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Synthesis, Characterization and Biological activity of new Metal Complexes Derived From 4-Phenyl-3-thiosemicarbazide

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ABSTRACT

The study involved the synthesis and characterization of the new compound 4-methoxy-N-(2-(phenylcarbamothioyl) hydrazine-1-carbonothioyl) benzamide (L). By combining the title ligand with metal ions of manganese (II), cobalt (II), and nickel (II), three transition metal complexes were synthesized. This reaction occurred through the combination of metal and ligand in a mole ratio of one-to-one utilizing ethanol as the solvent. The complexes generated have a chemical formula as follows: [$LMnCl_2.H_2O$], [$LCoCl_2H_2O$], and [$LNiCl_2.H_2O$]. A variety of physicochemical techniques have been utilized to demonstrate the entity of the predicted structure of the ligand and its metal complexes. including Fourier transform infrared spectroscopy, UV-Vis spectroscopy, (^1H and ^{13}C)-NMR, elemental analysis (C, H, N, S), content of chloride, content of metal, m.p., the molar conductivity measurements, and also magnetic susceptibility. Based on spectral and analytical data, the isolation complexes for the manganese (II), cobalt (II), and nickel (II) exhibit a six-coordinate with a distorted octahedral geometry. The antibacterial activity of the complexes was investigated against (G+) and (G-) bacteria.

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1. INTRODUCTION

Recent years have seen an increase in interest in the coordination chemistry of heteroleptic complexes due to their intriguing biological activities. In the fields of medicinal and bioinorganic chemistry, the coordination of metal based on thiosemicarbazone derivatives has become more significant [1]. The ligands of thiosemicarbazone derivative show biological activities including antiviral [2], antibacterial, antioxidant, fungicidal [3], and anti-parasitic effects [4]. Semicarbazone and thiosemicarbazone biological characteristics are frequently correlated with metal ion coordination. Secondly, coordination alters lipophilicity, hence regulating the pace of cellular entrance [5]. Researchers have investigated these species and their metal complexes in great detail, mostly because of their potential biological characteristics [6]. Among the most common nitrogen/oxygen compounds, semicarbazides offer a diversity of binding mechanisms, a wide range of structural options, and biological uses [7,8, 9]. The importance of these compounds is emphasized by using them as chelating agents that interact with transitional elements [10].

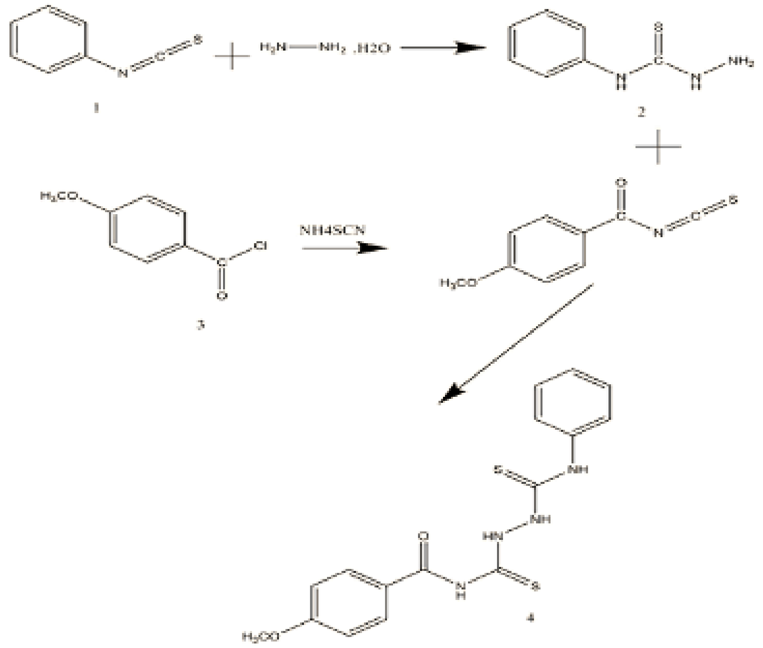
Different reaction conditions, the type of substituent on the carbonyl molecule, metal salts, and the nature of counter ions all have an impact on the coordination modes that semicarbazides exhibit with metal ions [11]. Semicarbazides are believed to possess diverse structural characteristics, and their metal complexes are considered to exhibit more physiological activity than the free ligands [12, 13]. Thiosemicarbazone transition metal complexes have been widely investigated due to their coordination capabilities and analytical uses [14,15]. This stady represents the preparation and spectral characterization of the new thiosemicarbazide ligand namely; 4-methoxy-N-(2-(phenylcarbamothioyl)hydrazine-1-carbonothioyl)benzamide (L) and its metal complex ions (manganese, cobalt, and nickel). Moreover, Ligand and its metal complexes' antibacterial efficacy is investigated.

