

# Preparation and Characterization of Fluorone Ligand Complexes for $\text{Co}^{+2}$ , $\text{Mn}^{+2}$ , and $\text{Fe}^{+2}$ Metallic Ions



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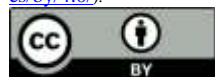
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## ABSTRACT

In this study, new complexes are produced by bonding of transition metal dinuclear chlorides ( $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ , and  $\text{FeCl}_2 \cdot 6\text{H}_2\text{O}$ ) with a polychelated ligand called 9-Phenyl-2,3,7-trihydroxy-6-fluorone, represented by (L), in a 1:2 (metal: ligand) ratio. Applying the appropriate chemical and physical methods, such as melting points, molar electrical conductivity, magnetic sensitivity, FT-IR, UV-Visible, and mass spectrometric studies, the produced complexes were characterized. All complexes are non-electrolytic, according to analyzes of molar electrical conductivity. All complexes have an octahedral structure, according to studies of magnetic sensitivity and Uv-Visible spectra. The mass spectrometry measurements showed the presence of the molar mass bands of the complex and the appearance of other bands resulting from the fragmentation of the complexes. For the metallic complexes spectra, FT-IR revealed a shift in the shape and intensity of the complexes bands at lower frequencies. The bonding of the metal to the oxygen atom can be observed by this shift in the bond stretch of the ligands. The size of the coordination between the ligand and the ions of the metallic elements is the cause. The measurement of the complexes' melting point is high, which suggests that the resultant complexes are quite stable.

## 1. INTRODUCTION

Since the discovery of  $\text{CoCl}_3 \cdot 6\text{NH}_3$  in 1789, chemists have been interested in coordinating chemical compounds. Due to its quick growth from the practical side of synthesizing complex coordination compounds and its involvement in understanding their structures, this branch of chemistry gained increased application in the last half of the 20th century[1]. These complexes played an important and growing role in various fields such as medicine[2]. Industry[3] and agriculture[4], which tested and confirmed their importance in modern life. When ligands, which can be either positive or negative ions or neutral molecules, come into immediate interaction with a metal, which is often a transition element, coordination compounds are produced[5,6]. The metal is known as the central ion or central atom when its d and f orbitals are partly filled with electrons.

The coordination ball is a term used to describe the metal and ligand group. The ligands donate a pair of electrons to the metal and are known as donor molecules, and they can be arranged in an unlimited number of possibilities to form the coordination complex. The charge of the metal ion and the charge of the ligands that surround it characterize the charge carried by the complex [7].

Many coordination complexes have been investigated for their potential as therapeutic compounds [8,9]. The effect of the metal has also been studied by including it in pharmaceuticals. where numerous studies and experiments were carried out to manage the levels of essential and dangerous metal ions in organisms by converting them into metal complexes. This necessitates understanding the common characteristics of the metal ion, which is peculiar due to its many oxidation states, and the collection of ligands that surround it in order to produce chelating complexes [10,11].

This research provided the path for several biologically significant application studies to be carried

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out [12]. Since coordination chemistry in particular and inorganic chemistry in general are employed as medications due to their vital function in inhibiting the growth of bacteria, biochemistry essentially informs us through these fields of chemistry. The compound 9-Phenyl-2,3,7-trihydroxy-6-fluorone used as a ligand is a xanthine dye. At 95 °C in an aqueous alcohol solution, Liebermann and Lindenbaum (1904) condensed hydroxy hydroquinone with benzaldehyde and acetaldehyde to produce dark red products. Both were known as 9-Phenyl-2,3,7-trihydroxy-6-fluorone(I) and 9-methyl-2,3,7-trihydroxy-6-fluorone (II) at the time as Figure (1). The color index did not list it at the time and it was not utilized as a textile dye[10]

