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Synthesis and Characterization Complexes of Cobalt(II), Nickel(II), Copper(II) and Palladium with [N'-{2-(4-isobutyl-phenyl)-propionyl} pyridine-4-carbohydrazide]

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Abstract

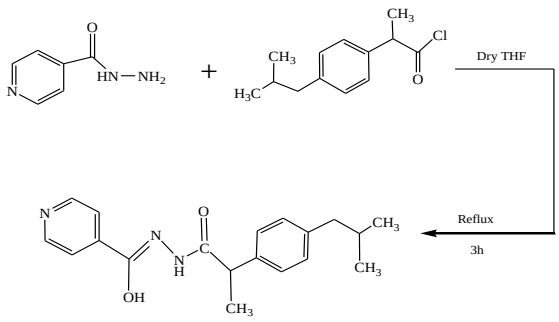
This research concern with the metal complexes of the ligand [N'-{2-(4-isobutyl-phenyl)-propionyl}pyridine-4-carbohydrazide] with Co(II), Ni(II) , Cu(II) and Pd(II) have been synthesized and characterized by atomic absorption , infrared, electronic spectra, molar conductivity and magnetic moment measurements. On the basis of the data obtained from the above measurements, the ligand act as a neutral bidentate and the octahedral structures are proposed for the Co(II), Ni(II) and Cu(II) while the square planer structures are assigned to Pd(II) complexes.

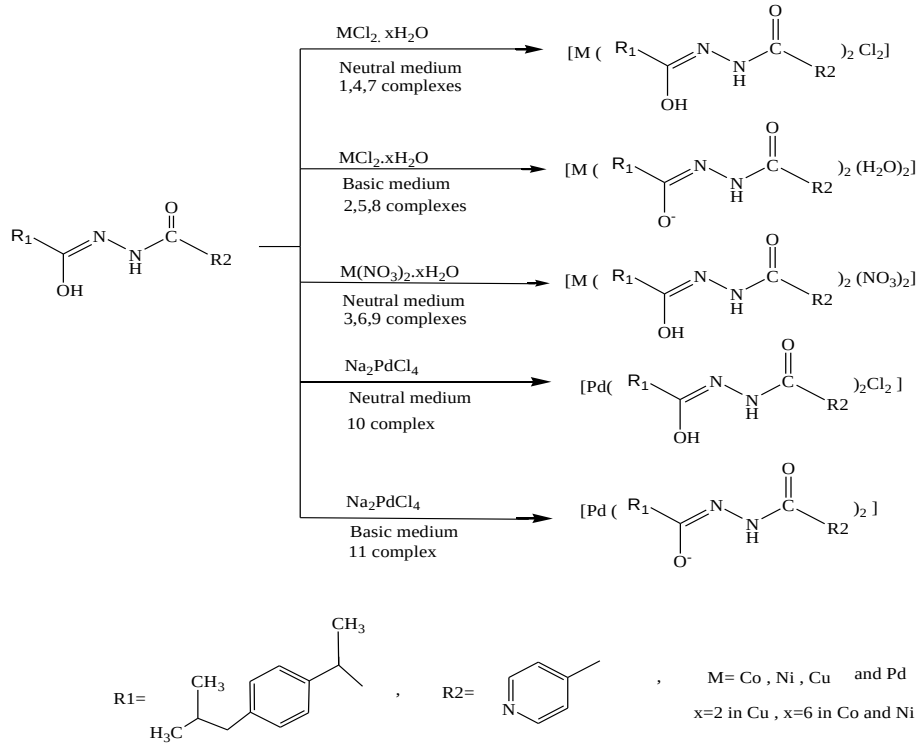
Keywords: diacylhydrazines, Transition metal Complexes, spectral data

1. Introduction.

Aroylhydrazine ligands have recently gained the increasing concern due to their quite interesting chemical properties [1,2]. Most of the studies were focused on unsymmetrical aroylhydrazines, used as ligands for coordination to metal ions, due to the tautomeric effect of the enol form and keto form, several electron-rich donor centers, variable bonding modes, structural diversity and promising biological implications[3-6]. As regards biological implications, complexes with these ligands have been intensively investigated for antitumoral, antimicrobial and anti-inflammatory activities. The inhibitory action is attributed due to their chelating properties [7,8]. In view of our interest in metal complexes with a variety of multidentate acylhydrazines [9], we report here in the synthesis and characterization of [N'-{2-(4-isobutyl-phenyl)-propionyl}pyridine-4-carbohydrazide] **(LH)**, which represents a prototype of new of asymmetric ligands, which combine two donor functionalities in one molecule (ibuprofen acid chloride and Isoniazid).

