

# A series of M-M-heterometallic coordination polymers: syntheses structures properties ( $M=Ni^{+2}/M=Ag^{+1}, Cd^{+2}, Zn^{+2}$ )

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## ABSTRACT

*In this paper a series of new mono and heterometallic Ni(II) complexes of the general formula  $[Ni(3-Hophdte)_2]$ ,  $[Ni(Ag(3-Hophdte)_2)_2]$ ,  $NiLn(Ag(3-Hophdte)_2)_2$ ,  $NiM(3-Hophdte)_4$ ,  $[Ni(L)nM(3-Hophdte)_4]$ . 3-Hophdte=3-hydroxy phenyl dithiocarbamate. when  $n = 2$ , L quinoline, 4-picoline, when  $n = 3$ , L = ethylenediamine, 1,10 phenanthroline,  $M=Zn^{+2}, Cd^{+2}$ .*

*All the isolated complexes have been characterized by atomic absorption , elemental analysis, molar conductivity , Infrared , electronic absorption spectra and magnetic measurement. The conductivity measurements proved the non-electrolytic behaviour of all the compounds, susceptibility measurements and electronic spectra shows a tetrahedral geometry for complexes of the type  $[Ni(3-Hophdte)_2]$ ,  $[Ni(Ag(3-Hophdte)_2)_2]$ ,  $NiM(3-Hophdte)_4$  and octahedral geometry for complexes of the type  $NiLn(Ag(3-Hophdte)_2)_2$ ,  $[Ni(L)nM(3-Hophdte)_4]$ .*

**Keywords:** dithiocarbamate , Nickel(II), cadmium(II), zinc(II).

## البوليمرات التناسقية المتعددة الفلز الحقيقية: دراسة خصائص المركبات



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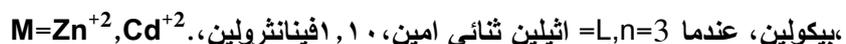
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### المخلص

في هذا البحث المعقدات المستخلصة الجديدة للنيل (II)Ni احادية ومتعددة الفلز ذات الصيغة العامة



، بيكولين، عندما  $L, n=3$  = اثيلين ثنائي امين، ١٠، ١٠، فينانثرولين،  $M=Zn^{+2}, Cd^{+2}$ ، عندما  $L, n=2$  = كوينولين، ٤ -



جميع المعقدات التي تم الحصول عليها شخضت بواسطة قياسات الامتصاص الذري، تحليل العناصر، التوصيلية

الكهربائية المولارية، اطياف الاشعة تحت الحمراء، الاطياف الالكترونية والقياسات المغناطيسية.

دلت قياسات التوصيلية على السلوك الغير الكتروليتي لجميع المعقدات ودلت القياسات المغناطيسية والاطياف

الالكترونية على الشكل الرباعي السطوح للمعقدات من نوع  $[Ni(3-Hophdtc)_2], [Ni(Ag(3-Hophdtc)_2)_2],$

$NiLn(Ag(3-Hophdtc)_2)_2, [Ni(L)_nM(3-Hophdtc)_4]$  وثمانى السطوح للمعقدات من نوع  $NiM(3-Hophdtc)_4]$

$Hophdtc)_4]$ .

كلمات دالة: dithiocarbamate , Nickel(II), cadmium(II), zinc(II)



## 1. INTRODUCTION

As a major branch of the coordination chemistry, heterometallic complexes have been given extensive attentions [1]. The simultaneous presences of heterometallic centers in the complexes could lead to not only fascinated structures but also novel properties, such as magnetism, catalysis, electrochemistry, optics, etc. [2]. There have been a number of investigations on heterometallic complexes and most were concentrated on 3d–3d' [3], 3d–4d [4], 3d–5d [5], 3d–4f [6], 4d–4f [7], etc. For example, Timco and Winpenny reported a series of Cr–3d' heterometallic complexes and found that there were anti-ferromagnetic exchanges in the molecules [8]. Niureported a series of d10–4f complexes and discussed the influences of d10 metal on NIR luminescence of 4f blocks [9]. A large number of heterometallic polymers bridged by CN– and SCN– groups were reported with ferromagnetic coupling and ferroelectricity in the most of them [10]. The magnetic researches of heterometallic complexes indicate that the introduction of heterometals develops the fashion and channels of electron transitions, on the basis of which the ferromagnetic materials may be designed [11]. But the reports about photoelectric properties are rare. As a high sensitive detection tool, surface photo voltage spectroscopy (SPS) has been applied widely to the detection of electron transitions on the surface and interface [12]. Dithiocarbamates (R<sub>2</sub>CNS<sub>2</sub>- or DTCs) are monoionic 1,1-dithio ligands and have been reviewed many times.

