# Preparation and Characterization of Some Transition Metal Complexes with a New Schiff base Ligand

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## الخلاصة

تم تحضير سلسلة جديدة من المعقدات المخلبية لأيونات المنغنيز(II) والحديد(II) والكوبلت(II) والنيكل(II) والنحاس(II) والخارصين(II) مع قاعدة شف جديدة مشتقة من الإيزاتين و N-(1-(4-إمينوفنيل) أيثليدين)-4- مثيل أنيلين. شخصت الليكاند و معقداتها المخلبية المحضرة بوساطة التحليل الدقيق للعناصر والقياسات المغناطيسية والتوصيل المولاري و الأشعة تحت الحمراء وكذلك الأطياف الإلكترونية. بينت نتائج أطياف الأشعة تحت الحمراء أن قاعدة شف تسلك كليكاند ثنائية المخلب متعادلة، تتناسق مع الأيونات الفلزية من خلال ذرة النتروجين لمجموعة الآزوميثين المرتبطة بحلقة الإيزاتين و ذرة أوكسجين مجموعة الكاربونيل للحلقة المذكورة. واستنادا إلى النتائج المستحصلة فقد تم اقتراح الشكل الفراغي المتوقع للمعقدات.

## Abstract

A new series of Mn(II), Fe(II), Co(II), Ni(II), Cu(II), and Zn(II) chelate complexes have been prepared with Schiff base ligand derived from isatin and N-(1-(4-aminophenyl) ethylidene)-4-methylaniline. The obtained ligand and its chelate complexes were characterized by their elemental analyses, magnetic measurements, molar conductance, FTIR, as well as electronic spectra. The FTIR spectral data revealed that the Schiff base behave as a neutral bidentate ligand and was coordinated to metal ions *via* azomethen nitrogen atom and the carbonyl group oxygen of isatin molecule. From the available data, the suggested structures for the complexes have been proposed.

## Introduction

Schiff bases are forming some important kinds of organic compounds, since they contain various donor atoms and are able to change reactivity depending on the starting reagent. This class of compounds has a wide variety of biological properties(1-3). Isatin derivatives are reported to show many biological activities like antibacterial, antifungal, antiviral, anti-allergic and anti-inflammatory(4-8) activities. Isatin, due to its cis-α-dicarbonyle moiety, is a potentially good substrate for the synthesis of metal complexes either a lone or with other ligands(9-11). Schiff base complexes have an important and popular area of research due to their simple synthesis, versatility, and diverse range of applications(12) such as biological, pharmaceutical, clinical and analytical applications(13,14). To our knowledge, no reports can be found in the literature regarding the preparation and characterization of metal chelate complexes of Schiff base derived from isatin and N-(1-(4-aminophenyl) ethylidene)-4-methylaniline.

The aim of the our work is there for to the preparation and characterization of new Mn(II), Fe(II), Co(II), Ni(II), Cu(II), and Zn(II) chelate complexes of 3-(4-(1-(p-tolylimino) ethyl) phenylimino) indolin-2-one (HL).