2. Materials and methods

All of the Aldrich-sourced reagents were utilized without further purification. Prior to being utilized in the preparation, the solvents were dried in accordance with established protocols.

Synthesis of; 4-methoxy-N-(2-(phenylcarbamothioyl)hydrazine-1-carbonothioyl)benzamide L

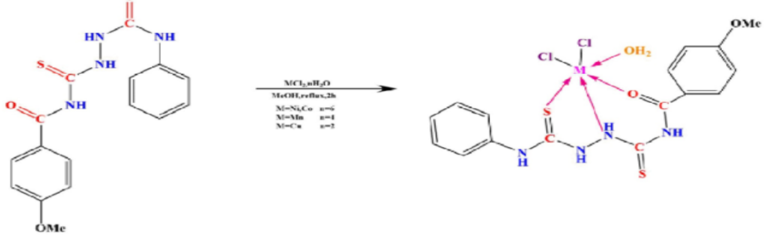
The ligand (4) was produced according to the procedure described in [16] in a two step. The first step included the preparation of 4-phenyl-3-thiosemicarbazide (2).It was achieved from the reaction of (1.35 g, 10 mmol) of phenylisothioyanate (1) with (0.5g, 10 mmol) of hydrazine hydrate using ethanol. The reaction took place below 0°C. The second step is a mixture of 4-methoxybenzoyl chloride (1.7g, 10mmol) and ammonium thiocyanate (0.76 g, 10mmol) in acetonitrile (40ml) It had been heated at reflux for one hour. At room temperature, the reaction mixture was subsequently cooled and then filtered. After adding 4-phenyl-3-thiosemicarbazide (1.67g, 10 mmol) in 20 ml of acetonitrile to the filter, the mixture was refluxed for two hours. The title chemical was obtained by filtering the white solid that had formed after cooling, washing it with (10mL) of acetonitrile, and drying it in a desiccator on anhydrous silica gel, Scheme 1.



Scheme 1. Synthetic route of ligand L

2.1 Synthesis of complexes

In a 100mL of a round-bottomed flask 4-methoxy-N-(2-(phenylcarbamothioyl)hydrazine-1-carbonothioyl)benzamide L (0.72g, 2mmole) dissolved in EtOH (10ml). A solution of KOH (0.11g, 2mmol) dissolved in EtOH (10ml) has been added to the L solution dropwise. The resulting mixture was refluxed for 1h and then an ethanolic solution (10ml) of the title metal ions $MCl_2.nH_2O$ (2 mmol) (M(II)=Mn, n=4. Co or Ni, n=6) was added dropwise. The colored solution that resulted was left to reflux for two hours and thereafter left to cool to room temperature. After filtering, 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 spectrumum of (L)

Solid state infrared of the prepared ligand L, Figure 1. was recorded in the range of 4000-370 cm^{-1}. The main FTIR bands of the ligand are presented in Table 1. In the FTIR spectra of thiosemicarbazone ligand, bands of about (3430-3152) cm^{-1}, 1614 cm^{-1} and 780 cm^{-1} were observed, assigned to ν(N-H), ν(C=O) carbamoyl and ν(C=S) thiosemicarbazide, respectively [15, 16,17]. The bands found at 1386 cm^{-1} and 1160 cm^{-1} were assigned to ν(C-O) and ν(N-N), respectively. ν(C=S) thiocyanate appeared at 721 cm^{-1}[18].

