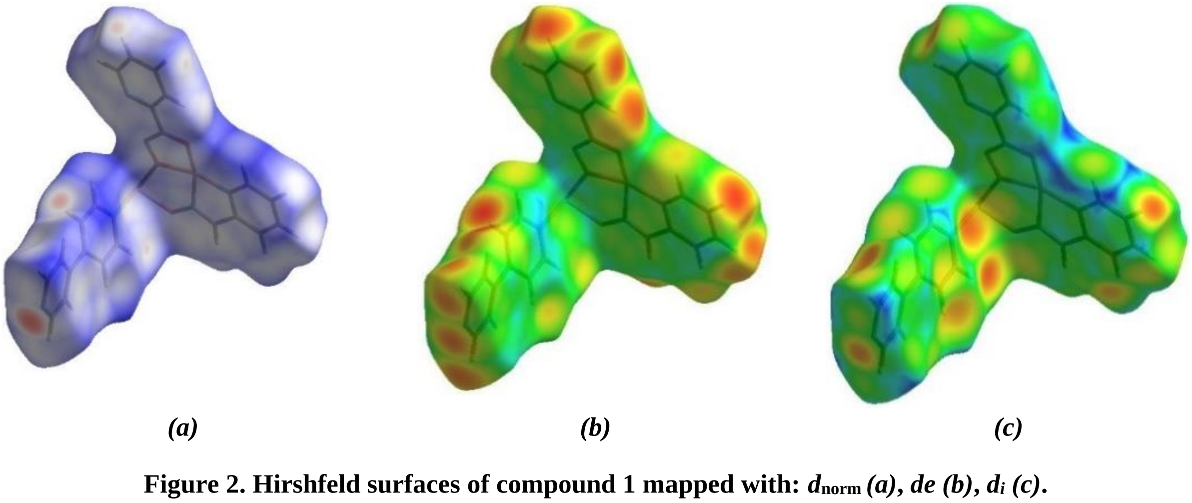
Hirshfeld Surface (HS) analysis

The HS analysis performed aims to investigate the contributions of different intermolecular contacts to the crystal packing of the components of compound $\{[Cu_4(Pao)_2(L)_2(bpy)_2](NO_3)_2\cdot 2dmf\cdot 4H_2O\}_n$ (**1**). This study focused on analysing sets of hydrogen bond pattern graphs to identify multiple intermolecular contacts that could easily be highlighted. The HS analysis was performed using the CrystalExplorer 17.5 program [18,19], which facilitates the definition of different regions surrounding the molecule in the crystal, thereby enabling the integration of the electronic distribution.

A detailed study of the intermolecular interactions present within the crystal structure of **1** was conducted, using the generation of two-dimensional footprint diagrams. These diagrams were then mapped onto the d_{norm} parameter, with the objective of elucidating variations in contact distances between molecules. The HS was also analysed using the normalised contact distance (d_{norm}), which is the sum of the external (d_e) and internal (d_i) distances reported against the van der Waals radii of the atoms in contact with the surface. This method allowed for a clear differentiation between the types of intermolecular interaction present in the crystal. Regions coloured red highlight close contacts (distances smaller than the sum of the *van der Waals*), while blue regions indicate electrostatic complementary areas. Green regions correspond to weak van der Waals-type interactions, where the contact distances are close to the van der Waals values (Figure 2). The presence of extended red spots near the carboxyl groups, pyridine nitrogen atoms, and coordinated water molecules confirms the potential for donor–acceptor bond formation, as highlighted by the mapping of the d_{norm} parameter. Hirshfeld surfaces for compound **1** were generated using standard high-resolution methods, and three-dimensional representations were displayed using a fixed colour scale ranging from –1.9220 to +2.3932.