## Materials and Methods

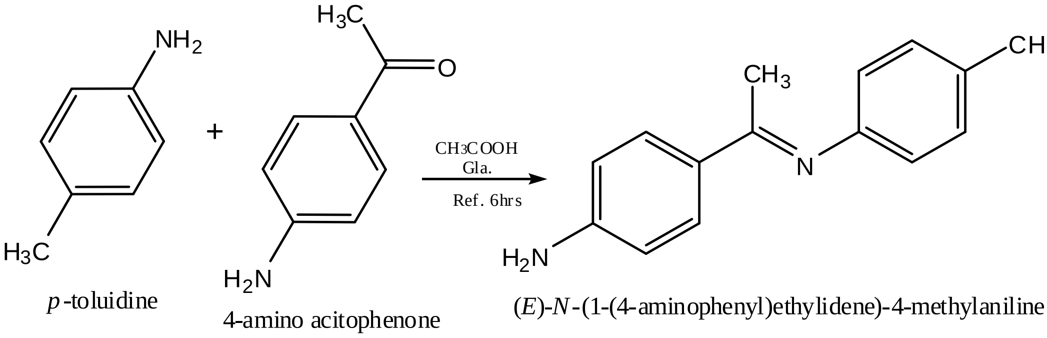
All employed chemicals were of analytical grad and purchased from (BDH, Aldrich and Merck) and used as resaved. The melting points of the compounds were taken in open capillary tube and are uncorrected by using a Stuart melting points SMP10. Elemental analysis C, H and N analysis were performed by using C.H.N.O.S EA 3000. The infrared spectra of the ligands and of their chelate complexes were recorded in KBr pellets in the 4000-400 $cm^{-1}$ region using a Shimadzu FTIR 8400S spectrophotometer. Electronic spectra were recorded on a Shimadzu UV-1700 spectrophotometer using DMSO as solvent in the 1000-200 nm. The metal contents of the complexes were measured using atomic absorption technique by Shimadzu AA-6300. The chloride ion was estimated by Mohr method (15). Magnetic susceptibilities were measured using Faraday method, balance magnetic (MSB-MKI) was used for this purpose and diamagnetic corrections for the ligand were calculated using Pascal’s constant. The molar conductance of the complexes was measured on digital conductivity meter Alpha -800 using ($10^{-3}$ M) solutions in DMSO.

### Preparation of Schiff base ligand (*p*-TEPIO =HL):

The preparation of the ligand 3-(4-(1-(p-tolylimino) ethyl) phenylimino) indolin-2-one include two steps the first one is a preparation of the Schiff base N-(1-(4-aminophenyl) ethylidene)-4-methylaniline which was resulted from condensation of p-toluidine and p-aminoacitophenone. The second step was preparation of the ligand by acid catalyzed condensation of the product of step one with Isatin in ethanol. The condensation proceeds, as usual, selectively on the carbonyl in position 3 in the Isatin ring(16).

