

Formation, Characterization of Some New Coⁱⁱ, Cuⁱⁱ, Niⁱⁱ, Auⁱⁱⁱpotassium (2-Carboxyphenyl)(2,3-Dimethylphenyl) Carbamodithioate complexes and Study Their Biological Activity

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Abstract. A new ligand (potassium (2-carboxyphenyl)(2,3-dimethylphenyl)carbamodithioate) is being synthesized and characterized. which has two sulfur donor atoms in it. The ligand was made by reacting one equivalent of carbon disulfide and 2-((2,3-dimethylphenyl)amino)benzoic acid for 4 hours in ethanol (as a solvent) in the presence of an alkali base (KOH). The FT-IR, ¹H NMR, ¹³C-NMR, (C.H.N.S), UV-visible, and ESI-mass range, as well as the melting point, were used to describe the dithiocarbamate ligand. Four metal complexes are prepared by refluxing. The complexes were made by reacting two equivalents of potassium dithiocarbamate ligand with one equivalent metal salt for four hours in refluxing ethanol. For certain complexes, FT-IR, UV-Visible, molar conductivity, magnetic susceptibility, solubility of flammable atomic absorption spectroscopy, melting point, element microanalysis, and mass spectroscopy were used to characterize them. The biological effectiveness of the prepared dithiocarbamate ligand and the complexes was investigated using the inhibition method in four different bacteria: two Gram positive and two Gram negative bacteria, as well as one pathogenic fungus.

Keywords: Dicarbodithioate, Mefenamic acid, Synthesis.

Introduction

DTCs (dithiocarbamates) are a class of small organic molecules that have a good ability to chelate metal ions. [1]. There have been several studies and reviews on transition and non-transition metals, suggesting a broad variety of anion compounds. [2]. Organosulfur compounds with dithiocarbamate ligands are more complex. This may be due to the CSS group's low bite angle, which allows it to interact with most metals in the periodic table and has sulfur atoms with the ability to add a single pair of electrons to central metal atom to form complexes [3]. DTCs can be stabilized in a variety of metal oxidation states, coordination geometries, and compounds with a wide range of structural variations, from monomeric to polymeric molecular assemblies. The physical and chemical properties of the resulting metal complexes will be determined by the binding properties of dithiocarbamates; monodentate, bidentate chelating, and bidentate bridging varieties [4]. Dithiocarbamate and dithiophosphate complexes have significant applications. [5] Dithiocarbamates are essential materials that have been studied extensively in coordination chemistry, ((medicine and radiopharmaceutical chemistry)), materials science, sensing engineering, and industry. DTCs have shown a wide range of biological effectiveness, inclusive, antibacterial activity [6].

Materials

Commercially available laboratory chemicals and reagents may be used without further purification.

measurements

For ligands and their metal complexes, an element analyzer (C. H. N. S.) was used (Euro EA 3000). Infrared spectra of potassium bromide disks were taken on a Biotic 600 FT-IR spectrophotometer in the range 4000-400 cm^{-1} , and potassium iodide disks in the range 4000-200 cm^{-1} on a Shimadzu 8400s FT-IR. Electronic spectra were obtained at room temperature using a Shimadzu 1800 (UV-Vis) spectrophotometer from 200 to 1000 for 10⁻³ M solutions in dimethyl sulfoxide using quartz cells that were 1.0 cm long. Mass spectra for ligand were obtained using the Shimadzu GC-Mass QPA spectrometer, and mass spectra for complexes were obtained using the Shimadzu LC-MSQP50A Shimadzu (E170Ev) spectrophotometer. Glucosamine Brucker, 400 MHz Ultra Shield model, Germany root, was used to use nuclear magnetic resonance spectra in Deuterated chloroform. The metal signification of complexes is determined using the atomic absorption (A.A) technique and a Shimadzu (A.A 680 GBC 933 plus) spectrophotometer. Glucosamine Brucker, 400 MHz Ultra Shield model, Germany root, was used to use nuclear magnetic resonance spectra in Deuterated chloroform. The metal signification of complexes is determined using the atomic absorption (A.A) technique and a Shimadzu (A.A 680 GBC 933 plus) spectrophotometer.

Synthesis the ligand

Ligands are made according to a published method [7] with a few tweaks, as discussed below:

Synthesis of potassium (2-carboxyphenyl)(2,3-dimethylphenyl)carbamodithioate

2-((2,3-dimethylphenyl)amino)benzoic acid (0.482g, 0.002 mol) was dissolved in (12 ml) ethanol. potassium hydroxides (0.112 g, 0.002mol) dissolved in double distilled water (1ml) with 30 minutes of stirring at room temperature, solution cool in the Snow bath. Pure CS₂ (0.152g, (0.002mol) be added to the commixture drop-wise with regular stirring kept at temperature (0-4 ° C) for 4 hours. The brown solution was allowed at room temperature and then evaporated before completing solid brown precipitation. The brown solid was collected by filtration and then recrystallized from methanol, washed with diethylether (10ml) twice and then dried at temperature (35 ° C) for 7 hours.

