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Crystal structures of *bis*-{*N*-[1-(pyridin-2-yl- κ N)ethylidene]nicotine hydrazide- κ^2 *N'*,*O*}cobalt(II)*bis*(perchlorate) dihydrate and *bis*-{*N'*-[1-(pyridin-2-yl- κ N)ethylidene]nicotinohydrazide- κ^2 *N'*,*O*}copper(II) perchlorate

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ABSTRACT



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Complexes of Co(II), [Co(C₂₆H₂₄N₈O₂)]·(ClO₄)₂·(H₂O)₂ (1), and Cu(II), [Cu(C₂₆H₂₃N₈O₂)]·(ClO₄) (2), have been synthesized. The prepared two compounds were characterized by elemental analysis, infrared and their structures were determined by single-crystal X-ray diffraction. The compound 1 crystallizes in the triclinic space group *P*-1 with the following unit cell parameters: *a* = 8.880 (5) Å, *b* = 10.529 (5) Å, *c* = 18.430 (5) Å, α = 99.407 (5)°, β = 102.174 (5)°, γ = 100.652 (5)°, *V* = 1618.2 (13) Å³, *Z* = 2, *T* = 293(2), μ (MoK α) = 0.77 mm⁻¹, *D*_{calc} = 1.582 g/cm³, 16135 reflections measured (5.050° ≤ 2 θ ≤ 59.152°), 7648 unique, *R*_{int} = 0.034 which were used in all calculations. The final *R*₁ was 0.066 (*I* ≥ 2 σ (*I*)) and *wR*₂ was 0.22 (all data). The compound 2 crystallizes in the monoclinic space group *P*2₁/*c* with the following unit cell parameters: *a* = 11.652 (5) Å, *b* = 16.540 (5) Å, *c* = 14.512 (5) Å, β = 93.495 (5)°, *V* = 2791.6 (18) Å³, *Z* = 4, *T* = 293(2), μ (MoK α) = 1.05 mm⁻¹, *D*_{calc} = 1.768 g/cm³, 15592 reflections measured (5.624° ≤ 2 θ ≤ 58.804°), 6630 unique, *R*_{int} = 0.025 which were used in all calculations. The final *R*₁ was 0.050 (*I* ≥ 2 σ (*I*)) and *wR*₂ was 0.144 (all data). In both complexes, the ligand acts in a tridentate fashion. In the structure of the mononuclear complex 1, the Co(II) cation is coordinated by two ligand molecules. The basal plane around the Co(II) cation is occupied by two pyridine nitrogen atoms and two carbonyl oxygen atoms. Two imino nitrogen atoms occupy the apical positions of the distorted square-pyramidal geometry. The mononuclear 2 consists of a Cu(II) coordinated by one ligand and one monodeprotonated ligand molecule. The metal center lies in a distorted square bipyramidal environment. The basal plane around the Cu(II) is occupied by two pyridine nitrogen atoms and two carbonyl oxygen atoms, the apical position being occupied by the two imino nitrogen atoms.

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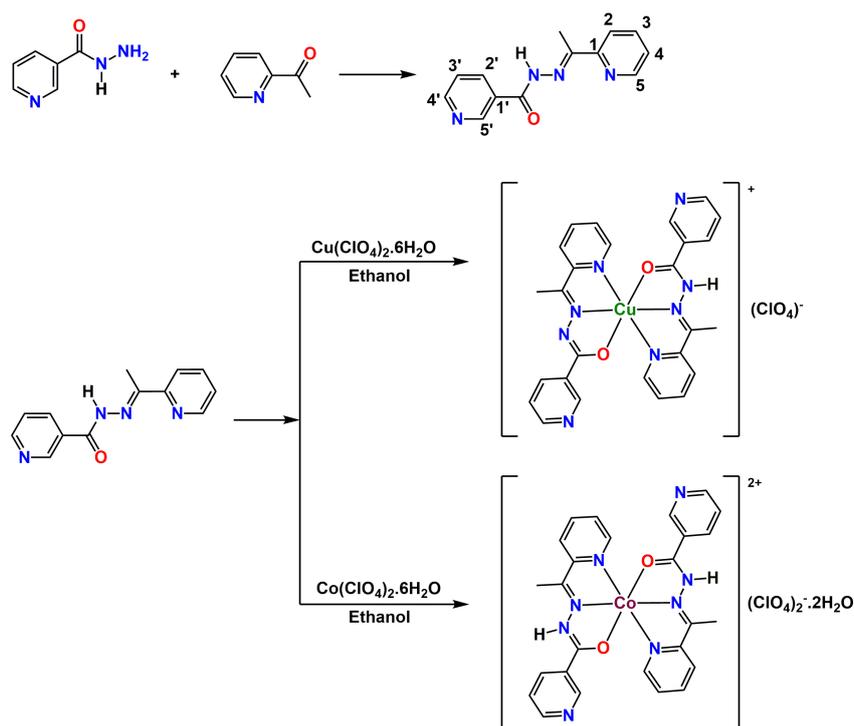
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1. Introduction

