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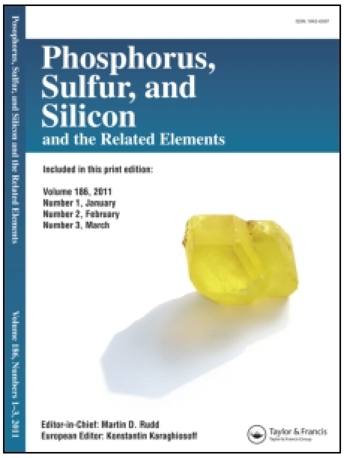

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Studies of Some New Aminocyclodiphosphazane Complexes of Co(II), Ni(II), and Cu(II)

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Studies of Some New Aminocyclodiphosphazane Complexes of Co(II), Ni(II), and Cu(II)

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The ligand aminocyclodiphosph(V)azane derivative (III) and its complexes with Co(II), Ni(II), and Cu(II) ions were prepared and characterized by microanalytical, FTIR, ^{1}H, ^{13}C, and ^{31}P-NMR, UV/Visible, thermogravimetric (TGA) analysis, and magnetic moments. The ligand acts in a tetrahedral manner forming 2:1 metal to ligand ratio. The copper complex is assigned to be tetrahedral while cobalt and nickel complexes were assigned to be octahedral structure.

Keywords ^{31}P-NMR ; aminocyclodiphosph(V)azane; electronic; IR

INTRODUCTION

The importance of phosphorus compounds has been realized as antioxidants, corrosion resistant materials, stabilizers in plastic manufacture, oil industry, flame-proofing as well as agricultural insecticides.[1–3] Also, cyclotriphosphazanes were been synthesized,[4,5] and their structures have been verified by X-ray single crystal analysis. Moreover, cyclodiphosphazanes such as hexachlorocyclodiphosph(V)-azanes have been synthesized and their structures were studied by many authors.[6–10] The reaction of hexachlorocyclodiphosph(V)azanes with aromatic and aliphatic amines, active methylene containing compounds, and bifunctional reagents and their metal complexes have been investigated.[11–15] In the present work, the interaction of hexachlorocyclodiphosph(V)azane **(I)** with amino thiophene derivative and its metal complexes have been reported.

Little is known about the interaction of hexachlorocyclodiphosph(V)azane with amino thiophene derivative and its metal complexes.

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EXPERIMENTAL

Starting Materials

The preparation and purification of hexachlorocyclodiphosph-(V)azanes **(I)** has been reported.[7–16] The preparation of 2-amino-3-cyanothiophene **(II)** was carried out according to the Gewald method.[17]

Synthesis of Ligand (III)

The solid of 2-amino-3-cyanothiophene **(II)** (2.48 g; 0.01 mol) was added in small portions to a well-stirred solution of the hexachlorocyclodiphosph(V)azane **(I)** (2.285 g; 0.005 mol) in 100 ml acetonitrile for 1.5 h. After the complete addition, the reaction mixture was heated under reflux for 2 h with continuous stirring. After completion of the reaction (HCl gas ceased to evolve) the reaction mixture was filtered while hot, and the filtrate was left to cool at room temperature. The obtained solid (bright yellow) was filtered washed several times with acetonitrile, followed by ether and then dried in vacuo to give the corresponding aminocyclodiphosph(V)azane derivative **(III)** (Figure 1). Yield = 92%; m.p. = 190°C; elemental analysis calculated (found) for $C_{22}H_{16}N_6P_2S_2Cl_4$ (632): C = 41.75% (40.87%), H = 2.53% (2.54%), N = 13.29% (13.28%), P = 9.80% (7.90%), S = 10.12% (10.10%).

