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# Synthesis, Characterization and Biological activity Study of Some New Metal Complexes With Schiff's Bases Derived from [*O*-Vanillin] With [2-Amino-5-(2-Hydroxy-Phenyl)-1,3,4-Thiadiazole]

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**Abstract**

This study involves the preparation of a new series of dinuclear complexes Cr(III), Co(II), Ni(II), Cu(II) and Zn(II) complexes of the Schiff base ($H_2L$) derived from Vanillin with 2amino-5-(2-hydroxy-phenyl)-1,3,4-thiadiazole has been synthesized tetradentate Schiff base ligands were used for complexation upon two metal ions of Cr(III),Co(II), Ni(II), Cu(II) and Zn(II) as dineucler formula $M_2L_2.4H_2O$ and $M_2L_2$. These ligands can be characterization IR ,UV-Vis , Mass, $^1$H-NMR and elemental microanalysis. The Synthesized complexes were characterized by IR, spectroscopy ,elemental microanalysis, electronic spectra, magnetic susceptibility conductive ,thermal Analysis (TGA) and atomic absorption on the basis of analytical data, the stoichiometry of metal to ligand in complexes and Schiff bases ligand. The Structures of complexes were proposed from the measurements. The bioactivity of the prepared complexes has been examined with antibacterial activity. Antimicrobial activities of the Schiff base ligand and their metal complexes reveal that the Schiff base transition metal complexes show significant activity against some fungi and bacteria.

**Keywords**: Tetradentate Schiff base, (1,3,4-thiadiazole), Vanillin, antimicrobial studies, TGA.

## 1. Introduction

Schiff bases are important special and effective multi-dentate Schiff bases are widely studied in coordination chemistry, especially those that possess compounds containing heterocyclic compounds with the azomethene group, as they have basic properties due to the presence of a pair of electron on the azomethene nitrogen atom (-C = N) and often they are pentagonal or hexagonal rings with the metallic ion [1-4].

The Schiff bases heterocyclic metals complexation have been intensively investigated in recent years in many applications such as in antibiotics and medicine, catalyst [5], Thiazole compounds are related and have diverse bioactivity Activity possibly via N-C-S binding, which is of good importance in many pesticides.The rules have recently gained great importance due to their diverse biochemical properties[6]. The Schiff rules are derived from Vanillin and 2-amino-5-(2-hydroxy-phenyl)-1,3,4-thiadiazole have an importance in the formation of complexes and in this study a number of Cr(III), Co (II), Ni (II), Cu (II) and Zn (II) complexes . were characterized and diagnosed by different spectroscopy methods.

## 2. Experimental *Section*

*2.1.Materials*

All chemicals were obtained from supplied ( Sigma-Aldrich ) companies.

*2.2. Instrumentation*

The electronic spectra registered by using Shimadzu 160 A- Spectrophotometer . Mass analysis of ligand has been done with LC-Mass 100P Shimadzu .The IR spectra of ligand and complexes have been obtained (as a discs of KBr) in the range (4000-400) $cm^{-1}$. (Shimadzu 8300) device was used to conduct magnetic sensitivity measurements at room temperature using the (Faraday Method) method. A.A.S.

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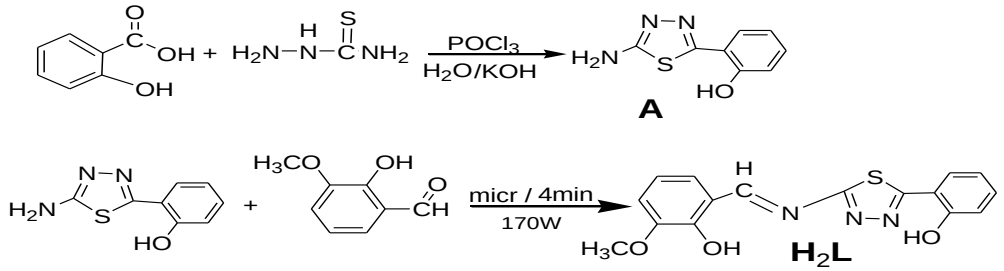
Spectrophotometer model Double-beam atomic absorption spectrometer, model: AA400 Analytic Jeana (made in Germany). Conductivity Measurements were performed with a conductivity meter Conductivity Meter Model PCM 3 - JENWAY. C.H.N.O analysis was carried out using analyzer model 5500 Carlo-Erba. Thermal analysis studies of the compounds were performed on Metther instrument TGA.

*2.3. Preparation of compound [A] [7]*

M.p. yield, C.H.N. analysis in Table (1).

