



Studying gold compounds analytically and spectroscopically using novel diamine donor ligands

Raghad k. khamass¹, Sattar R. Majeed²

¹Department of Chemistry, Education College for Women, University of Anbar, Ramadi, Anbar, Iraq.

²Department of Chemistry, College of Science, University of Anbar, Ramadi, Anbar, Iraq.
ragadkh030@gmail.com

Abstract

Whenever N-(Naphthylethylenediaminedihydrochloride) and 4-nitroaniline were combined, they yielded N-(2-Amino-5-nitrophenyl) diazenyl ethyl)-naphthalen-1-amine, an azo-ligand. We started with the primary compound: Azo, and then we added salts of sodium pyrophosphate, organic acids like malonic acid, and other secondary ligands like oxalic acid. Researchers conducted thorough analyses of the materials' spectroscopic (UV-Vis and FTIR), (molar conductivity), and magnetic properties (Ultraviolet-Vis and FTIR). The infrared spectrum and molar conductivity data show that all of the complexes are electrolytic, and the bidentate ligands are all coordinated to gold (III). Based on data from magnetic susceptibility, mass spectrometry, elemental analysis, ¹H-NMR, and UV-Vis spectra, the compounds were shown to have square planar geometries.

Keywords: Gold complexes , Synthesis , Azo dye

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1. Introduction

depending on the sum of the charges of the ligands and the central metal atom. The Lewis base, which is the donor atom or gainer of the electron pair, and the metal ion, which is the acceptor or gainer of the electron pair because it has vacant orbitals that might accept the double electrons, are the two types of ligands. It is referred to as a (monodentate ligand) if it only has one donor atom, and a (polydentate ligand) if it has more than one. Polydentate-containing compounds that are more likely than other polydentate to be attached to the metal are referred to as chelating complexes. The stability of the complexes depends on the chelating ring created as a result of coordination, where the pentagonal and hexagonal rings are the most stable. Bridge ligands are ligands that may

Since the 1789 discovery of the substance (CoCl₃.6NH₃), coordination chemistry has attracted the attention of researchers. Due to its rapid development, perhaps in the second half of the 20th century, it gained a large area in chemistry to prepare complex coordination compounds practically and to understand the structures of these complexes. These complexes have become more and more significant in a variety of fields, such as agriculture, industry, and medicine [1-2]. In the vast majority of complexes, an ion or molecule is surrounded by another ion or molecule. Covalent coordination connections or coordination bonds known as ligands (or organic reagents) can be used to connect to them. The complexes can be positive, negative, or neutral



Shimadzu Atomic Absorption / Flame Emission Spectrophotometer(AA680).

2.3 Methodology

2.3.1 Synthesis of azo ligands

(C₁₈H₁₇N₅O₂)

N-(2-(2-Amino-5-nitrophenyl)diazenyl)ethyl)naphthalen-1-amine

N-(1-

Naphthyl)ethylenediaminedihydrochloride (0.1300g, 0.0005mol) was dissolved in ethanol (10 ml) with hydrochloric acid (2ml) to make a clear solution .A drop-by-drop application of an aqueous sodium-nitrite solution (one gram in ten ml of water) was used to keep the temperature below 5 degrees Celsius. After reducing the acidity to (5–6) with an ice bath and potassium dihydrogenphosphate buffer, the liquid was agitated for 30 minutes. 0.069g (0.0005mol) of 4-nitro aniline was prepared by dissolving it in 10ml of ethanol and cooling it in an ice bath to between 0 and 5 °C. It was dried and filtered to produce a reddish brown strong precipitate, yielding 83 percent, with a melting point of (137-139) degrees Celsius.M.wt:(335g.mol⁻¹). Found: C; 64.48 : H;5.07 : N;20.9 : O;9.55 . Cal: C;63,88 : H;4.86: N;22.61 : O;9.19. IR(KBr): 3524 cm⁻¹; 3427cm⁻¹; 3307 cm⁻¹;1654 cm⁻¹;1469 cm⁻¹;1412 cm⁻¹;3070 cm⁻¹and 2924 cm⁻¹. UV-Vis(DMSO): 289 nm and 401 nm.

2.4 Synthesis of gold(III) complexes

2.4.1 Synthesis of [Au(C₁₈H₁₇N₅O₂)Cl₂]Cl complex

The ligand (C₁₈H₁₇N₅O₂) (0.17g) was dissolved in 30 ml of ethanol and slowly added to a solution of (AuCl₃) salt (0.152 g, 0.0005 mol) in 10 ml of distilled water and 20 ml of buffer solution, and the pH was set to (2-3) and the mixture increased for eight hours while stirring (60-70 °C). Afterwards, the sediment was rinsed with diethyl ether and dried, then filtered with

interact with more than one metal atom simultaneously [3-4]. The brilliant hues of inorganic complexes, which often emerge in the visible spectrum because the ligand contains an effective color-carrying group (chromophoric group), and examples of which include groups (N=N, C=N-), have made them popular in analytical science. A group (chromic Auxo) (OH,-Br,-Cl,-SH,-NH₂) can also be included in the ligand. It helps make ligands and their complexes more soluble in solvents. When used in the qualitative and quantitative determination of low ion concentrations, these complexes and their ligands exhibit high color sensitivity[5-6].

