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Preparation of the ligand (E)-2-amino-5-((4,5-dimethylthiazol-2-yl)diazenyl)-3,4-dimethylbenzoic acid and some Complexes

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ORIGINAL STUDY

Preparation of the Ligand (E)-2-Amino-5-((4,5-Dimethylthiazol-2-yl)diazenyl)-3,4-Dimethylbenzoic Acid and Some Complexes

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Abstract

The ligand was prepared with a new series of chelating complexes with the following metal ions chromium (II), cobalt (II), nickel (II), Copper (II), Silver (I), Gold (III) and in different molar ratios. The results showed that the molar ratio (metal–ligand) is (1:2) for all the complexes under study, except for the silver complex with its molar ratio (1:1). The new metal complexes of the elements were diagnosed using available methods such as microanalysis and calculation of the ratio of metal ions in their complexes using the technique of flame atomic absorption and ultraviolet-visible and infrared spectra, in addition to the electrical conductivity of solutions of complexes as well as the magnetic sensitivity of complexes in the solid state. Based on the results obtained, the octahedral forms of all canine complexes with d^2sp^3 hybridization were proposed) except for the gold complex, which has the shape of a planar square with dsp^2 hybridization, and the Silver Complex in a tetrahedral form with sp^3 hybridization. The research also included the study of the Bio-effect of ligand and its metal complexes in the growth of two types of bacteria using solvent DMSO and diffusion technique on Acar medium as Gram positive bacteria) and bacteria (*Escherichia coli*) are representatives of Gram-negative bacteria (gram negative bacteria, causing many diseases The rumor. This ligand and its metal complexes have been found to have varying bioavailability as antibiotics in inhibiting bacterial growth.

Keywords: Heterogeneous azo, Metal complexes, TGA and mass spectrum

1. Introduction

Azo compounds are organic compounds that contain a functional group called an azo group ($-N=N-$) in their molecular structure. The azo group consists of two nitrogen atoms connected by a double bond. Azo compounds are characterized by their vibrant and diverse colors, which arise from the presence of conjugated double bonds in their structures' compounds have been explored for various medicinal applications [1]. Here are some examples: Drug Delivery: Azo compounds have been utilized in drug delivery systems. They can be incorporated into drug formulations as prodrugs, which are inactive or less active compounds that

undergo a chemical transformation in the body to release the active drug. The azo group can act as a linker that undergoes cleavage under specific conditions, such as changes in pH or enzymatic activity, leading to the release of the active drug at the desired site. Anti-Inflammatory Agents: Some azo compounds have demonstrated anti-inflammatory properties [2]. They can inhibit the production or release of inflammatory mediators, such as cytokines and prostaglandins, thereby reducing inflammation. These compounds have potential applications in the treatment of inflammatory diseases, such as rheumatoid arthritis and inflammatory bowel disease [3,4]. Anticancer Agents: Azo compounds have been investigated for their

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potential as anticancer agents. Some azo compounds exhibit cytotoxic activity against cancer cells by inducing cell death or inhibiting cell proliferation. In addition, azo compounds can be designed to selectively target specific cancer cells or tumor tissues, minimizing the impact on healthy cells. **Antimicrobial Agents:** Certain azo compounds have shown antimicrobial activity against bacteria, fungi, and parasites [5]. They can disrupt microbial cell membranes, inhibit enzyme activity, or interfere with essential cellular processes, leading to the suppression of microbial growth. Azo compounds have been studied for their potential in developing new antimicrobial drugs or coatings for medical devices. **Diagnostic Agents:** Azo compounds have been used as diagnostic agents in medical imaging techniques [6]. They can be conjugated with imaging probes, such as fluorescent dyes or radioactive isotopes, to selectively target specific tissues or biomarkers. This allows for the visualization and detection of diseases or specific cellular processes. It's worth noting that while azo compounds hold promise for medicinal applications, further research and development are needed to fully explore their potential, optimize their properties, and ensure their safety and efficacy [7]. Rigorous testing and evaluation are essential before any azo compound can be approved for clinical use.