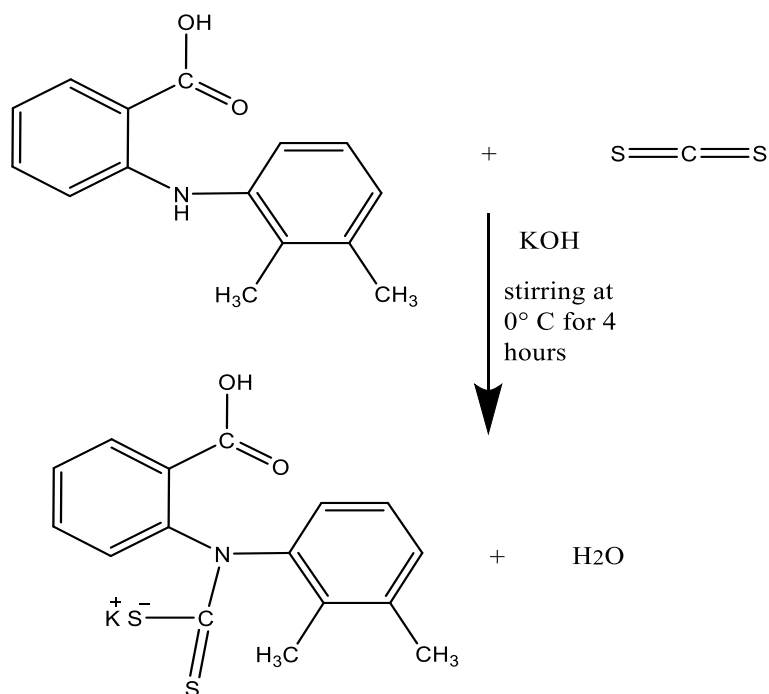
Synthesis of potassium (2-carboxyphenyl)(2,3-dimethylphenyl)carbamodithioate complexes

A general method for achieving dithiocarbamate complexes was used and as follows: Complexes were synthesis by the reaction of (2 potassiumdithiocarbamate salt) equivalent (DTC) as ligand in ethanol with 1 metal salt equivalent as central ion (2:1). Metal chloride solution in 10ml absolute ethanol has been added to a round bottom container, volume (100) ml containing a dithiocarbamate ligand salt solution Meltedat (10) ml ethanol. The commixture of the reaction was shaky and warmed for 4 h under reflux. Filtration was used to bank up the complex, which was then cleaned. with excess methanol and diethyl ether, dried for 48 h [8,9].

