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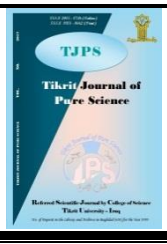

Synthesis and Characterization Complexes of Ni(II) that Contain Cyanoguanidine and Phosphines Ligands

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Abstract

The ligand of Cyanoguanidine showed an important coordination formula with Ni^{2+} to give a complex of tetrahedral geometrical shape $[Ni(CY)_2Cl_2]$ when the metal reacted with Cyanoguanidine of 1:2 ratio, and octahedral complexes of the types $[Ni(CY)_2(PPh_3)_2Cl_2]$, $[Ni(CY)_2)(PPh_3)_4]Cl_2$ and $[Ni(CY)_2(diphos)_2]Cl_2$ (where CY= Cyanoguanidine and diphos = dppm, dppe, dppp, dppb), since the structure depends on the conditions and type of the compounds involved in reaction.

The prepared complexes were characterized by element analysis CHN, conductivity measurements and magnetic susceptibility and also identified spectrally by Ultraviolet and Infrared spectroscopy.

1.Introduction

The chemistry of the transition metal complexes that contains Cyanoguanidine [1-4] have developed comparing with the complexes of organic Nitriles RCN [5 ,6]. One of the binary shapes for the Cyanamide ($NCNH_2$) is Cyanoguanidine ($NCN=NC(NH_2)_2$ which has the following two resonance structures:

Despite the large number of preparation complexes for the Cyanamide and its biological significance, the reality is the advancement of using the Cyanoguanidine complexes in polymers, composts, pesticides and pharmaceuticals[7] and many biological significances[8,9]. There are a limited number of the Cyanoguanidine complexes with the metals of transition elements like Molybdenum, Platinum and Copper[10-12]. Moreover, there are also complexes with the metals Manganese and Rhenium[13-14].

2.Experimental Part

All chemicals used in this work were purchased from commercial suppliers and used without further purification. Elemental analyses were carried on an Euro Ea 3000 CHN elemental analyzer at the center service laboratory in college of education for pure sciences, University of Baghdad. Infrared spectra were obtained on a Shimadzu FTIR 8400s spectrometer as KBr pressed pellets. UV- Visible spectra were obtained on a T90 UV–Visible spectrophotometer. Magnetic Susceptibility measurements were obtained on a Sherwood Scientific Cambridge MK1 at 25 C^o. measurements were obtained on an Ohaus starter Conductivity mete at 25 C^o and used DMSO as solvent. The IR, UV-Visible, Magnetic Susceptibility and Electrical conductivity were performed at laboratory of Chemistry Department in college of education for pure sciences, Tikrit University.

2.1.Preparation the $[(Ni)(Cy)_2Cl_2]$ (1) complex

A solution of Cyanoguanidine (0.070g; 0.834mmol) in 5ml of EtOH was added to light green solution of $NiCl_2.6H_2O$ (0.054g; 0.417mmol) in 5ml of EtOH with ratio 2:1 of the Ligand: metal. The mixture was refluxed for two hour to give a green precipitate, which was filtered off, washed with cold EtOH and dried under vacuum and recrystallized by chloroform to give dark green precipitate.(1.35g ,76%).

2.2.Preparation the $[(Ni)(Cy)_2(PPh_3)_2Cl_2]$ (2) complex

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A solution of Cyanoguanidine (0.070g; 0.834mmol) in 5ml of EtOH was added to light green solution of $NiCl_2.6H_2O$ (0.054g; 0.417mmol) in 5ml of EtOH with ratio 2:1 of the Ligand:metal, the mixture was refluxed for hour and added to the mixture a solution of PPh_3 (0.218g; 0.834mmol) in 5ml of EtOH with ration 1:2 of metal: Phosphine. The color of solution was change to light brown immediately after the addition. Then, the mixture was refluxed for two hour to give a reddish brown precipitate, which was filtered off, washed with cold EtOH and dried under vacuum and recrystallized by chloroform to give reddish brown precipitate.(3.62g , 74%).

The **$[Ni(CY)_2(PPh_3)_4]Cl_2$ (3)** complex was synthesized following the same procedure as in the case of $[(Ni)(Cy)_2(PPh_3)_2Cl_2]$ (2) complex.