In order to further investigate the coordination modes of **(LH)** with transition metals and to study the chemistry of its complexes, we synthesized and characterized and their Cu(II), Co(II), Ni(II) and Pd(II) complexes (Scheme 1). These studies have been mainly directed towards identifying the groups directly attached to the metal site and establishing the structure of the coordination compounds thus formed.





Scheme 1. Route of synthesis for (LH) and the corresponding Cu(II), Co(II), Ni(II) and Pd(II) complexes.

2-Experimental Part.

2-1. Physical measurements.

All the chemical substances were supplied by BDH and Fluka and of purity more than 99%.The NMR spectra were registered on a Varian Gemini 300 BB apparatus working at 300 MHz for a 1H and 75 MHz for 13C using TMS as internal standard. Chemical elemental analyses were done with Eurovectro EA3000 (for C, H and N). Infrared spectra were record by a SHIMADZU infrared spectrophotometer FT - IR model 8400S in the 4000-400 cm^{-1} Range using KBr disc, Ultraviolet spectra were recorded Shimadzu UV-VIS Recording UV-1800 Spectrophotometer using DMF as a solvent, melting point were determined by an Electrothermal melting point model 9300,Magnetic susceptibilities were measured on instrument type Bruker BM 6 were carried at room temperature by Faraday method , the molar conductivity of the complexes (0.001 M) in DMF were measured using HANNA model 214EC conductivity meter, determination of metals percentage were determined by atomic absorption spectrophotometer on PYEUNICAM SP9-Atomic Absorption Spectrophotometer (Phillips).

2-2. Preparation of the ligand.

The [N'-{2-(4-isobutyl-phenyl)-propionyl}pyridine-4-carbohydrazide] ligand was prepared as following(Scheme-1), was added slowly with stirring (0.001mol) from ibuprofen acid chloride to Isoniazid (0.001mol) in (25 ml) of dry THF. The solution was kept under reflux for 3 hours. Solution cooled down, and poured on crushed ice and neutralized using 10% sodium bicarbonate, a white solid precipitated directly. The white precipitate was isolated and recrystallized from ethanol .Yield: 79%; m.p.218-221 ºC; Anal. calcd. for C19H23N3O2 (345.4 g/mol): C, 70.06, H, 7.06, N, 12.90%. Found: C, 71.15, H, 6.79, N, 12.73%; IR (KBr, cm^{-1}): 3452 m(OH),3304 w (NH), 3115 m (C-H stretching of aromatic ring), 2954 m (CH2 as),1668 s (C=O), 1597 s(C=N).1219 m(C-O), 1H-NMR: (DMSO-d6) δ at 0.9 (d, (CH3)4), 1.9(m, 1H aliphatic), 2.5(d, - CH2-), 1.3(d, 3H aliphatic), 3.7(quartet,1H), 7.1(dd, Ar, J=8.14 for ibuprofen ring), 6.9(s, H, NH), 7.8(d.2H, pyridine ring), 8.7(d.2H, pyridine ring),10.4(s,OH). 13C-NMR: 167 (C=O), 19(CH3), 22(CH3)2, 44 (CH2aliphatic), 40(C-H aliphatic), 30(C-H aliphatic tertiary), 128.8,129.3(C=C Ar for ibuprofen ring), 139, 140(=C-Ar for ibuprofen ring), 151 (C-Ar in pyridine).

2.3. Synthesis of the complexes.

(**1,3,4,6,7, 9** and **10** complexes)

These complexes were prepared using $CoCl_2.6H_2O$, $NiCl_2.6H_2O$,$CuCl_2.2H_2O$,$Co(NO_3)_2.6H_2O$.$Ni(NO_3)_2.6H_2O$, $Cu(NO_3)_2.2H_2O$ and Na_2PdCl_4 salts scheme(1). The ethanolic solution of metallic salt (1 mmol/5 mL ethanol) was mixed with stirring with a hot clear ethanolic solution of the ligand **(LH)** (2 mmol/20 mL ethanol). After stirred the solution for 3 hrs, Then cooled down to room temperature. The resulting solution, was filtered off and set aside for slow evaporation at room temperature. A precipitates were filtered, washed successively with water and diethylether and finally dried in oven.

