Coordination of N,O-donorappended Schiff base ligand *N,N'*-bis(3-methoxy salicylidenimino-1,3-diaminopropane) towards Nickel (II), Copper (II) and Zinc (II) : Syntheses, crystal structure

ABSTRACT

New complexesprepared by the reaction of*N,N'*-bis(3-methoxysalicylidenimino-1,3-diaminopropane) (**H_2L**)withNi(II), Zn(II) andCu(II) ions arereported in thispaper. The **H_2L**ligand isstructurallycharacterized by elementalanalysis, NMR and infraredand UV-Visspectroscopies, conductance andmagnetic room temperaturemeasurement.The mononuclearcomplex of Ni(II) (**$C_{19}H_{24}N_2NiO_6$**) ischaracterizedby single X-ray diffraction. The compound crystallizes in the orthorhombic system in the space group Pnmawith the unit cellparametersa = 7.3754(5) Å, b = 21.9576(13) Å, c = 11.5546(6) Å, α = 90°, β = 90°, γ = 90°. The ligand **H_2L**acts in tetradentate fashion in its di-deprotonatedform. Twocoordinated water moleculescomplete the coordination sphere. The environmentaround the Ni(II) center is best described as an octahedralgeometry.

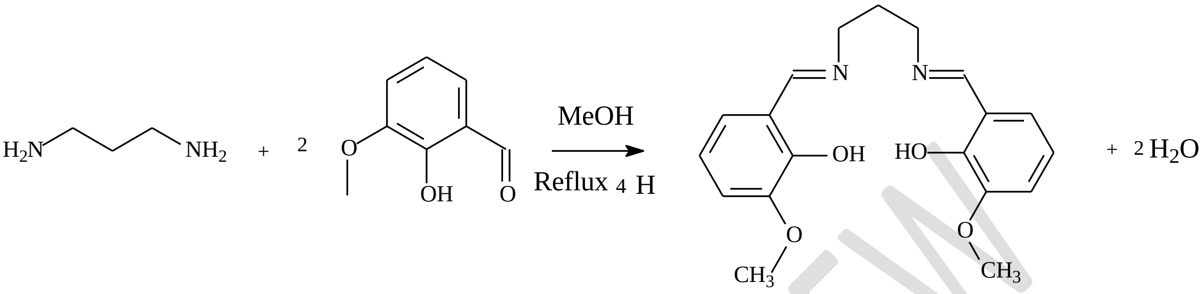
Keywords Schiff base, X-ray, mononuclearcomplex, tetradentate

1. INTRODUCTION

The use of Schiff' bases in coordination chemistry, to develop from molecular units with specific properties has undergone spectacular development in recent years thanks to very elaborate synthesis methods which make it possible to control the synthesis of complexes with original structures. Polyfunctional acyclic, symmetric, or asymmetric ligands and their complexes represent an essential component of this chemistry [1–6]. These ligands are widely used for the synthesis of complexes with very interesting biochemical and physical properties [7–10]. The formation of acyclic ligand complexes depends on the flexibility of the arms bearing the groups having donor sites [11,12], the nature of the donor atoms and the complexation properties of the cations and anions playing the role of charge balance [13,14]. These Schiff bases have made it possible to develop molecular materials with specific properties [15–19]. This article describes the synthesis of a tetradentate Schiff base obtained by the reaction between 2-hydroxy-3-methoxybenzaldehyde and 1,3-diamine propane. The flexibility and numerous coordination sites of the resulting acyclic ligands of this type offer several possible structures with lanthanide ions [20–25].

2. EXPERIMENTAL

2.1 Synthesis of *N,N’*-bis(3-methoxysalicylidenimino-1,3-diaminopropane)(H_2L)



Scheme 1.Synthesis of the ligand H_2L

The Schiff base *N,N’*-bis(3-methoxysalicylidenimino-1,3-diaminopropane) (**H_2L**) waspreparedwithslight modification of literatureprocedures.1,3-diamine propanedissolved in methanolwasrefluxedwith an methanolic solution of 2-hydroxy-3-méthoxybenzaldéhyde, in the presence of few drops of glacial aceticacid.In a 250 ml flask, dissolve 3.04 g (20 mmol) of o-vanillin or (2-hydroxy-3-methoxybenzaldehyde) in methanol. Add 0.83g (10mmol of density 0.888g/mL) of 1,3-diamine propane suspended in methanol. A yellow solution wasobtained. The reflux wasmaintained for 4hours. On cooling the reactant media, yellowprecipitatewasseparated. The compound wasthoroughlywashedwithether and dried over P_4O_{10}.

Yield: 77.15%; Melting temperature: 140 - 141°C

RMN ^{1}H : (500 MHz, DMSO-d_6, δ, ppm) : 3.71 (s, 6H, CH_3), 6.8-6.62 (m, 6H, Ar-H), 2.53 (t, 2H, CH), 1.63 (s, 2H, OH), 3.79 (t, 4H, CH_2).

