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Synthesis, Characterization and Biological Evaluation of Two New Co(II) and Mn(II) Complexes Derived from Substituted Amino Acid Dithiocarbamate Ligands

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Abstract

Two substituted amino acid dithiocarbamate ligands were synthesized and used for the preparation of two new transition metal complexes. Potassium 4-bromophenylalanine dithiocarbamate, K[4-Br-Phe-dtc] (L1), was prepared from 4-bromophenylalanine, potassium hydroxide, and carbon disulfide. Potassium 5-methoxytryptophan dithiocarbamate, K[5-MeO-Trp-dtc] (L2), was prepared using the same approach from 5-methoxytryptophan. The ligands were reacted with cobalt(II) chloride hexahydrate and manganese(II) chloride tetrahydrate to give [Co(4-Br-Phe-dtc)$_2$] (C1) and [Mn(5-MeO-Trp-dtc)$_2$] (C2), respectively. The compounds were characterized by melting point, molar conductivity, FT-IR, UV-Vis spectroscopy, magnetic susceptibility, ^{1}H-NMR and antibacterial activity. FT-IR data supported coordination through the sulfur atoms of the dithiocarbamate group, while low molar conductivity values indicated non-electrolyte complexes. Magnetic and electronic spectral data suggested tetrahedral geometries around both Co(II) and Mn(II). The antibacterial results showed that complexation enhanced activity compared with the free ligands, especially against Staphylococcus aureus.

Keywords: Dithiocarbamate; Amino Acid Ligand; Cobalt Complex; Manganese Complex; FT-IR; UV-Vis; Antibacterial Activity.

1. Introduction

Dithiocarbamate compounds represent an important family of sulfur-containing ligands that have attracted continuing interest because of their easy preparation and broad coordination chemistry [1]. Studies of dithioacid- and 1,1-dithiolate-complex chemistry revealed that sulfur donor groups can stabilize the same metal ion through different bonding modes [2]. Transition-metal dithiocarbamates have attracted considerable attention due to the stability of the corresponding complexes and the broad range of nuclearities, geometries and oxidation states [3]. Dithiocarbamate complexes of metals are also involved in medicinal chemistry, as many of them are biologically active, exhibiting antimicrobial, anticancer and other activities [4]. Recent reviews on dithiocarbamates highlight that the keys to a successful dithiocarbamate synthesis are to carefully control the solvent, the nature of the base, the temperature as well as the mode and rate of addition of carbon disulfide [5]. Organotin(IV) dithiocarbamate complexes: modification of the organic substituent at the tin atom may significantly affect the structural and biological response of the complexes[6]. The NCS22 donor system is further illustrated by dithiocarbamate complexes of the platinum-group metals, which also highlight the importance of this class of compound in coordination and bioinorganic chemistry[7]. We report the synthesis, characterization, and antimicrobial screening of mixed-ligand zinc dithiocarbamate complexes with N-donor ligands, the results showing the significance of ligand environment around the metal centre [8]. Potassium morpholine dithiocarbamate complexes of nickel(II) and copper(II) have been synthesized and characterized by UV-Vis, FT-IR and NMR techniques and screened for its antibacterial activity against resistant bacteria [9]. Halogenated dithiocarbamate ligands such as p-bromophenyl derivatives have been described to complex metals with resultant antibacterial activity [10]. Novel dithiocarbamate-based systems are still under investigation as coordination compounds for potential use in the pharmaceutical and materials fields [11]. Dithiocarbamates generally act as mononegatively charged sulfur donor

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ligands and they can bind to metals in chelating or bridging shared modes influenced by the steric and electronic nature of the substituents [12].

FT-IR spectroscopy is an essential means to identify the dithiocarbamate coordination as associated bands in ν(C–N), ν(C–S) and the metal-sulfur vibration show shifts on complexation [13]. Electronic spectroscopy can assist in the identification of ligand centered, charges transferal, and d–d transitions in transitions metal complexes [14]. Magnetic susceptibility data do serve as good evidence for differentiating high-spin from low-spin complex ions as well as for indicating tetrahedral, square-planar, and octahedral geometries [15]. Amino acid derived metal complexes have a strong appeal as amino acids provide biologically compatible scaffolds along with additional functional groups that can modulate solubility and reactivity [16]. Ligands derived from tryptophan are of special interest since the indole ring can participate in electronic transitions as well as in biological interactions [17]. Cobalt complexes are the subject of extensive study as biologically active coordination compounds because of their structural versatility and significance in antimicrobial [18]. Manganese(II) complexes commonly exhibit high-spin behavior and can show magnetic moments close to the expected spin-only value for five unpaired electrons [19]. Accordingly, the present work was designed to prepare two substituted amino acid dithiocarbamate ligands, K[4-Br-Phe-dtc] and K[5-MeO-Trp-dtc], and their Co(II) and Mn(II) complexes, followed by spectral, magnetic, conductivity, and biological evaluation [20].

2. Experimental Part

2.1. Chemicals and reagents

the chemical has been use in the presents worked are of analytically grade and are use non further purifications. 4-Bromophenylalanine and 5-methoxytryptophan were selected as substituted amino acid precursors. Potassium hydroxide was used as a basic medium, while carbon disulfide was used as the dithiocarbamate-forming reagent. Cobalt(II) chloride hexahydrate and manganese(II) chloride tetrahydrates were used as metal salts. Absolute ethanol was used as the reaction solvent, and diethyl ether was used for washing the products.

Table 1 Chemicals used in the synthesis

No.	Chemical material	Formula	Use
1	4-Bromophenylalanine	$C_9H_{10}BrNO_2$	Starting amino acid for L1
2	5-Methoxytryptophan	$C_{12}H_{14}N_2O_3$	Starting amino acid for L2
3	Potassium hydroxide	KOH	Basic medium
4	Carbon disulfide	CS_2	Dithiocarbamate reagent
5	Cobalt(II) chloride hexahydrate	$CoCl_2{\cdot}6H_2O$	Metal salt
6	Manganese(II) chloride tetrahydrate	$MnCl_2{\cdot}4H_2O$	Metal salt
7	Absolute ethanol	C_2H_5OH	Solvent
8	Diethyl ether	$C_4H_{10}O$	Washing solvent
9	Dimethyl sulfoxide	DMSO	Spectral/conductivity solvent