2. Experimental

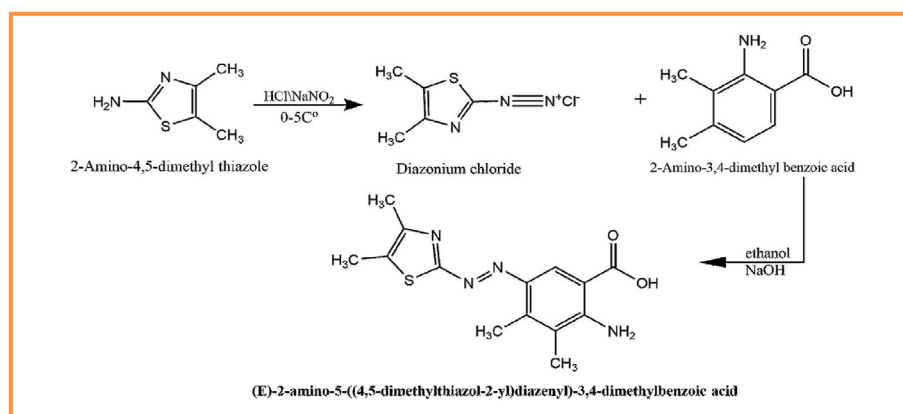
2.1. Preparation of ligand (E)-2-amino-5-((4,5-dimethylthiazol-2-yl)diazenyl)-3,4-dimethylbenzoic acid

The ligand was prepared by the addition of 0.128 g (0.001 mol) from 2-amino-4,5-dimethyl thiazole in a mixture of ml 5 (of concentrated HCl hydrochloric acid and) ml 5 (of distilled water) then we cooled the

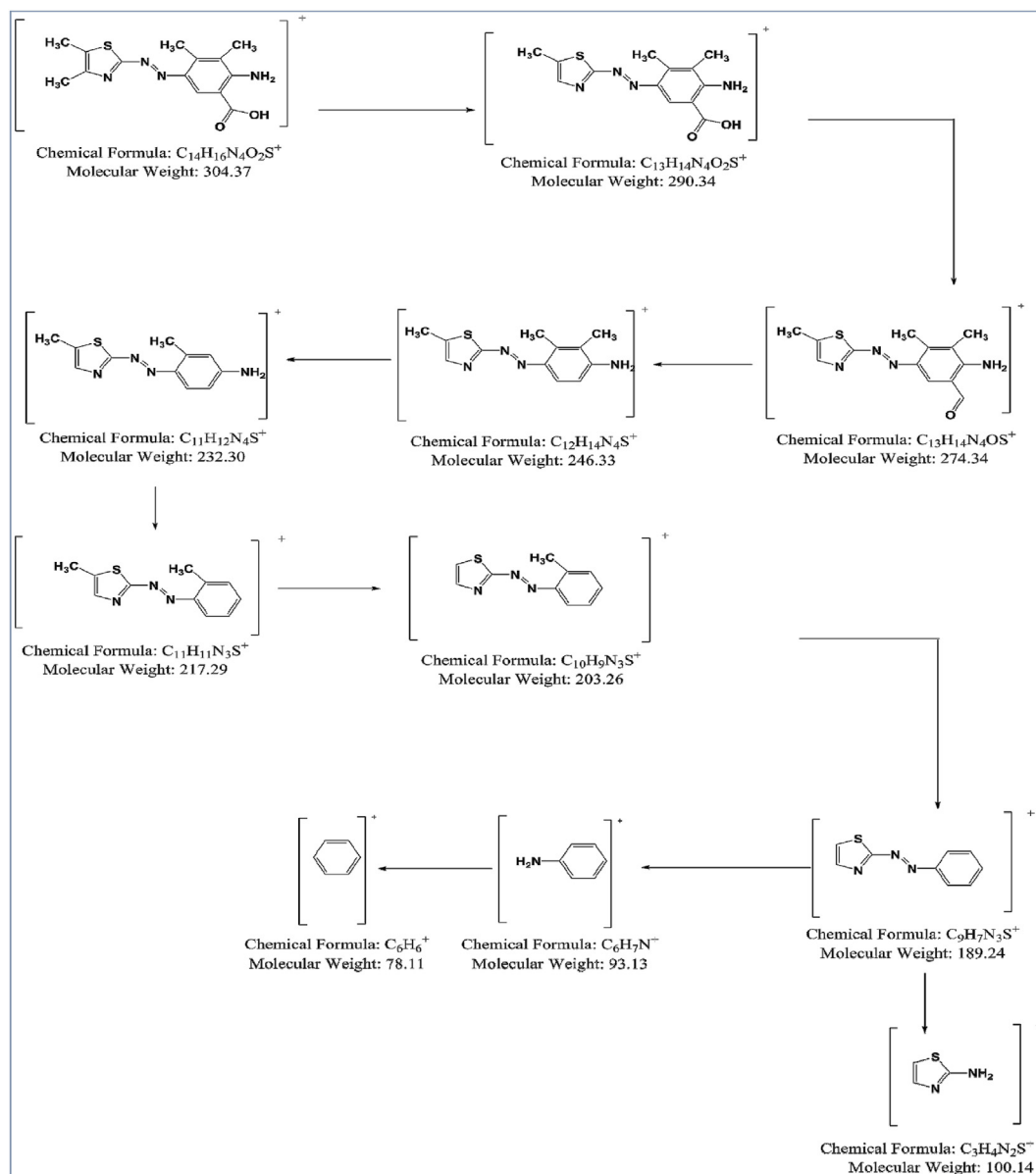
mixture to (0–2 °C) and then prepared a solution of 0.77 g (0.0097 mol (of sodium nitrite dissolved in 15 ml) (of distilled water and the two prepared solutions were mixed in the form of drop by drop with continuous stirring and cooling and for 15 min, noting that the temperature does not rise above °C5, then the solution is left to settle for half an hour to complete the process of azotation to obtain a diazonium chloride solution, this step is called the step of azotation (diazotization) the prepared diazonium chloride salt solution is added to the prepared solution This step is called pairing and preparing a solution (g 0.165 (mol 0.001) of 2-Amino-3,4-dimethyl benzoic acid dissolved in a mixture of ml 20 of ethanol alcohol at a concentration of 99.9 % in water and ml 4 of a solution of ethanol, which is dissolved in a mixture of 20 ml of ethanol with a concentration of 99.9 % in water and sodium hydroxide at a concentration of 2 % cooled to zero degrees Celsius, the prepared dark orange precipitate is filtered and left until the next day to dry and then recrystallized using absolute ethanol alcohol, We filter it and leave it to dry inside the electric oven, after which the dry matter is collected in a tight box, and the general equation below represents the reactions of both the steps of diazotization and coupling [8,9] (see Schemes 1, 2).