*2.4. Syntheses 2- [5-(2-Hydroxy-phenyl)-[1,3,4]thiadiazol-2-ylimino]-methyl-6-methoxy-phenol [$H_2L$] [8-9]*

In crucible add mixture (0.02 mol) of compound [A] with same amount of *O*-Vanillin , wer put in microwave irradiation 170W for (4) minutes , after completion the reaction the obtained solid was recrystallized by absolute ethanol, some of proparties are listed in Table (1).




**Scheme (1) Synthesis of ($H_2L$) ligand**

*2.4.Preparation of Metal Complexes*

An methanolic solution of the ligand (0.02 mol in 20 ml mthanol) was added to few drops of Triethylamine before mixing in 50ml round bottom flask , then we add (0.02 mol) metal ions Cr(III) ,Co(II), Ni(II), Cu(II) and Zn(II), Chloride dissolved in 20 ml Methanol was put in ultrasonic bath 60 $^0$C. After 60 miuntes crystalline colored precipitates formed after cooling at room temperature, the resulting solids were filtered off, washed with distilled water & ether ,dried in a desiccator. some roperties are shown in Table (1).

*2,5,Stoichiometric Determination of Complexes:*

Mole ratio and Continuous variation (JOB) method was used make sure to the correlation ratio between ions and ligand in equilibrium media.

**Table (1) ligand and Metal percentages with Yield**

| Compound Formula | Yield% | Analysis (calculated) | | | | | |
|---|---|---|---|---|---|---|---|
| | | %C | %H | % N | %O | %Cl | %M |
| A | %72 | 49.51 | 3.81 (3.65) | 21.61 | 8.22 (8.28) | --- | -- |
| $C_8H_7N_3OS$ | | (49.73) | | (21.75) | | | |
| $H_2L$ | %81 | 58.94 | 3.91 (4.00) | 12.71 | 14.49 | --- | -- |
| $C_{16}H_{13}N_3O_3S$ | | (58.70) | | (12.84) | (14.66) | | |
| $[Cr_2 (H_2L)_2 (H_2O)_4] Cl_2$ | %53 | 43.09 | 3.31 (3.37) | 9.22 (9.36) | 17.71 | 7.73 | 11.68 |
| | | (42.82) | | | (17.82) | (7.90) | (11.59) |
| $[Co_2 (H_2L)_2 (H_2O)_4]$ | %69 | 50.28 | 2.97 (2.89) | 10.69 | 12.37 | | 15.21 |
| | | (50.01) | | (10.93) | (12.49) | | (15.34) |
| $[Ni_2 (H_2L)_2]$ | %73 | 50.37 | 2.97 (2.89) | 10.73 | 12.37 | | 15.14 |
| | | (50.04) | | (10.94) | (12.50) | | (15.28) |
| $[Cu_2 (H_2L)_2(H_2O)_4]$ | %67 | 49.71 | 2.94 (2.85) | 10.67 | 12.18 | | 15.13 |
| | | (49.42) | | (10.81) | (12.34) | | (15.28) |
| $[Zn_2 (H_2L)_2(H_2O)_4]$ | %65 | 49.47 | 2.91 (2.84) | 10.59 | 12.15 | | 16.59 |
| | | (49.18) | | (10.75) | (12.28) | | (16.74) |

## 3.Results and Discussion

***3.1.*** *spectra of $H_2L$*

The method for preparing ligand (L) is illustrated in **Scheme** (1). The infrared spectra of the prepared ligand showed the disappearance of the bundles of the carbonyl group of the aldehyde in the region(1665) cm$^{-1}$ and the amino group (-$NH_2$) in the region (3402-3213) cm$^{-1}$ and the emergence of new beams, which are the bundles of the right group, and the absorption beams of the imine group of the prepared ligand were in the range (1630)cm$^{-1}$ which belongs to the azomethene group, and the frequencies of the thiadiazole ring appeared at (1178-1303) cm$^{-1}$ [10]. Fig. (1) & Table (2) contain the values of the infrared spectra of the prepared ligand.