### Preparation of Schiff base [N-(1-(4-aminophenyl)ethylidene)-4-methyl aniline]

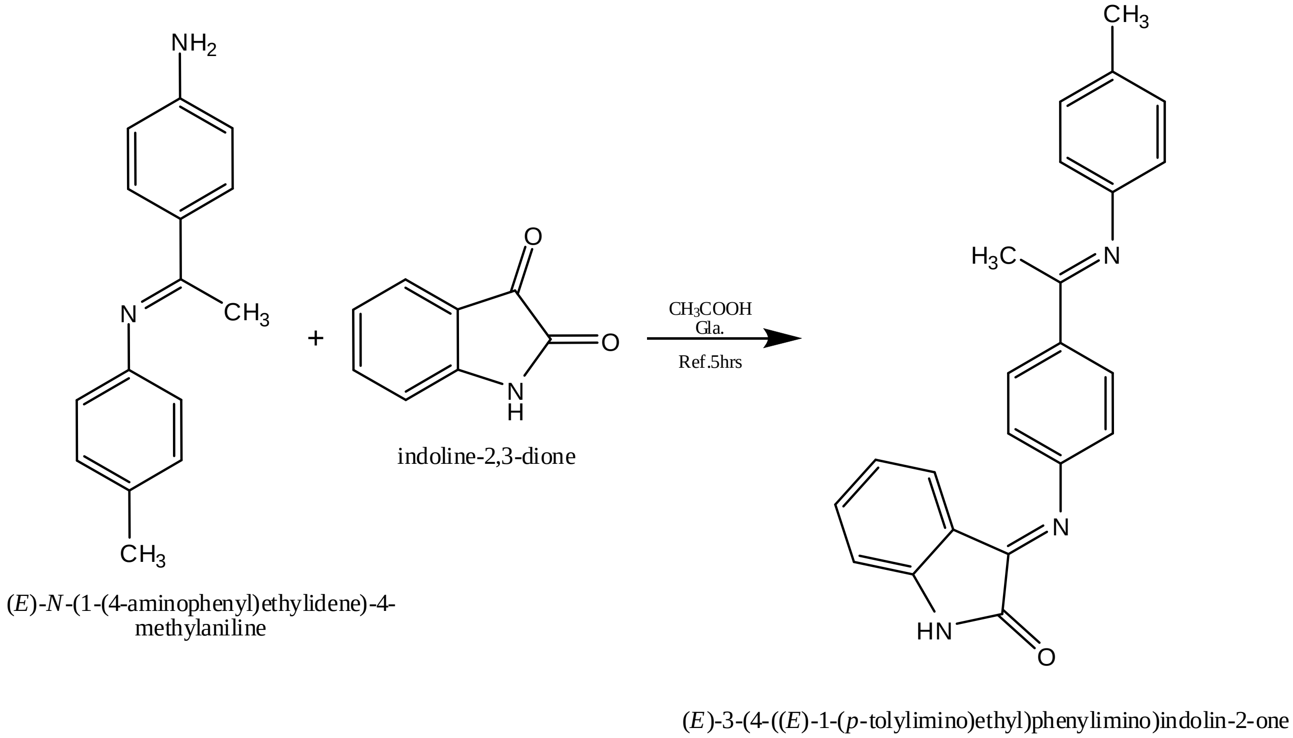
A hot ethanolic solution (20 mL) of *p*-toluidine (1.07 g, 0.01 mol) and an ethanolic solution (20 mL) of *p*-aminoacetophenone (1.35 g, 0.01 mol) were mixed slowly under constant stirring. This mixture was refluxed at (85±5 °C) for 6 hrs in the presence of a few drops of glacial acetic acid. On cooling, a white coloured compound precipitated out. This was filtered, washed with cold ethanol, and purified by recrystalization from hot ethanol. The preparation of this Schiff base is shown in Scheme.1.



**Scheme 1: Preparation of N-(1-(4-aminophenyl)ethylidene)-4-methylaniline**

### Preparation of the ligand 3-[4-{1-(*p*-tolylimino)ethylidene}phenylimino] indolin-2-one (*p*-TEPIO =HL)

A solution of isatin (1.47g, 0.01 mol) in ethanol (30 mL) was added drop wise to a refluxing solution of N-(1-(4-aminophenyl) ethylidene)-4-methylaniline (2.24g, 0.01 mol) in the same solvent (30 mL). Few drops of glacial acetic acid were added to the reaction mixture and were refluxed at (85±5 °C) for 5hrs. The progress of the reaction was monitored by TLC. After completion of the reaction the contents were poured in ice water the formed yellowish-orange precipitate was collected, filtered and finally washed with absolute ethanol (10 mL) and purified by recrystalization from hot ethanol and dried at ambient temperature. Scheme.2. shows the preparation of the ligand.



**Scheme 2: Preparation of the ligand (*p*-TEPIO =HL)**

## Preparation of metal complexes

The (1:2) chelate complexes of the metal and ligand are prepared by dissolving (0.3534g, 0.01 mol) Schiff base (HL) in 25 mL of hot ethanol. The corresponding hydrated metal chloride salts of Mn(II), Fe(II), Co(II), Ni(II), Cu(II), and anhydrous Zn(II) chloride of (0.005 mol) was dissolved in the same volume of the same solvent. The hot ethanol solution of metal salt was slowly added to the hot alcoholic solution of the ligand. The resulting mixture was refluxed for 1h. The coloured precipitate was separated by filtration and washed with distilled water, hot ethanol and air dried. The analytical data and some of the physical and chemical properties of the ligand and its chelate complexes were recorded in Table1.