2.2. Synthesis of metal complexes

Metallic complexes can be prepared through various methods depending on the desired complex and the properties of the metal and ligands involved. One of the most important methods of preparation is Precipitation methods involve the formation of a metal complex by mixing two or more solutions that contain the metal ion and the ligands. In some cases, the addition of a precipitating agent or changing the



Scheme 1. Preparation of new azo ligand (L₁).



Scheme 2. Proposed block fragmentation pathways for ligand.

pH of the solution can induce the formation of a solid metal complex, which can be isolated by filtration or centrifugation. The complex barn under study is prepared by gradually adding the required weight of the ligand dissolved in mL 40 of absolute ethanol alcohol with continuous stirring to 0.001 Mol of metal chloride dissolved in mL 20 of absolute ethanol with stirring and heating, then escalating the reaction mixture for 2 h at a temperature of C° 70 and after completion of the escalation, the desired precipitate is formed, then the precipitate is filtered, washed with distilled water and recrystallized with a small amount of absolute ethanol alcohol [10,11] (see Table 1).

3. Result and discussion

3.1. 1H -NMR spectra

DMSO- d_6 was used as a solvent and TMS as a standard reference, where the spectrum showed a single beam at the chemical displacement $\delta = 2.503$ ppm, where it belongs to the presence of two CH_3 isomer groups in the thiazole ring, while two single beams appeared at the chemical displacement $\delta = 2.656$ ppm and $2.737 = \delta$, which belong to the two methyl groups in the benzene ring, As for the mono-beam at the chemical displacement = 6.407 ppm, it returns to the protons

Table 1. Some physical properties of ligand and its metal complexes.

Molecular formula	Color	m.P °C	Percentage of output %	PH
Ligand B	Dark red	413	81	5.5
Complex Cr(III)	Green	015	60	7
Complex Co(II)	Dark purple	179	82	7.5
Complex Ni(I)	Light brown	187	73	8
Complex Cu(II)	Dark green	179	78	7
Complex Zn(II)	Josie	817	66	6.5
Complex Ag(I)	Reddish orange	176	74	8
Complex Au(III)	Reddish brown	218	72	7

of the aromatic ring NH₂, and a beam appeared at the chemical displacement $\delta = 7.565$ ppm, and it returns to the protons of the aromatic ring, while the hydroxyl group gave a mono-beam of 9.756 ppm, and the spectrum showed a beam at the chemical displacement $\delta = 2.202$ ppm and returns to the solvent DMSO-d₆ [12,13], and Fig. 1 the proton spectrum of the nuclear magnetic resonance of ligand.

