RESEARCH ARTICLE

Metal Complexes of Ligand Derived from Amine Compound: Formation, Spectral Characterization, and Biological Evaluation

Wisam A. Abdulrahman,[1] Iman A. Othman,[1] Enass J. Waheed[2]

[1]*Department of Chemistry, College of Education for Faculty, University of Samarra, Samarra, Iraq*
[2]*Department of Chemistry, College of Education for Pure Sciences, Ibn-Al-Haitham, University of Baghdad, Baghdad, Iraq*

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ABSTRACT

A new set of metal complexes by the general formula $[M(C)_2(H_2O)_2]Cl_2$ has been prepared through the interaction of the new Ligand [N1, N4-bis(4-chlorophenyl)succinamide] (C) derived from succinyl chloride with 4-Chloroaniline with the transition metal ions Mn(II), Co(II), Ni(II), Hg(II), Cu(II) and Cd(II). Compounds diagnosed by TGA, ^1H, $^{13}CNMR$ and Mass spectra (for (C)), Fourier-transform infrared and Electronic spectrum, Magnetic measurement, molar conduct, (%M, %C, %H, %N). These measurements indicate that (C) is associated with the metal ion in a bi-dentate fashion by nitrogen atoms (the amide group) and the octahedral composition of these complexes is suggested. The anti-bacterial action of the compounds towards three types of bacteria *Pseudomonas* (+), *Staphylococcus aureus* (+), and *Escherichia Coli* (–) were studied.

Keywords: 4-Chloroaniline, Biological activity, Succinyl chloride

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INTRODUCTION

Aniline is one of the most organic compounds (toxic). Aniline is involved in synthesizing many organic compounds via azo dyes, corrosion inhibitors, pesticides, antioxidants, rubber, antiseptics, pharmaceuticals, and fuel additives, due to the wastewater structure for pharmaceutical production dyestuffs and its high toxicity due to its aniline content harm aquatic ecosystems.[1,2] Aniline derivatives contribute to the manufacture of paints, pharmaceuticals, rubber, dyes, herbicides, pigments, and precursors to polyurethane. Although aniline compounds produced in some pharmaceutical factories are organic, they are very complex in composition, causing high resistance to biological degradation.[3] 4-Chloroaniline is aromatic amine-containing chlorine substituted for the benzene ring and is used as a medium during the degradation (microbial) of phenyl carbamate with phenylurea. Polymerizing it with guaiacol in an aqueous solution containing oxido reductases creates different oligomers.[4,5] It can be used in these industries (azo dyes, cosmetics, agricultural chemicals, pharmaceutical products, and pigments) as an intermediary. Hemoglobin and liver and kidney proteins covalently bind with metabolites reactions to 4-Chloroaniline. Due to continuous exposure to it, several damages to human health are cyanosis, methemoglobinemia, effects in blood, kidneys, liver, spleen, changes in hematological parameters, and haemosiderosis in the spleen.[6-8] The current study aims to define the structure and geometry of (C) and its complexes to ions Mn(ii), Co(ii), Ni(ii), Hg(ii), Cu(ii), and Cd(ii).

EXPERIMENTAL SECTION

Materials and Methods

From commercial sources, we obtained all chemical and analytical reagents and were used without further purification. From (the United States of America, Sigma - Aldrich, Merck, and India), various chemicals, metal salts, and solvents used in this research were purchased. Infrared spectroscopy was measured using the device (Shimadzu FTIR affinity-1s) and KBr disc within the range (400–4000) cm^{-1}. Using an electrothermal melting point device (SMP10 Stuart), the melting point of the compounds prepared in an open tube was determined. Using (Shimadzu UV-1800) visible, ultraviolet spectrophotometer with a concentration of 3^{-10} M samples in dimethylsulfoxide (DMSO) solvent at room temperature and a quartz cell length of 1.0 cm, the electron spectra of the prepared compounds were measured. Using the device (Bruker 300 MHz NMR spectrometer), the chemical displacements were recorded in (NMR spectra ^1H and ^{13}C) in DMSO-d6 with trimethylsilyl (TMS). Using a device (Shimadzu (AA 680)), %M in complexes is determined. Using a device (Philips pw-Digital conductivity meter), the molar conductivity of