The molecular weight was measured using mass spectroscopy (LC-MS) with (SIM) technology, which determines the molecular mass of the material to be analyzed without fragmentation. The mass spectral data of Schiff base showed molecular ion peaks, which were in good agreement with the expected values. The mass spectrum of ligand $H_2L$ gives a peak at *m/z*=327.2 Fig. (2) and the theoretically calculated mass 327.36**.** Fig.(3): $^1$H-NMR($CDCl_3$-400MHz) δ= 11.909, 13.074(s,2H, OH), 9.669 (s, H, CH=N), 7.041-7.885 (m,7 H, Ar-H), 3.753 (s, 3H, $CH_3$), 1.57-0.8 (solvent+$H_2O$)

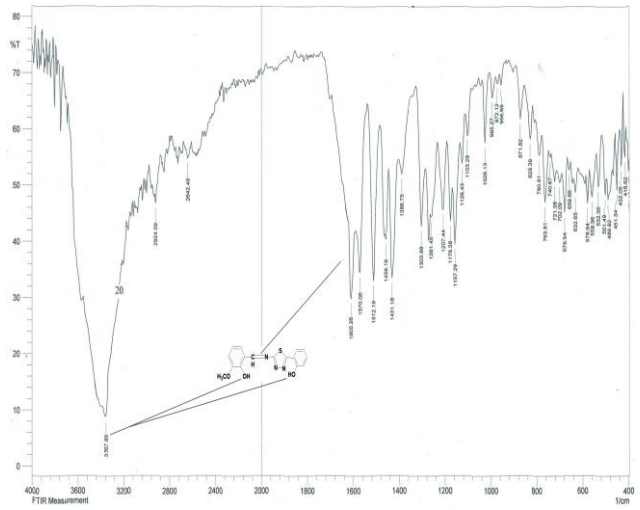


Fig. (1): IR for Ligand $H_2L$

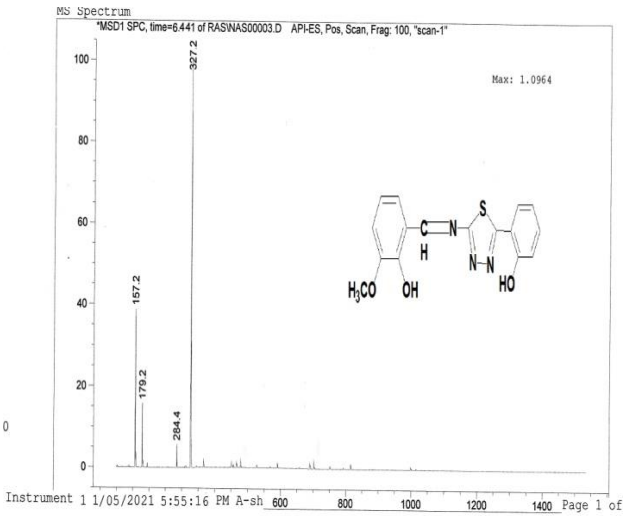



Fig. (2) : LC-Mass for Ligand H2L

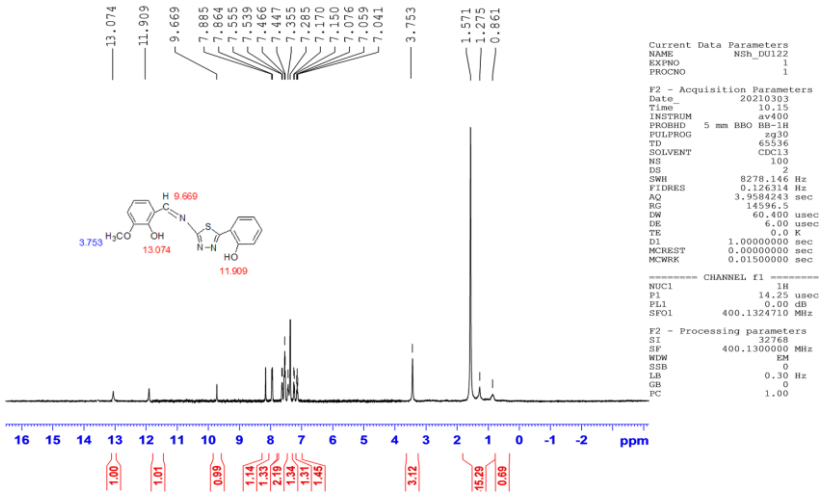



Fig. (3): $^1$H-NMR for Ligand $H_2L$

*3.2.Infrared (FT-IR) spectra of complexes.*

The all infrared bands assignments of the compounds are presented in (Table 2).The amplitude frequency of the amine group υ (C = N) in the ligand appeared the region (1630). It was also found that these stretchy frequencies in all the complexes shifted to lower frequencies that differ from what is in the ligand when linked by the nitrogen atom, which indicates the participation of (C=N) in coordination with the metal, this is consistent with the aforementioned researches related to Schiff's rules [11]. The absorption bands of ligand appear in the range (1174 -1303) $cm^{-1}$ and when complexes are formed are shifted are attributed to the bonds (= N-N =) in the thiadiazol ring, which confirms the metal's binding to the ligand by group (= N-N =), and this is in agreement with the aforementioned research related to thiazole [12]. As shown in the tables, the disappearance of the broad absorption bands ranging in the range (3414 $cm^{-1}$) to (3477 $cm^{-1}$) belonging to the hydroxyl phenolic (O) complexes is an evidence of its chelation by the phenolic oxygen atom [13]. The infrared spectrum showed a stretching of the group (M-N) of the prepared complexes in the bounded region between (419-453cm-1), confirming the metal's association with Schiff's bases via the nitrogen atom of the imine group [14] and also indicating the binding of the metal in the prepared complexes. Through the nitrogen atom of the thiadiazole group. In most of the complexes, a new beam appeared in the range (571-541) $cm^{-1}$ due to the vibrations of the group stretch (M-O) [11], indicating that the metal in these complexes is bound to the oxygen atom in the ligand. In addition to the three main forms of the free water molecule, water is the harmonic also shows other forms, oscillating and stretching [15]. The M–O modules are effective in the infrared spectrum if the M–O beam is sufficiently coherent. The presence of these beams in aqueous complexes at (783-758)$cm^{-1}$ for inorganic salts is a oscillating form of harmonic water evidence of its coordination of water[16]. The bending of the water complexes appear by about (481-756)$cm^{-1}$ [17] . All the infrared spectrum values for the complexes are shown in Table (2).