2. Experimental

2.1. Materials

The Chemicals are pure and used as delivered; AuCl₃(B.D.H), oxalic acid, malonic acid and tetra sodium pyrophosphate (Fluka), DMSO and ethanol (B.D.H).

2.2. Instruments

Melting points are determined using Stuart SMP20melting points system. Infrared spectra were carried out using a TetscanShimadzu FT.IR—8300s. The electronic spectra have been recorded in DMSO using a double-beamShimadzu UV-Visible Spectrophotometer.Proton nuclear magnetic spectrum¹H-NMR Bruker, Ultrasheid (400.81)MHz. Electrical conductivity measurements were recorded at 25 °C using aDigital conductivity meter. Elemental analysis of elements was performed via Euro vector. EA 3000, single V.3.Osingle. Magnetic susceptibility measurements were performed using a MEGNETICSUSCEPTIBILITY BALANCE-faraday scientific. Mass spectra were recorded withMass spectra GC- Shimadzu Qp-2010 Plus.atomic absorption

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In 30ml of ethanol, a solution of the first ligand ($C_{18}H_{17}N_5O_2$) (0.17g) was added slowly to solution of ($AuCl_3$) salt (0.152 g, 0.0005 mol) and 20ml buffer solution with stirring to make up the final concentration. Drop by drop, the malonic acid (Ma) solution (0.0528gm, 0.0005mol) in distilled water was added to the mixture while being stirred. It was heated for eight hours with constant stirring to (60-70 °C), and the solution was then cooled to (25 °C) a pH (2-3). Afterwards, the sediment was rinsed with diethyl ether and dried, then filtered with water and ethanol in a 1:1 ratio. 633.97 g.mol⁻¹ mass-weight. 60 percent of the time. m.p.: 120-124°C. Found: C; 36.97: H;2.01 : N; 9.88 : O;15.88 : Cl;4.71 : M;28.77.Cal: C;37.64 : H;2.84 : N;10.46 : O;14.34 : Cl;5.30 : M;29.42 for the time. The IR(KBr) is 3442 cm⁻¹; 3427 cm⁻¹; 1653 cm⁻¹; 3300 cm⁻¹; 1450 cm⁻¹; 1400 cm⁻¹; 3072 cm⁻¹; 2908 cm⁻¹; 1516 cm⁻¹; 1215 cm⁻¹; 495 cm⁻¹ and 570 cm⁻¹. 235, 313, 529, and 644 nm are the UV-Vis (DMSO) wavelengths. Its molar conductance is 29.0 S.cm².mol⁻¹ DMSO.

2.4.4 Synthesis of $[Au(C_{18}H_{17}N_5O_2)(Pyph)]Cl$ complex

In 30ml of ethanol, a solution of the first ligand ($C_{18}H_{17}N_5O_2$) (0.17g) was added slowly to solution of ($AuCl_3$) salt (0.152 g, 0.0005 mol) and 20ml buffer solution with stirring to make up the final concentration. Drop by drop, the pyph solution (0.223gm, 0.0005mol) in distilled water was added to the mixture while being stirred. It was heated for eight hours with constant stirring to (60-70 °C), and the solution was then cooled to (25 °C) a pH (2-3). Afterwards, the sediment was rinsed with diethyl ether and dried, then filtered with water and ethanol in a 1:1 ratio. A mol⁻¹ of M.wt = 751.97.

water and ethanol in a 1:1 ratio. Six hundred and two and ninety-seven percent g.mol⁻¹. M.p: 100-105 oC; yield: 76%; percentage of yield: For C; 33.83: H; 2.66: N; 10.96: O; 5.01: Cl; 16.68: M; 30.85, we found: C; 35.00: H; 3.01: N; 11.09: O; 4.96: Cl; 17.71: M; 31.13, with IR(KBr) 3423 cm⁻¹, 3372 cm⁻¹, 1651 cm⁻¹, 3301 cm⁻¹, 1452 cm⁻¹, 1390 cm⁻¹, 3070 cm⁻¹, 2926 cm⁻¹, and 433 cm⁻¹. 317 nm; 425 nm; 600 nm and 709 nm UV-Vis (DMSO). Molar conductance of DMSO is 36.0 S.cm².mol⁻¹.