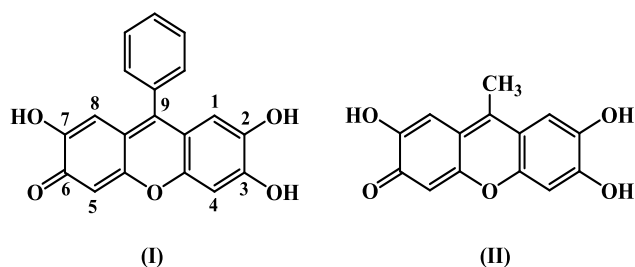


Fig. 1. 9-Phenyl-2,3,7-trihydroxy-6-fluorone (I) and the other 9-methyl 2,3,7-trihydroxy-6-fluorone (II)

The derivatives of 2,3,7-trihydroxy-6-fluorone belong to the fluorescein group, which is the second group of hydroxyxanthene dyes. In addition to the formation of colored claws of the elemental ions, these detectors are the most often utilized in spectrophotometric studies to identify the ions of the elements. In titration measurements of complexes, it is frequently employed to visually represent the equivalency point [14].

## 2. MATERIALS AND METHODS

In accordance with the manufacturer and country of origin, materials and apparatus were employed during our research. The chlorides of the aqueous iron, cobalt and manganese elements included Sigma-Aldrich USA, the ligand 9-Phenyl-2,3,7-trihydroxy-6-fluorone (MERCK-Germany), ethanol, Sigma-Aldrich USA, dimethyl sulfoxide (USA). As for the equipment used, it included an infrared spectrometer (FT-IR) Bruker Germany, a magnetic susceptibility device, a molar electrical conductivity meter (Folk) Germany, a melting point apparatus (Bicote) England, Molar mass spectrometry (GLC-mass QP SO A), UV-vis spectroscopy (Jenway USA), Alfa electric heater (Hs-860) Iran, USA MettlerToledo Sensitive balance.

## Complex preparation

Cobalt complex was prepared by adding 0.320g (0.1mol) of the ligand in 10ml of ethanol with 0.2mol (0.474g) of aqueous cobalt chloride. This synthesized the complex  $[Co_2(L)Cl_2 \cdot 7H_2O]$  The mixture was added to a round flask with a 100 ml capacity. In the reflux process, the mixture undergoes heating for two hours at 50 °C. Once a precipitate formed, the reaction mixture was cooled. Using ethanol, the precipitate was filtered, dried, and crystallized. It will produce a light brown precipitate, and following purification, 70% of the resultant complex was present [13]. As indicated in Table (1), other complexes with comparable characteristics were produced using the same technique, but with a different metal salt each time. When measuring the melting point of the complexes, it was higher than 300 °C, which is higher than the melting point of the free ligand, which was between (297-298 °C) as shown in Table (1).

Table 1. complexes with a different metal salt

No.	suggested formula	complex colour	Material used	Material weight (gm)	Melting point	Percentage %
1	9-Phenyl-2,3,7-trihydroxy-6-fluorone	reddish orange	Ligand $C_{19}H_{12}O_5$	0.320g	297-298□	99
2	$[Co_2(L)Cl_2 \cdot 7H_2O]$	light brown	$CoCl_2 \cdot 6H_2O$	0.474g	>300	70
3	$[Fe_2(L)Cl_2 \cdot 7H_2O]$	Dark brown	$FeCl_2 \cdot 6H_2O$	0.470	=	79
4	$[Mn_2(L)Cl_2 \cdot 7H_2O]$	coffee	$MnCl_2 \cdot 4H_2O$	0.0392	=	81

## 3. RESULTS AND DISCUSSION

### Spectroscopic studies

#### 1- Infrared spectroscopy:

In terms of the wave number between (400-4000  $cm^{-1}$ ), spectral measurements were carried out using KBr tablets in its solid state by mixing it with potassium

bromide in the form of tablets. By examining their infrared spectra and comparing them to the spectrum of the ligand being employed, the produced complexes were characterized[15].