(**2,5,8** and**11**complexes)

These complexes were prepared using $CoCl_2.6H_2O$, $NiCl_2.6H_2O$, $CuCl_2.2H_2O$ and Na_2PdCl_4 salts. A hot ethanolic solution of metallic salt (1 mmol/5 ml

ethanol) was mixed with stirring with a hot clear ethanolic solution of the ligand **(LH)** (2 mmol/20 ml ethanol). A few drops of triethylamine (Et_3N) was added to the solution with stirring. The mixtures were refluxed for 3 hours. The solid precipitates were filtered off, washed in ethanol , distilled water and dried. The solid precipitates were recrystallized from DMSO.

3.Results and Discussion

The ligand and it's metal complexes were insoluble in water but soluble in organic solvents such as DMSO,DMF the metal percentage in the complexes and physical data of ligands and complexes were given in table (1).

Table (1): metal percentage and physical data of ligands and it's complexes.

	Compound	Formula	M.Wt	Color	M.P c^0	Yield	%Metal Calc(found)
	LH	$C_{19}H_{23}N_3O_2$	325.4	Yellow	218-220	79	_
1-	$[Cu(LH)_2Cl_2]$	$C_{38}H_{46}N_6O_4Cl_2Cu$	785.26	Green	253-255d	72	9.04 (8.3)
2-	$[Cu(L)_2(H_2O)_2]$	$C_{38}H_{48}N_6O_6Cu$	748.37	Green	196-198	67	9.2 (8.8)
3-	$[Cu(LH)_2(NO_3)_2]$	$C_{38}H_{46}N_8O_{10}Cu$	838.38	Green	229-231d	69	8.4 (7.5)
4-	$[Co(LH)_2Cl_2]$	$C_{38}H_{46}N_6O_4Cl_2Co$	781.26	Brown	223-225d	66	7.9 (8.3)
5-	$[Co(L)_2(H_2O)_2]$	$C_{38}H_{48}N_6O_6Co$	744.37	Brown	167-169	75	7.9 (7.08)
6-	$[Co(LH)_2(NO_3)_2]$	$C_{38}H_{46}N_8O_{10}Co$	834.38	Brown	209-211	65	6.7 (7.5)
7-	$[Ni(LH)_2Cl_2]$	$C_{38}H_{46}N_6O_4Cl_2Ni$	780.4	Yellow	279-282d	62	7.4 (8.3)
8-	$[Ni(L)_2(H_2O)_2]$	$C_{38}H_{48}N_6O_6Ni$	743.51	Greenish-Yellow	266-269d	73	7.8 (7.04)
9-	$[Ni(LH)_2(NO_3)_2]$	$C_{38}H_{46}N_8O_{10}Ni$	833.52	Yellow	290-294d	65	10.38 (10.22)
10-	$[Pd(LH)_2Cl_2]$	$C_{38}H_{46}N_6O_4Cl_2Pd$	828.11	Yellow	175-177	73	
11-	$[Pd(L)_2]$	$C_{38}H_{44}N_6O_4Pd$	755.19	Brown	226-228	77	

d= decomposition temperature

3.1. Infrared spectra.

(1,3,4,6,7,9 and **10)** complexes

The IR spectra of all the complexes were listed in table(2), and compared with that of the ligand to study the structural changes in the ligand upon complexation. The IR spectra of the complexes showed bands at 3387-3444 cm^{-1} due to υ(O-H). In the IR spectra of the complexes showed a medium intensity band at 1602-1620 cm^{-1} due to υ(C=O), shifted to lower frequencies compared with that of the ligand, this indicates that C=O group is participate in coordination to metal ions[10]. There is a medium intensity band at 1597 cm^{-1} in the IR spectrum of ligand due to C=N group, with shifted lower frequency at 1546-1566 cm^{-1}, this indicates that C=N groups is participate in coordination to metal ions[11] . New strong intensity bands appears 1180-1215 cm^{-1} due to (C-O) group[12] .A new strong intensity bands at 1381-1392cm^{-1} **3,6** and **9** complexes in the spectra of the complexes which assigned to stretching frequency of $\upsilon(NO_3)$ bond [13]. The appearance of bands in the IR region at 410-443 cm^{-1} in the complexes may be assignable to M-N frequency.Additional bands in the complexes in the region 474-549cm^{-1} compared with IR spectrum of free ligand have tentatively been assigned to M-O bond [14,15]. From the obtained results it is clear that the metal ions associated with ligand through oxygen atom of one carboxyl groups and nitrogen atom of one amide groups.