## Results and Discussion

### General

The Schiff base ligand (HL) is yellowish-orange semicrystal, which is soluble in common organic solvents and partially soluble in water. The reaction of this ligand with the metal ions gives different color crystals. All complexes are quiet air-stable, insoluble in water, but soluble in most organic solvents. The elemental analysis of the ligand and its complexes which are recorded in table.1, are in a good agreement with those required by the proposed formula. In all cases (1:2) metal to ligand solid complexes are isolated.

### IR spectra

The IR spectra provide valuable information regarding the nature of the functional group attached to the metal ion(17). The IR spectrum of the free ligand (*p*-TEPIO =HL), Fig.1 showed a broad band that appears in 3176 $cm^{-1}$ is assigned to the stretching vibration of $\upsilon$(N–H) of the isatin moiety(16,18,19). The position of this band remained at nearly the same frequency in spectra of the metal complexes, suggesting the uncoordination of this group(5, 18). The band at 1742 $cm^{-1}$ in the spectrum of the free ligand, assigned to $\upsilon$(C=O) of isatin moiety(20,21), shifted towards lower values, around 1725-1707 $cm^{-1}$, in the complexes, indicating the coordination of the carbonyl oxygen atom of the isatin residue(22,23). The band appearing at 1610 $cm^{-1}$ in the free ligand, assignable to the $\nu$ (C=N) vibration mode(22,24), is shifted to lower wave numbers with a $\Delta\nu$ = (21-13) $cm^{-1}$ in the complexes spectra, this indicating the participation of azomethinic nitrogen atom in coordination(11,22). The appearance of two new bands in the region 491-478 and 597-588 $cm^{-1}$ in the spectra of the complexes, due to $\upsilon$(M–N) and $\upsilon$(M–O) stretching vibrations respectively(25), also confirmed the formation of metal complexes. The characteristic IR data are presented in Table.2. and Fig.2, showed the infrared spectrum of $[Co(LH)_2Cl_2]$.

### Electronic spectra

The electronic spectrum of ligand was displays two intense bands at (253 nm) and (413 nm) can be assigned to $n\rightarrow\pi^*$ and $\pi\rightarrow\pi^*$ respectively(18,26), while the manganese, iron, cobalt, nickel and copper chelate complexes appears a weak bands assigned to d→d transition. The manganese(II) complex shows two bands at (431 and 618 nm) due to $^6A_1g\rightarrow{}^4T_2g$ and $^6A_1g\rightarrow{}^4T_1g$ respectively. The spectrum resemble those reported for octahedral complexes(27). The iron(II) complex displays a band at (580 nm) attributed to the d→ d transition type $^5T_2g \rightarrow {}^5Eg$ which that supported with octahedral geometry studies(28). The solution spectrum of the cobalt(II) complex exhibited absorption bands at 970, 842 and 522 nm assigned to $^4T_1g\rightarrow{}^4T_2g(F)$, $^4T_1g\rightarrow{}^4A_2g(F)$ and $^4T_1g\rightarrow{}^4T_1g(P)$, respectively. The spectrum is consistent with the octahedral nature of this complex(29). The spectrum of nickel(II) complex shows two bands, observed in the visible region at (950 and 637 nm) which are suggesting the existence of $^3A_2g\rightarrow{}^3T_2g$ and $^3A_2g\rightarrow{}^3T_1g$ transitions with an octahedral spatial configuration(30). The electronic spectrum of copper(II) complex shows two bands at (817 and 529 nm) which may assigned to $^2B_1g\rightarrow{}^2Eg$

and ${}^2B_1g \rightarrow {}^2A_2g$ transitions in octahedral environment(29,30). The spectrum of zinc (II) exhibit high intense charge transfer transition in the visible region (524 nm) which are assigned to (MLCT) (29).

Therefore, it may be concluded that all the complexes formed by the reaction of (*p*-TEPIO =HL) with Mn(II), Fe(II), Co(II), Ni(II), Cu(II)and Zn(II) metal ions are octahedral geometry.