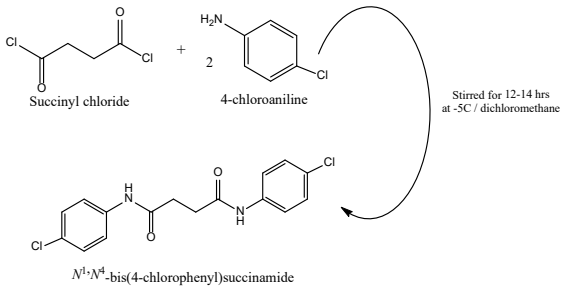
**Author for Correspondence: enasshassanali@gmail.com*

the prepared compounds was measured with a concentration of 10^{-3}M in DMSO and at room temperature. Using a device (magnetic sensitivity balance (Sherwood Scientific)), (μeff BM) of complexes were measured at room temperature. Using a device (Euro EA 300), (%C, %H, %N) was determined for the prepared compounds. An STA PT-1000 Linseis at the temperature range of 0–1000℃ and used argon gas, thermal gravity analysis (TGA) was performed.

The Organic Compound

Synthesis of Ligand (C)[9]

A solution of (0.999 g, 6.45 mmol) of Succinyl chloride in acetone (10 mL) was slowly added to a solution of 4-Chloroaniline (1.645 g, 12.9 mmol) in dichloromethane 10 mL over 1-hour. The mixture was stirred for 12 hours at -5°C and then for 2 hours at laboratory temperature. The precipitate formed was filtered and then washed with solvents (water and diethyl ether) and then recrystallized in ethanol and left to dry at laboratory temperature. m.p. 160–163°C (yield 88%) (Scheme 1 and Figure 1).



Scheme 1: Preparation course of the (C)

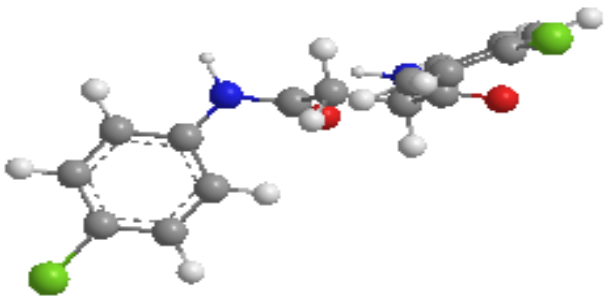


Figure 1: 3D-Structure of the (C)

In-Organic Compounds

Synthesis of $[M(C)_2(H_2O)_2]Cl_2$: To ligand (C) solution (0.66 gm, 2 mmol) in (10ml) EtOH, the metal chloride solution ($MnCl_2$. $4H_2O$, $CoCl_2$. $6H_2O$, $NiCl_2$. $6H_2O$, $CuCl_2$. $6H_2O$, $HgCl_2$. H_2O and $CdCl_2$. H_2O) (0.24–0.17 gm, 1 mmol) in (10ml) EtOH was added. Under reflux, the reaction mixture was heated for 4 hours. The precipitate formed was filtered and then washed with solvents (H_2O and diethyl ether) and then recrystallized in ethanol and left to dry at laboratory temperature (Figure 2)

RESULTS AND DISCUSSION

Thermal stability and the nature of the colored solid are the essential characteristics of the prepared metal complexes. In solvents DMSO and dimethylformamide (DMF), soluble. The theoretical and practical data of (AA) measurements for all prepared complexes were approximated (Table 1).

