

Preparation, Characterization and studied Biological Effect as Antioxidant of Azo Compound and Schiff base Complexes

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Abstract

The advance of compounds that can have different biological effects and effects on the cell of the organism and the study of their physical and spectral properties. The importance of studying the biological effectiveness of a number of prepared compounds containing different groups of compensators, which may have an impact in the biological field or the industrial field. And based on what the transition elements possess of distinctive properties such as their possession of multiple oxidation states, as well as their strong tendency to form ionic or neutral complexes with different degrees of complexity, therefore, selective preparation of a very wide range of therapeutic agents containing transition elements can be carried out and the behavior towards the formation of complexes with living ligands can be studied. Participating in different life processes, as well as forming mathematical and chemical models for life systems with these compounds, and finally controlling these systems and limiting the optimal conditions for their work. It is possible to design new materials with expected effectiveness within this field, and thus the life inorganic chemistry has made great efforts through its applications in the field of medicine not only the necessary elements but also the non-essential elements and even radioactive ones, and finally the medical inorganic chemistry has become a real possibility to discover types Drugs and drugs and reaction mechanics to form compounds with stable and stable dyes.

Keywords: *new mixed ligand complexes, azo and Schiff base ligands, antioxidants, Biological activity.*

1. Introduction

The importance of mixed ligand compounds has increased recently in every field , so they must used in many basic chemical, pharmaceutical and biological applications, where they acted as antioxidants, anticancer, antibiotics and anti-inflammatory, in addition to their effective role in oxidation and hydrogenation reactions.[1,2].Nitrogen-containing chalets are one of the furthermost important chalets in complexity and coordination with biological transition metals [3,4]. Complexes of transition elements with nitrogenous ligands stand of great rank in the

arena of organic and inorganic attraction because of their magnetic and electrical properties [3].Azo compounds are the azo bridge group (—N=N—) in which both ends of the two nitrogen atoms are linked to aliphatic or aromatic derivatives, which are characterized by high stability and are the most important and widely used. Mixed-ligand complexes derived from azo ligands and a Schiff base, with metal ions at two different sites, have given their biochemical significance and are of interest as a metalloenzymes model [3-5]. In addition of some imidazole derivatives can also be used as catalyst in industrial

applications [6-10]. Azo imidazole molecules as well as being an efficient π -acidic system, so these organic molecules use ligand functions to form stable complexes with transition metal ions [11-15]. In recent years, researchers have increased interest in involving ligands and their relations in medical treatment, as they have been used in the manufacture of medicinal drugs for diabetes and infections [16-20], as well as anti-cancer drugs, as in the case of triple gold complexes because they have high inhibitory effectiveness. In addition to its importance as antioxidants [21-25].

2. Chemical materials

These were delivered through many companies : A.K.Scie. and Germany, these materials were used without purification process

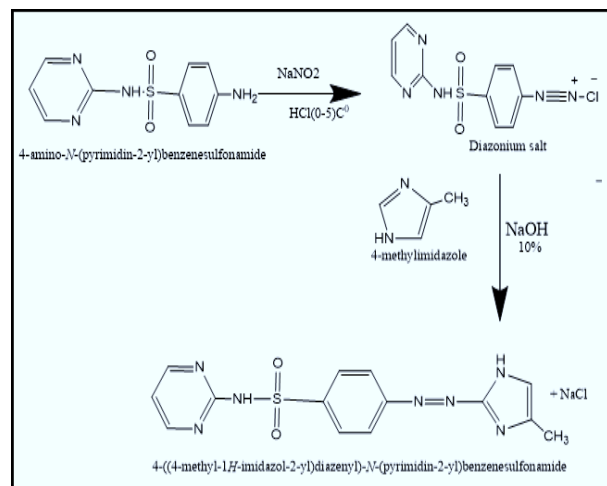
3. Measurement

All analysis have done in lab-center of measurement in kufa university ,while other resonance measurements in Iran universities .

4. Grounding of the azo Chalet

The chalet prepared via azotate of sulfadiazine compound with imidazole derivative to yield new chalet azo-imidazole that prepared according to studies [26-30].

