

Preparation, Characterization and Spectroscopic Study of New Tridentate Schiff Base and its Cu(II), Ni(II) and Zn(II) Metal Complexes

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Abstract :

A new tridentate ligand has been synthesized derived from phenyl(pyridin-3-yl)methanone. Three coordinated metal complexes were prepared by complexation of the new ligand with Cu(II), Ni(II) and Zn(II) metal salts. The new Schiff base "benzyl -2-[phenyl(pyridin-3-yl)methylidene]hydrazinecarbodithioate" and the new metal complexes were characterized using various physico-chemical and spectroscopic techniques. From the analysis results, the expected structure to the metal complexes are octahedral in geometry for Cu(II) complex, square planner for Ni(II) and tetrahedral for Zn(II) complex. The new compounds are expected to show strong bioactivity against bacteria and cancer cells.

Keywords: Schiff Base, tridentate, metal complexes

Introduction:

Schiff bases and their metal complexes have attracted the attention of researchers for several decades. Hhydrazinecarbodithioic acid $\text{NH}_2\text{NHCS}_2^-$ and its substituted derivatives remain high in interest and has been studied since the seventies of the last century [1-3]. These compounds warrant further study because they provide an interesting series of ligands whose properties can be greatly modified by introducing different Schiff bases, not only because of their intriguing coordination chemistry, but also because of their pronounced biological activities against microbes, viruses, and cancer cells. [4-10] A new Schiff base benzyl -2-[phenyl(pyridin-3-yl)methylidene]hydrazinecarbodithioate (L) from the condensation reaction of phenyl(pyridin-3-yl)methanone with benzyl hydrazinecarbodithioate while the three new metal complexes were prepared using copper(II) acetate

trihydrate, nickel(II) acetate and zinc(II) acetate. The new compounds were characterized using various physico-chemical and spectroscopic techniques.

Materials and Methods :

Materials

Chemicals that have been used in this research are: potassium hydroxide (BDH), hydrazine hydrate (Fluka), carbon disulphide (BDH), phenyl(pyridin-3-yl)methanone (BDH), benzyl chloride (Acros), Cupper(II) acetate trihydrate (BDH), Nickel(II) acetate and Zinc(II) acetate (fluka). All chemicals were used as supplied without any further purification. Absolute ethanol (99.99%, BDH), Diethyl ether (fluka) and dimethyl sulphoxide (DMSO) (99% BDH).

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Methods

Physical measurements

Melting point apparatus of Gallenkamp M.F.B 600.01 was used to measure the melting points of all prepared compounds. The infrared spectra of the prepared compounds were recorded using FT-IR-8300-Shimadzu, in the wave length range of (4000-400) cm^{-1} . The electronic spectra obtained using (UV-Vis-160A) Shimadzu Spectrophotometer, in the range of wave-length (200-1100 nm). Carbon, hydrogen, nitrogen and sulfur analyses were carried out using a Leco CHNS-932 analyzer. The molar conductivity measurements were carried out using Wissenschaftlich-Technisch Werk- -stätten 8120 Weilheim I.O.B, Drucker-Printer. ^1H NMR spectra and ^{13}C spectra were recorded on Jeol (JNM-ECA400) 400 MHz NMR spectrometer. The magnetic susceptibility values using (Magnetic Susceptibility Balance), of Johnson matthey catalytic system division using atomic absorption technique by Perkin-Elmer-5000 to (Cu^{+2} , Ni^{+2} and Zn^{+2}) metal ions.

Preparation of benzyl hydrazinecarbodithioate

This compound was synthesized as previously reported [11, 12]. Potassium hydroxide (0.2 mol) was dissolved in 95% ethanol (70 ml) and the solution was cooled in ice bath. To this solution, hydrazine hydrate (0.2 mol) was added. Carbon disulfide (0.2 mol) was then added dropwise. The resulting lower yellow layer was separated and dissolved in 40% ethanol (40 ml). Benzyl chloride (0.2 mol) was added slowly. The white resulted product was filtered, washed and dried in a vacuum desiccator over a night. The crude product was recrystallized from ethanol. The melting point was sharp 123 ± 1 °C. (Lit. 125°C ; yield: 92%). (Calc. C, 48.5; H, 5.1; N, 14.1; S, 32.3%) (Found: C, 48.3; H, 5.0; N,

13.9; S, 31.2%), [12]. The yield was 87%.