Dithiocarbamates are organosulphure compounds. Dithiocarbamates and their metal complexes have invited much research attention due to their diverse applications and interesting biological, structural, magnetic, electrochemical and thermal properties. [13]. They are used as accelerators in vulcanization, as high-pressure lubricants and as fungicides and pesticides. Also, dithiocarbamates are often used for the syntheses of transition metal complexes [14]. As the dithiocarbamates themselves, dithiocarbamate metal complexes have been used in agriculture for controlling insects and fungi [15], in the treatment of alcoholism, etc. The dithiocarbamate ligands reported in only the dithiocarboxy group as the ligand group. Dithiocarbamates have been found to act almost as uninegative bidentate ligands, coordinating through both sulphure atoms, and both tetra and hexa-coordinated complexes of many transition metal ions have been isolated [16]. We report in this article a new bidentate ligand dithiocarbamate of 3-amino phenol (3-HoPhdtc) was prepared.



## 2. EXPERIMENTAL (MATERIALS AND INSTRUMENTATION)

The reagents and solvents were of analytical grade. (1,10)- Phenanthroline, amine, carbon disulfide were purchased from Merck Company, 4-picoline, quinoline were purchased from BDH company. All melting points were determined on a Gallen Kamp and Electro thermal IA9300 Digital-Series (1998) apparatus and were uncorrected. The IR. – Spectra using (KBr disc) were recorded on Perkin – Elmer 590B Spectrophotometer. UV –Visible spectra were recorded using Shimadzu UV-160 spectrophotometer and DMF as solvent, the magnetic susceptibilities were measured by the Faraday method. The elemental analyses were detected on a PE-240 C Analyzer and TLASMA–II ICP instrument. Diamagnetic corrections were calculated using Pascal's constants. Conductivities were measured with a Conductivity Hand-Held Meter LF 330 (WTW GmbH) at 25 °C. Metal% contained was determined using atomic absorption elemental Analyzer Model 1106.

### 2.1. Synthesis of potassium 3-hydroxy phenyl dithiocarbamate (3-HOPhdtc)

This ligand is prepared by adding potassium hydroxide (5.61 gm, 0.1 mol) dissolved in water to (10.9 gm, 0.1 mol) from 3-amino phenol under stirring, followed by carbon disulfide (7.6 g, 0.01 mol). The mixture was stirred for 30 min. in the ice-salt bath, then allowed to reach room temperature and stirred for an additional one hour then dried under vacuum. A yellowish white precipitate formed ..

### 2.2. Synthesis of [Ni (3-HOPhdtc)<sub>2</sub>]

This complex was prepared by adding a solution of NiCl<sub>2</sub>.6H<sub>2</sub>O (0.2367 g, 0.001 mol) dropwise with constant stirring at room temperature. After 30 min of stirring, the green precipitate was filtered off and then dried by ether and in air. To a solution of potassium 3-hydroxy phenyl dithiocarbamate (0.446 g, 0.0002 mol) in water .

### 2.3. Synthesis of [Ni{Ag (3-HOPhdtc)<sub>2</sub>}]<sub>2</sub>

This complex was prepared by adding silver nitrate (0.169 gm, 0.001 mol) that dissolved in water to (0.446, 0.002 mol) of potassium 3-hydroxy phenyl dithiocarbamate which dissolved in water too. K<sub>2</sub>[Ag(3-HOPhdtc)<sub>2</sub>]<sub>2</sub> was formed. Then added to this solution (0.1183 gm, 0.005 mol) of NiCl<sub>2</sub>.6H<sub>2</sub>O that dissolved in water . After 20 min. of stirring , the precipitate was filtered off and then dried by ether and in air.

#### 2.4. Synthesis of $[\text{NiLn}]\{\text{Ag (3-HOPhdtc)}_2\}_2$

This complexes was prepared by adding nitrogen bases [L=qui.,4piq.(n=2),1,10ph,en(n=3) (0.002mol)] to the complex that prepared in the (2.3) . After 1 hour of sterring, the precipitate was filtered off and then dried by ether and in air.

#### 2.5. Synthesis of $[\text{NiCd (3-HOPhdtc)}_4]$

Amixture of  $\text{CdNO}_3$ (0.334gm,0.001mol)and potassium3-hydroxy phenyl dithiocarbamate (0.892gm,0.004mol)with30 min. sterring.  $\text{K}_2[\text{Cd(3-HOPhdtc)}_4]$ was formed. Then added to this solution(0.2367gm,0.001mol)of  $\text{NiCl}_2.6\text{H}_2\text{O}$  that dissolved in water .After 30 min. sterring, the precipitate was filtered off quiqlly to avoied containing $[\text{NiCd (3-HOPhdtc)}_2]$  and then dried by ether and in air.

#### 2.6. Synthesis of $[\text{NiLnCd (3-HOPhdtc)}_4]$

This complexes was prepared by adding nitrogen bases[L=qui.,4-piq. (n=2), 1,10ph, en (n=3) (0.002mol)]to the complex that prepared in the (2.5) . After 1 hour of sterring, the precipitate was filtered off and then dried by ether and in air.