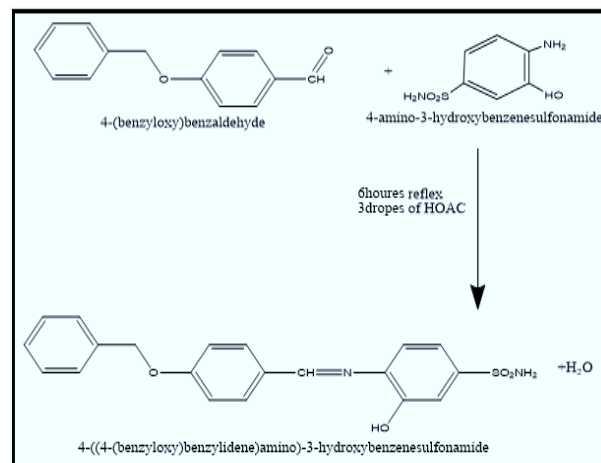
Scheme 1: Ligand azo (L1) preparation



5. Preparation of the Schiff base (HL2):

The chalet prepared via condensation reaction of primary amine compound with carbonyl derivative of aldehyde to yield new chalet of Imine-derivative that prepared according to studies [31-35].

Scheme 2 Ligand Schiff Base (HL2) preparation



6. Preparation complex of Mixed ligand L1 and HL2:

The reaction that includes the formation of complexes of mixed ligands is more stable than similar ligands, as both ligands can be prepared from the mixture of equal molar amounts of both compounds according to the optimal molar ratios for complex formation. As the factors on which the interference, consistency, or overlapping of the metal ion with the donor ligand atoms depends are well known and proven in many references, and this aspect is continuously enriched through ongoing research in the field of coordination chemistry, especially the study of factors affecting the stability of the complexes formed and the relationship of the nature of the ion. The nature of donor ligand atoms, their spatial distribution, and ligand size

7. Antioxidant commotion

Biological harmonious chemistry is a wide field through its presence in most life systems, cancer-preventing substances and tumors, metallic complexes with living systems that have an important role in the life of animals and plants. On the other hand, inorganic elements play a vital role in the fields of life and medical life, as it is clear that many organic compounds that are used in the field of medicine are not limited to organic mechanics only, but some organic compounds are active or biologically changed in the presence of metal ions, and it is indicated that Some of the heavy transition elements in particular (the second and third chain elements) have a toxic effect in the body, so it is necessary to design coordinating compounds (coordinating agents) that have the ability to remove the toxic effect

8. Cytotoxicity Assay

Some researchers began working on studying chemical compounds in the field of cancerous tumors and comparing infected cells with healthy cells using many metal complexes as a measure of the ability of drugs to treat diseases by knowing the ability of metal ion complexes

Table 1. Micro-element analysis result

Compound	Chemical formula	m.p	Y %	Color	C Found % (cal.)	H Found % (cal.)	N Found % (cal.)
Ligand=L1	C ₁₄ H ₁₃ N ₇ O ₂ S	177-180	82	Brown	48.93 (48.95)	3.76 (3.78)	28.63 (28.55)
Ligand=HL ₂	C ₂₀ H ₁₈ N ₂ O ₄ S	175-177	90	Greenish white	62.74 (62.79)	4.68 (4.70)	7.29 (7.30)
[Co(L ₁ L ₂)H ₂ OCl]	CoC ₃₄ H ₃₂ N ₉ O ₇ S ₂ Cl	310Dec.	73	Dark brown	23.86 (24)	3.16 (3.18)	41.14 (41.19)
[Ni(L ₁ L ₂)H ₂ OCl]	NiC ₃₄ H ₃₂ N ₉ O ₇ S ₂ Cl	148-150	78	Dark brown	23.88 (24)	3.11 (3.19)	41.17 (41.19)
[Cu(L ₁ L ₂)H ₂ OCl]	CuC ₃₄ H ₃₂ N ₉ O ₇ S ₂ Cl	148-150	76	Brown	23.77 (23.9)	3.15 (3.17)	40.66 (40.9)
[Zn(L ₁ L ₂)H ₂ OCl]	ZnC ₃₄ H ₃₂ N ₉ O ₇ S ₂ Cl	310Dec.	74	Red	23.76 (23.83)	3.14 (3.16)	44.39 (44.5)

to penetrate cellular membranes, while there are ligands and special ions This ability is often lost, and in this case it was found that antibiotics become highly effective and their activity increases in the presence of metal ions. Metal complexes differ in their mode of action and activity in the life system depending on their composition. For example, inert (stable) metal complexes have good effectiveness against microbes, fungi, and viruses. They are also used to control the spread of cancerous tissues.