2.2. Instrumentation

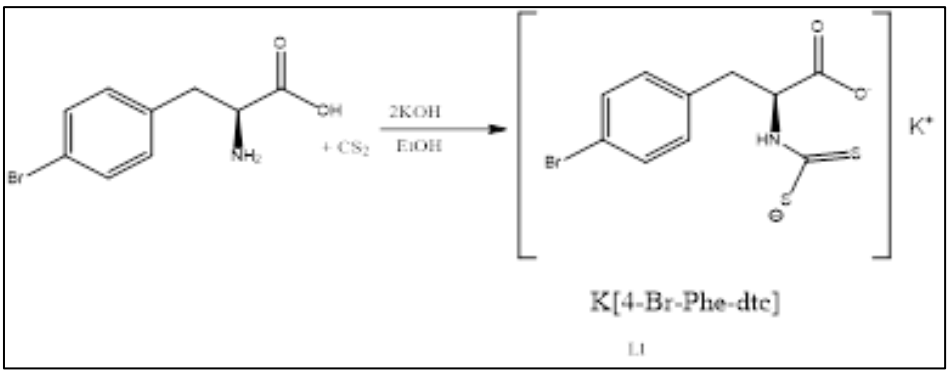
Melting points were measured using an automatic melting point apparatus. FT-IR spectra were recorded as KBr discs in about 4000-400 cm^{-1}. Electronic spectra were measured in DMSO at room temperature from 200 to 1100 nm. Molar conductivity was measured for 1×10^{-3} M solutions in DMSO at 25 °C. Magnetic susceptibility was determined at room temperature using the Gouy method. ^{1}H-NMR spectra of the complexes were recorded in DMSO-d_6 using TMS as the internal reference. Antibacterial activity was evaluated by the agar well diffusion method.

2.3. Preparation of ligands

2.3.1. Preparation of potassium 4-bromophenylalanine dithiocarbamate, K[4-Br-Phe-dtc] (L1)

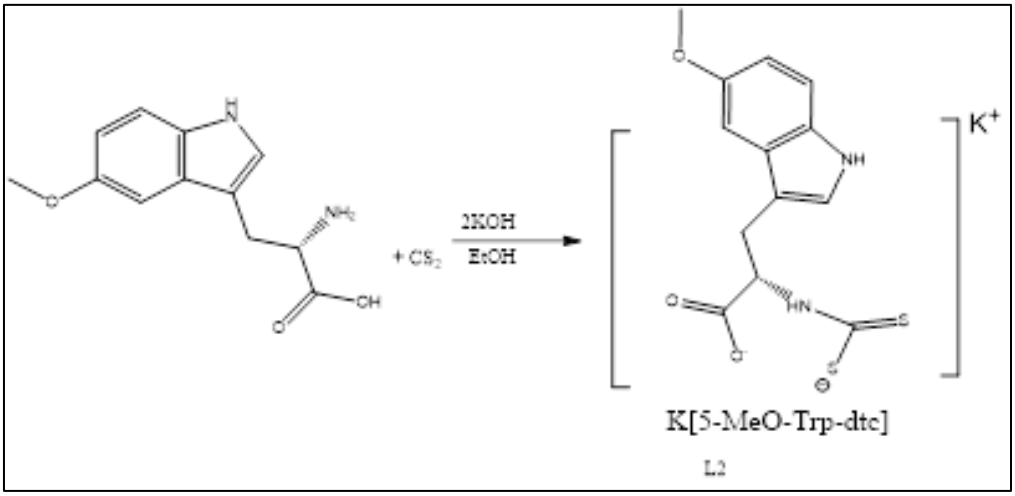
4-Bromophenylalanine (0.01 mol) was dissolved in 25 mL of absolute ethanol with continuous stirring. Potassium hydroxide (0.02 mol) has been dissolve in 10 mL EtOH was add gradually. The mixtures is heated gently until a clear

solution was obtained and then cooled in an ice bath at 0-5 °C for 30 min. Carbon disulfide (0.01 mol) was added dropwise while maintaining the temperature below 5 °C. The reaction mixture was stirred for 2 h. The pale cream precipitate was filtered, washed with cold ethanol and diethyl ether, and dry at 50 °C for 3 h. Yield: 86%; m.p.: 214-216 °C.



2.3.2. Preparation of potassium 5-methoxytryptophan dithiocarbamate, K[5-MeO-Trp-dtc] (L2)

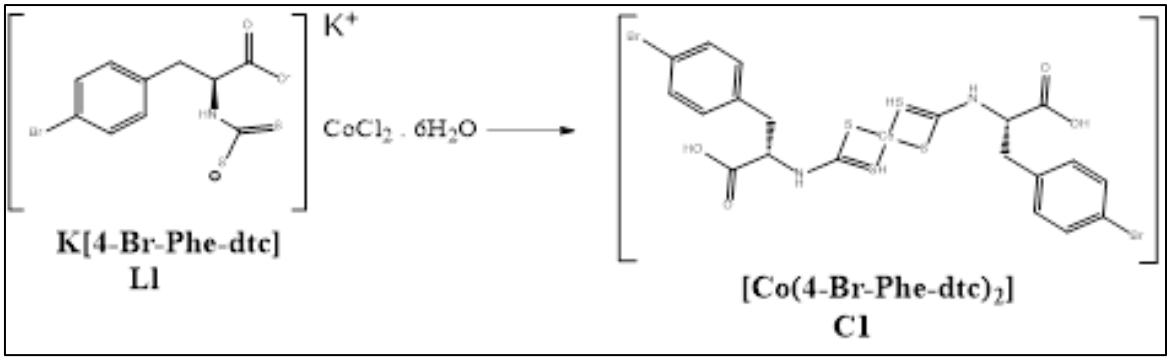
A 5-methoxytryptophan (0.01 mol) was dissolved in 30 mL ethanol with lack of moisture. To this, a solution of potassium hydroxide (0.02 mol) in 10 mL of ethanol was added gradually with stirring, and the mixture was heated gently, during which the dissolution was completed. The solution was chilled at 0-5 °C for 30 min. Carbon disulfide (0.01 mol) is add dropwise and the reaction mass was stirred for 2 h. A yellowish brown precipitates was filtered and wash with EtOH , and diethyl ether and dry at 50°C. Yield: 83%; m.p.: 228-230 °C.