**Table (2) FT-IR data of Ligand and its metal complexes ($cm^{-1}$)**

| Symbol | ν(C=N) | ν(H-O) | ν(C-N=N-C) | Wagging υ $H_2O$ | twisting υ $H_2O$ | ν ($H_2O$) | ν (M-N) | ν (M-O) |
|---|---|---|---|---|---|---|---|---|
| HL1 | 1630(s) | 3369 | 1174-1303 | - | - | | - | |
| $[Cr_2 (H_2L)_2 (H_2O)_4] Cl_2$ | 1598 | | 1157-1290 | | | 3345 | 445 | 588 |
| $[Co_2 (H_2L)_2 (H_2O)_4]$ | 1616(s) | -- | 1238-1311 | 622 | 756 | 3378 | 486 | 540 |
| $[Ni_2 (H_2L)_2]$ | 1600(s) | ---- | 1157-1327 | ----- | ------ | - | 449 | 597 |
| $[Cu_2 (H_2L)_2(H_2O)_4]$ | 1612(s) | ----- | 1240-1308 | 481 | 590 | 3417 | 497 | 570 |
| $[Zn_2 (H_2L)_2(H_2O)_4]$ | 1602(s) | ----- | 1230-1295 | 499 | 595 | 3393 | 452 | 551 |

*3.3.Electronic Spectra , Magnetic Moments and Molar Conductance of Complexes***:**

The Fig. (4) (UV-Vis) spectra of the summation prepared in the DMF solvent showed a main absorption peak, the first (230nm) representing local excitations ($\pi \rightarrow \pi^*$) of the thaidiazole ring and other aromatic rings and the second (290 nm) attributing to the transitions ($n \rightarrow \pi^*$) resulting from the presence of sums (-O-H) and (- C = N) carrying nonbonding pairs [18].

When comparing the ligand spectra with the spectra of the complexes Fig. (4) under study, a displacement was observed , it ranged between (5-25) nanometers and there is a difference between the spectra of the solutions of ligand and the metal ion, as well as the clear difference in the colors of the mixing solutions from the solutions of the ligand and the metal ion before mixing, which is clear evidence of a coordination between them [19]. The locations of these bands correspond to the complexes Cr(III) ,Co(II), Cu(II) and Zn(II) of the octahedral but Ni(II) squire planer [20-24]. In this case the magnetic moment for Cr(III) ,Co(II), Ni(II), Cu(II) and Zn(II) complexes are 3.9, 4.9, Dia,1.89 and Dia B.M respectively which confirmed the octahedral geometry but Ni(II) squire planer complex [19] . the theoretically calculated magnetic moment values differ from the practical due to the orbital contributions Table (3) gives the electronic spectral, magnetic moments and Molar Conductance data of the prepared compounds. The results of the magnetic susceptibility gave values for the magnetic moment which correspond to the suggested shape.

