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Synthesis, Characterization and Biological Activity of Novel Metal Complexes Derived from Cyclohexylthiosemicarbazide

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ABSTRACT

In recent years, thiosmicarpazide compounds and its complexes have attracted the interest of researchers due to their many applications. Three new complexes $[LMnCl_2(H_2O)]$ (C1), $[LCoCl_2(H_2O)]$ (C2) and $[LNiCl_2(H_2O)]$ (C3) with the ligand N^1-cyclohexyl-N^2-(dietheylcarbamoyl) hydrazine-1,2-bis(carbothioamide) (L), the reaction was carried out by mixing the metal chloride and ligand in a 1:1 mole ratio using methanol as the medium. Complex formation between the metal (II) moiety and the ligand occurred through the O, S and N donor atoms. The ligand and its complexes were characterized by elemental analysis, FT-IR and NMR (^{1}H and ^{13}C) spectroscopy. Accordingly, the complexes were proposed to have octahedral geometry. The ligand and its complexes were screened for their antimicrobial activities against some Gram-positive and Gram-negative bacteria. The studies demonstrated that complexation can increase the antimicrobial activity, compared with the free ligand. The results indicated that the ligand behave as coordinate in tridentate to give a octahedral around the Mn II, Co II, and Ni II ions.

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1. INTRODUCTION

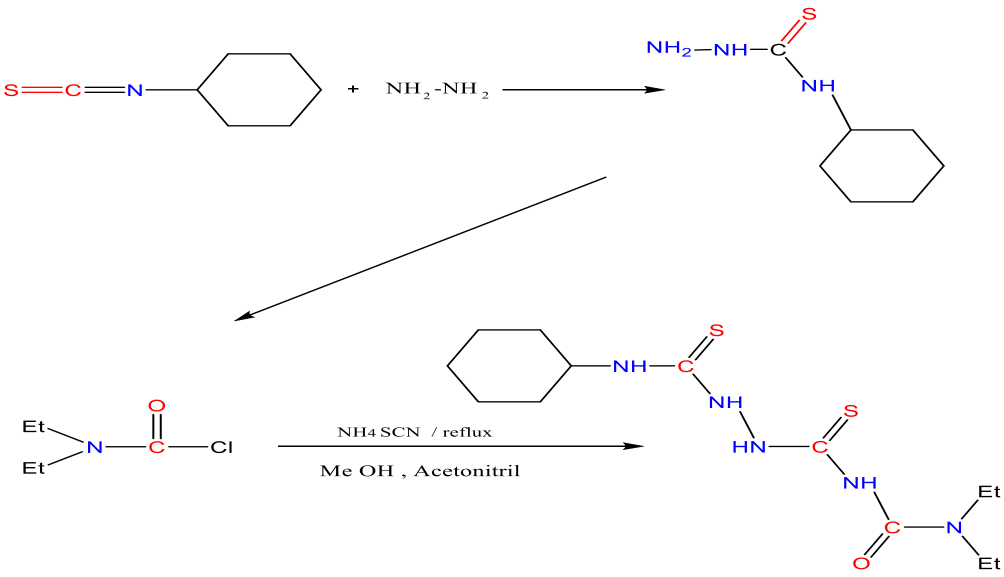
Nitrogen, oxygen, and sulfur compounds are regarded as a significant classes of organic chemicals that have been crucial to the advancement of organic chemistry [1], inorganic chemistry, and complex chemistry [2], Researchers have investigated many industrial methods to improve the production and stability of these compounds. They have played an important and crucial role in the development of chemistry, especially coordination chemistry [3]. and their potential applications in medicine, analytical chemistry, and the environmental spectrum [4]. Semicarbazide derivative ligands have biological properties including antiviral [5], antibacterial, bactericidal, antioxidant [6], and antiparasitic [7] Activities. The biological properties of semicarbazides and thio semicarbazides of ten correlates with the coordination of temptations. Second, coordination alters lipophilicity and modulates the rate of entry into the cell [8].

Semicarbazide softer varied binding mechanisms, a broad range of structural alternatives, and biological applications make it one among the most prevalent nitrogen/oxygen compounds. [9]. The importance of these compounds is under scored by their use as chelating agents that in treat with transition elements [10]. Semicarbazide transition metal complexes have been extensively researched due to their analytical applicability and excellent coordination capacity [11]. The chemistry of heterogeneous organic compounds has been and remains one of the most influential areas in organic chemistry. The importance of its physiological compounds in industrial and periodic chemistry remains heterogeneous, as they provide researchers with new compounds with many applications [12].