9. Results and Discussion

All mixed ligand complexes in this research were Freely dissolvable by dimethyl formamide and other solvent, through all results ,we briefed that all chalets gave good results on cancer cells.

9.1. Element investigation

In table (1), the results of element investigation for share chalets 1:1:1 [L:M:L] exhibited that the results of theoretical were a good agreement with the found results. That were used to test the purity of mixed ligand.

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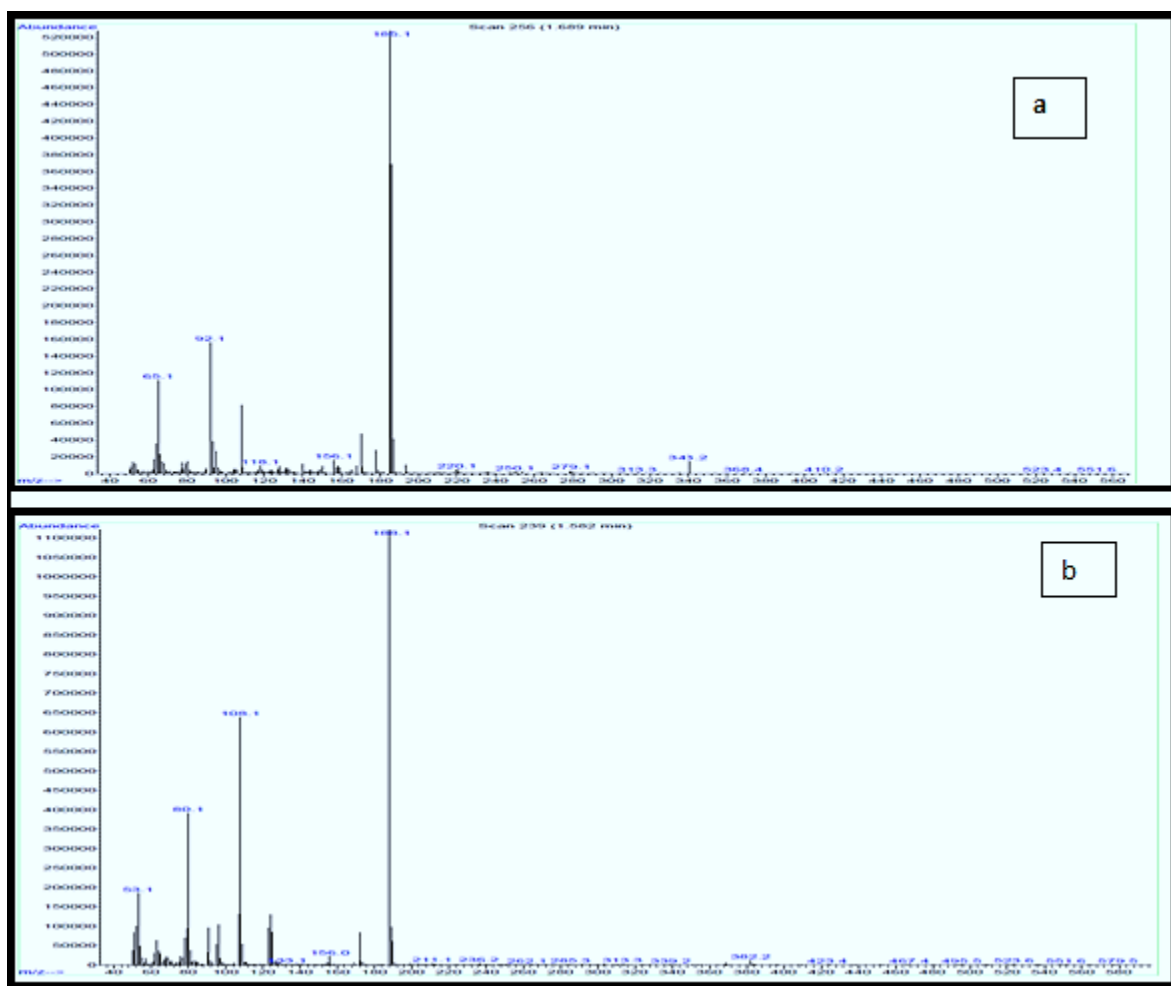
[Cd(L ₁ L ₂)H ₂ OCl]	CdC ₃₄ H ₃₂ N ₉ O ₇ S ₂ Cl	198-200	78	Dark orange	22.43 (22.54)	2.76 (2.99)	38.43 (38.96)
[Hg(L ₁ L ₂)H ₂ OCl]	HgC ₃₄ H ₃₂ N ₉ O ₇ S ₂ Cl	198-200	77	Dark red	20.55 (20.56)	2.46 (2.71)	34.89 (35)
[Au(L ₁ L ₂)]Cl ₂	AuC ₃₄ H ₃₀ N ₉ O ₆ S ₂ Cl ₂	198-150	70	Brown	19.72 (19.80)	2.58 (2.62)	33.74 (33.95)
[Fe(L ₁ L ₂) Cl ₂]	FeC ₃₄ H ₃₀ N ₉ O ₆ S ₂ Cl ₂	98-100	76	Brown	22.87 (23.10)	2.82 (3.06)	39.48 (39.60)