Preparation of Schiff base (L) by condensation reaction

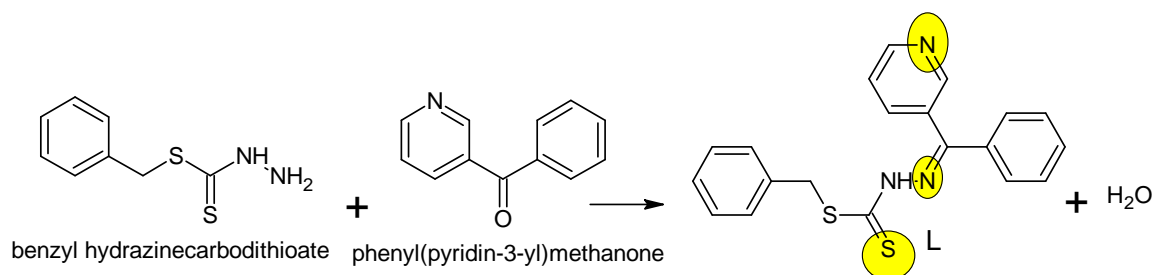
(0.01 mole) of benzyl hydrazinecarbodithioate was dissolved in 95% ethanol. An equimolar of 3-benzoylpyridine was dissolved in ethanol 95% by heating, the two solutions were mixed together and heated until the light yellow color crystals formed which were filtered off and washed with ethanol dried in room temperature. The Schiff base (L3) obtained has a melting point 156 ± 1 °C.

Preparation of metal complexes

0.5 g of Schiff base L3 was dissolved in 40 ml ethanol 95% and heated. A half molar of metal salt was dissolved in ethanol 95% and heated. Both solutions were mixed together and heated. Then the mixture was allowed to cool and the precipitate formed was filtered off. The complex formed was dark in color and was then dried at room temperature. The metal complex obtained has a melting point higher than the from Schiff base. All compounds had sharp melting points indicating that the complexes were relatively free of impurities.

Results and Discussions :

Scheme 1 shows the condensation reaction to prepare the new ligand L, while the physical and analytical data for the new ligand L and its three transition metals complexes [Cu(II), Ni(II) and Zn(II)] are shown in (Table 1). All analytical data obtained are in agreement with proposed structures. Also the analytical values of percentage of metal in the metal complexes were found very close to the calculated value which indicates that the structures are similar as what we predicted in CHNS result.



Scheme 1: Preparation of new ligand L

Table 1: Physical properties and (CHNS) Analyses data of the new compounds

Compound	Color	Melting Point (± 1 °C)	Yield %	% Found (calculated)				
				Carbon%	Hydrogen%	Nitrogen%	Sulfur%	Metal%
L C ₂₀ H ₁₇ N ₃ S ₂	Orange yellowish	155	58	70.23 (66.08)	5.23 (4.71)	13.11 (11.56)	19.83 (17.64)	-
Cu(L) ₂ C ₄₀ H ₃₂ CuN ₆ S ₄	Dark brown	202	44	60.07 (60.93)	4.26 (4.09)	10.81 (10.66)	16.26 (16.27)	8.92 (8.06)
Ni(L) ₂ C ₄₀ H ₃₂ NiN ₆ S ₄	Yellow brownish	201	43	56.87 (61.30)	4.41 (4.12)	10.59 (10.72)	12.73 (16.37)	6.95 (7.49)
Zn(L) ₂ C ₄₀ H ₃₂ NiN ₆ S ₄	Yellow	171	35	57.93 (60.78)	4.36 (4.08)	10.65 (10.63)	12.19 (16.23)	8.41 (8.28)

Fourier Transform Infrared Spectroscopy (FTIR)

The results of interpreted data from the spectrum of Fourier Transform Infrared Spectroscopy (FTIR) are as shown in (Table 2).

Table 2: Fourier Transform Infrared Spectroscopy (FT IR) data

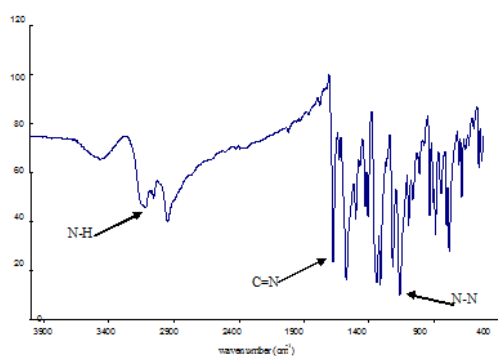
Compound	ν (C=N)	ν (C-S)	ν (N-N)	ν (OH)	ν (NH)	ν (M-N)
L	1631	-	1265	3441	3292	-
Cu(L) ₂	1565	961	1025	3453	3021	509
Ni(L) ₂	1570	977	1029	3431	3022	449
Zn(L) ₂	1583	829	1051	3440	3079	527

