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Synthesis, characterization, and biological activity assessment of new metal complexes of Schiff bases produced from benzoin

Hussein Ahmad Ismail & Rana R. Abed*

Department of Chemistry, College of Education of Pure Science, University of Mosul, Mosul, Iraq.

*Corresponding Author: ranaalbustani@uomosul.edu.i

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Keyword: Schiff Bases; Zn++ , Mn++ ,Ag+ Complexes ; Benzoin ; Biological Activities.

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Abstract:

By the reaction of Benzoin with melamine and Benzoin with 5-aminosalysilc acid a new Schiff base is obtained the reaction of the legends with Mn++, Zn++, and Ag + salts leads to the complexes [M (L)2] X at PH=7-6, and [M (L)2] at PH=9-10, L= BMA, BMS M= Mn++ Zn++ and Ag +, Several chemical and physical measurements, such as melting point, molar conductivity, and magnetic properties, as well as FT-IR spectroscopy, UV-VIS spectroscopy element analysis (C.H.N.), and (1 H-NMR)spectroscopy, and GC-MASS. was used to identify the produced ligands and complex structures, the complexes of Mn++, and Znand ++ give hexacoordinated complexes having the shape octane al but Ag+ gives tetracoordinate complexes. having shape tetrahedral. then they were screened for biological activity using agar plate diffusion technique against Gram(+) (Staphylococcus aureus bacteria, pseudomonas aeruginosa bacteria) and Gram(−) (Escherichia coli bacteria, Klebsiella pneumonia bacteria) When compared to streptomycin and trimethoprim drug, all these ligands and their complexes show good antimicrobial effectualness.

Keywords: Schiff Bases; Zn++ , Mn++ ,Ag+ Complexes ; Benzoin ; Biological Activities.

تحضير وتشخيص وتقييم الفعالية الحيوية لمعقدات فلزية جديدة لقواعد شف المشتقة من البنزوين

رنا رمزي عبد & حسين احمد إسماعيل الكيكاني

جامعة الموصل / كلية التربية للعلوم الصرفة / قسم الكيمياء

ranaalbustani@uomosul.edu.i

من مفاعلة البنزوين مع الميلامين مرة والبنزوين مع ٤-امينو حامض السالسيلك تم الحصول على ليكندات جديدة لقواعد شيف أعقبها تحضير معقدات للمنغنيز والخارصين الثنائي والفضة الأحادية التأكسد باستخدام أملاح الفلزات حيث نتجت معقدات لها الصيغة التركيبية X_2 [M $(L)_2$] في الوسط المتعادل PH=7-6 و [M (L)2] في الوسط القاعدي PH=9- 10 حيث M, $^+$BMA, BMS = L and Ag = Zn^{++} , Mn^{++} , أعطت معقدات للمنغنيز والخارصين (II) الشكل الهندسي ثماني السطوح في حين نتجت معقدات رباعية السطوح للفضة (١) تم إجراء العديد من القياسات التشخيصية الكيميائية والفيزيائية مثل قياس درجة الانصهار والتوصيلية المولارية والخصائص المغناطيسية وكذلك طيف الأشعة تحت الحمراء وطيف الأشعة فوق البنفسجية وقياس تحليل العناصر C.H.N. وطيف الرنين النووي المغناطيسي ١ H-NMR و GC-MASS (طيف الكتلة) كما أجريت قياسات لتقييم الفعالية الحيوية لليكندات ومعقداتها تجاه أنواع مختلفة من البكتيريا الموجبة الغرام مثل بكتيريا الستافيلوكوكس اوريس وبكتيريا السيدوموناس اوراجينوزا والبكتيريا السالبة الغرام مثل البكتيريا الاشيريكية ايكولاي وبكتيريا الكليبسيلا بينومونيا، حيث سجلت كلا من الليكندات ومعقداتها فعالية جيدة مقارنة بالأدوية القياسية مثل الستربتومايسين والترايميثوبريم.

الكلمات المفتاحية: قواعد شف، معقدات فلزية Ag, Mn++ , Zn+++ , بنزوين ، فعالية بيولوجية.

1. INTRODUCTION:

Schiff bases are considered one of the most important organic compounds that are easy to prepare and of wide importance because they possess important medicinal properties as substances with high efficacy against bacteria, fungi, and anti-tumors, which increases the effectiveness of these compounds, and their consistency with transition metals that have high effectiveness in increasing the immunity of living cells, such as zinc, manganese, and silver **[1]** Schiff base compounds are known to be prepared by a condensation reaction between amine and carbonyl compounds such as aldehydes and ketones, which produces a compound with a functional group called the azomethine which is considered a very effective group in terms of pharmacology. In addition to their consistency with metal atoms through the donation of the double electron to the nitrogen atom, where they are stable complexes with important therapeutic properties in the preparation of many anti-inflammatory drugs**[2]**.