2.3.Preparation of the $[(Ni)(Cy)_2(dppm)Cl_2]$ (4) complex

A solution of Cyanoguanidine (0.070g; 0.834mmol) in 5ml of EtOH was added to light green solution of $NiCl_2.6H_2O$ (0.054g; 0.417mmol) in 5ml of EtOH with ratio 2:1 of the Ligand:metal, the mixture was refluxed for hour and added to the mixture a solution of dppm (0.160g; 0417mmol) in 5ml of EtOH with ration 1:1 of metal: diphosphine, in order to change the solution color to light brown immediately after the addition. Then, the mixture was refluxed for two hour to give a reddish brown precipitate, which was filtered off, washed with cold EtOH and dried under vacuum and recrystallized by chloroform.

The following complexes **$[(Ni)(Cy)_2(dppe)Cl_2]$ (5)**, **$[(Ni)(Cy)_2(dppe)_2]Cl_2$ (6)**, **$[(Ni)(Cy)_2(dppp)_2Cl_2]$ (7)**, and **$[(Ni)(Cy)_2(dppb)_2Cl_2]$ (8)** were prepared and isolated by similar methods.

3.Results and discussion

The molecular formulas of the prepared complexes are confirmed by the results of the element analysis (CHN) as shown in (Table 1) which also illustrate the physical properties of the prepared complexes.

(Table 1) Color, Yield, M.p. and CHN analysis, magnetic momentum, molar electrical conductivity for complexes (1-8)

Seq.	Complexes	Color	m.p C°	W.t g	Yield %	Found(cal.)%			X_g ×10^{-6} c.g.s. u	X_M ×10^{-6} c.g.s.u	D ×10^{-6} c.g.s.u	X_A ×10^{-6} c.g.s.u	μ_{eff} B.M.	Λ (ohm^{-1}.cm^2.mol^{-1}) in DMSO
						C	H	N						
1	$[Ni(Cy)_2Cl_2]$	Green	330-332	1.35	76	16.23 (16.14)	2.95 (2.71)	37.64 (37.63)	12.8	3811.328	92	3903.32	3.05	15.8
2	$[Ni(Cy)_2(PPh_3)_2Cl_2]$	Reddish brown	260[d]	3.62	74	58.73 (58.42)	4.87 (4.66)	13.91 (13.63)	5.2	4276.168	264.06	4540.22	3.28	17.7
3	$[Ni(Cy)_2(PPh_3)_4]Cl_2$	Reddish brown	385[d]	6.25	78	67.98 (67.77)	5.32 (5.09)	8.40 (8.32)	3.2	4310.144	402.52	4712.66	3.35	75.2
4	$[Ni(Cy)_2(dppm)Cl_2]$	Reddish brown	254-256	3.33	82	51.34 (51.06)	4.68 (4.43)	16.57 (16.43)	6.2	4229.392	249.5	4478.89	3.26	18.2
5	$[Ni(Cy)(dppe)Cl_2]$	Reddish brown	264[d]	3.31	80	52.07 (51.76)	4.75 (4.63)	16.23 (16.10)	5.8	4037.844	261.36	4299.20	3.20	18.6
6	$[Ni(Cy)_2(dppe)_2]Cl_2$	Reddish brown	>360	5.08	78	61.28 (61.45)	4.92 (5.16)	10.13 (10.24)	4.2	4597.362	397.12	4994.48	3.45	72.6
7	$[Ni(Cy)_2(dppp)Cl_2]$	Reddish brown	265-267	3.42	81	52.70 (52.43)	5.04 (4.83)	15.82 (15.78)	6.1	4332.281	273.22	4605.50	3.31	19.3
8	$[Ni(Cy)_2(dppb)Cl_2]$	Reddish brown	273-276	3.32	77	53.31 (53.07)	5.34 (5.01)	15.57 (15.47)	6.5	4707.56	285.08	4992.64	3.44	17.0

d: decomposition

3.1.Measurements of electrical conductivity

The electrical conductivity of the solutions is widely used in the coordination chemistry to know the ionic formulas of the complexes in the solution and in the solid state[15]. The greater the number of ions that are released by the complexes in the solution, the higher its electrical conductivity and the complexes that cannot be ionized has low conductivity[16]. The electrolyte type can be deduced from the molecular conductivity measurement at a constant concentration (10^{-3}) for several types of electrolytes in different solvents. The electrical conductivity of the prepared complexes is measured at (10^{-3}) molar in (DMSO) solution at 25°C. The results of conductivity for the two prepared complexes **$[Ni(Cy)_2(PPh_3)_4]Cl_2$ (3)** and **$[Ni(Cy)_2(dppe)_2]Cl_2$ (6)** were (75.2, 72.6) respectively which Indicating that a 1:2 ratio of positive ions: negative ions, while The rest of complexes gave molecular conductivity values ranging from (15.8-19.3) which are not conductive and do not give ions in the solution[17].