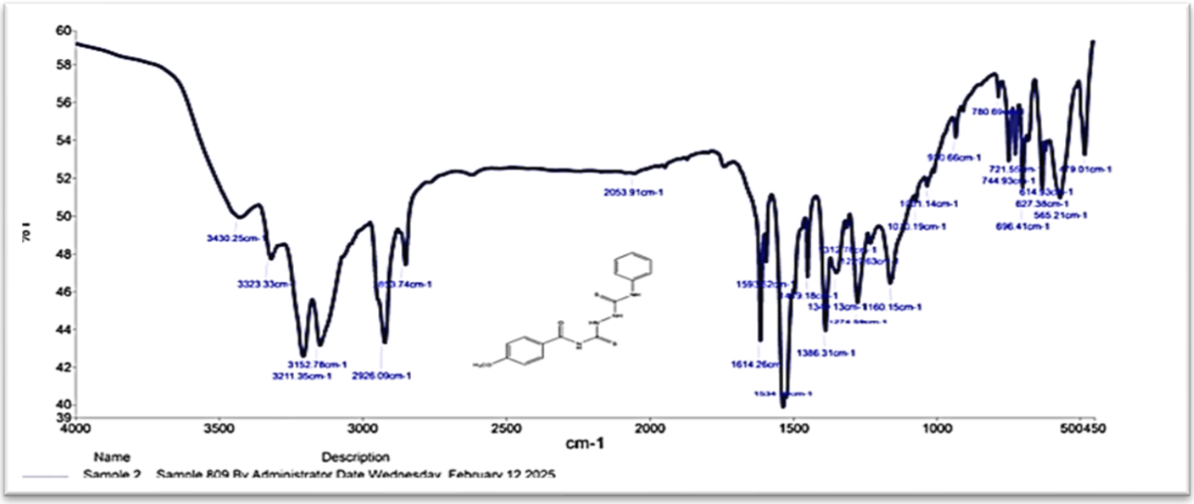


Figure 1. FTIR spectrum of 4-methoxy-N-(2-(phenylcarbamothioyl)hydrazine-1-carbonothioyl)benzamide *(L)*

3.2 Diagnostics of the complexes

T-IR spectra of the prepared complexes

Figure 2 show the FT-IR spectrum of complexes 1 and 2 while Table 1 includes the assignment of the distinct bands. Complexes spectrum of manganese (II), cobalt (II), and nickel (II) showed peaks related to ν(O-H) stretches around 3436, 3580 and 3595 cm^{-1}, respectively. These peaks were attributed to the OH of the aqua molecule [19, 20 , 21, 22, 23,24]. The spectra showed bands that were ascribed to the ν(N-H) in the 3435–3111 cm^{-1} region. These bands had been shifted to lower wavenumberes compared with that observed in the range 3430-3111 cm^{-1} in the spectrum of the free ligand, Table 1. A band detected at 1614 cm^{-1}in the free ligand, figure1, is related to the ν(C=O) carbamoyl. In complexes C_1–C_3, this band was visible at a lower shift in the range 1547–1545 cm^{-1}.

The shifting of the carbonyl bands indicates the involvement of these functional groups in the coordination to the metal centre [25,26,27,28,29]. The FT-IR spectra of complexes C_1,C_2 and C_3 revealed bands that are assigned to the ν(N-N) group in the range of (1188-1190) cm^{-1}. The involvement of this ν(N-N) group following coordination was confirmed by the change of these bands to a higher wavenumber when compared to that observed at 1160 cm^{-1} in the ligand's infrared spectrum [30,31,32].

The FT-IR spectra of complexes C_1,C_2 and C_3 revealed in the 751–749 cm^{-1} region that were assigned to the ν(C=S) thiosemicarbazide group. The involvement of this ν(C=S) thiosemicarbazide group throughout coordination was confirmed by the shift of these bands to a higher wavenumber when compared to that exhibited at 721 cm^{-1} in the ligand's infrared spectra [30,32]. Additional peaks between 600 and 200 cm^{-1} that were absent from the ligand spectrum were discovered in the spectra of metal complexes. ν(Mn-O), ν(Co-O), and ν(Ni-O) associated peaks were found at (690) cm^{-1}, (586) cm^{-1}, and (632) cm^{-1}, respectively [33, 34]. Bands detected at 543, 532 and 486, cm^{-1} assigned to ν(Mn-N), ν(Co-N) and ν(Ni-N), respectively. Bands detected at 462, 532 and 435 cm^{-1} assigned to ν(Mn-S), ν(Co-S) and ν(Ni-S), respectively. Peaks detected at(262,235), (254,223) and (254,219)cm^{-1} were correlated to ν(Mn--Cl), ν(Co-Cl) and ν(Ni--Cl), respectively[34,35,36].

Table1. The L and their complexes' most significant distinctive bands in the FT-IR spectrum

N O	ν(O-H)	ν(N_5-H)	ν(N_4-H)	ν(N_3-h)	ν (N_2-H)	ν (C=O) carbamoyl	ν (C=O) semicarbazide	ν(C-N)	ν (N-N)	ν(c=s) thiocyanate	ν(O-H)	ν (M-O)	ν (M-O)	ν (M -N)	ν(M-Cl)
L	-----	3294	3224	3149	3093	1658	1573	1149	1075	756	-----	----	----	----	----
C_1	3520	3322	3214	3186	3080	1674	1565	1153	1086	756	972	524	474	424	262
C_2	3566	3313	3219	3132	3073	1677	1562	1180	1049	757	980	532	473	422	270
C_3	3516	3309	3227	3183	3070	1667	1560	1144	1072	756	980	534	470	385	293