The development of new molecules with drug activity and without dangerous side effects is one of the major challenges of science. The compounds having a hydrazone unit -C=N-NH-R are characterized by their use in the synthesis of compounds having therapeutic properties [1-4]. Compounds with antibacterial [5,6], antifungal [7], antiviral [8], and anticancer [9] properties are listed. These compounds having donor sites such as N, O or S atoms are widely used in coordination chemistry. Complexes with diverse structures have been synthesized in recent years [10,11].

These hydrazone compounds have the ability to exhibit keto-enol tautomeric forms which can coordinate in a tridentate fashion. These tautomeric forms offer these molecules the possibility of coordinating in the neutral mode [12], in monoanionic mode [13], or dianionic mode [10,14]. These

complexes often exhibit important properties for industries such as magnetism [15], luminescence [16], catalysis [17], and optics [18]. These metal transition coordination compounds obtained with hydrazone ligands are also studied because of their broad profile in the pharmacological field, with antitumor [19], antimicrobial [20,21] and anti-tuberculosis [22] potential. It has been shown that hydrazone ligands with biological properties can see their activity increased after complexation with metal ions. Cindric *et al.* demonstrated that free benzo-hydrazone having cytotoxic activity against a THP-1 and HepG2 cell line saw its activity significantly increased after complexation with copper [23]. It is in this context that we have prepared transition metal and lanthanide complexes from ligands having hydrazine units [24-29].



Scheme 1. Synthetic scheme for HL and complex preparation.

In this work, we describe the synthesis and characterization of two complexes of cobalt(II) and copper (II) ions obtained from the *N'*-(1-(pyridin-2-yl)ethylidene)nicotinohydrazide (HL) ligand, which are characterized by elemental analysis, FT-IR, ^1H NMR, and ^{13}C NMR. The crystal structures of these two complexes have been elucidated by X-ray diffraction studies.

2. Experimental

2.1. Materials and instrumentation

Nicotinic hydrazide, 2-acetyl pyridine, cobalt perchlorate hexahydrate, and copper perchlorate hexahydrate were purchased from Sigma-Aldrich and used as received without further purification. All solvents used were of reagent grade. The ligand, *N'*-(1-(pyridin-2-yl)ethylidene)nicotinohydrazide (HL) was synthesized following the reported procedure [30].

Melting points were determined on a Büchi 570 melting-point apparatus and were uncorrected. Elemental analyses of C, H and N were recorded on a VxRio EL Instrument. Infrared spectra were obtained on an FTIR Spectrum Two of Perkin Elmer spectrometer in the 4000-400 cm^{-1} region. The molar conductance of 1×10^{-3} M in DMSO solution 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: $\text{Hg}[\text{Co}(\text{SCN})_4]$).

2.2. Synthesis and characterization of *N'*-(1-(pyridin-2-yl)ethylidene)nicotinohydrazide (HL)

The ligand HL was synthesized following the procedure reported in the literature [30]. To a solution of 2-acetyl pyridine (0.6627 g, 5.5 mmol) in 10 mL of methanol was added a solution of nicotinic hydrazide (0.7566 g, 5.5 mmol) in 30 mL of methanol. The mixture was heated under reflux for two hours. The solvent was evaporated to dryness and the solid was

collected and recrystallized from ethanol solution. After one week, the white powder was collected (Scheme 1).

