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Preparation and Characterization of Some Complexes of Nickel(II), Copper (II), and Zinc (II) With Decylxanthate and their Adducts with Nitrogen Base Ligands, and their Biological Activity

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Abstract: This research includes the synthesis and characterization of twelve new complexes of mono nuclear xanthate for a number of transition metal ions Ni(II), Cu(II), and Zn(II) and through the interaction with ligand (potassium decyl xanthate) and complexes of formula $[M(DEXANT)_2]$ were prepared: M= Ni(II), Cu(II), and Zn(II), and then the interaction of these complexes with Lewis bidentate bases, to give complexes with the formula $[M(DEXANT)_2.L]$, where L= 1,10-phenanthroline, 8-hydroxyquinoline, and 2,2-bipyridine. The prepared complexes were characterized by melting point, atomic absorption spectrometry, micro elemental analysis, infrared spectroscopy, proton nuclear magnetic resonance spectroscopy, molar electrical conductivity, electronic absorption spectra, and magnetic susceptibility measurements. Magnetic moment and electronic spectra indicated that the complexes of type $[M(DEXANT)_2]$ had a tetrahedral geometry, while complexes of type $[M(DEXANT)_2.L]$ had an octahedral geometry. The conductivity measurements proved the non-electrolytic behavior of all compounds. The biological evaluation against bacterial species indicated that the xanthate complexes were effective against all bacterial types.

Keywords: Xanthate, Complexes, Nickel(II), Copper(II), Zinc(II), Biological Activity.

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1. INTRODUCTION

Xanthates are sulfur- and oxygen-containing ligands that form a wide variety of coordination complexes with transition and main group metals (1). Xanthates are derived from xanthic acid, and organic molecules with a $-OCS_2$ functional group (2). Due to the important roles that several xanthate reactions play, the chemistry of xanthates holds a unique place among sulfur-containing reagents (3). Chemistry professionals have been drawn to the significant class of organic compounds known as xanthates because of their wide range of potential uses (4). Xanthates are much efficient for removing heavy metals from wastewater due to their low solubility products and high stability constants (5). Xanthates inhibit the replication of both DNA and RNA viruses in vitro and therefore, possess enhanced antiviral and antitumoral activity (6). Metal xanthates are extensively used as fungicides, pesticides, rubber accelerators, corrosion inhibitors, reagents in agriculture, and for treating HIV infections (7). The synthesis of xanthates may involve different processes and substances through a sequence of alkylation and xanthation reactions. However, they are mainly obtained by reacting an alkaline hydroxide with carbon disulfide and alcohol (8).

$$ROH + KOH \longrightarrow ROK + H_2O$$

$$ROK + CS_2 \longrightarrow ROCS^-_2K^+$$

Scheme 1: Preparation of xanthates.

2. EXPERIMENTAL PART

2.1 Materials and Instrumentation

The primary chemicals and solvents used were all processed by Aldrich Sigma and BDH: 1,10-phenanthroline monohydrate, 8-hydroxyquinoline, 2,2'-bipyridine, ethanol, decanal, diethyl ether, carbon disulfide, dimethylformamide (DMF), potassium hydroxide, $NiCl_2$, $CuCl_2$, and $ZnCl_2$.

The percentage of nickel, copper, and zinc in the complexes was determined by atomic absorption spectrometry (Analytik Jena NovA A350) in Mosul. NMR Ultrashield was used for analyzing ^{1}H-NMR spectra of synthesized ligand in Turkey, the sample was dissolved in deuterated (DMSO) at room temperature (298 K). FT-IR spectra of ligands and complexes were recorded on a spectrometer (Shimadzu) in the range (400 - 4000 cm^{-1}) using a KBr pellet, that was conducted at the University of Tikrit at 25 °C. The electrical conductivity was measured on the conductivity Meter-Model (Eutech pc700) of the complexes, and the samples were dissolved in DMF at a concentration of 10^{-3} M and a temperature of 25 °C. A UV spectrophotometer (PG Instruments) was used for recoding electronic spectra in dimethylformamide solvent with 10^{-3} M at 25 °C using 1 cm quartz cells. The melting point or decomposition temperature for the compounds was measured using (Aparatues-Stuart-SMP Melting point). magnetic measurements were made. using the Gouy method in the solid-state using (Magnetic Susceptibility Balance) at the University of Tikrit. The elements of carbon, hydrogen, nitrogen, and sulfur in the ligand and prepared complexes were estimated using an Eleminter Germany-type device at Tehran University/Iran.