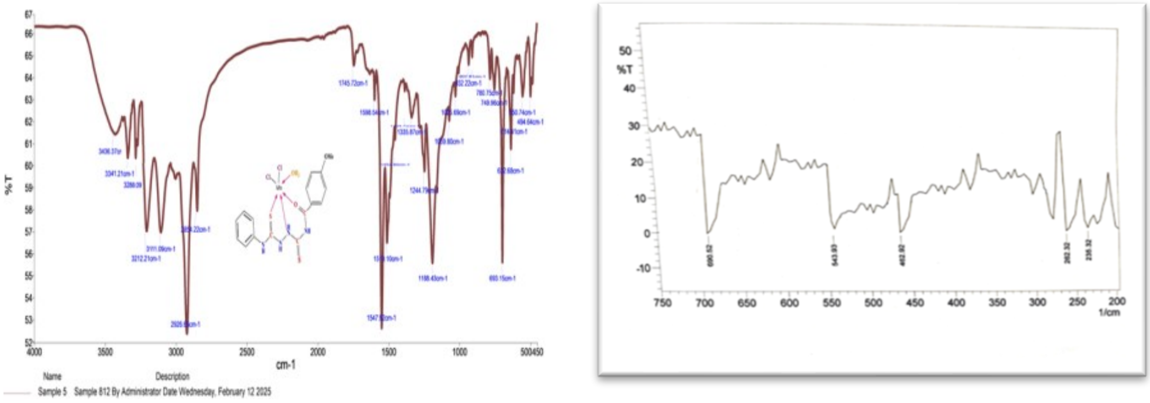


Figure 2. FTIR spectrum of [L $MnCl_2H_2O$](C_1)

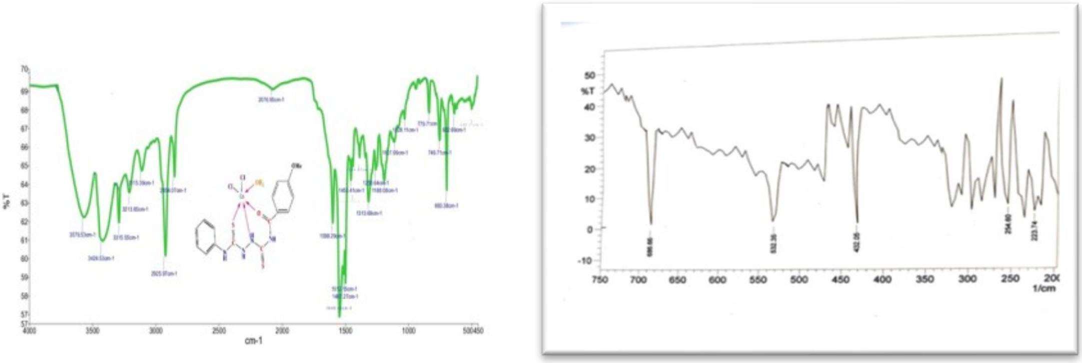


Figure 3. FTIR spectrum of ($LCoCl_2H_2O$](C_2)

3.4. U.v.-Vis Spectrum of Ligand

The UV spectrum for L, Figure 4, showed a high-intensity absorption peaks at 234nm and 298nm which are attributed to the ligand field (π →π*) and (n →π*) respectfully [19, 20].