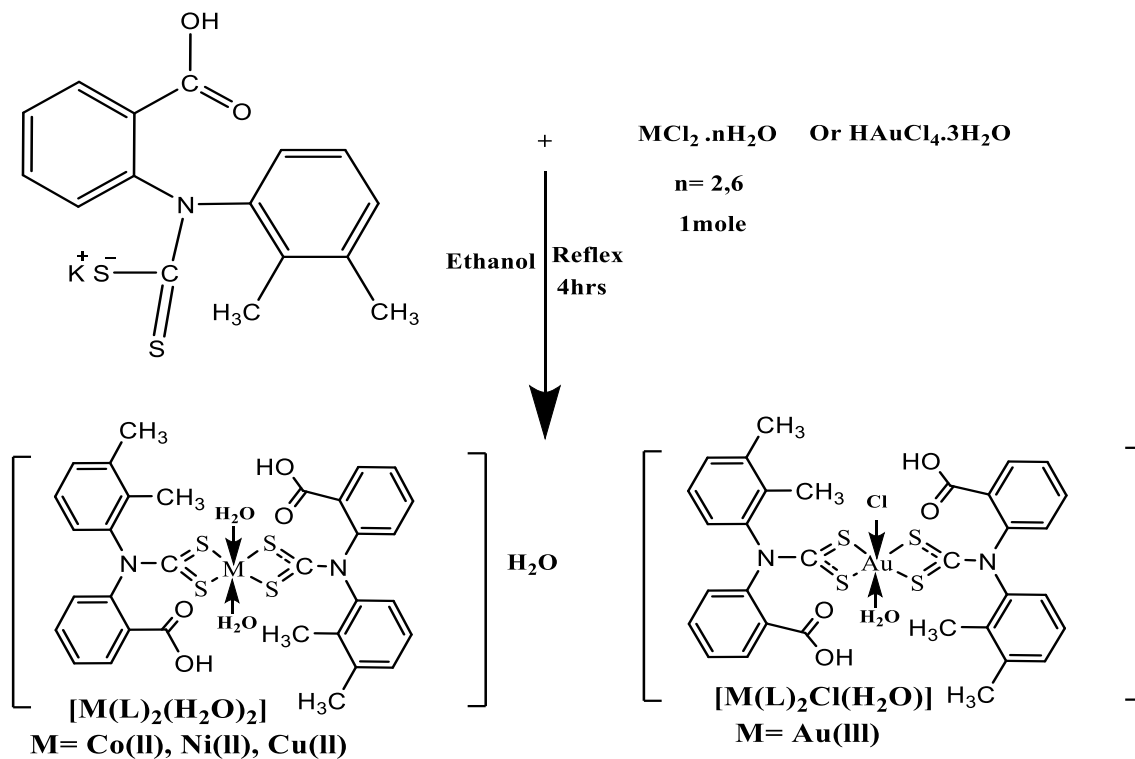
Results

The dithiocarbamate ligand was identified in this study. The prepare of this ligand was based on the reaction obtained between NH group with CS₂. (KL) was synthesized by combining 2-((2,3-dimethylphenyl)amino)benzoic acid with carbon disulfide and KOH as a base with the mole ratio of (1:1) ethanol is used as a solventscheme (1) shows that. The complexes were prepared from reaction (ligand KL with metal chloride salts) in (2:1) mole ratio heated under reflux in ethanol to produce

pure complexes, where isolated in moderate yield, as shown in scheme (2).



Scheme (1): Synthetic of ligand KL



Scheme(2):Synthetic of $[M(L)_2(H_2O)_2]H_2O$ and $[Au(L)_2(H_2O)(Cl)]$ complexes

Table (1) describes ligand and complexes physical properties.

Table(1)Elemental analysis andPhysical characteristics for the ligand DTC

The Compounds	M. Wt.	Yield (%)	M. P (°C)	Colour	Found /calcd.				
					C	H	N	S	M
C₁₆H₁₄KNO₂S₂ (KL)	355.51	81	130-132*	Brown	53.80 (54.06)	3.53 (3.97)	4.13 (3.94)	17.49 (18.04)	
[Co(L)₂(H₂O)₂]H₂O	745.80	74	200*	Blue	51.15 (51.54)	4.07 (4.60)	3.87 (3.76)	16.98 (17.19)	7.47 (7.90)
[Cu(L)₂(H₂O)₂]H₂O	750.42	79	215*	Gray	51.04 (51.22)	4.49 (4.57)	3.87 (3.73)	16.51 (17.09)	7.87 (8.47)
[Ni(L)₂(H₂O)₂]H₂O	745.56	64.5	270*	Light green	51.15 (51.55)	4.04 (4.60)	3.89 (3.76)	16.97 (17.20)	7.43 (7.87)
[Au(L)₂(H₂O)Cl]	883.26	77.4	293*	brown	43.74 (43.52)	3.28 (3.42)	3.48 (3.17)	14.23 (14.52)	21.74 (22.30)

*= decomposed; m.p= melting point; Calc.= Calculated

The ¹HNMR, ¹³CNMR and Mass spectra of ligand KL

¹H-NMR spectra of ligand KL (Fig.1) showed the characteristic chemical shift of the solvent CDCl₃ at δ=7.23. The spectrum presented singlet chemical shifts at δ =13.17 ppm attributed to the one proton for carboxylic acid [10]. The spectrum of two singlet signals was displayed at δ =2.13, 2.26 ppm attributed to the one proton for CH₃ [11]. The signal at δ=8.07, 7.57, 7.41, 7.30, 7.15, 7.04 ppm were assigned for protons of CH (benzene ring) [12,13]. The ¹³C-NMR spectrum, in CDCl₃ solvent was displayed in (Fig.2). The preparation of ligand has been revealed through spotting signal in δ =196.69 ppm, that can be referred to carbon atom C1 for CS₂ group [14]. The carbon atoms C2 at δ=166.66 ppm, that can be attributed to carboxyl group [10]. The carbon atoms (C3-C14) were received with the chemical shifts at δ= 140.97, 138.48, 135.32, 133.15, 130.71, 128.79, 125.64, 124.07, 120.69, 118.43, 115.33, 113.18 ppm, respectively were assigned for C (benzene ring) [15]. Finally, the chemical shift at δ=77.45 ppm attributed to CDCl₃ solvent. The electrospray (+) mass spectra for KL is obtainable in (Fig.3). The spectra exhibits fragments belong to ligand composition. Parent ion peak of the ligand is spotted in m/z = 355 which corresponds to (M+H)⁺(%) for C₁₆H₁₄KNO₂S₂; requires = 355.01.

