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SYNTHESIS AND CHARACTERIZATION OF Cu(II) AND Co(II) COMPLEXES WITH *m*-PHENYLENDIAMINE LIGAND

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ABSTRACT

Two new complexes, Cu(II) and Co(II) were successfully synthesized under reflux. A mixture of $Cu(NO_3)_2 \cdot 3H_2O$ and $CoSO_4 \cdot 7H_2O$ with *m*-phenylenediamine (MPD) ligands in methanol with molar ratios of 1:4 and 1:2, respectively resulted in the formation of two new complexes. Complexes characterized using UV-Vis Spectrophotometer, Atomic Absorption Spectrophotometry (AAS), Thermogravimetric Analysis (TGA), conductivity, Fourier-transform Infrared Spectroscopy (FTIR), Magnetic Susceptibility Balance (MSB), and powder XRD. Cu(II) complex transition shows $^2B_{1g} \rightarrow {}^2A_{1g}$ at 459 nm. Meanwhile, the Co(II) complex exhibits a peak at 503 nm, overlapping between $^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$ and $^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$. Complex formulas are $[Cu(MPD)_4](NO_3)_2 \cdot 4H_2O$ and $[Co(MPD)_2H_2O]SO_4 \cdot 2H_2O$, in which the H_2O molecule and MPD are coordinated into Cu(II) and Co(II) ion via nitrogen atom from primary amine group. Both complexes are electrolyte and paramagnetic with μ_{eff} values of 1.76 BM and 4.23 BM, respectively. The complex of Cu(II) is indicated as square planar, Co(II) complex is a trigonal planar with a monoclinic crystal system. There was no antibacterial act for *E. coli* ATCC 25922 and *S.aureus* ATCC 25923 for the two complexes.

Keywords: Copper, Cobalt, Complex, Synthesis, *m*-Phenylenediamine, Antibacterial.

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INTRODUCTION

Cu(II) and Co(II) are often used in complex compounds because they are easy to find, and have high stability and low toxicity.[1,2] Cu(II) and Co(II) can be easily coordinated via N, O, and S ligand donor atoms to form complexes. In general, Cu(II) complexes possess a geometry square planar, and Co(II) complexes possess octahedral or tetrahedral geometry.[3-4]

The complex has important applications in pharmacology and medicine such as antibacterial, anticancer, antifungal, antimalarial, anti-inflammatory, antiviral, and antipyretic.[5-6] One of the interesting things is that the complex can be applied as an antibacterial.[7] Therefore, Cu(II) and Co(II) complexes also possess potential as antibacterial. Ligand, 1,3-diaminobenzene ($C_6H_8N_2$) also called *m*-phenylenediamine (MPD) is a colorless solid, consisting of an aromatic group with two $-NH_2$ functional groups. Ligands containing N donor atoms attract research concern because they exhibit biological activity and have the potential as antibacterial, antifungal, and anti-inflammatory agents.[8] MPD structures tend to be easily coordinated to metals via meta-positioning. Complexes with the Schiff base ligand, *p*-phenylenediamine showed weak antibacterial activity.[9]

This is due to the existence of a substituent group at the position of para which causes the ligand molecule to become large so that it is difficult to penetrate the bacterial wall which has small pores so that the resulting complex is less effective as an antibacterial agent. Antibacterial activity did not appear in the Cu(II) complex of Schiff base o-phenylenediamine in resisting *P.aeruginosa*, *E.coli*, and *S.aureus*.[10]

This is because the steric effect of the ligand makes it difficult to form bonds between Cu(II) with bacterial proteins and DNA functional groups such as -SH and PO^{3-}, thus reducing its antibacterial properties.[11] Co(II) complex with Schiff base ligand *m*-phenylenediamine showed antibacterial activity towards *B.subtilis, S. aureus,* and *N. gonorrhoeae*.[12]

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EXPERIMENTAL

Materials

The substances used in synthesis such as $Cu(NO_3)_2 \cdot 3H_2O$, $CoSO_4 \cdot 7H_2O$, MPD, methanol, DMSO, HNO_3, NaCl, $CuSO_4 \cdot 5H_2O$, $CuCl_2 \cdot 2H_2O$, $NiCl_2 \cdot 6H_2O$, $CoCl_2 \cdot 6H_2O$ retrieved from E. Merck.