#### 2.7. Synthesis of $[\text{NiZn (3-HOPhdtc)}_4]$

Amixture of  $\text{ZnCl}_2$ (0.1362gm,0.001mol)and potassium3-hydroxy phenyl dithiocarbamate (0.892gm,0.004mol). Then added to this solution (0.2367gm,0.001mol) of  $\text{NiCl}_2.6\text{H}_2\text{O}$  that dissolved in water .After 20 min.sterring, the precipitate was filtered off and then dried by ether and in air.

#### 2.8. Synthesis of $[\text{NiLnZn (3-HOPhdtc)}_4]$

This complexes was prepared by adding nitrogen bases[L=qui.,4-piq.(n=2),1,10ph,en(n=3) (0.002mol)]to the complex that prepared in the (2.8).After 1 hour of sterring, the resulting was filtered off and then dried by ether and in air.

### 3. RESULTS AND DISCUSSION

The new ligand was prepared by the reaction of 3-hydroxy phenyl amine with potassium hydroxide and then added carbon disulphide the complex (1) was prepared through direct reaction of the metal chloride  $\text{NiCl}_2.6\text{H}_2\text{O}$ ,other complexes were prepared through direct reaction of the metal chlorides  $\text{NiCl}_2.6\text{H}_2\text{O}$  and  $\text{ZnCl}_2$  or  $\text{CdNO}_3$ ,and nitrogen bases.

With the above ligand in (1:2) Molar ratio. The values of conductivity in dimethyl formamide solution of the complexes range from (0.02-8.37) $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$ , which are typical values for non-electrolyte type [17].

### 3.1. Infrared spectral studies

The important IR bands of the ligand and its complexes are listed in Table (3) . The stretching frequency of the  $\nu(\text{C} - \text{N})$  band for the dithiocarbamates was intermediate between the stretching frequencies associated with typical single- and double-bonded carbon and nitrogen atoms [18]. The  $\nu(\text{C} - \text{N})$  and  $\nu(\text{C} - \text{S})$  were observed in the ranges of (1506-1581) $\text{cm}^{-1}$  and ( 930-1022) $\text{cm}^{-1}$ . The presence of only one band in the later region reports the bidentate coordination of the dithio ligand [19].

IR spectra showed a new band at (403-420) $\text{cm}^{-1}$  which is the evidence for the coordination of metal to sulfur  $\nu(\text{M}-\text{S})$ , This behavior may be attributed to the electron-releasing of the amines, which forces high electron density towards the sulfur atoms. [20].

### 3.2. Electronic spectral studies

The UV-Visible spectra of the ligands and their complexes in 10<sup>-3</sup> M solution DMF were recorded; the results were listed in Table (2). The Ni(II) complex (1,2,7,12) shows two absorption bands in the range (10163-11295  $\text{cm}^{-1}$ ), (13888-1497  $\text{cm}^{-1}$ ) which were assigned to ( $3\text{T}_1(\text{F}) \rightarrow 3\text{A}_2(\text{F})$ ) and ( $3\text{T}_1(\text{F}) \rightarrow 3\text{T}_1(\text{P})$ ) transitions, respectively in an tetrahedral geometry [21], the complexes (3-6,8-11,13-16) show two absorption bands in the range (9708-12016  $\text{cm}^{-1}$ ) and (21325-25641 $\text{cm}^{-1}$ ), which were assigned to ( $3\text{A}_2\text{g}(\text{F}) \rightarrow 3\text{T}_1\text{g}(\text{F})$ ) and ( $3\text{A}_2\text{g}(\text{F}) \rightarrow 3\text{T}_1\text{g}(\text{P})$ ) transitions in an octahedral geometry [22].

### 3.3. Magnetic susceptibility measurements

The magnetic moments of the complexes Table (1). Were measured at (25 °c). The magnetic moments for Ni(II) complexes (1,2,7, and 12) are (3.0-4.0 B.M) suggest a tetrahedral geometry. Finally the magnetic moments values of the other complexes (3-6,8-11,13-16) were in the range (2.2-4.0 B.M. ) in an octahedral geometry [21].