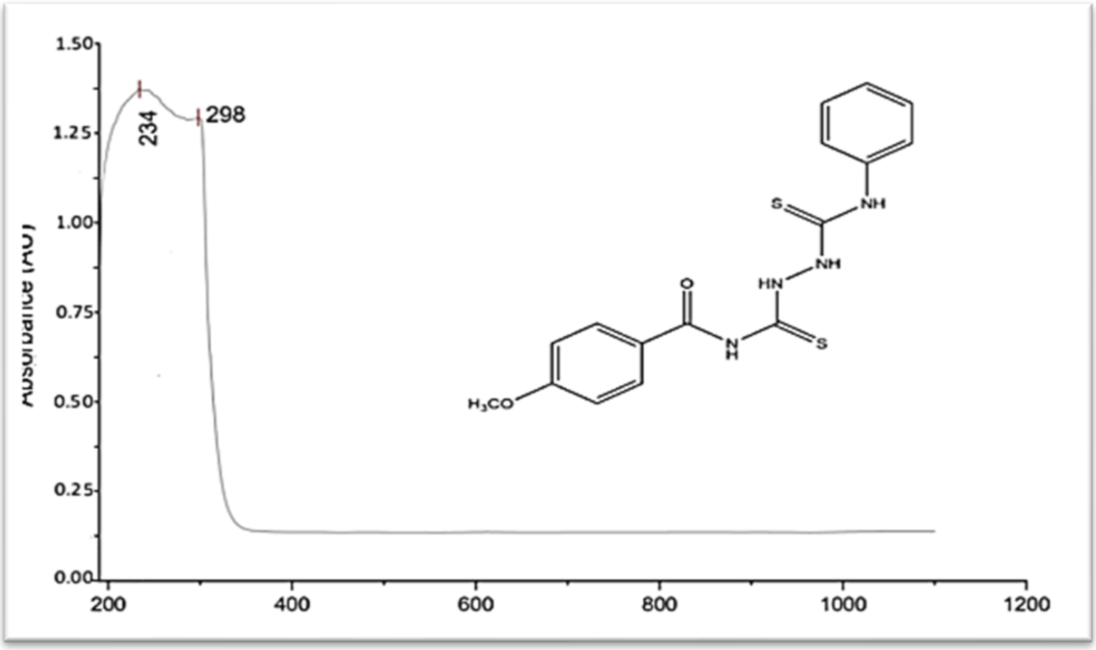


Figure 4. Electronic spectrum of Ligand

3.5 U.V.-Vis Spectra of the complexes

The electronic absorption spectrum of complexes was measured in dimethyl sulfoxide solutions (con. = 1x 10^{-3} M). The electronic spectra of the metal complexes of manganese (II) and cobalt (II) are exhibited in Figures 6 and 7, respectively. The spectra indicated peaks, (see Table 2), at the range (206-211),(256-299) nm attributed to **π→π***and **n→π*** sequentially (intra ligand) [37, 38]. In the d-d region of the Mn (II) complex, the band at 699 nm, attributed to^6A_1g →$^4Eg(D)$ indicated a distorted octahedral structure around the metal center [39,40]. In the C_2 complex peaks at 651 nm related to $^4T_1g_{(F)}$ →$^4A_2g_{(F)}$ that supported a distorted octahedral geometry around the metal center [41]. Peaks at 900 nm in the spectrum of C_3 referred to $^3A_2g_{(F)}$ →$^3T_2g_{(F)}$, establishing a distorted octahedral shape around the metal core [41,42, 43].

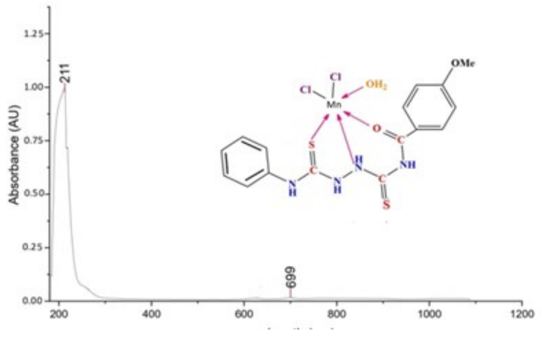


Figure 5. Electronic spectrum of [(L)MnCl$_2$H$_2$O]C1

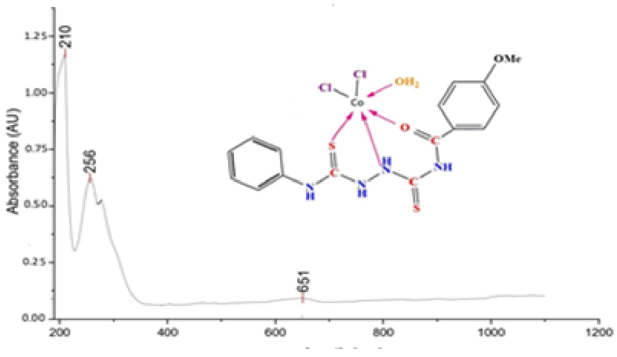


Figure 6 .Electronic spectrum of [(L)CoCl$_2$H$_2$O]C$_2$

Table 2. UV-visible spectrum of ligand and complexes

Complex	nmλ	cm^{-1}λ	ΣMax (dm^3 mo^{-1} cm^{-1})	Assignment	Suggested geometry
L	234 298	42735 33557	1350 1300	π? π* n? π*	
C_1	211 699	47393 14306	1020 62	π? π $^6A_{1g}$? ^4E_g	Distorted Octahedral
C_2	210 256 651	47619 39062 15360	1130 625 90	π ? π* n? π* $^4T_{1g(F)}$? $^4A_{2g(F)}$	Distorted Octahedral
C_3	206 299 900	48543 33444 11111	1030 960 86	π? π* n? π* $^3A_{2g(F)}$? $^3T_{2g(F)}$	Octahedral