RMN ^{13}C : (125 MHz, DMSO-d_6, δ, ppm): 55,47 (C_2 ; $C_{2'}$), 117,88 (C_6 ; $C_{6'}$), 120,041 (C_4 ; $C_{4'}$), 120,47 (C_5 ; $C_{5'}$), 147,38 (C_3 ; $C_{3'}$), 146,82 (C_9 ; $C_{9'}$), 46,11 (C_{10}), 111,00 (C_7 ; $C_{7'}$), 28,82 ($C_{1;}$), 147,38 (C_8 ; $C_{8'}$).

I**R: ν(cm^{-1}) :** 1628 (C=N)$_{imine}$; 3078 (H-C=N) ; 3189(OH), (1466 - 1358) (C=C) ; 1245 (C-O), 2946 (C-H).

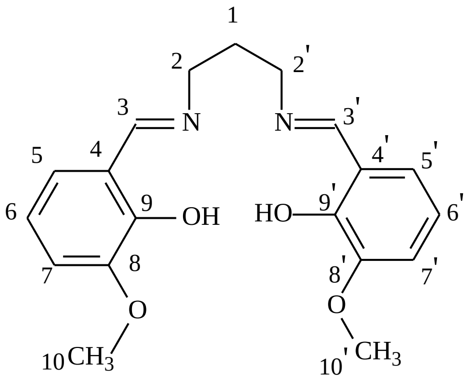
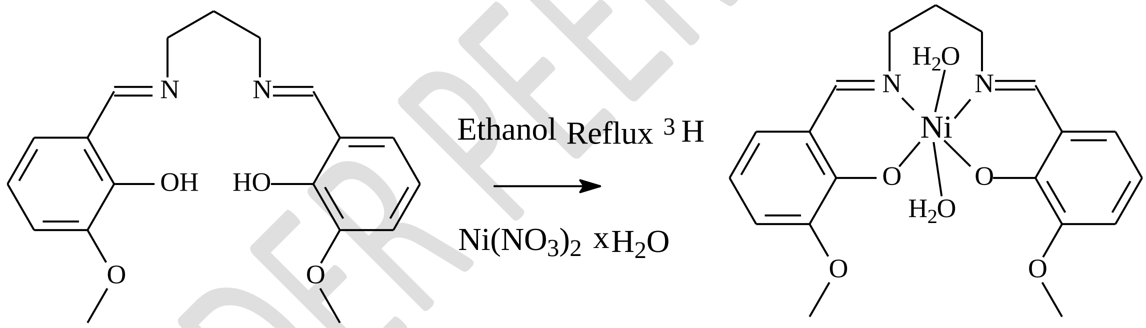


Figure 1. H2L ligand

2.2 Synthesis of the complexes

In a 100 mL flask containing 20 mL of ethanol, dissolve 0.15 g (0.44 mmol) of the H_2L ligand. Add (0.44 mmol) metal nitrate x hydrated $M(NO_3)_2.xH_2O$ with (M = Ni, Cu, Zn and x = 3; 4; 6) previously dissolved in 10 mL of ethanol. A precipitate immediately forms for all complexes. The mixture obtained is stirred and heated to reflux for three hours. Collect the precipitates by filtration, wash them with ether and dry them in the open air. Leave the filtrates to slowly evaporate for a few days; green crystals form for the nickel (II) complex filtrate. Yields range from 78.7% to 97.77%.



Scheme 2. Synthesis of nickel (II) complex

3. RESULTS AND DISCUSSION

3.1 General Studies

The infrared spectrum of the ligand reveals the disappearance of the intense band pointed at 1637 cm^{-1} attributed to the ν(C=O) vibration of o-vanillin and the appearance of a new strong band at 1628 cm^{-1} corresponding to the vibration ν(C=N). This proves that condensation has indeed taken place. The bands attributed to the C-H vibrations of the aromatic skeleton are noted at 3078, 1076, 958 and 833 cm^{-1}[26]. The valence vibration of the phenolic (C-O) bond is pointed at 1245 cm^{-1} and those of the CH2 groups at 2843 cm^{-1}. The very strong band at 730 cm-1 accompanied by a shoulder justifies the presence of an aromatic core. We observe a broad band at 3189 cm^{-1} attributable to the ν(OH) vibrations of the ligand.

The analysis of infrared data of the H_2L ligand complexes reveals a decrease towards low frequencies of the vibration bands characteristic of the C=N bonds of the C-O imine group of the phenol group. This reduction ranging from 1628 to 1612 cm^{-1} for the imine group and 1245 to 1216 cm^{-1} testifies to the coordination of the electron donor atoms of these groups to the metal [27]. However, let us recall that despite the slight reduction in the vibration band characteristic of the C-O bond of the methoxy group observed at 1076 cm^{-1} on the spectrum of the ligand, its donor atom does not participate in the coordination of the metal. On the spectra of all complexes, we note the presence of new vibration bands in the regions (1470 – 1762), (1298 – 1292) and (1040 – 1008) cm^{-1}. These triplet bands indicate the presence of coordinated nitrate in the complexes. The magnitude values estimated respectively at 169, 172 and 167 cm^{-1} for the copper, nickel and zinc complexes prove that the nitrates are coordinated to the metal in a bidentate chelate manner. On the spectrum of the zinc complex, we also observe a characteristic vibration band of free nitrate group at 1367 cm^{-1}.