Chemical sciences are currently heading towards the preparation of more drugs, antibiotics, and anti-cancer drugs that can be said to be new drugs or drugs developed from existing ones to help humans combat stubborn germs that are beginning to show resistance to traditional drugs used at the present time. In our research, after preparing complexes of Schiff bases with Zinc, Manganese, and Silver, we studied their biological effect against different types of bacteria, as they showed clear effectiveness against these pathogenic bacteria **[3]**.

2. EXPERIMENTAL SECTION

2.1 Materials

All the chemicals and solvents required to create the compounds came from several suppliers, including Merck, BDH, Fluke, and Sigma Aldrich.

2.2 Synthesis Methods

Synthesis of ligand ((E)-2-((4,6-diamino-1,3,5-triazin-2-yl) imino)-1,2-diphenylethan-1-ol) BMA (0.047 mole of Benzoin (10g) dissolved in 50 mL ethanol then (0.047 mole, 5.94 g of melamine) as 1:1 molar ratio dissolved in 20 mL ethanol with few drops of glacial acetic acid, the mixture of the two solutions was refluxed for (2 h.). The yellow precipitate was filtrated and washed with water after cooling to room temperature. and recrystallization by hot ethanol then dried in the oven at 40 °C. Synthesis of ligand) (E)-2-hydroxy-5-((2- hydroxy-1,2- diphenyl ethylidene)amino) benzoic acid) BMS follows the same method as (0.047 mole of Benzoin (10g) dissolved in 50 mL ethanol then (0.047 mole, 7.21 g of mesalazine) 1:1 molar ratio **Fig.1** and **Fig.2** (Scheme 1, 2) then the metal complexes were prepared **[4]**, depicts the general reactions of prepared compounds, while **Table 1** lists all physical properties.

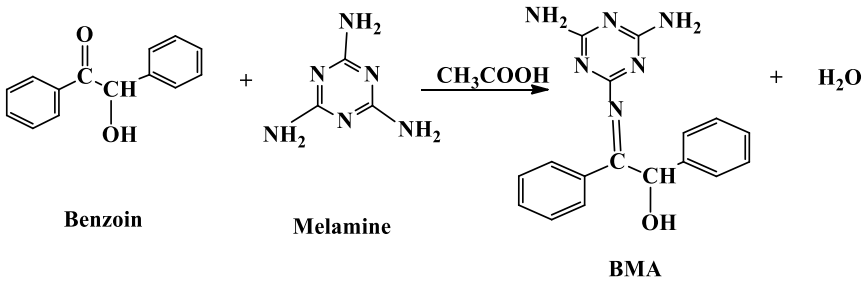

Fig. 1: Scheme 1

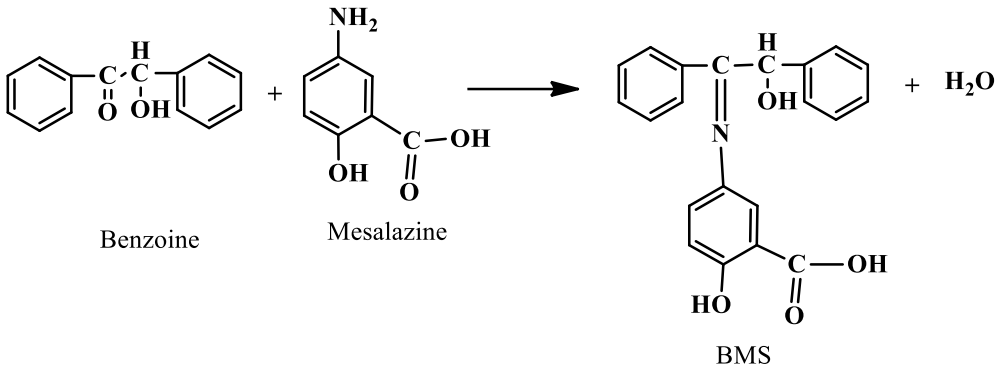

Fig. 2: Scheme 2

2.3. Preparation of complexes

The coordination of Manganese (II), Zinc(II), and Silver (I) with ligands is carried out with a 1:2, (M-L) molar ratio. The coordination reaction is carried out in the neutral medium and basic medium as follows (Scheme. 3) **Fig. 3**.

2.3.1. In a neutral medium.

[Zn(BMS)$_2$] AOc$_2$ was prepared by dissolving 0.945 g (0.0027 mole) of BMS ligand in 15 mL of ethanol with 0.25 g (0.0013 moles) of Zn(AOc)$_2$. 2H$_2$O which dissolved in distilled water in a (2:1) ratio. For three hours, the mixture has been refluxing. half of its capacity evaporated before being allowed to cool. The resultant complex was filtered out, washed twice with diethyl ether, then cold distilled water, and dried. Using the same method, BMA complexes with the other Zn^{++}, Mn^{++}, and Ag^{+} salts were synthesized **[4]**. (Scheme 2) **Fig. 2**. According to the combination ratio the suggested formula of the complex in the neutral medium and basic medium is presented in **Table 1**.