Mass Spectral

The mass spectral data fragmentation of (C) [N1,N4-bis(4-chloro phenyl)succinamide], Figure 3 showed (M+)

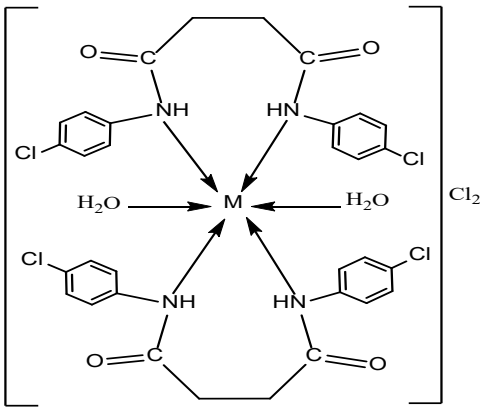


Figure 2: Suggested structure for (C) complexes

Table 1: Different physical properties of the prepared compounds

Compound	*Empirical Formula (Formula wt.)*	*m.p (°C)*	*Color*	*Metal% Calculated (Actual)*	*Conducts $Ohm^{-1}cm^2mol^{-1}$ In solvent (DMSO)*
Ligand (C)	C16H14Cl2N2O2 337.20	160–163	Yellow	-----	1.68
Mn-C	C32H32Cl6MnN4O6 836.27	188–190	Off-White	6.57 (6.56)	74.3
Co-C	C32H32Cl6CoN4O6 840.26	197–200	Green	7.01 (7.03)	75.5
Ni-C	C32H32Cl6NiN4O6 840.02	117–120	Green	6.99 (7.01)	72.1
Cu-C	C32H32Cl6CuN4O6 844.88	168–170	Brown	7.52 (7.51)	70.4
Cd-C	C32H32Cl6CdN4O6 893.74	214–216	Yellow	12.58 (12.56)	81
Hg-C	C32H32Cl6HgN4O6 981.92	177–180	Yellow	20.43 (20.44)	69

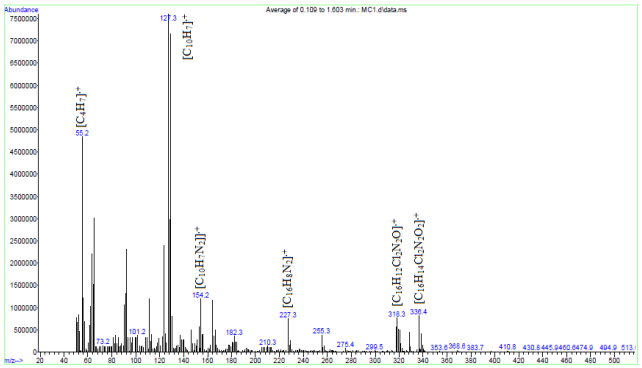


Figure 3: EI mass spectrum of (C).

Table 2: The fragmentation pattern of (C)

Fragment	*Mass/charge (m/z)*	*Relative abundance*
$[C16H14Cl2N2O2]^{\cdot+}$	336	12%
$[C16H12Cl2N2O]^{\cdot+}$	318	12%
$[C16H8N2]^{\cdot+}$	227	9.3%
$[C10H7N2]]^{\cdot+}$	154	22.7%
$[C10H7]^{\cdot+}$	127	100%
$[C4H7]^{\cdot+}$	55	64%

at (m/z+=336) Due to (the original molecular weight of C) (337), $[C_{16}H_{14}Cl_2N_2O_2]$. Additional peaks are displayed in Table 2.[10,11]

Nuclear Magnetic Resonance (NMR) Spectra

^{1}H-NMR Spectra of (C)

In order to confirm the structure of the ligand (C), ^{1}HNMR of the synthesized ligand was recorded. Figure 4 display the ^{1}HNMR of the (C), the peak at (δ 10.25–10.29 ppm) corresponds to the proton of NH group. The peaks at (δ 7.23–δ 7.82 ppm) correspond to all the aromatic protons of the ligand. The peak at (δ 2.71–2.78 ppm) corresponds to four aliphatic protons of the succinyl group). Finally, the peaks at (δ 2.47–2.58 ppm) correspond to residual protons in DMSO-d6 and moisture protons in DMSO-d^6.[12,13] The results are summarized in Table 3.

