Synthesis, Structural and Spectroscopic Study of Complexes of Tetradentate Schiff Base Derived from Malonyldihydrazide with Cr(III), Mn(II), Co(II), Cu(II) and Zn(II) Ions

Abstract—This paper describes the synthesis of a new derivative dihydro-3H-indol-3-ylidene propane dihydrazide [L], which have been obtained from the reaction between malonyl di-hydrazide and an ethanolic solution of isatin. Tetradentate macrocyclic ligand [L] was characterized by (C,H,N), (FT-IR), (UV-Vis), and 1H NMR. A series of the used metal complexes have been synthesized by the prepared ligand [L] reaction with some transition metal ions. The isolated complexes were Cr(III), Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) also characterized using different techniques such as (FT-IR), and (UV-Vis) spectroscopies, (C,H,N), flamm atomic absorption in addition to the magnetic susceptibility and molar conductivity measurement. Al complexes were octahedral in geometry except for Zn (II) complex, which was tetrahedral.

Keywords: Tetradentate Ligand, Transition Metals, Spectroscopic Studies.

1. Introduction
Malonyldihydrazide and isatin compounds are important class of polydentate ligands in coordination chemistry and find extensive applications in different fields [1], also, the presence of imine group (−N=C=), which imports in elucidating the mechanism of transformation and rasemination reaction in biological system [2]. The transition metal complexes of hydrazone and its derivatives have been extensively examined due to its wide applications ranging from antimicrobial [3], anti-tubercular, carbonic anhydraz inhibitors, and anti-inflammatory [4]. The developments of the field of bioorganic chemistry have increased the interest in macrocyclic complexes containing oxygen and nitrogen atoms, since it has been recognized that many of these complexes may have some applications in industry and medicine [5,6]. The present paper describes some of the new Schiff base [L] applications, which is derived from isatin reaction with malonyldihydrazide. Ligand [L] provides four sites that are potential donor and forms complexes with Cr(III), Mn(II), Co(II), Ni(II), Cu(II), and Zn(II). The ligand [L] and its complexes have been fully characterized.

2. Experimental
All the chemical were used as received (from Fluka and BDH) and they were of highest purity available by using metal chloride salts (CrCl₃,6H₂O, MnCl₂,4H₂O, CoCl₂,6H₂O, NiCl₂,2H₂O, CuCl₂,2H₂O and ZnCl₂).

I. Physical Measurement and Analysis
The carbon, hydrogen and nitrogen contents were determined on Cario-Érba 1108, C.H.N, elemental analyzer at Al-Bait University, Jorden. FT-IR spectra were recorded on Shimadzu FT-IR-8100 spectrometer at Al-Mustansiriya University. H-NMR spectra of the ligand [L] were measured in d₆-DMSO solvent using TMS as internal standard on Bruker 300 MHz at Al-Bait University, Jordan. The electronic spectra were measured using (UV-1650) PC Shimadzu spectrophotometer at 25°C. The results were recorded by using (10⁻³M) concentration of the complex in the absolute ethanol. Molar conduct -ivity (µs.cm⁻¹) of the complexes were recorded at 25°C for (1×10⁻³M) solution of the samples in DMF, using a coming conductivity meter (220). Magnetic susceptibility results were also obtained at 25°C at the solid state by applying Gouy balance at Al-Mustansiriyah University. Moreover, all melting points results were recorded on (Gallen Kamp melting apparatus), and left uncorrected.

I. Synthesis of Malonyldihydrazide
This compound was prepared according to the literature preparations [7-9].

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(0.01Mole, 1.32g) with isatin (0.02Mole, 2.9g). The mixture was then refluxed for 6hrs. Then filtered, washed with ethanol and dried under vacuum, (yield 75%).

3. Synthesis of Metal Complexes
Ethanol solution (20ml) of metal chloride CrCl₃.6H₂O (0.22g,1mmole), MnCl₂.4H₂O (0.197g,1mmole), CoCl₂.6H₂O(0.237g,1mmole), NiCl₂.6H₂O (0.237g, 1mmole), CuCl₂.2H₂O (0.170g,1mmole), and ZnCl₂ (0.136g, 1mmole) were added with stirring to an ethanol solution of ligand (0.390g, 1mmole) and refluxed on water bath temperature for 2hrs., then the crude solid complexes were separated by filtration under suction, washed for several times with hot ethanol to afford colored purified metal complexes.