**Table (1):** Analytical and some physical properties of the prepared complexes

No.	Compound	Color	m.p (°c)	Molar conductivity $\Omega^{-1}.cm^2.mol^{-1}$	%MetalFound (calcula)	%yield	$\mu_{eff}$ B.M
L	C7H6NOS2K	Light yellow	110*	----	---	72.2	---
1	[Ni(3-HoPHdte)2]	Light green	200	0.45	13.76(13.24)	70.4	3.5
2	[Ni{Ag(3-HoPHdte)2}2]	Orange	260	0.42	5.81(6.30)	33.7	3.71
3	[Ni(qui)3{Ag(3-HoPHdte)2}2]	Gray	268	3.47	4.62(5.08)	24.4	3.45
4	[Ni(4-pic)3{Ag(3-HoPHdte)2}2]	Gray	280	0.32	4.91(4.85)	25.1	4.0
5	[Ni(en)2{Ag(3-HoPHdte)2}2]	Gray	282	8.37	4.96(5.34)	5.9	2.2
6	[Ni(1,10phen)2{Ag(3-HoPHdte)2}2]	Gray	282	5.22	3.43 (2.58)	20.4	3.5
7	[NiZn(3-HoPHdte)4]	Yellowish green	210	0.62	6.83(7.02) 7.59(7.32)	80.2	3.0
8	[Ni(qui)3Zn(3-HoPHdte)4]	Yellowish green	210	0.02	5.25(5.62) 5.84(6.22)	61.7	4.0
9	[Ni(4-pic)3Zn(3-HoPHdte)4]	Yellowish green	210	5.0	5.61(6.08) 6.24(6.76)	59.3	3.5
10	[Ni(en)2Zn(3-HoPHdte)4]	Dark brown	179	2.9	5.68(6.13) 6.31(7.44)	29.0	3.28
11	[Ni(1,10phen)2Zn(3-HoPHdte)4]	Yellowish green	178	2.3	3.76(2.81) 4.18(3.88)	51.8	3.7
12	[NiCd(3-HoPHdte)4]	Light green	180	1.1	6.47 (6.80)	68.3	4.0
13	[Ni(qui)3cd(3-HoPHdte)4]	Yellowish green	180	3.3	5.04(4.18)	54.9	3.83
14	[Ni(4-pic)3Cd(3-HoPHdte)4]	Green	176	1.7	5.37(4.48)	79.6	2.78
15	[Ni(en)2Cd(3-HoPHdte)4]	Brown	190	5.0	5.43(5.18)	32.4	3.22
16	[Ni(1,10phen)2Cd(3-HoPHdte)4]	Yellowish green	205	5.7	3.65(2.77)	50.9	3.16