9.2. Mass spectra

The compound (C₁₄H₁₃N₇O₂S) and this confirms the proposed formula of the synthesized compounds. As well as, the mass

continuum of the chalet (HL2) exhibited the peak of derivative ion at m/z 382.2 (0.8%) compound(C₂₀H₁₈N₂O₄S)) and this confirms the proposed formula of the synthesized compounds.

Order.(1): (a) Mass continuums of the azo-chalet(L1),(b) Mass continuums of the ligand (HL2)



9.3.11H NMR Spectra of azo- ligand L1

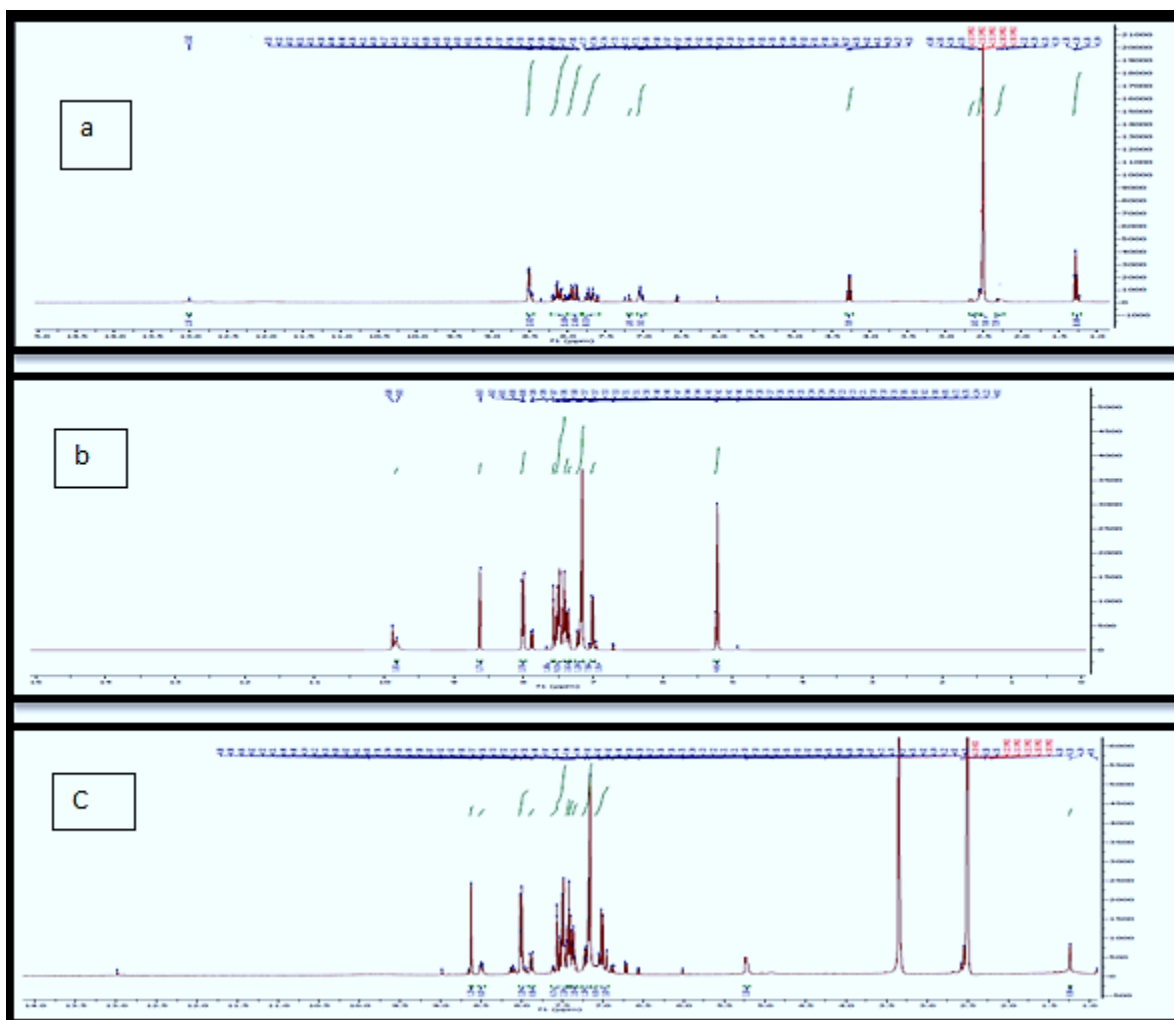
¹H NMR spectrum of this compound was recorded by DMSO-d₆. The singlet signal at δ 2.5 ppm was due to the solvent protons. The perfumed protons were seen in the range of δ 6.5 –8.5 ppm as multiple signals. CH₃ imidazole ring at 1.2 ppm, another signal at δ 13.0 ppm accredited to imidazole (-NH-) proton besides singlet at δ 4.3 ppm owing to (NH-) sulfadiazine [15,16,17]

9.3.2.1H NMR Spectra of schiff base- ligand HL2

The assignment of the main signal in the ¹H NMR spectra of Schiff-base ligand

showed a signal at δ 4.9 ppm was due to group (NH₂-) of sulfonamide ring. The aromatic protons were seen in the range of δ 6.7 –8.02 ppm as multiple signals. the singlet single at δ 8.6 ppm belong to proton (CH=N) of Schiff group, also another signal at δ 9.8 ppm attributed to group (OH). While complex (Cd) of mixed ligand show nearly the same signals which belong to L1, HL2 except disappear signal attributed to proton of (OH) since it contributed to coordination as the complex new signal at (δ 3.5)ppm refers to protons of water molecule inside coordination sphere.

Figure. (2). The ¹H NMR spectrum of ligand (a) (L1), (b) (HL2) (c) Cd complex of Mix ligand



10. Infrared continuums

The IR continuums of the chalets in all shapes by appearance many bands in figures as

formation ligands and chalet-ion as a results of coordination process [36-38].

Table 2 Characteristic IR frequencies (in cm⁻¹) of the ligand (L1) & (HL2) & Mixed ligand relations