Changes in the intensity and form of the bands, as seen in Table (2), were used to detect the shifting of certain bands and generate of other bands. The free ligand spectrum showed a broad absorption band at (3456.44  $\text{cm}^{-1}$ ) belonging to the phenol ligand ( $\nu\text{OH}$ ) group. While this band disappeared in the prepared iron, manganese and cobalt complexes. Which indicates its participation in coordination with the metal ion. The absorbance band ( $\nu\text{C=O}$ ) appeared at the range (1639  $\text{cm}^{-1}$ ) in the ligands, while it shifted to a lower extent in the complexes due to the occurrence of coordination. In the case of the ligands, the stretch vibration's absorption band ( $\nu\text{C=C}$  ring.) was visible in the range of (1480 - 1620  $\text{cm}^{-1}$ ), however it varied in the case of the complexes.

The ( $\nu\text{C-O}$  ring.) vibration absorption bands appeared at the range (1203  $\text{cm}^{-1}$ ), while their ranges changed at the complexes as well. While it was observed that the spectrum of complexes appeared new absorption bands within the range (574-582) due to the association of the metal with the oxygen of the ligand, and these bands were not present in the spectrum of the free ligand. Additionally, in the complexes that were prepared, new absorption bands showed up in the (833.25-860.55  $\text{cm}^{-1}$ ) range, indicating the presence of coordination water. The occurrence of these changes in band and shape is a preliminary indication of the validity of the experimental method used to prepare these compounds [12], as shown by Table (2).

**Table 2.** FT-IR absorption band values for ligand and complexes

Comp. Symb.	$\nu\text{OH}$	$\nu\text{C=O}$ Ketone.	$\nu\text{C=C}$ ring		$\nu\text{C-O}$ ring	$\delta(\text{H}_2\text{O})$	M-O
L	3456.44	1639	1620	1480	1203	-	-
Cl.7H <sub>2</sub> O] [Co <sub>2</sub> (L)	-	1620	1585	1473	1222	833.25	582

Cl.7H <sub>2</sub> O] [Fe <sub>2</sub> (L)	-	1620	1593	1469	1234	860.25	574
Cl.7H <sub>2</sub> O] [Mn <sub>2</sub> (L)	-	1616	1593	1485	1207	833.25	582

**UV spectroscopy:** By measuring the apparent absorption in their electronic spectra, ultraviolet-visible spectra are crucial for characterizing complexes of transition elements. In fact, these complexes are colored a result of the presence of electron-partially filled d orbitals. To determine these complexes' shapes, the electronic spectra of the produced complexes are being studied. It also shows the type and degree of bonding between the central metal and the ligands related to it through electronic effects within the d-orbitals of the transition metal or within the molecular orbitals formed between it and the ligands. In general, the electronic spectra of complexes of transition elements include absorptions, which are different from the spectra of ligands.

Due to an electronic transition ( $\pi \rightarrow \pi^*$ ), the ligand depicted in Table (3). UV spectrum displayed two high-intensity absorption peaks, one at (266nm, 37593.9  $\text{cm}^{-1}$ ), and the other at (294nm, 34013.6  $\text{cm}^{-1}$ ). [16-17] It alludes to the electronic transition ( $n \rightarrow \pi^*$ ). Figure (7) depicts the ultraviolet-visible spectrum of the cobalt binary complex, which has two peaks in the ligand spectra at (256nm, 39062.5  $\text{cm}^{-1}$ ), and (309 nm, 32362.4  $\text{cm}^{-1}$ ). This peak, which has moved from where it was in the case, shows that coordination between the ligand and the metal is taking occurred. The presence of two peaks at (574 nm, 17421.6  $\text{cm}^{-1}$ ), and (694 nm, 14409.2  $\text{cm}^{-1}$ ), in the visible spectrum, is also confirmed by this. The transitions ( ${}^4\text{T}_{1g(p)} \rightarrow {}^4\text{T}_{1g(F)}$ ) ( ${}^4\text{A}_{2g(F)} \rightarrow {}^4\text{T}_{1g}$ ) that are particular to the metal itself (d-d) are responsible for these peaks. This finding suggests that the ligand-cobalt combination produced an octahedral structure.