Complexes Synthesis

The Cu(II) and Co(II) complexes were synthesized within 1 h. For the Cu(II) complex, 0.242 g; 1 mmol of $Cu(NO_3)_2 \cdot 3H_2O$ and 0.433 g; 4 mmol of MPD in 5 mL of methanol. As for the Co(II) complex, mix $CoSO_4 \cdot 7H_2O$ (0.562 g; 2 mmol) and MPD (0.433 g; 4 mmol) of 10 mL methanol. Then each solution was processed by filtering also drying in a vacuum desiccator for 24 h.

Physical Measurements

The content of metal ions in both complexes was identified by the 'Shimadzu AA-6650 AAS spectrophotometer. Thermal analysis was being conducted on the 'Diamond STA Linseis PT-1600' at 10°C/min. Molar conductivity measurement was performed on a 'Jenway CE 4071' conductivity meter in DMSO solution at 1 x 10^{-3} M. IR spectrum of complexes was being shown on the 'Shimadzu Prestige-21' Spectrophotometer (400-4000 cm^{-1}). The complexes' spectra electronic were analyzed via a 'Lamda 25 Perkin Elmer Spectrophotometer and UV-Vis Double Beam in DMSO. At 25 °C, the 'Auto Sherwood Scientific 10169' MSB was showing the effective magnetic moment (μ_{eff}). Powder XRD complexes were made with 'D8 Advan Bruker'. The analysis of X-ray diffractograms was performed on the Rietica program of the Le Bail method. In order to exact the complexes' space group, the diffractogram of complexes was compared with compounds registered in JCPDS (Joint Powder Diffraction Standards Committee) space groups. The inhibition zone of the antibacterial activity test was determined on the 'OEM 25363' digital electronic caliper with an accuracy of ± 0.025 mm.

Antibacterial Activity

The agar disc diffusion method (Kirby-Bauer) is a method utilized to test the antibacterial activity of metals, MPD, and complexes. The bacteria used are *E. coli* ATCC 25922 Gram-negative and *S. aureus* ATCC 25923 Gram-positive. Agar media (MHA) infected with bacterial suspensions in 0.9% NaCl solution. The samples were evaluated in DMSO 1000, 500, and 250 (g/mL) at various concentrations. On the 6 mm blank paper disc, drops of each sample and DMSO (the negative control) totaling 15 L were made. DMSO, gentamycin, and the samples were added to the MHA medium. At 37°C for 24 h was incubated. An electronic digital caliper is used to measure the zone of inhibition.

RESULTS AND DISCUSSION

Synthesis of Complex

In methanol for 1 h by refluxing method, the synthesis of complexes was run using the ratio of metal to the ligand of 1:1 and 1:2, for Cu(II) and Co(II) complexes. The complexes were found in the form of dark purple precipitate (yield: 61.3%) for the Cu(II) complex and purple precipitate (yield: 44.6%) for the Co(II) complex.

Metal Content Analysis

The copper content in the complex was found to be 9.45% while the cobalt content in the complex was found to be 13.79%. Calculation of the copper and cobalt content of various theoretically possible complex formulas that shown in Table-1. Thus, estimated empirical formula of complex are $Cu(MPD)_4(NO_3)_2(H_2O)_n$ (n = 3, 4 or 5) and $Co(MPD)_2(H_2O)_nSO_4$ (n = 2, 3, or 4).

Table-1: Copper and Cobalt Contents in Various Theoretically Possible Complex Formulas

No.	Empirical Formula	Molecular weight (g/mol)	Cu content theoretically (%)
1.	$Cu(MPD)_4(NO_3)_2(H_2O)_3$	674.12	9.43
2.	$Cu(MPD)_4(NO_3)_2(H_2O)_4$	692.12	9.23
3.	$Cu(MPD)_4(NO_3)_2(H_2O)_5$	710.12	8.95
4.	$Co(MPD)_2(H_2O)_2SO_4$	407.28	14.49
5.	$Co(MPD)_2(H_2O)_3SO_4$	425.28	13.87
6.	$Co(MPD)_2(H_2O)_4SO_4$	443.28	13.31

