Synthesis and Characterization of Some Metal Complexes of [N-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylcarbamothioyl) acetamide]

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Abstract

A new ligand [N- (1,5- dimethyl -3- oxo- 2 – phenyl - 2 ,3 – dihydro -1H- pyrazol -4-ylcarbamothioyl) acetamide] (AAD) was synthesized by reaction of acetyl isothiocyanate with 4-aminoantipyrine, The ligand was characterized by micro elemental analysis C.H.N.S., FT-IR ,UV-Vis and ^{1}H-^{13}CNMR spectra, some transition metals complex of this ligand were prepared and characterized by FT-IR, UV-Vis spectra, conductivity measurements, magnetic susceptibility and atomic absorption. From the obtained results the molecular formula of all prepared complexes were $[M(AAD)_2(H_2O)_2]Cl_2$ (M^{+2} = Mn, Co, Ni, Cu, Zn, Cd and Hg),the proposed geometrical structure for all complexes were octahedral.

Key Word: 4-Aminoantipyrine, acetyl isothiocyanate, complexes.

Introduction

In recent years, there has been increasing interest in synthesis of heterocyclic compounds that have biological and commercial importance. Antipyrine compounds play an important role in modern organic synthesis, not only because they constitute a particularly useful class of heterocyclic compounds, but also because they are of great biological interest. They have been found to have biological clinical, and pharmacological activities. One of the most important derivatives of antipyrine is 4-aminoantipyrine, which is used as a synthetic intermediate to prepare poly functionally substituted heterocyclic moieties with anticipated biological activity, analgesic, antiinflammatory, antimicrobial, and anticancer activities[1,2]. Properties of 4-aminoantipyrine to coordinate with metal is varied by condensing it with aldehydes, ketones, thiosemicarbazides and carbazides etc. [3]. Ligand complexes of metals (Fe(III), Co(II), Ni(II), Cu(II) and Zn(II)) with L_1 and L_2 (L_1: obtained through the condensation of 4-aminoantipyrine with furfuraldehyde); (L_2: derived from 2-aminophenol and 3-nitrobenzaldehyde) were synthesized [4]. Noble metal complexes of Schiff bases derived from 2-thiophenecarboxaldehyde and 4-aminoantipyrine have been synthesized[5]. The aim of this work is to prepare and characterize a new ligand [N- (1,5- dimethyl -3- oxo-2 – phenyl - 2 ,3 – dihydro -1H- pyrazol -4-ylcarbamothioyl) acetamide] (AAD),and it's metal complexes with Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) ions.

Experimental

Chemicals

All reagents were analar or chemical pure grade by BDH, Merck and Fluka. All metal chloride salts and solvents were purchased from Merk and Fluka com., and used without purification.

Instruments

^{1}H and ^{13}C–NMR were recorded using Ultra Shield 300 MHz Switzerland at University of Al al-Bayt, Jordan. Melting point was recorded by using Stuart- melting point apparatus. FT-IR spectra were recorded as KBr disc using 3800 Shimadzu in the range of (4000-400) cm^{-1}. Electronic spectra were obtained using UV-160 Shimadzu spectrophotometer at 25℃ for 10^{-3} M solution DMSO with 1.000 ± 0.001 cm matched quartz cell. Molar Conductivity was measured at 25℃ for 10^{-3}M solution of DMSO by using Philips PW. Digital. micro elemental analysis (C.H.N.S) were performed using Acrlo Erba 1106 elemental analyzer. Magnetic susceptibility measurements were obtained by balance magnetic susceptibility model MSB-MKI. Metal contents of the complexes were determined by atomic absorption technique by using Shimadzu (AA680G).