3. Results and Discussion
The reaction of one mole of malonyldihydrazide and two moles of isatin afforded the tetradeptatemacroyclic ligand [L] in good yield, scheme 1. The suggested mechanism of the formation of the ligand (L)can be shown in scheme 2.

I. Physical properties and micro elemental analysis
The physical properties and micro elemental analysis of the prepared ligand and its metal complexes are summarized in Table 1. Results are quite related to the suggested structural formula.
II. $^1$H-NMR Spectrum

$^1$H-NMR spectrum of the ligand in DMSO-d$_6$ solution was mentioned in addition to all experimental possible assignments [10]. Proton NMR spectrum showed peaks at 10.810ppm (4H, NH-C=O) 6.910-7.951 (m, 8H, Ar-H), 4.137-4.161ppm (s, CH$_2$) [11]. The integration curves were used to calculate protons numbers and further details of the chemical shifts with their assignments can be found in Figure 1, which reveals the proposed structure of the ligand [L].

III. Infrared Spectra

The FT-IR provides very interesting information about the metal atom attached functional group nature. The free ligand [L] and its prepared metal complexes behave as a tetra dentate coordination with these ions of metal through the carbonyl oxygen and the nitrogen of azomethine group. FT-IR spectrum of free ligand band shows peculiar absorption at 1728cm$^{-1}$ for $\nu$C=O in addition to a strong band in the 1612cm$^{-1}$ region, due to $\nu$C=N which undergone to down shift in all spectra of metal complexes what indicates azomethine group nitrogen involvement in the coordination [12,13], as shown in Figure 2. Moreover, $(-$C=O) absorption negative shift gives a coordination significant investigation of the metal ions through carbonyl group oxygen atom [14]. Furthermore, the spectrum of each complex shows a weak to medium band in (530-619)cm$^{-1}$, and (420-474)cm$^{-1}$ regions for (uM-N) and (uM-O) bands, respectively [15,16]. The spectrum of each complex except for Zn(II) complex exhibits a broad absorption band in (3412-3435)cm$^{-1}$ that is attributed to ($\nu$O-H) of the coordinated water molecules and the coordinated water molecules O-H stretching [17,18]. Further details of the characteristic are shown in Figure 3 and Table 2.

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**Table 1:** Ligand (L) and its Metal Complexes Physical and Analytical Data

| Compound          | Mol. Formula | Colour       | M.P °C  | Yield | M % | C % | H % | N % | Elemental Analysis Found (Calc.) |
|-------------------|--------------|--------------|---------|-------|-----|-----|-----|-----|----------------------------------|
| C$_{19}$H$_{14}$N$_{6}$O$_{4}$ | 390          | Light Green  | 203-205 | 75    | -   | 58.35 | 3.32 | 21.52 |
| [CrL(H$_2$O)$_2$]Cl | 584.5        | Dark Green   | 235d*   | 70    | 9.022 | 39.22 | 3.21 | 14.13 |
| [MnL(H$_2$O)$_2$]Cl | 551.9        | Light Orange | 166-168 | 65    | 10.242 | 41.00 | 3.43 | 15.12 |
| [CoL(H$_2$O)$_2$]Cl | 555.9        | Brown        | 220d*   | 73    | 10.233 | 41.32 | 3.30 | 15.22 |
| [NiL(H$_2$O)$_2$]Cl | 555.7        | Orange       | 210d*   | 76    | 10.952 | 41.21 | 3.22 | 15.00 |
| [CuL(H$_2$O)$_2$]Cl | 560          | Brown        | 146-148 | 80    | 11.40 | 40.03 | 3.00 | 15.21 |
| [ZnL]Cl$_2$       | 526.4        | Light Green  | 190-192 | 76    | 12.424 | 40.41 | 3.18 | 14.55 |

**d*= decomposition
Figure 1: The $^1$H-NMR Spectrum of the Ligand (L)

Figure 2: FT-IR Spectrum of the Ligand [L]