Thermal Analysis

Thermal analysis can be used to determine water molecules that are possible to be either lattice water or central metal ion coordinated ligands. TGA curve of Cu(II) complex displayed initial weight loss of complex compound by 13.049% at 40-125°C. This specific weight loss is the equivalent of the evaporation of five H_2O molecules. The weight loss at 220°C is where the organic molecules of ligand got decomposed.[13] TGA curve of Co(II) complex exhibited two-step weight loss. In the first step at 40-135°C, the loss of 7.19% weight is in equivalent to the release of two molecules of H_2O. Then, in the second step weight loss at 135 -220°C by 3.10%, equivalent to the loss of one molecule of H_2O. It was reported that weight loss at 30-130°C showed the release of H_2O molecules as lattice water, while weight loss at 150-200°C showed the release of H_2O molecules as coordinated ligands.[14] Thus, the Cu(II) complex has only H_2O molecules as lattice water, whereas Co(II) complex has two molecules of H_2O as crystalline water and one molecule of H_2O as coordinated water. Figure-1 shows the thermograms of both complexes.

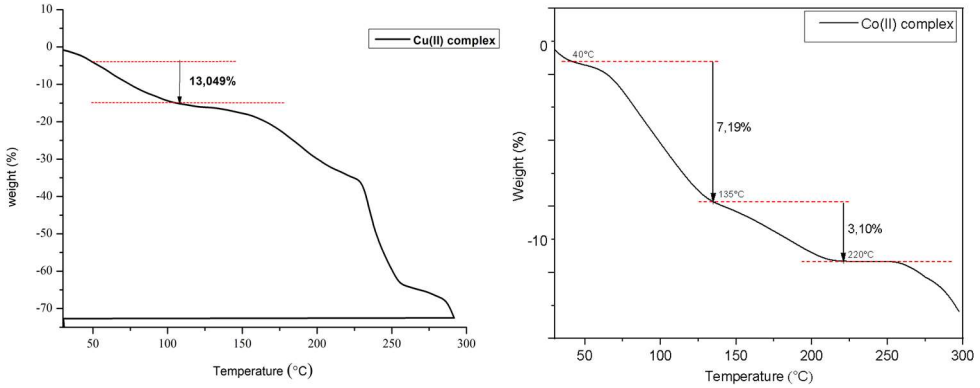

Fig.-1: The TGA Thermogram of Cu(II) (left) and Co(II) (right) Complexes

Electrical Conductivity Analysis

The measurements result of the electrical conductivity of standard solutions and complexes in 1.10^{-3} M DMSO are presented in Table-2. The charge of cation and anion ratio of the Cu(II) complex is indicated as 2:1 (electrolyte), similar to the electrical conductivity of $CuCl_2 \cdot 2H_2O$. The suggested complex formula is $[Cu(MPD)_4](NO_3)_2 \cdot 5H_2O$ with nitrite ion as a counter ion. The Co(II) complex is similar to the electrical conductivity of NaCl, $CuSO_4 \cdot 5H_2O$, and $CoSO_4 \cdot 7H_2O$ which indicated the charge of cation and anion ratio is 1:1. Thus, Co(II) complex is electrolyte and sulphate ion is being counter ion and the suggested formula of complex Co(II) is $[Co(MPD)_2H_2O]SO_4 \cdot 2H_2O$.

Table-2: Standard Solutions' and Complexes' Electrical Conductivity in DMSO (± 1.10^{-3} M)

No.	Solution	Λ_m ($S.cm^2.mol^{-1}$)	Cation: anion
1.	DMSO	-	-
2.	NaCl	2	1:1
3.	$CuSO_4 \cdot 5H_2O$	2	1:1
4.	$CoSO_4 \cdot 7H_2O$	2	1:1
5.	$CuCl_2 \cdot 2H_2O$	18	2:1
6.	$CoCl_2 \cdot 7H_2O$	43	2:1
7.	$NiCl_2 \cdot 6H_2O$	62	2:1
8.	$Cu(NO_3)_2 \cdot 3H_2O$	77	2:1
9.	Cu(II) complex	16	2:1
10.	Co(II) complex	2	1:1