Figure (8) depicts the ultraviolet-visible spectrum of the iron complex. It has three peaks, two of which are from the ligand spectrum and are located at (254 nm, 39370  $\text{cm}^{-1}$ ) and (301 nm, 33222.6  $\text{cm}^{-1}$ , respectively). The metal's (d-d) transitions also cause a peak to occur in the visible range, and the iron in question possesses binary valence or the d6 system. Based on this, the transition ( ${}^3\text{E}_{1g} \rightarrow {}^5\text{T}_{1g}$ ) is what causes the peak to arise

with a low absorbance at (396nm, 20161.3cm<sup>-1</sup>). Giving the octahedral geometry.

Because the 3d<sup>5</sup> orbital becomes (half filled), has the term symbol <sup>6</sup>S, and is a monomeric state that does not divide, the oxidation state of the binary manganese ion is thought to be among the most stable oxidation states. It also has the term symbol <sup>6</sup>A<sub>1g</sub>. Therefore, electronic transitions occur from the stable state <sup>6</sup>A<sub>1g</sub> to the excited states <sup>4</sup>G, <sup>4</sup>P, <sup>4</sup>D, <sup>4</sup>F which are prohibited according to Laporte's law and the law of spinning. On this basis, the electronic spectra of the manganese binary complex showed multiple peaks, as it showed two absorption peaks at (254nm, 39370cm<sup>-1</sup>) and (282nm, 35460.9cm<sup>-1</sup>). This evidence confirms the presence of coordination between the ligand and the metal ion. The origin of these peaks is related to the ligand's spectrum, with displacement and change in shape. As for the peaks that appeared, three weak peaks at (466nm, 21459.2cm<sup>-1</sup>), (498nm, 20080.3cm<sup>-1</sup>) and (536nm, 18656.7cm<sup>-1</sup>), the origin of these peaks is due to the d-d electronic transitions that are prohibited by the law of spin and Laporte's law type (<sup>4</sup>A<sub>1g</sub>, <sup>4</sup>E<sub>g(G)</sub> → <sup>6</sup>A<sub>1g</sub>), (<sup>4</sup>T<sub>2g(G)</sub> → <sup>6</sup>A<sub>1g</sub>), (<sup>4</sup>T<sub>1g(G)</sub> → <sup>6</sup>A<sub>1g</sub>) respectively. This is consistent with what was mentioned in the previous spectral studies of the manganese complex, which has an octahedral shape [18-21], as shown in Table (3).

**Table 3.** Electron spectra data for the ligand and its complexes

Compounds	$\lambda_{max}$ (nm)	$\nu$ (cm <sup>-1</sup> )	ABS	$\epsilon_{max}^{\xi}$ L.mol <sup>-1</sup> cm <sup>-1</sup>	Assignment	Suggested formula
Ligand	266 294	34013.6 37593.9	3.265 3.724	3265 3724	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	-
Mn-Complex	254 282 466 498 536	39370 35460.9 21459.2 20080.3 18656.7	0.883 0.643 0.786 0.931 0.968	883 643 786 931 968	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ <sup>4</sup> A <sub>1g</sub> <sup>4</sup> E <sub>g(G)</sub> → <sup>6</sup> A <sub>1g</sub> <sup>6</sup> A <sub>1g</sub> → <sup>4</sup> T <sub>2g</sub> <sup>6</sup> A <sub>1g</sub> → <sup>4</sup> T <sub>1g(G)</sub>	Octahedral
Fe-Complex	254 301 496	39370 33222.6 20161.3	0.905 0.446 0.297	905 446 297	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ <sup>5</sup> E <sub>1g</sub> → <sup>5</sup> T <sub>1g</sub>	Octahedral

Co-Complex	256	39062.5	0.222	222	$\pi \rightarrow \pi^*$	Octahedral
	309	32362.4	0.168	168	$n \rightarrow \pi^*$	
	574	17421.6	0.215	215	<sup>4</sup> T <sub>1g(P)</sub> → <sup>4</sup> T <sub>1g</sub>	
	694	14409.2	0.154	154	<sup>4</sup> A <sub>2g(F)</sub> → <sup>4</sup> T <sub>1g</sub>	

The values in Table 3 show that the absorption bands of the complexes shifted towards a higher wavelength (red shift), and this is because coordination with metals shifts the electronic structure and energy level of the ligand.