2.4.2 Synthesis of $[Au(C_{18}H_{17}N_5O_2)(Ox)]Cl$ complex

In 30ml of ethanol, a solution of the first ligand ($C_{18}H_{17}N_5O_2$) (0.17g) was added slowly to solution of ($AuCl_3$) salt (0.152 g, 0.0005 mol) and 20ml buffer solution with stirring to make up the final concentration. Drop by drop, the oxalic acid (OX) solution (0.0639gm, 0.0005mol) in distilled water was added to the mixture while being stirred. It was heated for eight hours with constant stirring to (60-70 °C), and the solution was then cooled to (25 °C) a pH (2-3). Afterwards, the sediment was rinsed with diethyl ether and dried, then filtered with water and ethanol in a 1:1 ratio. At 619.97 grams per mol⁻¹ M.wt, A whopping 87% of the crop was harvested. M.p. temperature ranges from 102-104 degrees Celsius. C,35.65: H, 3.09: N, 11.97: O, 15.57: Cl, 4.88: M, 31.33 were found and Cal: C;36.61 : H;2.59 : N;10.68 : O;14.65 : Cl;5.42 : M;30.05. The following measurements IR(KBr): 3501 ; 3402 ; 1651 ; 3306 ; 1443 ; 1393 ; 3070 ; 2924; 1516;1215 ;459; and 592cm⁻¹. Each of the DMSO UV-Vis (333, 398, 489, 713 and 780nm) wavelengths has its own unique properties. S.cm².mol⁻¹ of DMSO molar conductance: 26.0 .

2.4.3 Synthesis of $[Au(C_{18}H_{17}N_5O_2)(Ma)]Cl$ complex



$^1A_{1g} \rightarrow ^1B_{1g}$ and $^1A_{1g} \rightarrow ^1E_g$ transition for the complexes (1), (2), (3) and (4); respectively [8].

3.2 IR spectral data

Table 2 summarizes the Four complexes' FTIR spectral data. The IR spectra of the four complexes (Figures 6-10) were compared with the free ligand to configure the groups that may be involved in coordination. The amine group's bending frequency ($\nu_{\delta}(\text{NH}_2)$) is represented by the band at 1654 cm^{-1} in the spectrum of the free ligand [9]. The stretching frequencies of the amine group ($\nu_{\text{asym}}(\text{NH}_2)$), ($\nu_{\text{sym}}(\text{NH}_2)$) and ($\nu_{\text{asym}}(\text{NH})$) are represented by the bands at 3524 cm^{-1} , 3427 cm^{-1} , and 3307 cm^{-1} , respectively. The bands at 1469 cm^{-1} and 1412 cm^{-1} stretching vibration, designated for the stretching frequency of the azo group ($\nu(\text{N}=\text{N})$), were discovered modified to (1452; 1390), (1443; 1393), (1450; 1400), and (1454; 1396) cm^{-1} in complexes (1), (2), (3), and (4), respectively. The liberation of hydrogen bonding with the NH_2 may compensate for the anticipated shift in the frequency of the $\text{N}=\text{N}$ group upon coordination [10]. The presence of (PO_3) in complex spectra suggests that the $[\text{P}_2\text{O}_7]^{4-}$ ring adopts a bent conformation in conjunction with gold. Complex (4)'s IR spectrum revealed new bands at 1213 cm^{-1} and 950 cm^{-1} attributed to asymmetric and symmetric stretching vibrations of the terminal $\nu(\text{PO}_3)$ [11]. Au(III) complex spectra revealed two novel bands assigned to (M-N) and (M-O), respectively, at 433 (1), 459 (2), 495 (3), and 461 (4) cm^{-1} and 592 (2), 570 (3), and 503 (4) cm^{-1} [12]. Measurements of the magnetic susceptibility measurements of the complexes confirmed the diamagnetic nature and the existence of divalent Gold (d^8) in a square planar geometry.

Percentage yield: 70%. melting point: 115-120 °C. Found: C; 28.97; H; 3.08; N; 9.77; O; 17.11; Cl; 3.76; M; 26.61. Cal: C; 27.43; H; 2.16; N; 8.89; O; 18.29; Cl; 4.51; M; 25.01. IR(KBr): 3427 cm^{-1} , 3372 cm^{-1} , 1653 cm^{-1} , 3307 cm^{-1} ; 1454 cm^{-1} ; 1396 cm^{-1} ; 3072 cm^{-1} ; 2924 cm^{-1} ; 950 cm^{-1} ; 1213 cm^{-1} ; 461 , and 503 cm^{-1} of IR(KBr). Each of the UV-Vis(DMSO) wavelengths has an intensity of between 317; 413; 479 and 765 nanometers. Conductance molar of $21.0 \text{ S.cm}^2.\text{mol}^{-1}$: (DMSO).