2. METHOD

2.1 Synthesis of; N^1-cyclohexyl-N^2-(dietheylcarbamoyl) hydrazine-1,2-bis(carbothioamide) (L)

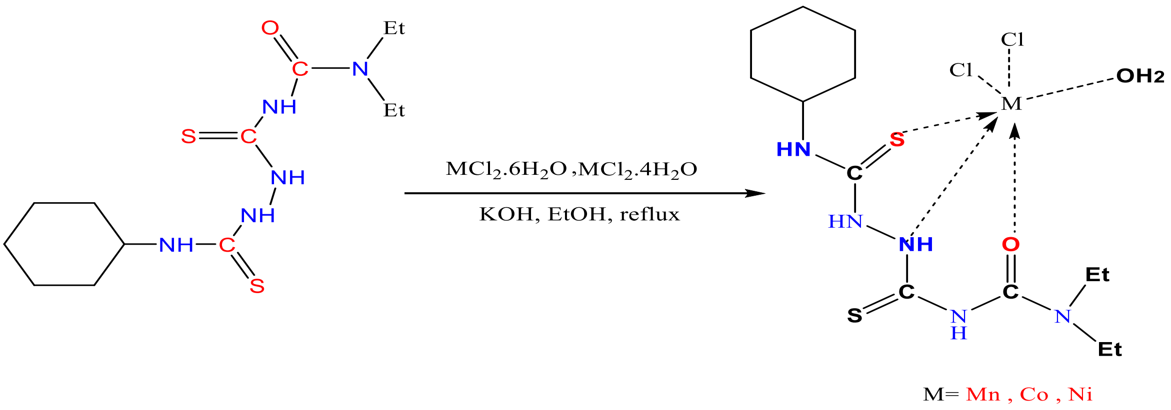
The ligand was prepared following a reported method ,involving a two-step synthesis process. The first step included the preparation of N- cyclo hexyl hydrazine carbothioamide. It was achieved from the reaction of (0.710 g, 10 mmol) of Cyclo hexyl isothiocyanate and (0.5 g, 10 mmol) of hydrazine hydrate using methanol the reaction took place below 0℃. The second step A mixture of diethyl carbamoyl chloride (6.77g, 10mmol) and ammonium thiocyanate (3.806 g, 10mmol) in methanol (20 mL) was heated for one hour at reflux. After the reaction mixture had reached room temperature, it was filtered out. The filtrate was mixed with N-cyclo hexyl hydrazine carbothioamide (1.03g, 10 mmol) in 20 mL of methanol, and the combination refluxed for two hours. The resulting white material, known as Scheme 1, was recovered by filtration, washed with 10 mL of methanol, and dried in a desiccator on anhydrous silica gel after chilling.



Scheme 1. Synthetic route of ligand

2.2 Synthesis of complexes

N^1-cyclohexyl-N^2-(dietheylcarbamoyl) hydrazine 1,2-bis(carbothioamide)(L) (0.67g 2mmole) dissolved in methanol (10mL) was combined in a 100mL round-bottom flask. The L solution was gradually mixed with a drop wise addition of KOH (0.011g, 2mmol) diluted in 10mL of ethanol. An ethanolic solution (10 mL) of the title metal ions MCl2.6H2O (2 mmol) (M(II)=Mn, Co, or Ni) was then added drop wise to the mixture after it had refluxed for one hour. After two hours of reflux, the resulting colorful solution was cooled to room temperature. Following filtration, the metal complexes were gathered and allowed to air dry (Scheme 2).



Scheme 2. Synthetic route of complexes

3. RESULTS AND DISCUSSION

3.1 FT-IR spectrum of L

The synthesized compounds' solid state infrared spectrum (Figure 1) was recorded between 4000-370 cm^{-1}. Table (1) displays the ligand's primary FTIR bands. Thiocimecarbazide ligand's FTIR spectra revealed bands at around 3280-3163, 894, and 825 cm^{-1}, which were attributed to v(N-H), v(N-H), v(C=S) thiocimecarbazide and (C=S) thiocyanate, respectively [13]. v(C-N) and v(N-N) were identified as the sources of bands found at 1103 and 979 cm, respectively. The band appears at 1620cm^{-1} attributed to v(C=O).