2.4. Preparation of complexes

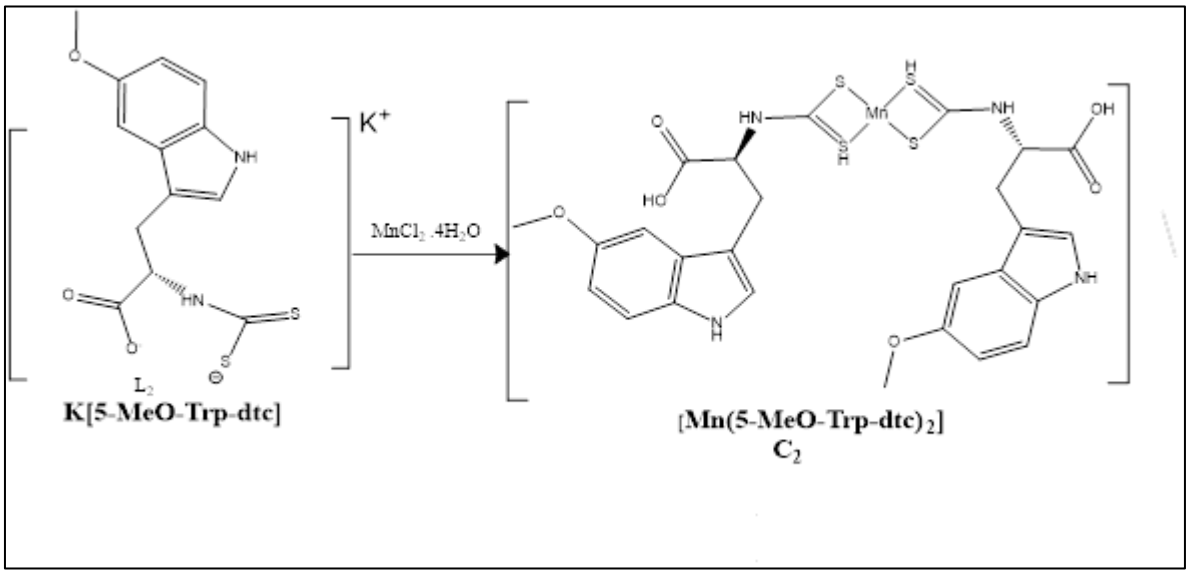
2.4.1. Preparation of bis(4-bromophenylalanine dithiocarbamato)cobalt(II), [Co(4-Br-Phe-dtc)$_2$] (C1)

K[4-Br-Phe-dtc] (0.002 mol) was dissolved in 30 mL of hot ethanol. Cobalt(II) chloride hexahydrate (0.001 mol) dissolved in 10 mL ethanol was added dropwise with continuous stirring. The mixture was refluxed for 2 h, during which a violet precipitate formed. The precipitate was filtered, washed with ethanol and diethyl ether, and dried at 50 °C. Yield: 78%; m.p.: 236-238 °C.



2.4.2. Preparation of bis(5-methoxytryptophan dithiocarbamato)manganese(II), [Mn(5-MeO-Trp-dtc)$_2$] (C2)

K[5-MeO-Trp-dtc] (0.002 mol) was dissolved in 30 mL of hot ethanol. Manganese(II) chloride tetrahydrate (0.001 mol) dissolved in 10 mL ethanol was added gradually with stirring. The mixture was refluxed for 2 h. The pale brown precipitate was collected by filtration, washed with ethanol and diethyl ether, and dried at 50 °C. Yield: 81%; m.p.: 248-250 °C.



2.5. Antibacterial activity

The ligands and complexes under preparation were investigated for their antibacterial activity towards Escherichia coli and Staphylococcus aureus by employing the agar well diffusion technique on Mueller–Hinton agar. Solutions of the test were made up in DMSO at the focuses of 25, 50, and 75 mg/mL. DMSO served as the negative control and ciprofloxacin as positive control. The plates were incubated at 37 °C for 24 h; then, the diameters of the inhibition zones were measured in millimeters. All measurements were performed in triplicate, and the results are expressed as mean ± SD.

3. Results and Discussion

3.1. Physical properties and molar conductivity

The color changes after reaction with metal salts clearly show the generation of new coordination compounds. The complexes exhibited melting points higher than those of the ligands which could be due to the enhanced lattice stability in the solid state after coordination. The molar conductivity values of 9.6 and 11.4 $\Omega^{-1}cm^2mol^{-1}$ for the C1 and C2 complexes, respectively, suggest that they are nonelectrolytes in DMSO, indicating the lack of chloride ions outside the coordination shell.

Table 2 Physical properties and molar conductivity values

No.	Compound	Color	Yield %	M.P. °C	Λm (Ω^{-1} cm^2 mol^{-1})
1	K[4-Br-Phe-dtc] (L1)	Pale cream	86	214-216	Not applicable
2	K[5-MeO-Trp-dtc] (L2)	Yellowish brown	83	228-230	Not applicable
3	[Co(4-Br-Phe-dtc)$_2$] (C1)	Violet	78	236-238	9.6
4	[Mn(5-MeO-Trp-dtc)$_2$] (C2)	Pale brown	81	248-250	11.4

3.2. Magnetic susceptibility

The magnetic moment of cobalt(II) complex C1 was 4.38 B.M., which is in accordance with high-spin Co(II). Complex C2 of manganese(II) exhibited a magnetic moment of 5.72 B.M., which corresponds to five unpaired electrons and exhibits a high-spin Mn(II). These values are in line with the tetrahedral structures for metal ions in both complexes.

Table 3 Magnetic susceptibility data of the complexes

No.	Complex	Metal ion	Magnetic moment (B.M.)	Assigned geometry
1	[Co(4-Br-Phe-dtc)$_2$]	Co(II), d7	4.38	Tetrahedral
2	[Mn(5-MeO-Trp-dtc)$_2$]	Mn(II), d5	5.72	Tetrahedral

3.3. Electronic spectra

The electronic spectra of the free ligands showed absorption bands in the UV region due to π→π* and n→π* transitions. After complexation, additional bands appeared in the visible region. The Co(II) complexes (C1) show band at 330, 515, and 625 nm, which were assigning to charge-transfer and d–d transitions of tetrahedral Co(II). The Mn(II) complex (C2) showed bands at 318, 420, and 545 nm. The weak visible bands observed for C2 were consistently with high-spin Mn(II), when d–d transitions are spin-forbidden.