^{13}C-NMR Spectra of (C)

The ^{13}C-NMR spectrum for ligand (C) portrays is shown in Figure 5. The spectrum exhibits signals at chemical shift (δ=177.17 ppm), which assign to ($C_{1,1}^-$). The chemical shifts at (δ =138.76, 135.99, 129.28–129.82 and 120.91 ppm) attributed to the aromatic ring ($C_{2,2}^-$),($C_{3,3}^-$),($C_{4,4}^-$) and ($C_{5,5}^-$) respectively. Chemical shift at (δ =39.10–40.78 ppm) related to DMSO-d^6. Finally chemical shift at (δ =31.52–31.63ppm) attributed to ($C_{6,6}^-$).[14,15]

Fourier Transform Infrared Spectroscopy (FTIR) Spectra

Ligand (C)

The band at 3299 cm^{-1} the spectrum of the (C) determined to the υ(NH), while another absorption band showed at (1652) cm^{-1} could be interpreted as υ(C=O amide), in addition to

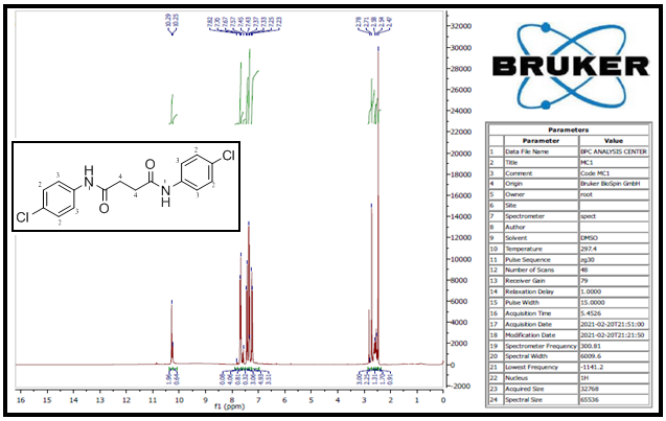


Figure 4: ^{1}H-NMR spectra of (C)

Table 3: Chemical shifts in (^{1}H-NMR) spectra of (C)

Compound	*Functional groups*	*δ (ppm)*
Ligand (C)	proton (N-H) group	10.25–10.29 (2H),d
	Aromatic protons C(2,2$^-$),(3,3$^-$)	7.23–7.82 (8H),m
	succinyl group protons C(4,4$^-$)	2.71–2.78 (4H),d
	DMSO-d^6	2.47–2.58

m = multiplet, d = doublet

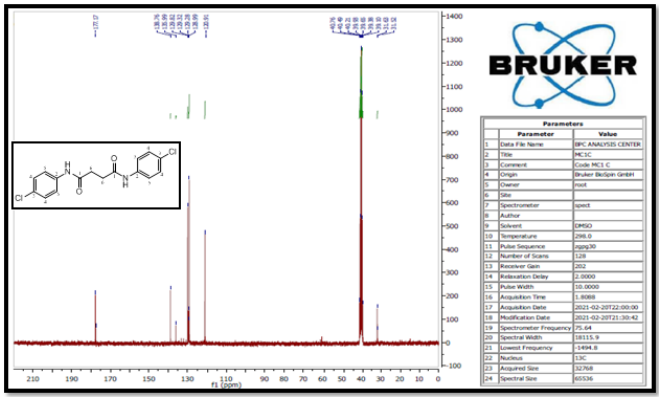


Figure 5: ^{13}C-NMR spectra of (C)

Table 4: Chemical shifts in (^{13}C-NMR) spectra of (C)