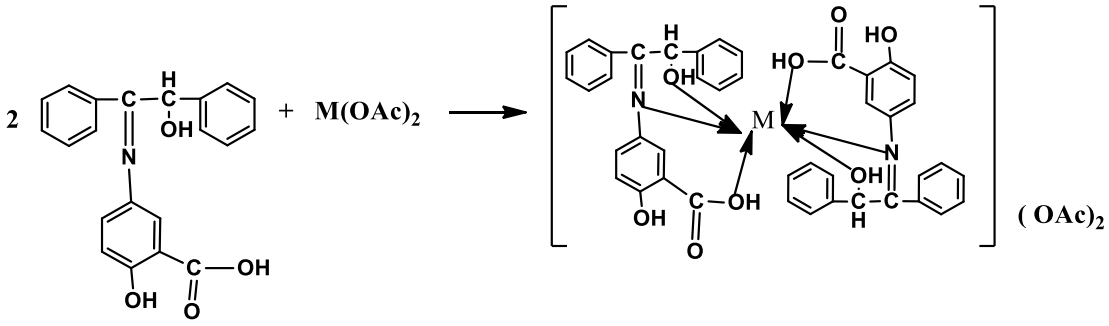

Fig.3: Scheme 3

2.3.2. In a Basic medium

$[Zn(BMS)_2]$ OAc_2 was prepared by dissolving 0.945 g(0.0027 moles) of BMS ligand in 15 mL of ethanol with 0.25 g (0.0013 moles) of $Zn(Ac)_2$. $2H_2O$ which dissolved in distilled water (2:1) ratio. A few drops of KOH (1M) were then added, and the precipitation was finished. The resultant complex was filtered out, washed twice with diethyl ether, and dried after each wash with cold distilled water. Using the same method, BMA complexes with the other Zn^{++}, Mn^{++}, and Ag^{+} salts were created [4]. (Scheme 4) Fig. 4. The complex's recommended formula in the neutral medium and basic medium is as follows, depending on the combination ratio in Table 1.