chalets	(O-H) H ₂ O	NH ₂	H ₂ O	(C=N) Schiff	ν (C=N) pyrimidine	ν (C=N) imidazole	(N=N)	(SO ₂)	M-N	(M-O)
L ₁	-----	-----	-----	-----	1685	1664	1446	1157 1328	----- -	----- -
HL ₂	3600 (OH)	3383 3280	-----	1641	-----	-----	-----	1157 1323	----- -	----- -
[Co(L ₁)(L ₂)H ₂ OCl]	3100 3600	3375 3232	933	1591	1664	1504	1427	1155 1317	584	422
[Ni(L ₁)(L ₂)H ₂ OCl]	3487	3319 3251	931	1587	1644	1542	1413	1145 1311	588	426
[Cu(L ₁)(L ₂)H ₂ OCl]	3464	3385 3348	937	1591	1665	1508	1417	1153 1309	588	416
[Zn(L ₁)(L ₂)H ₂ OCl]	3431	3365 3251	933	1589	1666	1506	1429	1141 1334	586	424
[Cd(L ₁)(L ₂)H ₂ OCl]	3100 3400	3342 3246	935	1589	1676	1504	1408	1157 1315	540	422
[Hg(L ₁)(L ₂)H ₂ OCl]	3371	3336 3238	943	1585	1665	1508	1427	1153 1332	578	424
[Au(L ₁)(L ₂)]Cl ₂	----- --	3138 3045	----- -	1589	1687	1589	1489	1159 1328	576	430
[Fe(L ₁)(L ₂) Cl ₂]	-----	3361 3172	-----	1589	1664	1504	1414	1157 1328	514	433

11. Electronic Spectra

At room temperature, the spectra of electronic absorption of all the compounds were logged

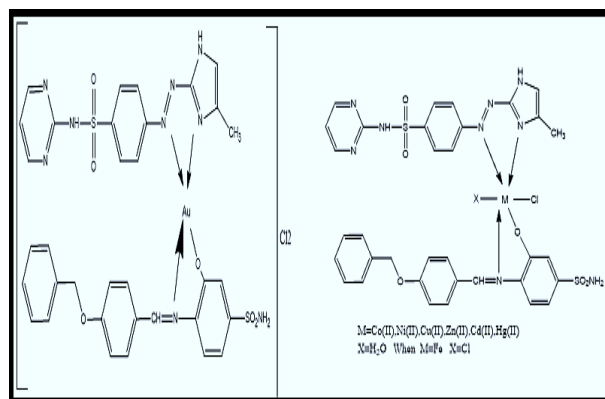
by exhausting solution of ethanol in the series 200-1100 nm. The spectral data of azo L₁ , Schiff base HL₂ ligands and their metal complexes are summarized in Table (3).

Table 3 Magnetic moment, and electronic spectra of complexes

Compounds	λ (nm)	ϵ (cm ⁻¹)	Transitions	Geometr y	Hybridization	μ_{eff} (B.M)
L ₁	208 248 380	48076 40322 26315	$\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	-----	-----	L ₁
HL ₂	217 268 380	46.82 43859 62315	$\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	-----	-----	

[Co(L ₁ L ₂) H ₂ OCl]	858 703 489 432	11655 14224 20449 23148	⁴ T _{1g} (F)→ ⁴ T _{2g} (F)= ν ₁ ⁴ T _{1g} (F) → ⁴ A _{2g} (F) = ν ₂ ⁴ T _{1g} (F)→ ⁴ T _{1g} (p) = ν ₃ M→L,CT	Octahedra 1	Sp ³ d ²	4.25
[Ni (L ₁ L ₂) H ₂ OCl]	703 671 586	14224 14903 17064	³ A _{2g} → ³ T _{2g} (F)=(ν ₁) ³ A _{2g} → ³ T _{1g} (F)=(ν ₂) ³ A _{2g} → ³ T _{1g} (p)=(ν ₃)	Octahedra 1	Sp ³ d ²	2.92
[Cu (L ₁ L ₂) H ₂ OCl]	858 505 435	11655 19841 22988	² B _{1g} → ² A _{1g} = ν ₁ ² B _{1g} → ² B _{2g} = ν ₂ ² B _{1g} → ² E _g = ν ₃	Octahedra 1	Sp ³ d ²	1.74
[Zn (L ₁ L ₂) H ₂ OCl]	529	18903	M→L,CT	Octahedra 1	Sp ³ d ²	Dia
[Cd (L ₁ L ₂) H ₂ OCl]	516	19379	M→L,CT	Octahedra 1	Sp ³ d ²	Dia
[Hg (L ₁ L ₂) H ₂ OCl]	511	19569	M→L,CT	Octahedra 1	Sp ³ d ²	Dia
[Au L ₁ L ₂]Cl ₂	431	23201	¹ A _{1g} → ¹ B _{1g}	square planer	dsp ²	Dia
[FeL ₁ L ₂ Cl ₂]	529 671 702 858 1072	18903 14903 14245 11655 9328	⁶ A _{1g} → ⁴ T _{1g} ⁶ A _{1g} → ⁴ T _{2g} ⁶ A _{1g} → ⁴ E _g , ⁴ A _{1g} ⁶ A _{1g} → ⁴ T _{2g} ⁶ A _{1g} → ⁴ E _g	Octahedra 1	Sp ³ d ²	5.41

