Synthesis, Characterization and Spectral Studies of a New Azo-Schiff base Ligand Derived from 3,4-diamino benzophenone and its Complexes with Selected Metal Ions

Review Article

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Article History:
Received: 2020-03-23
Revised: 2020-06-24
Accepted: 2020-06-28
Published: 2020-07-01

Keywords:
Azo-Schiff base
3,4-diamino benzophenone
spectral studies
transition metal complexes

How to cite?
Hadi, M. A., & Kareem, I. K. (2020). Synthesis, Characterization and Spectral Studies of a New Azo-Schiff base Ligand Derived from 3,4-diamino benzophenone and its Complexes with Selected Metal Ions. *Research Journal in Advanced Sciences*, 1(1). Retrieved from https://royalliteglobal.com/rjas/article/view/251

Published in Nairobi, Kenya by Royallite Global in the **Research Journal in Advanced Sciences**

Abstract

The complexes of Co(II), Ni(II), Cu(II), Zn(II), Cd(II), Pt(II), and Au(III) were prepared from the complexation reaction between the new Azo -Schiff base ligand and Selected Metal Ions. This ligand(3,4-bis(((1E)-1-(2-((2,4-dihydroxy-6-methylphenyl) diazenyl) phenyl) ethylidene) amino) phenyl) (phenyl) methanone) was derived from the condensation reaction of 3,4-diamino benzophenone, 2-amino acetophenone, and 3,5-dihydroxy toluene. The ligand was characterized by C.H.N elemental analysis, Uv-Vis, FT-IR, ^{1}H-NMR, and mass spectra studies, its complexes characterized by C.H.N elemental analysis, Uv-Vis, FT-IR, mass of Ni(II) complex and 1H-NMR of Zn(II) complex , molar conductivity, Atomic absorption, magnetic moment measurements. The results of this studies show the coordination sites for the ligand with the metal ion were to be through oxygen of the hydroxyl group, and the nitrogen of azomethine group and the nitrogen of azo group. The electronic spectral and magnetic measurement data predict octahedral structure of the complexes, except for Pt(II) and Au(III) complexes were suggested a square planar geometry. All complexes show that non electrolytes properties and no conductive species but Pt(II) and Au(III) complexes are electrolyte nature.

Public Interest statement

This short review focuses on ligand and complexes of Co(II), Ni(II), Cu(II), Zn(II), Cd(II), Pt(II), and Au(III) and how they are prepared from the complexation reaction between the new Azo -Schiff base ligand and Selected Metal Ions. This ligand (3,4-bis(((1E)-1-(2-((2,4-dihydroxy-6-methylphenyl) diazenyl) phenyl) ethylidene) amino) phenyl) (phenyl). These compounds have many applications, including in analytical chemistry, for example in the manufacture of selective and industrial electrodes as inhibitors of corrosion, inks and compact disc (CD), it can also be used in biology as anti-cancer.

Introduction

Schiff bases are the most important class and studies extensively class because of wide spread biological applications, easily prepared, chelating properties and stability [1,2]. In addition to, Schiff bases ligands have played important role in the development of inorganic biochemistry and pharmacological activities as antibacterial [3], antioxidant [4], anticancer [5], antifungal [6]. Schiff bases of 3,4-diamino benzophenone and their complexes have a variety of application biological, analytical and pharmacological areas, many researches had been carried out to study the uses in various fields like antimicrobial and molecular docking studies as well as thermodynamics studies of complexes formation with metal ions [7,8]. Azo-Schiff bases compounds are a new class of chemical compounds that are receiving increasing concern in scientific research [9], Metal complexes containing azo-Schiff bases are of interest because of their electronic properties and the synthetic flexibilities, selectivity and sensitivity towards the metal ions. In this work, we have prepared and studies physiochemical properties of new ligand (3,4-bis(((1E)-1-(2-((2,4-dihydroxy-6-methylphenyl) diazenyl) phenyl) ethylidene) amino) phenyl) (phenyl) methanone) and its metal complexes with selected metal ions.

Experimental

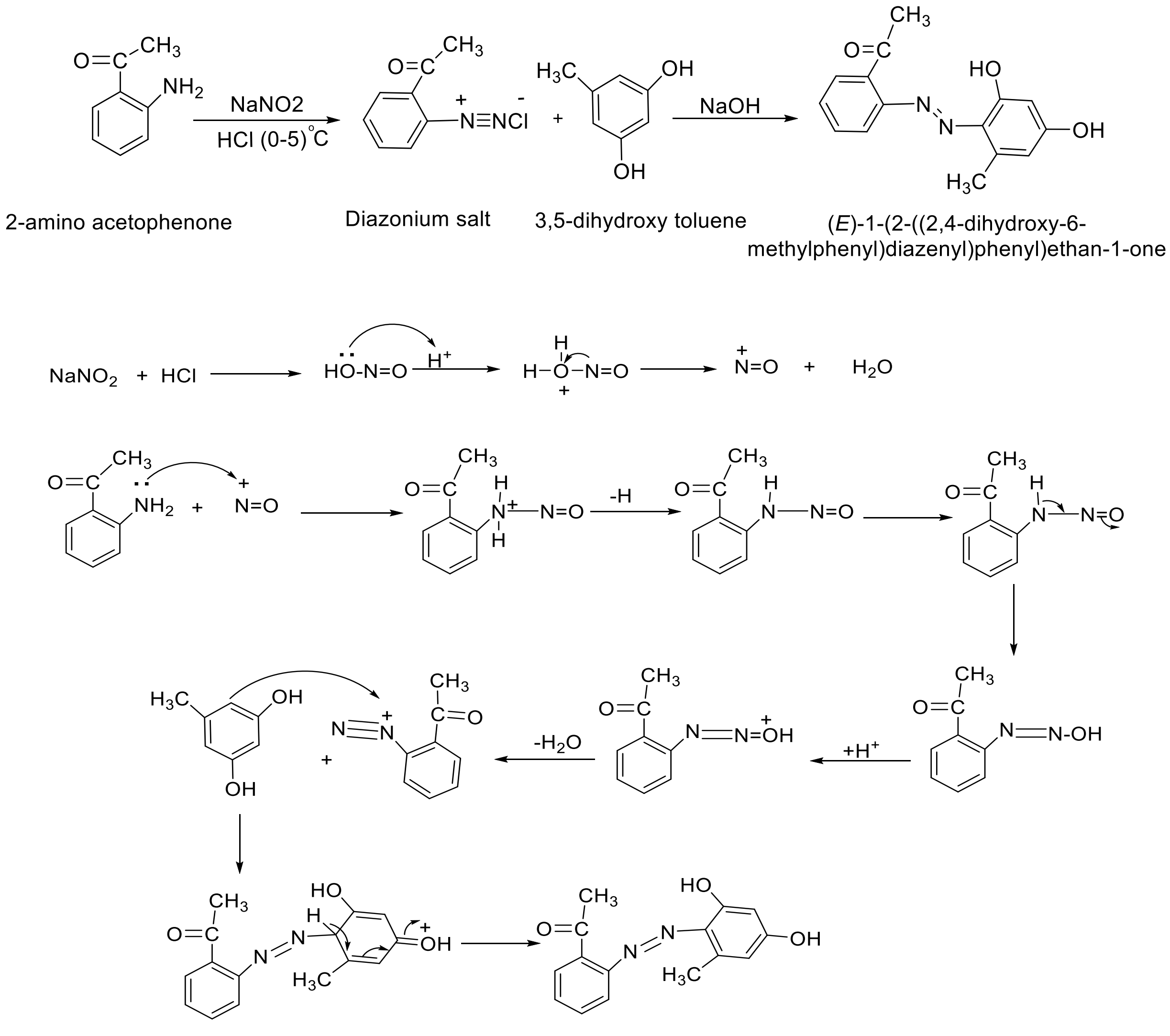
Materials and Methods

All chemicals were obtained from Sigma - Aldrich, and Merck and used without further purification. Melting point were determined using model 9300 of ligand and its complexes. The UV-Visible spectra were recorded on Shimadzu spectrophotometer double beam model 1700. IR spectra were recorded on Shimadzu FTIR 8400 spectrometer using KBr pellet in the wavelength range 4000-400 cm^{-1}. Magnetic susceptibility measurements were carried out on a balance magnetic MSB-MKI using faraday method. The diamagnetic corrections were made by Pascal's constants. Elemental analyses were performed by means of EURO 2012EA 300 C.H.N Elemental analysis. ^{1}HNMR spectra were recorded as solution in DMSO d^6 as solvent using (varian 500MHZ Spectrophotometer), and Mass Spectra were recorded on Shimadzu Agilent Technologies 5975C.