Infrared Spectrum Analysis

Figure-2 and Fig.-3, as well as in Table-3 and Table-4 show the IR spectra of free ligands and complexes. An infrared spectrum of the Cu(II) complex displayed shifting from (N-H) stretch and (N-H) bend to a lower level. ν(N–H) stretch at 3391 cm^{-1}; 3315 cm^{-1}; 3207 cm^{-1} have shifted to 3176 cm^{-1} and ν(N–H) bend has shifted from 1597 cm^{-1} to 1496 cm^{-1}.[15] This showed that MPD is coordinated to Cu(II) by the N atom

of the group N-H. This coordination was corroborated by the existence of one new absorption band at 459 cm^{-1} representing Cu-N.[16]

New absorption band at 3320 cm^{-1} is the absorption of the O-H group by hydrated H_2O molecules.[17] Due to this absorption band, the N-H stretch absorption overlapped, so the N-H stretch absorption became almost invisible because each of the O-H and N-H stretch spectra are adjacent. The Co(II) complex infrared spectrum displayed new absorption at 445 cm^{-1} that indicated Co-N. Infrared absorption at 447-449 cm^{-1} indicated the existence of coordinated N atoms in metal ions.[18] Furthermore, a new absorption at 848 cm^{-1} indicated coordination of H_2O molecule on Co^{2+} metal ion. Infrared absorption at 836-867 cm^{-1} pointed out the presence of H_2O molecules coordinated with metal ions.[19]

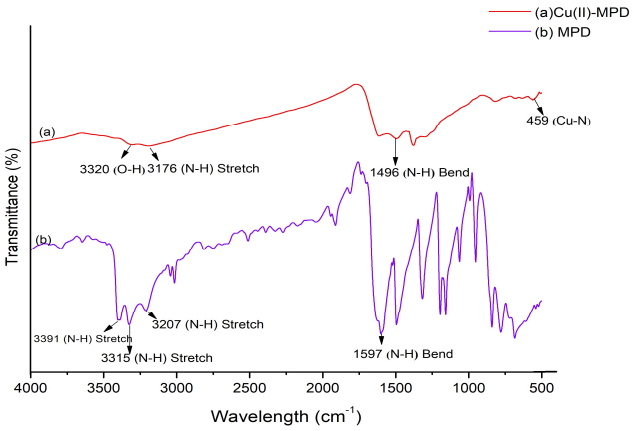

Fig.-2: The Infrared Spectrum of (a) Cu(II) Complex and (b) MPD

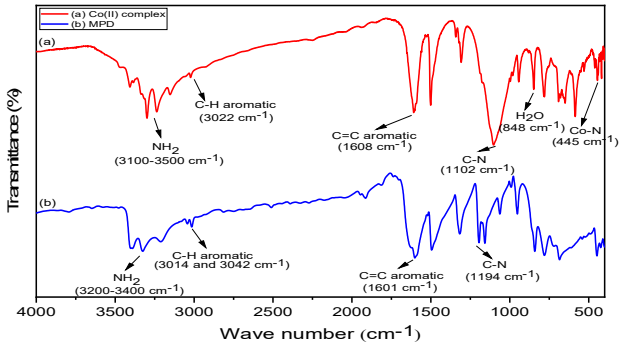

Fig.-3: The Infrared Spectrum of Co(II) Complex (a) and MPD (b)

Table-3: Infrared Absorption of Cu(II) Complex and MPD

No.	Compound	Infrared absorption (cm^{-1})			
		v(Cu-N)	v(N-H) stretch	v(N-H) bend	v(O-H)
1.	MPD	-	3391 3315 3207	1597	-
2.	Cu(II) complex	459	3176	1496	3320

Table-4: Infrared Absorption of Co(II) Complex and MPD

No.	Compound	Infrared absorption (cm^{-1})					
		v(Co-N)	v(Co-H_2O)	v(-NH_2)	v(C-H)	v(C=C)	V(C-N)
1.	Co(II) complex	445	848	3100-3500	3022	1608	1102
2.	MPD	-	-	3200-3400	3014,3042	1601	1194