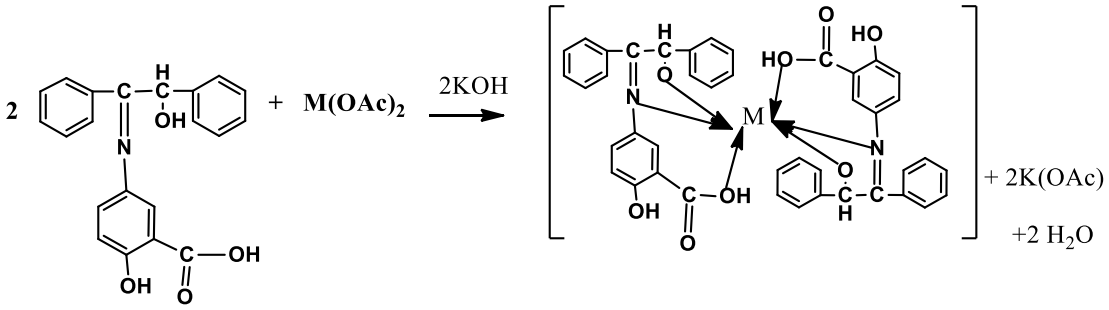

Fig. 4: Scheme 4

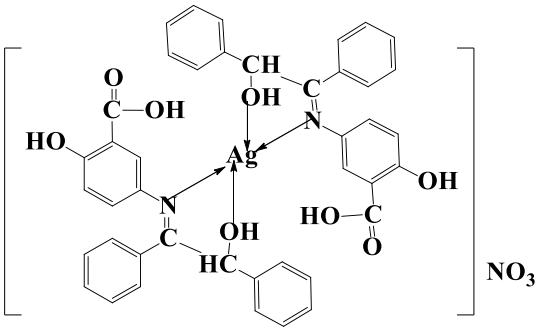

Fig. 5: tetra coordination Silver(I) complex

Table 1: Amounts, Yield, and Formulae of the prepared complexes

No.	Wt. Of BME, BMS in gram	Salts 0.25 g	yield %	Complexes
1	0.872	$Zn(OAc)_2 2H_2O$	79	$[Zn(BMA)_2](OAc)_2$
2	0.872	$Zn(OAc)_2 2H_2O$	77	$[Zn(BMA)_2]$
3	0.652	$Mn(OAc)_2 4H_2O$	75	$[Mn(BMA)_2](OAc)_2$
4	0.652	$Mn(OAc)_2 4H_2O$	87	$[Mn(BMA)_2]$
5	0.941	$AgNO_3$	81	$[Ag(BMA)_2]No_3$
6	0.941	$AgNO_3$	83	$[Ag(BMA)_2]$
7	0.945	$Zn(OAc)_2 2H_2O$	89	$[Zn(BMS)_2](OAc)_2$
8	0.945	$Zn(OAc)_2 2H_2O$	87	$[Zn(BMS)_2]$
9	0.707	$Mn(OAc)_2 4H_2O$	88	$[Mn(BMS)_2](OAc)_2$
10	0.707	$Mn(OAc)_2 4H_2O$	80	$[Mn(BMS)_2]$
11	1.021	$AgNO_3$	83	$[Ag(BMS)_2]No_3$
12	1.021	$AgNO_3$	86	$[Ag(BMS)_2]$

4. RESULTS AND DISCUSSION

4.1. physical and Analytical measurements

The produced ligands and complexes are identified using a variety of approaches. The elements of carbon, hydrogen, and nitrogen (CHN), as well as the complex's infrared spectrum, melting point, and molar conductivity, have all been analyzed. NMR of the "Bruker Ultra Shield 300 MHz" type was used to record the H-NMR for the ligand [4].

4.2. Physical properties

The compounds that were created were yellow solids that were air-stable, insoluble in water, but soluble in DMSO. The complexes with the metal ions Mn^{++}, Zn^{++}, and Ag^{+} had relative molecular weights that could be estimated, and it was discovered that these complexes might have the molecular formulas $[M(L)_2]X2$ in neutral medium and [M(L)2] in basic medium. The ligands are tridentate when it is ready. Using the nitrogen and oxygen atoms that formed Octahedral geometry with Mn (II), Zn (II) [5], it acts as bidentate when it is ready. Using the nitrogen and oxygen atom that formed tetrahedral geometry with Ag (I).

4.3. The molar conductivity.

The determined values of the molar conductivities of the prepared complexes of Mn^{++} and Zn^{++} were found to be in the range of (77-80 Ω^{-1} cm^2 mol^{-1}). This range approaches the values expected for complexes of 1:2 electrolytes, and 1:1 electrolyte. the molar conductivities of the

prepared Ag $^{+}$complexes were found to be in the range of (32-35 Ω^{-1} cm^2 mol^{-1}). This range approaches the values expected for complexes of 1:1 [6]. In basic medium, all the complexes are non-electro let.

4.4.Elemental analysis (CHN).

CHN elemental analysis is carried out on the isolated complexes to prove their formation. The results obtained from this analysis are given in **Table 3**. The results in Table 3 show good consistency between the calculated and experimental ratio of the elements CHN, of the proposed structure. This agreement supports the formation of the synthesized complex [7].

4.5. Determination of Zn^{++}, Mn^{++}, and Ag^{+} ions.

By using flame atomic absorption spectrometry to a monomeric structure, the concentrations of Zn and Mn ions are determined. The findings demonstrated that the compounds under study have octahedral geometries [8], **Table 2**.

Table 2: CHN analysis, Metal contents of the prepared complexes

NO.	Structure	M.wt	color	M.P	Mol\Cond	C% Cal/Exp	H% Cal/Exp	N% Cal/Exp	M% Cal/Exp
BMA	$C_{17}H_{16}N_6O$	320	Off White	155	-	63.74 64.01	5.03 5.21	26.23 26.43	-
BMS	$C_{21}H_{17}NO_4$	347	yellow	152	-	72.61 72.13	4.93 4.57	4.03 3.98	-
1	Zn C_{38} $H_{38}N_{12}O_6$	823	Pale yellow	140	87	55.4 55.5	4.61 4.87	20.41 21.00	9.26 8.49
2	Zn C_{34} $H_{30}N_{12}O_2$	703	Pale yellow	144	16	57.87 57.99	4.53 4.93	23.82 24.03	9.28 8.98
3	Mn C_{38} $H_{38}N_{12}$ O_6	813	Pale yellow	143	79	56.08 55.87	4.67 4.98	20.66 21.04	6.75 6.87
4	Mn C_{34} $H_{30}N_{12}$ O_2	693	Pale yellow	145	11	58.70 59.09	4.60 4.80	24.17 23.99	7.92 8.06
5	Ag C_{34} $H_{32}N_{13}$ O_5	810	Pale yellow	149	35	50.37 51.14	3.95 4.23	22.46 22.56	13.30 12.95
6	Ag C_{34} $H_{30}N_{12}$ O_2	746	Pale yellow	137	15	54.54 55.05	4.27 4.33	22.45 23.22	14.45 14.44
7	Zn C_{46} $H_{40}N_2$ O_{12}	877	Pale yellow	135	80	62.94 61.99	4.56 4.65	3.19 3.42	7.44 7.73
8	Zn C_{42} $H_{32}N_2$ O_8	757	Pale yellow	142	16	66.40 66.87	4.47 5.06	3.68 4.12	8.62 8.56
9	Mn C_{46} $H_{40}N_2$ O_{12}	867	Pale yellow	148	77	63.66 64.31	4.61 4.89	3.22 3.76	6.33 7.07
10	Mn C_{42} $H_{32}N_2$ O_8	747	Pale yellow	146	19	67.28 67.66	4.53 5.05	3.73 3.84	7.34 7.44
11	Ag C_{42} $H_{34}N_3$ O_{11}	864	Pale yellow	144	32	58.33 58.06	3.93 4.00	3.24 3.54	12.47 12.82
12	Ag C_{42} $H_{32}N_2$ O_8	800	Pale yellow	141	11	62.84 61.97	4.23 4.33	3.49 3.53	13.47 13.13