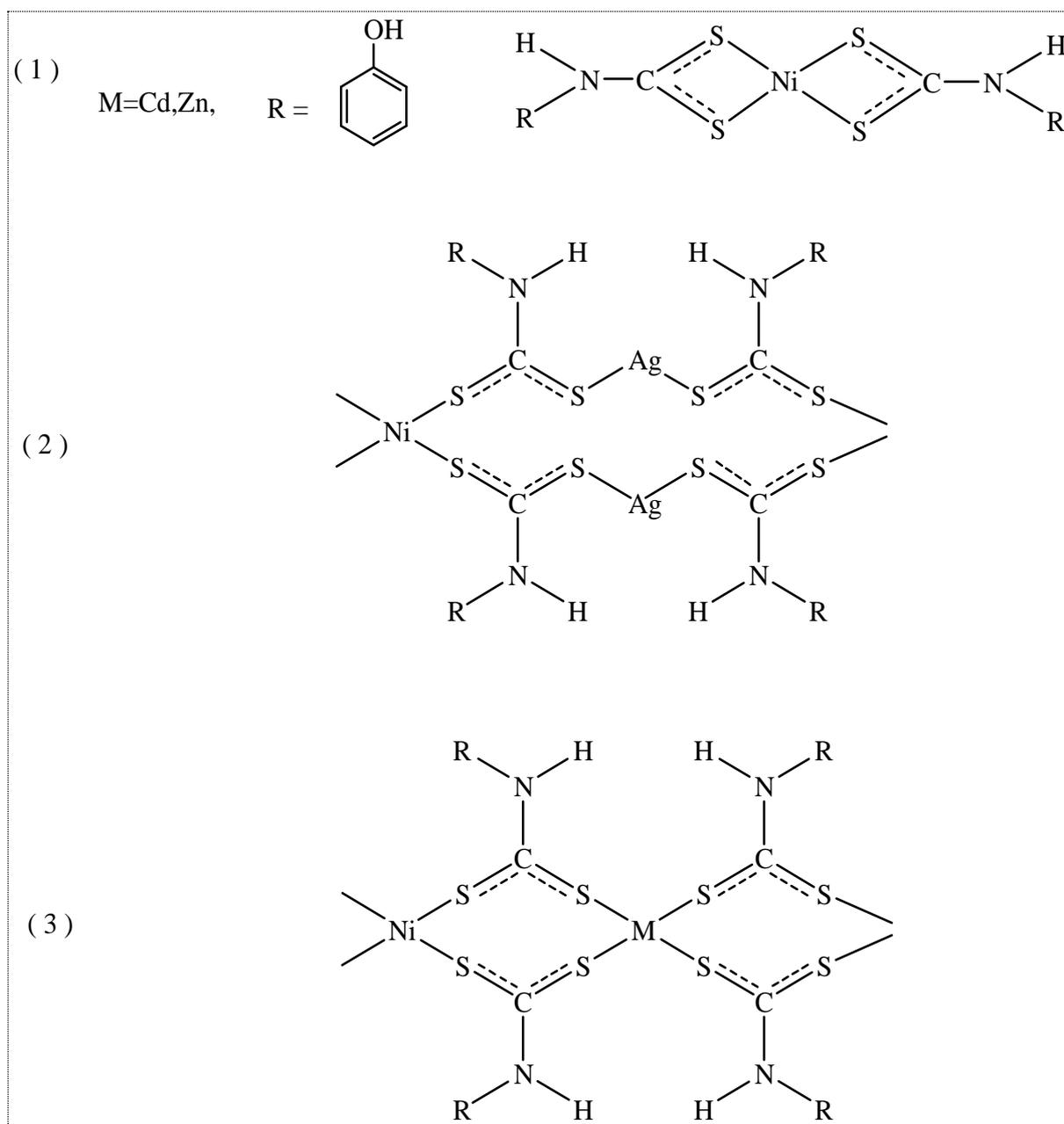
**Table (2): CHN measurements**

No.	Compound	C%	H%	N%
L	C7H6NOS2K	37.7(37.1)	2.7(3.4)	6.3(6.8)
1	[Ni(3-HoPHdtc)2]	39.4(40.2)	2.8(1.9)	6.6(6.3)
2	[Ni{Ag(3-HoPHdtc)2}2]	33.3(33.7)	2.4(2.7)	5.5(5.10)
3	[Ni(qui)3{Ag(3-HoPHdtc)2}2]	43.5(43.80)	2.1(2.5)	6.6(7.2)
4	[Ni(4-pic)3{Ag(3-HoPHdtc)2}2]	40.1(40.6)	3.2(3.5)	7.0(7.4)
5	[Ni(en)2{Ag(3-HoPHdtc)2}2]	34.4(34.8)	3.6(3.2)	11.8(11.3)
6	[Ni(1,10phen)2{Ag(3-HoPHdtc)2}2]	49.5(49.2)	3.1(3.4)	9.0(8.8)
7	[NiZn(3-HoPHdtc)4]	39.1(39.7)	2.8(2.5)	6.5(6.3)
8	[Ni(qui)3Zn(3-HoPHdtc)4]	49.4(48.7)	3.4(3.6)	7.5(7.9)
9	[Ni(4-pic)3Zn(3-HoPHdtc)4]	45.9(45.7)	3.6(3.3)	8.0(7.8)
10	[Ni(en)2Zn(3-HoPHdtc)4]	39.5(38.7)	4.1(4.6)	13.5(13.8)
11	[Ni(1,10phen)2Zn(3-HoPHdtc)4]	54.9(54.4)	3.4(3.5)	10.0(10.6)
12	[NiCd(3-HoPHdtc)4]	37.0(37.8)	2.7(2.4)	6.2(6.5)
13	[Ni(qui)3cd(3-HoPHdtc)4]	47.4(46.9)	3.3(3.5)	7.2(7.7)
14	[Ni(4-pic)3Cd(3-HoPHdtc)4]	43.9(43.4)	3.5(2.8)	7.7(7.3)
15	[Ni(en)2Cd(3-HoPHdtc)4]	37.7(36.8)	3.9(3.4)	13.0(12.6)
16	[Ni(1,10phen)2Cd(3-HoPHdtc)4]	53.1(53.6)	3.3(3.7)	9.7(9.3)