N'-(1-(pyridin-2-yl)ethylidene)nicotinohydrazide (HL): Color: White. M.p.: 171-172 °C. Yield: 66 %. FT-IR (ATR, ν , cm^{-1}): $\nu(\text{NH})$ 3201, $\nu(\text{C}=\text{O})$ 1663, $\nu(\text{C}=\text{N})$ 1622, 1581, $\nu(\text{N}-\text{N})$ 1150. ^1H NMR (500 MHz, $\text{DMSO}-d_6$, δ , ppm): 2.52 (s, 3H, CH_3), 7.50-9.07 (m, 8H, Ar-H), 11.29 (s, 1H, NH). ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$, δ , ppm): 13.52 (CH_3), 120.92 (H-C2), 120.96 (H-C4'), 123.89 (H-C4), 123.92 (C1'), 130.20 (H-C3), 136.36 (H-C5'), 137.16 (H-C3'), 149.18 (H-C5), 152.63 (H-C2'), 152.84 (C1), 155.48 (C=N), 163.37 (C=O). Anal. calcd. for $\text{C}_{13}\text{H}_{12}\text{ON}_4$: C, 64.99; H, 5.03; N, 23.32. Found: C, 64.96; H, 5.01; N, 23.30%.

2.3. Synthesis and characterization of complexes 1 and 2

To a solution of 2-acetyl pyridine (0.6627 g, 5.5 mmol) in 10 mL of ethanol was added a solution of nicotinic hydrazide (0.7566 g, 5.5 mmol) in 30 mL of ethanol. The mixture was heated under reflux for two hours. After cooling, the volume is completed to 50 mL. To 25 mL of the above solution was added a solution of $\text{Co}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ (0.0915 g, 2.5 mmol) or $\text{Cu}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ (0.0926 g, 2.5 mmol) in 5 mL of methanol. The mixture was stirred at room temperature for two hours. The precipitate was discarded, and the filtrates were left for slow evaporation. On standing for five days, crystals suitable for X-ray single crystal diffraction analysis were formed, brown prisms of complex 1 and light-yellow prisms of complex 2 were collected (Scheme 1).

Bis-{*N'*-[1-(pyridin-2-yl- κN)ethylidene] nicotinohydrazide $\kappa^2\text{N',O}$ }cobalt(II)bis(perchlorate) dihydrate (1): [$\text{Co}(\text{HL})_2$](ClO_4) $_2 \cdot 2\text{H}_2\text{O}$ (1). Color: brown. Yield: 53 %. FT-IR (ν , cm^{-1}): 3100, 1602, 1583, 1502, 1462, 1409, 1373, 1161, 1070, 1028, 912, 823, 780, 620. μ_{eff} (μB): 4.17. Λ_{M} ($\text{S}\cdot\text{cm}^2\cdot\text{mol}^{-1}$): 154.4. Anal. calcd. for $\text{CoC}_{26}\text{H}_{28}\text{N}_8\text{Cl}_2\text{O}_{12}$: C, 40.33; H, 3.64; N, 14.47. Found: C, 40.31; H, 3.62; N, 14.44%.

Bis-{*N'*-[1-(pyridin-2-yl- κN)ethylidene] nicotinohydrazide- $\kappa^2\text{N',O}$ }copper(II) perchlorate (2): [$\text{Cu}(\text{L})(\text{HL})$](ClO_4) (2). Color: Yellow-light. Yield: 59 %.

Table 1. Crystal data, X-ray data collection, data reduction and structure refinement for complexes **1** and **2**.