Figure 3: FT-IR Spectrum of Cu(II) Complex

Table 2: The Stretching Vibration Significant Frequencies (cm$^{-1}$) Situated in (L) and its Metal Complexes FT-IR spectra

| Compound                  | $\nu$C=O | $\nu$C=N | $\nu$M-N | $\nu$M-O | $\nu$O-H |
|---------------------------|----------|----------|----------|----------|----------|
| C$_{19}$H$_{14}$N$_{6}$O$_{4}$ [L] | 1728     | -        | 1612     | -        | -        |
| [Cr L(H$_2$O)$_2$]Cl$_3$   | (1693-1622) | 1498     | 545      | 474      | 3412 br  |
| [MnL(H$_2$O)$_2$]Cl$_2$   | 1616     | 1506     | 619      | 447      | 3429 br  |
| [CoL(H$_2$O)$_2$]Cl$_2$   | 1680     | 1592     | 561      | 439      | 3435 br  |
| [NiL(H$_2$O)$_2$]Cl$_2$   | (1651-1616) | 1464     | 588      | 420      | 3419 br  |
| [CuL(H$_2$O)$_2$]Cl$_2$   | 1645     | 1590     | 530      | 430      | 3431 br  |
| [Zn L]Cl$_2$              | 1703     | 1502     | 619      | 470      | -        |

IV. Electronic Spectra, Magnetic Susceptibility Measurements

The electronic spectroscopy is available tool for coordination chemists to obtain important information regarding structure of complexes.

The free ligand solution in ethanol ($10^{-3}$M) concentration showed high intensity absorption at (280, 320)nm, which are assigned to $\pi\rightarrow\pi^*$, and $n\rightarrow\pi^*$ respectively[19], Table 3, Figure 4.

The dark green chromium (III) complex gave three absorption bands that appeared at (15151, 18181, and 21739) cm$^{-1}$ following octahedral field with [d$^3$] configuration. It was found that the effective magnetic moment at 25°Cequals (3.79 B.M) which reveals an octahedral stereo chemistry of ligand around Cr(III) metal ion[20]. The prepared light orange Mn(II) complex showed two band at (16666,27777) cm$^{-1}$ due to the transition $^6$A$_{2g}$$\rightarrow$$^4$T$_{2g}$, and $^6$A$_{2g}$$\rightarrow$$^4$E$_{g}$, $^4$A$_{2g}$ respectively, which associated withoctahedral Mn(II) complex spin-forbidden transition of high spin (5.08 B.M) [21]. The brown solution of cobalt(II) complex in the ethanol presented well resolved band at 172413 cm$^{-1}$ and 20408 cm$^{-1}$ that maybe designated to $^4$T$_{2g}$$\rightarrow$$^4$T$_{2g}$, $^4$T$_{2g}$$\rightarrow$$^4$T$_{2g}$ transitions respectively, which confirm the high spin octahedral geometry for Co(II) ion [22].
Table 3: Ligand (L) and its Metal Complexes Electronic Spectrum, Molar Conductance in (DMF) and Magnetic Moments (B.M)