### Molar conductivity

The molar conductance for ($10^{-3}$ M) solutions of the complexes in DMSO solvent at (25 $C^o$) were in the ranges (8.15-13.77 $ohm^{-1}$ $cm^2$ $mol^{-1}$) (Table.3), which suggested the presence of nonconductive species(23) so the propose of existing of non-ionic complexes in the solvent used was made.

### Magnetic susceptibility measurement

The magnetic susceptibility measurements (Table.3) show that the complexes of Mn(II), Fe(II), Co(II), Ni(II) and Cu(II) are paramagnetic at ambient temperature. Where Mn(II) has a magnetic moment value of 5.91 B.M which is due to an octahedral geometry (31). The magnetic moment value for Fe(II) complex is 4.92 B.M this value is characteristic of octahedral geometry for this complex (32). Ni (II) and Co (II) complexes have magnetic moment values of 3.88 and 3.1 B.M. respectively. These values are expected for octahedral geometry (16, 33) of Ni (II) and Co (II) complexes at room temperature. The magnetic moment value of the Cu (II) complex is 1.73 that is suggests octahedral geometry(32). The Zn(II) complexes were found to be diamagnetic, as expected for $d^{10}$ configuration(29).

## Conclusion

From the elemental analysis, molar conductivity, IR spectral data it was possible to determine the type of coordination of our ligand with the metal ion complexes mentioned. In all above complexes, only two positions of the ligand is coordinated to the metal ion resulting mononuclear complexes. The ligand coordinates essentially through the carbonyl oxygen of the isatin fragment and the nitrogen atom of the azomethine group to give five membered rings. According to these results the structural formula of prepared complexes may be proposed in (Fig.5).

**Table 1: Some physical and analytical data of prepared compounds.**

| Compounds | M.P. ($C^{\circ}$) | Color | Yield (%) | M. wt (g/mol) | M. formela | C (cal.) | H (cal.) | N (cal.) | M (cal.) |
|---|---|---|---|---|---|---|---|---|---|
| HL | 231-233 | Yellowish -orange | 88.74 | 353.42 | $C_{23}H_{19}N_3O$ | 77.92<br>(78.16) | 5.36<br>(5.42) | 11.81<br>(11.89) | -<br>- |
| $[Mn(HL)_2Cl_2]$ | >300 | Brown | 61.25 | 832.68 | $[Mn(C_{23}H_{19}N_3O)_2Cl_2]$ | 66.22<br>(66.32) | 4.51<br>(4.60) | 9.89<br>(10.09) | 5.89<br>(6.60) |
| $[Fe(HL)_2Cl_2]$ | >300 | Deep brown | 65.48 | 869.04 | $[Fe(C_{23}H_{19}N_3O)_2Cl_2]$ | 63.29<br>(63.58) | 4.29<br>(4.41) | 9.78<br>(9.97) | 6.21<br>(6.43) |
| $[Co(HL)_2Cl_2]$ | >300 | Brown | 71.58 | 836.67 | $[Co(C_{23}H_{19}N_3O)_2Cl_2]$ | 65.87<br>(66.03) | 4.40<br>(4.58) | 9.93<br>(10.04) | 6.93<br>(7.04) |
| $[Ni(HL)_2Cl_2]$ | >300 | Lightened brown | 63.59 | 836.43 | $[Ni(C_{23}H_{19}N_3O)_2Cl_2]$ | 65.91<br>(66.05) | 4.49<br>(4.58) | 10.18<br>(10.05) | 6.89<br>(7.02) |
| $[Cu(HL)_2Cl_2]$ | 256-258 | Brown | 68.22 | 841.28 | $[Cu(C_{23}H_{19}N_3O)_2Cl_2]$ | 66.42<br>(66.67) | 4.43<br>(4.55) | 9.77<br>(9.99) | 7.31<br>(7.55) |
| $[Zn(HL)_2Cl_2]$ | 159-161 | Yellow | 77.26 | 843.15 | $[Zn(C_{23}H_{19}N_3O)_2Cl_2]$ | 66.35<br>(66.53) | 4.48<br>(4.54) | 9.76<br>(9.97) | 7.59<br>(7.76) |