3.2. Mass spectra

The proposed mass fractionations of the ligand are shown below in diagram (2), where the main peak appeared at 304.1 m/z+ = due to the molecular weight of the ligand above, which is equal to (304.37) with the formula [C₁₄H₁₆N₄O₂S]⁺, while other peaks appeared at (78.7, 93.9, 100.8, 189, 203, 217, 232.4, 246.1, 274, 290.1 (m/z+ and belong to the ions C₁₃H₁₄N₄O₂S⁺), C₁₃H₁₄N₄O₂S⁺ + C₁₂H₁₄N₄S⁺, C₁₁H₁₂N₄S⁺, C₁₁H₁₁N₃S⁺, C₁₀H₉N₃S⁺,

C₉H₇N₃S⁺, C₃H₄N₂S⁺, C₆H₆⁺, C₆H₇N⁺, respectively (125,126). The table below shows the proposed mass fractionation products for ligand (see see Table 2 and Fig. 2).

3.3. Infrared spectra of azo ligand and their metal complexes

The infrared spectrum of ligand shown in Fig. 3 showed a medium-intensity stretching beam at a frequency (3463 Cm⁻¹) due to the stretching vibrations of the sphincter (N-H) of ligand and its metal complexes and a medium-intensity beam at a frequency (3317 Cm⁻¹) attributed to the sphincter (O-H) of the hydroxyl group in ligand, and this beam does not disappear for all spectra of metal complexes of ligand indicative the spectrum of the free ligand showed a weak absorption beam at (C-M 13183) (which belongs to the actin (C-H) aromatic ring [14], and this beam is formed by the oh phenolic group after its protons [15]. lose an indication of the presence of moisture, and the spectrum of the free ligand showed a weak absorption beam at (13,183 Cm⁻¹) (which belongs to the actin (C-H) aromatic ring [16], The region of the spectrum confined between (400–1700 Cm⁻¹) is very important in the infrared spectrum when interpreting the prepared metal complexes because it includes the absorption beams of the effective aggregates in both the spectra of the ligand and its metal complexes, including the aggregates N=N, C=N, C=C and other effective aggregates, in addition to the vibrations of the N-M and O-M the ligand spectrum showed the heterogeneous

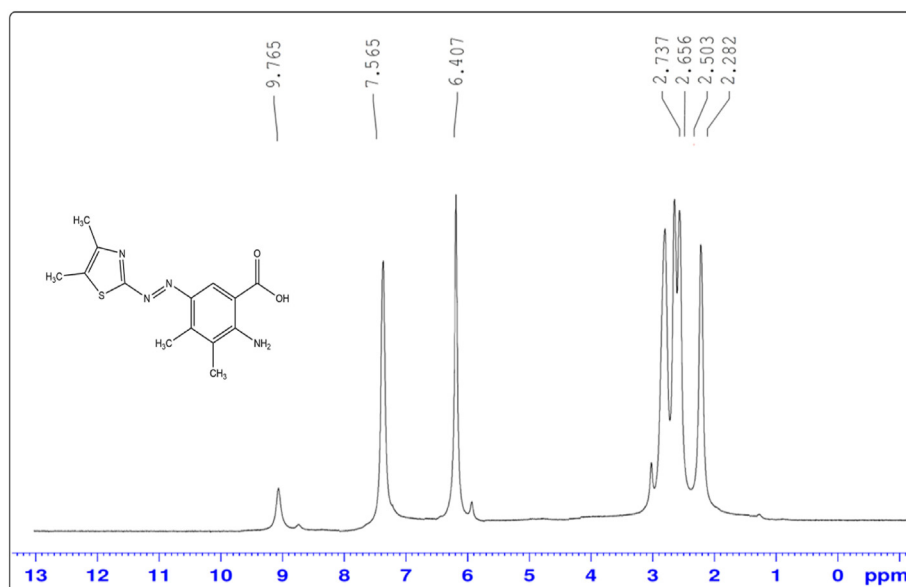


Fig. 1. Of the 1H-NMR proton magnetic resonance spectrum of ligand in DMSO solvent.

Table 2. Mass fractionation products of ligand.