*3.4.Biological Activity*

The drilling method experiment was conducted and the experiment was conducted under aerobic conditions (temperature of 37 ° C), four types of pathogenic bacteria were grown *Staphylococcus aureus, Escherishia coli, Pseudononas aeroginosa and*

*Streptococcus pneumonia*. (two negative for Gram stain and two positive for Gram stain, the compound is effective against positive stain bacteria. *Staphylococcus* aurous and *Streptococcus pneumonia*) are effective against *Escherichia coil* negative bacteria only at 200 ml / mg [25,26).

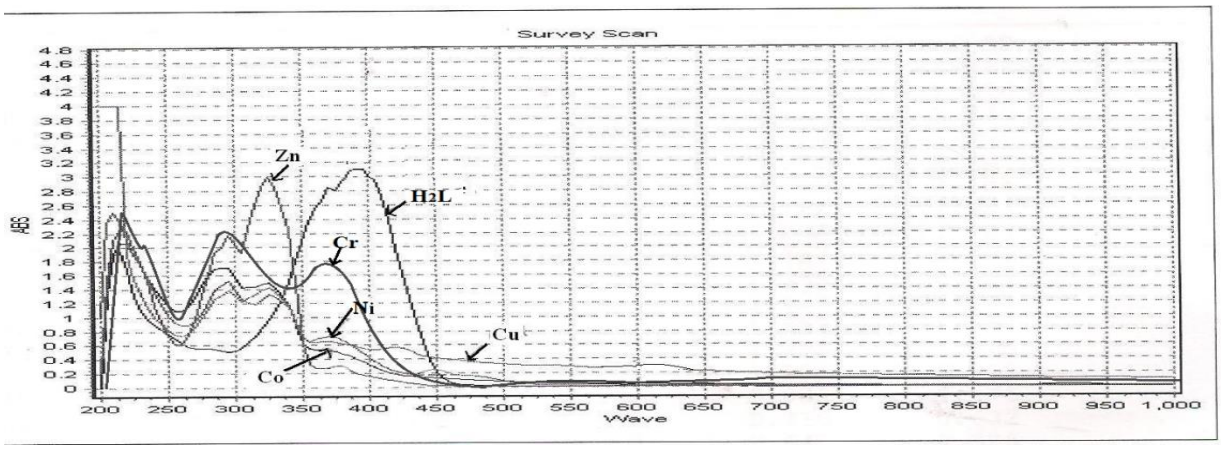



**Fig. (4): (UV-Vis) spectra**

**Table (3) Some physical data electronic spectra for ligand and complexes in DMF**

| Symbol | Dec. Point $^0C$ | Conductivity $ohm^{-1}cm^2mol^{-1}$ $25^0C$ | Magnetic Moment (B.M) (Calc. Thior) Found | Color | Absorption Bands (nm) | Assigned Transition |
|---|---|---|---|---|---|---|
| A<br>$C_8H_7N_3OS$ | 244 | 3 | - | White-yellow | 215<br>285 | $\pi \rightarrow \pi^*$<br>$n \rightarrow \pi^*$ |
| $H_2L$<br>$C_{16}H_{13}N_3O_3S$ | 221-223 | 4 | - | yellow | 230<br>390 | $\pi \rightarrow \pi^*$<br>$n \rightarrow \pi^*$ |
| $[Cr_2(H_2L)_2(H_2O)_4]Cl_2$ | 300d | 153 | (3.87)<br>3.9 | Violet | 693<br>549<br>370 | $^4A_2g \rightarrow {}^4T_2g$ (F)<br>$^4A_2g \rightarrow {}^4T_1g$ (F)<br>Charge Transfer |
| $[Co_2(H_2L)_2(H_2O)_4]$ | 290d | 15 | (5.2)<br>4.90 | Dark Brawn | 631<br>485<br>370 | $^4T_1g(F) \rightarrow {}^4A_2g(F)$<br>$^4T_1g(F) \rightarrow {}^4t_1g(p)$<br>Charge Transfer |
| $[Ni_2(H_2L)_2]$ | 300d | 23 | (zero)<br>Dia | Dark olive | 529<br>445 | $^1A_1g \rightarrow {}^1A_2g$<br>$^1A_2g \rightarrow {}^1B_1g$ |
| $[Cu_2(H_2L)_2(H_2O)_4]$ | 285d | 17 | (1.8)<br>1.89 | Brawn | 436<br>644 | Charge Transfer<br>$^2Eg \rightarrow {}^2T_2g$ |
| $[Zn_2(H_2L)_2(H_2O)_4]$ | 290d | 21 | (zero)<br>Dia | Light-yellow | 364 | Charge Transfer |