Compound	*Functional groups*	*δ (ppm)*
Ligand (C)	C1,1$^-$ for (CO-NH) amide group	177.71
	C2,2$^-$ for aromatic ring	138.78
	C3,3$^-$ for aromatic ring	135.99
	C4,4$^-$ for aromatic ring	129.28-129.32
	C5,5$^-$ for aromatic ring	120.91
	C6,6$^-$ for (CH2) Succinyl group	31.52-31.63
	DMSO	39.10-40.78

absorption bands at (2921) due to υ(C-H aliphatic), (3053) cm^{-1} due to υ(C-H aromatic) and (2835) cm^{-1} which determined to υ(C-Cl), Figure 6.[16]

Complexes of Ligand (C)

These spectra showed a marked various between bands belonging to the stretching vibration of υ(NH, amide group) in the range between (3427–3402) cm^{-1} shifted to higher

frequencies by 128–103 cm^{-1} proposing the probability of the coordination of (P) by the nitrogen atom at the amide group.[17,18] The υ(C=O, amide) in ligand was not correlated with the central ion and confirmed by no changes in frequencies of this group set at (1654–1649) cm^{-1} in the complexes spectrum of the ligand complexes.[19,20] New bands υ(M-O, water group) and υ(M-N, amide group) appeared in the range of (823–819) cm^{-1} and (487–495) $cm^{-1,}$ respectively, indicating the formation of the metal complexes.[21,22] In Table 5, FTIR data is displayed. In Figure 7, the spectra of the (C) and its complexes were presented.

Electronic Spectra

Ligand (C)

The electronic spectrum of (C) in DMSO solution is including in Figure 8. The spectrum display absorption peaks (two) at (254 nm =39370 cm^{-1}; ε_{max}= 3720 $molar^{-1}$ cm^{-1} and (292 nm =34246 cm^{-1} ; ε_{max}= 3853 $molar^{-1}$ cm^{-1} which assigned to ($n\rightarrow\pi^*$) and ($\pi\rightarrow\pi^*$) respectively.[23] In Table 6 data are recorded.

Complexes of Ligand (C)

The electronic spectrum of the Mn (ii) - complex display peaks in the (d-d) region at 887 nm, which is due to $^6A_1g\rightarrow{}^4T_2g_{(G)}$ transition and 910 nm assigned to $^6A_1g\rightarrow{}^4T_1g_{(G)}$. Another peak in the275 nm and 314 nm which assigned to intra ligand, these peaks indicating octahedral geometry about Mn ion.[24]

The Co (ii) - complex show peak at 272 nm and 310 nm attributed to intra ligand. Another peak in the (d-d) region 600 is due to $^4T_{1g\ (F)}\rightarrow{}^4A_{2g\ (P)}$ transition and 680 due to $^4T_{1g\ (F)}\rightarrow{}^4T_{2g(F)}$. These electronic transitions revealed by the cobalt complex refer to the octahedral geometry of the aforementioned complex.[25]

The electronic spectrum of the Ni^{II} - complex display in the (d-d) region (peaks) at 686 nm assigned to $^3A_{2g}\rightarrow{}^3T_{1g\ (P)}$ transition and 774nm due to $^3A_{2g}\rightarrow{}^3T_{2g(F).}$ Other peaks at 272 nm and 312 nm due to intra ligand indicate octahedral geometry about Ni ion[26] (Figure 9).

The electronic spectrum of the Cu (ii)- complex display in the (d-d) region (peak) at 944 nm attributed to $^2E_g\rightarrow{}^2T_{2g}$. Another peak at 264 nm and 310 nm due to intra ligand, these all confirming distorted octahedral geometry about Cu ion.[27]

The electronic spectra of the Cd-complexes exhibited peaks at (267, 310) nm, which were due to (intra-ligand) and another peak at 399 nm due to (CT) and the Hg-complexes exhibited peaks at (278, 310) nm, which were due to (intra-ligand) and another peak at 410 nm due to CT. These are all indicating octahedral geometry about Cd and Hg ions.[28] In Table 6, UV data of compounds was displayed.