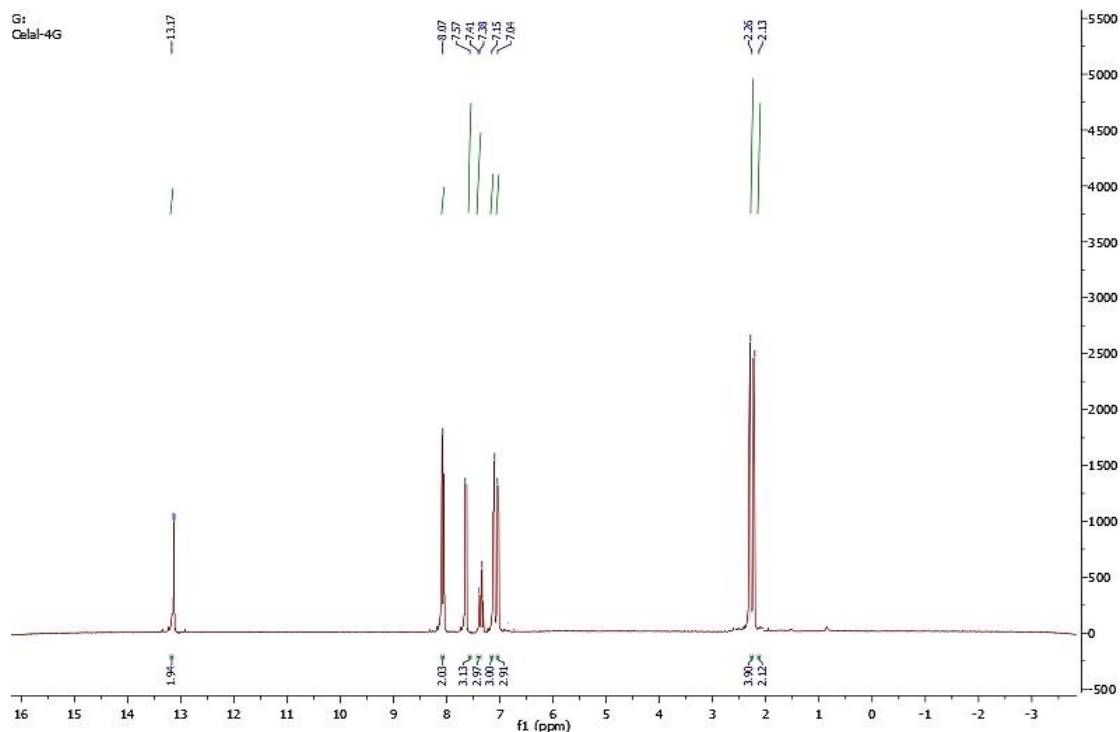


Figure (1) ¹H NMR spectra for ligand KL (CDCl₃ as solvent).