Fragment	m/z ⁺ Exact mass	Relative Abundance (%)
C ₁₄ H ₁₆ N ₄ O ₂ S ⁺	304.1	2.5
C ₁₃ H ₁₄ N ₄ O ₂ S ⁺	290.1	3.5
C ₁₃ H ₁₄ N ₄ OS ⁺	274.0	4.5
C ₁₂ H ₁₄ N ₄ S ⁺	246.1	11
C ₁₁ H ₁₂ N ₄ S ⁺	232.4	33
C ₁₁ H ₁₁ N ₃ S ⁺	217.0	88
C ₁₀ H ₉ N ₃ S ⁺	203.0	47
C ₉ H ₇ N ₃ S ⁺	189.0	53
C ₃ H ₄ N ₂ S ⁺	100.8	31
C ₆ H ₇ N ⁺	93.9	100
C ₆ H ₆ ⁺	78.7	23

AZO thiazolyl dye ring ligand a strong absorption beam at the frequency (1688 cm⁻¹), which belongs to the C=N actin in the thiazole ring [17,] and this beam was obtained by A slight change in the location, shape, intensity and displacement at the site [18]. and this change is caused by the synergy between the metal ions and the nitrogen atom present in the composition of the thiazole ring [19], and the spectrum of free ligand showed a sharp intensity absorption beam at Frequency (14,581 Cm⁻¹), which belongs to the C=C Group in the aromatic ring and the spectrum of free ligand showed a beam at Frequency (1600 Cm⁻¹), which belongs to the azo Group N=N spectra of the metal complexes under study in terms of. The Shape of the beams, their intensity and their locations, which indicates the involvement of the azo group in the process of coordination with the ions of metal elements by the nitrogen atom of the azo

group The spectrum of free ligand showed a bundle at (1267 Cm⁻¹), which returns to the C-S sphincter frequency of the thiazole ring, which does not change in intensity and location, this is evidence of its lack of involvement in the symmetry [20], as well as the spectrum of free ligand showed a bundle at the frequency (1384 Cm⁻¹), which returns to the N-C sphincter, while the spectrum of free ligand showed a bundle at the frequency (1330 Cm⁻¹) the prepared metal complexes showed absorption spectra with frequencies of new beams that did not appear in the free ligand spectrum, as they showed frequencies at (420-400 cm⁻¹), as these beams return to the frequencies of the M-N. Therefore, the infrared spectrum indicates that the ligand exhibits a bicyclic alkane during the coordination process by means of the nitrogen atom of the heterocyclic thiazole ring and the nitrogen atom of the azo bridging group close to the phenyl ring and the oxygen atom of the phenolic hydroxyl group to give two pentacyclic metal rings that lead to an increase in the stability of the covalent metal complexes formed. The many changes mentioned in this paragraph are illustrated by Table 3 the consistency of the ligand with metal ions is under study for the formation of metal complexes.

3.4. Magnetic susceptibility and electronic spectra measurements

The UV–visible spectrum of the Chromium complex showed only one absorption peak at the wavelength of 476 Nm (21,008 Cm⁻¹), which is due

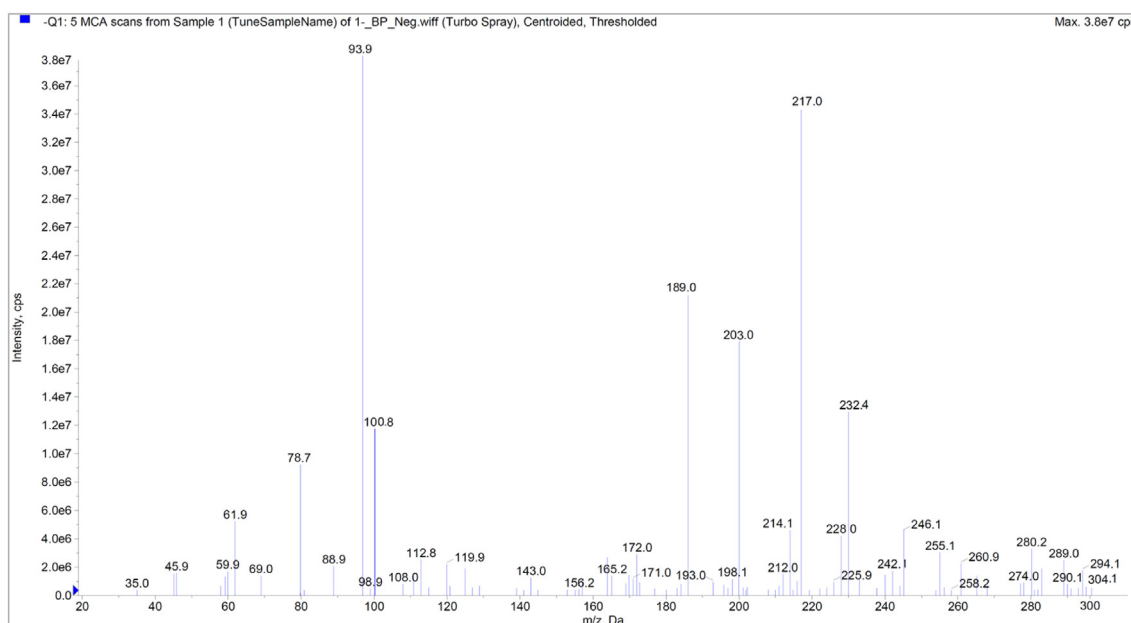


Fig. 2. Represents the mass spectrum of the ligand.