2.2. Synthesis of Potassium Decyl Xanthate Ligand

Decanol (38.11 mL, 0.199 mol) was added to potassium hydroxide (11.20 g, 0.199 mol) with stirring. The mixture was cooled in an ice bath, and carbon disulfide was added drop wise to the mixture (12.06 mL, 0.199 mol) while stirring was continued for 30 minutes. The yellow precipitate formed was filtered off in an ice bath, washed with diethyl ether, and dried in vacuum (9).

2.3 Synthesis of Complexes [M (DEXANT)$_2$] (1:2)

M = Ni(II), Cu(II), and Zn(II) in which $NiCl_2$ (0.237 g, 0.0018 mol) or $CuCl_2$ (0.246 g, 0.0018 mol) or $ZnCl_2$ (0.250 g, 0.0018 mol) potassium hydroxide in ethanol, and decyl xanthate (**1,** 0.0036 mol) was added drop wise to the ethanolic solution while stirring 30 min to achieve complete precipitation. The precipitate was filtered off, rinsed with ethanol, dried in vacuum, and washed again with diethyl ether.

2.4 Synthesis of Complexes [M(DEXANT)$_2$.L] (1:2:1)

L = 1,10-phenanthroline, 8-hydroxyquinoline, or 2,2'-bipyridine were prepared similarly to section 2.3. The resulting precipitate was treated with (0.0018 mol) (1,10-phenanthroline, 8-hydroxyquinoline and 2,2'-bipyridine), which were added drop wise for 30 minutes while stirring constantly. The resulting precipitate was filtered off, washed with ethanol and dried.

3. RESULT AND DISCUSSION

3.1. Molar Conductivity

Molar conductance values of the millimolar solutions of adducts in DMF were found in the range of (2.5-21.5) $ohm^{-1}mol^{-1}cm^2$ (Table 1). The values were much smaller than that expected for any univalent electrolyte suggesting that these complexes were neutral and non-ionic in nature (10).

3.2. Magnetic Susceptibility Measurements

The effective magnetic moment (eff) of Complexes was calculated at 25 °C as shown in Table 2. The magnetic moments for Ni(II) and Cu(II) complexes from **1**, **2** and **3** are in the range in (1.65 – 2.12 B.M) suggesting a tetrahedral geometry (11). The low values of the effective magnetic moments compared to the spin-only magnetic moment for complexes **4** and **5** are due to antiferromagnetic interaction. The magnetic moment values of the other complexes **7**-**12** were in the range (1.83 - 2.75 B.M) suggesting an octahedral geometry (12). All Zn(II) complexes are diamagnetic.

3.3. Atomic Absorption

The proportions of Ni, Cu, and Zn in the resulting complexes were estimated from the results obtained in Table 1 and compared with theoretically calculated values, it was shown that the resulting complexes were consistent with the proposed formula (10).

3.4.Elemental Analysis (CHN)

Elemental analysis was carried out on the isolated complexes in order to prove their formation. The results obtained from this analysis are given in Table 1. Looking at the results in Table 1, it can be seen that there is a good consistency between the calculated and experimental ratio of the elements C,H,N and S of the proposed structure. This

agreement supported the formation of synthesized complex (10).

3.5. Electronic Spectral Studies

The UV-Vis spectra of the 10^{-3} M (DMF) solution of the ligand and its complex were recorded; the results are shown in (Table 2). For ligands, the high-intensity absorption peaks appearing in the 37037, 49504 cm^{-1} region are related to $\pi \rightarrow \pi*$, $n \rightarrow \pi*$ intraligand transitions (13) .