**Table 2: Infrared spectra of the preparation compounds ($cm^{-1}$)**

| Compounds | $\nu_{(N-H)}$ | $\nu_{(C=O)}$ | $\nu_{(C=N)}$ | $\nu_{(M-N)}$ | $\nu_{(M-O)}$ |
|---|---|---|---|---|---|
| HL | 3176 | 1742 | 1610 | - | - |
| $[Mn(HL)_2Cl_2]$ | 3173 | 1700 | 1593 | 478 | 588 |
| $[Fe(HL)_2Cl_2]$ | 3175 | 1699 | 1591 | 478 | 591 |
| $[Co(HL)_2Cl_2]$ | 3173 | 1711 | 1589 | 480 | 597 |
| $[Ni(HL)_2Cl_2]$ | 3175 | 1707 | 1590 | 482 | 598 |
| $[Cu(HL)_2Cl_2]$ | 3178 | 1725 | 1593 | 486 | 594 |
| $[Zn(HL)_2Cl_2]$ | 3172 | 1727 | 1589 | 491 | 596 |

**Table 3: Electronic spectral data, magnetic measurements and molar conductivity of the ligand and its complexes**

| Compounds | $\lambda_{max}$ (nm) | | | Magnetic Measurements (μB) | $\Omega_M$ $ohm^{-1}.cm^2.mol^{-1}$ |
|---|---|---|---|---|---|
| | d–d | C–T | n→π * Azomethine | | |
| HL | - | - | 413 | - | - |
| $[Mn(HL)_2Cl_2]$ | 618, 431 | 413 | 390 | 5.91 | 9.19 |
| $[Fe(HL)_2Cl_2]$ | 580 | 420 | 388 | 4.92 | 8.15 |
| $[Co(HL)_2Cl_2]$ | 970, 842 & 522 | 417 | 383 | 3.88 | 10.32 |
| $[Ni(HL)_2Cl_2]$ | 950, 637 | 425 | 395 | 3.1 | 12.05 |
| $[Cu(HL)_2Cl_2]$ | 817, 529 | 419 | 389 | 1.73 | 11.28 |
| $[Zn(HL)_2Cl_2]$ | - | 524 | 388 | Dia. | 13.77 |

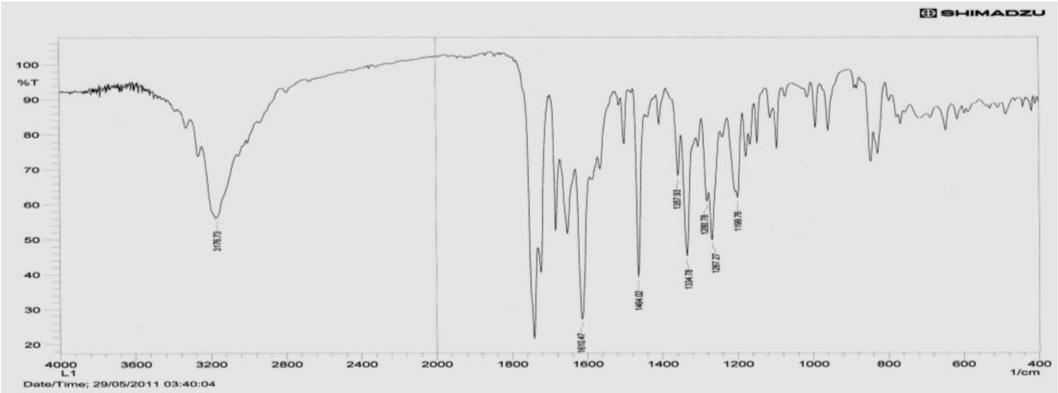


Fig.1: The FTIR spectrum of the Schiff base ligand (HL).