The study of the molar conductivity of these complexes in a millimolaire solution of DMF confirms the position of the nitrate groups in the complexes. Indeed, the newly found values for the copper and nickel complexes indicate that their electrolytes are of neutral type while that of the zinc complex gives an electrolyte of type 1: 1. Fifteen days later the values found show that the complexes are all stable in DMF[28].

The UV-visible spectra of the complexes are recorded in a millimolaire solution of DMF. They present bands attributed to the $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ transitions of the chromophore groups and charge transfer bands from the ligand to the metal (CTLM). These transitions are observed in the range 410 – 460 nm. In addition to these bands, we note on the spectrum of the copper complex a broad band pointed at 630 nm which characterizes the $d \rightarrow d$ transitions of a copper ion in an octahedral environment [29]. This environment of the metal is confirmed by the magnetic moment which is equal to 1.77 µB, a value which is typical of an octahedral copper mononuclear complex [30].

On the spectrum of the nickel (II) complex, the broad band observed at 638 nm is attributed to the sum of the transitions ${}^3A_{2g} \rightarrow {}^3T_{2g}$ and ${}^3A_{2g} \rightarrow {}^3T_{1g}$ of an octahedral nickel (II) complex [30]. The magnetic moment found is 2.76 µB. This value is that of a mononuclear nickel complex in an octahedral environment.

On the spectrum of the zinc (II) complex the charge transfer band from the ligand to the metal is observed at 460 nm. It suggests the presence of zinc in the product.

X–ray Crystallography :Details of the crystal structure solution and refinement are given in table 2. Single-crystal X-ray diffraction data were measured on a Rigaku Synergy S diffractometer at the MoKα radiation (λ = 0.71073 Å).

Table 1.UV-visible and magnetic data of metal nitrate complexes with H_2L ligand

Complexes	**UV-Visible**		**μ_{eff} (μB)**
	λ(nm)	**Attributions**	
$[Cu(HL)(NO_3)]^{\cdot}xH_2O$	344 ; 392 ; 410 ; 630	π→ π*, n→ π*, CTLM, d → d	1.77
$[Ni(HL)(NO_3)]^{\cdot}xH_2O$	348 ; 388 ; 408 ; 638	π→ π*, n→ π*, CTLM M, d → d	2.77
$[Zn(H_2L)(NO_3)]^{\cdot}(NO_3)^{\cdot}xH_2O$	332 ; 385 ; 460	π→ π*, n→ π*, CTLM , d → d	Diam

Table 2. Crystal data and details of the structure determination

Chemical formula	$C_{19}H_{24}N_2NiO_6$
Mr (g/mol)	431.11
Température (K)	100 K
Radiation (Å)	0.71075
Crystal size (mm^3)	0.22 × 0.1 × 0.06
Crystal system	Orthorhombique
Space group	Pnma
Z	4
a ; b ; c(Å)	7.3754 (5) ; 21.9576 (13) ; 11.5546 (6)
α ; β ; γ(°)	90
V ($Å^3$)	1871.22 (19)
Calculateddensity($Mg.m^{-3}$)	1.544
Radiation type	Mo Kα
μ (mm^{-1})	1.08
F(000)	912
θ (°)	3.3 – 31.8
h, k, l	$-9\leq h \leq 9$; $-24\leq k \leq 29$; $-15\leq l \leq 15$
No. of measuredreflections, independentand	2468 ; 1639 ; 2166

observed[$I \geq 2u(I)$] ; Final Indices R [I>2σ(I)]	
GOF	1.06
$R[F^2 > 2\sigma(F^2)]$; $wR(F^2)$; R_{int}	0.045 ; 0.120 ; 0.027
No. of parameters	66
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.81 ; −0.68

Table 3. Bond lengths (Å) and angles around Ni (°) in the complex [$Ni(H_2L)(H_2O)_2$]

Bond	Bond lengths(**Å**)	Corner	**Value (°)**
Ni2-N1^i	2.073(2)	O2-Ni-N1	91.93(7)
Ni2-N1^i	2.073(2)	N1-Ni-N1^i	97.38(11)
Ni2-O1	2.0141(17)	O2-Ni-O2^i	85.98(10)
Ni2-O2^i	2.0141(17)	O2^i-Ni-O3	91.93(7)
Ni2-O4	2.095(2)	N1^i-Ni-O4	89.04(6)
Ni2-O3	2.112(3)	O2-Ni-N1^i	174.29(7)

Symmetry code: (i) x ; −*y*−1/2, *z*

Table 4.Intermolecular hydrogen bond lengths in (Å) for the complex [$Ni(H_2L)(H_2O)_2$]