Electronic Spectrum

The spectrum of Cu(II) complex exhibited one broad peak of 459 nm (21786 cm^{-1}) due to $^2B_{1g} \rightarrow {}^2A_{1g}$ transition, which indicated a geometry square planarly. Co(II) complex has one wide absorption peak at 503 nm (19880 cm^{-1}), due to the overlap between the $^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$ and $^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ transition. This indicated Co(II) complex is near the point of intersection between the energy levels $^4A_{2g}(F)$ and $^4T_{1g}(P)$

in d^7 (Co^{2+}) octahedral field orgel diagram so there seems to be only one absorption peak. Meanwhile, the $^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ transition was not observed because it was around the absorption wavelength of 1200 nm (outside the range of the UV-Vis spectrophotometer). The same thing happened to the complex $[Co(ur)(asn)(H_2O)_2]Cl$ (ur = urea and asn = asparagine) where the $^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ transition could not be observed because of the limited range of UV-Vis spectrophotometer instruments. used (200-1100 nm).[20]

Magnetic Properties

Cu(II) complex's effective magnetic moment (μ_{eff}) is measured to be 1.70 BM. It indicated Cu(II) complex is paramagnetic with an unpair electron. The complex formed is mononuclear.[21] Cu^{2+} is coordinated through the N ligand donor atom from MPD, thus being a four-coordinated square planar. The cobalt complex has an effective magnetic moment (μ_{eff}) of 4.23 BM. It indicated that the complex is paramagnetic with the number of unpaired electrons being three. The value of the effective magnetic moment (μ_{eff}) of the cobalt complex ranges from 4.1-5.2 BM.[22] This supported the complex's trigonal planar geometry with two MPD ligands and one H_2O molecule coordinated to the metal ion Co^{2+}. The probable structures for the complexes are given in Fig.-4.

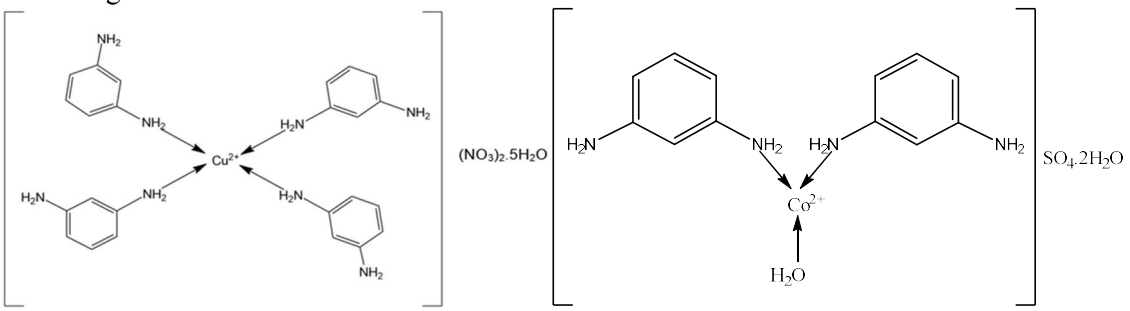

Fig.-4: Approximate Structure of the $Cu(MPD)_4](NO_3)_2 \cdot 5H_2O$ (left) and $[Co(MPD)_2H_2O]SO_4 \cdot 2H_2O$ (right)

Powder X-ray Diffraction

The crystal system can be determined by comparing the three strongest peaks d(Å) of the complex with another three strongest peaks comparing compounds. If the complex has the same d value as the reference compound, then the complex crystal system is the same as the reference compound. Based on Miller's equation, the value of d is equal to the a, b, and c values. If each value of the a, b, and c complex is equivalent to the a, b, and c comparing compounds, then the d value is similar.[23] In this case, the Cu(II) complex does not form crystals. The diffractogram of Co(II) complex is displayed in Fig.-5. There are three strong peaks of Co(II) complex which are presented in Table-5. Peaks of Co(II) complex is not similar to $CoSO_4 \cdot 7H_2O$ which indicated that the complex is formed. The Co(II) complex formed is crystalline.