Preparation of new azo-Schiff base ligand

Preparation of the diazo dye (DMPDE)

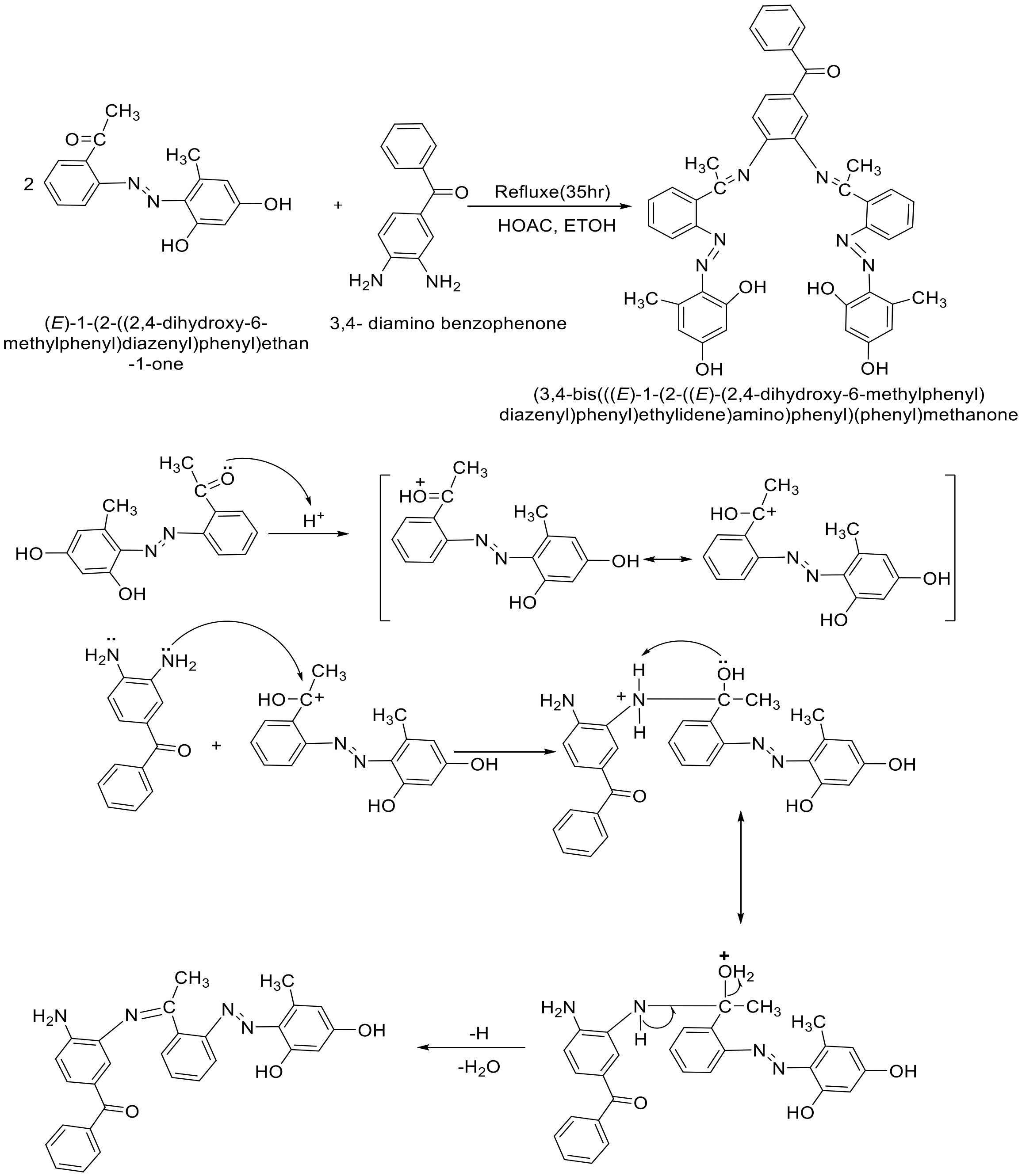
The preparation of ligand included dissolving (1.35g, 0.01mol) of 2-amino acetophenone in (4ml) concentrated HCl and (30ml) distilled water. After cooling this solution to 0℃, the solution was treated with (0.75g,0.01mol) of sodium nitrite in (20 ml) of distilled water was added drop wise and stirred for 30 min at (0-5℃), the diazoinum chloride solution was added wise with stirred to alkaline solution of 3,5-dihydroxy toluene (1.24g, 0.01mol) which dissolved in (50ml)of ethanol and (70ml) of 10% sodium hydroxide was added. The mixture was allowed to stand overnight and acidified with dilute hydrochloric acid until pH=6, then washed several times with distilled water, air dried and recrystallized twice from ethanol, then dried in the oven at 60℃ for two hours, m.p (210-212℃). Yield: 80%. **Scheme 1**.



Scheme 1: Preparation and mechanism of the azo dye (DMPDE**)**

Preparation of the anew azo-Schiff base ligand (3,4-bis(E)-(2,4- dihydroxy-6- methylphenyl) diazenyl) phenyl)ethylidene) amino)phenyl)(phenyl)methanone.

The new azo- Schiff base ligand was prepared by condensation (5.4g, 0.02 mol) of (DMPDE) in (30 ml) ethanol absolute with 3,4-diamino benzophenone (2.12g ,0.01 mol) in the same solvent, equimolar (1:2) mole ratio. Few drops of glacial acetic acid were added to the reaction mixture and refluxed for (35h). The product was recrystallized from ethanol and dried over anhydrous $CaCl_2$.m.p (170-172℃), Yield: 88%. As showing in **Scheme 2.**



Scheme 2: Preparation and mechanism of the new azo –Schiff base ligand.

Preparation of metal complexes

Complexes of the ions $CoCl_2.6H_2O$, $NiCl_2.6H_2O$, $CuCl_2.2H_2O$, $ZnCl_2.H_2O$, $CdCl_2.H_2O$, K_2PtCl_4, $NaAuCl_4.2H_2O$ were prepared by the mixing of (50ml) ethanolic solution with (0.716g, 1mmol) of Azo-Schiff base ligand dissolved in (50ml) from the same solvent in (1:1) (metal: ligand) ratio. The resulting mixture was refluxed for (2 hours) at (70-80℃) and cooled to room temperature. The final product was filtered and washed with ethanol, dried in air.

Results and Discussion

The new Azo- Schiff base ligand and the metal complexes are colored. All complexes are stable towards air. Generally, the complexes are insoluble in water, but they are soluble in common organic solvents (DMSO, DMF, Methanol and ethanol). The metal complexes were characterization by (C.H.N) Micro elemental analysis, FT-IR, UV-Vis, ^{1}HNMR, molar conductivities and the magnetic moment. The analytical data of the complexes are agreed with the experimental data. The molar conductivity values show that of the complexes were (1:1) (metal: ligand), this result proves that complexes have non-electrolytic nature, except the Pt(II) and Au(III) complexes which showed higher conductivity value, this support the electrolytic nature of the chelate complex. The magnetic moments value of the complexes are agreement with geometry structure. Some physical and chemical propertied for the new azo-Schiff base ligand and its chelate complexes are recorded at **Table 1**.