Preparation of ligand (AAD)

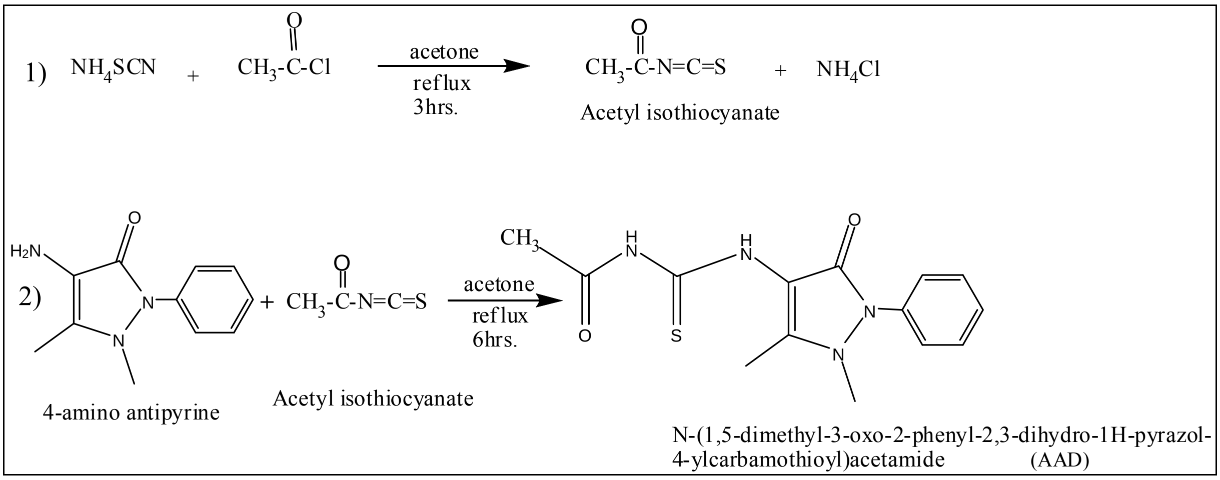
The ligand was prepared by two steps (scheme-1)

(A)- Preparation of the (Acetyl isothiocyanate)[6]

Mixture of acetyl chloride(1.86 ml,1mmol) and ammonium thiocyanate (2g, 1mmol) in (25 ml) of acetone was stirred under refluxed for 3 hrs and then filtered, the filtrate was used for further reaction.

(B)- Preparation of N-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylcarbamothioyl) acetamide (AAD)

(5.33g, 1mmol) of 4-aminoantipyrine in (20ml) acetone was rapidly added to Acetyl isothiocyanate and maintaining reflux. After refluxing for 6 hrs, the resulting solid was collected, washed with acetone and recrystallization from ethanol, yield (68%), (m.p = 218-220) ˚C, C% found (55.58) calc.(55.26), H% found (5.47) calc.(5.26), N% found (18.35) calc.(18.42), S% found (10.69) calc.(10.53).



Synthesis ligand complexes

Synthesis of the $[Cu(AAD)_2(H_2O)_2]Cl_2$ complex

A solution of (0.17g, 1mmol) $CuCl_2.2H_2O$ in (10ml) ethanol was added to solution of (0.61g, 2mmol) (AAD) in (10ml) ethanol. The mixture was stirred for 6 hours at room temperature, the brown solid was collected by filtration, washed with (1:1) mixture of water: ethanol, recrystallized from ethanol and dried in an oven (50˚C).

Synthesis of $[Mn(AAD)_2(H_2O)_2]Cl_2$, $[Co(AAD)_2(H_2O)_2]Cl_2$, $[Ni(AAD)_2(H_2O)_2]Cl_2$, $[Zn(AAD)_2(H_2O)_2]Cl_2$, $[Cd(AAD)_2(H_2O)_2]Cl_2$, $[Hg(AAD)_2(H_2O)_2]Cl_2$complexes.

A similar method to that mentioned for preparation of $[Cu(AAD)_2(H_2O)_2]Cl_2$ complex was used to prepare the complexes of [Mn^{+2},Co^{+2},Ni^{+2},Zn^{+2},$Cd^{+2}$and Hg^{+2}] ions with (AAD), Table (1) showed some physical properties of the prepared complexes.