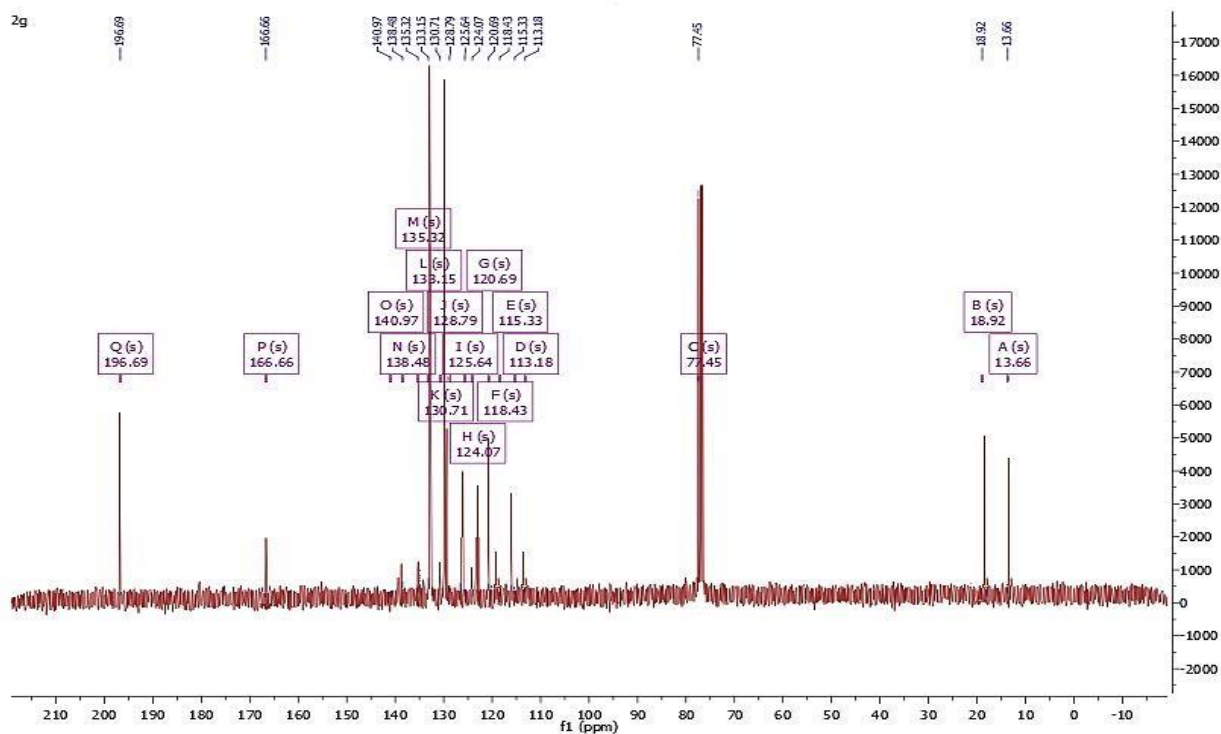


Figure (2) ¹³C NMR spectra for ligand KL (CDCl₃ as solvent).

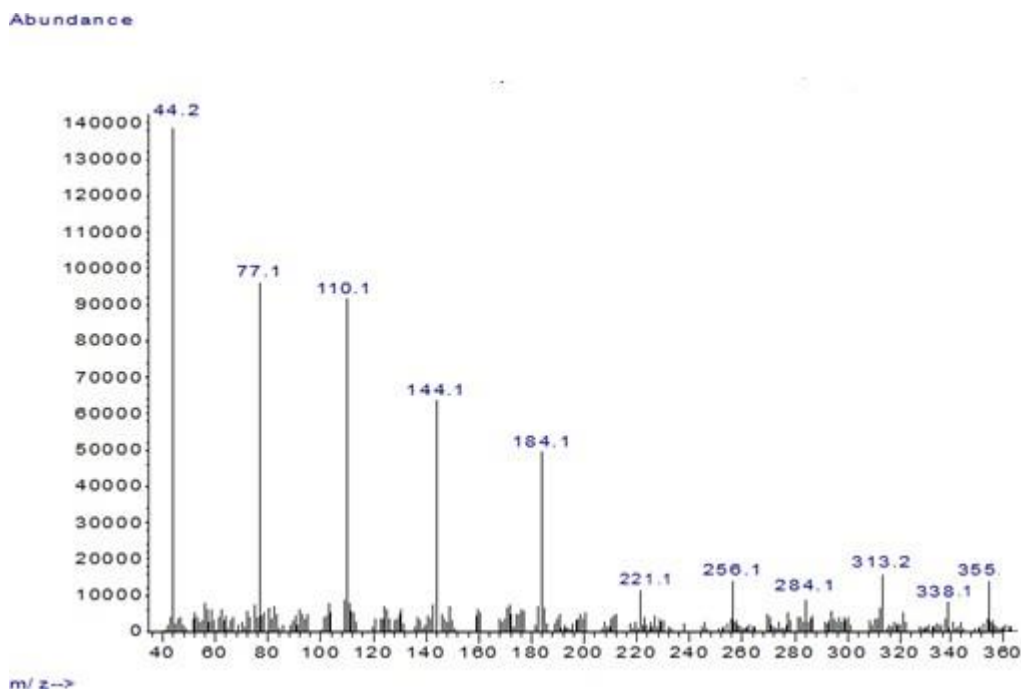


Figure (3) mass spectra for ligand KL

Mass Spectra for the complexes

Mass spectra for some complexes is presented at (Fig 4-5). Spectra exhibits fragments refer to complexes composition. Parent ion peaks are observed at $m/z = 745$ and 755 which corresponds to $(M+H)^+$ (%) for $[Co(L)_2(H_2O)_2]H_2O$, $[Cu(L)_2(H_2O)_2]H_2O$; requires = 745.06 and 749.05 respectively.