3. Results and Discussion

The complexes are totally soluble in DMSO but only slightly soluble in ethanol, acetone, and diethyl ether. When the complexes' molar conductance in DMSO was tested, it was found to range between 21 and $36 \text{ S.cm}^2.\text{mol}^{-1}$, indicating that they had an electrolytic characteristic. The structures of the gold complexes were elucidated via elemental analyses, magnetic, spectroscopic data as the following:

3.1 UV-Vis spectral

Table 1 summarizes Au(III) spectral data of the complexes (Figures 1 - 5). The free ligand spectrum showed a strong absorption band at 289 nm (34602 cm^{-1} , $15100 \text{ L.mol}^{-1}.\text{cm}^{-1}$) which reflected $\pi \rightarrow \pi^*$ transition and a peak at 401 nm (24937 cm^{-1} , $9960 \text{ L.mol}^{-1}.\text{cm}^{-1}$) attributed to $n \rightarrow \pi^*$ transition (charge transfer within the azo group). In addition to the above two bands of the free ligands the spectra of complexes showed additional peaks characteristic to $d-d$ transition in a square planar geometry [7]. The bands at 600 nm (16666 cm^{-1} ; $8760 \text{ L.mol}^{-1}.\text{cm}^{-1}$); 709 nm (14104 cm^{-1} ; $4770 \text{ L.mol}^{-1}.\text{cm}^{-1}$); 713 nm (14025 cm^{-1} ; $8.7 \text{ L.mol}^{-1}.\text{cm}^{-1}$); 780 nm (12820 cm^{-1} ; $10 \text{ L.mol}^{-1}.\text{cm}^{-1}$); 529 nm (18903 cm^{-1} ; $1420 \text{ L.mol}^{-1}.\text{cm}^{-1}$); 644 nm (15527 cm^{-1} ; $1060 \text{ L.mol}^{-1}.\text{cm}^{-1}$) and 479 nm (20876 cm^{-1} ; $3560 \text{ L.mol}^{-1}.\text{cm}^{-1}$); 765 nm (13071 cm^{-1} ; $870 \text{ L.mol}^{-1}.\text{cm}^{-1}$) refer to



show that 1.0 moles of azo ligand and 1.0 moles of gold are coordinated (III).

Mole-ratio method

A series of solutions containing fixed amounts of a gold (III) solution and variable volumes of the same concentration of the ligand solution were monitored at an optimal acidic function (pH = 4). The point of intersection of the straight lines represents the molar ratio of the reactants (ligand: metal). Colour intensity increases until a point where stability is seen, which indicates the development of complexes.. **Figure 16** depicts the absorbance readings associated with the molar ratio of the Au (III) ion solution at these crossroads. As predicted by Job, the mole-ratio approach confirmed the creation of 1:1 complexes (M:L). And for each mole of azo ligand, there was a 1.0 mole of Au(III) coordination .

Conclusions

The azo dye ligand was properly synthesized and spectroscopically reported. Four Au (III) complexes were made using the bidentate azo ligand as a primary. The four complexes have square planar geometries, as determined by magnetic and UV-Vis studies. The Au (III) ions were identified using spectroscopy and the azo ligand.

3.3 Au(III) complex mass spectra

The mass spectrum of the complex $[\text{Au}(\text{azo})\text{Cl}_2]$ (**Figure 11**) (M. wt. = 602.97 g/mol) showed a parent peak at $m/z = 603$ (M^+). The mass spectrum of the complex $[\text{Au}(\text{azo})(\text{Ox})]$ (**Figure 12**) (M. wt. = 619.97 g/mol) showed a parent peak at $m/z = 621$ (M^+). The mass spectrum of the complex $[\text{Au}(\text{azo})(\text{Ma})]$ (**Figure 13**) (M. wt. = 633.97 g/mol) showed a parent peak at $m/z = 634$ (M^+). The mass spectrum of the complex $[\text{Au}(\text{azo})(\text{pyph})]$ (**Figure 14**) (M. wt. = 751.97 g/mol) gave a parent peak at $m/z = 753$ (M^+). These values confirm the proposed formulae molecular weights. Additionally, using analytical techniques such as Job's continuous and mole-ratio methods, the complexes' stoichiometries were verified as follows [13-14-15]:

3.4 Job's continuous and the mole-ratio methods

An acidic pH of 4 was used to prepare a series of solutions that were mixed with different volumes of the ligand, with the final volume remaining constant, in order to create the graphs between the absorbance and the mole fraction. An example of a Job's approach curve with Au(III) ion solution and ligand at (max = 600) is shown in **Figure 15**. The results



The azo ligand and the Gold complexes synthesis scheme 1

Analyses of complicated solutions by UV-Vis spectroscopy, as shown in Table 1

No.	Comp.	WL (nm)	Transition
1	Ligand N-(2-(2-Amino-5-nitrophenyl)diazenyl)ethyl)naphthalen-1-amine. [C ₁₈ H ₁₇ N ₅ O ₂]	289 401	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$, C.T
2	[Au(C ₁₈ H ₁₇ N ₅ O ₂)Cl ₂]Cl	269 317 425 600 709	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ C.T $^1A_{1g} \rightarrow ^1B_{1g}$ $^1A_{1g} \rightarrow ^1E_g$
3	[Au(C ₁₈ H ₁₇ N ₅ O ₂) (Ox)]Cl	333 398 489 713 780	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ C.T $^1A_{1g} \rightarrow ^1B_{1g}$ $^1A_{1g} \rightarrow ^1E_g$
4	[Au(C ₁₈ H ₁₇ N ₅ O ₂)(Ma)]Cl	235 313 529 644	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ $^1A_{1g} \rightarrow ^1B_{1g}$ $^1A_{1g} \rightarrow ^1E_g$
5	[Au(C ₁₈ H ₁₇ N ₅ O ₂)(Pyph)]Cl	317 413 479 765	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$, C.T $^1A_{1g} \rightarrow ^1B_{1g}$ $^1A_{1g} \rightarrow ^1E_g$