Results and Discussion

Ligand (AAD)

The FT-IR spectrum of the free ligand (AAD) ,Fig.(3) showed bands at(1635) cm^{-1},(1600) cm^{-1} and (1367) cm^{-1} due to υ C=O(in ring) , υ C=O(amidic) and υ C=S respectively. While another absorption band at (3138) cm^{-1} could be explained as υ N-H [7-9]. The FT-IR spectral data of the free ligand were listed in table(2).

The UV-Vis spectrum of the free ligand (AAD). Fig.(5) exhibits a high intense absorption peak at (33444) cm^{-1} which may be attributed to electronic transition type $\pi \longrightarrow \pi^*$[10].The data of electronic spectrum of the free ligand (AAD) were listed in table (3).

The ^{1}H-NMR spectrum of free ligand (AAD),Fig.(1) which was recorded in DMSO-d_6 solvent showed the following signals: singlet at δ(1.93-1.95) ppm refers to (3H, CH_3), singlet at δ(2.36-2.38) ppm refers to (3H, CH_3CO), singlet at δ(3.10) ppm refers to (3H,CH_3N), singlet at δ(3.34) ppm refers to (1H, NH sec. amine), the multiplet signals δ(7.31-7.53) ppm were attributed to aromatic protons, singlet at δ(11.37) ppm refers to (1H,NHsec. amide).

^{13}CNMR spectrum of the free ligand (AAD),Fig.(2)showed chemical shift at δ(11.96) ppm refers (CH_3) for (CH_3CO) group, signal at δ(24.21) ppm refers to (CH_3CO), signal at δ(36.13) ppm for (CH_3N), signals at δ(39.22-41.48) ppm for DMSO, signal at δ(108.53) ppm refers to (**C**=C-CH_3), The chemical shifts at range δ(123.85-129.95) ppm due to aromatic carbons, signal at δ(135.47) ppm for (=C-N), signal at δ(153.47) ppm for (=C-N, aromatic carbons), while the signals at Fig. (2) showed the following signals at δ(161.55) ppm , δ(173.05) ppm and δ(182.32) ppm were attributed to ((C=O), antipyrine ring), (CONH) and (C=S) [11,12].

Complexes of the ligand (AAD)

The solid complexes soluble in some common solvent such as dimethylformamide, dimethylsulphoxide and relatively thermally stable. The molar conductivity values of all complexes in DMSO solvent in 10^{-3} M at 25˚C (table-1) indicated electrolyte nature with 1:2 ratio [13].The atomic absorption measurements for all complexes gave approximated values when its comparison with theoretical values, Table (1) includes the physical properties for the ligand and its complexes.

FT-IR Spectra

These spectra exhibited marked difference between bands Fig. (4) belonging to the stretching vibration of υ(C=O amido) in the range between (1635-1620) cm^{-1} shifted higher frequencies by (27-20) cm^{-1} suggesting of the possibility of the coordination of ligand through the oxygen atom at the carbonyl group[14] while the band caused by υ(C=S) appeared between (1434-1411) cm^{-1} shifted to higher frequencies by (67-44) cm^{-1} which indicates to the coordination of ligand through the sulfur atom at the thiol group to the central ion[15]. The stretching vibration band υ(C=O in ring), υ(N-H) either show no change or very little in their frequencies (1635-1650) cm^{-1}, (3147-3186) cm^{-1} respectively there for indicating do not coordinate to the metal ion. Metal-oxygen and metal-sulfur bonds were confirmed by the presence of the stretching tremor of υ(M-O) and υ(M-S) around (501-455) cm^{-1} and (447-423) cm^{-1} respectively the spectra of complexes showed the appearance of bands in the range (918-805) cm^{-1} attributed to υ(OH), these bands confirm the coordination of the water with metal[14], Table (2) describes the important bands and assignment for all prepared complexes.