From the observation, the free benzyl hydrazinecarbodithioate ligand does not display band at ν (S-H) which is attributed to *ca*(S-H), but its display a very strong intensity at ν (1048 cm⁻¹, bending) which represent as C=S which clearly indicates it remains in the thione form in the solid state. In L3 Schiff base, there is no peak assigned to C=S indicating that the Schiff bases appear in thiole form. There is peak at (1631 cm⁻¹) which attribute to C=N, from this observation, it is concluded that the Schiff bases are formed via condensation reaction with 3-

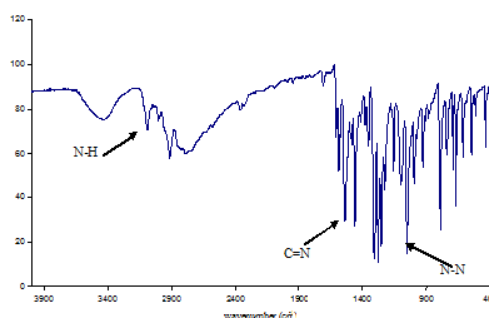
benzoylpyridine in which the primary amine reacted with the carbonyl group to yield the respective product. For complexes of copper, nickel and zinc there is peaks assigned at (961 cm⁻¹, 977 cm⁻¹ and 929 cm⁻¹) which attribute to C-S, This only can happen if ligand in thiole form. It indicating that the ligand were undergoes deprotonated and acted as tridentate uni-negative (NNS-) chelating agents. In benzoylpyridine where pyridine nitrogen atom were at 3 coordination, C=N showed at peak *ca*(1631cm⁻¹) but as we can see that in the metal

complexes, it is shift to the lower frequency to 1583 cm^{-1} - 1565 cm^{-1}) this indicating that the metal bonded through azomethine nitrogen atom. A strong band at 1265 cm^{-1} in the IR spectrum of the Schiff base is assigned to the hydrazinic $\nu(\text{N-N})$ band. Its shift to lower wavenumbers in spectra of all the metal complexes (1025 - 1051 cm^{-1}) reflects reduction in the repulsion between the lone pairs of electrons on

the nitrogen atoms and is certainly due to coordination *via* the azomethine nitrogen atom.[13-18]The appearance of new weak low frequency bands around 449 - 527 cm^{-1} which are due to $\nu(\text{M-N})$ [19] while $\nu(\text{M-S})$ cannot be distinguished due to overlap between the bands. (Figure 1) show the spectra of the Schiff base and its copper complex. [19]



Schiff base Spectra



Copper complex

Figure 1: FT-IR Spectrum off the Schiff base (L) and its copper complex

Molar Conductance Results

The molar conductivities were determined using the prepared solution

of metal complexes with concentration of 10^{-3} M in DMSO. (Table 3) shows these conductivities.

Table 3: Molar Conductivity and Magnetic Susceptibility data

Compounds	$\lambda_M (\text{ohm}^{-1}\text{cm}^2\text{mole}^{-1})$	μ_{eff}
Cu(L)_2	7.32	2.59
Ni(L)_2	19.78	1.63
Zn(L)_2	4.39	Diamagnetic

The conductivities in the range of 19.78 - $4.39\ \Omega^{-1}\text{cm}^2\text{mole}^{-1}$ indicating that all the complexes are non-electrolytes. Furthermore, the non-electrolytic nature of the complexes proves that the Schiff bases are coordinated to the metal ion as uninegatively charged tridentate species. [20]The magnetic susceptibility measurements of all metal complexes at room temperature are also tabulated in (Table 3).

Complexes without unpaired electrons are classified as diamagnetic, where they tend to move out of magnetic field and paramagnetic complexes are those with unpaired electrons and they tend to move into a magnetic field. Zinc is diamagnetic at room temperature because zinc have ten electrons (d^{10}) that field in orbital in term of pair. For others complexes, there are two tridentate attach on the metal, so its indicating octahedral geometry by

attachment via pyridine nitrogen atom, azomethine nitrogen atom and also through thiolato sulphur atom. [21-22]

Nuclear Magnetic Resonance (NMR) Spectroscopic Analyses

For the NMR analysis, the Schiff base was dissolved in dimethyl sulphoxide (DMSO). Both proton NMR and C-13 NMR were done. The results are shown in units of parts per million (ppm).