Table 1. the analytical and the physic-Chemical data of the new azo –Schiff base ligand and its metal complexes.

No.	***Compound***	***Formula Weight***	***Colour***	***M.p.℃***	***Yield %***	***Found (Calc.)%***			
						%C	*%H*	*%N*	*%M*
1	$HL=C_{43}H_{36}N_6O_5$	716	Deep red	170-172	88	(72.06) 71.60	(5.02) 5.42	(11.73) 11.18	-----
2	$[CoL].H_2O$	790.9	Green	238-240	78	(65.24) 64.88	(4.29) 3.92	(10.62) 10.78	(7.44) 7.29
3	[NiL]	772.6	Brown	>310	70	(66.78) 65.53	(4.40) 4.52	(10.87) 9.98	(7.58) 6.89
4	$[CuL].H_2O$	795.5	Green	>310	80	(64.86) 64.61	(4.27) 4.02	(10.55) 9.92	(7.98) 7.35
5	$[ZnL].H_2O$	797.3	Colorless	193-195	72	(64.71) 65.74	(4.26) 4.31	(10.53) 10.42	(8.19) 7.87
6	$[Cd L].H_2O$	844	Colorless	187-190	74	(61.13) 60.89	(4.02) 4.54	(9.95) 9.21	(13.27) 13.50
7	$[PtL]Cl_2$	982	Green	>310	70	(52.54)	(3.66)	(8.55)	(19.85)

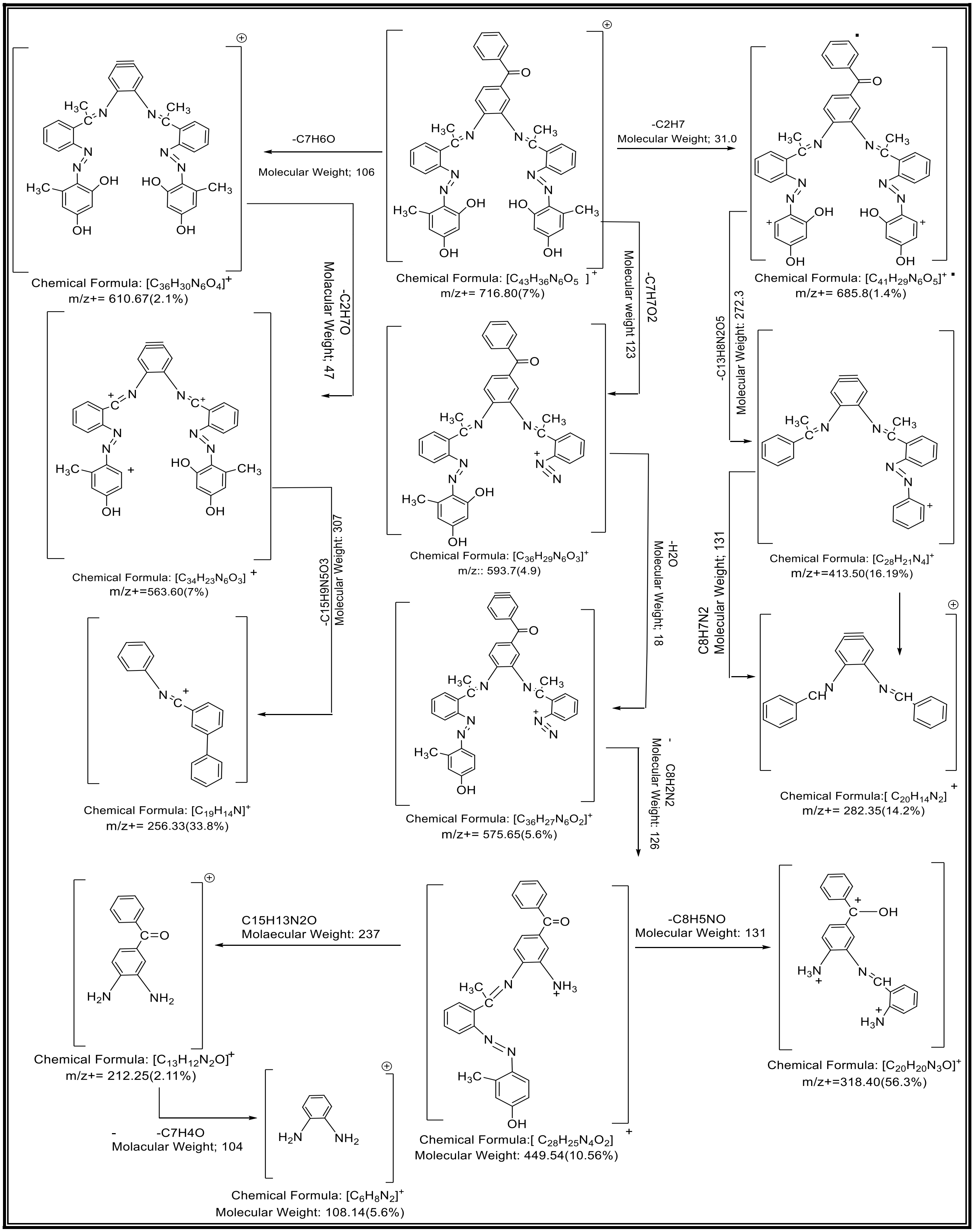
						52.22	3.13	8.34	18.65
8	[AuL]Cl_3	1019.4	Green	>310	75	(50.61)	(3.53)	(8.24)	(19.31)
						50.27	3.13	8.12	19.20