3.2.Susceptibility measurements

The magnetic susceptibility of the prepared complexes were calculated at a room temperature and were correct diamagnetic (D) of atoms in organic molecules, inorganic roots and metal ions using

Pascal constants in the constituent atoms of the prepared complexes, since D which represent ($g.atom^{-1}$) = The total number of ions or atoms of an element multiplied by the value of its Pascal constant. The values of the effective magnetic momentum (μ_{eff}) were calculated according to the following relationship:-

$$\mu_{eff.} = 2.828\sqrt{\chi_A T} \quad B.M.$$

The values of gram susceptibility (X_g), molecular susceptibility (X_M), atomic susceptibility (X_A), the electromagnetic correction factor (D) and μeff of the prepared complexes are shown in (Table 1).

With respect to the nickel complexes of octahedral, they are derived from ionic state d^8 and have two lonepair electrons. In the nickel complexes of octahedral there is no orbital contribution, so the magnetic momentum is approximately (2.83-3.50 B.M)[18].

The prepared complex (1) exhibit magnetic momentum at (3.05B.M) this value is consistent with the tetrahedral structure[19]. While the prepared complexes (2-8) exhibit magnetic momentum within the range (3.20-3.45B.M) and these values are consistent with the octahedral structure[20].

3.3.Electronic spectra

The electronic spectra of the prepared complexes were measured using a (DMSO) solvent within the range of (900-200nm) and at a concentration of (10^{-3}) molar for the visible area and ultraviolet. The prepared complexes gave d-d spectra, charge transfer spectra and ligand spectrum. The prepared tetrahedral complex **(1)** showed a pack in the region (20161 cm^{-1}) due to the transition $^3T_1(F) \rightarrow {}^3T_1(P)\upsilon_2$. The pack that appeared in the region (19193cm^{-1}) is due to electronic transition $^3T_1g(F) \rightarrow {}^3T_2(F)\upsilon_3$. The charge transfer pack is shown in the region (30058cm^{-1}) and confirms the appearance of these bands in the range shown that the Ni(II) complexes has a tetrahedral structure[19].

The prepared octahedral complexes **(2-5,7,8)** showed absorption bands in the region (11689-12121cm^{-1}) due to transition $^3A_2g(F) \rightarrow {}^3T_1g(F)\ \upsilon_2$. The bands that appeared in the region (21739-18667cm^{-1}) due to electronic transition $^3A_2g(F) \rightarrow {}^3T_1g(P)\ \upsilon_3$. The charge transfer bands were observed in the region (38128-30137cm^{-1}). The appearance of these bands in the indicated range confirms that the prepared complexes **(2-8)** have octahedral structure[21,22], as illustrated in (Table 2) and (Figures 1-4).

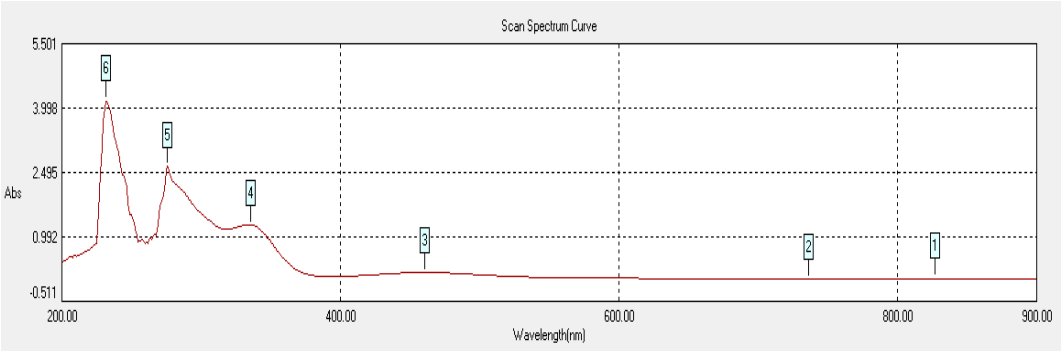

Figure (1) shows the UV-Visb. spectrum of the complex (2)