Magnetic Moments and Conductivity Measurements

In Table 5, the ⁋(μeff) values of Mn(ii), Co(ii), Ni(ii), and Cu(ii) complexes are display. These complexes exhibit μeff (5.494, 4.370, 3.491 and 2.134) B.M respectively, these normal values are consistent with high spin octahedral complexes.[29] The electrolytes nature (1:2); M^{+2}= Mn(ii), Co(ii), Ni(ii), and Cu(ii), Hg(ii) and Cd(ii) complexes of all metal complexes was confirmed by molecular conductivity measurements,[30] (Table 1).

Table 5: FT-IR data of (C) (cm^{-1}) and their complexes.

Comp.	*υ(C-H) Arom.*	*υ(C-H) Aliph.*	*υ(N─H)*	*υ(C=O) amide*	*υ(C-Cl)*	*υ(M-N)*	*δ(M-O) of water*
Ligand (C)	3053	2921	3299	1652	721	-	-
Mn-C	3033	2925	3421	1650	717	823	487
Co-C	3051	2904	3402	1650	717	821	491
Ni-C	3053	2920	3423	1649	717	821	491
Cu-C	3035	2991	3413	1654	717	819	489
Cd-C	3014	2929	3421	1652	717	823	487
Hg-C	3037	2927	3427	1652	713	821	495

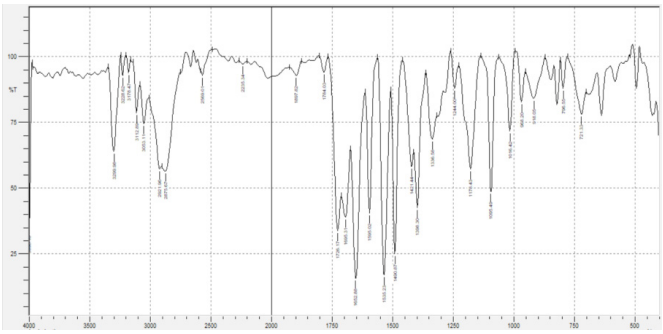


Figure 7A: FTIR spectrum of [C] complex.

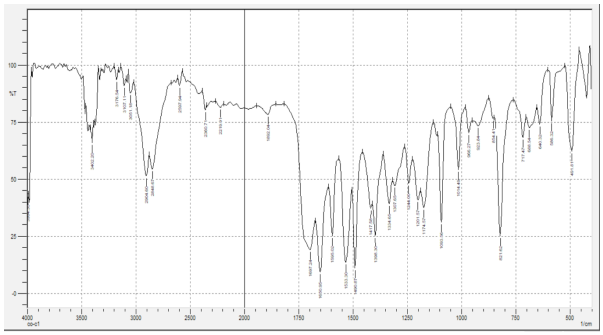


Figure 7B: FTIR spectrum of [Co-C] complex.

Thermal Analysis

The ligand (C) was prepared and subjected to thermal analysis using a STAPT-1000 Linseis company1 Germany. In an atmosphere of argon gas, this measurement was done within the temperature range (0–1000)°C and heating rate 10°C/min.[31,32] Where it was recorded, all results are derived from the TG curves for the ligand (C) examined (Table 7 and Figure 10).