Preparation of the Solid Metal Complexes

A solution of the salts (0.002 mol) in 50 ml absolute ethanol was added dropwise to a solution of aminocyclodiphosph(V)azane derivative **(III)** (0.001 mol) in 50 ml absolute ethanol at room temperature with continuous stirring. After the complete addition of the metal salt solution, the reaction mixture was heated under reflux for three hours. Then,

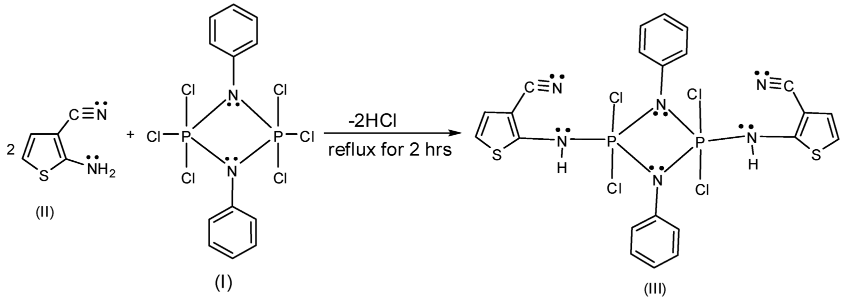

FIGURE 1 Structure of ligand III: 1,3-diphenyl-2,2,4,4-tetrachloro-2,4-bis(2-imino-3-cyanothiophene) cyclodiphosph(V)azane.

the reaction mixture was evaporated to give solid compounds, which recrystallized from ethanol to give the products $\mathbf{IV_{a-c}}$. The analytical data of both ligand and its metal complexes are listed in Table I.

Measurements

Elemental analysis of C, H, and N and S were carried out using the Perkin-Elmer 2400 apparatus and phosphorus was determined gravemetrically as phosphoammonium molybdate using the Voy method.[18] Infrared spectra were recorded in the solid state on a Mattson 5000 FTIR spectrometer using KBr disc technique. The absorbance of solutions was measured in the UV/VIS range (200–800 nm) using Unicam spectrophotometer model UV 2-100 and 1 cm matched quartz cells. The ^{1}H and ^{13}C NMR spectra were recorded on a Varian Spectrometer at 90 (22.5) MHz, using TMS as an internal reference. ^{31}P NMR spectra were run, relative to external H_3PO_4 (85%), with a Varian FT-80 spectrometer at 365 MHz. Magnetic measurements were recorded by the Gouy method at room temperature using a magnetic susceptibility balance (Johnson Mathey), Alfa product. Model No. (MK). The conductometric measurements in solutions were carried out using conductivity TDS model 72. The thermal analyses of the complexes were carried out using a Shimadzu thermogravimetric analyzer with a TGA-50H detector in nitrogen atmosphere. The percent weight loss was measured from the room temperature to 1000 °C at a heating rate of 20°C/min.

RESULTS AND DISCUSSION

The structure of the free ligand was investigated using elemental analyses, IR, UV/VIS, ^{1}H, ^{13}C, and ^{31}P NMR.

IR Spectra

The assignments of the important bands of the free ligand are given in Table II. The infrared spectrum of free ligand exhibits band at 2600 cm^{-1} which was assigned to ν_{P-NH} stretching vibration similar to those assigned previously.[19] The band observed at 2190 cm^{-1} [20] in the compound **(II)** which was assigned to be $\nu_{C\equiv N}$stretching vibration was shifted to higher frequency in the ligand at 2216 cm^{-1} due to the ligand formation. The ν_{P-Cl} stretching vibration is observed at 459 cm^{-1}.[21,22] The observed band at 1250 cm^{-1} was attributed to the ν_{P-N} stretching vibration.[23,24] Multibands appear in the range 1654–1435 cm^{-1} may be ascribed to $\nu_{C=C}$of the phenyl rings as well as attached compound **(II)**.[24]

TABLE I Elemental Analyses, Yields, Colors, and Melting Points of Ligand (III) and Its Corresponding Metal Complexes