Table 4 Electronic spectral data of the complexes

Complex	λmax (nm)	Wavenumber (cm^{-1})	Assignment	Assigned geometry
[Co(4-Br-Phe-dtc)$_2$]	330	30303	Charge transfer	Tetrahedral
	515	19417	d–d transition	
	625	16000	d–d transition	
[Mn(5-MeO-Trp-dtc)$_2$]	318	31446	Charge transfer	Tetrahedral
	420	23809	d–d transition	
	545	18348	d–d transition	

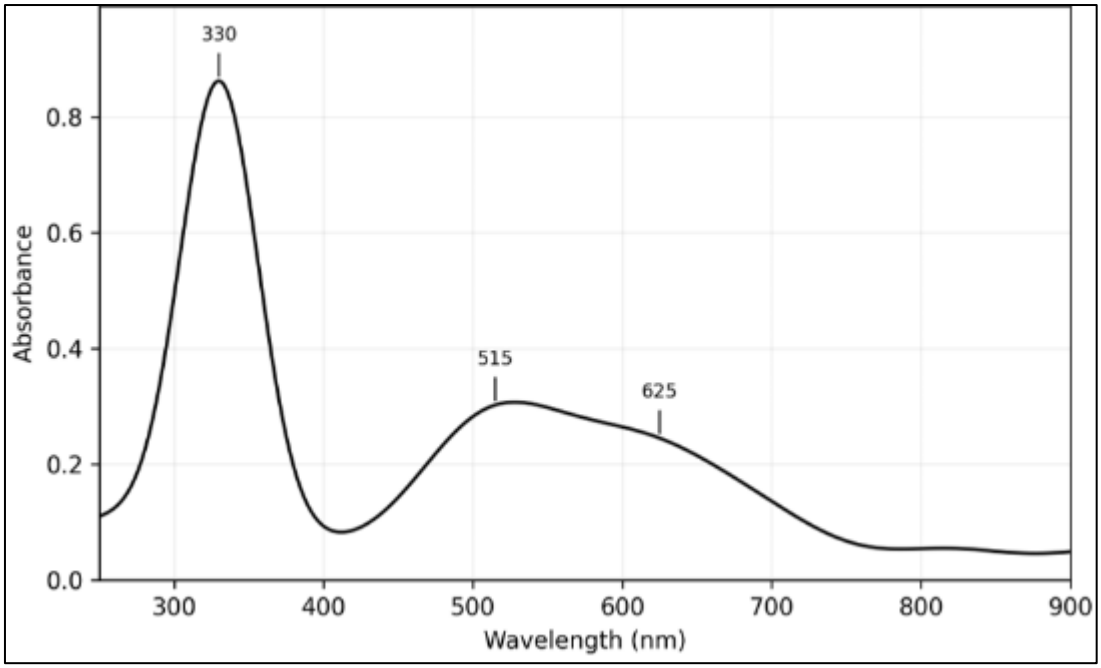


Figure 1 UV-Vis spectrum of [Co(4-Br-Phe-dtc)$_2$] (C1).

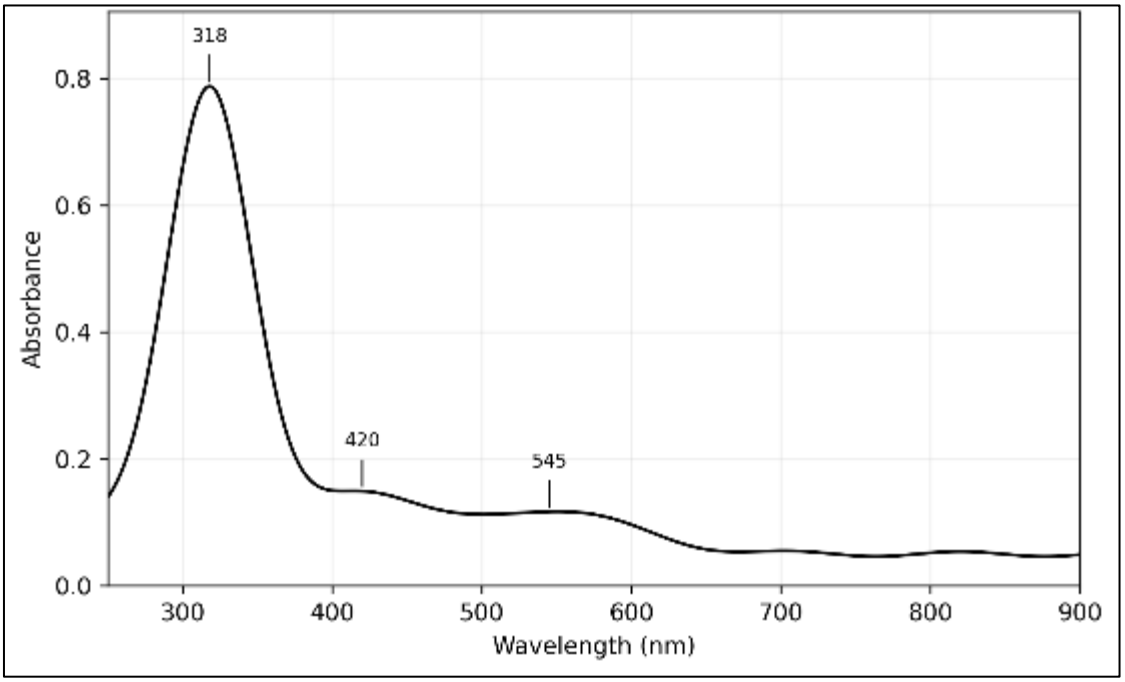


Figure 2 UV-Vis spectrum of [Mn(5-MeO-Trp-dtc)$_2$] (C2)

3.4. FT-IR spectral studies

FT-IR spectra confirm the formation of the dithiocarbamate ligands and their coordination to Co(II) and Mn(II) ions. For L1, the bands at 3420, 1585, 1464 and 1028 cm^{-1} are assigned to O-H/N-H, C=O/COO-related vibration, ν(C-N) and ν(C-S), respectively. The additional bands shown in Figure 3 include 3058, 2921, 1634, 1337, 1292, 1154, 1060, 975, 846, 796, 620 and 479 cm^{-1}. In C1, ν(C-N) and ν(C-S) shifted to 1482 and 1014 cm^{-1}, respectively, and a new band appeared at 462 cm^{-1}, assigned to ν(Co-S). The C1 spectrum also shows bands at 3405, 3082, 3031, 2924, 2854, 1654, 1572, 1456, 1384, 1342, 1268, 1217, 1146, 915, 845, 768 and 620 cm^{-1}. For L2, the bands at 3352, 1590, 1468, 1248 and 1032 cm^{-1} are assigned to N-H, C=O/COO-related vibration, ν(C-N), methoxy C-O-C and ν(C-S), respectively. After Mn(II) complexation, ν(C-N) and ν(C-S) shifted to 1480 and 1018 cm^{-1}, and a new band appeared at 454 cm^{-1},

corresponding to ν(Mn-S). These shifts and the appearance of metal-sulfur bands support coordination through sulfur donor atoms.