(**2,5** and **8**) complexes

The IR spectrum of the ligand shows band at 3305 cm^{-1} due to the NH group, IR spectra of the prepared complexes showed disappeared of this band that mean not found N-H in these complexes. The υ(C=O) band was disappeared in spectra of these complexes. There is one medium intensity bands at 1597 cm^{-1} in the IR spectrum of ligand due to C=N group , and this was shifted to lower frequencies at 1548 -1566 cm^{-1}, this indicates that C=N groups is participate in coordination to metal ions. New strong intensity bands at 1604-1610 cm^{-1} due to new C=N. New strong intensity bands appears 1172-1219 cm^{-1} due to (C-O) group[11]. The stretching vibration of the CH group observed at 3029-3053cm^{-1} and 2954-2978cm^{-1} the aromatic and aliphatic respectively. The IR spectra of the complexes exhibit a broad band's centered at 3402-3425 cm^{-1} due to the symmetric and asymmetric stretching modes of coordinated H_2O. Weak bands in the 934 -945 cm^{-1} in complexes, and these bands represent the coordinated water[16] . The appearance of bands in the IR region at 403-443 cm^{-1} in the complexes may be assignable to M-N frequency. The appearance of new bands in the complexes in the region at 501-564 cm^{-1} compared with IR spectra of free ligand have tentatively been assigned to M-O bond[17,18].

(**10**) complex.

In the IR spectrum of $[Pd(L3H)_2Cl_2]$ complex(**10**) showed strong band at 3442 cm^{-1} due to OH ,as well as the disappearance of the N-H groups and also disappearance of C=O in spectrum of complex. There is one medium intensity band at 1597 cm^{-1} in the IR spectrum of ligand due to C=N group , with shifted lower frequency at 1546 in complex, this indicates that C=N groups is participate in coordination to

metal ions[10]. New intensity band at 1608 cm^{-1} due to new C=N. New strong intensity band appears 1068cm^{-1} due to (C-O) group.

(**11**) complex.

In the IR spectrum of $[Pd(L)_2]$ complex (**11**) showed weak band at 3197 cm^{-1} due to υ(N-H) group, and this was shifted to lower frequency, this indicates that N-H group is participate in coordination to metal ions. In the IR spectra of complex showed the one medium intensity band at 1600 cm^{-1} due to υ(C=N). One medium intensity band at 1660 cm^{-1} due to υ(C=O), with shifted to slightly lower frequencies compared with that of the ligand, this indicates that ^{2}C=O group is not participate in coordination to metal ion.

Table (2): Important infrared spectral bands (cm^{-1}) and their assignments.

	Compounds	U(O-H)	U(N-H)	U(C=O)	U(C=N)	U(C-O)	U(NO_3)	H_2O Coord	U(M-N)	U(M-O)
	LH3	3452 m	3304 m	1668 m	1597 m	1062 m	_	_	_	_
1-	$[Co(L3H)_2Cl_2]$	3387 m	3208 w	1609 w	1579 m	1066 m	_	_	426 w	528 w
2-	$[Co(L3)_2(H_2O)_2]$	3402 m	_	_	1548 m 1610 m	1060 S	_	934 w	403 s	524 w
3-	$[Co(L3H)_2(NO_3)_2]$	3396 m	3234 w	1610 m	1552 m	1058 m	1381 s	_	416 w	532 w
4-	$[Ni(L3H)_2Cl_2]$	3398 m	3210 w	1602 m	1579 w	1070 m	_	937 w	430 w	549 m
5-	$[Ni(L3)_2(H_2O)_2]$	3410 m	_	_	1566 s 1604 s	1037 S	_	_	407 w	564 m
6-	$[Ni(L3H)_2(NO_3)_2]$	3405 w	3155m	1604 s	1549 w	1029 m	1392 m	_	410 w	474 m
7-	$[Cu(L3H)_2Cl_2]$	3444 m	3309 w	1620 s	1556 m	1060 s	_	_	443 w	499 w
8-	$[Cu(L3)_2(H_2O)_2]$	3425 m	_	_	1552 m 1610 m	1060 S	_	945 w	441 w	501 w
9-	$[Cu(L3H)_2(NO_3)_2]$	3425 s	3240 w	1610 s	1554 m	1043 m	1388 s	_	443 w	501 w
10-	$[Pd(L3H)_2Cl_2]$	3442 s	_	_	1546 s 1608 s	1068 s	_	_	_	_
11-	$[Pd(L3)_2]$	_	3197 w	1660 w	1546 s	1060 m	_	_	_	_