D*—H···*A	***D*—H**	**H···*A***	***D*···*A***	***D*—H···*A***
O4—H4A···O2^i	0.88	1.99	2.75(3)	148.6

Symmetry code:(i) x-1/2, −*y*−1/2, −*z*+3/2

Crystallographic analysis of the [$Ni(HL)(H_2O)_2$] complex reveals that it crystallizes in an Orthorhombic system with a Pnma space group. The unit cell parameters recorded in table 2are a = 7.3754 (5) Å; b = 21.9576 (13) Å; c = 11.5546 (6) Å and α = β = γ = 90°. The asymmetric unit consists of a mononuclear complex in which a deprotonated ligand (L^-) coordinates through its two iminic nitrogen atoms and its two phenolate oxygen atoms forming an ONNO equatorial plane around the metal.However, it should be noted that the axial plane of the octahedral environment of nickel Ni^{2+} is formed by two water molecules. Analysis of crystallographic data reveals that the angles around nickel (II) in the equatorial plane O2-Ni-N1, N1-Ni-N1i and O2-Ni-O2i are respectively 91.93(7)°, 97.38(11)° and 85.98(10)° while those of the axial plane O2i-Ni-O3 and N1i-Ni-O4 are 91.93(7)° and 89.04(6)°. These values suggest that the octahedral geometry around the nickel (II) is slightly distorted. This observation is confirmed by the lengths of the equatorial and axial connections. Indeed the lengths of these bonds are 2.073(2) Å for Ni—N1 and 2.0141(17) Å Ni—O2 all equatorial.For the bond lengths with nickel in the axial plane, the values are 2.112(3) Å for Ni—O3 and 2.095(2) Å for Ni—O4, respectively comparable to the axial bond lengths [Ni1—O1W = 2.0955 (19) Å and Ni1—O2W = 2.1071 (18) Åwith Barr and al.[30]. The crystallographic structure of the complex is stabilized by intermolecular hydrogen bonds of the Ophenol-H---Owater type.

3.2 Structure description of the complex of nickel (II)

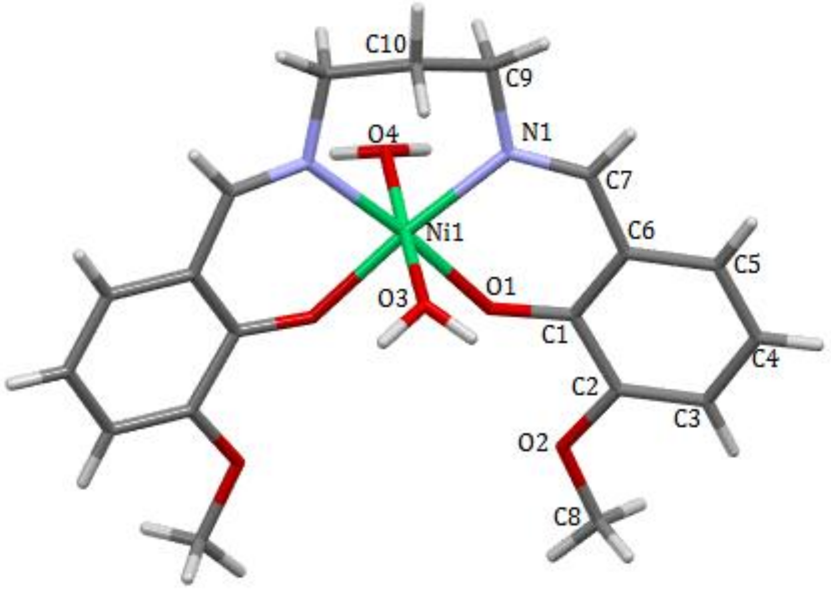


Figure 2.Crystallographic structure of the complex [Ni(L)(H_2O)$_2$]