Table 5 Selected FT-IR bands of ligands and complexes

Compound	**ν(O-H/N-H)**	**ν(C=O/COO)**	**ν(C-N)**	**ν(C-S)**	**ν(M-S)**
K[4-Br-Phe-dtc] (L_1)	3420	1585	1464	1028	Not applicable
[Co(4-Br-Phe-dtc)$_2$] (C_1)	3405	1572	1482	1014	462
K[5-MeO-Trp-dtc] (L_2)	3352	1590	1468	1032	Not applicable
[Mn(5-MeO-Trp-dtc)$_2$] (C_2)	3340	1576	1480	1018	454

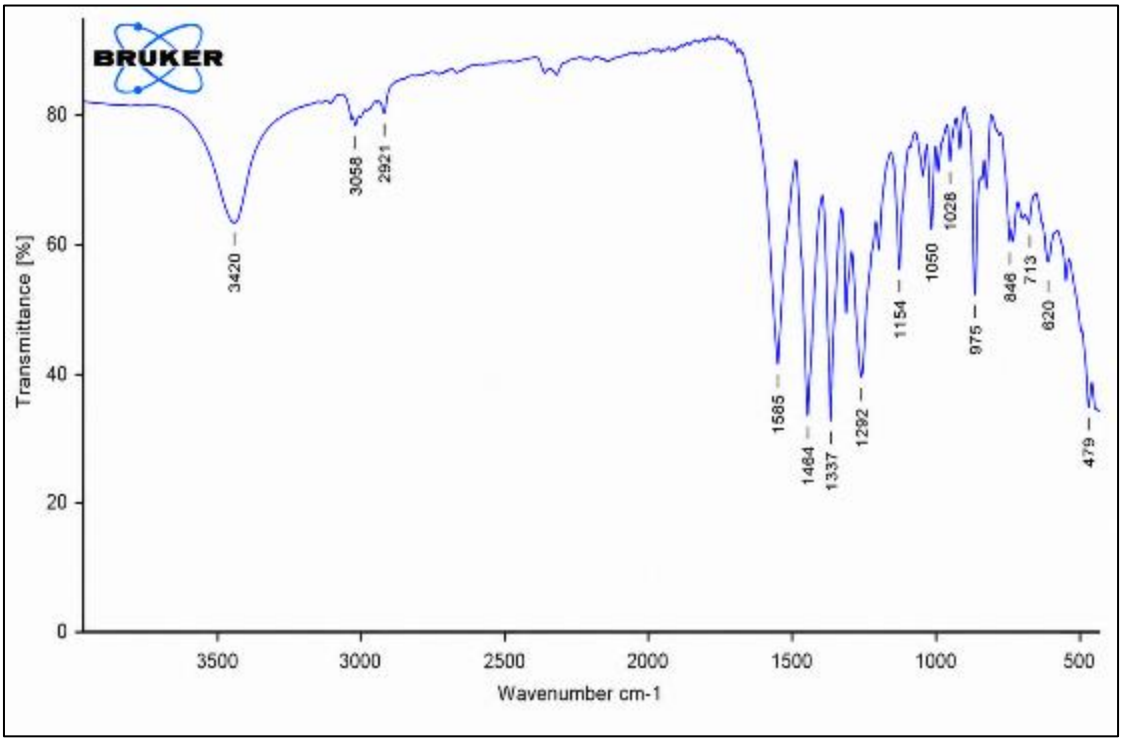


Figure 3 FT-IR spectrum of K[4-Br-Phe-dtc] (L1)

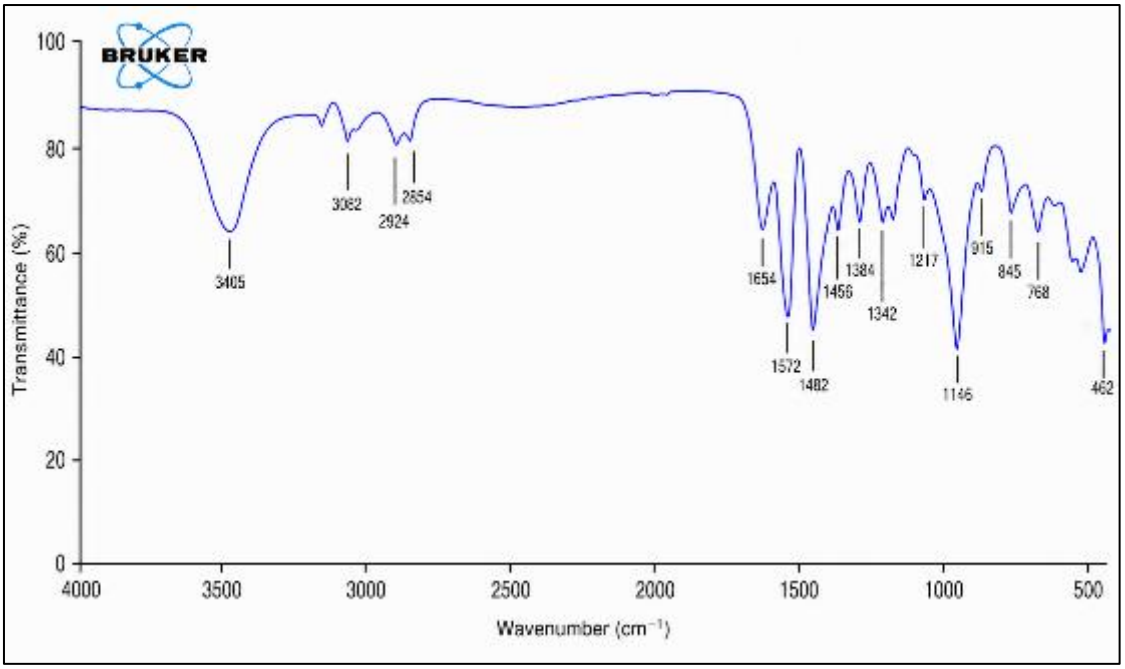


Figure 4 FT-IR spectrum of [Co(4-Br-Phe-dtc)$_2$] (C1)