Mass Spectra

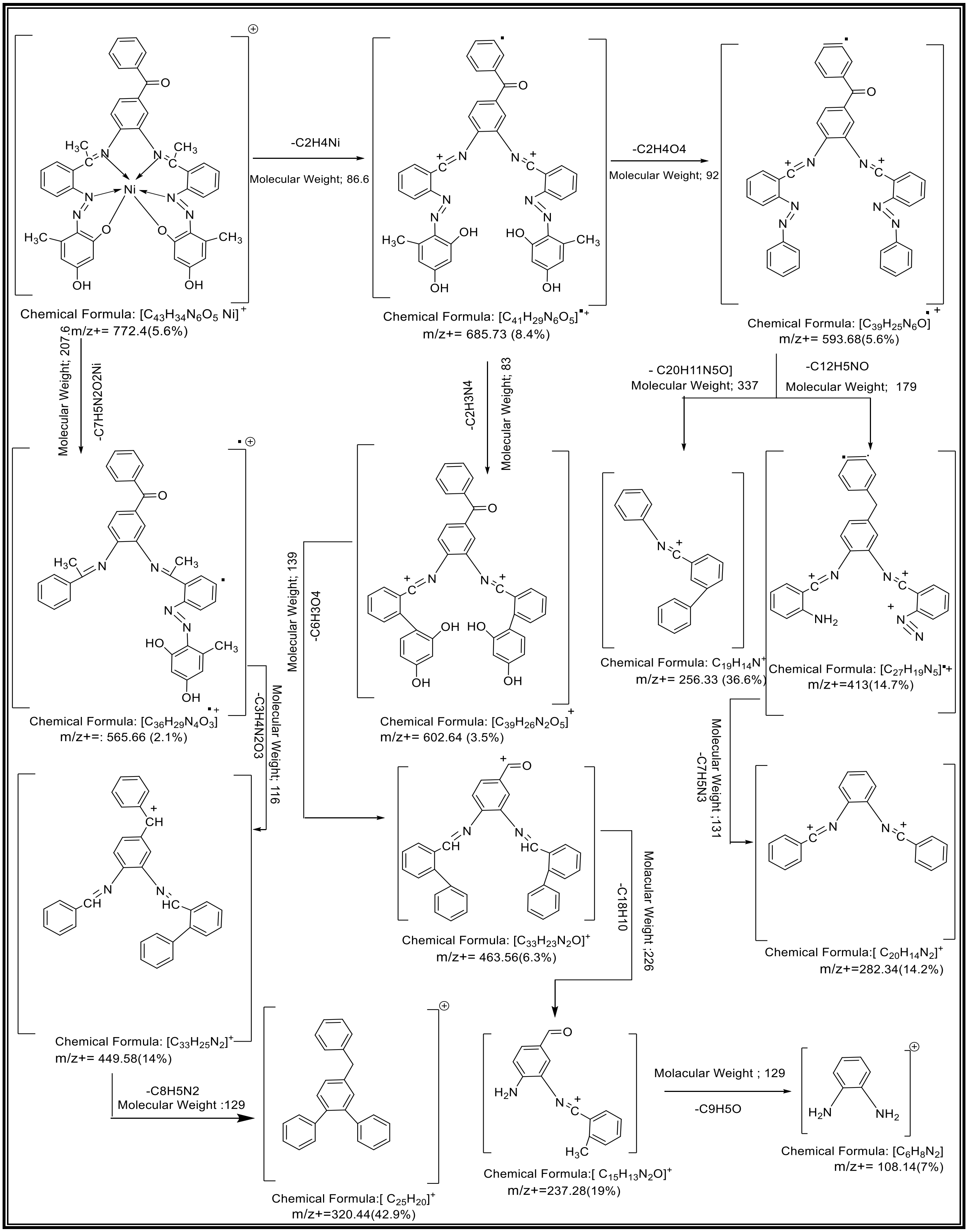
Mass spectra **(Figure.1 and 2)** of the new azo-Schiff base ligand and its Ni(II) complex**,** clearly shows the mother ion peak of ligand at m/z^+ = 716.8(7%) which assigned to M^+, suggested fragmentation pathways and structural assignments of fragments. The molecular ion peak for Ni(II) complex observed at m/z^+ =772.4(5.6%) which confirms to the check molecular weight of the complex. The obtained molecular ion peaks confirm the proposed formulae for ligand and Ni(II) complex, the mass spectrum supports the proposed empirical formula for the synthesized compounds, the data confirming were in agood agreement with the suggested structure. The mass fragmentation pattern of the new azo-Schiff base ligand and its chelate complex is shown in **schemes 3 and 4.**

^{1}HNMR spectra

The ^{1}HNMR spectra of the new azo-Schiff base ligand and Zn(II) complex are show in **(Figure 3 and 4)**, DMSO-d^6 solvent with TMS as internal standard, the spectrum of the ligand exhibits signals at δ= (9.5ppm, 9.7 ppm) belong to proton of hydroxyl groups (-OH), the phenyl multiples at δ= (6.1-8.3)ppm is endorsed for aromatic protons, the peaks at δ= (1.7)ppm (-N=C-CH_3) is belong to methyl protons of the azomethine group, (CH3-phenol) at δ= (2.3ppm) [10,11]. The position phenolic (-OH) proton of the first peak is disappear in the spectra of Zn(II) complex indicating the involvement in bonding and absence proton, while the second peak of (-OH) proton was noted in the spectra of complex[2, 12].



Scheme 3. Suggested fragmentation pathways of the new azo-Schiff base ligand and structural assignments of fragments



Scheme 4. Suggested fragmentation pathways of Ni- complex and structural assignments of fragments

FT-IR Spectra of ligand and its metal ion complexes

FTIR spectra the new azo- Schiff base ligand and their metal complexes are scheduled in **(Figure 5 and 6).** In The infrared spectrum of the ligand exhibit two broad bands at (3431, 3375) cm^{-1} were assigned to **υ**(OH) group in the free azo-Schiff base ligand confirmed that hydroxyl group was present in azo-Schiff base ligand, which is absent in all metal complexes except the Pt(II) and Au(III) complexes, revealed that it was coordinated to metal ions via deprotonation of phenolic **υ**(OH) [13,14]. Band at (1666) cm^{-1} due to carbonyl **υ** (C=O) of benzophenone group, unchanged this band in the complexes [15].

The strong bands at (1653, 1622, 1602) cm^{-1} that was assigned to **υ**(CH=N) in the free azo-Schiff base ligand, the azomethine peak in ligand is shifted, indicating the coordination through **υ** (-C=N-) group [16,17]. The spectra of ligand show bands at (1487, 1456, 1390) cm^{-1} due to the presence of **υ** (-N=N-) group displaced into lower or longer wave number for change during shape at spectra for all the complexes [18]. The appearance of new absorption bands at (489-447) cm^{-1} and (567-516) cm^{-1} refer to **υ**(M-N), **υ**(M-O) sequence in some complexes, confirmed that O and N atoms of the ligand banded with the metal ions [19]. The complexes Co(II), Cu(II), Zn(II), and Cd(II) showed new abroad bands at (3356-3433)cm^{-1}, suggesting presence of the water molecular outside the coordination sphere [20]. Results showed that The ligand behavior as a hexadentate chelating, bonded to the metal ion via the two nitrogen atoms azomethine **υ**(-C=N-), azo **υ**(-N=N-) groups, and phenolic **υ**(OH) groups and of the azo –Schiff base ligand, except the Pt(II) and Au(III) complexes the ligand acts as a tetra dentate chelating agent, indicates that the coordination of azo methine nitrogen atoms and azo nitrogen atoms. The spectra data of the new azo – Schiff base ligand and the metal complexes were shown in **table (2)**

Table .2: FT-IR spectral data (cm^{-1}) of azo-Schiff base ligand and its complexes

Compound	*υ(OH)*	*υ(OH) H_2O*	*υ(C=N)*	*υ(N=N)*	*υ(M-N)*	*υ(M-O)*
Ligand $C_{43}H_{36}N_6O_5$	3431, 3375	-----	1653,1622 ,1602	1487, 1456,1390	-----	-----
[Co L].H_2O	-----	3433	1641, 1622 1600	1485, 1450, 1406	484	555
[Ni L]	3441	-----	1651, 1552	1408, 1367	474	524
[Cu L].H_2O	-----	3406	1639,1624, 1600	1485, 1450,1402	484	567

[Zn L].H_2O	-----	3356	1691,1662 1624	1487, 1465,1448	487	516
[Cd L]. H_2O	-----	3415	1625,1598,1568	1487, 1467, 1448	447	534
[Pt L] Cl_2	3454, 3356	-----	1640,1622	1486, 1458, 1385	489	-----
[Au L] Cl_3	3527, 3437	-----	1654,1598, 1573	1489, 1467,1450	482	-----

Electronic spectra and magnetic susceptibility measurements

Ultraviolet–visible spectra is one of the important methods used in the field of coordination chemistry, is studied and compared metal complexes spectra with free ligand spectra. The electronic absorption spectra of the new azo –Schiff base ligand and its complexes were recorded in freshly ethanol solution (10^{-3}) at room temperature. The absorption region band assignment and the proposed geometry of the complexes Co(II), Ni(II), Cu(II), Zn(II),Cd(II) are octahedral except Pt(II), Au(III) complexes, which had square planar geometry. Their relevant data shown in **Table (3)**.

The new azo-Schiff base ligand is red in colour and shows two charge transfer (CT) absorption bands in the region Uv-Visible at (246)nm (40650) cm^{-1}, and (429)nm (23310) cm^{-1}. the first band identified for (π -π^*) transition involving molecular orbitals of aromatic rings, the second band is accounts for (n-π^*) transition, which involves molecular orbitals essentially localized of the chromophore groups [1,21]. In the metal complexes, these absorption bands undergo of red-shifted indicating that the azo-Schiff base ligand are coordinated to the metal ions [22].