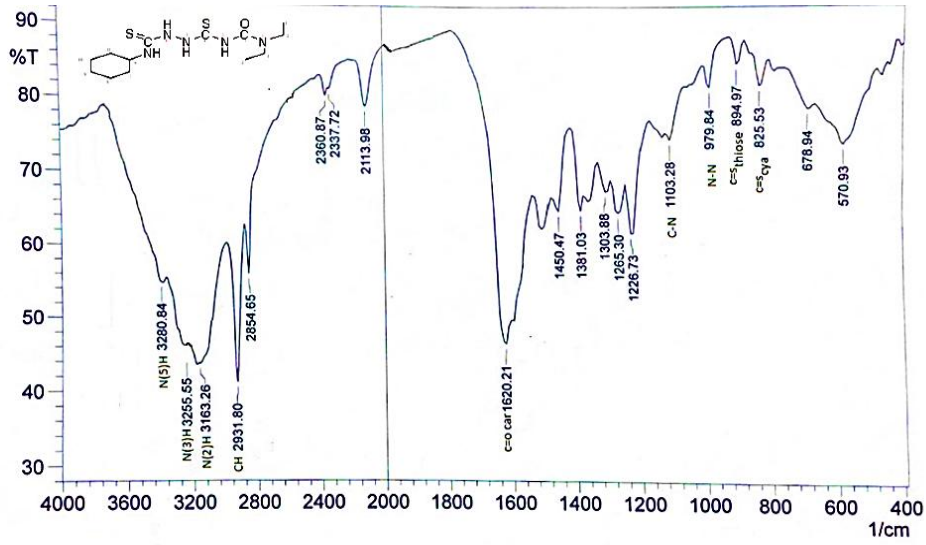


Figure 1. FTIR spectrum of N^{1}-cyclohexyl-N^{2}-(dietheylcarbamoyl) hydrazine-1,2- bis(carbothioamide)((L)

3.2 UV-Vis Spectrum of Ligand

The UV spectrum for L (Figure 2) showed a high-intensity absorption peak at 253 nm, attributed to the ligand field $\pi \rightarrow \pi^*$ transitions [14,15].

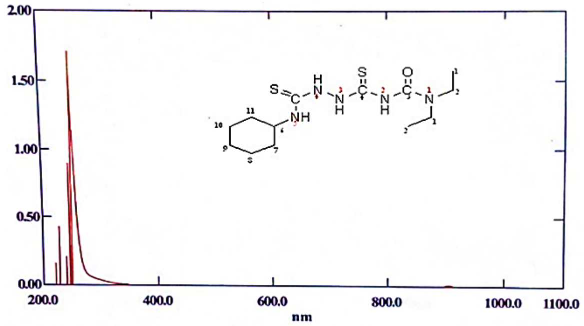


Figure 2. Electronic spectrum of Ligand L in DMSO Solvent

a. Nuclear Magnetic Resonance (NMR) spectra of Ligand

The ^{1}HNMR spectra for L was recorded in DMSO-d6 using tetramethyl silane as an internal standard, Figure 3 displays the ^{1}H-NMR spectra of N^{1}-cyclohexyl-N^{2}-(dietheylcarbamoyl) hydrazine-1,2-bis(carbothioamide). The singlet peak at (10.50 ppm) equivalent to proton is related to [1H, N (2)H, s]. A singlet peak at (10.03 ppm) is equivalent to one proton attributed to [1H, N (5)H, s][16]. The singlet peak at (5.93 ppm) equivalent to two protons is related to [2H, N (3,4) H, s] [17]. The quartet peak at (5.03 ppm) is associated with [4H, C (2,2) H, q], J=12 MHz][18]. The chemical shift related to H-aliphatic was detected as multiple at (2.72-2.09) which is due to [11H, C(6-11)-H aliphatic] and the chemical shift was found as a triplet at 1.17 ppm, attributed to [6H, C (1,1)-H aliphatic=16MHz][19]

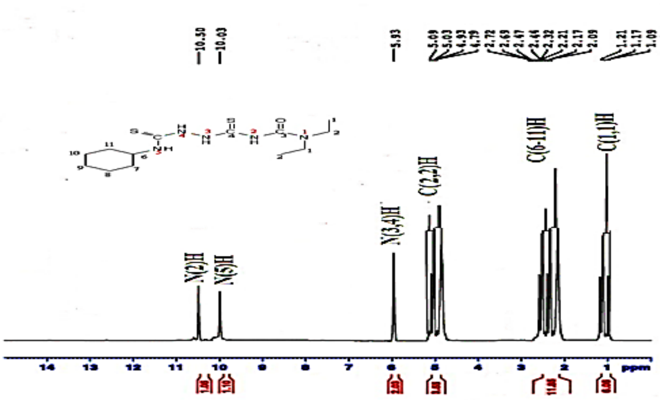


Figure 3. ^{1}H NMR of N^{1}-cyclohexyl-N^{2}-(dietheylcarbamoyl) hydrazine-1,2- bis(carbothioamide)((L)

The ^{13}C NMR spectrum of the ligand in DMSO-d6 is shown in Figure 4. The spectrum of L showed downfield shifts at 184.9 and 180.31 ppm assigned to (C=S) for thiocimecarbazide and thiocyanate, respectively. The slight differences of the chemical shifts of these peaks in the spectrum of ligand are probably due to different environments around (C=S. The spectrum showed signal at 154.99 ppm, attributed to (C=O) group. The aliphatic carbons signals of the ligand appeared as expected at the range (15.09-55.13) ppm respectively. All 13C-NMR chemical shifts positions of ligand L are in agreement with values reported of other thiocimecarbazide ligands [20].