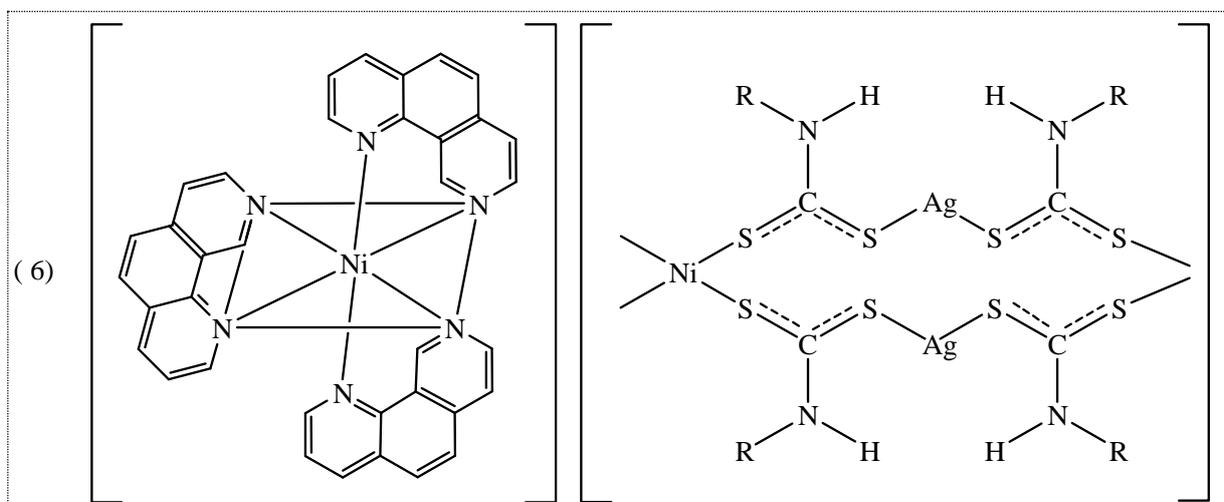
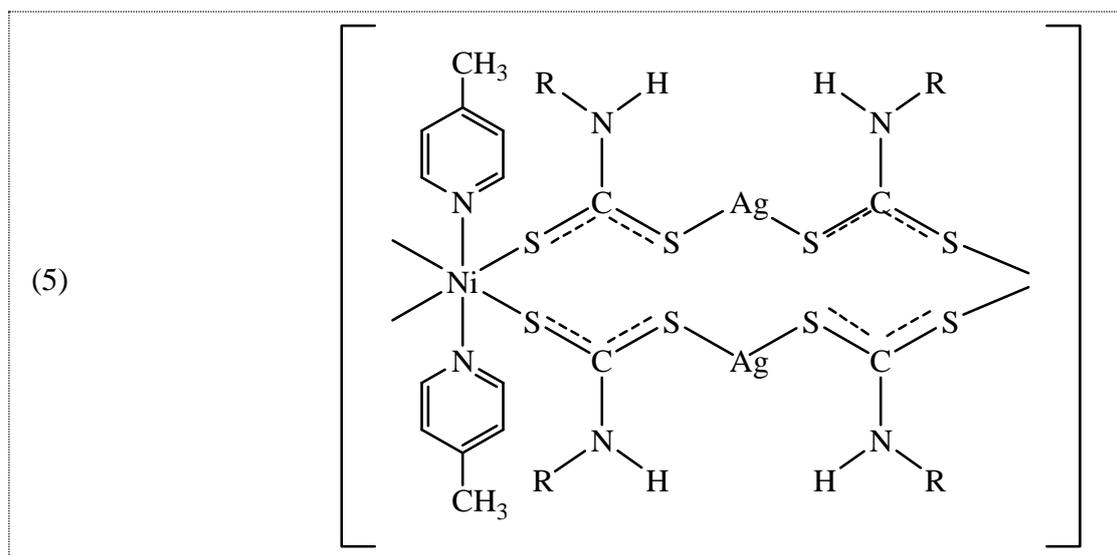
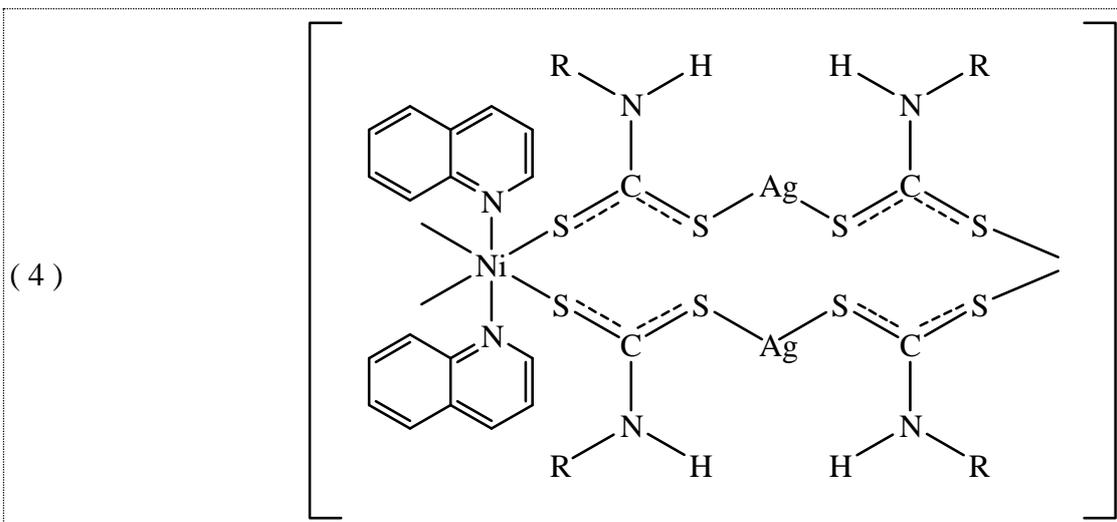
**Table (3):** IR bands (cm-1) and electronic spectral data of ligand and prepared complexes.

No	Compound	U-V bands (cm-1)	IR spectra		
			$\nu(\text{C}=\text{N})$	$\nu(\text{C}=\text{S})$	$\nu(\text{M}-\text{S})$
L	C7H6NOS2K	---	1518	1001	---
1	[Ni(3-HoPHdtc)2]	10166,1497,29585,31645	1581	930	420
2	[Ni{Ag(3-HoPHdtc)2}2]	10163,13888,29411	1581	930	410
3	[Ni(qui)3{Ag(3-HoPHdtc)2}2]	9727,24324,27777	1506	1020	411
4	[Ni(4-pic)3{Ag(3-HoPHdtc)2}2]	9708,23961,30864,28901	1506	1020	411
5	[Ni(en)2{Ag(3-HoPHdtc)2}2]	9708,25641,32258,39682	1506	1020	405
6	[Ni(1,10phen)2{Ag(3-HoPHdtc)2}2]	10575,23546,28409	1506	1020	405
7	[NiZn(3-HoPHdtc)4]	10893,14308,27472,31055	1581	930	418
8	[Ni(qui)3Zn(3-HoPHdtc)4]	12016,21843,31847,28571	1508	1022	403
9	[Ni(4-pic)3Zn(3-HoPHdtc)4]	11724,21325,30120	1508	1022	410
10	[Ni(en)2Zn(3-HoPHdtc)4]	11576,22348,29761,28089	1508	1022	407
11	[Ni(1,10phen)2Zn(3-HoPHdtc)4]	11069,23659,31055,28409	1508	1022	411
12	[NiCd(3-HoPHdtc)4]	11295,14450,27777,31446	1581	930	418
13	[Ni(qui)3cd(3-HoPHdtc)4]	13909,24589,33112,29585	1506	1020	409
14	[Ni(4-pic)3Cd(3-HoPHdtc)4]	12833,21932,29585,31446,	1541	1020	410
15	[Ni(en)2Cd(3-HoPHdtc)4]	9618,22459,29585	1506	1020	413
16	[Ni(1,10phen)2Cd(3-HoPHdtc)4]	13828,22932,32051	1506	1020	408