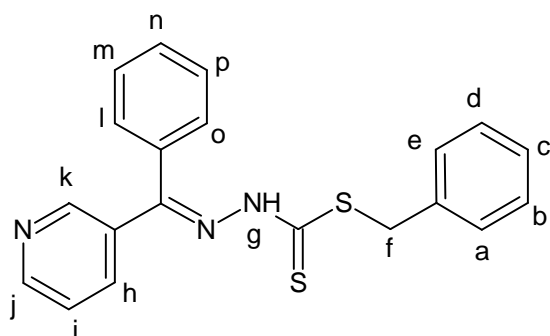


Table 4(a): protocol of the ^1H assignment

Shift (ppm)	Node
8.7381	K
8.7276	J
8.5845	I
8.4881	H
7.5844	P
7.4491	N
7.4353	M
7.4146	E
7.3275	D
4.4741	F

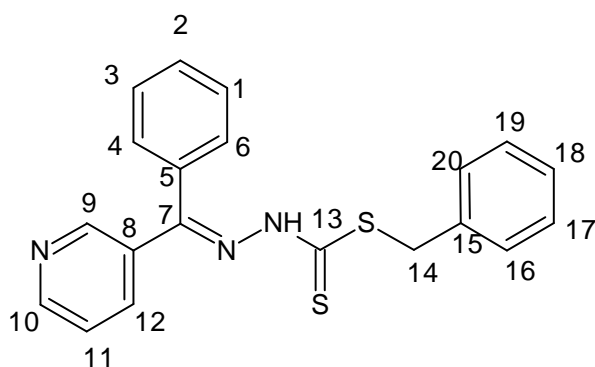


Table 4 (b): Protocol of the ^{13}C assignment

Shift(ppm)	Node
198.4890	13
150.5945	7
149.1226	9
148.5706	12
129.3668	4
129.3208	3
128.7574	19
128.5274	20
127.6535	18

A singlet peak appears at 4.47 ppm (2H) consistent with the presence of the S-CH₂ group thus supporting the proposed structure of the Schiff base. This signal occurs slightly downfield from assignments given for related compounds reported in the literature [23]. This is presumed to be because of the deshielding effect of the neighbouring electronegative sulphur and the aromatic ring.[24]The ^{13}C assignments of the structure of L, the measurements peaks are satisfied with expected structure. There are some peak do not appear in the spectrum because there were some over lapping of the symmetrical structure. For L, the carbon group 13 was a singlet group and it was a 198.4890 ppm because it was deshielded by two sulphur atom and a nitrogen atom. The C=N signal of the Schiff base occurred at 150.59 ppm, indicated that the coordination of the azomethine bond which is formed in the preparation of Schiff bases.

Ultra Violet /Visible Analyses

The rise of the electronic absorption bands in the UV/V that is spectra of the complexes are the results from the transition electrons between the levels [25].

As listed in (Table 5) below, the Schiff base ligand shows the absorption on $\pi-\pi^*$ and $n-\pi^*$, which give bands at (242 nm- 390 nm). For zinc(II) complex, the spectra shows bands at (256 nm

and 384 nm) which are attributed to charge transfer. While for copper (II) and nickel(II) complexes, mostly gave

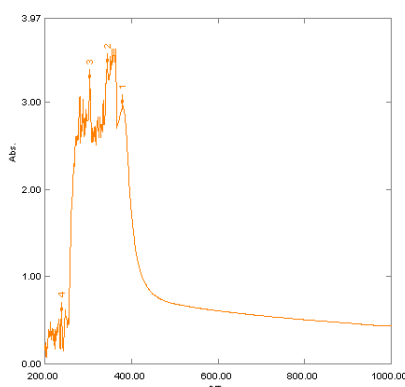
bands at (≈ 644 , shoulder) which represent as d-d transition region [26].

Table 5: Ultraviolet /Visible analysis data

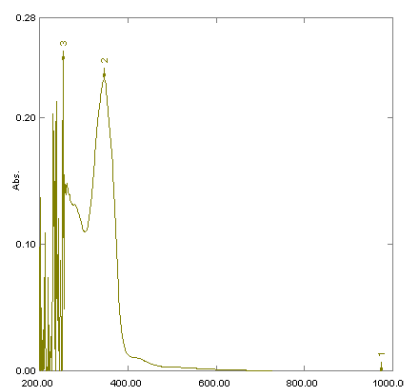
Compound	λ_{\max} (nm)
Schiff base ligand (L3)	242(4.04), 256(4.62), 293(3.90), 390(3.90)
Cu(L3) ₂	256(4.76), 644(3.00), 906(3.00)
Ni(L3) ₂	256(4.84), 644(3.00), 906(3.00)
Zn(L3) ₂	256(4.89), 384(4.18)