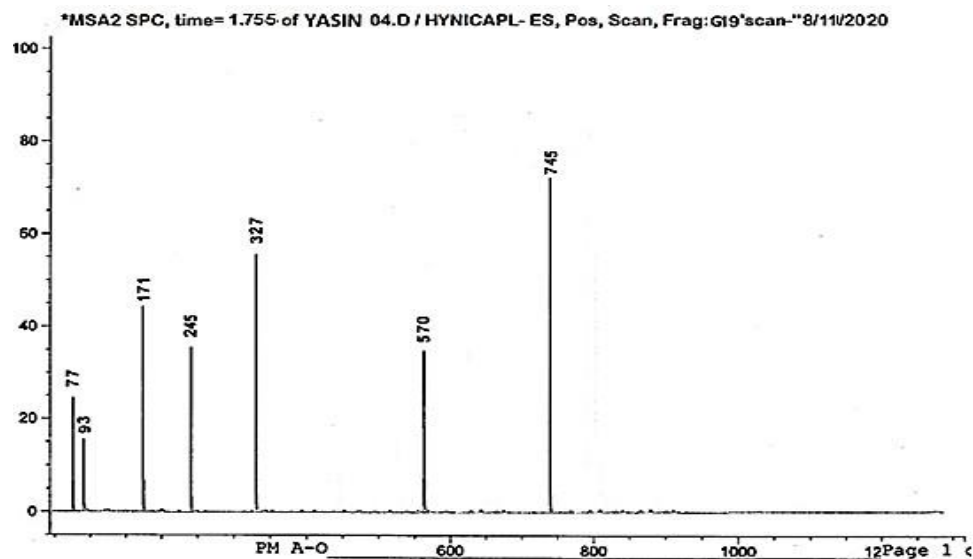


Figure (4): mass spectra for complex $[Co(L)_2(H_2O)_2]H_2O$

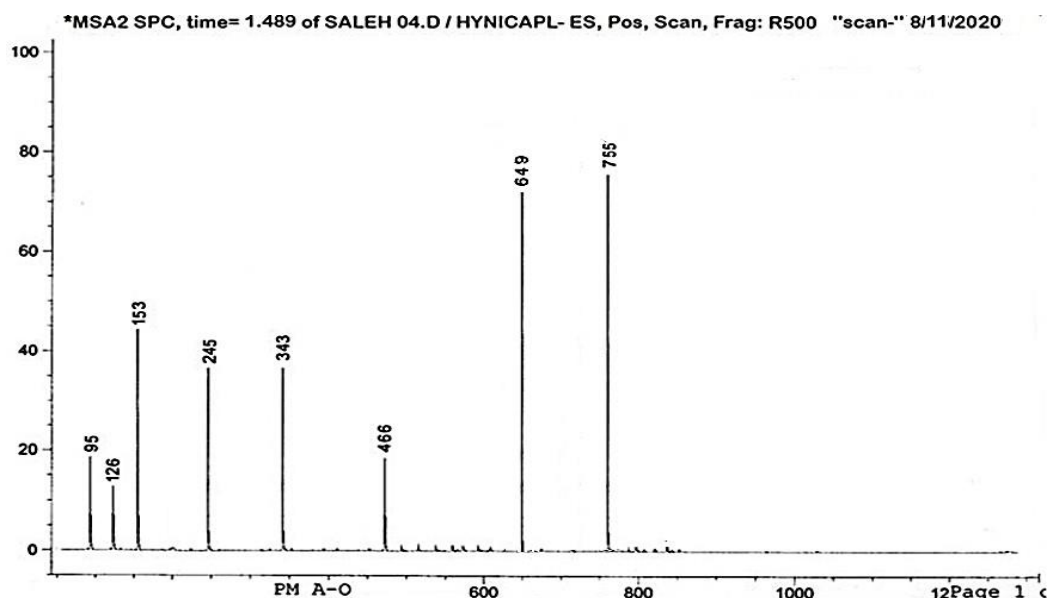


Figure (5): mass spectra for complex $[Cu(L)_2(H_2O)_2]H_2O$

The FTIR spectrum for ligand and complexes

The FTIR spectrum of (KL) shows no bands (disappeared) around 3339.5 cm^{-1} which belong to $\nu(\text{NH})$ [16]. FTIR spectrum reveals new bands in 1469 cm^{-1} refer to $\nu(\text{C-N})$ for (N- CS_2) group [17]. Also, two new bands are revealed by the FTIR spectrum at 1001 cm^{-1} and 885 cm^{-1} belong to $\nu_{\text{as}}(\text{CS}_2)$ and $\nu_{\text{s}}(\text{CS}_2)$, respectively [18]. The band in 1595 cm^{-1} is refer to $\nu_{\text{ar}}(\text{C=C})$ for aromatic ring [19]. In addition, the spectra displayed a bands in 1235 cm^{-1} and 648 cm^{-1} attributed for the $\nu(\text{C-N})$ and $\nu_{\text{ar}}(\text{C-S})$ respectively [20]. Bands are outlined in Tables (2).