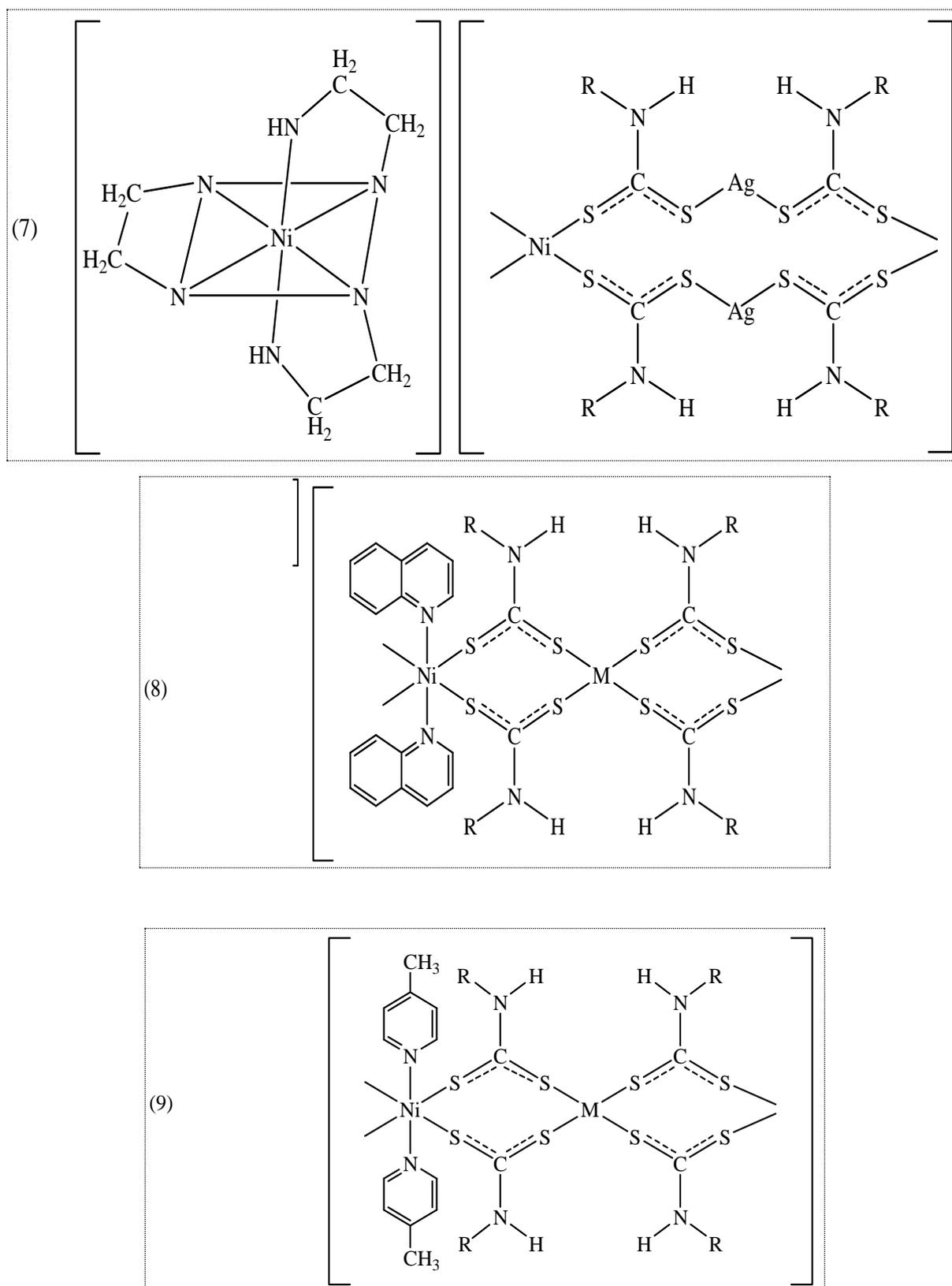
From the above discussion the following structures were suggested which indicated a four coordinate geometry around the metal ion with abidentate coordination ligand through two sulphur atoms as shown in Fig.(1-3) expected have a tetra hedral geometry as shown as Fig.(4-11) expected have octa hedral geometry.