The ²D free ion term of the six-coordinated Cu(II) complexes will split into E_g and T_{2g} levels which will split into B_{1g}, A_{1g}, B_{2g}, E_g levels, respectively. Thus three spin-allowed transitions are expected in the visible and near IR region. But only a few complexes are known in which such bands are resolved completely. Hence, the absorption maximum band observed at ca. 644 to 720 nm results from ²B_{1g} ← ²A_{1g} and/or ²B_{1g} → ²E_g (Figure 2) show the electronic UV-Vis spectra of the ligand Zn(II) complex.

transitions [27]. The electronic spectra of the Ni(II) complexes containing 3- and 4-acetylpyridine show a d-d band at ca. 440 to 560 nm and 700 – 800 nm. A square-planar Ni(II) configuration gives rise to three bands corresponding to ¹A_{1g} → ¹A_{2g}, ¹A_{1g} → ¹B_{1g} and ¹A_{1g} → ¹E_g transitions, but in some cases these bands were too weak or were overshadowed by other stronger absorptions in the same region [28].



UV-Vis Spectra for the Schiff base L



UV-Vis Spectra for the Zn complex

Figure 2: The UV-Vis spectra of the ligand and its Zn(II) complex

Conclusion :

New Schiff base with three metal complexes were successfully synthesized, the ratio metal: ligand equal to 1:2 in all new complexes. The result of the magnetic susceptibility measurement indicates the octahedral geometry for Cu(L4)₂ while Ni(L4)₂

tends to be square planar geometry and tetrahedral for Zn(II) complexes. The new compounds expected to show strong bioactivity against bacteria, fungi and cancer cells. (Figure 3) shows the expected structures of the metal complexes.

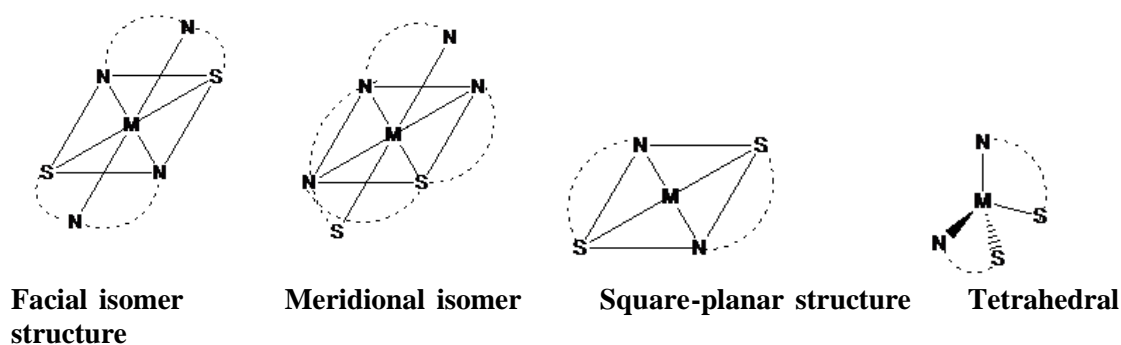


Figure 3: Expected structures of the metal complexes (NNS = Schiff bases)

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تحضير, تشخيص و دراسة طيفية لقاعدة شيف الثلاثية المنح الجديدة و معقداتها
لفلزات النحاس(II), النيكل(II) و الخارصين(II)

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الخلاصة :

تم تخليق معقد ثلاثي المنح جديد مشتق من فنيل(بريدين-3-يل)ميثانول. وتم تحضير ثلاثة معقدات فلزية تناسقية بواسطة تفاعل المعقد الجديد مع املاح فلزات النحاس(II), النيكل(II) و الخارصين(II). وقد تم تشخيص قاعدة شيف الجديدة بنزيل-2-[فنيل(بريدين-3-يل)مethylidene]هيدرازين كاربودايتاوات و معقدات الفلزات الجديدة باستخدام تقنيات فيزيو كيميائية و تقنيات طيفية. وقد تبين من نتائج التحليل بأن الشكل الهندسي المتوقع لمعقدات الفلزات هو الشكل ثماني الاضلاع لمعقد النحاس(II), و الشكل رباعي السطوح لمعقد النيكل(II) و رباعي الاضلاع لمعقد الخارصين(II). ويتوقع بأن تظهر المركبات الجديدة فعالية بايولوجية قوية ضد البكتريا و الخلايا السرطانية.