Parameters	Complex 1	Complex 2
Formula	C ₂₆ H ₂₈ Cl ₂ CoN ₈ O ₁₂	C ₂₆ H ₂₃ ClCuN ₈ O ₆
FW	774.39	742.97
Crystal shape / color	Block / brown	Block / yellow-light
Crystal size (mm)	0.10 × 0.10 × 0.05	0.30 × 0.10 × 0.09
Crystal system	Triclinic	Monoclinic
Space group	<i>P</i> -1	<i>P</i> 2 ₁ / <i>c</i>
<i>a</i> (Å)	8.880 (5)	11.652 (5)
<i>b</i> (Å)	10.529 (5)	16.540 (5)
<i>c</i> (Å)	18.430 (5)	14.512 (5)
α (°)	99.407 (5)	90.000
β (°)	102.174 (5)	93.495 (5)
γ (°)	100.652 (5)	90.000
<i>V</i> (Å ³)	1618.2 (13)	2791.6 (18)
<i>Z</i>	2	4
<i>D</i> _{calc} (g cm ⁻³)	1.582	1.768
λ (MoKα)	0.71073	0.71073
T (K)	293(2)	293(2)
μ (mm ⁻¹)	0.77	1.05
Index ranges	-11 ≤ <i>h</i> ≤ 11; -14 ≤ <i>k</i> ≤ 14; -24 ≤ <i>l</i> ≤ 22	-15 ≤ <i>h</i> ≤ 12; -22 ≤ <i>k</i> ≤ 18; -16 ≤ <i>l</i> ≤ 19
<i>F</i> (000)	730	1516
θ range (°)	2.525-29.576	2.812-29.402
No. of measured reflections	16135	15592
No. of Independent reflections	7648	6630
No. of observed [<i>I</i> > 2σ(<i>I</i>)] reflections	5762	4967
<i>R</i> _{int}	0.034	0.025
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)]	0.066	0.050
<i>wR</i> (<i>F</i> ²)	0.22	0.144
Goodness-of-fit (Gof) on <i>F</i> ²	1.08	1.02
No. of parameters	509	381
No. of restrains	0	0
Δρ _{max} , Δρ _{min} (e.Å ⁻³)	0.97, -0.64	0.63, -0.75

FT-IR (ν, cm⁻¹): 3092, 1612, 1588, 1504, 1463, 1380, 1113, 1097, 1041, 920, 838, 788, 616. μ_{eff} (μ_B): 1.77. Λ_M (S.cm².mol⁻¹): 75.1. Anal. calcd. for CuC₂₆H₂₃N₈ClO₆: C, 48.26; H, 3.61; N, 17.44. Found: C, 48.24; H, 3.59; N, 17.41%.

2.4. X-ray data collection, structure determination, and refinement

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 MoKα radiation (λ = 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 [31]. Structure solution and refinement were performed using SHELXT [32] and SHELXL-2018/3 [33]. All hydrogen atoms were added in calculated positions and refined in riding mode on the parent atom. Molecular graphics were generated using ORTEP-3 [34].

3. Results and discussion

3.1. Synthesis

The IR spectrum of the ligand shows main bands at 3194, 1656, and 1605 cm⁻¹, attributable, respectively, to ν(N-H), ν(C=O) and ν(C=N) vibration mods. Additional bands due to the aromatic ring are located in the range 1428-1582 cm⁻¹. Comparison of the infrared data of the ligand and those of the complexes obtained upon coordination with cobalt or copper ions shows that no iminization undergoes during the complexation. In fact, in the spectra of the complex band due to ν(C=O) appears ca. 1602 cm⁻¹ for complex **1** and ca. 1612 cm⁻¹ for complex **2**. The shift of the ν(C=N) band from 1605 cm⁻¹ in the ligand spectrum to 1583 cm⁻¹ for complex **1** and 1588 cm⁻¹ for complex **2** is indicative of the involvement of the azomethine atoms in coordination to the Co(II) and Cu(II), respectively [35]. The broad band centered in the range 3090-3110 cm⁻¹ is probably due to the stretching of H-N [36]. The bands located at ca. 1070 cm⁻¹ (ν_{as}) and 620 cm⁻¹ (δ_{as}) on the spectrum of

complex **1** and 1097 cm⁻¹ (ν_{as}) and 616 cm⁻¹ (δ_{as}) for complex **2** are indicative of the presence of free tetrahedral perchlorate anions [37].