The UV-Visible spectrum of Ni(II) complex (**1**) shows two absorption bands at 11235 cm^{-1} and 17793 cm^{-1} which were assigned to $^3T_1(F) \rightarrow ^3A_2(F)$ and $^3T_1(F) \rightarrow ^3T_1(P)$ transitions respectively in a tetrahedral geometry, at the same time, the complexes **4**, **7** and **10** exhibit three absorption bands in the range 11185-11363 cm^{-1} , 14577-15337 cm^{-1} and 24154-21789 cm^{-1} which corresponded to $^3A_{2g}(F) \rightarrow ^3T_{2g}(F)$, $^3A_{2g}(F) \rightarrow ^3T_{1g}(F)$, and $^3A_{2g}(F) \rightarrow ^3T_{1g}(P)$ octahedral transitions respectively (14).

The Cu(II) complex (**2**) shows absorption band at 11235 cm^{-1} which correlates to $^2T_2 \rightarrow ^2E$ transition in the tetrahedral geometry, whereas octahedral complexes (**5**, **8**, and **11**) exhibited broadband in the region 12886-13227 cm^{-1}, which was assigned to $^2E_g \rightarrow ^2T_{2g}$ in an octahedral geometry (13). The Zn(II) complexes have no peaks in the visible range (d^{10} system). This means that no d-d electronic transitions occurred. These metal complexes do not show d-d transitions (14).

3.6 IR Spectra

The critical IR bands of the ligand (decylxanthate)K and its complexes are listed in Table 3. The band of FT-IR spectrum at 1128 cm^{-1} was assigned to υ(C–O) in ligand spectra, this band was shifted to a higher frequency and observed in the range 1143-1200 cm^{-1}. The band at 1075 cm^{-1} due to υ(C- S) of the ligand which shifted to a lower frequency 995-1052 cm^{-1} (15).

The range (418-470 cm^{-1}) of the IR spectra revealed a new band of moderate to strong intensity that provides support for the coordination of metal to sulfur υ(M-S). This phenomenon could be explained by alcohol's release of electrons, which pushes a high electron density toward the sulfur atoms, where the υ(M-N) was seen in the 483 - 555 cm^{-1} region (13).

Table 2: Data on the electronic spectra and effective magnetic moment of the ligand and produced compounds (cm^{-1}).

No	Formula of complexes	μeff B.M	U-Vis. Bands (cm^{-1})	Charge transfer	Proposed Structure
L	K-DEXANT	----	----	37037, 49504	----
1	$[Ni(DEXANT)_2]$	2.12	11235, 17793	37037	Tetrahedral
2	$[Cu(DEXANT)_2]$	1.65	11235	35460	Tetrahedral
3	$[Zn(DEXANT)_2]$	Dia	----	33211	Tetrahedral
4	$[Ni (DEXANT)_2(phen)]$	2.75	11173, 15337, 21786	34722	Octahedral
5	$[Cu (DEXANT)_2(phen)]$	1.83	13227	35971	Octahedral
6	$[Zn (DEXANT)_2(phen)]$	Dia	----	35211	Octahedral
7	$[Ni (DEXANT)_2 (Bipy)]$	2.65	11363, 14577, 24154	37878	Octahedral
8	$[Cu (DEXANT)_2 (Bipy)]$	1.94	12886	37878	Octahedral
9	$[Zn (DEXANT)_2 (Bipy)]$	Dia	----	37971	Octahedral
10	$[Ni (DEXANT)_2(8\text{-}Qui)]$	2.70	11185, 14925, 23923	35211	Octahedral
11	$[Cu (DEXANT)_2 (8\text{-}Qui)]$	2.03	13989	37593	Octahedral
12	$[Zn (DEXANT)_2 (8\text{-}Qui)]$	Dia	----	35791	Octahedral

Dia: Diamagnetic.

Table 1: The closed formulas, the compounds' physical characteristics, molar conductivity, and elemental analysis of the compounds prepared in this study.