3-2.Magnetic measurement and electronic spectra.

The magnetic moment and spectral data of prepared complexes are given in table (3). The magnetic moment of Cobalt (II) has been found to be (4.15-4.83 B.M) which is with the range of octahedral Cobalt (II) complexes [19] . The electronic spectrum of **LH** show strong bands in the range 38638-39975 cm^{-1} which can be assigned to π→π* and n→π* transitions, the electronic spectra of Cobalt (II) complexes show three absorption bands at 11986-12704 cm^{-1} ,15105-16116 cm^{-1} and 18087-18762 cm^{-1} these were assigned to $^4T_{1g}$ (F) →$^4T_{2g}$ (F) *(v 1)*, $^4T_{1g}$(F) →$^4T_{1g}$(P) (v2), and $^4T_{1g}$ (F) → $^4A_{2g}$(v3) transitions respectively, which are characteristic of octahedral stereochemistry[20,21]. For Nickel (II) complexes its magnetic moment (3.39-3.67 B.M) and spectra of these complexes show bands at 9825-9922 cm^{-1} , 16464-16743 cm-1 and 21927-22162 cm^{-1} which suggesting the existence of $^3A_{2g}$ (F) → $^3T_{2g}$ (F) (ν1), $^3A_{2g}$ (F)→$^3T_{1g}$(F) (ν2) and $^3A_{2g}$ (F) → $^3T_{1g}$ (P) (ν3) transitions which an octahedral spectral configuration[22, 23]. The magnetic moment value of Copper (II) complexes (1.78-2.08 B.M) which may suggest an distorted octahedral structure. Its electronic spectrum show at 14931-15371 cm^{-1} which may assigned to 2E2g→ 2T2g transition in octahedral structure [24,25]. The magnetic moment value of Palladium (II) complexes (0.00 B.M) which may suggest an square planer structure. Its electronic spectrum show at 17548-18239 cm^{-1} which may assigned to $^1A_{1g}$ → $^1B_{1g}$ transition in square planer structure [16,27].