The Co(II) complex is green in colour exhibits new three absorption bands at 781 nm (12804)cm^{-1}, 679 nm (14727)cm^{-1},611 nm (16366cm^{-1}) assigned to $^4T_1g\rightarrow{}^4T_2g_{(F)}(\nu_1)$, $^4T_1g\rightarrow{}^4A_2g_{(F)}(\nu_2)$, $^4T_1g\rightarrow{}^4T_2g_{(p)}(\nu_3)$ transitions respectively [23]. This indicate the present of an octahedral geometric structure of cobalt (II) complex. The magnetic moment value 4.21 B.M may be taken as additional evidence for octahedral geometry [24]. The Ni(II) complex is brown in colour exhibits absorption bands at 749nm (13351) cm^{-1}, 564nm (17730) cm^{-1}, 460nm (21739)cm^{-1} which assigned to $^3A_2g\rightarrow{}^3T_2g_{(F)}(\nu_1)$, $^3A_2g\rightarrow{}^3T_1g_{(F)}(\nu_2)$, $^3A_2g\rightarrow{}^3T_1g_{(P)}(\nu_3)$ transition respectively [25]. The magnetic moment value of this complex is 3.02 B.M which was very close to the octahedral geometry structure [26].

The spectrum of the Cu(II) complex appears abroad absorption band around at 703nm (14224 cm^{-1}) described to electronic transition type $^2Eg \rightarrow {}^2T_2g$, the magnetic moment of this complex was found at (1.81 B.M) which was very close to octahedral environment [27]. The spectrum of Zn(II), Cd(II) complexes showed band at 484nm, (20661cm^{-1}),and 574nm (17421cm^{-1}) respectively assigned to a charge transfer transition, no (d-d) field transition band but always shows prominent (C.T) transition. The Zn(II), and Cd(II) complexes is diamagnetic moment because possess completely filled d10configuration [28]. Based on these data, an octahedral geometry is proposed to the complexes.

The spectrum of the Pt(II) complex is green in colour display one absorption bands at 738 nm (13550 cm^{-1}) showed the spin-allowed transition which designed $^1A_1g \rightarrow {}^1T_1g$ transition, the magnetic moment are diamagnetic nature because has ($5d^8$) system, according to electronic spectra data suggest a square planar geometry around Pt(II) ion [29, 30]. The electronic spectrum of the Au(III) complex exhibit one band at 544 nm (18382cm^{-1}) which assigned to $^1A_1g \rightarrow {}^1B_1g$ transition [31], the Au(III) complex is diamagnetic moment and a square planar [32]. The UV-Vis spectra of the new azo-Schiff base ligand and Cu(II) complex are shown in **(Figure 7 and 8).**

Molar conductivity measurements

The conductivity values (Λμ) of prepared complexes in DMSO solvent at a concentration of (10^{-3}M) in room temperature are listed in **Table (3).** The result explained molar conductance in the range between (13.43 – 17.76) $S.cm^2.mol^{-1}$, indicating that the compounds are non- electrolytes [33], except the molar conductance of Pt(II) and Au(III) complexes are (76, 134) $S.\ cm^2.mol^{-1}$ respectively suggesting that the electrolytic nature, indicate are present chloride ions outside the coordination sphere [32].

Table(3):- Electronic spectra (nm, cm^{-1}) magnetic moments, geometry, hybridization and conductivity

Compounds	λ_{max} (nm)	Absorption bands(cm^{-1})	Transitions	μ_{eff} (B.M)	Geometry	Hybridization	Conductivity S.mol^{-1}. cm^2
Ligand= $C_{43}H_{36}N_6O_5$	246	40650	$\pi \rightarrow \pi^*$	----	----	----	----
	429	23310	$n \rightarrow \pi^*$				
[Co L].H_2O	781	12804	$\nu_1 = {}^4T_1g \rightarrow {}^4T_2g_{(F)}$	4.21	Octahedral	Sp^3d^2	14
	679	14727	$\nu_2 = {}^4T_1g \rightarrow {}^4A_2g_{(F)}$				
	611	16366	$\nu_3 = {}^4T_1g \rightarrow {}^4T_2g_{(P)}$				
[Ni L]	749	13351	$\nu_1 = {}^3A_2g \rightarrow {}^3T_2g_{(F)}$	2.7	Octahedral	Sp^3d^2	16
	564	17730	$\nu_2 = {}^3A_2g \rightarrow {}^3T_1g_{(F)}$				
	460	21739	$\nu_{3=}\ {}^3A_2g \rightarrow {}^3T_1g_{(P)}$				
[Cu L].H_2O	703	14224	${}^2Eg \rightarrow {}^2T_2g$	1.76	Octahedral	Sp^3d^2	17
[Zn L].H_2O	484	20661	$d\pi(Zn)^{+2} \rightarrow \pi^*(L)$	Dia	Octahedral	Sp^3d^2	13
[Cd L].H_2O	574	17421	$d\pi(Cd)^{+2} \rightarrow \pi^*(L)$	Dia	Octahedral	Sp^3d^2	15
[Pt L]Cl_2	738	13550	${}^1A_1g \rightarrow {}^1T_1g$	Dia	Square planer	dsp^2	76
[Au L]Cl_3	544	18382	${}^1A_1g \rightarrow {}^1B_1g$	Dia	Square planer	dsp^2	134

The suggested structures of the metal complexes

According to the analytical data and spectroscopic studies of the prepared metal complexes are show in **Figure(9)**. The new azo-Schiff base ligand behaves as a hexadentate chelating agent coordination through the nitrogen atoms (-N=N-) of azo group and (CH=N-) of azomethine group, and oxygen atom of phenolic (-OH) group, except the Pt(II) and Au(III) complexes, the ligand acts as a tetradentate chelating agent.

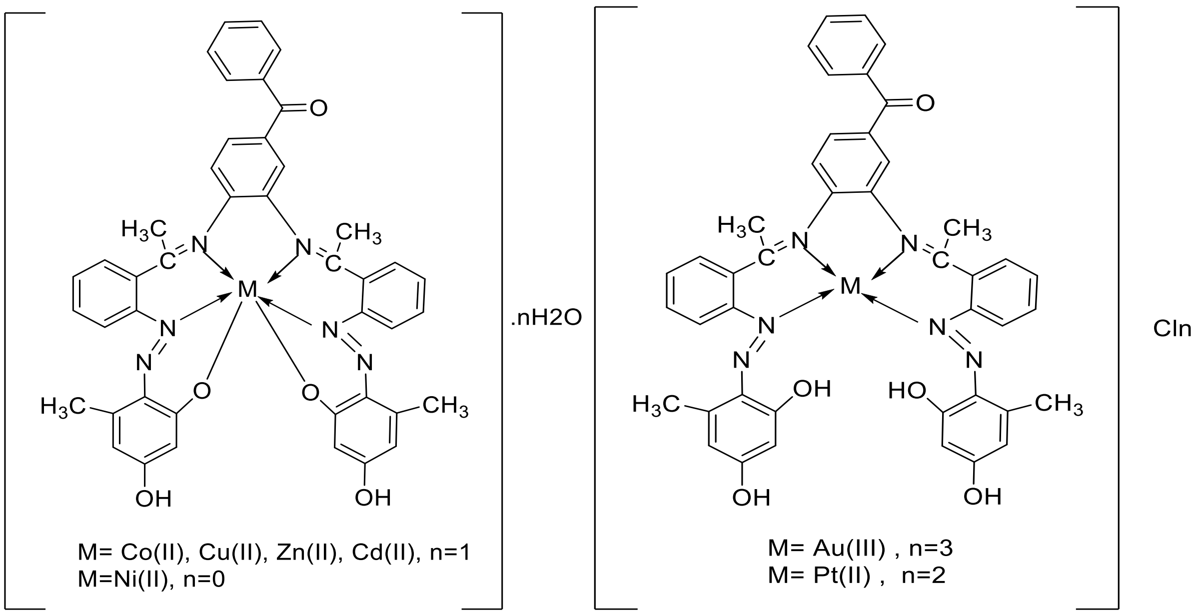


Fig (9). The proposed structural formula of the complexes.