NO	Formula of complexes	Color	M.P °C	Molar conductivity $\Omega^{-1}.cm^2.mol^{-2}$	Yield	Elemental analysis theoretical (Practical)				
						C%	H%	N%	S%	M%
L	K-DEXANT	Pale yellow	*260	---	87%	---	23.53 (22.77)	---	7.77 7.13	48.48 (47.83)
1	[Ni(DEXANT)$_2$]	Dark yellow	185	21.5	85%	11.17 (10.67)	24.40 (24.27)	---	8.06 (8.10)	50.28 (49.97)
2	[Cu(DEXANT)$_2$]	Blue	172	16.0	91%	11.98 (11.2)	24.18 (23.77)	---	7.98 (7.91)	49.82 (48.63)
3	[Zn(DEXANT)$_2$]	off White	220	19.3	72%	12.28 (11.65)	24.10 (23.69)	---	7.95 (7.87)	49.65 (49.73)
4	[Ni (DEXANT)$_2$(phen)]	Pink	*300	18.0	77%	8.32 (7.55)	18.17 (17.59)	3.97 (3.32)	7.14 (7.09)	57.87 (56.93)
5	[Cu (DEXANT)$_2$(phen)]	Deep Blue	120	6.9	85%	8.94 (8.47)	18.05 (18.23)	3.94 (3.69)	7.59 (7.47)	57.47 (57.09)
6	[Zn (DEXANT)$_2$(phen)]	Elegant White	*290	10.0	78%	9.18 (8.66)	18.00 (17.79)	3.93 (3.82)	7.57 (7.61)	57.32 (58.02)
7	[Ni (DEXANT)$_2$ (Bipy)]	Light green	*320	7.0	92%	8.61 (7.81)	18.81 (18.44)	4.11 (3.56)	7.39 (7.23)	56.38 (55.59)
8	[Cu (DEXANT)$_2$ (Bipy)]	Green	*295	18.0	89%	9.26 (8.55)	18.68 (18.17)	4.08 (3.47)	7.34 (7.39)	55.98 (54.88)
9	[Zn (DEXANT)$_2$ (Bipy)]	off White	180	6.0	82%	9.50 (8.76)	18.63 (17.87)	4.07 (3.83)	7.32 (7.37)	55.83 (54.67)
10	[Ni (DEXANT)$_2$(8-Qui)]	Light brown	200	4.7	87%	8.75 (7.45)	19.12 (19.56)	2.09 (1.87)	7.36 (7.41)	55.52 (54.77)
11	[Cu (DEXANT)$_2$ (8-Qui)]	Dark green	230	2.5	73%	9.41 (8.23)	18.98 (19.12)	2.07 (2.27)	7.31 (7.25)	55.12 (55.67)
12	[Zn (DEXANT)$_2$ (8-Qui)]	Yellow	*315	15.0	79%	9.65 (8.27)	18.93 (18.13)	2.07 (2.36)	7.29 (7.22)	54.97 (55.13)

*=decomposition

Table 3: IR bands (cm^{-1}) data of ligand and prepared complexes.

N0	Formula of complexes	ν(C-O)	ν(C-S)	ν(M-S)	ν(M-N)
L	K-DEXANT	---	1075	1128	---
1	$[Ni(DEXANT)_2]$	448	995	1200	---
2	$[Cu(\ DEXANT)_2]$	465	1005	1190	---
3	$[Zn(DEXANT)_2]$	425	1008	1195	---
4	$[Ni\ (DEXANT)_2(\ phen)]$	465	1035	1175	511
5	$[Cu\ (DEXANT)_2(\ phen)]$	458	1015	1182	483
6	$[Zn\ (DEXANT)_2(\ phen)]$	420	1047	1178	525
7	$[Ni\ (DEXANT)_2\ (Bipy)]$	470	1028	1162	510
8	$[Cu\ (DEXANT)_2\ (Bipy)]$	455	1048	1167	484
9	$[Zn\ (DEXANT)_2\ (Bipy)]$	438	1018	1148	493
10	$[Ni\ (DEXANT)_2(\ 8\text{-}Qui)]$	462	1043	1151	512
11	$[Cu\ (DEXANT)_2(\ 8\text{-}Qui)]$	423	1052	1143	487
12	$[Zn\ (DEXANT)_2\ (\ 8\text{-}Qui)]$	418	1037	1180	555