The molar conductivity values of the freshly DMF (1×10⁻³ M) complex solution and fifteen days later are, respectively, 154.4 (S.cm².mol⁻¹) and 158.2 (S.cm².mol⁻¹) for complex **1** and for 75.1 (S.cm².mol⁻¹) and 76.2 (S.cm².mol⁻¹) for complex **2**. These values are in accordance with those reported for 2:1 electrolyte for complex **1** and 1:1 electrolyte for complex **2** [38]. The stability of the conductivity values fifteen days later showed that the complexes are stable in DMF.

The values of the magnetic moment at room temperature for the diamagnetic complexes are indicative of the presence of one metal atom per molecule. The magnetic moment value of 4.17 μ_B complex **1** is in accordance with three unpaired electrons by *d*⁷ configuration. The magnetic moment value of 1.77 μ_B for complex **2** is in accordance with one unpaired electron in *d*⁹ configuration [39].

3.2. Single crystal structure

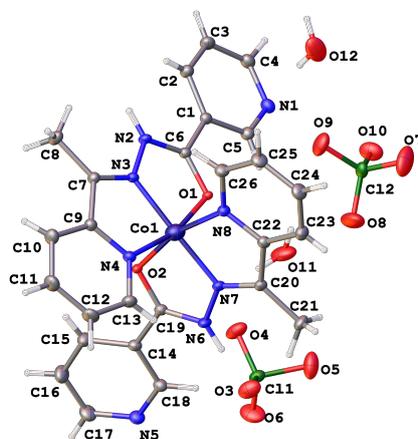
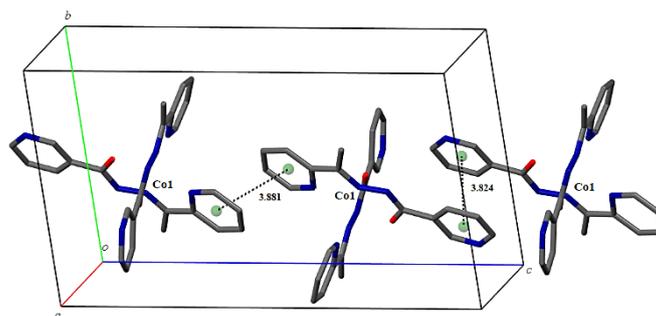
3.2.1. Complex I

In the crystal structure of the coordination compound [Co(C₂₆H₂₄N₈O₂)]·(H₂O)₂·(ClO₄)₂ (**1**), the Co(II) center is hexacoordinated by two pyridine nitrogen atoms, two imino nitrogen atoms and two carbonyl oxygen atoms of the neutral molecule ligand. The coordination environment can be best described as severely distorted square bipyramidal (Figure 1). The basal plane around the Co(II) cation is occupied by the pyridine nitrogen atoms N4 and N8 with Co-N distances of 1.920(3) and 1.915(3) Å and the carbonyl oxygen atoms O1 and O2 with Co-O distances of 1.887(3) and 1.905(3) Å (Table 2).

The bond lengths are slightly shorter than the values reported for related complexes [40]. The apical positions are occupied by the imino nitrogen atoms N3 and N7 with distances of Co-N of 1.853(3) and 1.856(3) Å. These values are shorter than the reported values for a similar complex [41].

Table 2. Selected bond lengths and bond angles for complexes 1 and 2.