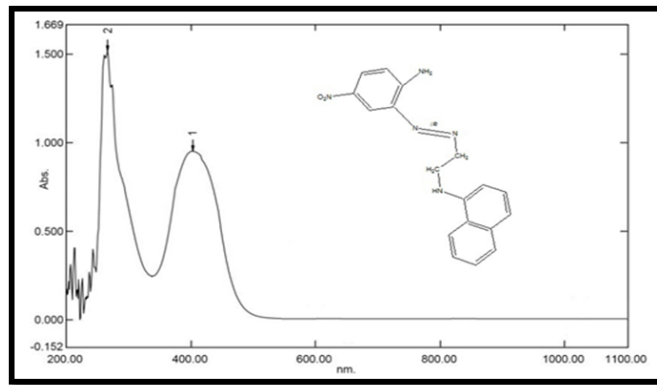


Figure 1: Primary ligand electronic spectrum ($C_{18}H_{17}N_5O_2$)

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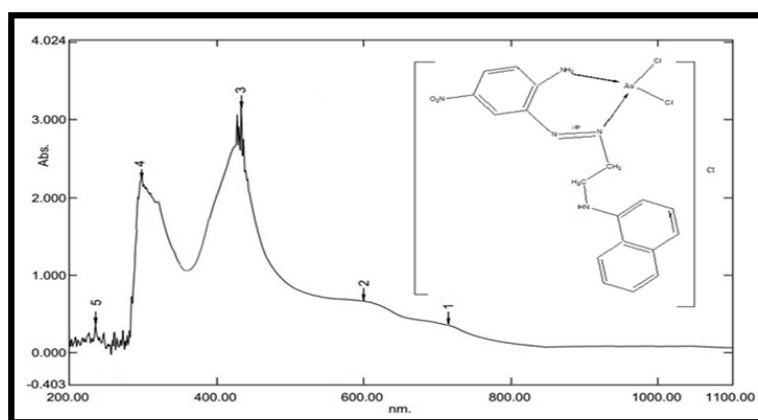


Figure 2 shows the $[Au(C_{18}H_{17}N_5O_2)Cl_2]Cl$ electronic spectrum.