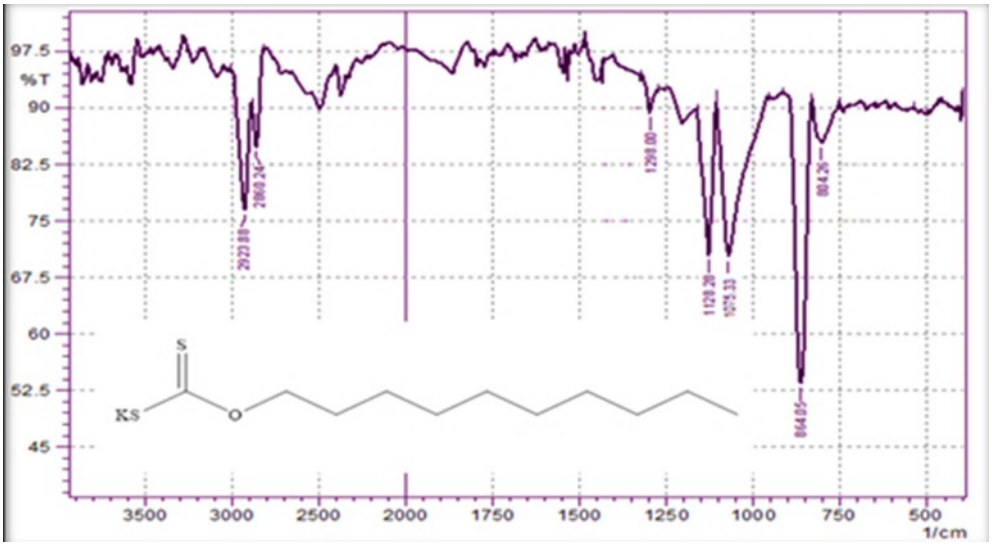


Figure 1: FTIR Spectrum of Ligand (K-DEXANT).

3.7 ^{1}H-NMR

The prepared ligand was studied by ^{1}H-NMR spectrometry, and the measurement reference is in this technique is tetramethylsilane ($SiMe_4$) and using (DMSO-d_6) solvent, or the results were interpreted depending on the values of the chemical signals, where the ligand spectrum was shown (16). ^{1}H-NMR (DMSO-d_6; 400 MHz) δH (ppm): 0.85- 0.88 (3H, m, CH_3), 1.25-1.30 (12H, m, CH_2), 1.39- 1.42 (4H, m, CH_2), 4.39-4.40 (2H, t, CH_2).

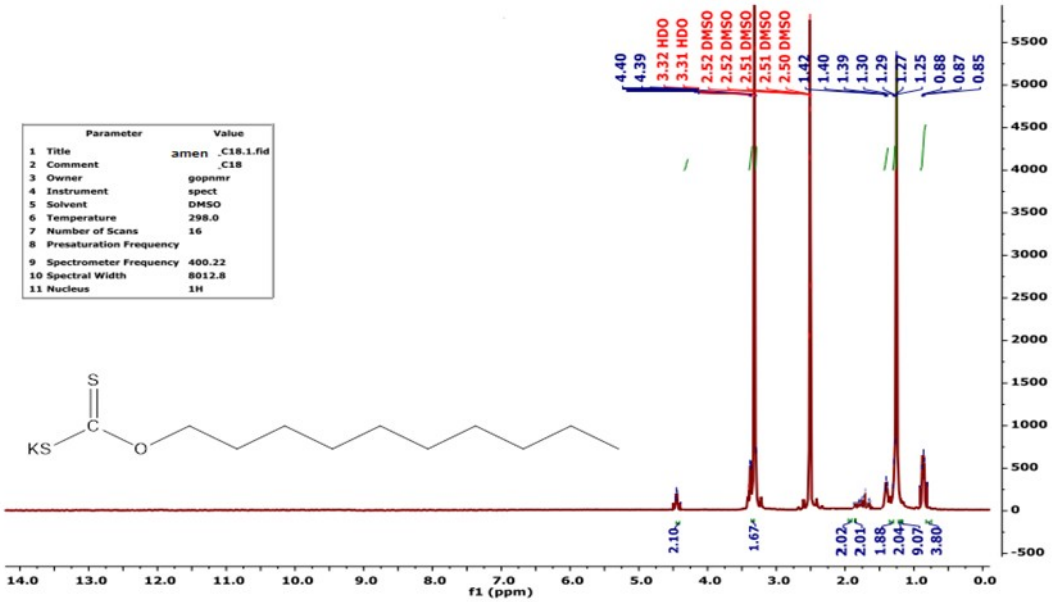


Figure 2: ^{1}H-NMR Spectrum of Ligand (K-DEXANT) in DMSO-d^{6}.