Atom-Atom	Bond lengths (Å)	Atom-Atom	Bond lengths (Å)
Co1-N7	1.856 (3)	Cu1-N6	2.050 (2)
Co1-O2	1.905 (3)	Cu1-N2	2.058 (3)
Co1-O1	1.887 (3)	Cu1-O1	2.099 (2)
Co1-N3	1.853 (3)	Cu1-O2	2.130 (2)
Co1-N4	1.920 (3)	Cu1-N1	2.220 (3)
Co1-N8	1.915 (3)	Cu1-N5	2.277 (3)
Atom-Atom-Atom	Bond angles (°)	Atom-Atom-Atom	Bond angles (°)
N7-Co1-O2	82.93 (12)	N6-Cu1-N2	165.60 (10)
N7-Co1-O1	93.01 (12)	N6-Cu1-O1	113.34 (9)
N7-Co1-N4	101.36 (13)	N2-Cu1-O1	75.61 (8)
N7-Co1-N8	82.50 (13)	N6-Cu1-O2	76.15 (9)
O2-Co1-N4	87.59 (13)	N2-Cu1-O2	114.89 (9)
O2-Co1-N8	165.37 (12)	O1-Cu1-O2	97.37 (8)
O1-Co1-O2	91.33 (12)	N6-Cu1-N1	97.01 (10)
O1-Co1-N4	165.34 (12)	N2-Cu1-N1	74.57 (9)
O1-Co1-N8	90.75 (13)	O1-Cu1-N1	149.64 (9)
N3-Co1-N7	175.12 (13)	O2-Cu1-N1	90.06 (9)
N3-Co1-O2	95.12 (12)	N6-Cu1-N5	73.66 (9)
N3-Co1-O1	82.56 (11)	N2-Cu1-N5	95.25 (9)
N3-Co1-N4	82.98 (13)	O1-Cu1-N5	92.56 (8)
N3-Co1-N8	99.51 (13)	O2-Cu1-N5	149.74 (8)
N8-Co1-N4	93.98 (14)	N1-Cu1-N5	95.66 (9)

**Figure 1.** The crystal structure of complex 1. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as a small sphere.**Figure 2.** Intermolecular π - π stacking between the pyridyl rings in the title complex 1.

Upon coordination, each of the two ligand molecules, which act as a tridentate, formed two five-membered rings with the Co (II) center: N-C-C-N-Co and O-C-N-N-Co. For each ligand, the two membered rings share one nitrogen atom N3 and N7, respectively. The angles imposed by the five-membered rings are severely deviated from the ideal angles of 90° for a regular square bipyramid ($82.56(11)$ - $101.36(12)^\circ$). These angles are smaller than those reported by Cocu *et al.* [41]. In the basal plane, the *transoid* angles O2-Co1-N8 and O1-Co1-N4 are, respectively, $165.37(12)^\circ$ and $165.34(13)^\circ$ while the *cisoid* angles are in the range [$87.59(13)$ - $93.98(14)^\circ$]. The sum of the angles subtended by the donor atoms of Co(II) in the equatorial plane [O1-O2-N4-N8] is 363.65° , indicating approximate

coplanarity for these atoms as shown by the rms value of 0.0422 Å. The angle value of $175.12(13)^\circ$ is defined by the atoms in apical position: N3-Co1-N7. A geometrical analysis has been performed on π - π stacking in the cobalt complex. Considering the crystal structure of the cobalt complex as shown in Figure 2, there are two types of π - π stacking interactions between the 2-substituted pyridyl rings (N4-C9-C13 and N8-C22-C26) and those between the 3-substituted pyridyl rings (N1-C1-C5 and N5-C14-C18) with $Cg \cdots Cg$ distances of 3.881 and 3.824 Å, respectively. The π - π stacking interactions link the molecules into a ribbon along the *c*-axis, Figure 2.

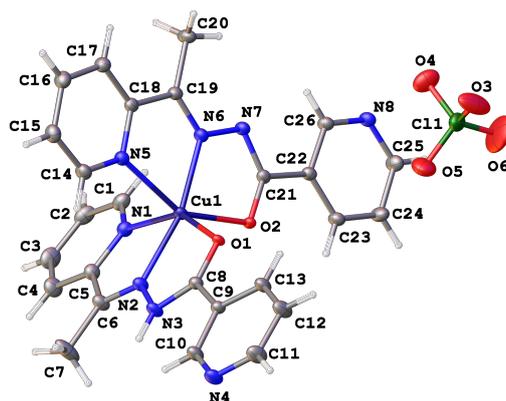


Figure 3. The crystal structure of complex 2. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as a small sphere.