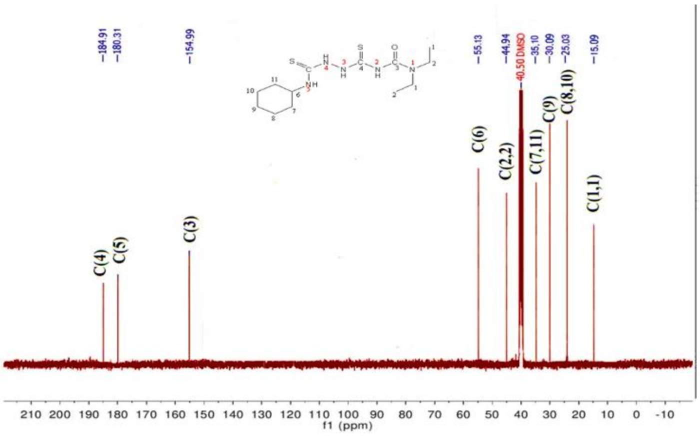


Figure 4. ^{13}C NMR spectrum of N^{1}-cyclohexyl-N^{2}-(dietheylcarbamoyl) hydrazine-1,2- bis(carbothioamide)((L)

b. Mass Spectroscopy of L

The electrospray (+) mass spectroscopy of L Figure 5 shows the molecular ion peak at m/z=331.9 amu (17%) this peak is related to (L $^{+}$) calculated (331.15) amu for ($C_{13}H_{25}N_5OS_2$)

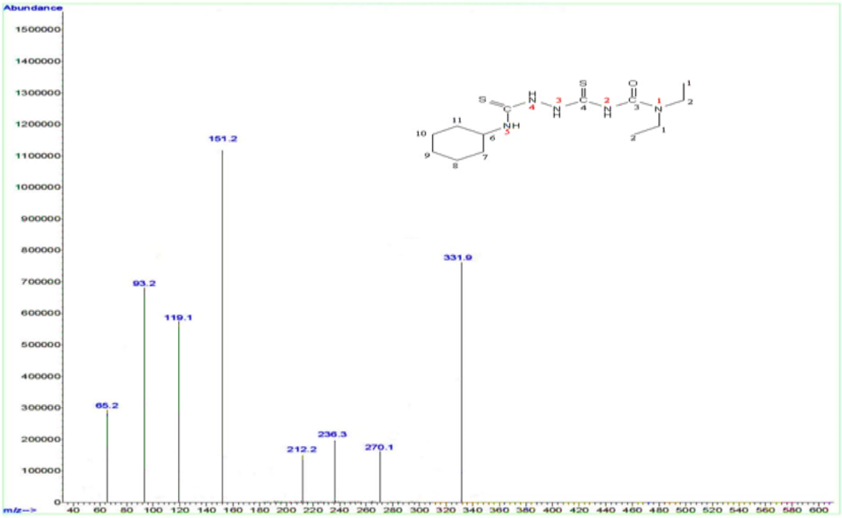


Figure 5. The electrospray (+) mass chart of L

3.5 Diagnostics of the complexes

3.5.1 FT-IR spectra of the prepared complexes

The FT-IR spectra of C1, C2 and C3 are displayed in Figures 6–8, and Table (1) lists the various bands. The FTIR spectra of Mn (II), Co (II), and Ni (II) complexes revealed peaks corresponding to v(O-H) at (3640 – 3516 cm^{-1}) [21–22]. The spectra showed bands that were identified as belonging to the v(N-H) in the range 3379-3163 cm^{-1}. These bands were moved to a higher wavenumber in comparison to the range 3280–3163 cm^{-1} in the free ligand's spectrum [23]. Table 1. In the free ligand, figure 1, a band associated to the v(C=O) was found at 1620 cm^{-1}. This band appeared at the lower shift in complexes C1-C3 at the range (1589-1519 cm^{-1}) that is related to the involvement of these moieties in the coordination of the metal centre [24]. The v(C=S) thiocimecarbazide group was identified by bands at the range (979-887) cm^{-1} in the FT-IR spectra of complexes C1, C2, and C3, these bands shifted to higher and lower wavenumbers compared to that at 894 cm^{-1}in free ligand, indicating that this group was involved upon coordination [25]. The metal complex spectra showed extra peaks between 600 and 200 cm^{-1} that were not presented in the ligand spectrum. Spectra detected peaks correlated to (M-O) (M-N) and (M-S) in the range (563,540), 478-462) cm^{-1} and 401-385) cm^{-1}, respectively [26]. Peaks measured at the rang (285- 208) related to v(M-Cl) [27–29].