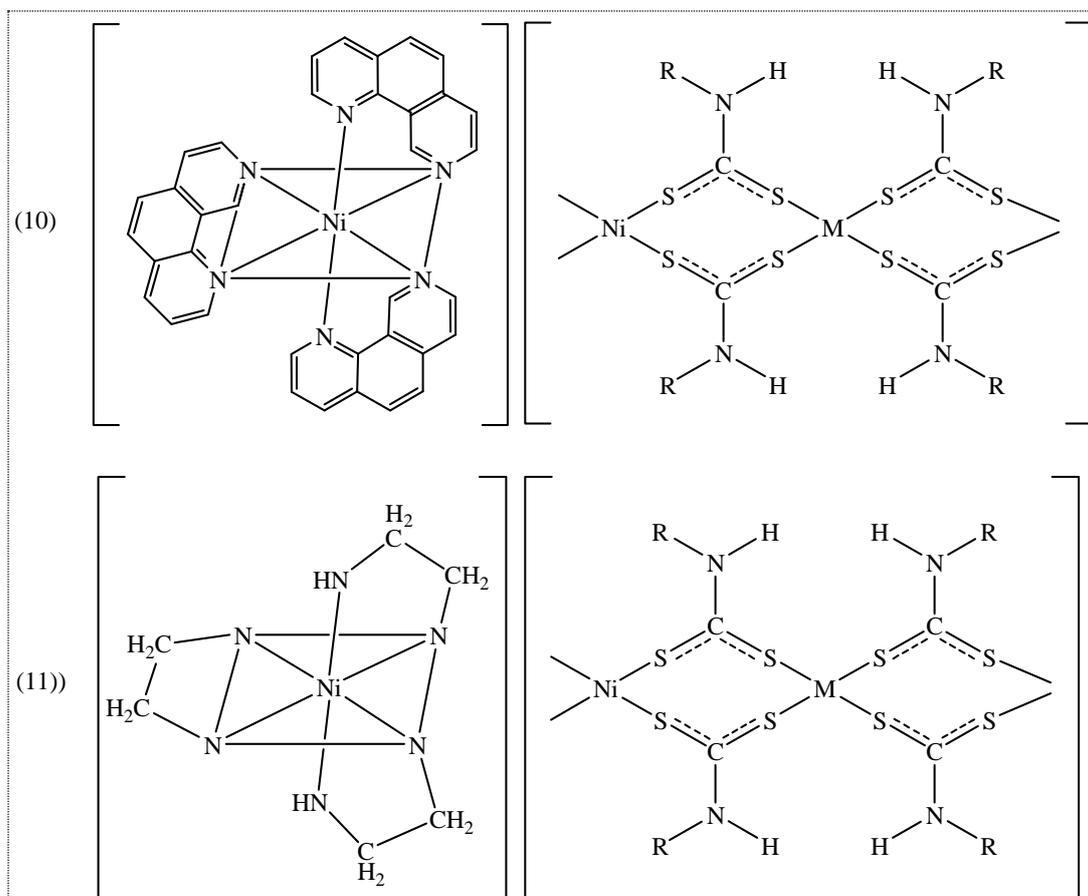
**Fig. (1):** a tetra hedral geometry around the metal ion( $Ni^{+2}$ ), **(2)** a tetra hedral geometry around the metal ion( $Ni^{+2}$ ) with ( $Ag^{+1}$ ), **(3)** a tetra hedral geometry around the metal ion( $Ni^{+2}$ ) and  $M=Cd^{+2}$  or  $Zn^{+2}$



**Fig. (4- 6):** octa hedral geometry around the metal ion( $Ni^{+2}$ )with ( $Ag^{+1}$ )



**Fig. (7):** octa hedral geometry around the metal ion( $\text{Ni}^{+2}$ )with ( $\text{Ag}^{+1}$ ), **(8,9)** octa hedral geometry around the metal ion( $\text{Ni}^{+2}$ )and  $\text{M}=\text{Zn}^{+2}$  or  $\text{Cd}^{+2}$



**Fig. (10, 11):** octa hedral geometry around the metal ion( $\text{Ni}^{+2}$ ), $\text{M}=\text{Cd}^{+2}$  or  $\text{Zn}^{+2}$

#### 4. CONCLUSIONS

The following structures were suggested which indicated:

- 1- A four coordinate geometry around the metal ion with abidentate coordination ligand through two sulphur atoms expected have atetra hedral geometry.
- 2- A six coordinate geometry around the metal ion with abidentate coordination ligand through two sulphur atoms expected have octa hedral geometry.
- 3- These compounds represent a class of versatile ligands, owing to the occurrence of resonance phenomena, which result in electron delocalization.
- 4- Their coordination chemistry with the transition metals commands much interest, on account of the rich diversity of the complexes.
- 5- Their numerous and potential applications in industry and agriculture.



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