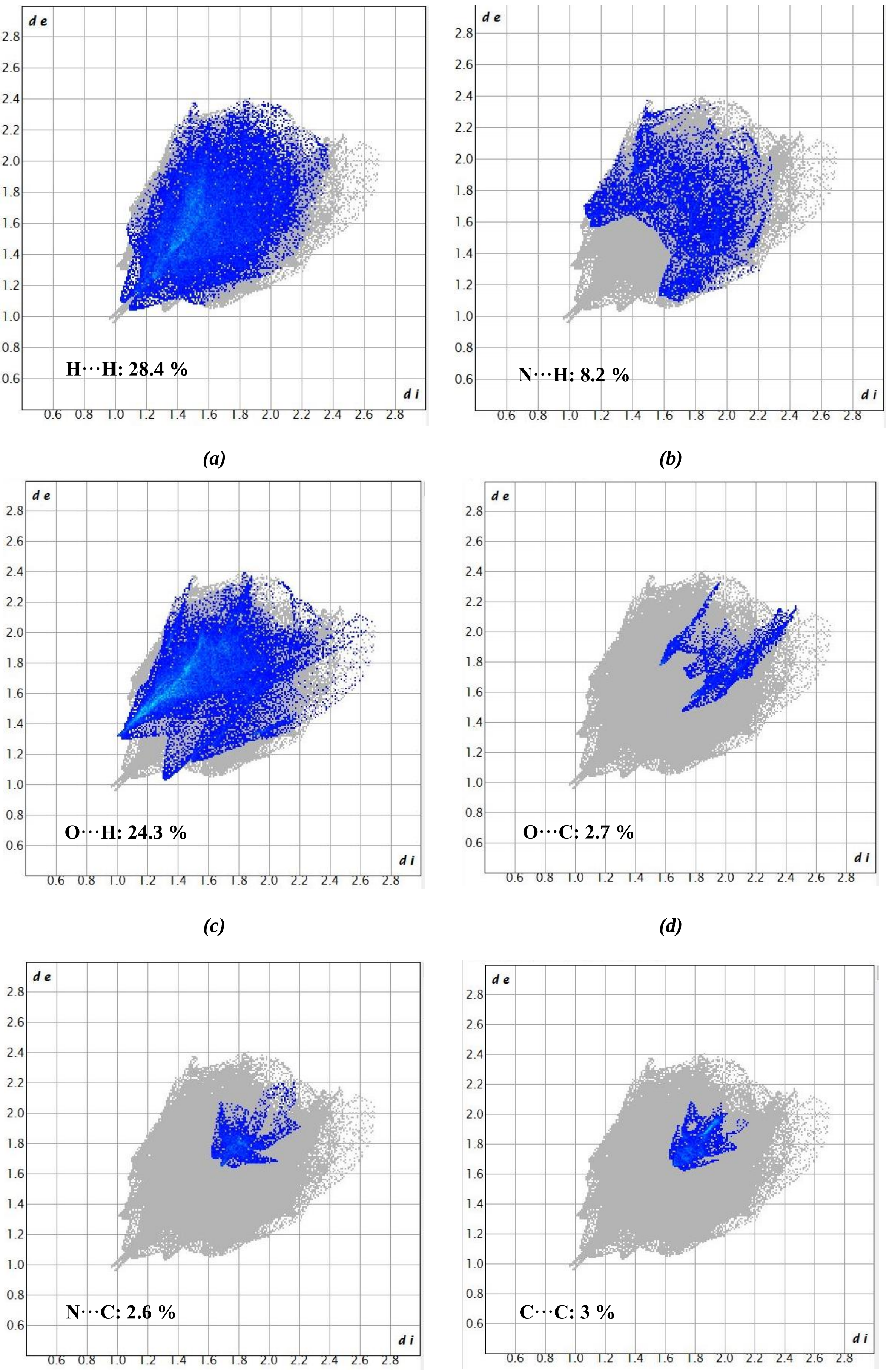
Two-dimensional fingerprint plots obtained and constructed as a function of the d_e and d_i distances allowed to estimate the percentage contribution of different interaction types (H···H, H···O/O···H, H···N/N···H etc.) to the total Hirshfeld surface area, providing an in-depth view of the molecular packing pattern in the crystal.

A summary of the Hirshfeld surface analysis of compound **1** shows that the dominant interactions are H···H (28.4%) and O···H/H···O (24.3%) types. These are highlighted in the two-dimensional footprint diagrams. The H-H contacts, which are characteristic of van der Waals interactions, appear as broad regions in the centre of the diagrams. In contrast, the O···H/H···O contacts, which correspond to hydrogen bonds, are well defined, which highlights their important role in stabilising the crystal structure. There are also less frequent, but significant, contacts that contribute to supramolecular organisation, such as C···C (3%) and C···N/N···C (2.6%). These contacts extend molecular fragments into supramolecular layers. The 2D footprint diagrams (Table 3) resulting from this analysis can be used to identify and quantify patterns associated with specific interactions (H···H, N···H, C···O, O···H *etc.*) in the crystal structure of the new compound.



(a) ***(b)*** ***(c)***

Figure 2. Hirshfeld surfaces of compound 1 mapped with: d_{norm} *(a)*, *de (b)*, d_i *(c)*.



(a) *(b)*

(c) *(d)*

(e) *(f)*

Figure 3. Two-dimensional footprint graphs showing the relative contributions of all contacts in 1.

Conclusions

The present study is devoted to the copper(II) complex with *syn*-2-pyridinealdoxime (PaoH), which also contains a bridging ligand 4,4′-bipyridine (bpy). The crystal structure of the compound was determined by single crystal X-ray diffraction. The obtained compound is ionic with the formula $\{[Cu_4(Pao)_2(L)_2(bpy)_2](NO_3)_2{\cdot}2dmf{\cdot}4H_2O\}_n$ (**1**) in which the cation is a symmetric 1D coordination polymer. For the first time, two distinct forms, besides $(Pao)^-$ it also contains a derivative of this ligand $(L)^{2-}$, which are two different forms of PaoH obtained by an *in situ* chemical transformation and involved in the formation of the coordination polymer, have been stabilized within the same compound. The components of the outer sphere of the crystal are linked to the polymer complex cations and to each other by hydrogen bonds. The intra- and intermolecular interactions present in compound were identified and quantified, and their 2D footprint diagrams were illustrated using HS analysis.

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