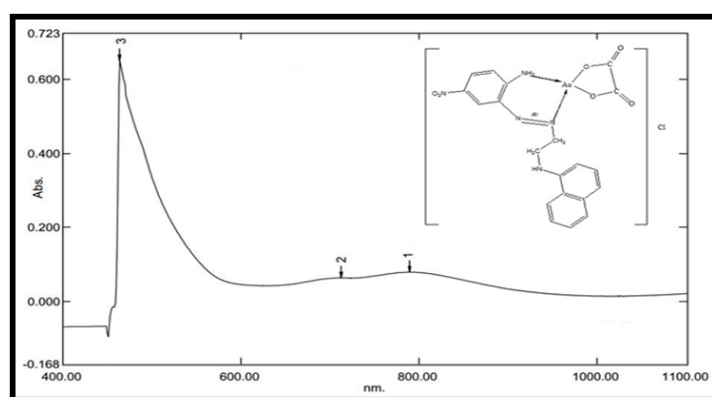


Figure 3: $[Au(C_{18}H_{17}N_5O_2)(Ox)] Cl$ electronic spectrum

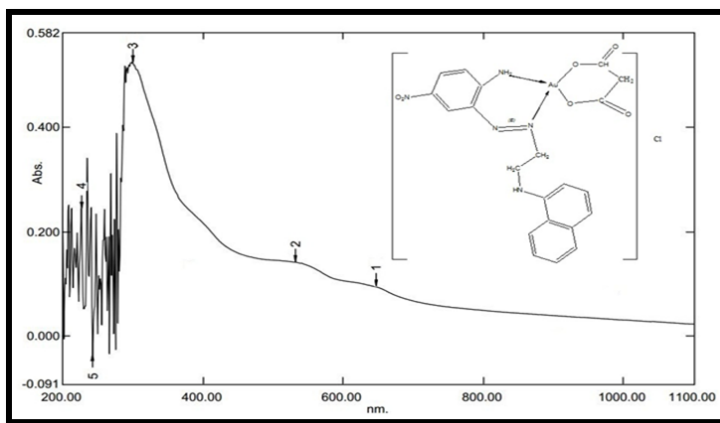


Figure 4: [Au(C₁₈H₁₇N₅O₂)(Ma)] Cl electronic spectrum

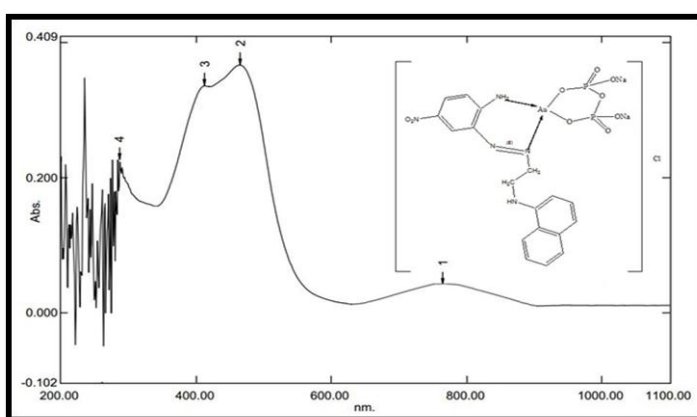


Figure (5) Electronic spectrum of [Au(C₁₈H₁₇N₅O₂)(Pyph)]Cl

Table 2 shows the FTIR spectrum of the ligand and the produced compounds

Compounds	NH ₂ asy V	NH ₂ syV	NH ₂ δV	NH	N=N	COO asy V	COO sy V	P=O	O-P-O	M-N	M-O	CH Arom	CH Aliph
Primary Ligand	352 4	342 7	1654	3307	146 9 141 2	-----	----	----	-----	----	----	307 0	292 4
[Au(C ₁₈ H ₁₇ N ₅ O ₂)Cl ₂]Cl	342 3	337 2	1651	3301	145 2 139 0	-----	----	----	-----	433	----	307 0	292 6
[Au(C ₁₈ H ₁₇ N ₅ O ₂)(Ox)]Cl	350 1	340 2	1651	3306	144 3 139 3	151 6	121 5	----	-----	459	592	307 0	292 4
[Au(C ₁₈ H ₁₇ N ₅ O ₂)(Ma)]Cl	344 2	342 7	1653	3300	145 0	151 6	121 5	----	-----	495	570	307 2	290 8



					140								
					0								
[Au(C₁₈H₁₇N₅O₂)(Pyph)]Cl	342	337	1653	3307	145	----	----	950	1213	461	503	307	292
	7	2			4		-					2	4
					139								
					6								

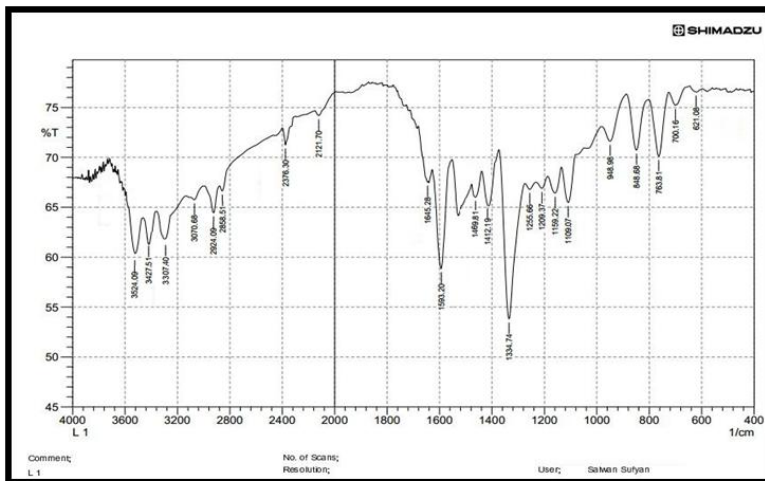


Figure (6) FTIR-Spectrum of the Primary Ligand (C₁₈H₁₇N₅O₂)

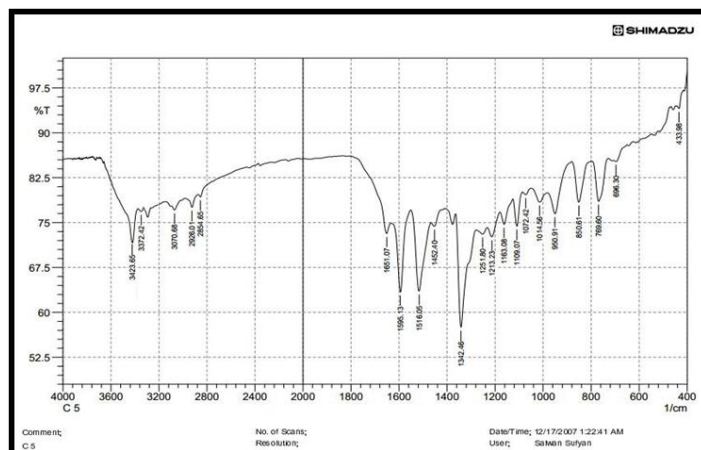
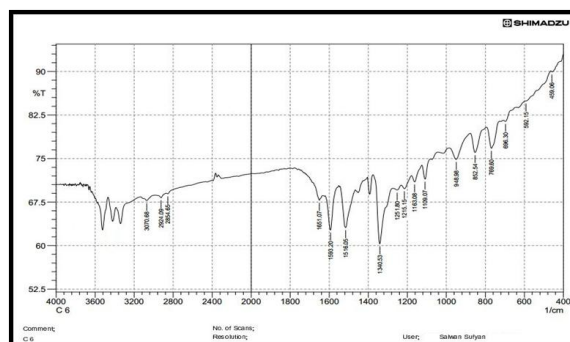