**Table (4)Antibacterial activity of the prepared compounds**.

| *Symbol* | *Staphylococcus aureus* | *Escherishia coli* | *Pseudononas aeroginosa* | *Streptococcus pneumonia* |
|---|---|---|---|---|
| $H_2L$ | ++ | ++ | - | ++ |
| $[Cr_2(H_2L)_2(H_2O)_4]Cl_2$ | +++ | + + | + | +++ |
| $[Co_2(H_2L)_2(H_2O)_4]$ | +++ | + + | - | +++ |
| $[Ni_2(H_2L)_2]$ | +++ | + + | - | +++ |
| $[Cu_2(H_2L)_2(H_2O)_4]$ | +++ | + + | + | +++ |
| $[Zn_2(H_2L)_2(H_2O)_4]$ | +++ | + + + | + | +++ |

Note (-) = no inhibition, (+) = (5-10) mm,

(+ +)=(11-20) mm, (+ + +) = more than (20)mm

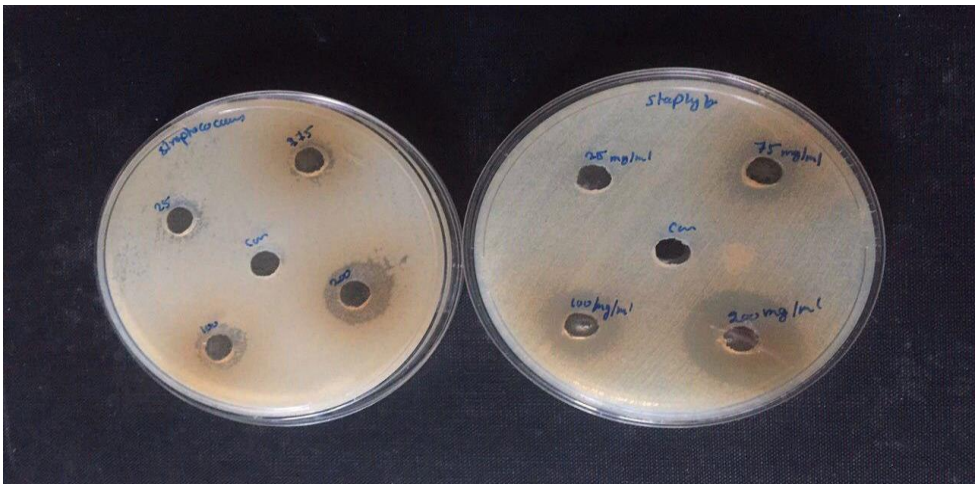
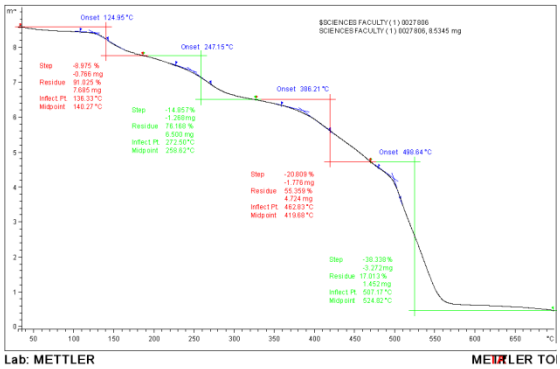
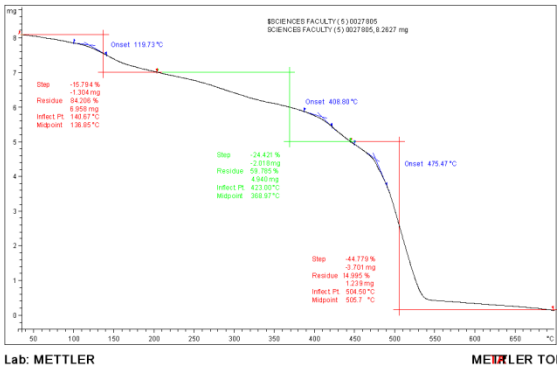


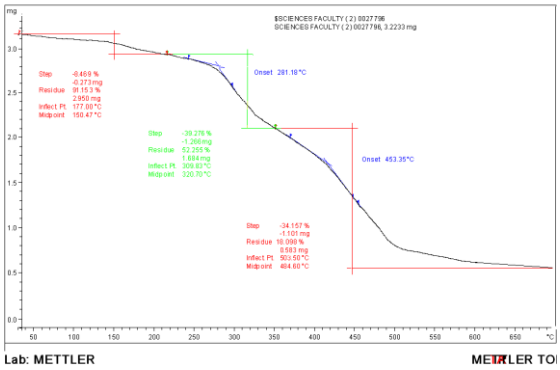
**Fig. (5):- Antibacterial activity of Schiff base ligand**