						Elemental analyses found (calc.), %					
Compd. no.	Reactants		Mol. formula (m. wt.)	Color	M.p. °C (yield %)	C	H	N	P	S	M
IV_a	III (0.63 g; 0.001 mmol)	$Co(OAc)_2.4H_2O$ (0.45 g; 0.002 mmol)	$C_{32}H_{44}N_6P_2S_2Cl_4O_{10}Co_2$ (1058.48)	Brown	234 (92.8)	36.15 (36.31)	4.08 (4.19)	7.92 (7.94)	5.84 (5.85)	6.00 (6.06)	11.00 (11.14)
IV_b	III (0.63 g; 0.001 mmol)	$Ni(OAc)_2.4H_2O$ (0.45 g; 0.002 mmol)	$C_{32}H_{44}N_6P_2S_2Cl_4O_{10}Ni_2$ (1058.00)	Brown	238 (92.7)	36.13 (36.33)	4.11 (4.19)	7.42 (7.94)	5.83 (5.86)	6.02 (6.06)	11.08 (11.10)
IV_c	III (0.63 g; 0.001 mmol)	$CuBr_2$ (0.47 g; 0.002 mmol)	$C_{22}H_{20}N_6P_2S_2O_2Br_4Cl_4Cu_2$ (1115.03)	Dark brown	265 (97.6)	23.24 (23.70)	1.92 (1.81)	7.40 (7.54)	5.37 (5.56)	5.21 (5.75)	11.20 (11.40)

TABLE II IR Spectra of Ligand and Its Metal Complexes

Compd.no.	ν_{NH}	ν_{P-NH}	$\nu_{Disubstitutedring}$	$\nu_{C\equiv N}$	ν_{OCOCH3}	ν_{p-N}	ν_{P-Cl}	ν_{M-N}	ν_{M-O}
III	3375 (br)	2600 (w)	776 (m)	2216 (m)	—	1252 (m)	459 (m)	—	—
IV_a	3321 (br)	2600 (v.w)	775 (m)	2207 (m)	1461 (m) 1563 (m)	1223 (m)	450 (m)	250 (m)	550 (m)
IV_b	3326 (br)	2600 (v.w)	770 (m)	2207 (m)	1465 (m) 1561 (m)	1220 (m)	445 (m)	300 (m)	525 (m)
IV_c	3279 (br)	2600 (v.w)	775 (m)	2208 (m)	—	1234 (m)	460 (m)	265 (m)	—

Electronic Spectra

The electronic spectrum of the ligand (III) showed an absorption band at longer wavelength 282 nm than that associated with its dimer (I) at 277 nm.[25] This bathochromic shift could be expected due to the replacement of one chlorine atom of each phosphorus atom of the phosphazo ring of dimer **(I)** by chromophoric group containing conjugated system such as 2-amino-3-cyanothiophene **(II)**. It is worthwhile that the expected band at 275 nm,[25] characteristic for the delocalization of the nonbonding electrons on the nitrogen atoms along the phosphazo ring of the dimeric structure was observed in the spectrum of ligand **(III)** at 282 nm, indicated the presence of the phosphazo ring. Moreover, the new band observed at 355 nm corresponding to n-π^* the transition of attached compound **(II),** was absent in its dimer **(I),** that is considered as an evidence for the ligand formation.

^{1}H, ^{13}C, and ^{31}P-NMR Spectra

The ^{1}H-NMR spectrum of the ligand **(III)** showed characteristic proton signals at $\delta = 7.80$ ppm corresponding to phenyl protons and the signal at $\delta = 8.3$ ppm due to N-H proton, which disappeared on the addition of D_2O because of the proton exchange. Presence of carbonitrile group in the ligand is also attested by a signal at $\delta = 104.7$(-C≡N) in its ^{13}C NMR spectrum.[26] The ^{31}P NMR of the ligand records a signal at $\delta =$ 116.4, 96.6 ppm, which supports the phosphazo ring structure.[27]