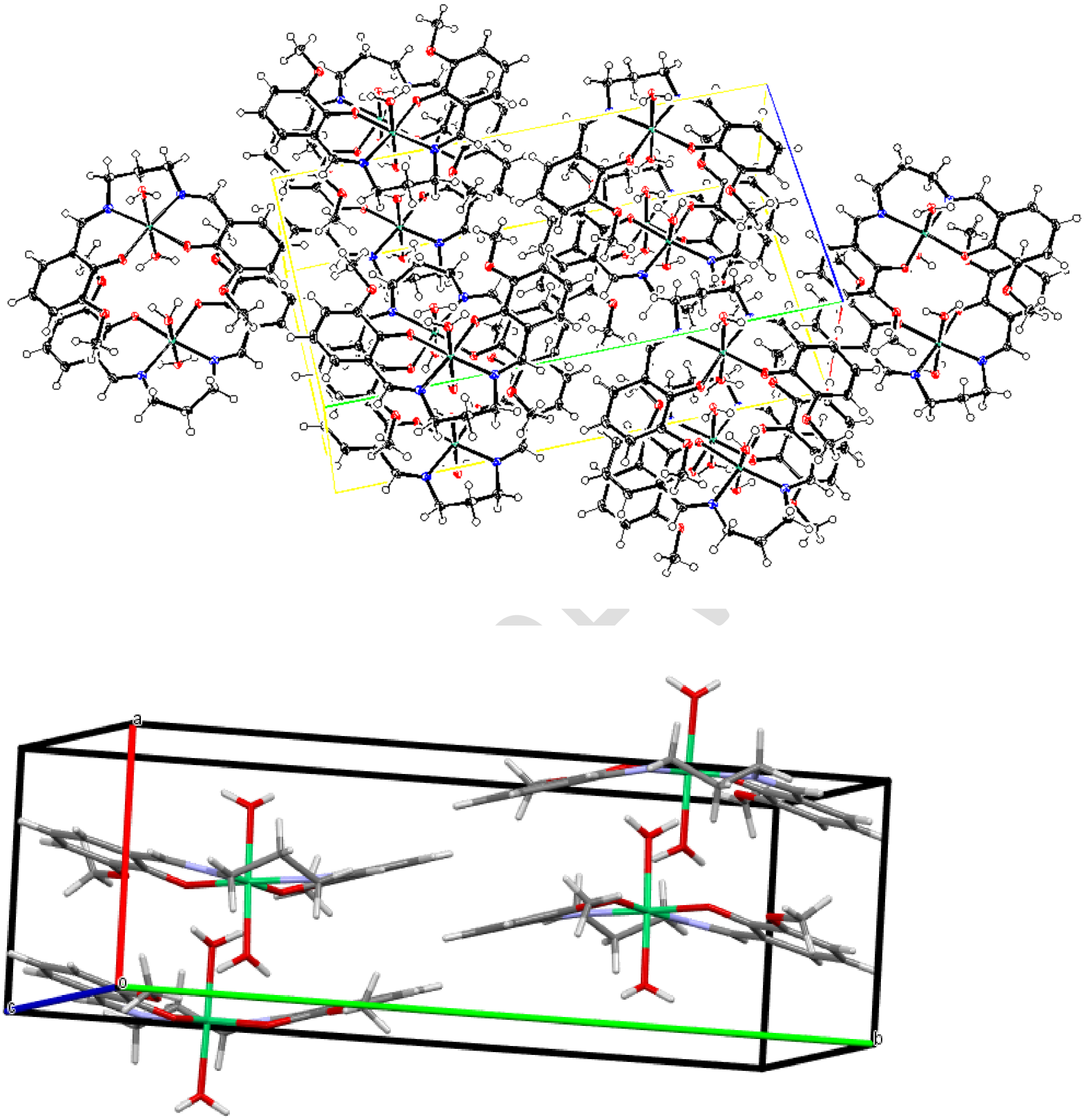


Figure 3. Three-dimensional structure of the complex [Ni(L)(H_2O)$_2$]

3.3 **Proposed other structures**

Based on spectroscopic, magnetic and conductometric analyses, we propose the following structures:

- The complexes are mononuclear.
- In the structure of the complexes, the ligand acts in a tetradentate manner through its two phenolic oxygen atoms and two iminic nitrogen atoms.

- The nitrate ions are coordinated to the metal in a bidentate chelate manner and a free nitrate which plays the stabilizing role in the zinc (II) complex.
- The complexes contain free water molecules.

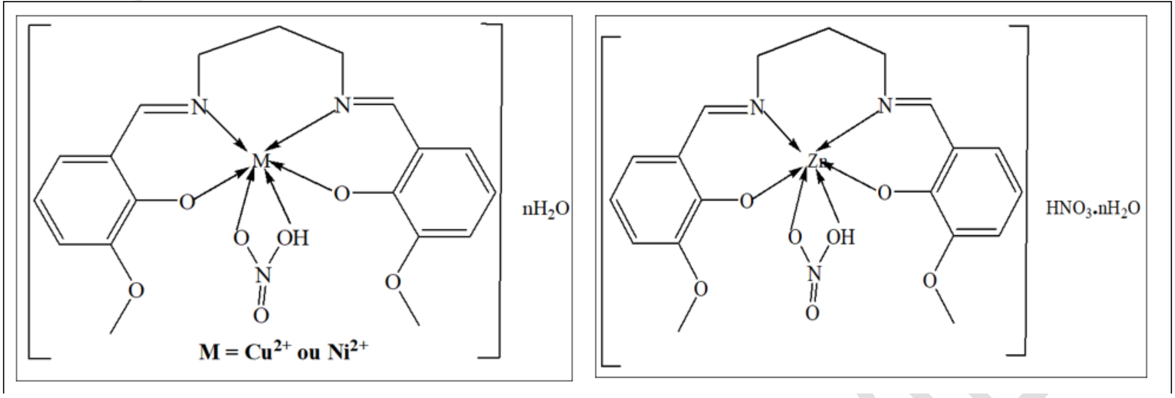


Figure 4.Proposed structure of copper, nickel and zinc (II) complexes with the H$_2$L ligand

4. CONCLUSION

The synthesis and characterization of the Schiff base ligand *N,N'*-bis(3-methoxysalicylidenimino-1,3-diaminopropane), threemetal transition complexes weresynthesized and werestudiedusingspectroscopic and spectrophotometricmethods as well as X-ray diffraction technique for nickel(II) complex. The dianionic ligand acts in tetradentate mode via twoazomethinenitrogenatoms and twooxygenphenolateatoms. Infraredspectroscopyconfirms the coordination between the Schiff base ligand and the cations Ni(II), Cu(II) and Zn(II). The structure of nickel(II) complexisdetermined by single crystal x-ray diffraction. The environmentaroundeach cation isoctahedralUV-Vis spectrophotometryindicatesinternal transition in the ligand and metal.This paperdescribes a mononuclear nickel (II) complex. The structure revealstetradentate coordination of the ligand via twonitrogenatoms and twodeprotonatedphenolicoxygenatoms. In thiscomplexthe central metal center ishexacoordinated. These complexeswerecharacterized by spectroscopicstudies (IR, UV-Vis),molarconductivity and roomtemperaturemagnetic moment measurements.Suitable X-ray crystal diffraction wereobtained forthe Nickel (II)complex.