Scheme 3: The proposed structural formula of the complex



12. Anticancer commotion of complex

It observed that half of the inhibitory concentration of cancer cells, IC₅₀, was (60.88 μg / ml), which is very low compared to healthy cells, where it was (675.51 μg / ml), and this is a good result. That is, the gold nano Au-complex kills breast cancer cells with high efficiency and has no effect on healthy cells. This is a very important result in the use of a complex of gold as a highly selective treatment for the treatment of breast cancer.

Figure (3): Percentage of inhibition in cells of (a) MCF-7 breast cancer line cell against the concentration of complex [Au L1L2]Cl₂ (b) normal line cell against the concentration of complex [Au L1L2]Cl₂

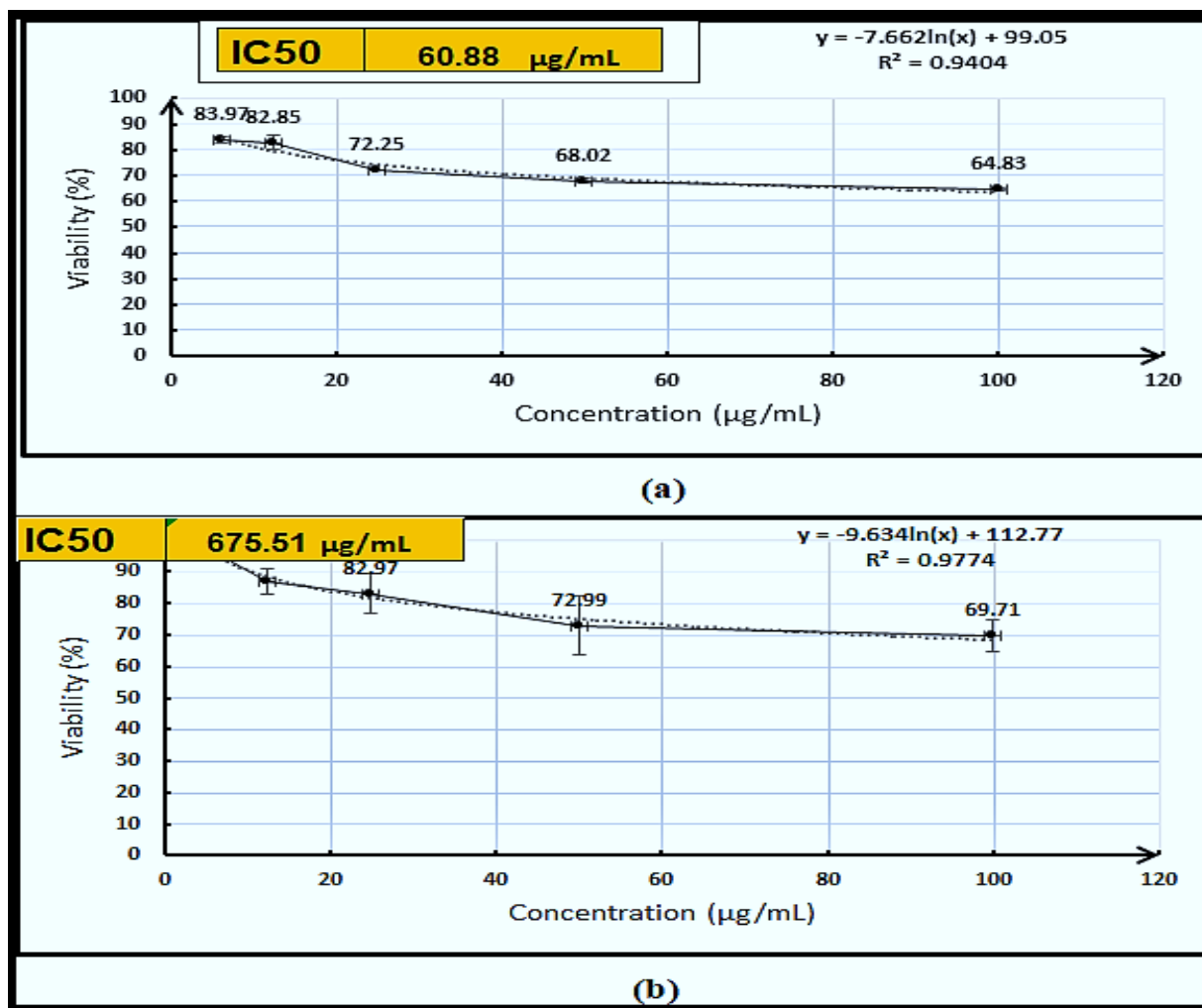
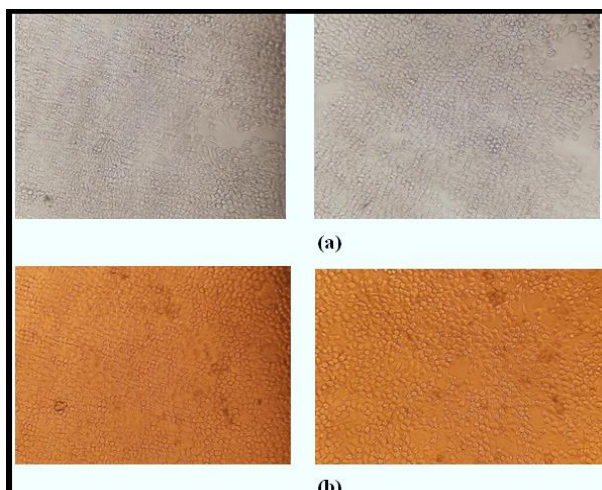