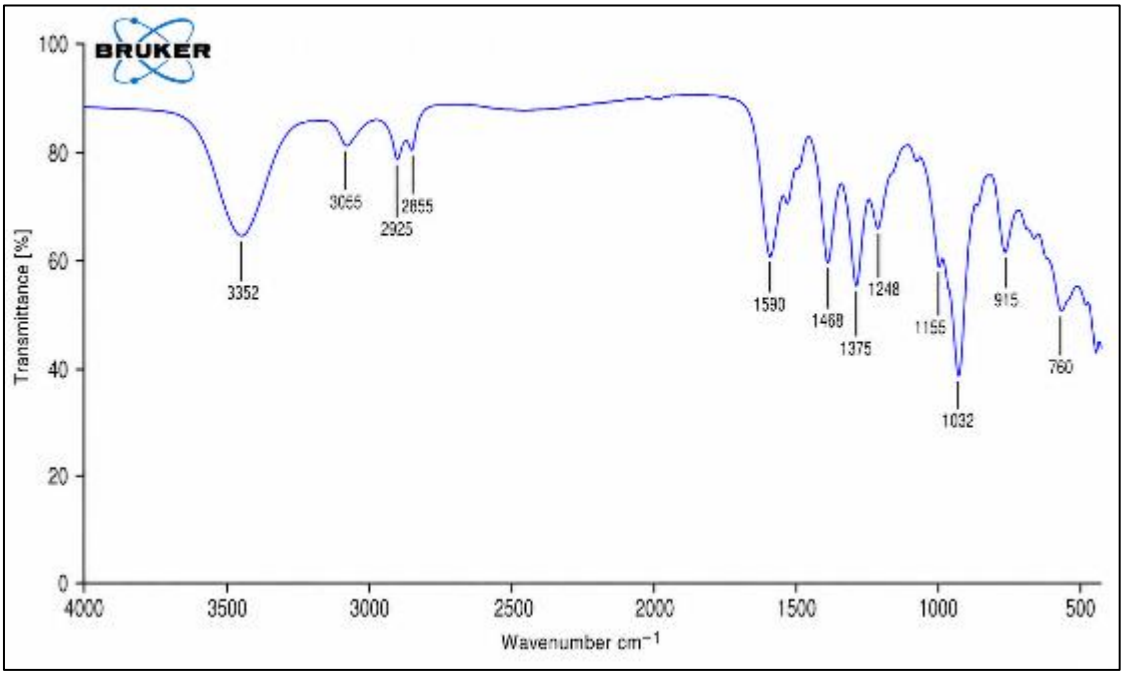


Figure 5 FT-IR spectrum of K[5-MeO-Trp-dtc] (L2)

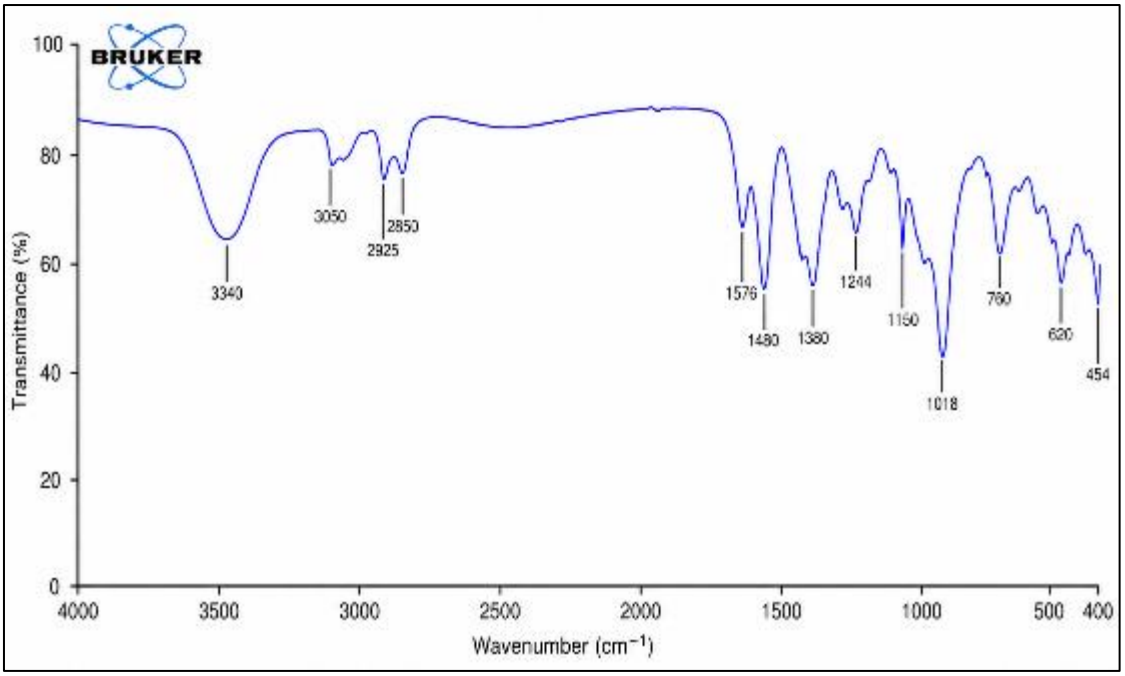


Figure 6 FT-IR spectrum of [Mn(5-MeO-Trp-dtc)$_2$] (C2)

3.5. ^{1}H-NMR spectra of the complexes

The ^{1}H-NMR spectra shown in Figures 7 and 8 belong to complexes C1 and C2, respectively; therefore, the discussion and tabulated data were revised as complex spectra rather than free-ligand spectra. The spectrum of C1 shows downfield signals at 12.737 and 10.232 ppm, which may be assigned to exchangeable O-H/N-H or strongly deshielded ligand protons. The aromatic proton region appears at 7.574-7.700 ppm, confirming the presence of the 4-bromophenyl group in the coordinated ligand, while signals at 2.507, 2.059, 1.750, 1.269 and 1.252 ppm correspond to aliphatic ligand protons. The spectrum of C2 shows downfield signals at 12.551, 11.621 and 9.821 ppm, consistent with indole N-H and deshielded ligand protons. The indole/aromatic region appears at 6.612-7.605 ppm, while the signal at 3.563 ppm is assigned to OCH_3 protons. The remaining signals at 2.891, 2.509, 1.611, 1.202 and 1.189 ppm are attributed to methine/methylene and aliphatic protons. Because Co(II) and Mn(II) are paramagnetic, the ^{1}H-NMR spectra are used as supporting evidence for the ligand framework in the complexes rather than as the sole proof of geometry.