Figure 7: FTIR-Spectroscopy of [Au(C₁₈H₁₇N₅O₂)Cl₂]Cl



FTIR-Spectroscopy of [Au(C₁₈H₁₇N₅O₂)(Ox)]Cl Figure 8:



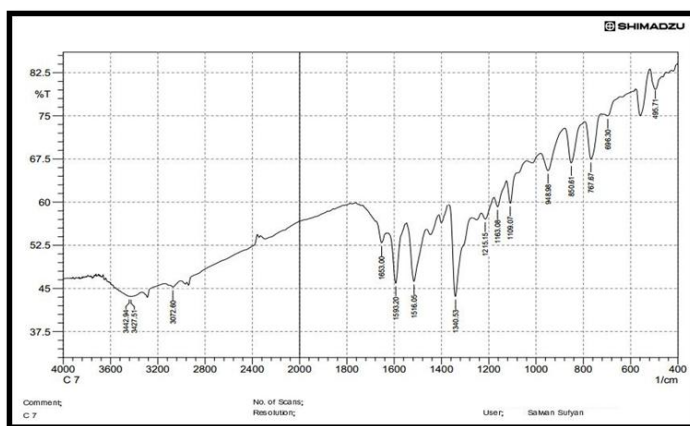
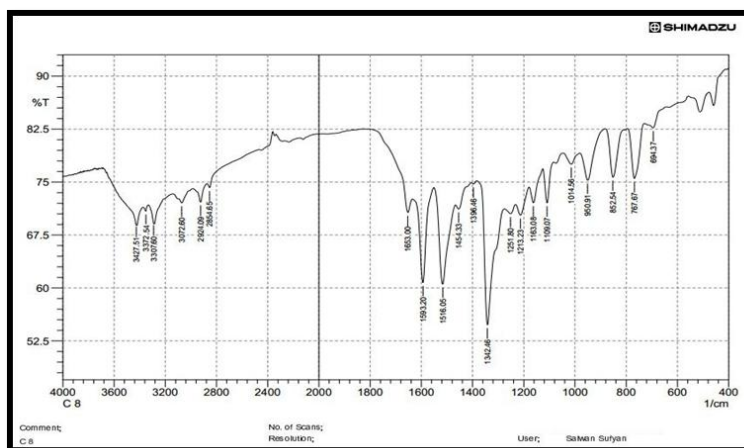
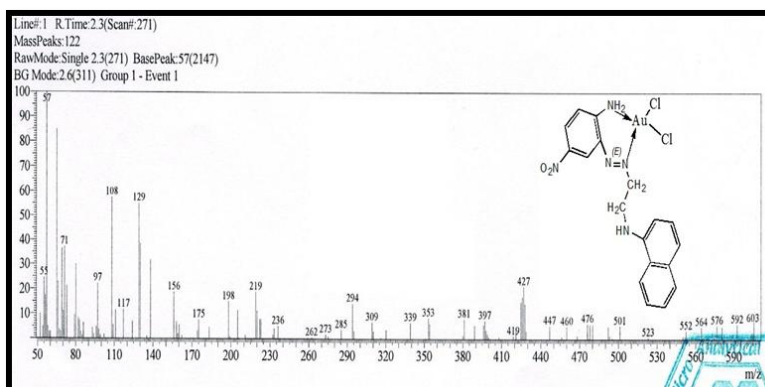


Figure (9): FTIR-Spectroscopy of $[Au(C_{18}H_{17}N_5O_2)(Ma)]Cl$



FTIR-Spectrum of $[Au(C_{18}H_{17}N_5O_2)(Pyph)]Cl$ (Figure 10)