### Magnetic Susceptibility

At a temperature of 25 °C, the Faraday method was used to determine the magnetic sensitivity of the solid states. The oxidation state and electronic configuration of the transition metal atoms are inferred from measurements of the magnetic characteristics. Therefore, whether a compound has low-spin or high-spin is determined by the number of single electrons that make up a transition metal ion<sub>[22,23]</sub>. The magnetic moment for the manganese (II) complex has been measured to be equal to (5.86B.M), whereas the magnetic moment for the iron complex was measured to be (5.80B.M). All complexes have highly twisted octahedral geometries, which are compatible with the magnetic moment of cobalt, which was measured to be (3.96B.M). The lists of magnetic sensitivity values of atoms in organic molecules (Pascal's constants) were used [24], and the values of magnetic moments were fixed in Table (4).

**Table (4)** Values of the effective magnetic moment for prepared complexes

Complexes	$X_g \times 10^{-6}$ Gramic susceptibility	$X_M \times 10^{-6}$ Molar susceptibility	$X_p \times 10^{-6}$ Pascal's constant	$X_A \times 10^{-6}$ Atomic susceptibility	$\mu_{eff}$ B.M.	Suggested structure
[Mn <sub>2</sub> (L)Cl(H <sub>2</sub> O) <sub>7</sub> ]	18.09	1398.42	-433.04	1441.73	5.86	Oh
[Fe <sub>2</sub> (L)Cl(H <sub>2</sub> O) <sub>7</sub> ]	16.85	1370.17	-449.14	1415.08	5.80	Oh
[Co <sub>2</sub> (L)Cl(H <sub>2</sub> O) <sub>7</sub> ]	7.56	6147.48	-451.14	6598.62	3.96	Oh

### Molar conductivity of complexes

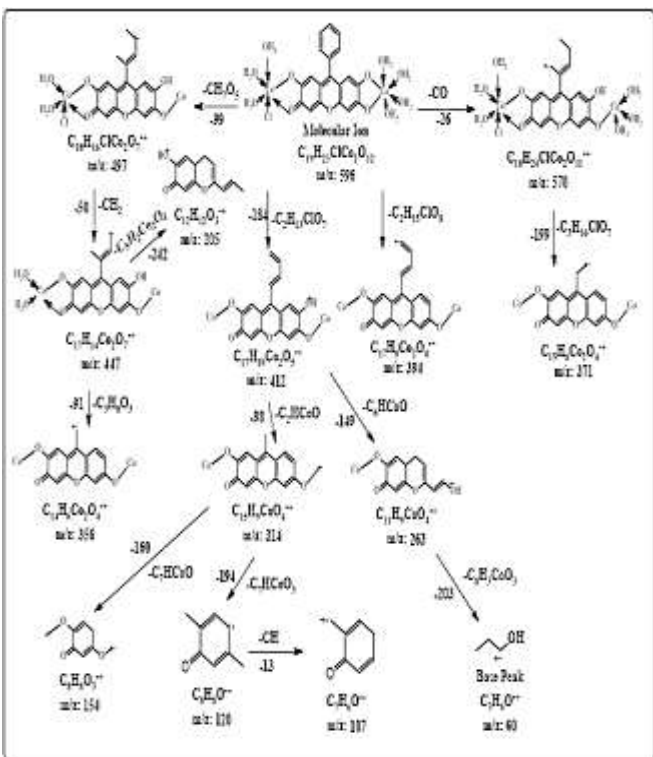
When dissolving the complexes prepared using a solvent (DMSO) and calculating the molar conductivity of the complexes, the measurements showed a non-electrolytic behavior (neutral) As it is consistent with the proposed structural formulas of the complexes. The values of molar conductivity ranged from (1.84-3.13), as shown in Table (5). [25-27]