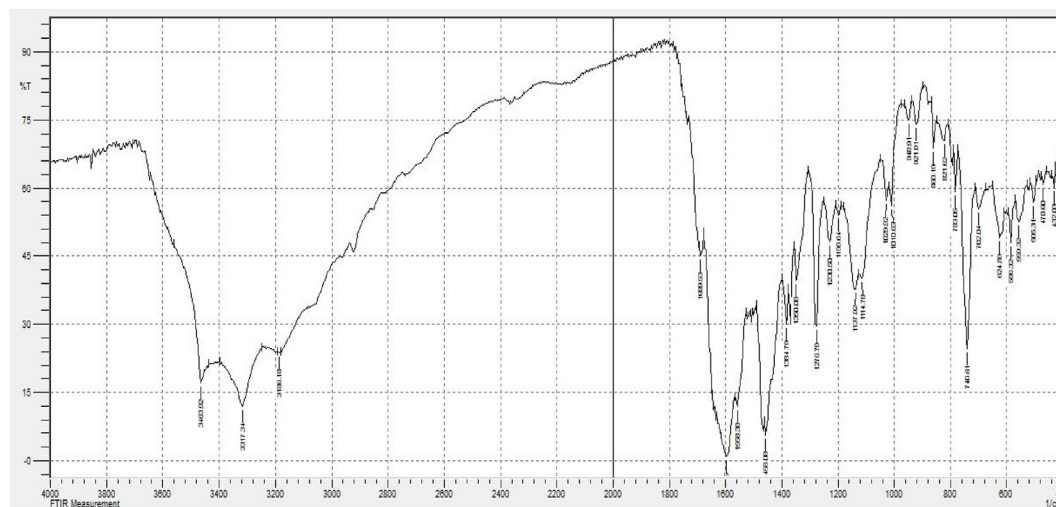


Fig. 3. Infrared spectrum of ligand.

Table 3. Represents the values of the frequencies of the infrared spectra in units (Cm^{-1}) of Ligand and its metal complexes.

Compound	$\nu(\text{N-H})$	$\nu(\text{O-H})$	$\nu(\text{C-H})$	$\nu(\text{C=N})$ Thiazole	$\nu(\text{N=N})$ Azo	$\nu(\text{C=C})$	$\nu(\text{N-C})$	$\nu(\text{C-S})$ Thiazole	$\nu(\text{O-C})$	$\nu(\text{M-N})$
Ligand (B)	3463	3317	3183	1688	1600	1458	1384	1267	1230	—
Complex (Cr)	3387	3183	3053	1630	1595	1483	1311	1220	1200	428
Complex (Co)	—	3370	3190	1658	1520	1420	1320	1235	1233	430
Complex (Ni)	3455	3375	3200	1650	1583	1435	1325	1240	1215	445
Complex (Cu)	3463	3317	3186	1689	1600	1458	1350	1230	1276	432
Complex (Zn)	3301	3179	2912	1655	1598	1465	1315	1245	1277	447
Complex (Ag)	—	3055	2900	1685	1595	1439	1315	1238	1276	432
Complex (Au)	—	3317	3163	1677	1600	1481	1315	1230	1168	428

to the permissible electronic transmission $A_{2g} \rightarrow 4T_{1g}$. The UV–visible spectrum of the cobalt complex showed two absorption peaks, the first peak at the wavelength of 984 Nm ($11,872 \text{ Cm}^{-1}$) belongs to the permissible electronic transitions $3A_{2g} \rightarrow 3T_{2g}$ and the second peak at the wavelength of 534 Nm ($18,727 \text{ cm}^{-1}$), which belongs to the permissible electronic transitions $A_{2g} \rightarrow 3T_{1g}$ and gives the geometric figure Octahedron [21,22]. The third peak at a frequency of 243 Nm ($41,667 \text{ cm}^{-1}$), which belongs to the electronic transition $3A_g \rightarrow T_{1g} (p)$. The UV–visible spectrum of the nickel (II) complex showed three absorption peaks, the first peak being medium and wide at 980 Nm ($10,204 \text{ cm}^{-1}$), which is due to the transition $3A_{2g} \rightarrow 3T_{2g}$, the second peak is wide at the frequency 558 Nm ($17,921 \text{ cm}^{-1}$), which is due to the transition $3A_{2g} \rightarrow 3T_{1g}$, while the third peak appeared at the frequency 438 Nm ($22,831 \text{ cm}^{-1}$) returns to the transition $3A_{2g} \rightarrow 3T_{1g}$. the nickel complex has the stereo spectral octahedral shape and two more peaks of the complex appear at the frequency of 258 Nm ($38,760 \text{ cm}^{-1}$) and at the frequency of 230 Nm ($43,478 \text{ cm}^{-1}$) return to the transitions within the ligand. The Copper (II)