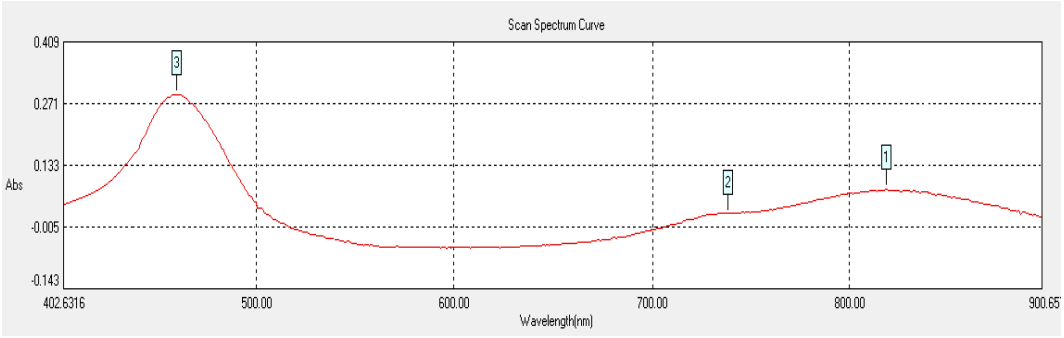

Figure (2) shows the Visb. spectrum of the complex (2)

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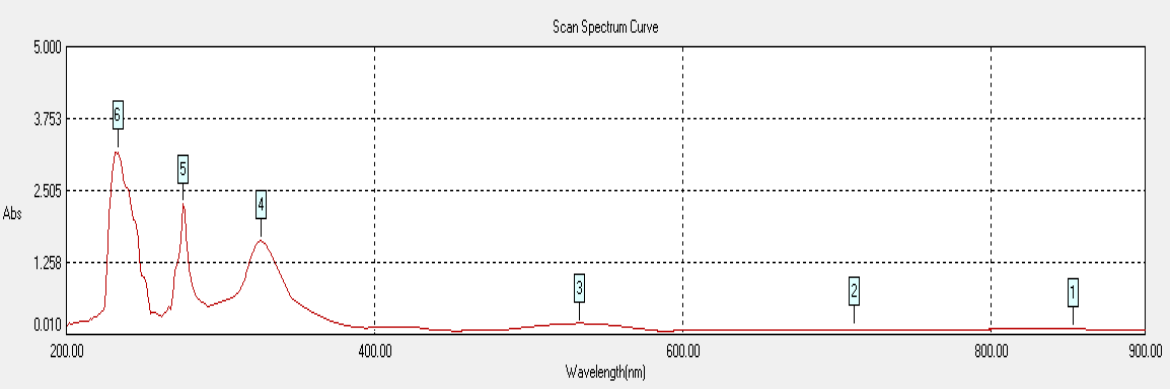


Figure (3) shows the UV-Visb. spectrum of the complex (7)

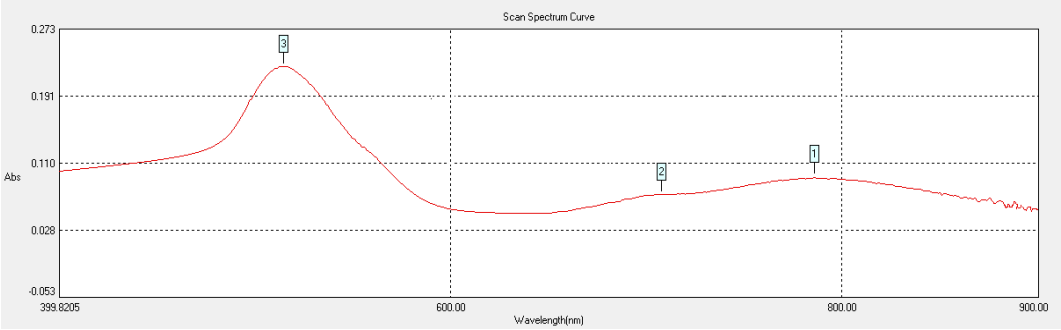


Figure (4) shows the Visb. spectrum of the complex (7)

3.4.Infrared spectra

The infrared spectrum of the complex $[Ni(Cy)_2(Cl)_2]$ (1) Figure (6) shows the shift of the υ(C≡N) band from (2163-2206cm^{-1}) for free ligand to (2187-2227cm^{-1}) of the complex is an indicative of the coordinate of this group[23]. The band υ(C=N) appeared at (1683cm^{-1}) in the complex and also a band appeared at (3550cm^{-1}) attributed to υ(NH), while another band appeared at (383cm^{-1}) attributed to the (Ni-Cl)[24].

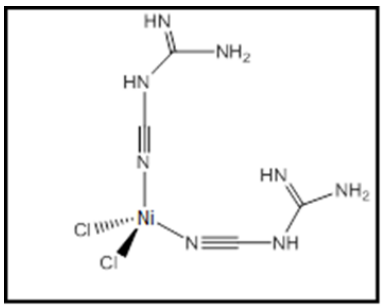
The two complexes (2,3) showed a significant shift of υ(C≡N) to (2192-2237cm^{-1}) and (2136-2181cm^{-1}) respectively, also the bands υ(C=N) appeared at (1660cm^{-1}) and (1629cm^{-1}) respectively. The two complexes showed a band at (3319cm^{-1}) and (3384 cm^{-1}) attributed to υ(NH). The (P-Ph) band was found in the complexes at (1429cm^{-1}) and (1440 cm^{-1}) respectively while the band b(P-C) appeared at (694cm^{-1}) and (686 cm^{-1}) respectively[23].