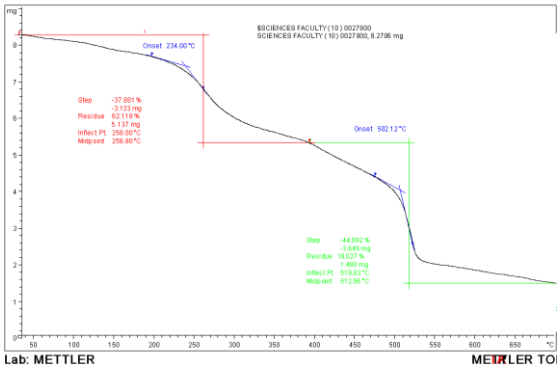

$[Co_2(H_2L)_2(H_2O)_4]$



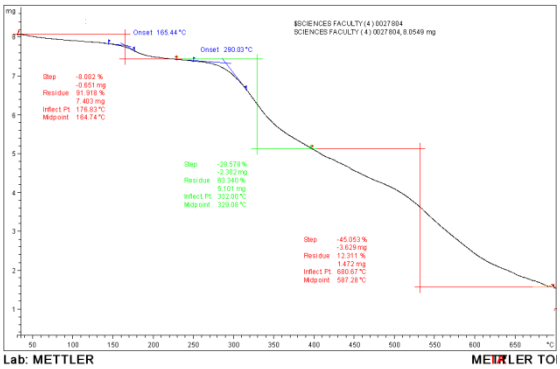

$[Cr_2(H_2L)_2(H_2O)_4]\ Cl_2$




$[Cu_2(H_2L)_2(H_2O)_4]$




$[Ni_2(H_2L)_2]$




$[Zn_2(H_2L)_2(H_2O)_4]$

**Fig. (6):Thermal Analysis of Complexes**

*3.5.Thermal Analysis (TGA)*

The TGA have been carried out in the range of (37–700 °C) at a heating rate of 5 °C/min in nitrogen atmosphere (TGA) weight loss curves and the corresponding (TGA) curves for the complex are shown in Fig 6. The complex showed (2-4) well-defined steps. The first step in the thermal range (37-140) °C for Cr(III) loss in weight 15.7% represent ($4H_2O$+2Cl) but Co(II) (37-140) loss in weight 8.963% , Cu(II) (37-177) loss in weight 8.48% and Zn (II) (37-176), loss in weight 8.08% respectively represents the loss of four water molecules, and this is evidence of the two coordinated water molecules in complexes [27]. The second, third and fourth steps weight losses are explained in Table (5). These steps are a loss of mass in the form of gases. Final step large weight drop can be explained by considering that the residue is a 1 : 1 mixture of (2MO).

## 5.Conclusions

We have observed new ligand compounds and their complexes from the first series transitional metals (studies of their physical properties and various analyzes). The collected data demonstrated that the ligand behaves a tetradentate ligand of $N_2O_2$; binuclear complexes Stable. From the electronic spectra, infrared spectrum , magnetic measurements it is indicated that most of Cr(III) ,Co(II), Cu(II) and Zn(II) complexes contain hexa coordinate and have octahedral geometry, while the Ni (II) complexe tetra coordinate have square planner geometry . Molar conductivity measurements of the prepared Complexes indicates

that complexes with the formula $[M_2(H_2L)_2(H_2O)_4]$ with M(II)= Co, Cu and Zn and $[Ni_2(H_2L)_2]$ were neutral (non electrolyte), while the other complexes with the formula $[Cr_2(H_2L)_2(H_2O)_4]$ $Cl_2$ and were electrostic type (1:2).