4.6. FT-IR

Some important bands of the infrared spectra of BMA ligands and their complexes are listed in **Table 4**. Comparison between bands of ligands and complexes may help to predict useful information **[9]**. The band at 1632-1650 cm^{-1} is due to C=N stretching of the azomethine group, in complexes, this band is observed in the range between (1604 −1621) cm^{-1} which refers to coordination between these groups and the metal ion **[10]**. Another band is observed in the infrared spectra at 1338 cm^{-1} due to C-N pyridine groups· On coordination, this band was observed at the same frequency This demonstrates that is no coordination of the nitrogen atom with the metal ion.

Various local bands 3416-3329 cm^{-1} in ligand due to OH group of benzoin, in complexes, this band is observed in the range between 3318-3435 cm^{-1}, This demonstrates that these groups and the metal ion are coordinated. The band at 3160-3476 cm^{-1} is due to two NH_2 groups stretching in complexes this band is observed in the range between (3109 – 3379 cm^{-1}) which refers to coordination between one of these groups and the metal ion **[10]**.

Some important bands of the infrared spectra of BMS ligand and their complexes. The band at 1617- 1645cm^{-1} is due to C=N stretching groups of Schiff bases in complexes this band is observed in the range between (1598 – 1556) cm^{-1} which refers to coordination between these groups and the metal ion **[10]**. Another band is observed in the infrared spectra of the BMS ligand at 1725-1645cm^{-1} due to C=O groups· On coordination, this band was observed at the same frequency that demonstrates no coordination between the oxygen atom of the C=O groups· with metal ions **[11]**.Other bands in the region 3395 cm^{-1} in ligand due to OH group of Benzoin part. in complexes in a neutral medium this band is observed in the range between 3302-3374 cm^{-1}, This gives an indication there is coordination between this group and the metal ion. The band in the region 2498-3200 cm^{-1} in ligand due to OH group carboxylic acid in complexes this band is observed in lower frequency in the range between 2317-3210cm^{-1}, This gives an indication there is coordination between the oxygen of this group and the metal ion .but this band observed in the same frequency in silver complexes This demonstrates that is no coordination between the oxygen atom with the silver ion. The band in the region 3375 cm^{-1} in ligand due to OH phenolic group this band was observed at the same frequency and demonstrates no coordination between the oxygen atoms and metal ion.

The bands in the range between 415-421 and 450-496cm^{-1} in complexes are due to the M-N and M-O connection which demonstrated the coordination of two nitrogen and Oxygen atoms

with a metal ion [11]. Important Bands in FT- IR spectra of the ligand and their complexes are listed in Table 3.

Table 3: Some Important Bands in FT- IR Spectra

No.	C=N	C=O	C-N $_{Py}$	OH $_{benzoin}$	OH $_{phenol}$	OH, $_{salicylic\ acid}$	NH_2	M-O	M-N
BMA	1650-1632	-	1338	3416-3329	-		3160-3467	-	
BMS	1617-1645	1677	-	3395	3375	2498-3200	-		
1	1604	-	1337	3337-3321	-		3110-3405	479	416
2	1614	-	1337	-	-		3109-3379	480	415
3	1620	-	1338	3404-3318	-		3111-3376	480	416
4	1615	-	1337	-	-		3113-3379	481	416
5	1621	-	1327	3336-3435	-		3466-3125	485	418
7	1634	1678	-	3302	3396	2354-3138	-	460	421
8	1638	1678	-	-	3378	-	-	462	420
9	1648	1678	-	3378	3406	2522-3210	-	481	417
10	1595	1678	-		3409	-	-	482	420
11	1619	1678	-	3374	3394	3217	-	481	416

4.7. UV-VIS

UV-VIS spectroscopy of the complexes is recorded by using dimethyl sulfoxide as a solvent the results refer to the manganese complexes is Hight spin containing (5 electrons in d orbitals) the obtained band was church transfer also $Mn^{+2,}$ $Zn^{+2,}$ and Ag^{+1} complexes [11].as Table 4.