The complexes (4-8) showed a significant shift of υ(C≡N) with a range of (2187-2231cm^{-1}) to (2196-2243cm^{-1}) while the bands υ(C=N) appeared in the range of (1689-1643cm^{-1}) and the bands (P-Ph) was shown in the complexes (4-8) at (1437-1433cm^{-1}), while the bands b(P-C) appeared at (694-680cm^{-1})[25] as illustrated in Table (3).

3.5.The suggestion of complex formulas

Based on the results obtained and compared with the literature[26,27], we suggest the following formulas for the prepared complexes:

1- The complex $[Ni(Cy)_2Cl_2]$ (1) shall be a tetrahedral where ligand Cy is coordinate by the (C≡N) group.



2- The complexes of (3,6) are becoming octahedral where the Cl ion is outside the coordinate ball and the ligand Cy is coordinated by the(C≡N) group. These complexes may be have two isomers cis and trans.

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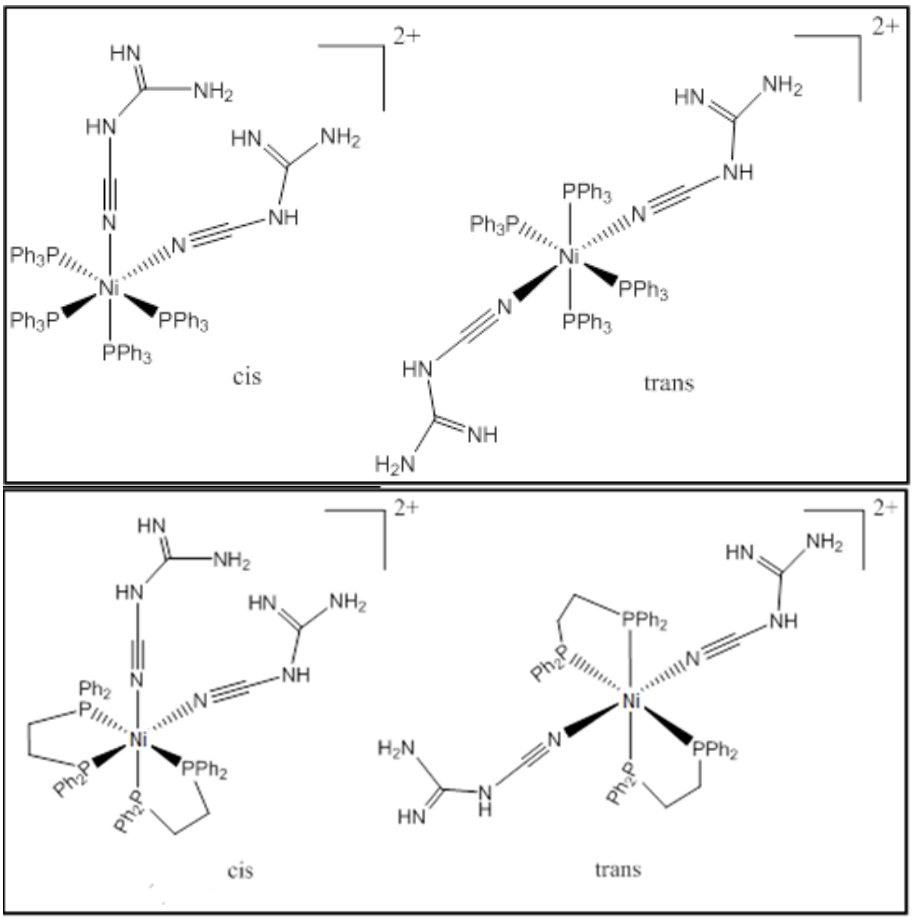
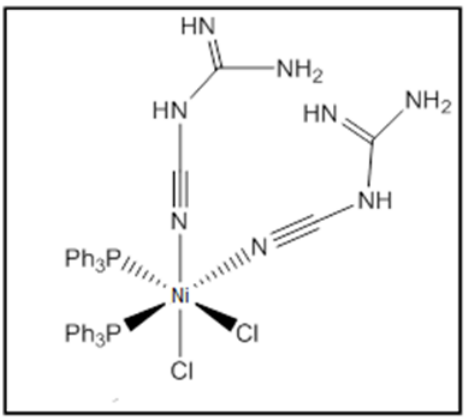
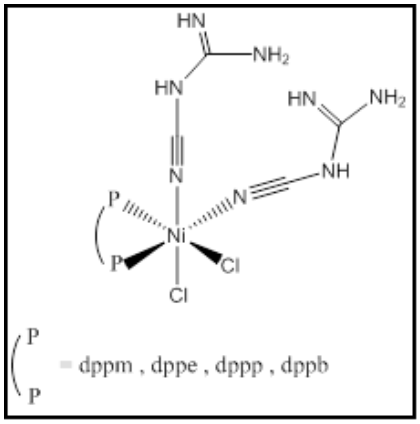