3.8 Thermogravimetric Analysis

The thermal behavior of the as-prepared complexes was studied at a heating rate of 25 °C per minute in a temperature range of 30–400 °C. The results showed the thermogravimetric analysis of the complexes of the type [M(DEXANT)$_2$L], there is no loss in the molecule above (120 °C) and this indicates the absence of a consistent water molecule within the crystalline network of the complexes as in (Figure 3) and the thermal dissociation of the complex ended with the transformation of the complex completely to a metal oxide at a temperature higher than 400 °C (17).

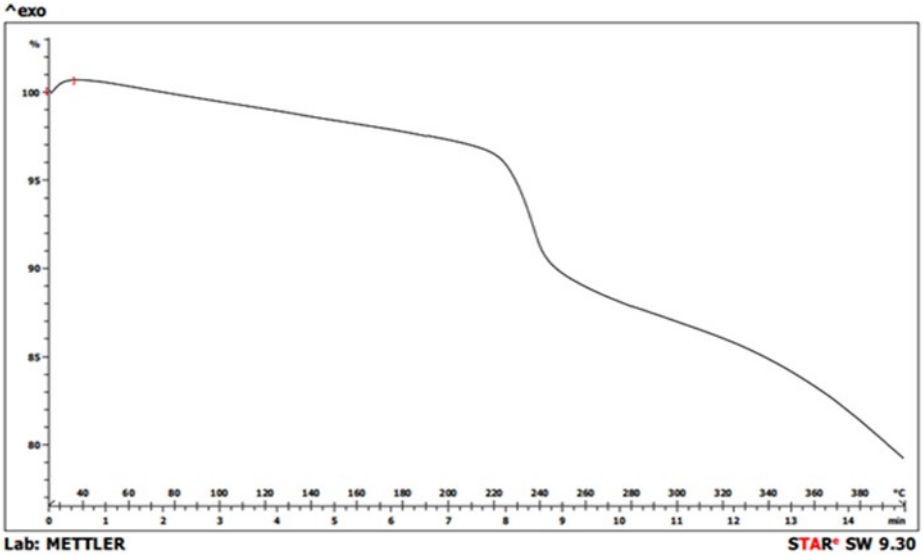


Figure 3: TGA For complexes [M(DEXANT)$_2$(Bipy)].

3.9 Gas chromatography- Mass spectrometry

The Gas Chromatography-Mass Spectrometry for K-DEXANT showed multiple peaks with relative abundance of Table 4. This shows where a peak appeared at 272 m\z at an abundance of 17.39% that thickened the partial weight of ligand itself, while the mass spectrum of the complex [Zn(DEXANT)$_2$] Peak at 532 m\z approved for theoretically calculated complex mass, while the mass spectrum of the complex [Ni(DEXANT)$_2$(Bipy)] Peak 681 m/z approved the theoretically-calculated complex mass, the mass spectrum for complex [Cu(DEXANT)$_2$(8-Qui)] peak at 675.12 m/z approved the calculated complex mass (10,18).

Table 4: Mass spectral data of ligand and the metal(II) complexes.