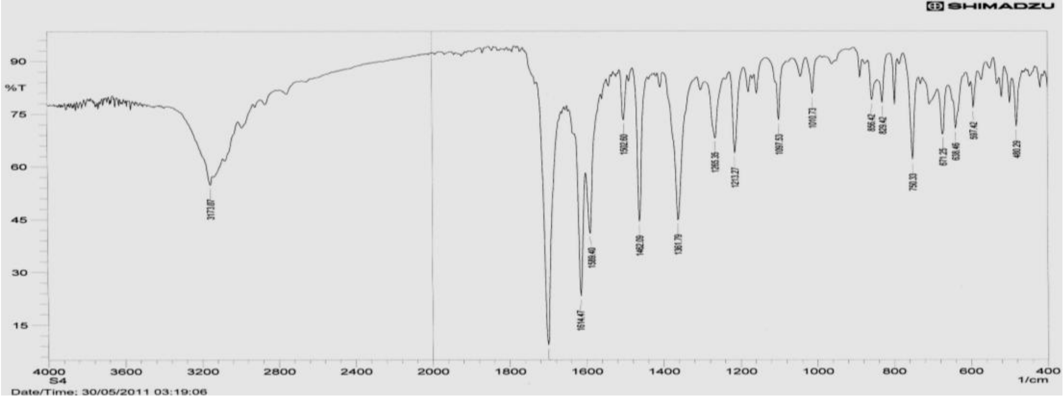


Fig.2: The FTIR spectrum of the $[Co(HL)_2Cl_2]$ complex.