3- The complexes of (2).(4,5),(7,8) are becoming octahedral where the Cl ion is inside the coordinate ball and the ligand Cy is coordinated by the(C≡N) group.





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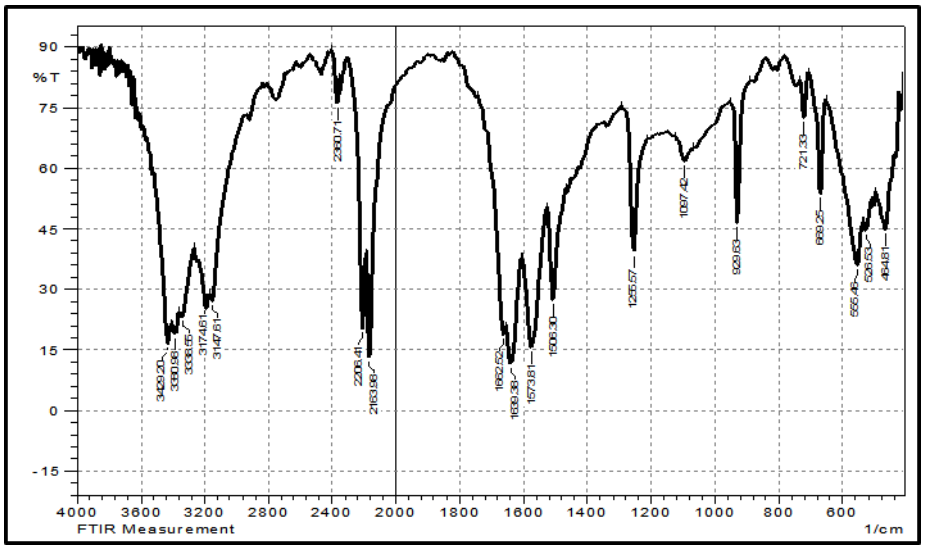

Figure (5) IR Spectrum of free cyanoguanidine

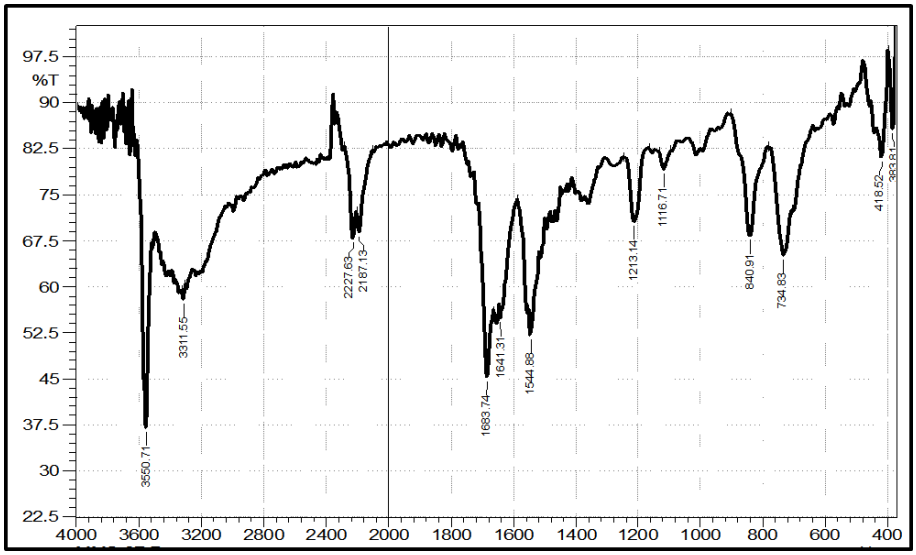

Figure (6) IR Spectrum of complex (1)