NO	Compound	Peak m\z	Relative % Abundance
L	K-DEXANT	272.1	17.39
1	$[Ni(DEXANT)_2]$	525.21	32.55
2	$[Cu(DEXANT)_2]$	530.16	49.23
3	$[Zn(DEXANT)_2]$	532.10	17.10
4	$[Ni(DEXANT)_2(phen)]$	705.12	28.32
5	$[Cu(DEXANT)_2(phen)]$	710.17	52.12
6	$[Zn(DEXANT)_2(phen)]$	712.21	21.44
7	$[Ni(DEXANT)_2(Bipy)]$	681.10	18.10
8	$[Cu(DEXANT)_2(Bipy)]$	686.15	45.12
9	$[Zn(DEXANT)_2(Bipy)]$	688.28	12.29
10	$[Ni(DEXANT)_2(8\text{-}Qui)]$	670.17	62.22
11	$[Cu(DEXANT)_2(8\text{-}Qui)]$	675.12	38.76
12	$[Zn(DEXANT)_2(8\text{-}Qui)]$	677.16	53.36

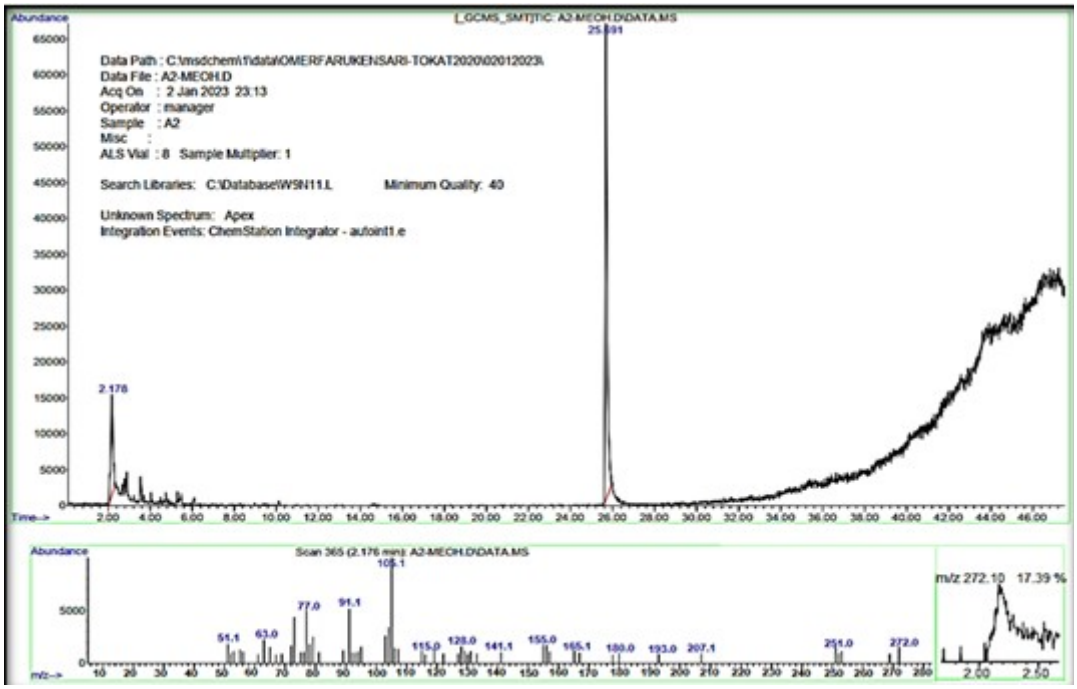


Figure 4: Mass Spectrum of ligand.

3.10 Biological Activity

The results of evaluating the biological activity of the ligand and the complexes under study on pathogenic bacteria (*Staphylococcus aureus, Escherichia coli, Pseudomonas aeruginosa* and *Proteus mirabilis* (*Klebsiella* spp) showed that the ligand and the complexes under study have an inhibitory ability towards these bacteria. These complexes bind with SH groups of enzyme cells, so they act more strongly than donor atoms in ligands, which should have the lowest (MIC) inhibitory concentration and came in agreement with what was published by many researchers (17,18) (see Table 5).

Table 5: Antibacterial activity (inhibition zone) of different concentrations of the ligand and complexes (µg/mL).