Table 6: Electronic transfers in the spectra of the prepared compounds

Com.	*λmax (nm)*	*υ (cm^{-1})*	*εmax ($moler^{-1}.cm^{-1}$)*	*Transition*	*Shape geometry*
Ligand (C)	254 292	39370 34246	3720 3853	n→π* π→* π*	-
Mn-C	215 314 887 910	36363 31847 11273 10959	3768 3653 15.8 16.2	Intra-ligand Intra-ligand ^{6}A1g→^{4}T2g(G) ^{6}A1g→^{4}T1g(G)	Octahedral
Co-C	272 310 600 680	36764 32258 16667 14705	7048 6920 212.9 428.9	Intra-ligand IntrA-ligand ^{4}T1g(f)→^{4}A2g ^{4}T1g(f)→^{4}T2g(f)	Octahedral
Ni-C	272 312 686 774	36764 32051 14577 12919	7048 3597 17.2 16.8	Intra-ligand Intra-ligand ^{3}A2g→^{3}T1g(f) ^{3}A2g→^{3}T2g(f)	Octahedral
Cu-C	264 310 944	37878 32258 10593	7222 6920 264.1	Intra-ligand Intra-ligand 2Eg→ 2T2g	Octahedral
Cd-C	267 310 399	36231 32258 25062	7390 7429 132.9	Intra-ligand Intra-ligand C.T	Octahedral
Hg-C	278 310 410	35971 32258 24390	7369 7001 628.5	Intra-ligand Intra-ligand C.T	Octahedral

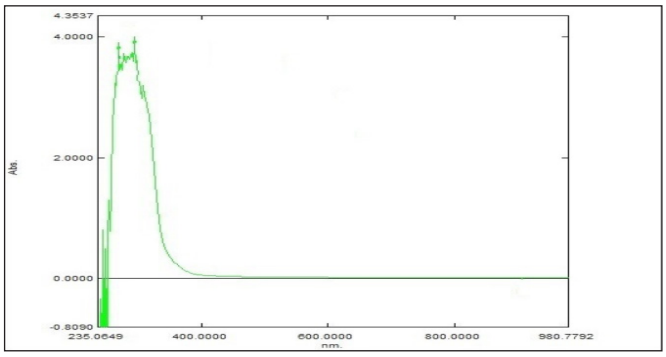


Figure 8: Electronic spectrum of the C.

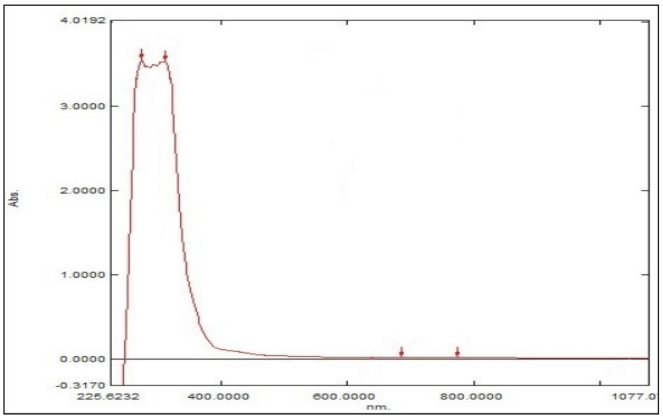


Figure 9: Electronic spectrum of [Ni-C] complex density.

Table 7: Temperature values for analysis along with corresponding weight loss values

Com.	*Stage*	*TGA Thermo Gravimetry range (°C)*	*Found (calculate) loss of Mass (mg)*	*Total mass lose(mg)*	*Fragmentation*
Ligand (C)	1	160–250	2.056 (2.046)	5.464	- C_2H_6, 1/2Cl_2, N_2O_2
	2	251–40	2.742(2.741)	(5.441)	-11C + 1/2 Cl
	3	341–1000	0.665(0.654)		-C_3H_4

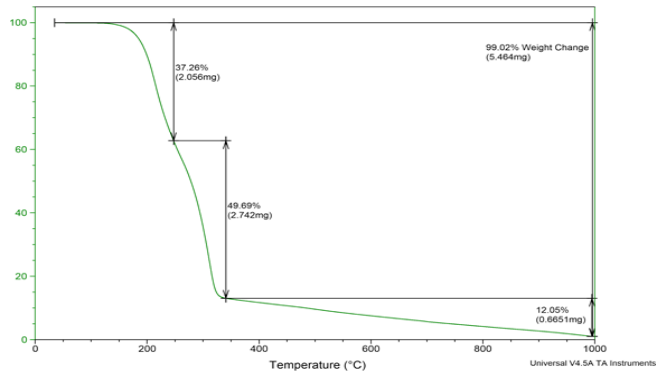


Figure 10: Thermal study of (C)

Table 9: Results obtained from the biological activity of the prepared compounds.