Magnetic Moment

The values of measured magnetic susceptibility and effective magnetic moment (μeff) for the Mn(II), Co(II), Ni(II), Cu(II)complexes are shown in table(1). Mn(II), Co(II), Ni(II) and Cu(II) complexes exhibit μeff (5.94, 4.83, 3.05, 1.76) B.M respectively ,which can be a normal values for high spin octahedral complexes.[16]

Electronic Spectra for Complexes

-$[Mn(AAD)_2(H_2O)_2]Cl_2$ d^5

The Brown complex of Mn(II) shows band at (33557) cm^{-1} due to ligand field and other bands at (14084) cm^{-1} and (10729) cm^{-1} which are caused by the electronic transfer $^6A_1g \longrightarrow {}^4T_2g_{(G)}$ and $^6A_1g \longrightarrow {}^4T_1g_{(G)}$ respectively, suggesting octahedral geometry around Mn(II) ion [17].

-$[Co(AAD)_2(H_2O)_2]Cl_2$ d^7

The spectrum of the Violet complex gave four bands at (33783) cm^{-1}, (27624) cm^{-1}, (14662) cm^{-1} and (12048) cm^{-1} attributed to (L.F),C.T mixed with $^4T_1g_{(F)} \longrightarrow {}^4T_1g_{(P)}$, $^4T_1g_{(F)} \longrightarrow {}^4A_2g$ and $^4T_1g_{(F)} \longrightarrow {}^4T_2g_{(F)}$ respectively and the rach interelectronic repulsion parameter (B^-) was found to be (409.46) cm^{-1}, from the relation $\beta = B^- / B^0$, was found to be equal (0.42), these parameter are accepted to Co(II) octahedral complex[18].

-$[Ni(AAD)_2(H_2O)_2]Cl_2$ d^8

The spectrum of green complex of Ni(II) has revealed the following electronic transfer (L.F),C.T mixed with $^3A_2g \longrightarrow {}^3T_1g_{(P)}$, $^3A_2g \longrightarrow {}^3T_1g_{(F)}$, and $^3A_2g \longrightarrow {}^3T_2g_{(F)}$, transition at (38610) cm^{-1}, (27322) cm^{-1}, (22123) cm^{-1} and (12515) cm^{-1}respectively, the (B^-) value is found to be (793) cm^{-1}, while β was equal to (0.76) these are the characteristics for octahedral complexes of Ni(II)[19].

-[Cu $(AAD)_2(H_2O)_2]Cl_2$ d^9

The spectrum of Brown complex of Cu(II) Fig. (6) shows two bands at (33898) cm^{-1}, (11750) cm^{-1} caused to (C.T), $^2Eg \longrightarrow {}^2T_2g$ transition respectively , which was a good agreement for distorted octahedral complex for Cu(II) ion [20,21].

-The Complexes of $[Zn(AAD)_2(H_2O)_2]Cl_2$,$[Cd(AAD)_2(H_2O)_2]Cl_2$ and **$[Hg(AAD)_2(H_2O)_2]Cl_2$** show only charge transfer of (M→L) in range (38610-27322) cm^{-1}[22,23]. All transition with their assignments are summarized in Table (3).

Suggested structures for complexes on the basis of molar conductivity, magnetic moment, spectroscopic studies(FT-IR,UV-Vis and atomic absorption) and (^1H-^{13}C NMR for ligand(AAD)only)for the ligand and all prepared complexes, we suggested that the ligand (AAD) behaves as bidentate on coordination with Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) ions via oxygen atom of (C=O) amido group and sulfer atom of (C=S) group, suggesting octahedral geometry around metal ions for all prepared complexes.