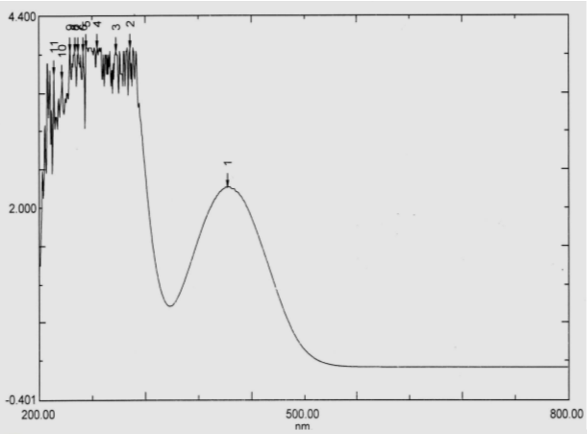


**Fig.3: The Uv-Vis. spectrum of the Schiff base ligand (HL).**

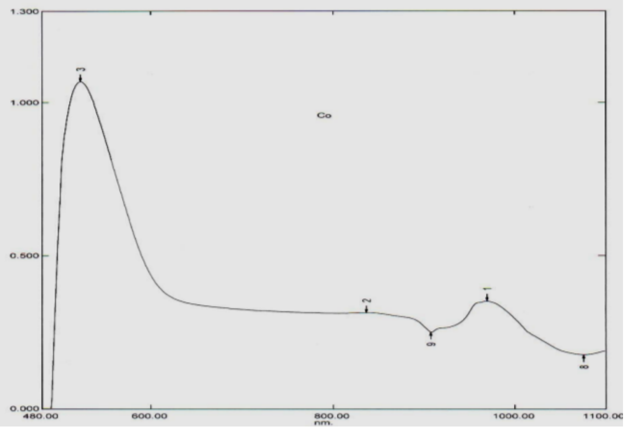
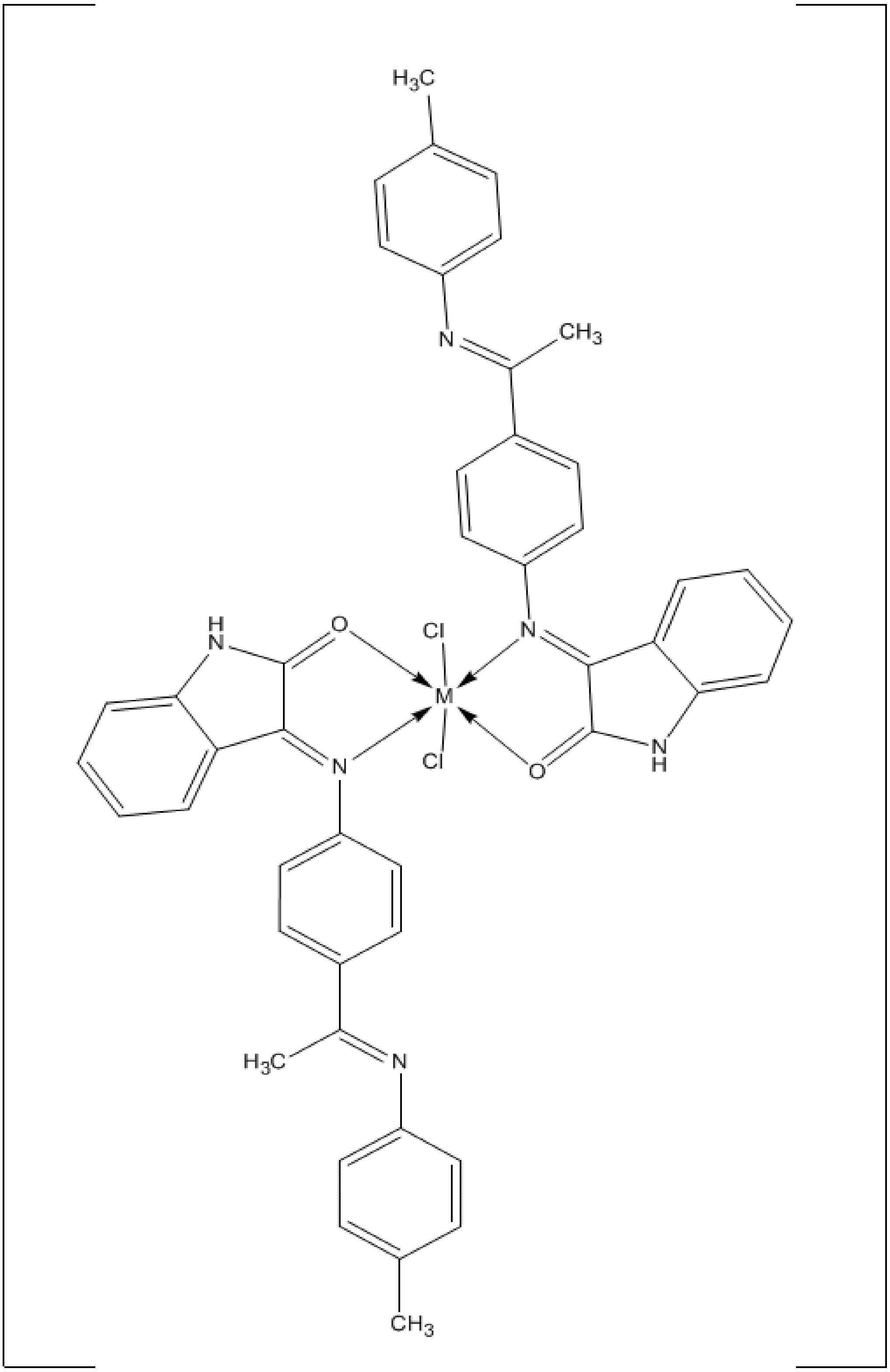


**Fig.4: The Uv-Vis. spectrum of the $[Co(HL)_2Cl_2]$ complex.**



M= $Mn^{2+}$,$Fe^{2+}$, $Co^{2+}$, $Ni^{2+}$, $Cu^{2+}$ and $Zn^{2+}$

**Fig.5: The stereo shape of the complexes.**

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