Nitrate ioncompletethe sphere of coordination forming Ni(II), Zn (II) and Zn(II) complexes with acoordination number of six. Sometime later two water molecules replaced the nitrate ion in Nickel (II) complex. The environmentaround the metal center is best described as a as aoctahedralgeometry. The ligands show similar fashion of coordination mode for eachmetal ion, acting as tetradentate. Eachmetal center iseightcoordinatedwithtwonitrogenatomfrom the ligand and twooxygenatomfromtwo water molecules. The environmentaroundeachmetal for both complexes is best described as a octahedralgeometry.

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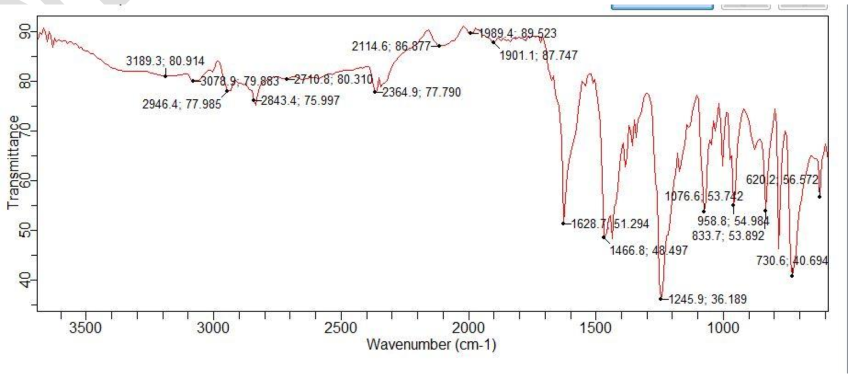
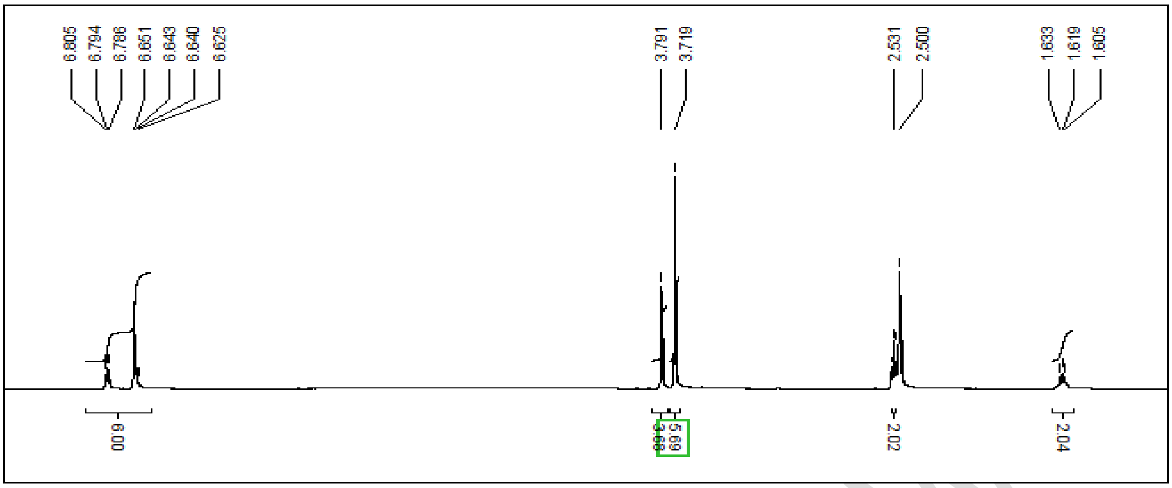
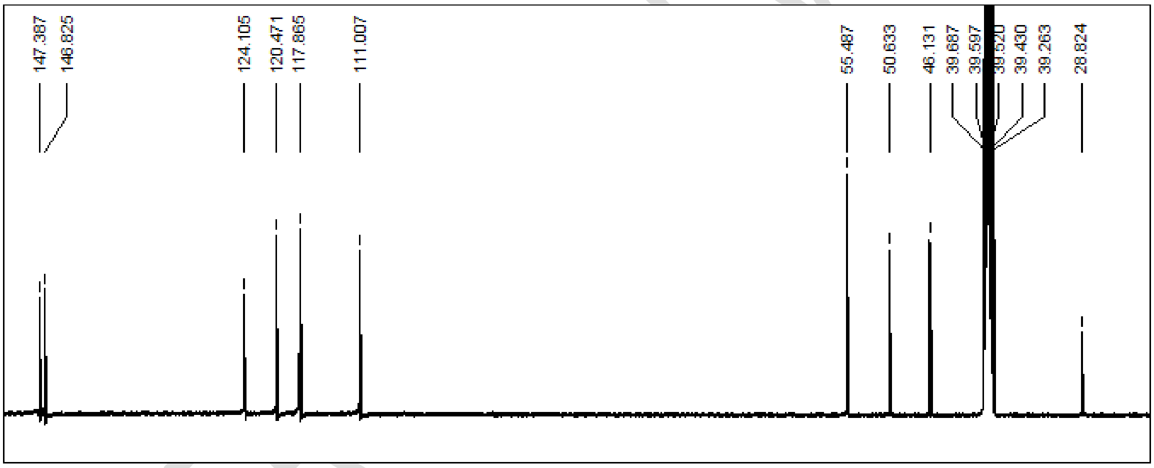
SUPPLEMENTARY MATERIALS