Table 1. The L and their complexes most significant characteristic bands in the FT-IR spectrum

NO	*V*(O-H)	*V*(N_5-H)	*V*($N_{3,4}$-H)	*V*(N_2-H)	*V*(CH) Aliph	*V*(C-O) Carba moyl	*V*(C=S) Thiosemi carbazide	*V*(C-N)	*V*(N-N)	*V*(C=S) cyanate	*V*(O-H)	*V*(M-O)	*V*(M-N)	*V*(M-S)	*V*(M-Cl)
L_1	-	3280	3255	3163	2931	1620	894	1103	979	825	-	-	-	-	-
C_1	3640	3348	3302	3186	2931	1519	979	1103	1026	825	887	563	740	401	254,208
C_2	3566	3379	3255	3163	2931	1589	972	1081	1064	757	887	540	462	385	262,223
C_3	3516	3348	3271	3186	2939	1523	887	1103	1040	786	887	563	478	401	285,254

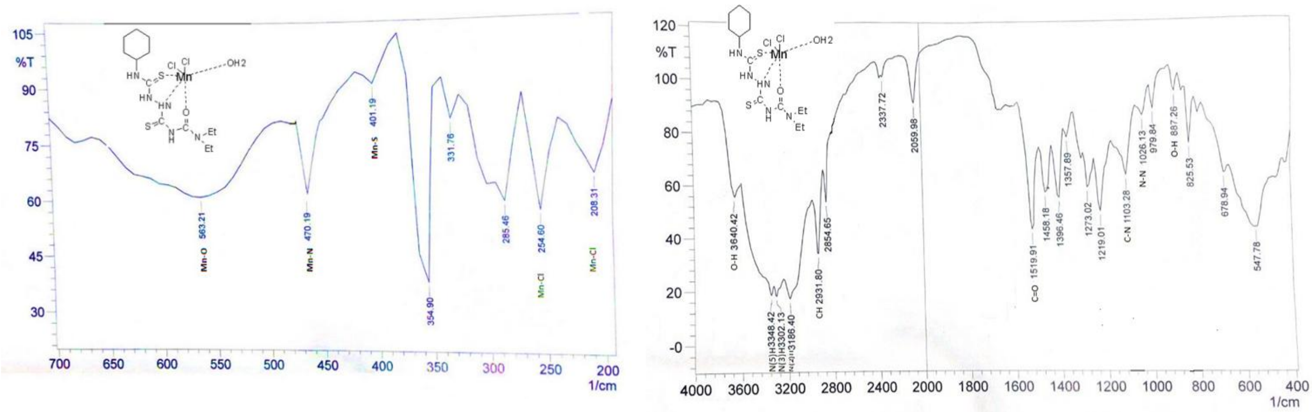


Figure 6. The Infrared spectrum of(L) $MnCl_2$ H_2O] (C1)

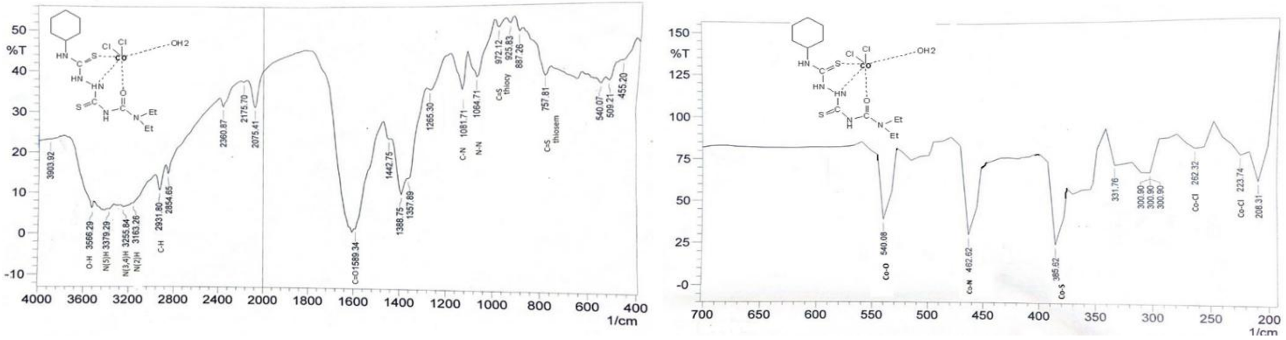


Figure 7. The Infrared spectrum of (L)[$CoCl_2$ H_2O](C_2)