Table 4:UV-VIS ligand bands

No.	Ligand	n → π* cm^{-1}	π → π* cm^{-1}
2	BMA	33783.78	39215.68
3	BMS	34013.60	37453.18

Table 5:UV-VIS of complexes, charge transfer bands

No.	cm^{1-} C.T
1	37313.43
2	37313.43
3	37313.43
4	37037.03
5	36764.70
6	35971.22
7	36496.35
8	32467.53
9	36496.35
10	37037.03
11	35971.22
12	32467.53

4.8. Magnetic properties :

The magnetic sensitivity of the manganese complexes was measured only, where the magnetic moment values proved the presence of five single electrons in the d orbitals, which confirms to us that the manganese complexes have para-magnetic properties and have a highly spiral octahedral geometry, while the zinc and silver complexes have Daya-magnetic properties.

Table 6: magnetic properties values

NO	Molecular structure	D. 10^{-6}	$\chi_g.10^{-6}$	$\chi_M.10^{-6}$	$\chi_A \times 10^{-6}$	μeff B.M	ن ح
3	Mn C_{38} $H_{38}N_{12}$ O_2	398.28	17.41477	14158.20801	14556.48801	5.89	Oh
4	Mn C_{34} $H_{30}N_{12}$ O_2	332.4	17.30034	11989.13562	12321.53562	5.41	Oh
9	Mn C_{46} H_{40} N_2 O_{12}	464.4	16.86179	14619.17193	15083.57193	5.99	Oh
10	Mn C_{42} H_{32} N_2 O_8	398.52	17.79901	13295.86047	13694.38047	5.71	Oh

4.9. H^1-NMR

The H -NMR spectra of the ligand are recorded by using deuterated dimethyl sulfoxide as an internal reference. The results as following bands appeared at chemical shifts supporting the structure of the ligand under investigation [11]. The spectrum was recorded using deuterated dimethyl sulfoxide as a solvent and at a frequency equal to 400 MHZ.

4.9.1. BMA Giving Nuclear Magnetic Resonance Data:

($CDCl_3$, 400 MHZ) ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm):

(1H, s, C*H*) δ: 6.06 ppm).

(H, m, OH), δ: 6.10 ppm).

(10H, m, Ar-*H*), δ: 7.24 – 8.02ppm). :

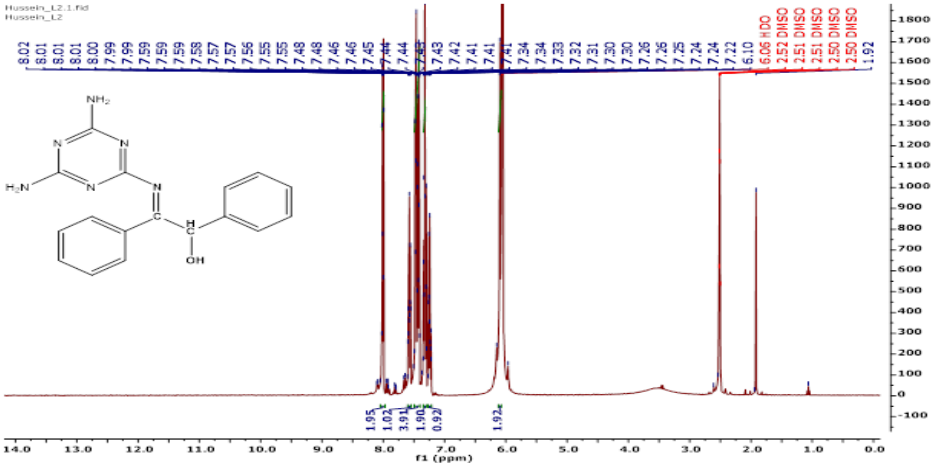

Fig 5: BMA Nuclear Magnetic Resonance

4.9.2 BMS Giving Nuclear Magnetic Resonance Data ($CDCl_3$, 400 MHZ)

1H NMR (400 MHz, $CDCl_3$, ,):

1H, s, C*H*) δ: 6.07 ppm).

(1H, s, O*H cholic*) δ: 6.71 ppm).

(2H, m, AR-*H*), δ: 7.17 – 8.01ppm).

(1H, s, O*H phenol*) δ:6.73 ppm

(1H, s, O*H carboxylic*) δ: 10.4 ppm).