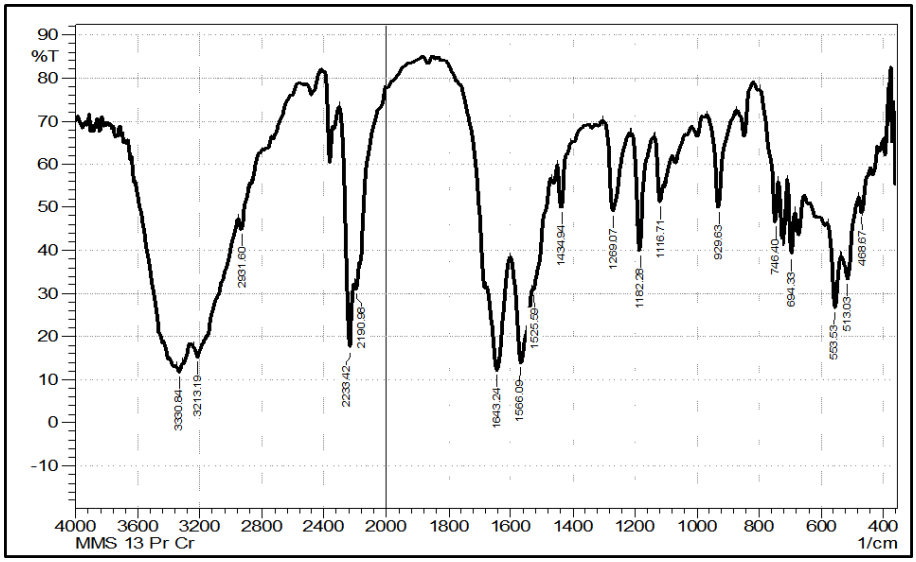

Figure (7) IR Spectrum of complex (5)

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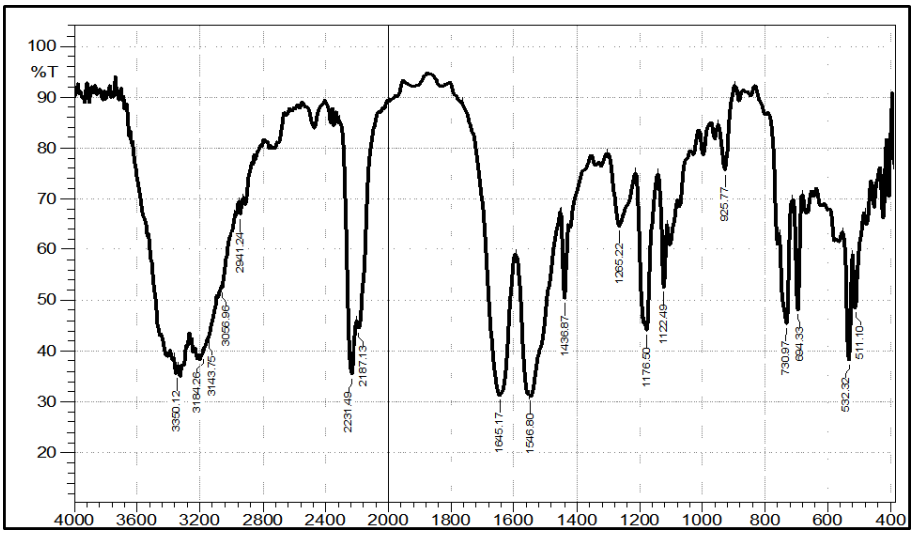

Figure (8) IR Spectrum of complex (7)

Table (2) the electronic transition of complexes (1-8)