Table (5). TGA analysis data of complexes

| Sample (step) | T.range ºC | Weight mass loss (calc) found% | Reaction |
|---|---|---|---|
| Cr(1) | 37-140 | (15.94) 15.70 | $(4H_2O+2Cl)$ |
| Cr(2) | 140-423 | (24.06) 24.421 | $C_{14}H_{16}O_2$ |
| Cr(3) | 423-506 | (44.77) 45.26 | $C_{18}H_{10}N_6O_2S_2$ |
| | Final residual | (14.77) 15.16 | $2CrO^+$ |
| Co(1) | 37-140 | (8.572) 8.963 | $4H_2O$ |
| Co(2) | 140-272 | (14.28) 14.85 | $C_6H_{16}O_2$ |
| Co(3) | 272-462 | (20.24) 20.80 | $C_{10}H_{18}O_2$ |
| Co(4) | 462-525 | (38.60) 38.338 | $C_{16}H_{12}N_4S_2$ |
| | Final residual | (17.84) 17.13 | 2CoO |
| Ni(1) | 37-256 | (38.83) 37.88 | $C_{16}H_{14}N_2O_4$ |
| Ni(2) | 256-512 | (44.09) 45.14 | $C_{16}H_{10}N_4O_2S_2$ |
| | Final residual | (19.45) 18.027 | 2NiO |
| Cu(1) | 37-177 | (8.72) 8.48 | $4H_2O$ |
| Cu(2) | 177-320 | (38.17) 39.27 | $C_{16}H_{12}N_4S_2$ |
| Cu(3) | 320-503 | (35.10) 34.16 | $C_{16}H_{14}N_2O_4$ |
| | Final residual | (18.72) 18.09 | 2CuO |
| Zn(1) | 37-176 | (8.44) 8.08 | 4H2O |
| Zn(2) | 176-332 | (28.85) 28.57 | $C_{14}H_{14}O_4$ |
| Zn(3) | 332-680 | (44.56) 45.10 | $C_{16}H_8N_6O_2S_2$ |
| | Final residual | (19.072) 18.09 | 2ZnO |

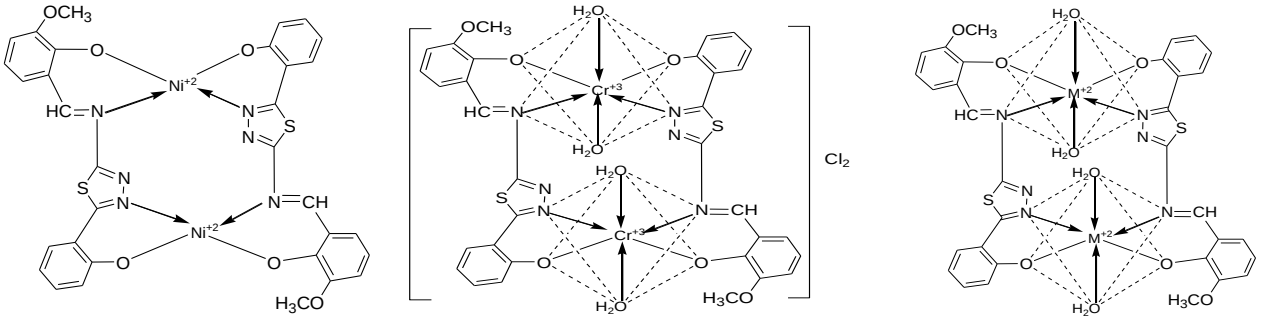


**Fig (7) suggested structure for complexes / M= Co(II), Cu(II) and Zn(II)**

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**الخلاصة:-**

تضمن هذه الدراسة تحضير سلسلة جديدة من المعقدات ثنائية النواة من عناصر السلسلة الانتقالية الأولى الكروم (III) الكوبلت (II) والنيكل (II) والنحاس (II) فضلاً عن الخارصين (II) لقاعدة شيف ($H_2L$) المشتقة من اورثو الفانيليين مع 2- امينو-(5- هيدروكسي-فينيل ) 4,3,1 - ثايو دايزول .ليكاند رباعي السن حيث تم تحضير معقدات ثنائية النوى من أيونات معدنية من Cr (III)، Co (II)، Ni (II)، Cu ( II ) و Zn (II) بصيغة $M_2L_2.4H_2O$ و $M_2L_2$. تم التحقق من الصيغ التركيبية لليكاندات المعقدات المحضرة بالطرائق الفيزياوية المعروفة مثل درجة الانصهار والتحليل الدقيق للعناصر (C.H.N.O), التوصيلية الكهربائية المولارية والحساسية المغناطيسية والتحليل الحراري (TGA) والأطياف الالكترونية وطيف الأشعة تحت الحمراء وتم تقدير نسبة الفلزات بطريقة الامتصاص الذري و ايجاد النسبة المئوية للكلوريد في المعقدات قياس الكتلة (LC-MS) و $^1$H-NMR لليكاندا المحضر على ضوء نتائج أطياف الاشعة فوق البنفسجية - المرئية، الحساسية المغناطيسية والتوصيلية المولارية أظهرت النتائج ان المعقدات ذات شكل ثماني السطوح بينما اعطى النيكل شكل مربع القاعدة.درست الفعالية البكتيرية لكل من الليكاند ومعقداته ضد انواع منتخبة من البكتريا.