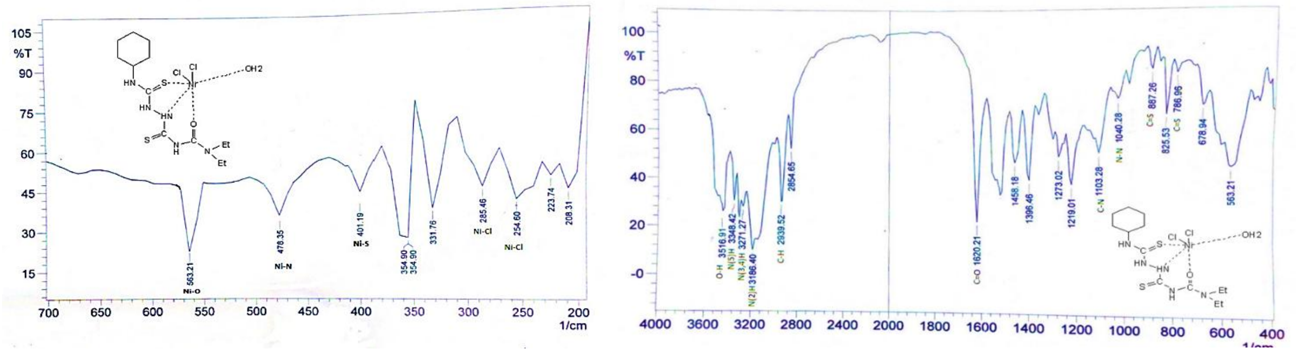


Figure 8. The infrared spectrum of [L $NiCl_2$H2O](C3)

Table 2. UV-visible spectrum of ligand and complexes

Complex	nmג	cm^{-1}ג	∑Max ($dm^3mo^{-1}cm^{-1}$)	Assignment	Suggested geometry
L	253	39525	1717	$\pi \rightarrow \pi^*$	
C_1[LMn]	255 907	39215 9718	1594 30	$\pi \rightarrow \pi^*$ $^6A_1g \rightarrow {}^4T_{1g}$	Distorted Octahedral
C_2[LCo]	273 848	36630 11792	733 20	$\pi \rightarrow \pi^*$ $^4T1g \rightarrow {}^4A_2g$	Distorted Octahedral
C_3[LNi]	254 907	39370 11025	1115 2	$n \rightarrow \pi^*$ $^3A_2g \rightarrow {}^1Eg$	Distorted Octahedral

3.5.2 U.v -Vis Spectra of the complexes

The electronic absorption spectra of complexes in DMSO solutions (con. = 1x 10-3 M) were determined, Figures (6), (7) and (8) display the electronic spectra of the metal complexes of Mn (II), Co (II) and Ni (II), respectively. According to Table (2), the spectra of L Figure showed peak at 253 nm that were associated with the π → π*, this peak was shifted to the rang (273-254nm) in the spectra of the complexes C1, C2 and C3 [30]. A distorted octahedral geometry was revealed by the band at 907 nm in the d-d region of the Mn (II) complex, which was attributed to 6A1g → 4T1g [31]. A deformed octahedral geometry surrounding the metal center was corroborated by the C2 complex peak at 848 nm, which was assigned to 4T1g → 4A2g[32]. The C3 spectrum's peak at 907 nm was identified as 3A2g →1Eg, indicating a deformed octahedral geometry surrounding the metal center [33].

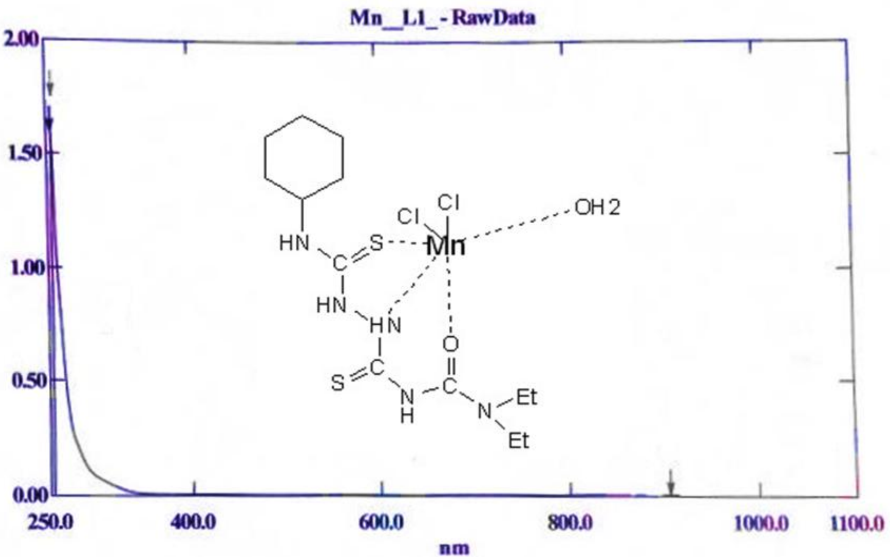


Figure 9. Electronic spectrum of [(L)MnCl_2H_2O] (C1)