No.	Complexes	Absorption		Possible assignment	البنية
		(nm)	(cm^{-1})		
1	$[(Ni)(Cy)_2Cl_2]$	233.59	42810.05	Ligand	T.h
		268.47	37248.11	Ligand	
		332.69	30058.01	C.T	
		496	20161.29	$^3T_1(F) \rightarrow {}^3T_1(P)$	
		521	19193.86	$^3T_1(F) \rightarrow {}^3T_2(F)$	
2	$[(Ni)(Cy)_2(PPh_3)_2Cl_2]$	232	43103.45	Ligand	O.h
		267	37453.18	Ligand	
		335	29850.75	C.T	
		460	21739.13	$^3A_2g(F) \rightarrow {}^3T_1g(P)$	
		729.81	13702.2	$^3A_2g \rightarrow {}^1Eg$	
		820.61	12186.06	$^3A_2g(F) \rightarrow {}^3T_1g(F)$	
3	$[(Ni)(Cy)_2(PPh_3)_4]Cl_2$	233.18	42885.32	Ligand	O.h
		265.2	37707.39	Ligand	
		331.81	30137.73	C.T	
		467.1	21408.69	$^3A_2g(F) \rightarrow {}^3T_1g(P)$	
		723.6	13819.79	$^3A_2g \rightarrow {}^1Eg$	
		825	12121.21	$^3A_2g(F) \rightarrow {}^3T_1g(F)$	
4	$[(Ni)(Cy)_2(dppm)Cl_2]$	232.87	42942.41	Ligand	O.h
		269.43	37115.39	Ligand	
		278.31	35931.16	C.T	
		478.34	20905.63	$^3A_2g(F) \rightarrow {}^3T_1g(P)$	
		83.21	120177.9	$^3A_2g(F) \rightarrow {}^3T_1g(F)$	
5	$[(Ni)(Cy)_2(dppe)Cl_2]$	233.29	42865.1	Ligand	O.h
		273	36630.04	Ligand	
		275.42	36308.18	C.T	
		481	20790.02	$^3A_2g(F) \rightarrow {}^3T_1g(P)$	
		850.67	11755.44	$^3A_2g(F) \rightarrow {}^3T_1g(F)$	
7	$[(Ni)(Cy)_2(dppp)Cl_2]$	234	42735.04	Ligand	O.h
		276	36231.88	Ligand	
		262.27	38128.65	C.T	
		535.68	18667.86	$^3A_2g(F) \rightarrow {}^3T_1g(P)$	
		715.45	13977.22	$^3A_2g \rightarrow {}^1Eg$	
		855.45	11689.75	$^3A_2g(F) \rightarrow {}^3T_1g(F)$	
8	$[(Ni)(Cy)_2(dppb)Cl_2]$	233.9	42753.31	Ligand	O.h
		271.5	36832.41	Ligand	
		263.1	38008.36	C.T	
		533.85	18731.85	$^3A_2g(F) \rightarrow {}^3T_1g(P)$	
		718.1	13925.64	$^3A_2g \rightarrow {}^1Eg$	
		852	11737.09	$^3A_2g(F) \rightarrow {}^3T_1g(F)$	

Table (3) the IR bands of complexes (1-8)

Seq.	Complexes	υ(NH)	υ(C≡N)	υ(C=N)	υ(P-Ph)	b(P-C)	M-Cl
-	Cyanoguanidine	3429s	2163s 2206s	1662s	-	-	-
1	$[Ni(Cy)_2Cl_2]$	3550b	2187m 2227m	1683s	-	-	383w
2	$[Ni(Cy)_2(PPh_3)_2Cl_2]$	3319b	2192s 2237s	1660s	1429m	694w	-
3	$[Ni(Cy)_2(PPh_3)_4]Cl_2$	3330b	2136m 2181m	1629s	1440m	686w	-
4	$[Ni(Cy)_2(dppm)Cl_2]$	3342b	2190s 2240s	1677s	1438m	694w	-
5	$[Ni(Cy)(dppe)Cl_2]$	3344b	2196s 2243s	1689s	1437m	680w	381w
6	$[Ni(Cy)_2(dppe)_2]Cl_2$	3340b	2192s 2241s	1688s	1437m	685w	-
7	$[Ni(Cy)_2(dppp)Cl_2]$	3350b	2187s 2231s	1645s	1436m	694w	-
8	$[Ni(Cy)_2(dppb)Cl_2]$	3330b	2190s 2233s	1643s	1434m	694w	-

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تحضير وتشخيص معقدات Ni(II) الحاوية على ليكاندات سيانوكوانيدين والفوسفينات

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الملخص

اظهر ليكاند السيانوكوانيدين صيغه تناسق مهمة مع Ni^{2+} مكوناً معقداً ذا شكل هندسي رباعية السطوح من نوع $[Ni(CY)_2Cl_2]$ عند تفاعل الفلز بوجود السيانوكوانيدين بنسبة 2:1 ومعقدات ثمانية السطوح من نوع $[Ni(Cy)_2(PPh_3)_2Cl_2]$ و $[Ni(Cy)_2(PPh_3)_4]Cl_2$ و $[Ni(Cy)_2(diphos)Cl_2]$ (حيث ان Cy= Cyanoguanidine و diphos= dppm, dppe, dppp, dppb) حيث يعتمد التركيب على ظروف والمركبات التي تدخل في التفاعل.

وشخصت المعقدات المحضرة بواسطة التحليل الدقيق للعناصر (CHN) وقياسات التوصيلية والحساسية المغناطيسية وشخصت طيفيا بواسطة طيف الاشعة فوق البنفسجية وطيف الاشعة تحت الحمراء.