Spectrum IR of ligand H_2L



SPECTRUM RMN ^{1}H



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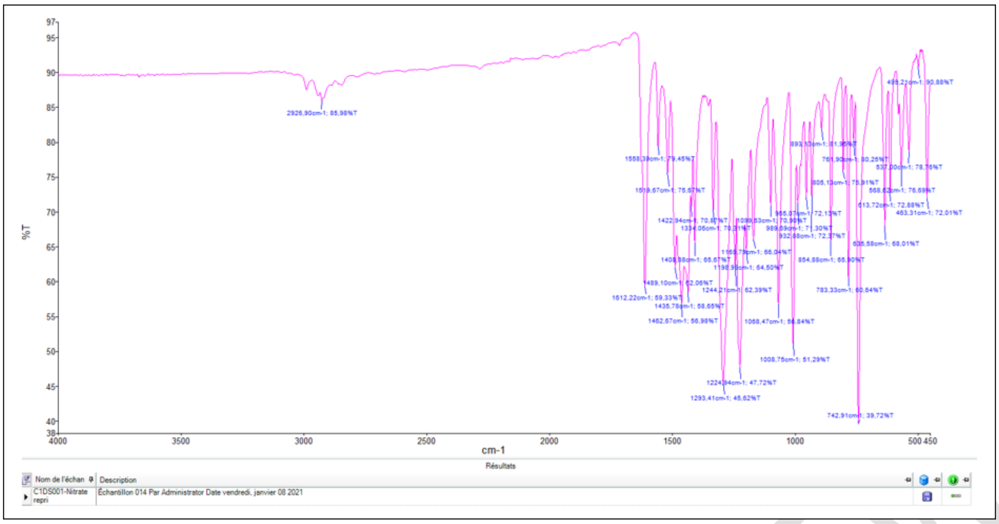

Nom de l'échan	Description
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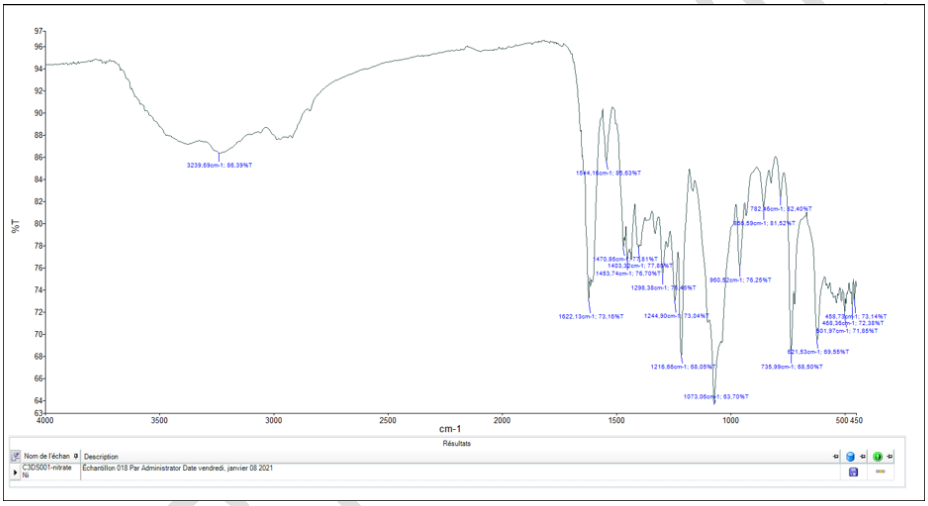
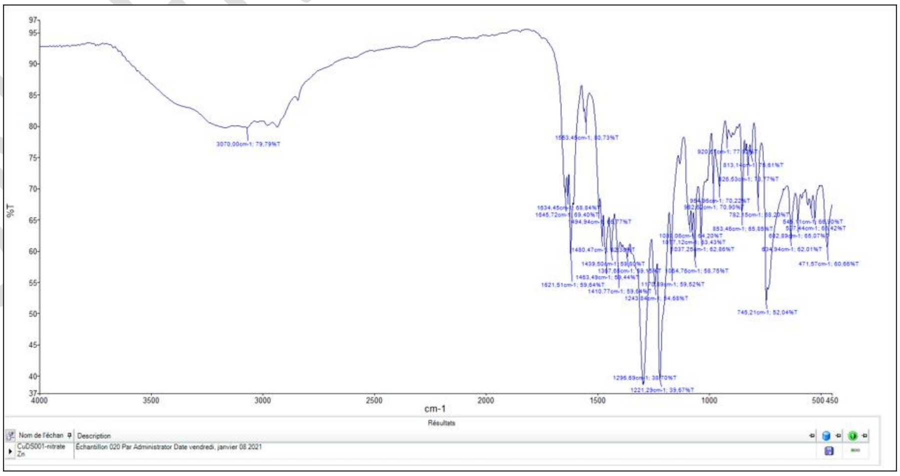
Spectrum IR of complex[$Cu(L)(NO_3)]^{\cdot}nH_2O$