Figure(11) Mass spectrum of $[Au(C_{18}H_{17}N_5O_2)Cl_2]Cl$



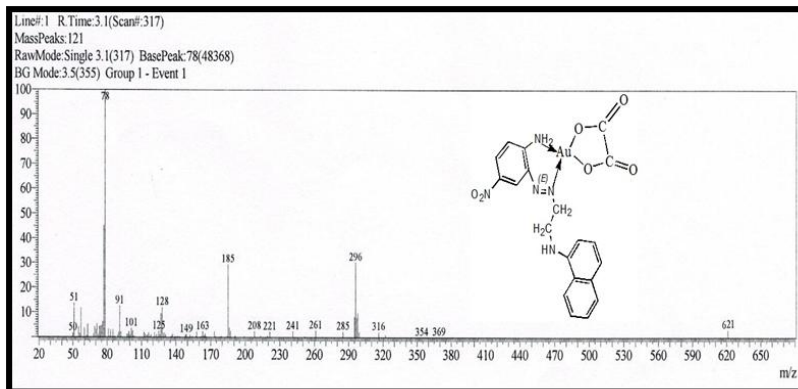


Figure 12: $[\text{Au}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Ox})]\text{Cl}$ mass spectrum

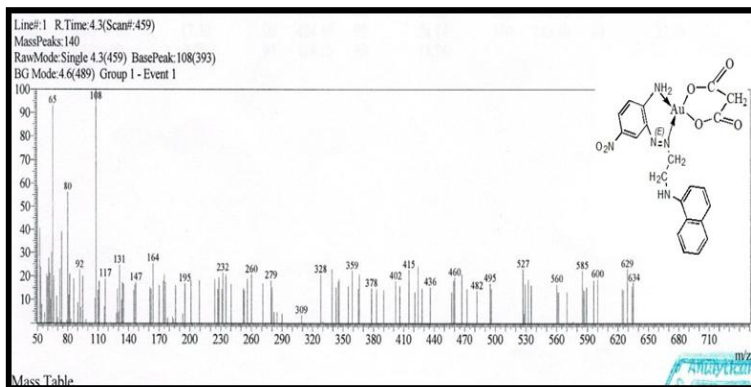


Figure (13) $[\text{Au}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Ma})]\text{Cl}$ mass spectrum

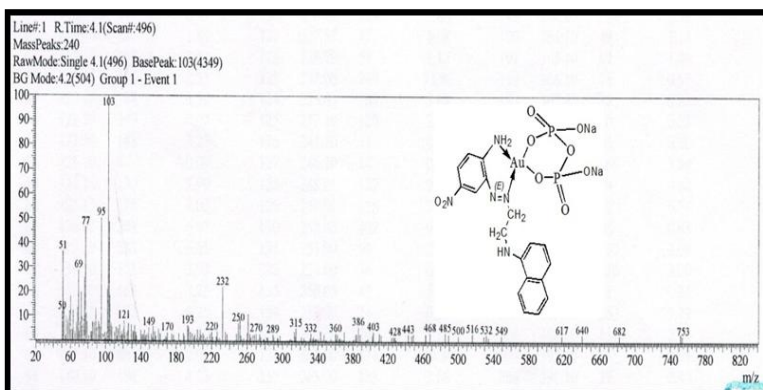


Figure (14) The mass spectrum of $[\text{Au}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Pyph})]\text{Cl}$

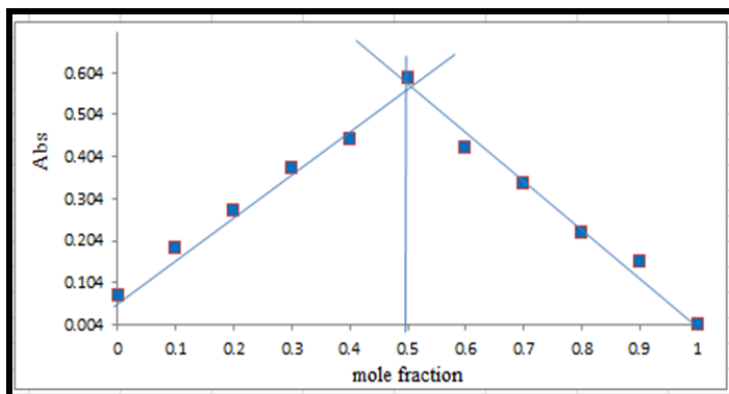


Figure (15): The Gob method's curve for the Au: L ratio.



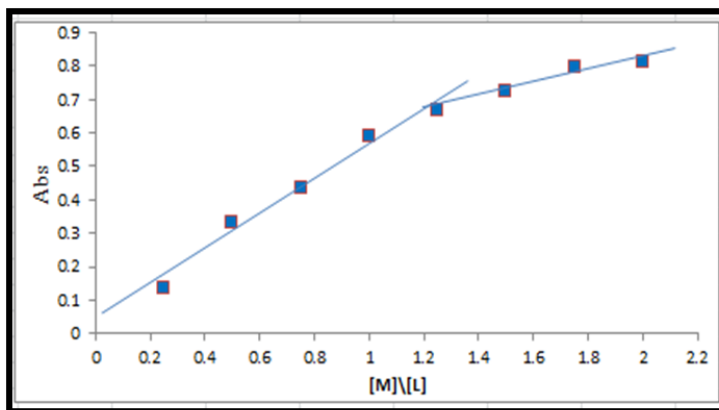


Figure 16: Au:L curve mole ratio technique

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