octahedral complex showed a wide broad band beam representing the electronic transition $2 E_g \rightarrow 2T_{2g}$ at a frequency of 550 Nm ($18,182 \text{ cm}^{-1}$) and this beam in the octahedral domain is divided into three beams due to the deformation of these complexes due to the Jean-teller effect. The electronic spectra of the outliers II complexes were studied in absolute ethanol with a concentration of M 0.001 and it turned out that this complex does not give the D-D transition, because it has in its outer shell on d^{10} , but it gave an absorption peak at a frequency of 542 Nm ($18,450 \text{ cm}^{-1}$), which is due to the transition (M-L, CT) [23,24]. In low-perm tetrahedral monovalent silver complexes, the transition beam d-d does not appear because all d orbitals are filled with electrons, and therefore the charge transfer transition beam charge transfer transition appears at a frequency of 466 Nm ($21,459 \text{ cm}^{-1}$) in the visible region of the spectrum, returning to the transition M-L, CT)). When measuring the UV–visible spectrum of the triple gold complex, it gave one absorption beam at a frequency of 542 Nm ($18,450 \text{ cm}^{-1}$), which is due to the transition $1A_{1g} \rightarrow 1T_{1g}$, which confirms that this complex is a planar Square, where it takes the same

Table 4. Represents the physical properties and electronic transitions of Ligand and its metal complexes.

Complexes	$\lambda_{\max}$ (nm)	Absorption bands (cm^{-1})	Transition	μ_{eff} (B.M)	Geometryt	Hybridization
Ligand	234	42,735	$\pi \rightarrow \pi^*$	—	—	—
	338	29,586	$n \rightarrow \sigma^*$	—	—	—
	412	24,272	$n \rightarrow \pi^*$	—	—	—
Complex-Cr(III)	558	17,921	${}^4A_{2g} \rightarrow {}^4T_{1g}(F)$	2.93	Octahedral (regular)	d^2sp^3
Complex-Co(III)	580	17,241	${}^3A_{2g} \rightarrow {}^3T_{2g}(F) (\nu_1)$	Dia	Octahedral (regular)	d^2sp^3
	402	24,876	${}^3A_{2g} \rightarrow {}^3T_{1g}(F) (\nu_2)$			
Complex-Ni(II)	654	15,291	${}^3A_{2g} \rightarrow {}^3T_{2g}(F)$	2.79	Octahedral (regular)	$sp^3 d^2$
	436	22,936	${}^3A_{2g} \rightarrow {}^3T_{1g}(F)$			
Complex-Cu(II)	536	18,657	${}^2E_g \rightarrow {}^2T_{2g}$	1.59	Octahedral (distorted) (Z-in, Z-out)	$sp^3 d^2$
Complex-Zn(II)	488	20,491	$M \rightarrow L$, CT	Dia	Octahedral (regular)	$sp^3 d^2$
Complex-Ag(I)	510	19,608	$M \rightarrow L$, CT	Dia	Tetrahedral (distorted)	sp^3
Complex-Au(III)	460	21,739	${}^1A_{1g} \rightarrow {}^1B_{1g}(\nu_1)$	Dia	Square planer (distorted)	dsp^2

geometric shape in calculating the electronic transitions of all elements that have an outer shell and a planar square d8, and the figure below represents all electronic transitions (see Table 4 and Figs. 4–8).

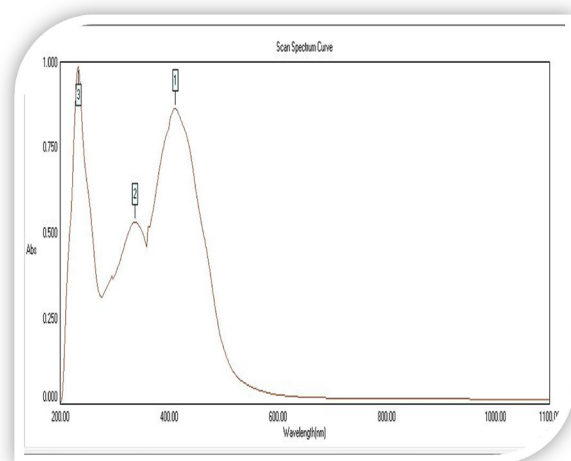


Fig. 4. UV–visible spectrum of the free ligand.