Table (2) KL ligand's FTIR spectral information and its complexes

Compound	$\nu(\text{O-H})$ carboxyl	$\nu(\text{C-H})$ aromatic	$\nu(\text{C=O})$ carboxyl	$\nu(\text{C=C})$ aromatic	$\nu(\text{N-CS}_2)$	$\nu_{\text{as}}(\text{CS}_2)$	$\nu_{\text{s}}(\text{CS}_2)$	$\nu(\text{M-S})$	$\nu(\text{M-O})$
(KL)	3077	3070	1658	1595	1469	1001	885	-	-
$[Co(L)_2(H_2O)_2]H_2O$	3082	3080	1660	1573	1489	1015	990	400	598
$[Cu(L)_2(H_2O)_2]H_2O$	3079	3054	1660	1565	1455	1034	887	398	587
$[Ni(L)_2(H_2O)_2]H_2O$	3086	3023	1660	1589	1479	1147	899	375	583

[Au(L) ₂ (H ₂ O) Cl]	3090	3065	1660	1584	1500	1109	993	315	580
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UV-Vis spectral data for DTC (KL) and the complexes

In dimethyl sulfoxide solution, the UV-Vis spectrum of KL shows peaks at (273nm = 36630 cm⁻¹ ; ε_{max} = 1310 molar⁻¹ cm⁻¹) and (360 nm = 28571 cm⁻¹; ε_{max} = 1000 molar⁻¹ cm⁻¹) refer to “π → π*”, “n → π*” transitions, respectively [22], show in Table (3). Most complexes are colored. Such colors vary from ligands and transition metal salts, so these colors are considered essential indicators of coordination, so the colored complexes show different bands of absorption in location and intensity[23]

Table 3. UV, Vis spectrum for ligand and complexes(in 10⁻³ M DMSO)

Compound	λ	ν	ε _{max} .	Assignment	Suggested Formula
(KL)	273	36630	1310	π→π *	
	360	27777	1000	n→π *	
[Co(L) ₂ (H ₂ O) ₂] H₂O	310	32258	1580	L. F	Octahedral
	370	27027	700	L. F	
	460	21739	733	C. T overlap ⁴ T _{1g} (F)	
	650	15384	300	⁴ T _{1g} (F) → ⁴ A _{2g} (F)	
[Ni(L) ₂ (H ₂ O) ₂] H₂O	272	36764	480	L. F	Octahedral
	390	25641	400	L. F	
	450	22222	300	C. T overlap	
	700	14285	200	³ A _{2g} (F) → ³ T _{1g} (F)	
[Cu(L) ₂ (H ₂ O) ₂] H₂O	276	36231	580	L.F	Distorted Octahedral
	370	27027	330	L.F	
	430	23255	250	C.T	
	724	13812	115	² E _g → ² T _{2g}	

[Au(L)₂(H₂O) Cl]	300	33333	2252	L.F	Octahedral
	346	28901	1360	L.F	
	456	21929	1210	C.T overlap	
				³ A _{2g} (F)	
	630	15873	50	→ ³ T _{1g} (P)	
			³ A _{2g} (F)		
			→ ³ T _{1g} (F)		

Molar conductivities and magnetic susceptibility

The low molar conductivities of Co^{II}, Ni^{II}, Cu^I, and Au^{III} complexes at DMSO (17.4, 18.3, 19.2, and 16.4 ohm⁻¹.cm².mol⁻¹), respectively, mean the complexes are non - electrolytes [24]. Measuring the Co complex magnetic moment yields a meff value at 4.27 B.M. Co^{II} complex to octahedral geometries is given this value. 3.01 B.M. values were obtained from the Ni-complex. The meff value for this complex is included in the octahedral array, indicating a high geometry spin around Ni^{II} ion. Cu-complex meff value is 1.98 B.M, refer to the octahedral formulato the Cu^{II} ion. The complex Au^{III} is diamagnetic. Two unpaired electrons should be paramagnetic if the complex is in complete octahedral configuration. Despite this, the Au^{III} ion inspirextreme ligand field splitting due to its high oxidation state and heavy metal character, and the ideal octahedral configuration must be unstable since two electrons can occupy e.g. high-energy orbitals. As a result of the tetragonal distortion, the eg orbitals will be divided, making the Au^{III} complex diamagnetic. [25].

Biological effect

Biological effectforsynthesiseddithiocarbamate ligandand its complexes were calculatedthrough using inhibition wayfor four types of bacteria and one kind of pathogenicfungus [26]. twokind of bacteria was gram positive which is(*Staphylococcus aureus*,*Bacillus subtilis*) the second two was gram negative which is(*Escherichia coli*, *Pseudomonas aeruginosa*) and thepathogenic fungus was Candida. This is toestimate their potential antimicrobial activity. the biological activity of DMSO showed no activity against (the bacterial strains) [27]. Inhibition circle diameters in millimetre unit are listed in Tables (4). Figures (5) screen the effect of the synthesized dithiocarbamate compounds on bacterial strains andpathogenic fungus.