No.	*Compound*	*Escherichia coli (G -)*	*Pseudomonas (G -)*	*Staphylococcus aureus (G +)*
	Control	-	-	-
1	Ligand C	4	8	-
2	$[Mn(C)_2(H_2O)_2]Cl_2$	14	-	-
3	$[Co(C)_2(H_2O)_2]Cl_2$	9	10	-
4	$[Ni(C)_2(H_2O)_2]Cl_2$	9	-	9
5	$[Cu(C)_2(H_2O)_2]Cl_2$	14	4	5
6	$[Cd(C)_2(H_2O)_2]Cl_2$	19	12	-
7	$[Hg(C)_2(H_2O)_2]Cl_2$	4	29	-

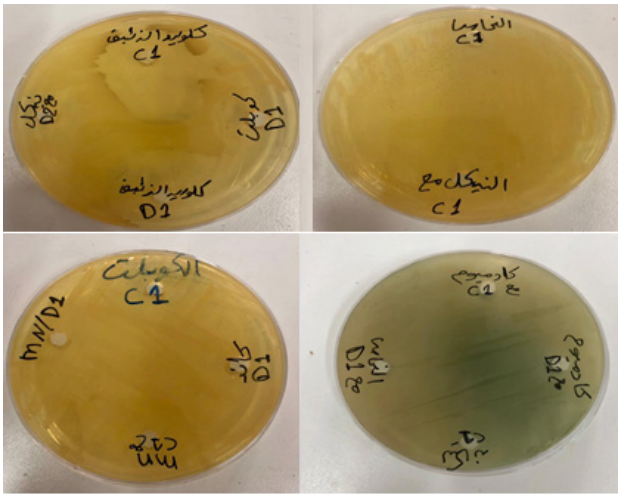


Figure 13: Figures of the biological activity of the prepared compounds

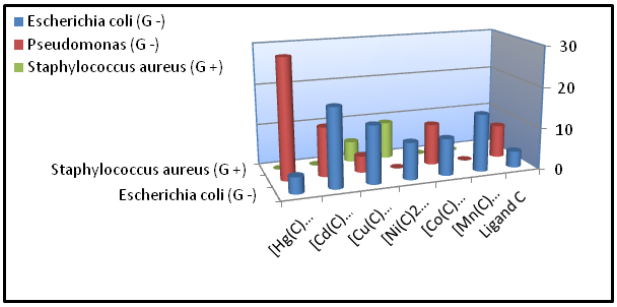


Figure 14: Graph of the results of the bioactivity of the prepared compounds

Anti-bacterial Activity Studies

Pseudomonas (+), *Staphylococcus aureus* (+), and *Escherichia Coli* (–) were used as test micro-organisms. The medium surface was inoculated and covered to perform the assay on test organisms. Before applying disks, allow the agar surface to dry for 3 to 5 minutes. The tablets were dipped in a chemical beaker using sterile forceps and placed in the medium previously. To grow the bacteria, plates were incubated at 37°C for 48 hours. The complexes show different activity in limiting bacterial proliferation compared to ligand (C) in concentrations prepared.[33-34] The results obtained are recorded (Table 8, Figure 11 and 12)

CONCLUSION

This search showed the prepared and identification of (C) and its Mn (ii), Co (ii), Ni (ii), Cu (ii), Hg (ii), and Cd (ii) compounds. The octahedral geometry of the prepared complexes was suggested depending on the results (TGA, ^{1}HNMR, Mass spectra, and ^{13}CNMR of (C) only), magnetic measurement, and molar conductivity of the compounds. The (C) acts as bidentate and coordinates by the N atoms in (NH-C=O) group. The thermal decomposition steps of the different parts of the prepared ligand (C) were determined by studying TGA. The (C) and its complexes were examined for their anti-bacterial activities against three types of bacteria.

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