Conclusion

Anew azo-Schiff base ligand was synthesized and characterization by different techniques. The data of the complexes suggested an octahedral geometry of the Co(II), Ni(II), Cu(II), Zn(II), and Cd(II) complexes, and a square planar of Pt(II) and Au(III) complexes. The metal complexes were coordinated to the ligand through the nitrogen atoms of (-N=N-), and (-C=N-) groups,and oxygen atom of (-OH) group. According to the mole ratio method and the analytical data showed that the metal to ligand ratio of all complexes are (1:1) (metal: ligand).

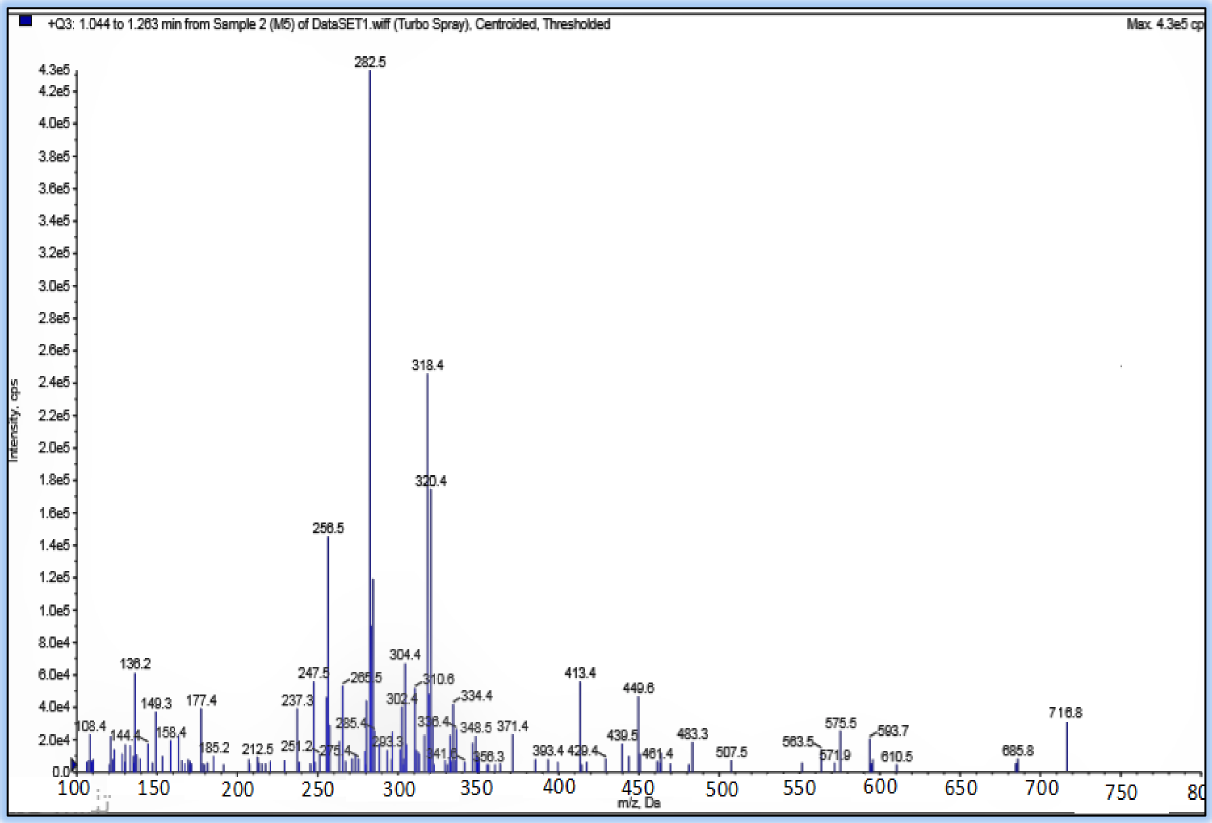


Fig. 1: the mass spectrum of the new azo-Schiff base ligand

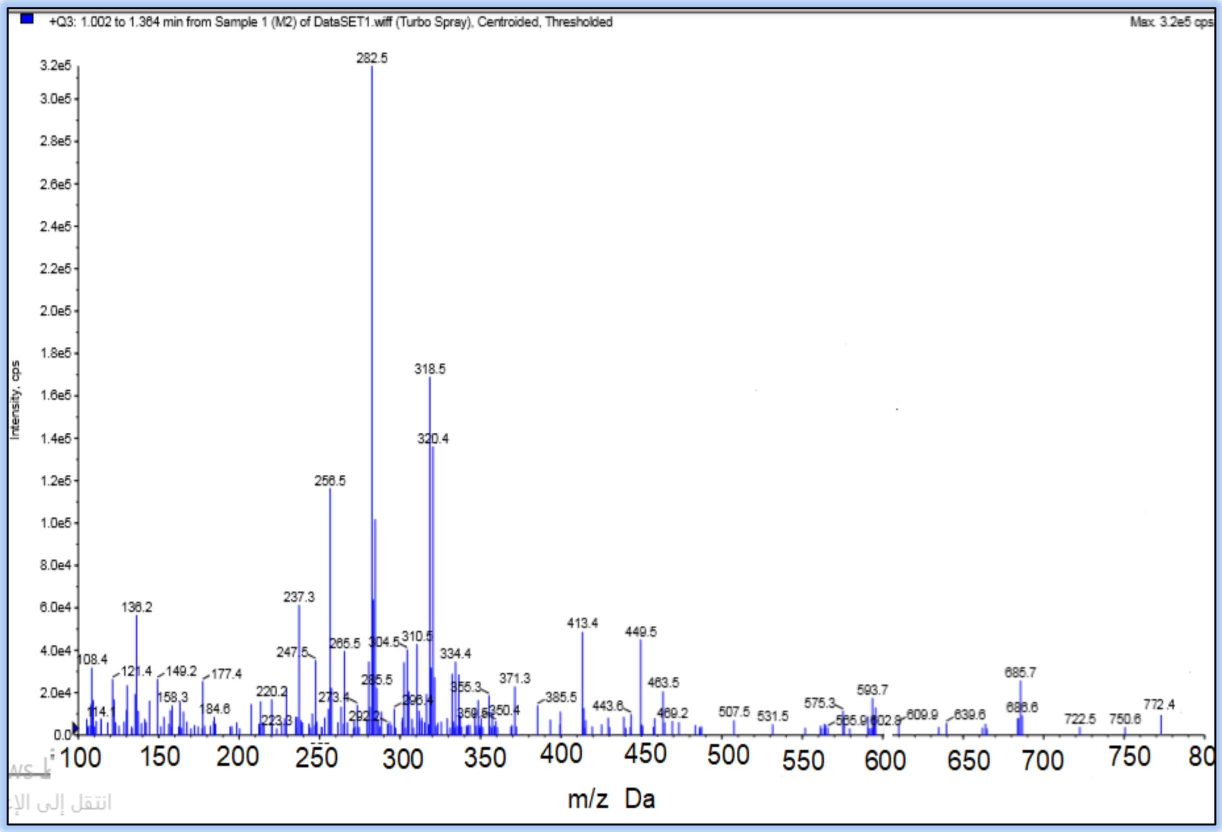


Fig. 2: the mass spectrum of the Nickel (II) complex.