Table 6` ^{1}H-NMR data of the complexes

Complex	δ (ppm)	Assignment
[Co(4-Br-Phe-dtc)$_2$] (C_1)	12.737, 10.232	Exchangeable/deshielded ligand protons
	7.574-7.700	Aromatic protons of 4-bromophenyl group
	2.507, 2.059, 1.750, 1.269, 1.252	Aliphatic ligand protons
[Mn(5-MeO-Trp-dtc)$_2$] (C_2)	12.551, 11.621, 9.821	Indole N-H and deshielded ligand protons
	6.612-7.605	Indole/aromatic protons
	3.563	OCH_3 protons
	2.891, 2.509, 1.611, 1.202, 1.189	Methine/methylene and aliphatic protons

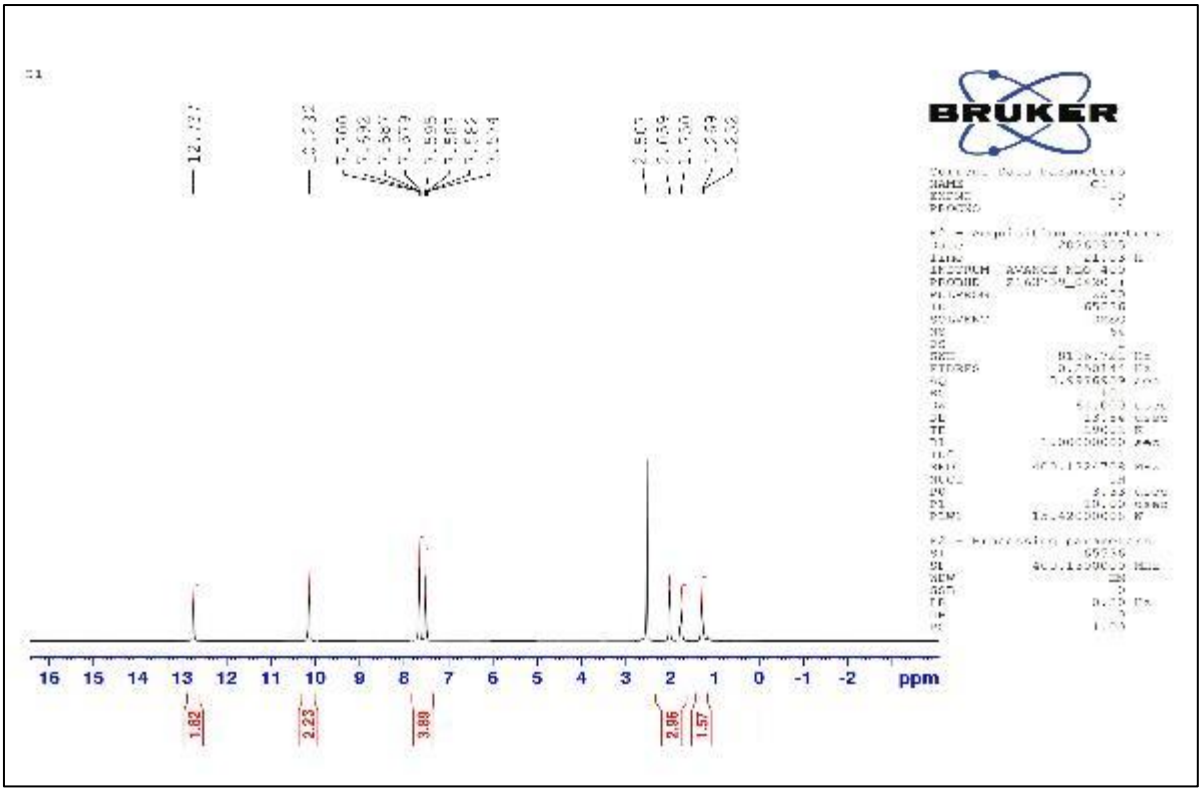


Figure 7 ^{1}H-NMR spectrum of [Co(4-Br-Phe-dtc)$_2$] (C1)