3.6 Nuclear Magnetic Resonance (NMR) spectra of Ligand

The ^{1}H-Nuclear Magnetic Resonance spectra of **N-cyclohexyl-2-((4-methoxybenzoyl)carbamothioyl) hydrazine-1-carboxamide** (L) in DMOS-d^6 solvent are shown in Figure 7. A singlet peak at (10.57 ppm) is equivalent to one proton due to [s,1H, N (1)H]. A singlet peak at (10.07ppm) equivalent to a proton is due to [s,1H, N(4)H] [44]. A singlet peak at (5.03ppm) this is related to protons are caused by [s,2H, N(2,3)H] [45]. The chemical shift due to the H- $_{aromatic}$ was found a multiple at (7.72-7.09) ppm which is due to [9H, C (3,4,6,7,12-16)-H $_{aromatic}$,m]. The chemical shift at (2.09ppm) which is due to protons of the methyl groups [3H, C(1)-H $_{aliphatic}$, s])

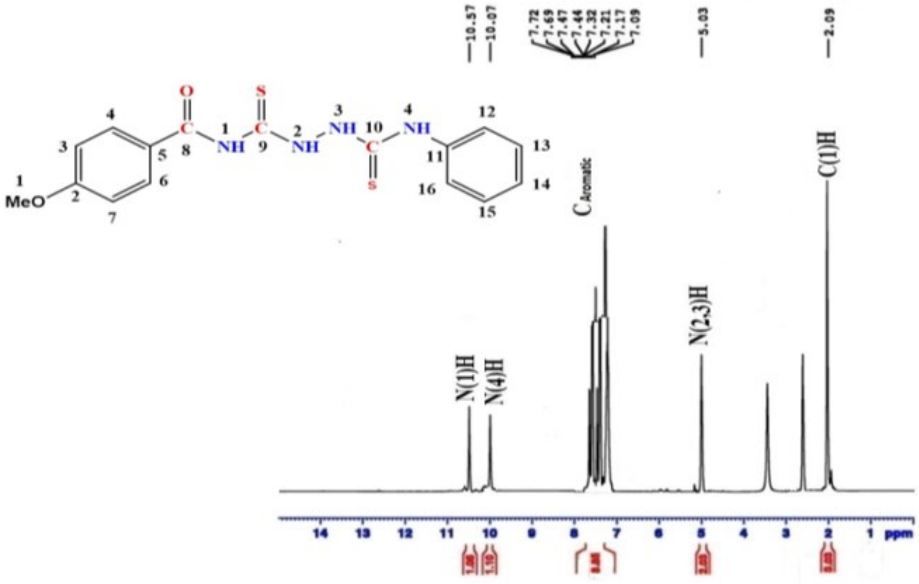


Figure 7. ^{1}H NMR of N-cyclohexyl-2-((4-methoxybenzoyl) carbamothioyl) hydrazine-1-carboxamide(*L*)

The ^{13}C Nuclear Magnetic Resonance spectra of the ligand in the dimethyl sulfoxide-d^6 solution are showed in Figure 8. The spectrum showed the compound's appropriate resonances. The spectrum indicated C_9 and C_{10} (C=S) signals at 185 and 180 ppm, attributed to the thiocyanate and thiosemicarbazide groups. The ^{13}C NMR spectrum of the ligand L^1 showed downfield shifts at 158 and 153 ppm assigned to C_8 (benzoyl carbon) (C=O) and (C_2-O)) respectively [45]. The aromatic carbon signals of (C_3-C_7) and(C_{11}-C_{16}) appeared as expected at the range (139-110) ppm. The aliphatic carbon signal of the(C_1) appeared at (49) ppm. All ^{13}C-NMR chemical shift positions of ligand L corroborate the results obtained from many different thiosemicarbazide ligands.