Table (3): biological effect for KLligand and the complexes (M(L)₂)

No	Compounds	<i>Bacillus subtilis</i> (G+)	<i>Staphylococcus aureus</i> (G+)	<i>Pseudomonas aeruginosa</i> (G-)	<i>Escherichia coli</i> (G-)	<i>condida</i>
1	Control (DMSO)	0	0	0	0	0
2	KL	22	23	30	18	24
3	[Co (L) ₂ (H ₂ O) ₂]H ₂ O	20	21	34	22	19

4	$[\text{Cu}(\text{L})_2(\text{H}_2\text{O})_2]\text{H}_2\text{O}$	24	22	29	30	22
5	$[\text{Ni}(\text{L})_2(\text{H}_2\text{O})_2]\text{H}_2\text{O}$	19	20	25	24	21
6	$[\text{Au}(\text{L})_2(\text{H}_2\text{O})\text{Cl}]$	25	23	27	27	25

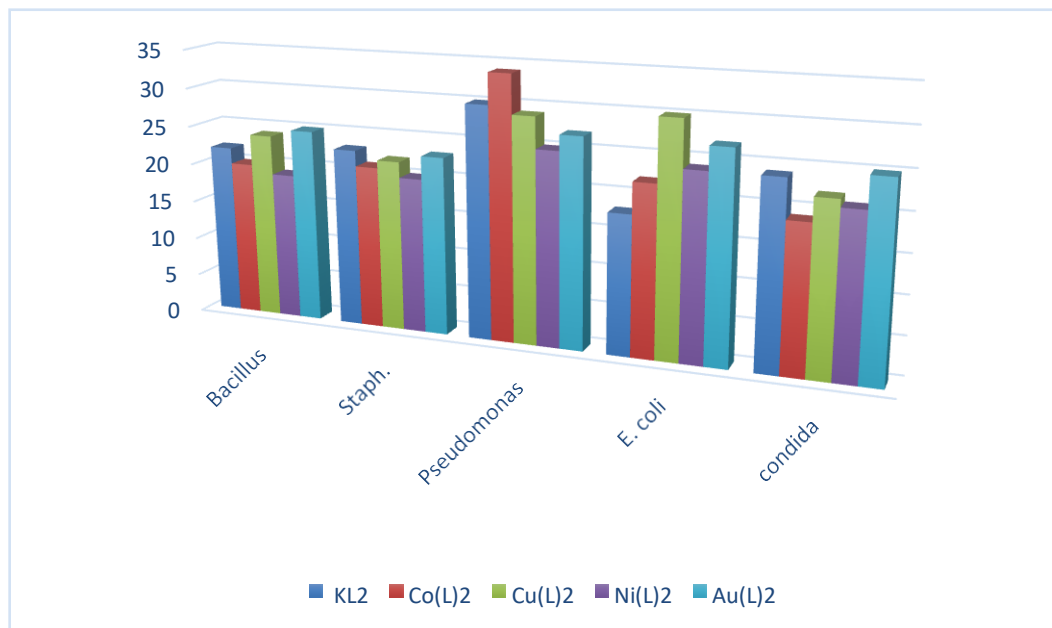


Figure (5): Evolution of diameter zone (mm) for inhibition of KL ligand and the complexes (M(L)₂) with the growth of various bacterial strains and pathogenic fungus.

Conclusion

The study of ligands and their complexes, as well as their preparation. The ligand complexes are made by reacting 2 potassium dithiocarbamate salt equivalent (DTC) with 1 metal salt equivalent as the central ion in ethanol. To check bonding mode and over all complex structure, physicochemical and spectroscopic methods have been implemented. These findings led to the development of four coordinate complexes, with the octahedral structure for complexes being proposed on fundamental the previous dispute. Biological effect of the prepared dithiocarbamate ligand and the complexes was investigated using inhibition method in four types of bacteria: (two gram positive and two gram negative bacteria), Add to one pathogenic fungus. It will be determined if they have antimicrobial activity. In general, the tests show that the ligands and their complexes have strong antibacterial and antifungal activity against various bacteria and pathogenic fungi.

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- π (chelate) interactions in $[\text{Ni}(\text{dtc})(\text{PPh}_3)(\text{NCS})]$ (dtc= N-(2-phenylethyl)-N-(4-methoxybenzyl)-dithiocarbamate and N-(2-phenylethyl)-N-(4-chlorobenzyl) dithiocarbamate). *Journal of Molecular Structure*, 1119, 385-395.
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