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Table No. (1): Some physical properties of the ligand(AAD) and its complexes

dec =decomp

Compound	**M.wt (gm/mole)**	**Color**	**M.P(℃) or dec.**	**M% Calculation (Found)**	**Molar Cond. Ohm^{-1}cm^{2}mol^{-1} in DMSO**	**μ_{eff} (B.M)**
$C_{14}H_{16}N_2O_4S$ AAD	304	White	218-220℃	–	1.67	–
$[Mn(AAD)_2(H_2O)_2]Cl_2$	768.94	Brown	175-177℃	7.14 (7.26)	78	5.94
$[Co(AAD)_2(H_2O)_2]Cl_2$	772.93	Violet	139-141℃	7.62 (7.46)	79.11	4.83
$[Ni(AAD)_2(H_2O)_2]Cl_2$	772.71	Green	164-166℃	7.59 (8.12)	76	3.05
$[Cu(AAD)_2(H_2O)_2]Cl_2$	777.54	Brown	136 dec.	8.17 (8.62)	71.2	1.76
$[Zn(AAD)_2(H_2O)_2]Cl_2$	779.38	Brown	164-166℃	8.39 (8.19)	83	0
$[Cd(AAD)_2(H_2O)_2]Cl_2$	826.41	Brown	230 dec.	13.60 (13.77)	82	0
$[Hg(AAD)_2(H_2O)_2]Cl_2$	914.59	Brown	158-160℃	21.93 (22.13)	72	0

Table No. (2): The characteristic infrared band for free ligand (AAD) and its metal complexes

Com.	υ(N-H)	υ(C=O) in Ring	υ(C=O) Amide	υ(C=S)	υ(O-H)	υ(M-O)	υ(M-S)
Ligand AAD	3138 (s)	1635 (s)	1600(S)	1367(S)	—	—	—
$[Mn(AAD)_2(H_2O)_2]Cl_2$	3147(s)	1635(s)	1620(m)	1434(w)	856(s)	470(m)	425(w)
$[Co(AAD)_2(H_2O)_2]Cl_2$	3178(m)	1635(w)	1620(s)	1419(m)	825(m)	455(m)	425(w)
$[Ni(AAD)_2(H_2O)_2]Cl_2$	3147(m)	1635(s)	1623(m)	1411(m)	856(s)	501(m)	439(m)
$[Cu(AAD)_2(H_2O)_2]Cl_2$	3170(m)	1635(w)	1620(w)	1411(s)	918(s)	501(m)	447(w)
$[Zn(AAD)_2(H_2O)_2]Cl_2$	3155(m)	1650(s)	1635(w)	1419(s)	833(s)	473(s)	425(m)
$[Cd(AAD)_2(H_2O)_2]Cl_2$	3186(s)	1635(s)	1620(m)	1419(s)	805(s)	475(m)	423 (m)
$[Hg(AAD)_2(H_2O)_2]Cl_2$	3155(s)	1635(w)	1623(s)	1434(s)	870(s)	470(m)	425(w)

Table No. (3): The peaks electronic transitions and structure geometries of the ligand (AAD) and its complexes