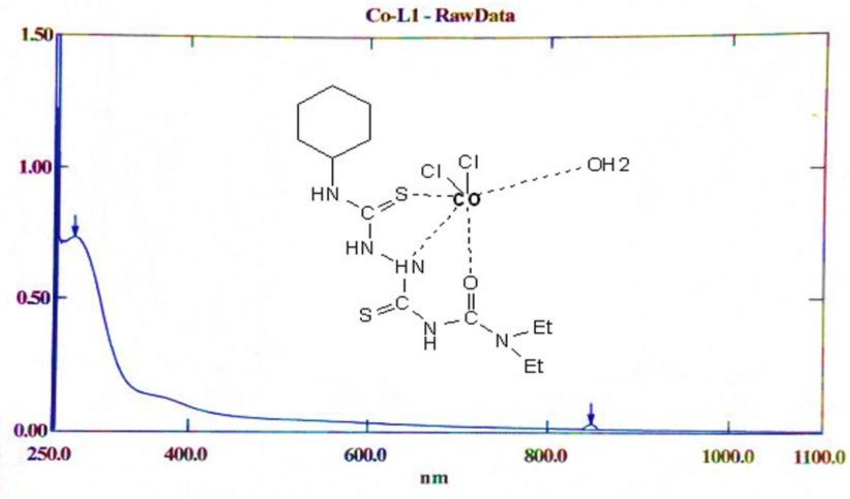
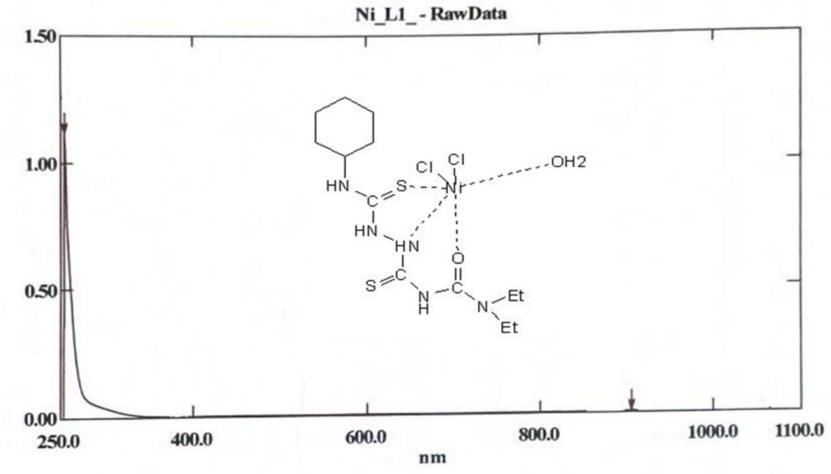


Figure10. Electronic spectrum of [(L)$CoCl_2H_2O$] (C2)



Figur11. Electronic spectrum of [(L)$NiCl_2H_2O$] (C3)

4. Biological Activity

The antimicrobial activity of the ligand and its metal complexes was tested against two bacterial isolates: Escherichia Coli (Gram-negative) and Staphylococcus aureus (Gram-positive). A disc diffusion approach was used to carry out the antibacterial activitis. [34]. The 50 mg/ml, 25 mg/ml, 12.5 mg/ml, and 6.25 g/ml doses were tested against pathogenic bacterial strains in the in vitro antibacterial investigations. Penicillin (10 micrograms/disc) served as the negative control and Cefotaxime (CTX 10 µ g) as the positive control. Filling the sanitized Petri dishes with the nutritional agar solution created the agar medium. The nutrient agar was covered with the cultures of all the bacteria that had been grown in the nutrient broth and incubated for 24 hours at 37°C. Cut from Whatman No. 1 filter paper, 5mm diameter discs were autoclaved at 15 psi for 15 minutes in an aseptic environment. The chemical will permeate into the agar when a filter paper disc impregnated with the chemical is placed on it. The chemical in the agar will only be distributed around the disc by diffusion. If the disc is chemically susceptible, the extent of the chemical infiltration surrounding it will depend on the chemical's solubility and molecular size. ‘Zone of inhibition' refers to the region where growth is inhibited. To create a consistent suspension, a loopful of the test organism's overnight slant culture was injected with 5µL of sterile physiological saline. To create a homogeneous grass culture, this suspension culture was surface distributed over a nutrient agar plate using a sterile cotton swab. On the swabbed surfaces of the plates (five discs per plate), the test sample discs that had been produced as previously indicated were inserted. After a 24-hour incubation period at 37°C, the plates were examined to look for zones of inhibition surrounding the discs.