Compd	*Staphylococcus aureus*			*Escherichia coli*			*Pseudomonas auruginosa*			*Klebsiella spp*		
Conc\ µg	**125**	**250**	**500**	**125**	**250**	**500**	**125**	**250**	**500**	**125**	**250**	**500**
L	-	-	6	-	-	7	-	6	7	-	8	6
1	-	-	8	-	-	9	-	7	7	7	9	8
2	-	-	7	-	-	7	-	9	6	-	8	7
3	-	-	8	-	-	8	-	8	9	-	9	6
4	-	-	11	-	-	7	-	7	8	8	7	8
5	-	6	7	-	-	6	-	6	8	-	9	7
6	-	-	6	-	-	7	-	7	7	-	8	6
7	-	7	7	-	-	9	-	6	6	-	7	8
8	-	8	9	6	7	8	-	9	9	7	8	7
9	-	-	10	-	-	6	-	7	7	-	9	10
10	-	-	7	-	-	8	9	9	7	-	6	7
11	-	7	7	-	-	8	-	8	11	-	10	8
12	-	-	11	-	-	7	-	10	8	-	7	11
CIPS		10			10			10			10	
TMP		12			4			10			0	

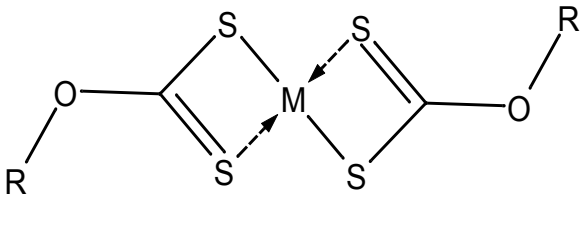
4. CONCLUSION

According to the analytical, physical, and spectral results the data observed have brought about the following points:

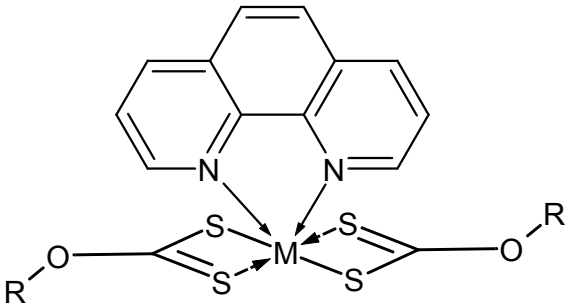
A- Complexes having a molar ratio of (2:1) are unsymmetrically tetrahedral and they have the formula [M(DEXANT)2] :

B-complexes with a molar ratio of (1:2:1) hexagonal symmetry octahedral and having the formula [M(DEXANT)2 L]

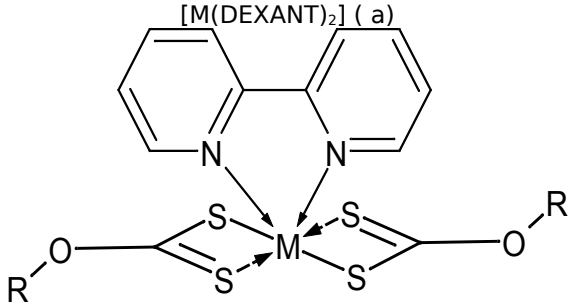
C-The ligand and metal complexes showed very good antimicrobial Properties.



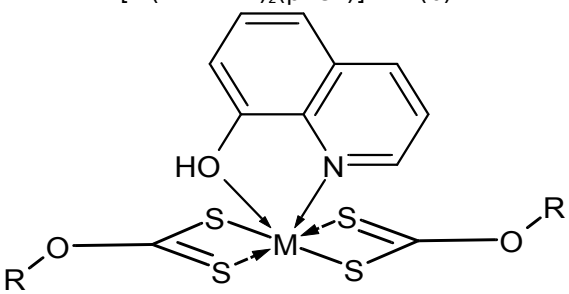
$[M(DEXANT)_2]$ (a)



$[M(DEXANT)_2(phen)]$ (b)



$[Mn(DEXANT)_2(Bipy)]$ (c)



$[M(DEXANT)_2$ (8-Qui)] (d)

Figure 3: Suggested structures (a) complexes (**1,2,3**), (b) complexes (**4,5,6**), (c) complexes (**7,8,9**), (d) complexes (**10,11,12**), M = Mn(II),Fe(II),Co(II) $R=(CH_2)_9CH_3$

5. ACKNOWLEDGMENTS

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