| Compound       | $\lambda_{\text{max}}$ nm | Bands Cm$^{-1}$ | Transitions       | $\mu_{\text{eff}}$ B.M | Molar Cond. $\mu$S.cm$^{-1}$ | Suggested Structure |
|----------------|---------------------------|-----------------|-------------------|------------------------|-------------------------------|---------------------|
| C$_{10}$H$_{12}$N$_{4}$O$_{4}$ [L] | 280 | 35714 | $\pi \to \pi^*$ | -                       | -                             | -                   |
|                | 320 | 31152 | $n \to \pi^*$ | -                       | -                             | -                   |
| [CrL(H$_2$O)$_2$]Cl$_3$ | 660 | 15151 | $^4A_{2g} \rightarrow ^3T_{2g}$ | 3.79                    | 223                           | Octahedral           |
|                | 550 | 18181 | $^4A_{2g} \rightarrow ^3T_{1g}$ | -                       | -                             | -                   |
|                | 460 | 21739 | $^4A_{2g} \rightarrow ^3T_{1g}(P)$ | -                       | -                             | -                   |
| [MnL(H$_2$O)$_2$]Cl$_2$ | 600 | 16666 | $^6A_{1g} \rightarrow ^3T_{1g}$ | 5.08                    | 155                           | Octahedral           |
|                | 360 | 17777 | $^6A_{1g} \rightarrow ^3E_{g}(G)$, $^4A_{1g}$ | -                       | -                             | -                   |
| [CoL(H$_2$O)$_2$]Cl$_2$ | 580 | 17241 | $^4T_{1g}(F) \rightarrow ^3T_{2g}(F)$ | 4.05                    | 132                           | Octahedral           |
|                | 490 | 20408 | $^4T_{1g}(F) \rightarrow ^3T_{1g}(P)$ | -                       | -                             | -                   |
|                | 340 | 29411 | $^4T_{1g}(F) \rightarrow ^3A_{1g}$ | -                       | -                             | -                   |
| [NiL(H$_2$O)$_2$]Cl$_2$ | 650 | 15384 | $^3A_{2g}(F) \rightarrow ^3T_{2g}(F)$ | 2.80                    | 150                           | Octahedral           |
|                | 530 | 18867 | $^3A_{2g}(F) \rightarrow ^3T_{1g}(F)$ | -                       | -                             | -                   |
|                | 430 | 23255 | $^3A_{2g}(F) \rightarrow ^3T_{1g}(P)$ | -                       | -                             | -                   |
| [CuL(H$_2$O)$_2$]Cl$_2$ | 620 | 16129 | $^3E_{g} \rightarrow ^3T_{2g}$ | 1.79                    | 140                           | Octahedral           |
| [ZnL]Cl$_2$ | 280 | 35714 | L.MCT | 0.0                     | 130                           | Tetrahedral          |
|                | 390 | 25641 | L.MCT | -                       | -                             | -                   |

Figure 4: UV-Vis Spectrum of the Ligand [L]

The high intensity band at (29411 cm$^{-1}$) has been allocated to the charge transfer from the metal to the ligand [23], which would support the high delocalization between the groups of C=O and C=N with the ion of Co(II) that maybe referred to high covalence factor of Co-O and Co-Nb and in complex [22], with (4.05 B.M) magnetic moment for Co(II) complex in the solid state. The Ni(II) complex electronic spectrum exhibited three bands at(15384,18867,23255)cm$^{-1}$, which have been attributed to $^3A_{2g}(F) \rightarrow ^3T_{2g}(F)$, $^3A_{2g}(F) \rightarrow ^3T_{1g}(F)$, and $^3A_{2g}(F) \rightarrow ^3T_{1g}(P)$ transitions, respectively, that indicates an octahedral geometry [23]. Ni(II) complex magnetic moment value equals of (2.80)B.M, which confirmed its high spin octahedral geometry [24], Figure 5, Table 3.

Copper (II) complex electronic spectra in ethanol demonstrated a band that is low energy and weak at (16129) cm$^{-1}$ with high intensity one at (26315)cm$^{-1}$, the visible region of the first band is typically expected for 10Dq, which corresponds to $^3E_{g} \rightarrow ^3T_{2g}$ transition [25]. Moreover, the magnetic moment (1.79B.M) corresponds to one unpaired electron, which offers possibility of an octahedral geometry [26]. On the other hand, Zinc(II) complex showed two high intensity absorption band at 35714cm$^{-1}$, and 25641 cm$^{-1}$ regions that are assigned to ligand field (L.MCT) transition [27], Table 3.

V. Molar Conductivity Measurements

The results of the molar conductance is (130-223)ohm$^{-1}$.cm$^{2}$.mol$^{-1}$ for all complexes, that were carried out in DMF solvent, which indicates that all of the study complexes are (1:2) electrolytic nature [28, 29] and Cr(III) is (1:3). These results suggest that all of the study complexes are electrolytes, thereby showing the chloride ions in counter ion, Table 3.
4. Conclusion

In this study, all spectral data demonstrated that the prepared Schiff base compound behaves as tetradentate ligand and binding to the metal ion through the oxygen of the carbonyl and nitrogen of azomethine group. Moreover, the analytical data demonstrated that the ratio of M:L is 1:1 in all the prepared complexes and consequently suggest a mononuclear structure for all complexes. Depending on the results that obtained from the spectral and elemental analysis in addition to the magnetic moment and the molar conductivity of the complexes in DMF solution, all complexes were electrolyte and have octahedral configuration except for Zn(II) complex, which has tetrahedral geometry, Figure 7.

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