Table-5: The Three Strongest Peaks of the Co(II) Complex and $CoSO_4.7H_2O$

Compounds	d(Å)			I		
Co(II)-MPD	7.0660	4.7120	3.6678	5202	7669	4676
$CoSO_4 \cdot 7H_2O$	4.8869	3.7609	3.2480	10000	5686	2084

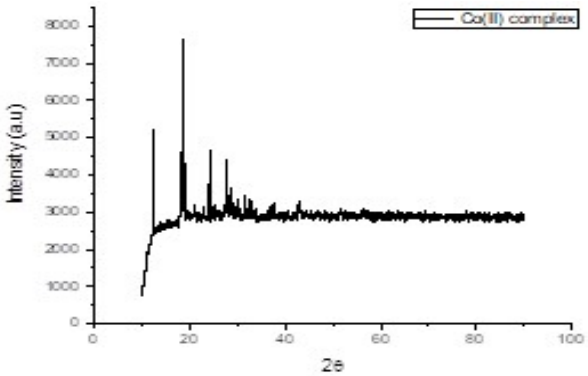

Fig.-5: Powder X-ray Diffractogram of Co(II) Complex

The d(Å) values of the comparing compounds with various crystal systems are shown in Table-6. The d(Å) values of Co(II) complex are comparable to the d(Å) values of the comparing compounds that possess a monoclinic crystal system. Thus, Co(II) complex is monoclinic.

Table-6: The Three Strongest Peaks of the Comparison Compounds with Various Crystal Systems

Compounds	System crystal	d(Å)			I			Ref
$C_{34}H_{28}CoN_{12}O_7$	Monoclinic	7.3769	4.4602	3.3137	10000	9550	9540	[24]
$C_{12}H_{16}Cl_2CoN_8$	Monoclinic	7.3869	5.7637	3.7039	10000	4565	7415	[25]
$C_{28}H_{40}CoN_8O_4C_{l2}$	Triclinic	10.1140	7.2824	4.5733	10000	2949	2717	[26]
$C_{33}H_{38}CoN_8O_{11}$	Triclinic	11.8190	9.0637	9.3943	10000	7744	5303	[27]
$C_{28}H_{26}CoIN_{10}$	Orthorhombic	8.9414	5.6818	3.6792	9062	6669	9982	[24]
$C_{27}H_{31}N_3CoCl_2$	Orthorhombic	9.2846	5.9140	5.3073	10000	3706	3616	[28]
$C_{71}H_{78}Co_2N_8O_9$	Tetragonal	14.8870	10.6100	8.5777	4644	10000	2314	[29]

Antibacterial Activity

In Table-7, the clear area around the paper disc indicates the presence or absence of antibacterial activity. Antibacterial activity test of MPD, metals, and complexes on both *E. coli* and *S. aureus* performed with agar disc diffusion at concentrations of 1000, 500, and 250 g/mL. The results stated that none of them showed antibacterial activity. This is due to the fact that counter ions reduce the solubility of the complex, making it more difficult to reach the bacterial membrane walls.[30]

Table-7: The Outcome of Testing the Complex, Metal Salt, and Free Ligand for Antibacterial Activity

No.	Compounds	Concentration (µg/mL)	Inhibition zone (mm)	
			S. aureus ATCC 25923	*E. coli* ATCC 25922
1.	MPD	1000	6	6
		500	6	6
		250	6	6
2.	$Cu(NO_3)_2{\cdot}3H_2O$	1000	6	6
		500	6	6
		250	6	6
3.	$CoSO_4{\cdot}7H_2O$	1000	6	6
		500	6	6
		250	6	6
4.	$[Cu(MPD)_4](NO_3)_2{\cdot}5H_2O$	1000	6	6
		500	6	6
		250	6	6
5.	$[Co(MPD)_2H_2O]SO_4{\cdot}2H_2O$	1000	6	6
		500	6	6
		250	6	6
6.	DMSO (-)	0	0	0
7.	Gentamicin (+)	10	27.18	23.29

CONCLUSION

$[Cu(MPD)_4](NO_3)_2{\cdot}5H_2O$ and $[Co(MPD)_2H_2O]SO_4{\cdot}2H_2O$ were successfully synthesized by refluxing $Cu(NO_3)_2{\cdot}3H_2O$ and $CoSO_4{\cdot}7H_2O$ with MPD at a mole ratio of 1:4 and 1:2, respectively. The binding of ligands to metal ions was preconcerted by IR spectra, corroborated by TGA and conductivity analysis. Both complexes are electrolytes with nitrate and sulfate ions as counter ions. The proposed geometry for the Cu(II) complex is in the form of square planar while for Co(II) complex it is trigonal planar. These are both paramagnetic complexes. The Co(II) complex's predicted crystal structure is monoclinic. None of them have antibacterial acts in resisting *E. coli* ATCC 25922 and *S.aureus* ATCC 25923.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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