Table (3): magnetic moment, Electronic spectra, Conductivity

	Compound	Electronic Spectra cm^{-1}			Transition	μ eff. (B.M)	Conductivity $cm^2.ohm^{-1}.mol^{-1}$
		v 1	*v 2*	*v 3*			
	L3H	38638	39975		$n \rightarrow \pi^*$(*v 1*) $\pi \rightarrow \pi^*$(*v 2*)		–
1-	$[Co(L3H)_2Cl_2]$	12704	15105	18087	$^4T_{1g}$ (F)→$^4T_2$ (F) (*v 1*) $^4T_{1g}$(F)→$^4A_{2g}$(F)(v2) $^4T_{1g}$ (F)→$^4T_{1g}$(P)(v3)	4.19	2.6
2-	$[Co(L3)_2(H_2O)_2]$		16841	22524	$^4T_{1g}$(F)→$^4A_{2g}$(F)(v2) $^4T_{1g}$ (F)→$^4T_{1g}$(P)(v3)	4.83	14.8
3-	$[Co(L3H)_2(NO_3)_2]$	11986	16161	18762	$^4T_{1g}$ (F)→$^4T_2$ (F) (*v 1*) $^4T_{1g}$(F)→$^4A_{2g}$(F)(v2) $^4T_{1g}$ (F)→$^4T_{1g}$(P)(v3)	4.15	2.22
4-	$[Ni(L3H)_2Cl_2]$	9922	16549	21927	$^3A_{2g}$ (F) → $^3T_{2g}$ (F) (v1) $^3A_{2g}$ (F)→$^3T_{1g}$(F) (v2) $^3A_{2g}$ (F) → $^3T_{1g}$ (P) (v3)	3.40	2.1
5-	$[Ni(L3)_2(H_2O)_2]$	9825	16464	22162	$^3A_{2g}$ (F) → $^3T_{2g}$ (F) (v1) $^3A_{2g}$ (F)→$^3T_{1g}$(F) (v2) $^3A_{2g}$ (F) → $^3T_{1g}$ (P) (v3)	3.67	14.4
6-	$[Ni(L3H)_2(NO_3)_2]$	9892	16743	22158	$^3A_{2g}$ (F) → $^3T_{2g}$ (F) (v1) $^3A_{2g}$ (F)→$^3T_{1g}$(F) (v2) $^3A_{2g}$ (F) → $^3T_{1g}$ (P) (v3)	3.39	1.92
7-	$[Cu(L3H)_2Cl_2]$		14931		$^2E_{2g} \rightarrow {}^2T_{2g}$	1.91	3.1
8-	$[Cu(L3)_2(H_2O)_2]$		15132	21281	$^2E_{2g} \rightarrow {}^2T_{2g}$ (v2) charge transfer (v3)	2,08	13.4
9-	$[Cu(L3H)_2(NO_3)_2]$		15371		$^2E_{2g} \rightarrow {}^2T_{2g}$	1.78	2.9
10-	$[Pd(L3H)_2Cl_2]$		17584		$^1A_{1g} \rightarrow {}^1B_{1g}$	0.00	0.9
11-	$[Pd(L3)_2]$		18239		$^1A_{1g} \rightarrow {}^1B_{1g}$	0.00	16.2

3-3. Conductivity measurements:

The molar conductance of these complexes 0.001 M in DMF at room temperature show that all the complexes are non-electrolytes [28]. The value in the range 0.9-16.2 $cm^2.ohm^{-1}.mol^{-1}$ table (3).

According to these results the structural formula of prepared complexes may be proposed in (fig- 1).