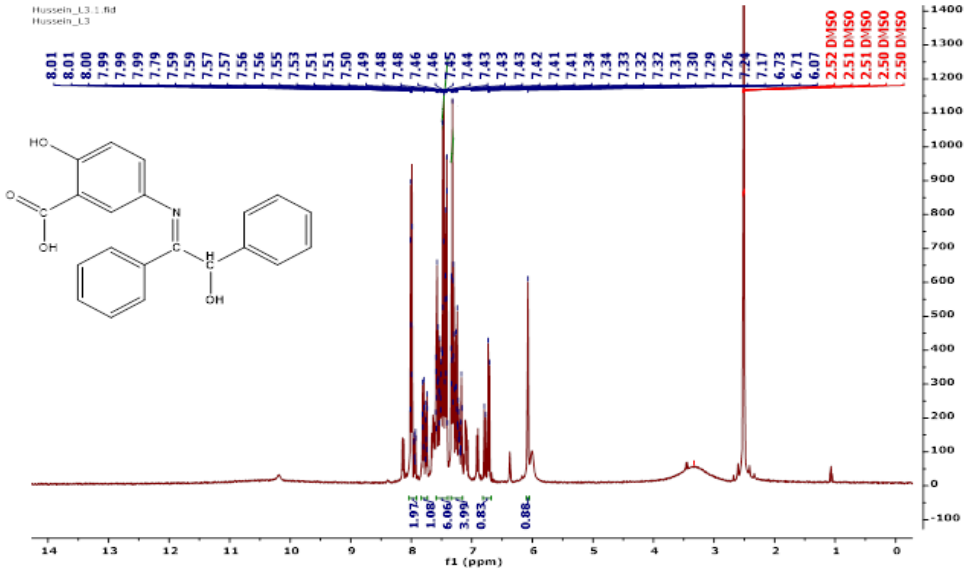

Fig 5: BMS Nuclear Magnetic Resonance

4.10. GC-MASS :

The suggested compounds of GC-MASS type Agilent GC-MS/ Turkey approach 90% of prepared compounds.

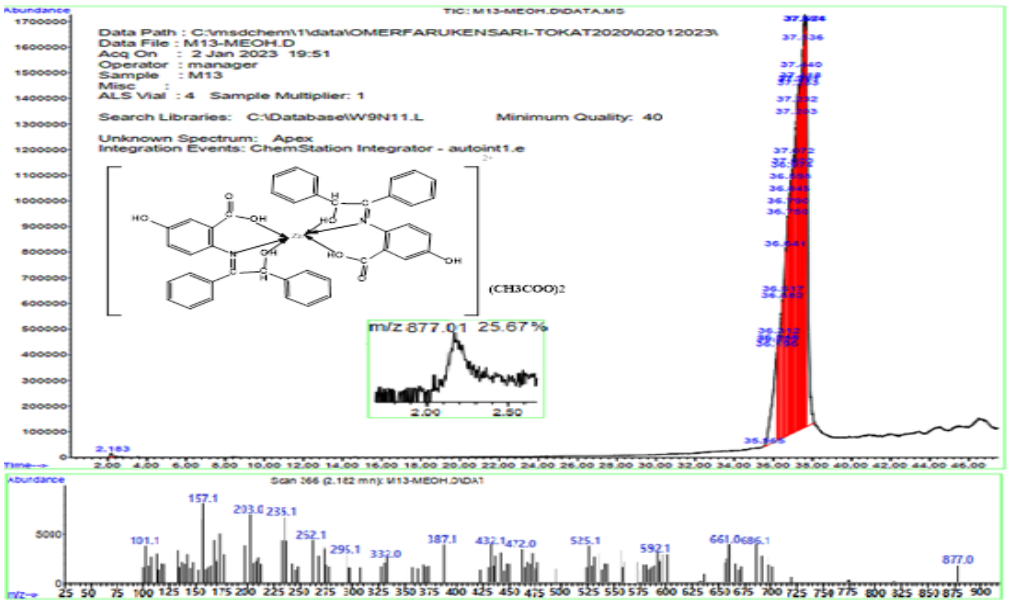

Fig 6: GC-MASS of complex NO.7

4.11. Biological Activity

A variety of bacteria, including "*Staphylococcus aureus, Escherichia coli, Pseudomonas aeruginosa, and Klebsiella spp*.," were used to screen the antibacterial properties of the ligand and their metal complexes The -C=N- group of the cell enzyme was one of the examined complexes that were engaged in a competitive equilibrium. **[12]** The molecules that were predicted to be attached to the cell enzyme's -OH group in this instance had a stronger reaction than the oxygen in the ligand's donor atom effect.

An active diffusion technique was used in agar plates to evaluate the synthesized ligand and complexes that were dissolved in DMSO **[13]**. After that, the plates were incubated for 24 hours at 37^{o}C. The zones of inhibition were measured in millimeters following the incubation period. The ligand and metal complexes showed impressive antibacterial properties **[14]**.