Spectrophotometric Measurements of the Solution Stoichiometry

The absorption spectra of the complexes $\mathbf{IV}_{a-c}$ are shown in Figure 2. The diagrams in Figure 3, showed two straight line se gents, the intersection of which corresponding to 1:2 [ligand] / [M^{+2}], where M^{+2} corresponding to Mn^{+2}, Co^{+2}, Ni^{+2}, and Cu^{+2} respectively, indicating the formation of 1L: 2M species. These results are in agreement with the elemental analyses and conductometric data. The stoichiometry[15] of these complexes was also defined by conductometric titration (Figure 4), in order to follow up the behavior of the ligand III in solution with each of the metal ions studied. The curves were plotted between the conductance of the solution and the volume of ligand added. The results revealed the complex formation of 1L: 2M with all metal ions used. The reason for increase in conductivity after the complex formation

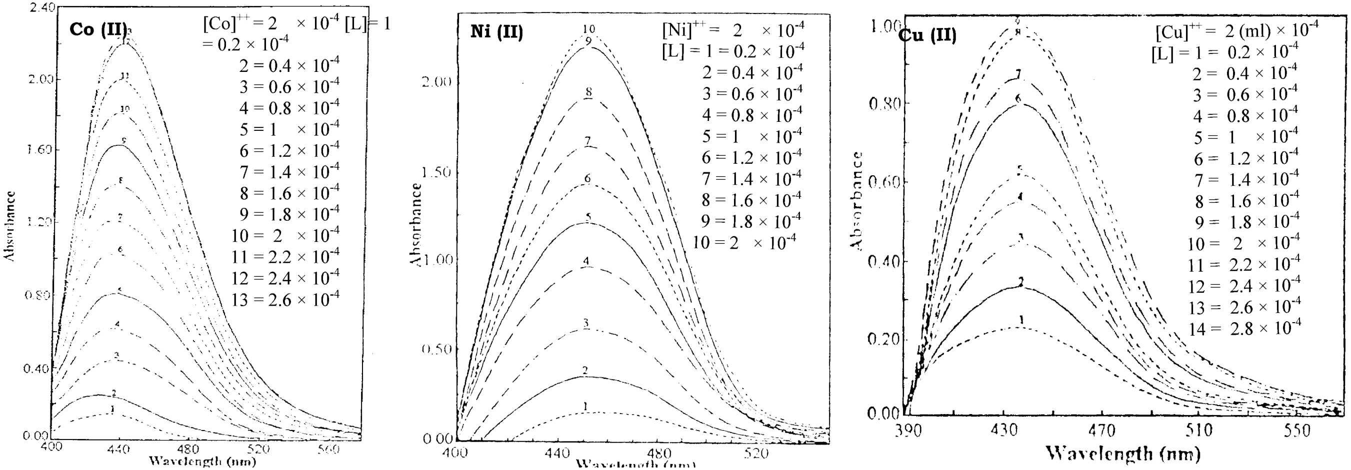

FIGURE 2 Absorption spectra for Co(II), Ni(II), and Cu(II) complexes.