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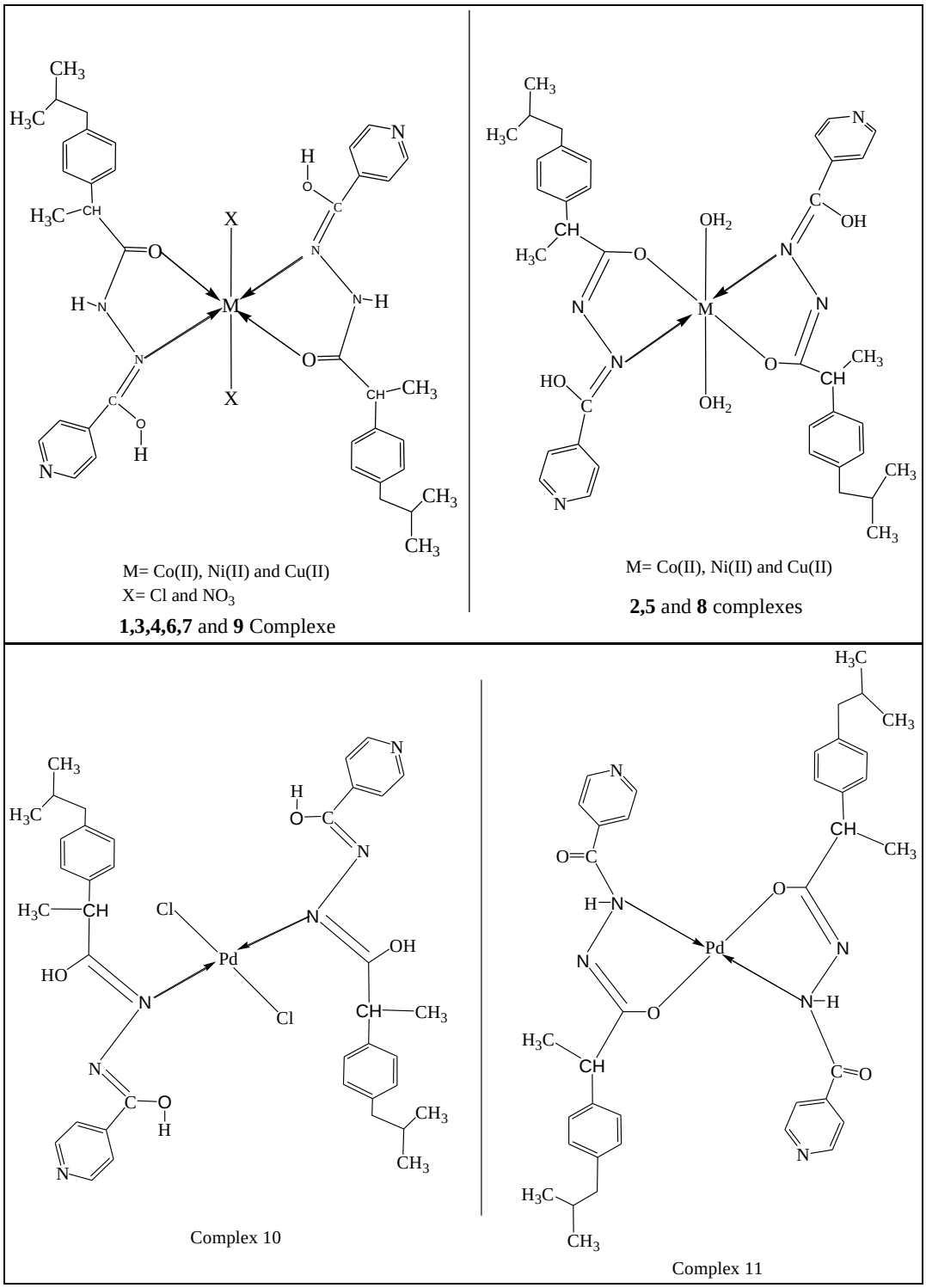

Figure(1): the proposed structural formula of the complexes.

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تحضير وتشخيص معقدات الكوبلت والنيكل والنحاس الثنائية مع الليكاند (N–[2–(4–ايزوبيوتيل–فنيل)–بروبونيل] بريدين–4–كاربوهايدرازايد)

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[2]قسم الكيمياء ، كلية العلوم ، جامعة تكريت ، تكريت ، العراق

الملخص

يتضمن البحث تحضير معقدات فلزية لايونات الكوبلت (II)،النيكل (II)،النحاس (II) و البلاديوم (II) مع اليكاند N4 -[2 -(–ايزوبيوتيل–فنيل)–بروبونيل [بريدين–4–كاربوهايدرازايد) شُخص الليكاند المحضر بواسطة التحليل الدقيق للعناصر و مطيافية الاشعة تحت الحمراء ومطيافية الرنين النووي المغناطيسي لـ^{1}H, ^{13}C اما المعقدات المحضرة فقد تم تشخيصها بواسطة مطيافية الأشعة تحت الحمراء ومطيافية الأشعة فوق البنفسجية والحساسية المغناطيسية والتوصيلية الكهربائية و الامتصاص الذري.

حيث حضر جزء من هذه المعقدات في وسط متعادل بينما الجزء الأخر في وسط قاعدي وبنسبة 2:1واثبتت النتائج ان هذه الليكاندات ترتبط بالايون الفلزي بشكل ثنائي السن حيث ترتبط هذه الليكاندات بالفلز عن طريق إحدى ذرتي النيتروجين وإحدى ذرات الأوكسجين لمجموعة الكاربونيل. كما بينت النتائج ان جميع هذه المعقدات كانت ذات اشكال ثمانية السطوح. كما بينت النتائج ان الليكاند مع معقدات Pd(II) تسلك سلوكا حادي السن في الوسط المتعادل حيث ترتبط بالبلاديوم من خلال احدى ذرات النيتروجين اما في الوسط القاعدي تسلك سلوك ثنائي السن ترتبط من خلال إحدى ذرات النيتروجين واحدى ذرات الاوكسجين لاحدى مجاميع الكاربونيل, كما بينت النتائج ان جميع معقدات البلاديوم هي ذات شكل مربع مستوي .