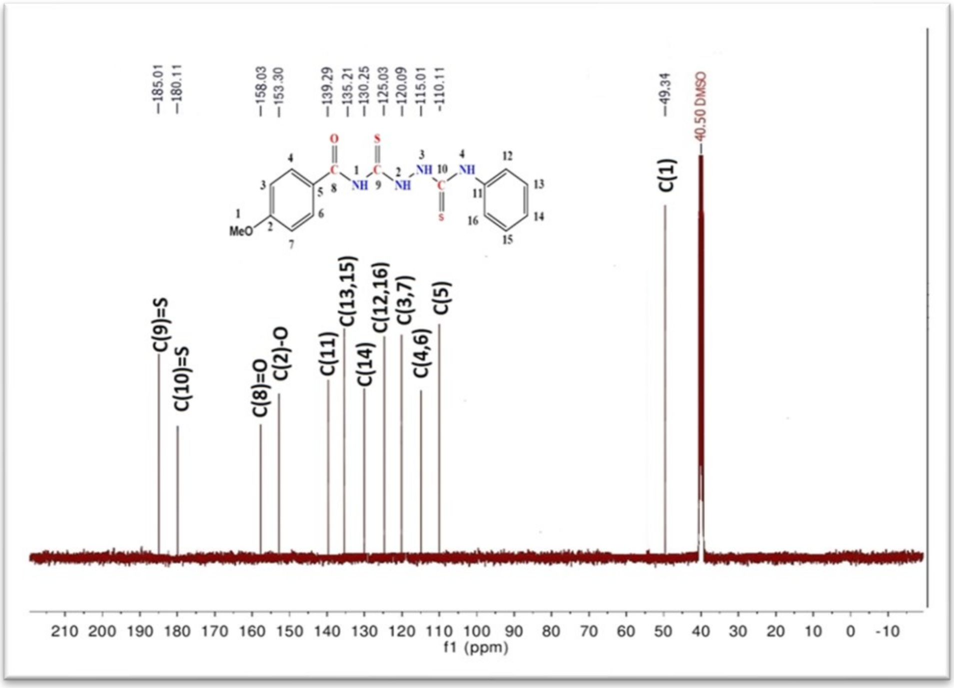


Figure 8. ^{13}C NMR spectrum of N-cyclohexyl-2-((4-methoxybenzoyl)carbamothioyl) hydrazine-1-carboxamide (L)

3.7 Mass Spectrum of (L)

The electrospray (+) mass spectroscopy of L. Figure 9 shows the molecular ion peak at m/z=360.2 amu (70%) this peak is related (360.45) amu for ($C_{16}H_{16}N_4O_2S_2$)

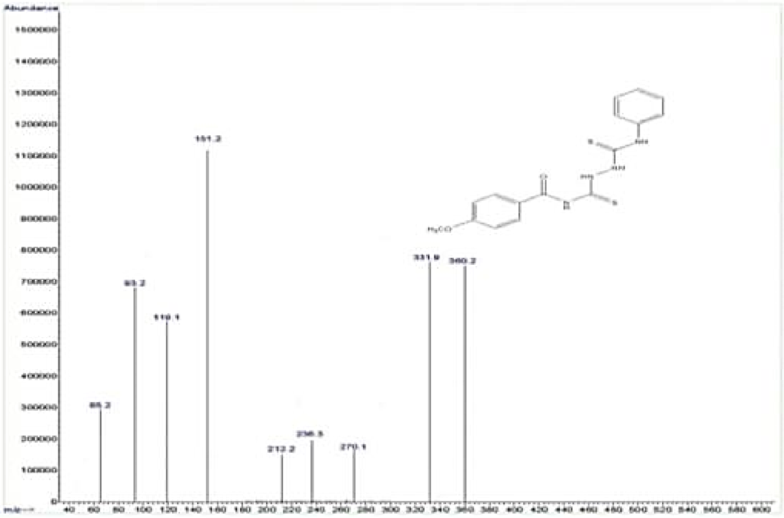


Figure 9. the electrospray (+) mass chart of L

3.8 Biological Activity

The microbiological activity of the L and its metal complexes was investigated against two specific types of bacteria: (G+) *(Staphylococcus aureus*) and (G-) (*Escherichia Coli)* Table 3. The antibacterial activity was performed using a disc diffusion method. The *in vitro* antibacterial studies were conducted at a range of concentrations of 10^{-5}mg/10ml, 10^{-4}mg/10ml and 10^{-3}mg/10ml against pathogenic bacterial strains. Tetracycline (10 µ g/disc), was employed as both a positive and negative control.

The nutrient agar solution was poured into the Petri plates that had been sterilized to create the agar medium. For a full day of incubation at 37°C, all bacteria were cultivated in the nutrient broth and subsequently spread out over the nutritional agar surface. Utilizing Whatman No. 1 filter paper, 5 mm diameter discs were cut out and autoclaved for 15 minutes at 15 psi in an aseptic environment. A chemical that has been put into a filter paper disc will permeate into the agar from the filter paper. Only the area around the disc will have the chemical in the agar due to diffusion. If the disc is sensitive to the chemical, the extent of the region of chemical infiltration around it will depend on the chemical's solubility and molecular size. The 'zone of inhibition' is the region that doesn't expand. To create a uniform solution, 5µL of 140 sterile physiological Salines was injected with a loopful of an overnight slant culture of the test organism. To generate a consistent grass culture, this suspension culture was surface dispersed over a nutrient agar plate utilizing a sterile cotton swab. Using sterile forceps, the test sample-containing discs—five per plate—were deposited on the swabbed surfaces of the plates. After a full day of incubation at 37°C, the plates were examined for zones of inhibition surrounding the discs. Staphylococcus aureus complexes C_2 and C_3 had more biological activity in the third concentration (10^{-3}), but ligands L and C_1 (figure 10) did not exhibit any action against the bacteria under investigation in the first concentration. At the first concentration, none of the synthesized compounds exhibited biological activity against Escherichia coli; nevertheless, at the third concentration, their activity greatly increased [45].