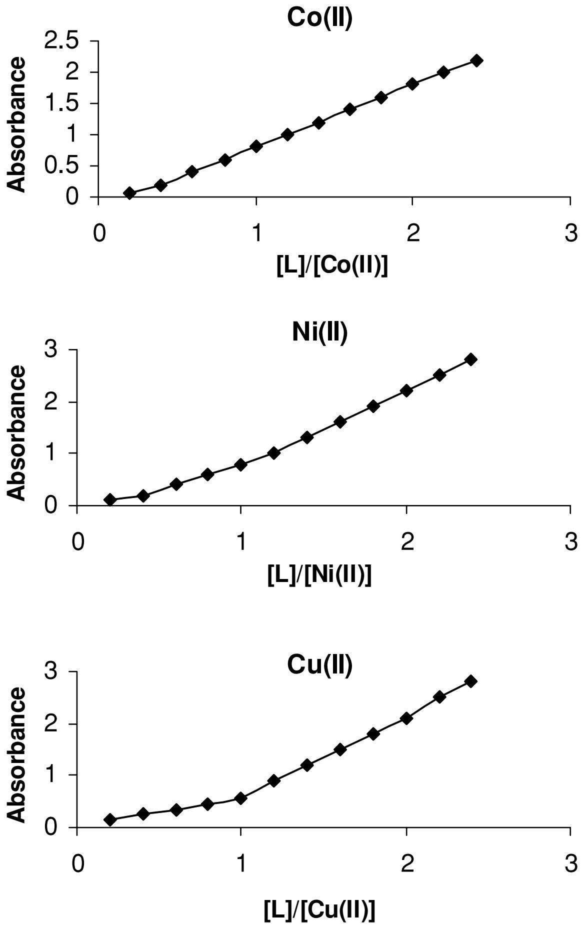

FIGURE 3 Results of molar ratio method for Co(II), Ni(II), and Cu(II) complexes.

was investigated to the presence of the ligand in ionic form in the medium (ethanol) which a rises the conductivity.

The Thermal Analysis of the Complexes

Table I shows the analytical data of the ligand **III** and its studied complexes. The elemental analysis of the complexes indicate that these compounds exhibit the formula $[Co_2(II)L(4OAc).4H_2O].2H_2O$, $[Ni_2(II)L(4OAc).4H_2O].2H_2O$, $[Cu_2(II)L(4OAc)].3H_2O$. Conductivities of the

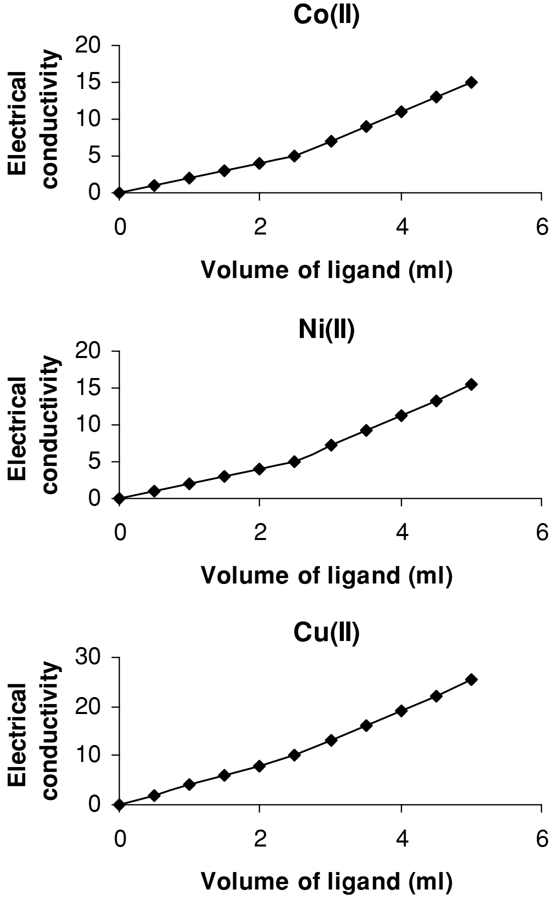

FIGURE 4 Conductometric titrations for Co(II), Ni(II), and Cu(II) complexes.

three complexes in DMF (0.001 M) are 3.2, 2.1, and 2.87 S cm^2 mol^{-1} for Co(II), Ni(II), and Cu(II) complexes, respectively, indicate that the complexes are behaving as non-electrolytes.

The thermogram of Co(II) complex shows that there are four water molecules as water of coordination, the decomposition range was (110–180°C) and two water molecules are involved as hydration water in the range (75–105°C). The initial mass loss observed for Ni(II) complex in the temperature range (80–120°C) shows the presence of two water molecules attributed as water of hydration. In the temperature range 180–210°C, the mass loss shows the presence of four coordinated water molecules. For Cu (II) complex, the TGA analysis indicate the complex contain three hydration water molecules (60–115° C).