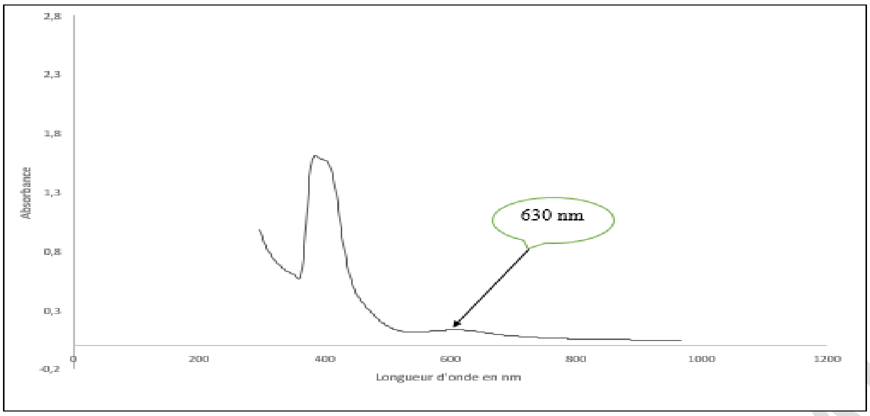
Nom de l'échan	Description
C3DS001-nitrate Ni	Échantillon 018 Par Administrator Date vendredi, janvier 08 2021

Spectrum IR of complex[$Ni(L)(NO_3)]^{\cdot}nH_2O$

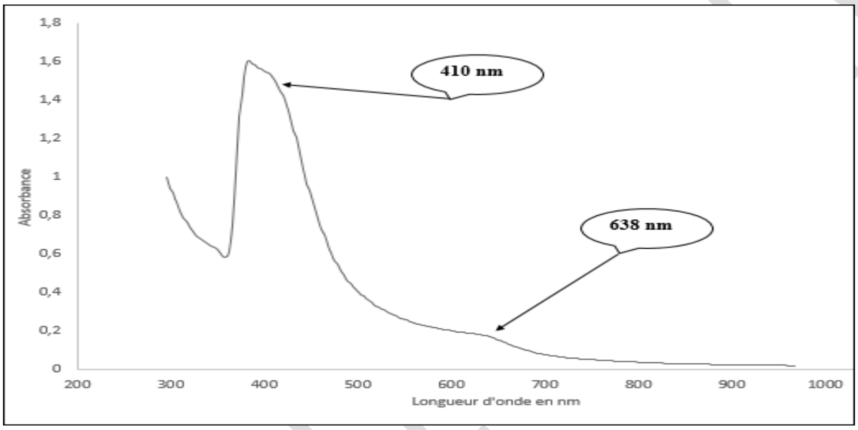


Nom de l'échan	Description
CuDS001-nitrate Zn	Échantillon 020 Par Administrator Date vendredi, janvier 08 2021

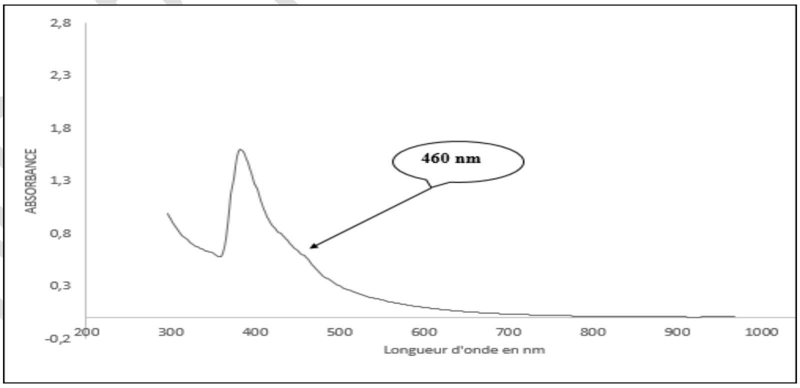
Spectrum IR of complex[$Zn(L)(NO_3)]^{\cdot}nH_2O$



Spectrum UV-Vis of complex$[Cu(L)(NO_3)]^{\cdot}nH_2O$



Spectrum UV-Vis of complex$[Ni(L)(NO_3)]^{\cdot}nH_2O$



Spectrum UV-Vis ofcrystal$[Ni(L)]^{\cdot}2H_2O$