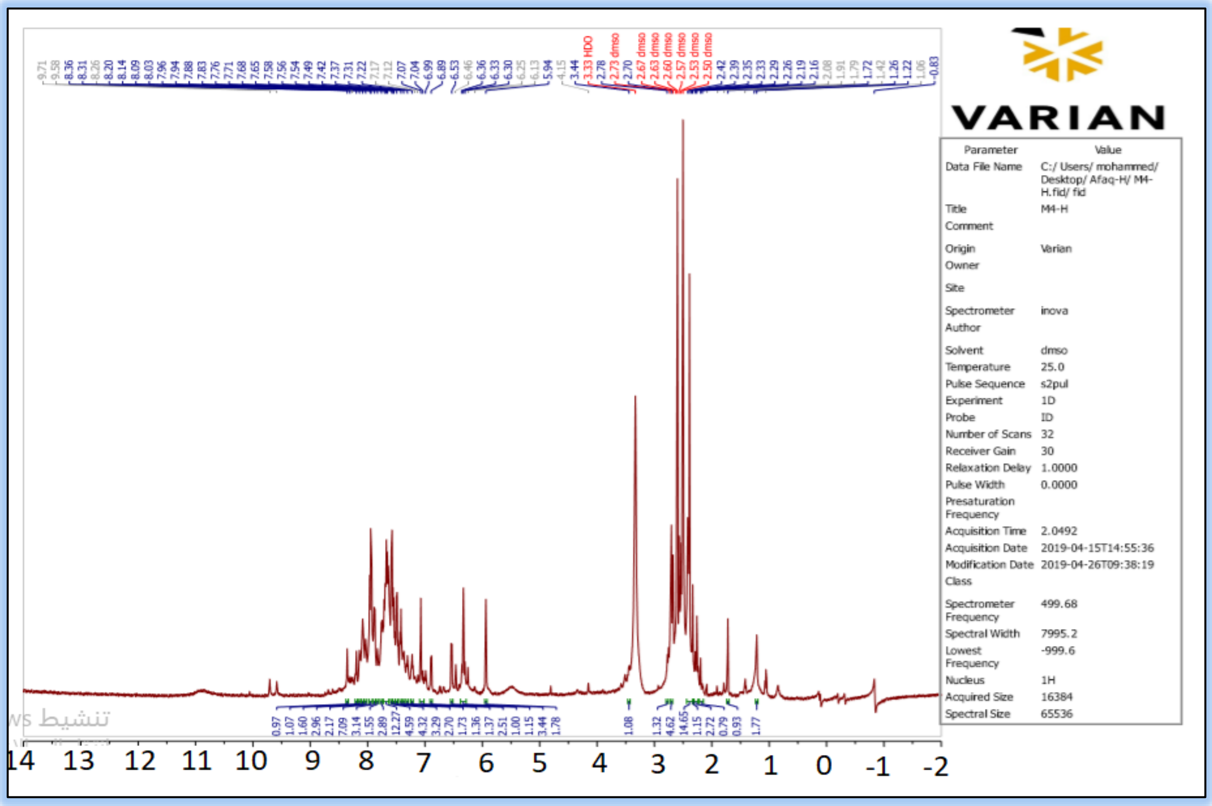


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Figure 3: ^{1}H-NMR. Spectrum of the new Azo-Schiff base ligand

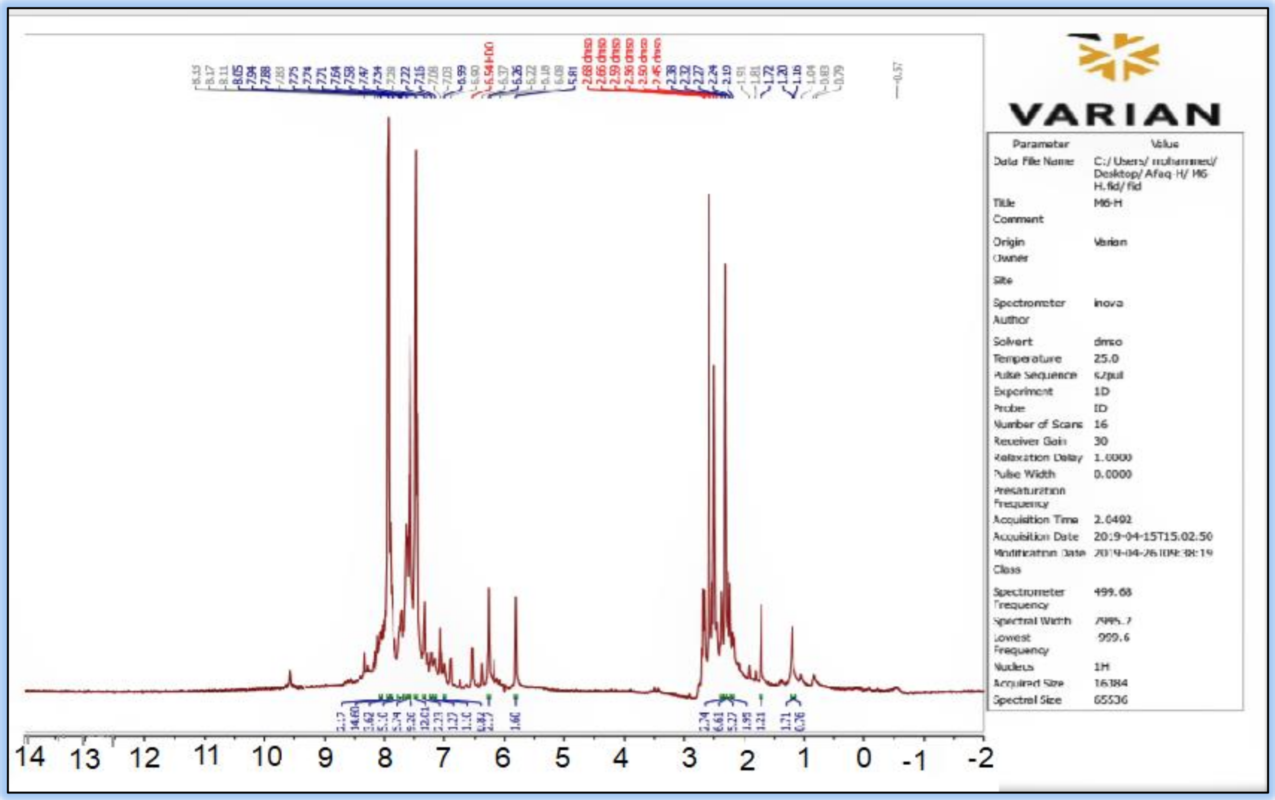

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Figure 4: ^{1}H-NMR. Spectrum of Zn(II) complex