**Table 5.** Molar conductivity of the prepared complexes

Compound	$\Lambda_m$ (S.cm <sup>2</sup> .mol <sup>-1</sup> )
[Mn <sub>2</sub> (L)Cl(H <sub>2</sub> O) <sub>7</sub> ]	1.84
[Fe <sub>2</sub> (L)Cl(H <sub>2</sub> O) <sub>7</sub> ]	5.40
[Co <sub>2</sub> (L)Cl(H <sub>2</sub> O) <sub>7</sub> ]	3.13

### Mass Spectrum Measurements:

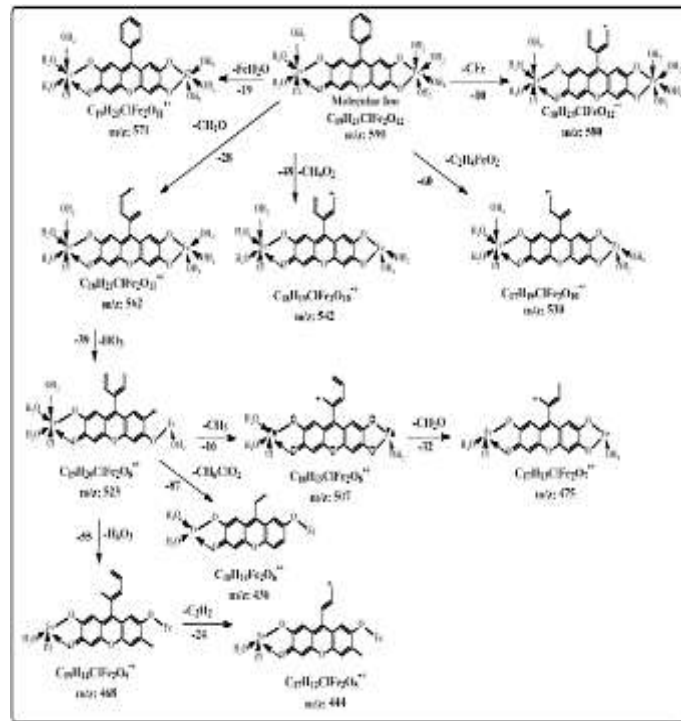
The prepared compounds' mass spectra revealed that the charge ratio m/z+ was compatible with their molar masses [28–30]. The cobalt complex's mass spectra revealed the charge m/z+ -to-mass (M.wt=596) ratio, which is related to the complex's molecular weight. As in scheme (1)



Scheme 1. Fragmentation pattern of Cobalt complex

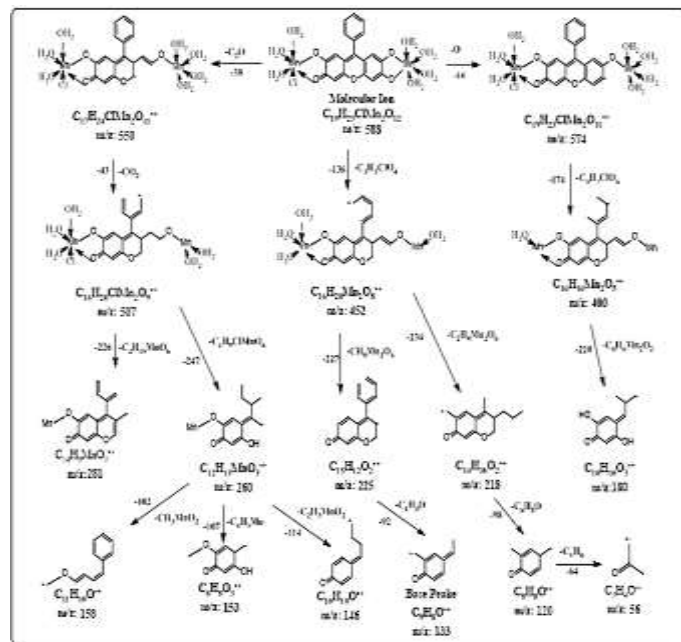
While the iron complex's mass spectrum is shown in Figure 11, the ratio of charge m / z + to mass (M.wt =