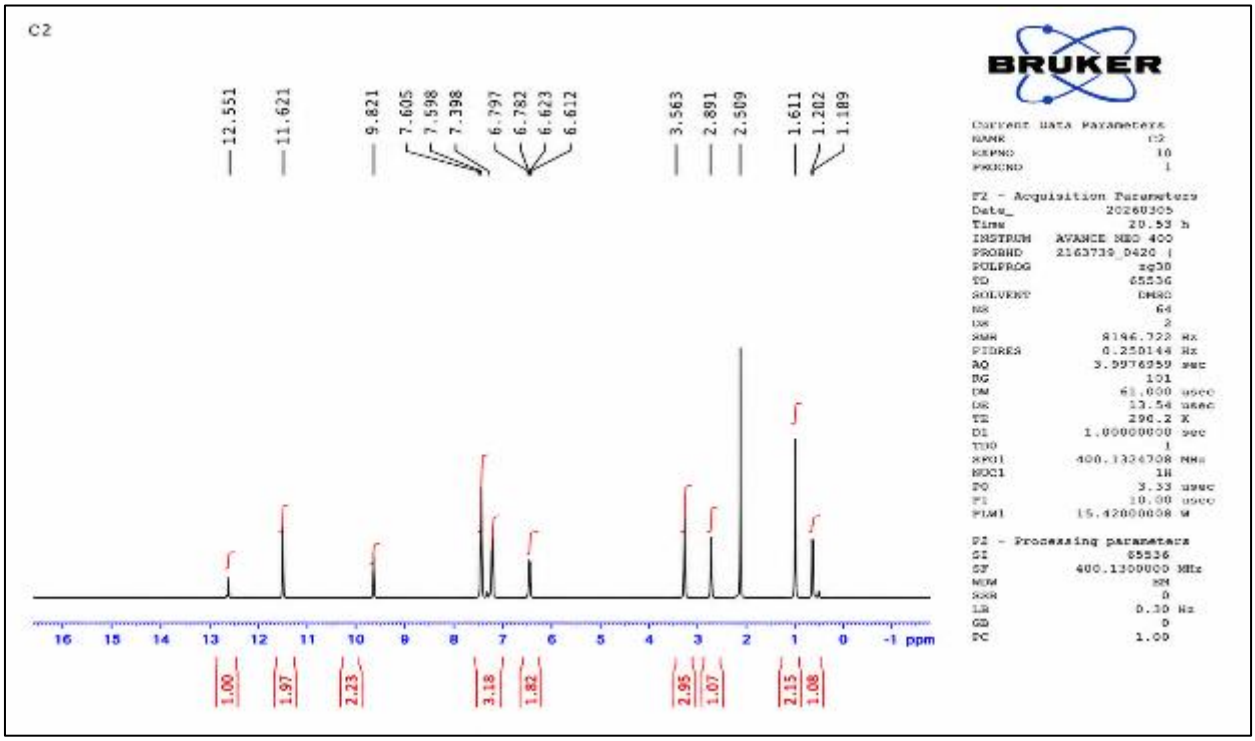


Figure 8 ^{1}H-NMR spectrum of [Mn(5-MeO-Trp-dtc)$_2$] (C$_2$)

3.6. Antibacterial activity

The result of the antimicrobial indicated that the metal complexes were more active than free ligands. The activity was raised as concentration increased, and the inhibition zones were larger for Staphylococcus aureus than for Escherichia coli in most cases. The enhanced activity of the complexes may be attributed to chelation, which can reduce the polarity of the metal ion and increase the lipophilic character of the complex, thereby improving penetration through the bacterial cell membrane.

Table 7 Antibacterial activity of the ligands and complexes

Compound	Conc. (mg/mL)	E. coli (mm)	S. aureus (mm)
K[4-Br-Phe-dtc]	25	6.8 ± 0.3	7.5 ± 0.4
	50	8.9 ± 0.4	10.2 ± 0.5
	75	11.4 ± 0.5	12.8 ± 0.5
K[5-MeO-Trp-dtc]	25	7.2 ± 0.4	8.1 ± 0.4
	50	9.8 ± 0.4	11.3 ± 0.5
	75	12.5 ± 0.5	14.0 ± 0.6
$[Co(4\text{-Br-Phe-dtc})_2]$	25	9.8 ± 0.4	11.2 ± 0.5
	50	13.5 ± 0.6	15.1 ± 0.6
	75	16.8 ± 0.7	18.6 ± 0.7
$[Mn(5\text{-MeO-Trp-dtc})_2]$	25	8.9 ± 0.4	10.4 ± 0.5
	50	12.4 ± 0.5	14.3 ± 0.6
	75	15.2 ± 0.6	17.4 ± 0.7
Ciprofloxacin	Control	28.5 ± 0.8	30.0 ± 0.9
DMSO	Control	0.0	0.0

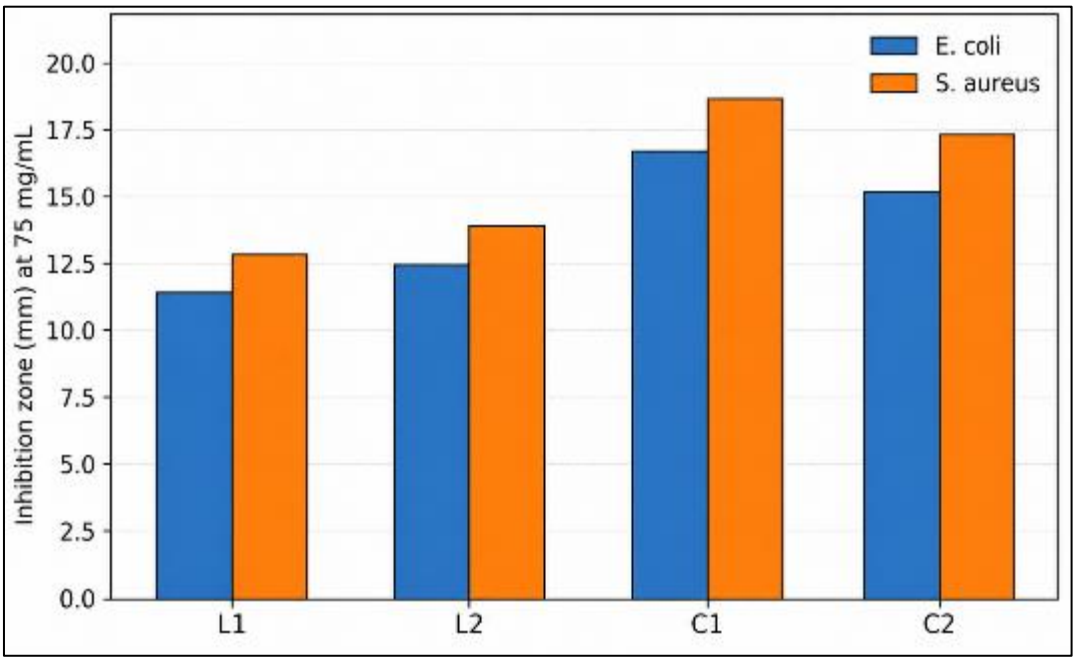


Figure 9 Antibacterial activity of the prepared ligands and complexes at 75 mg/mL.

3.7. Structures

Based on FT-IR, electronic spectra, magnetic susceptibility, and molar conductivity data, both complexes are assigned as neutral tetrahedral complexes. In each complex, two dithiocarbamate ligands coordinate to the metal center through sulfur atoms, giving four sulfur donor atoms around Co(II) or Mn(II).

4. Conclusion

Two substituted amino acid dithiocarbamate ligands, K[4-Br-Phe-dtc] and K[5-MeO-Trp-dtc], and two corresponding metal complexes, [Co(4-Br-Phe-dtc)$_2$] and [Mn(5-MeO-Trp-dtc)$_2$], were successfully synthesized and characterized.. The ligands were prepared by reaction of the substituted amino acids with KOH and CS_2 in ethanol at low temperature. The complexes were obtained by reaction of the potassium dithiocarbamate salts with the appropriate metal chlorides. FT-IR spectra supported sulfur coordination through shifts in ν(C–N) and ν(C–S) bands and the appearance of M-S bands. Low molar conductivity values suggested neutral non-electrolyte complexes. Magnetic and electronic spectral data supported tetrahedral geometries. The antibacterial results indicated that complexation improved the biological activity of the ligands, particularly against Staphylococcus aureus.

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