The first concentration of Escherichia coli complexes C1 and C2 shown a greater biological activity, but the third concentration of ligand L and C2 exhibited no action against the tested bacteria. In the first, second, and third concentrations, L showed little effectiveness.

The complexes (C3) in the first concentration 50 showed good activity against this type of bacteria, (C1) in the third concentration showed no activity against this type of bacteria, and the remaining complexes and the free ligand showed medium activity against Staphylococcus aureus, according to the biological activity of the tested compounds against this type of bacteria This particular kind of bacterium After a 24-hour incubation period at 37°C, the plates were examined to look for zones of inhibition surrounding the discs. The first concentration of Escherichia coli complexes C1 and C2 shown a greater biological activity, but the third concentration of ligand L and C2 exhibited no action against the tested bacteria. In the first, second, and third concentrations, L showed little effectiveness. The complexes (C3) in the first concentration 50 showed good activity against this type of bacteria, (C1) in the third concentration showed no activity against this type of bacteria, and the remaining complexes and the free ligand showed medium activity against this type of bacteria, according to the biological activity of the tested compounds against Staphylococcus aureus table(3). [38-37] .

Table 3. Biological Activity values of some prepared compounds against isolates of pathogenic bacteria

Compound	Concentration (µg/ml)	Escherichia coli (E)	S.aureus (S)
L^1	100	n.a	n.a
	50	n.a	n.a
	25	n.a	n.a
C1	100	n.a	n.a
	50	n.a	n.a
	25	n.a	n.a
C2	100	16	13
	50	13	11
	25	0	0
C3	100	24	18
	50	17	16
	25	14	12

n.a= No activity

Figure 12. Antiviral activity of the compounds against Escherichia aureus of the L ligand and its complexes [C1, C2, C3]

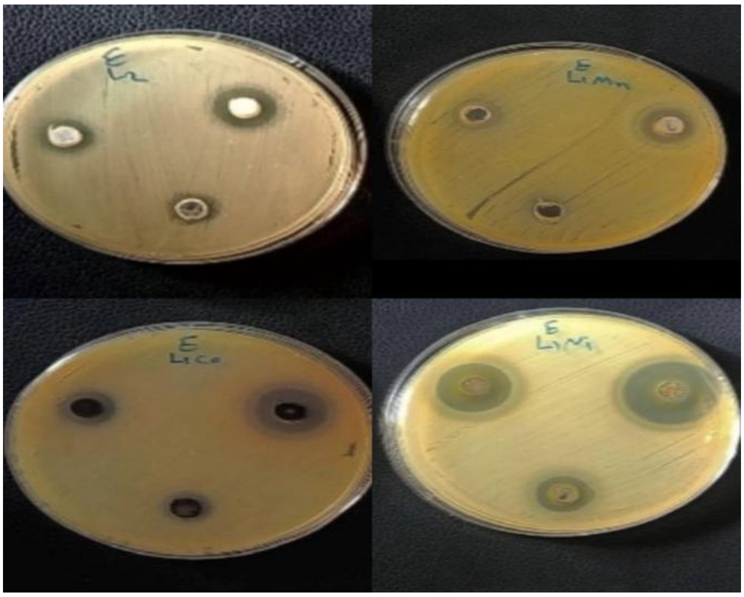


Figure 13. Antiviral activity of the compounds against Staphylococcus Escherichia coli of the L ligand and its complexes [C1, C2, C3]

5. CONCLUSION

The reaction of 2-(dimethylcarbamothioyl)-N-ethylhydrazine-1-carboxamide (L) with metal ions—Mn(II), Co(II), and Ni(II) resulted in the formation of the required complexes with octahedral geometry around ion complexes. Physical, chemical, and spectroscopic methods were used to investigate the mode of bonding and overall structure of the ligand and it's complexes, , which have been synthesized and characterized by spectroscopic techniques, The mass spectrum, ^{1}H-NMR, ^{13}CNMR, F.T-I.R. measurements, U.V-Vis . The biological activity testing was done on the ligand and its complexes bio against (G+ and G-) microorganisms.

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