Table 7:Antibacterial activity (inhibition zone) of different concentrations of the ligand and complexes (µg/mL)

Compound	***Staphylococcus aureus***			***Escherichia coli***			***Psudomonus auruginosa***			***Klebsiella spp***		
Cons\µg	125	250	500	125	250	500	125	250	500	125	250	500
BMA	10	14	17	9	11	13	10	12	15	11	14	15
BMS	9	16	18	9	13	15	8	11	14	8	9	9
1	9	10	13	6	12	13	7	11	13	9	11	14
2	7	11	15	7	11	12	8	10	12	8	14	11
3	8	12	14	8	9	9	6	10	11	9	15	13
4	9	11	14	8	14	17	7	12	11	7	14	17
5	8	14	11	6	8	9	7	15	16	9	13	18
6	9	15	13	6	13	14	8	9	12	8	14	17
7	7	14	17	8	12	12	8	12	11	7	11	15
8	9	13	18	8	14	18	9	12	19	9	12	18
9	8	14	17	7	12	19	8	14	18	8	11	21
10	7	11	15	8	16	18	7	16	20	7	9	12
11	7	12	19	7	11	17	6	13	20	7	10	18
12	8	10	17	9	17	20	7	12	18	8	11	19
CIPS	10			10			10			10		
TMP	12			4			10			0		

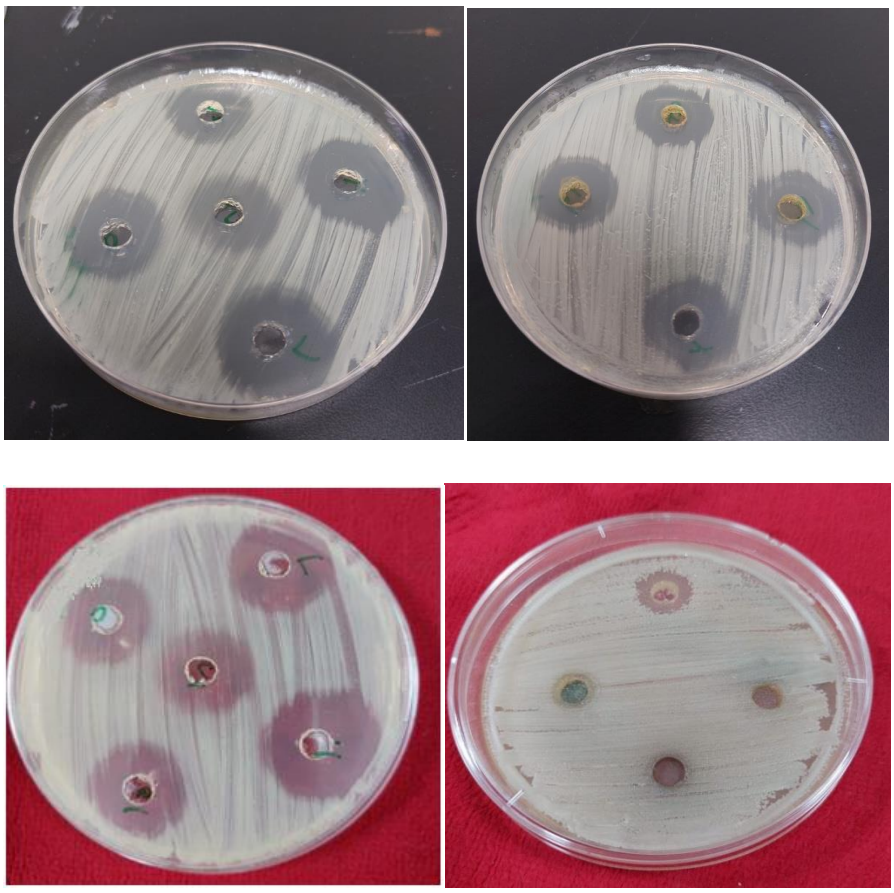

Fig 7: petri dish showing the inhibition zone of different bacteria

5. Conclusion.

According to the analytical, physical, and spectral results the data observed have established the following points: Both ligands BMA and BMS acted as tridentate chelating ligands, joined to the metal ion through the nitrogen and oxygen atoms.in neutral and basic medium All the resulting complexes in the neutral medium were ionic and had the formula [M $(L)_2$]$(X)_2$, X= NO_3 or CH_3COO, and having the formula [M $(L)_2$] basic medium. Zn^{++}, Mn^{++} (II), and Ag^{+} complexes of Zn^{++}, and Mn^{++} were proposed to be hexa-coordinated and forming octahedral geometries. But Ag^{+} complexes were proposed to be tetra-coordinated and form tetrahedral geometries The ligand and metal complexes showed very good antimicrobial properties.

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