590), which is proportional to the molecular weight of the complex. The following Scheme shows the method of fragmentation of the mass spectrum of the complex



Scheme (2) Fragmentation pattern of Iron complex

, which shows the mass spectra of the manganese complex, shows the charge m/z + to mass (M.wt = 588) ratio, which is related to the molecular weight of the complex. The complex mass spectrum's mechanism of fragmentation is depicted in the following scheme (3).



Scheme (3) Fragmentation pattern of Manganese complex

## Conclusions

It was apparent from the spectrum characterization results and the suggested structural formulas that this ligand behaves as a polydentate (four-teethed) ligand with all the metal ions under study. Where the coordination of these metal ions with the ligand occurs through oxygen atoms.

The study of molar conductivity (which provides information about the oxidation state in which the metal ion is present in the formed complexes), the low value of conductivity proved that all complexes have no charge, i.e. neutral.

The study showed that complexes with ligands have high stability, and this is supported by their high melting points.

From the suggestion of the stereoscopic shapes of the solid complexes and based on the available techniques and measuring the magnetic sensitivity, it was found that all the complexes take the form of octahedral.

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## تحضير وتشخيص معقدات ليكند الفلورين لبعض الايونات الفلزية $Fe^{+2}$ و $Mn^{+2}$ و $Co^{+2}$

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### الخلاصة:

اتجهت الدراسة المقدمة الى تحضير معقدات جديدة من تفاعل كلوريدات العناصر الانتقالية ثنائية النواة ( $CoCl_2 \cdot 6H_2O, MnCl_2 \cdot 4H_2O, FeCl_2 \cdot 6H_2O$ ) مع الليكند متعدد المخالب الحاوي على ذرات اوكسجين واهبة والمسمى 9-فنايل-2،3،7-ثلاثي هيدروكسي-6-فلورون والممثل (L) وبنسبة (1:2) (فلز: ليكند) وتم تشخيص المعقدات المحضرة باستخدام الطرائق الكيمائية والفيزيائية المناسبة مثل التوصيلية الكهربائية المولارية والحساسية المغناطيسية واطياف الاشعة تحت الحمراء (FT-IR)، والاطياف الالكترونية (Uv.-Visivle) وقياسات طيف الكتلة ودرجات الانصهار فبينت قياسات التوصيلية الكهربائية المولارية بان جميع المعقدات غير الكتروليتية، بينت اطياف (Uv.-Visible) وقياسات الحساسية المغناطيسية ان جميع المعقدات تمتلك شكل ثنائي السطوح . بينما بينت قياسات طيف الكتلة بوجود حزم الكتلة المولية للمعقد وظهور الحزم الاخرى الناتجة من تشطي المعقدات وبينت اطياف (FT-IR) تغير شكل وشدة مواقع الحزم المعقدات الى ترددات اقل لطيف المعقدات الفلزية ويدل هذا الانزياح في مط الاصرة لليكاندات على ارتباط الفلز بذرة الاوكسجين ويعود السبب في حجم تناسق بين الليكاند مع ايونات العناصر الفلزية. وان قياس درجة انصهار المعقدات عالية وهذا يبين بان المعقدات الناتجة ذات استقراره عالية.

الكلمات المفتاحية: (العناصر الانتقالية)، (معقدات ثنائية النواة)، (9-فنايل-2،3،7-ثلاثي هيدروكسي-6-فلورون)، (اطياف الكتلة).