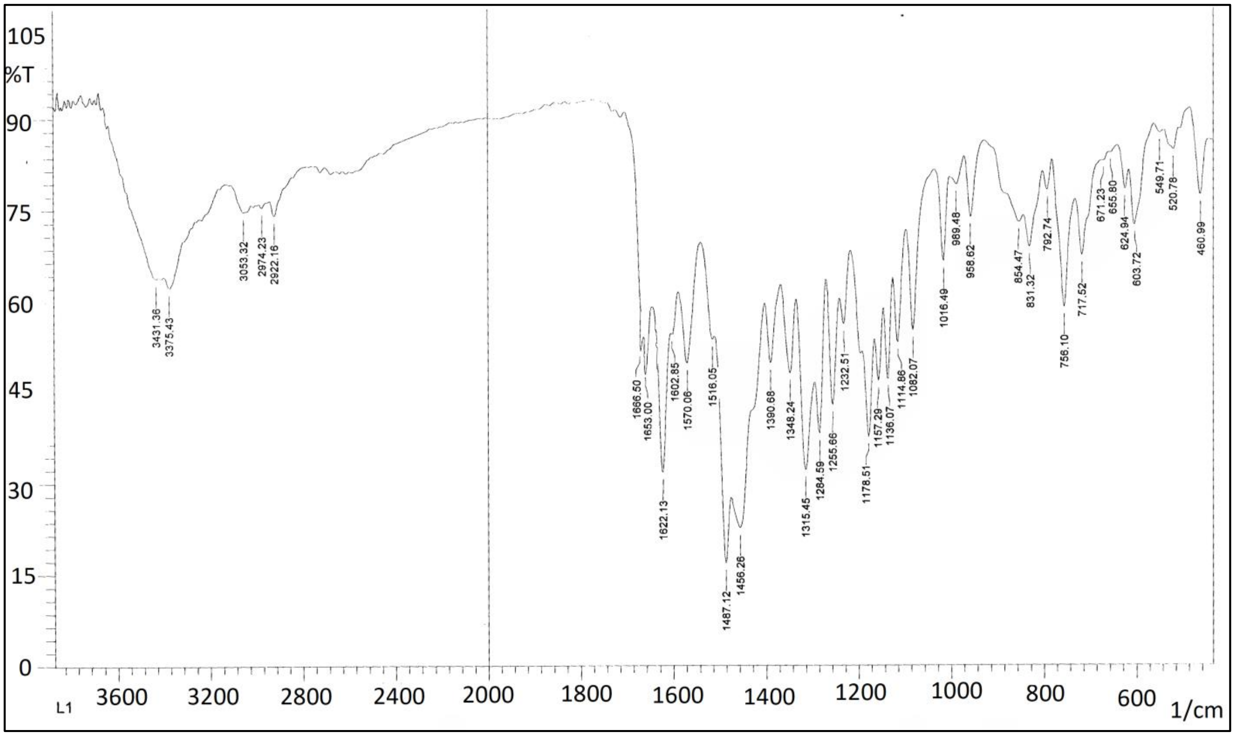

Figure 5: FT-IR bands of the new Azo-Schiff base ligand

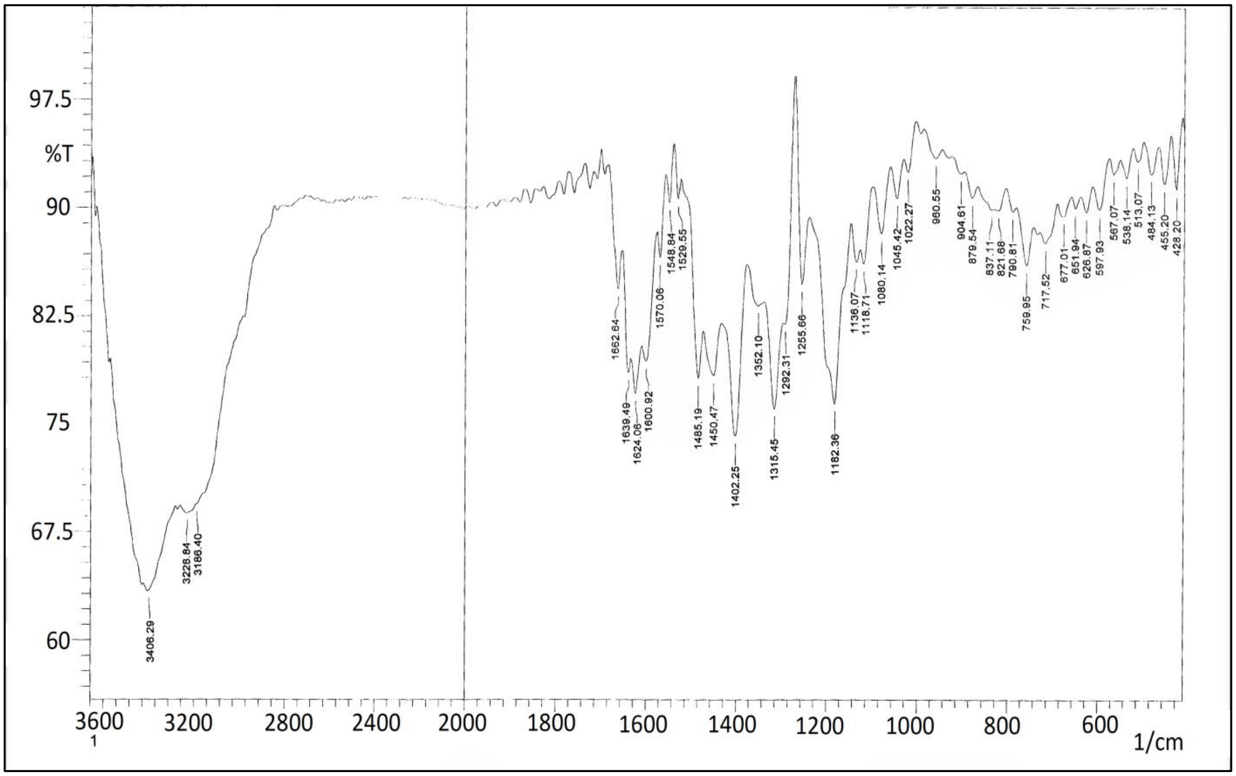

Figure 6: FT-IR bands of Cu(II) complex.

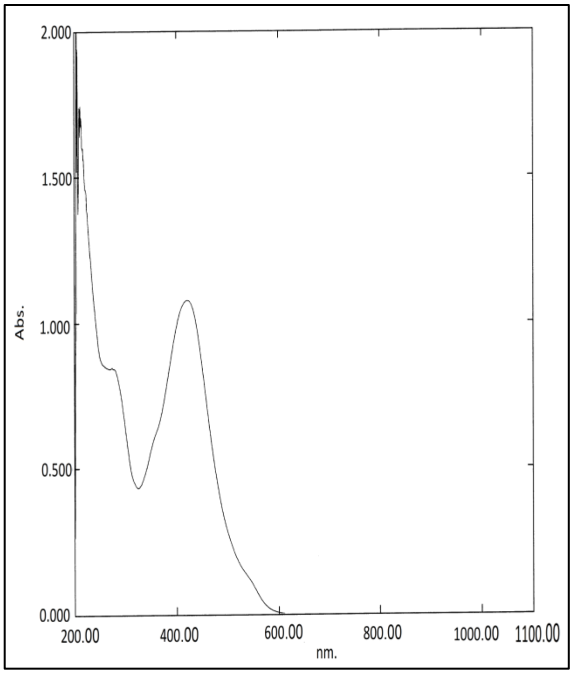

Figure 7: UV-Vis. Spectrum of the new Azo-Schiff base ligand

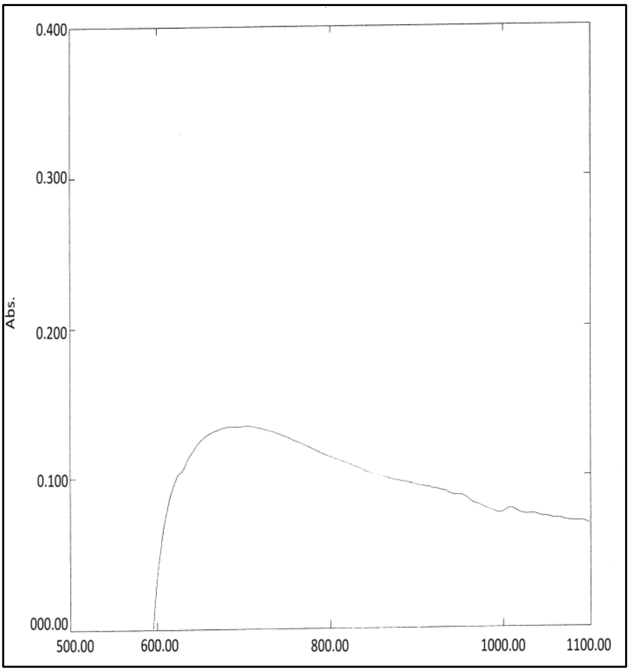

Figure 8: UV-Vis. Spectrum of Cu(II) complex

Bionote

Muna Abass Hadi is an assistant professor and a researcher in inorganic chemistry at the University of Kufa, Iraq. His research interest is in organic compounds and metal complexes that can be used in many areas.

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