Figure (6): (a) health cell (b) cancer cell

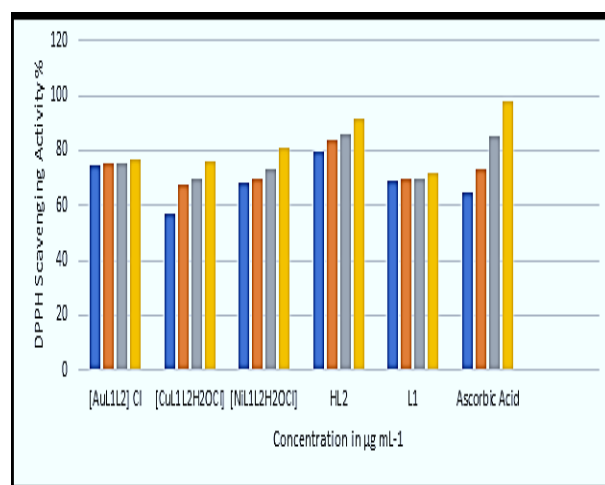


13. Antioxidant broadcast (DPPH radical rummaging commotion)

All results of evaluating activity of antioxidants showed that all the prepared compounds have antioxidant assets when equated with typical antioxidants such as ascorbic acid as a reference in search of their antioxidant activity by the stable free radical method [36-37]

Table 4 Scavenging activity of some synthetic compound

DPPH Scavenging activity						
Conc.	Ascorbic acid	L ₁	HL ₂	[NiL ₁ L ₂ H ₂ OCl]	[CuL ₁ L ₂ H ₂ OCl]	[AuL ₁ L ₂]Cl ₂
50	65	69.3	79.6	68.4	56.9	74.3
100	73	69.7	84.1	69.6	67.6	75
150	85.3	69.8	86	73	70	75.3
200	98.2	71.7	91.9	81.3	76.3	76.6

Figure (7) Scavenging activity of the compound using DPPH.

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