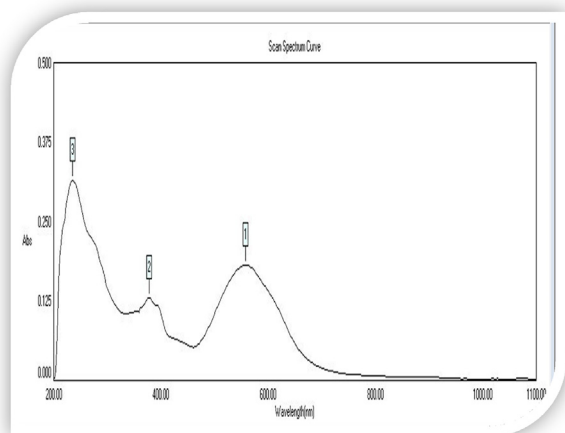


Fig. 5. UV–visible spectrum of the Chromium complex.

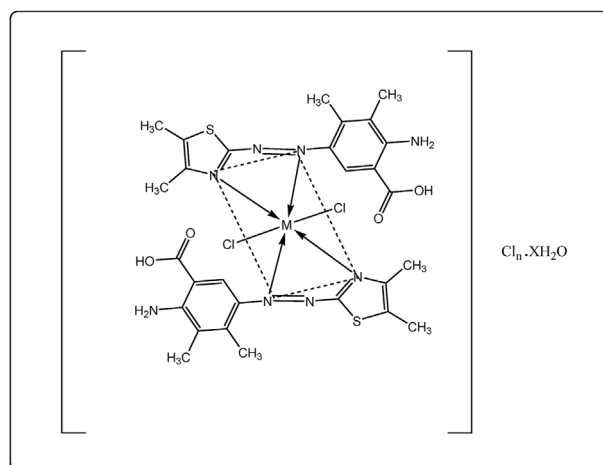


Fig. 6. Represents the symmetry of metals with a ligand.

Based on the results of the obtained diagnostic measurements, we can formulate the proposed formulas for the complexes under study:

$M = \text{Cr(III), Co(III), Ni(II), Cu(II), Zn (II)}$

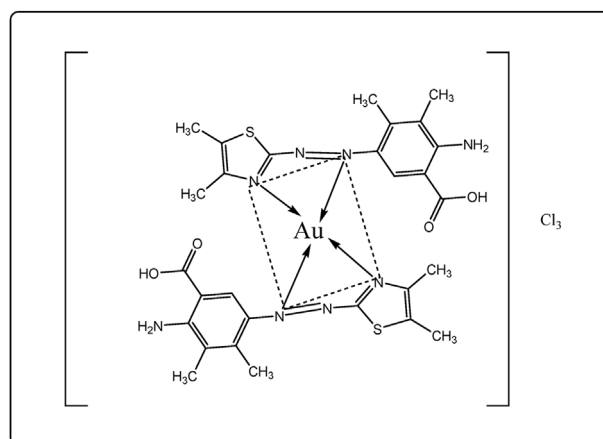


Fig. 7. Represents the consistency of gold with the ligand.

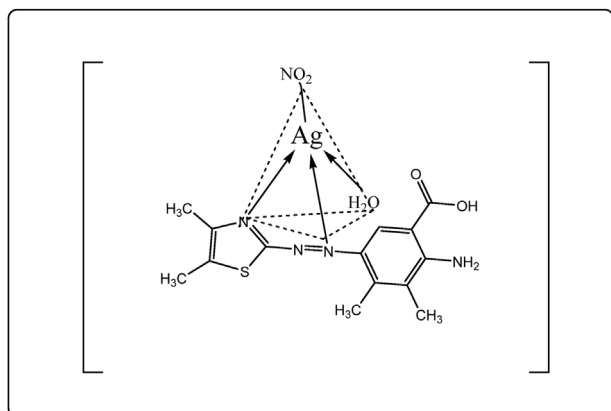


Fig. 8. Represents the symmetry of silver with a ligand.

$n = 1$ Cr (III), Co (III)

$n = 0$ Ni (II), Cu(II), Zn (II)

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