Table 3. The biological activity of ligand and complexes

Compounds	***Conc. (mg/10ml)***	***G+***	***G-***
		Staph. aureus	*E. Coli*
L	10^{-5}	n.s	n.s
	10^{-4}	9	9
	10^{-3}	13	18
C_1	10^{-5}	n.s	n.s
	10^{-4}	8	9
	10^{-3}	14	17
C_2	10^{-5}	8	n.s
	10^{-4}	13	8
	10^{-3}	19	21
C_3	10^{-5}	10	n.s
	10^{-4}	14	8
	10^{-3}	23	19
Tetracycline	10^{-3}	28	26

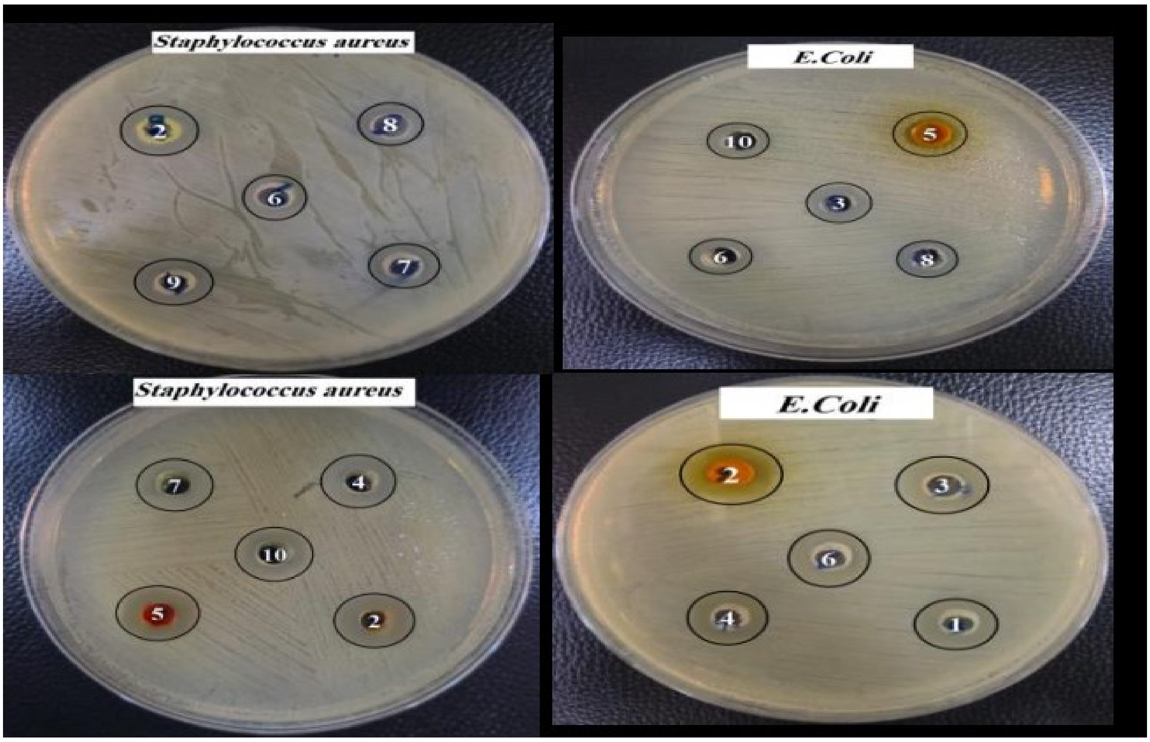


Figure 10. Antibacterial actions against Staphylococcus aureus of ligand and its complexes [C_1, C_2, C_3]

4. CONCLUSION

The synthesis and characterisation of 4-methoxy-N-(2-(phenylcarbamothioyl)hydrazine-1-carbonothioyl)benzamide (L) and its new metal complexes are reported. Monomeric complexes were isolated as a consequence of the ligand's interaction along with metal ions of manganese(II), cobalt(II), and nickel(II) in one to one (ligand:metal) mole ratio. The employed physicochemical techniques to confirm the compounds' chemical structures and the complexes' general bonding behaviour. The six-coordinate monomeric complexes with general formula; [$LMnCl_2H_2O$], [$LCoCl_2H_2O$] and [$LNiCl_2H_2O$] were isolated, as verified by the characterisation results. At last, the antibacterial activity of the L and corresponding complexes was evaluated against (G+) and (G-) bacteria.

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