Magnetic and Electronic Spectral Studies

Table III lists the electronic spectral bands and the magnetic moments of the metal ions in their complexes in the solid state. On complexation, the lower-energy band observed at 345 nm in the spectrum of the free ligand **III**, which was assigned to n-π^*, is shifted to a red shift, while the other one at 282 nm of the $\pi - \pi^*$ transition is slightly The magnetic susceptibility measurement at room temperature[15,16–28] of the green Co(II) complex $\mathbf{IV}_a$, gave magnetic moment value μ_{eff} of 5.04 B.M.[16]; this high value may be due to orbital contribution corresponding to three unpaired electrons is expected for a week field ligand. The electronic spectra of the Co(II), Ni(II), and Cu(II) complexes as nujol mulls and/or solution in ethanol (Table III) were recorded in the range 200–900 nm. Two Peaks at 705 nm and 554 nm, which may be assigned to $^4T_{1g}(F) \rightarrow {}^4A_{1g}(F)$ and $^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ are consistent with the octahedral geometry.[15] The peaks observed at 255 nm, 310 nm, and 380–405 nm regions, were assigned to $\pi \rightarrow \pi^*$ and n→ π^* transitions respectively.

The deep brown Ni-complex $\mathbf{IV}_b$ gives value μ_{eff} 2.92 B.M.,[16] which is indicative of two unpaired electrons. The electronic spectrum of the Ni (II) complex $\mathbf{IV}_b$ shows absorption band at 712 nm which may be investigated to the $^4A_{2g}(F) \rightarrow {}^4T_{1g}(F)$, week bands observed on the high and low energy sides of the 712 nm band have been assigned to spin forbidden bands.

The Cu (II) complex $\mathbf{IV}_c$ gives value μ_{eff} of 1.74 B.M.[15,16] corresponding to one unpaired electron, which is indicative to tetrahedral structure. The electronic spectrum of the complex $\mathbf{IV}_c$ shows absorption band at 683 nm, which is assigned to $^2B_2 \rightarrow {}^2E$ transition. The observed band

TABLE III Magnetic Moment and Electronic Spectral Data, nm for the Ligand III and Its Metal Complexes

Compound no.	μ_{eff} (B.M.)	$\pi \rightarrow \pi^*$,n→ π^* and charge transfer transitions	d→d transitions
III	—	282, 345	—
IVa	5.04	255,295, 310, 380, 392, 405	554, 705
IVb	2.92	244, 274, 296, 319, 355, 397, 490	594, 712
IVc	1.74	250, 282, 325, 414, 465	683

Where M is Co^{2+} and Ni^{2+} and Ac = acetate

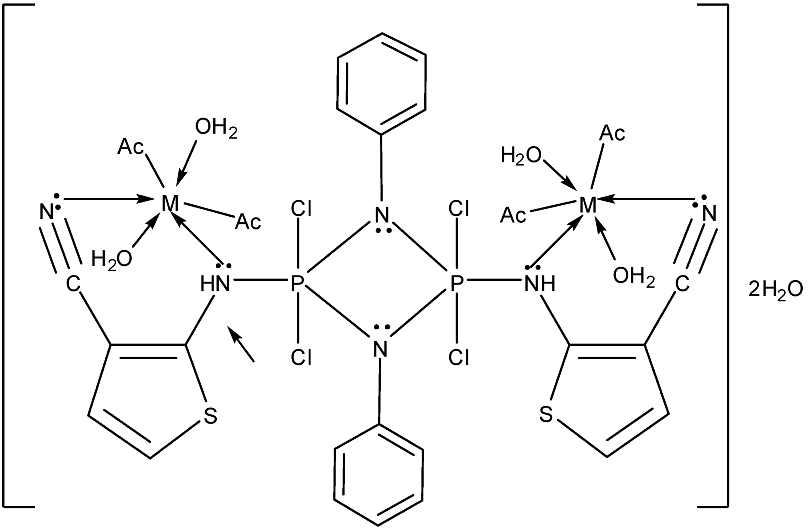

FIGURE 5 The structure of the three complexes.

at 414 nm is attributed to the charge transfer via L→M (Cu^{+2}). The observed band at 250 nm, and 282 nm were investigated to n→ π^*.