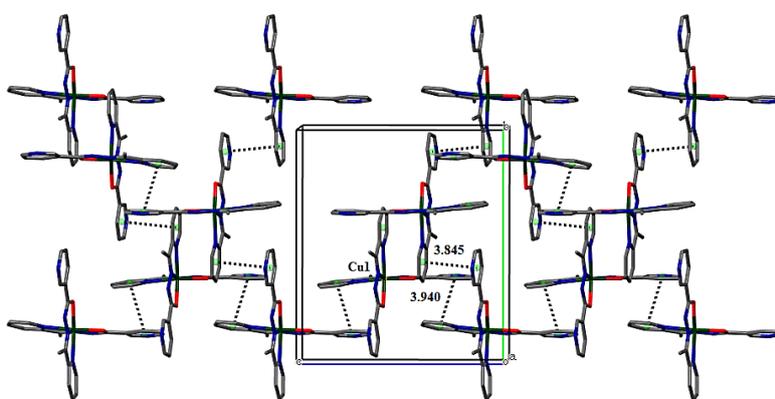


Figure 4. Intermolecular π - π stacking between the pyridyl rings in the title complex 2.

3.2.2. Complex 2

In complex **2**, the Cu (II) lies in an octahedral environment (Figure 3). One neutral ligand molecule coordinates the Cu (II) center through its pyridine nitrogen atom, its imine nitrogen atom, and its carbonyl oxygen atom. Additionally, a monodeprotonated ligand molecule coordinates the Cu (II) center through its pyridine nitrogen atom, its imine nitrogen atom and its iminolate oxygen atom. In each case two five-membered rings N-C-C-N-Cu and O-C-N-N-Cu are formed. The result is a severely distorted square bipyramid environment as shown by the *transoid* angles (149.64(9) and 165.60(10)°), the *cisoid* angles (90.06(9)-97.37(8)°) and the apical angle O1-Cu1-N1 of 149.64(9)° (Table 2). Two imino nitrogen atoms, one iminol oxygen atom and one pyridine nitrogen atom occupy the basal plane, while one pyridine nitrogen atom and one carbonyl oxygen atom occupy the apical positions. The Cu-N distances are in the range 2.050(2)-2.277(3) Å and agree with the values reported by Santiago *et al.* [42]. The Cu1-O1 and Cu1-O2 are, respectively, 2.099(2) and 2.130(2) Å showing the negative charge difference of these two oxygen atoms. In fact, the iminolate oxygen atom O1 is more negatively charged than the carbonyl oxygen atom O2 and the link O2 to Cu1 is stronger, resulting in a shorter distance as observed for similar complexes [42]. Additionally, the C-O bonds in the two ligand molecules are slightly different. The C21-O2 (1.271 (3) Å) bond is longer than the C8-O1 (1.264 (3) Å) bond which has a double bond character. The sum of the angles subtended by the donor atoms at the Cu (II) in the equatorial plane [N2 O2 N6 N5] is 359.95°, indicating approximate coplanarity for these atoms as shown by the rms value of 0.0780 Å. The crystal structure of the copper complex illustrated in Figure 4 shows two types of π - π

stacking interactions between the 2-substituted pyridyl ring and the 3-substituted pyridine ring with $Cg \cdots Cg$ distances of 3.845 and 3.940 Å respectively. The π - π stacking interaction link the molecules into sheet parallel to the *bc* plane (Figure 4).

4. Conclusion

The acetylpyridineticotinohydrazone ligand well known in the literature is used to synthesize two new complexes from perchlorate salts of cobalt or copper. Magnetic measurements, spectroscopic studies, and X-ray diffraction indicated the formation of a dicationic mononuclear complex in the case of cobalt (**1**) and a monocationic mononuclear complex in the case of copper (**2**). In complex **1**, two ligand molecules act in a tridentate fashion in a neutral form, while in complex **2** one ligand molecule acts in a tridentate manner in a neutral form while a second monodeprotonated ligand molecule acts in tridentate fashion. In both structures, we have a hexacoordinate environment with a distorted octahedral geometry for the two complexes.

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Supporting information

CCDC-2054420 and 2054421 contain the supplementary crystallographic data for compounds **1** and **2**, respectively. These data can be obtained free of charge via

<https://www.ccdc.cam.ac.uk/structures/>, or by e-mailing 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.

Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.

Author contributions: All authors contributed equally to this work.

Ethical approval: All ethical guidelines have been adhered.

Sample availability: Samples of the compounds are available from the author.

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