It is concluded from the above results that the proposed structure of the investigated complexes coordination with metal ions occurs through the nitrogen of the NH and CN groups as shown in Figure 5.

IR Spectra

The most important vibrational bands of the ligand and its metal complexes are given in Table II. The results of IR spectra of the metal complexes show absorption bands of both $\nu_{C\equiv N}$ and ν_{N-H} at lower frequencies than those of the free ligand **III**, indicating that the metal ions are coordinated to the nitrogen atoms of both C≡N and NH groups of the ligand **III**. Further, in all the metal complexes, there are new medium to weak bands observed at lower frequencies in the range (250–300 cm^{-1}) assigned to ν_{M-N} and two bands at 550 cm^{-1}, 525 cm^{-1} attributed to ν_{m-o} for **IVa** and **IVb,** respectively.[15] The bands observed at 1563 cm^{-1}, 1619 cm^{-1} and 1470, 1561 cm^{-1} in both Co (II) and Ni(II) complexes are attributable to ν_{sym}OCO and ν_{asym}OCO, respectively, which indicated that the acetate groups coordinate as a monodentate to the central metal cation of Co(II), Ni(II), and Cu(II) complexes, respectively. This assumption is supported with the observed characteristic ν_{M-O} band. Moreover, the characteristic vibrational bands corresponding to the ν_{P-NH}, ν_{P-N} and ν_{p-Cl}, which were associated with all the complexes under investigation are given in Table II.

^{13}C-NMR Spectrum

The ^{13}C NMR (125.7 MHz, DMSO-d_6) spectral data also support the authenticity of the proposed structures. The considerable shifts in the positions adjacent to the δ 106.7–107.5 (-C≡N), 123.5–124.4, 127.6–128.2, 129.4–129.8, 135.8–136.4, 139.5–140.3, 148.4–149.2 (6C, Ar), 114.8–115.6, 153.9–154.7 (2S-C=C), 17.2–18.2, 18.3–18.9 (2CH_3), 51.3–52.7, 53.2–54.7 (2OCH_3), 52.1–52.8, 113.2–113.8 (HN-C=C), and 162.4–164.2, and 166.4 (2C=O) ppm, support the proposed coordination in the complexes. Thus, the shifts in the position of carbon atoms adjacent to the coordinating atoms indicate the bonding of the carbonitrile nitrogen and amino nitrogen to the cobalt, nickel, and copper atoms.

Antimicrobial Activity

Table IV shows the results of the antimicrobial activity of the ligand and its metal complexes. The disc diffusion method was used to measure the antimicrobial activity of these complexes. The test compounds

TABLE IV Antimicrobial Activity of III and Corresponding Metal Complexes

		Gram-ve			Gram +ve	
Samples	Concentration (mg/mL)	*E. coli*	*Klebseilla*	*Pseudomonas aeroginosa*	*Staph. aureus*	*Staph. albus*
III	1	+++	++	+++	+++	++
	2.5	+++	+++	++	+++	+
	5	+++	+++	+++	++	+
IV$_a$	1	+++	++	++	++	+
	2.5	++	+++	++	++	+
	5	+++	+++	+++	++	+
IV$_b$	1	++	+++	++	++	+
	2.5	+++	+++	++	++	+
	5	+++	+++	+++	++	+
IV$_c$	1	+++	+	++	+	+
	2.5	+	+++	+	++	+
	5	++	++	++	++	+

were dissolved in dimethylformamide (DMF) (2% w/v) and added at a concentration of 0.5mL/disc to Whatman number 3 filter paper, 5 mm diameter. The antimicrobial activity of some of the prepared compounds of the type III, $IV_{a,}IV_b$, and IV_c were examined with different species of gram-positive bacteria such as *Staphylococcue albus* and *Staphylococcue aurues*, and gram-negative bacteria such as Pseudomonas aeroginosa, *Klebseilla,* and *Escherichia coli*.

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