Compounds	λ_{max} nm	Wave number cm^{-1}	ABS	ε_{max} $molar^{-1}cm^{-1}$	Transitions
Ligand AAD	299	33444	2.392	2392	$\pi \longrightarrow \pi^*$
$[Mn(AAD)_2(H_2O)_2]Cl_2$	298	33557	2.301	2301	L.F
	710	14084	0.010	10	${}^6A_{1g} \longrightarrow {}^4T_{2g(G)}$
	932	10729	0.015	15	${}^6A_{1g} \longrightarrow {}^4T_{1g(G)}$
$[Co(AAD)_2(H_2O)_2]Cl_2$	296	33783	2.119	2119	L.F
	362	27624	0.432	432	C.T mixed with ${}^4T_{1g(F)} \longrightarrow {}^4T_{1g(P)}$
	682	14662	0.033	33	${}^4T_{1g(F)} \longrightarrow {}^4A_{2g}$
	830	12048	0.010	10	${}^4T_{1g(F)} \longrightarrow {}^4T_{2g(F)}$
$[Ni(AAD)_2(H_2O)_2]Cl_2$	259	38610	1.484	1484	L.F
	366	27322	1.623	1623	C.T mixed with ${}^3A_{2g} \longrightarrow {}^3T_{1g(P)}$
	452	22123	0.391	391	${}^3A_{2g} \longrightarrow {}^3T_{1g(F)7}$
	799	12515	0.091	91	${}^3A_{2g} \longrightarrow {}^3T_{2g(F)}$
$[Cu(AAD)_2(H_2O)_2]Cl_2$	295	33898	2.051	2051	C.T
	851	11750	0.017	17	${}^2Eg \longrightarrow {}^2T_{2g}$
$[Zn(AAD)_2(H_2O)_2]Cl_2$	295	33898	2.015	2015	C.T
$[Cd(AAD)_2(H_2O)_2]Cl_2$	298	33557	2.211	2211	C.T
$[Hg(AAD)_2(H_2O)_2]Cl_2$	259	38610	1.483	1483	C.T
	366	27322	1.730	1730	C.T

C.T = Charge transfer

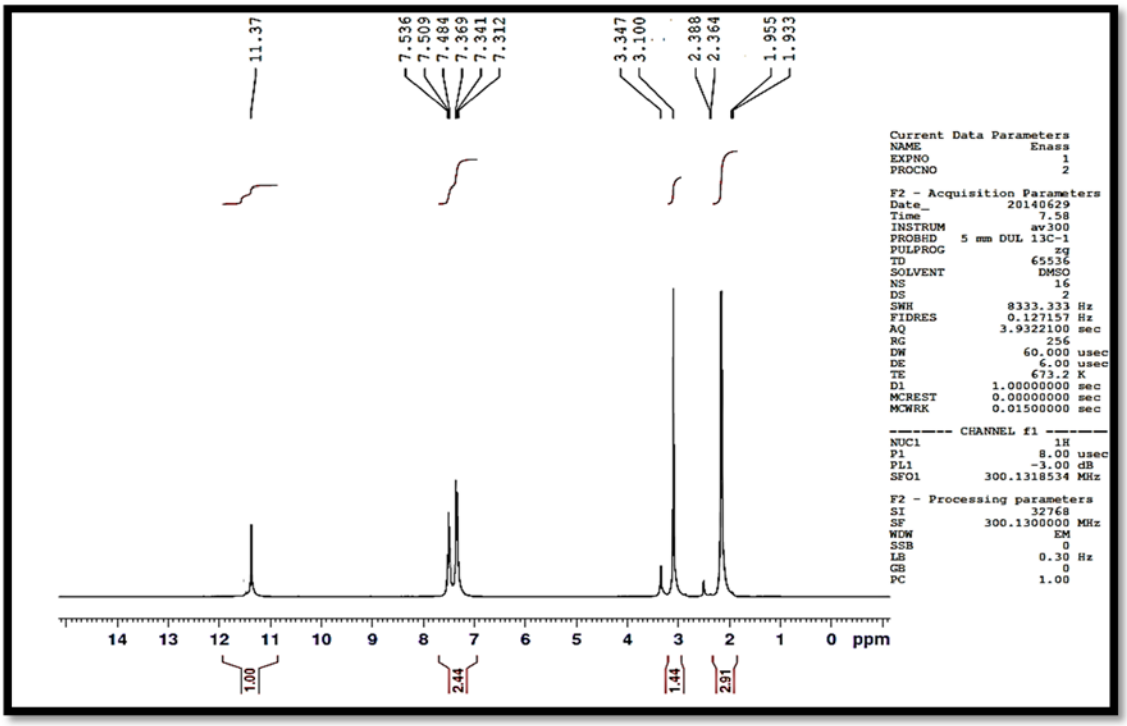

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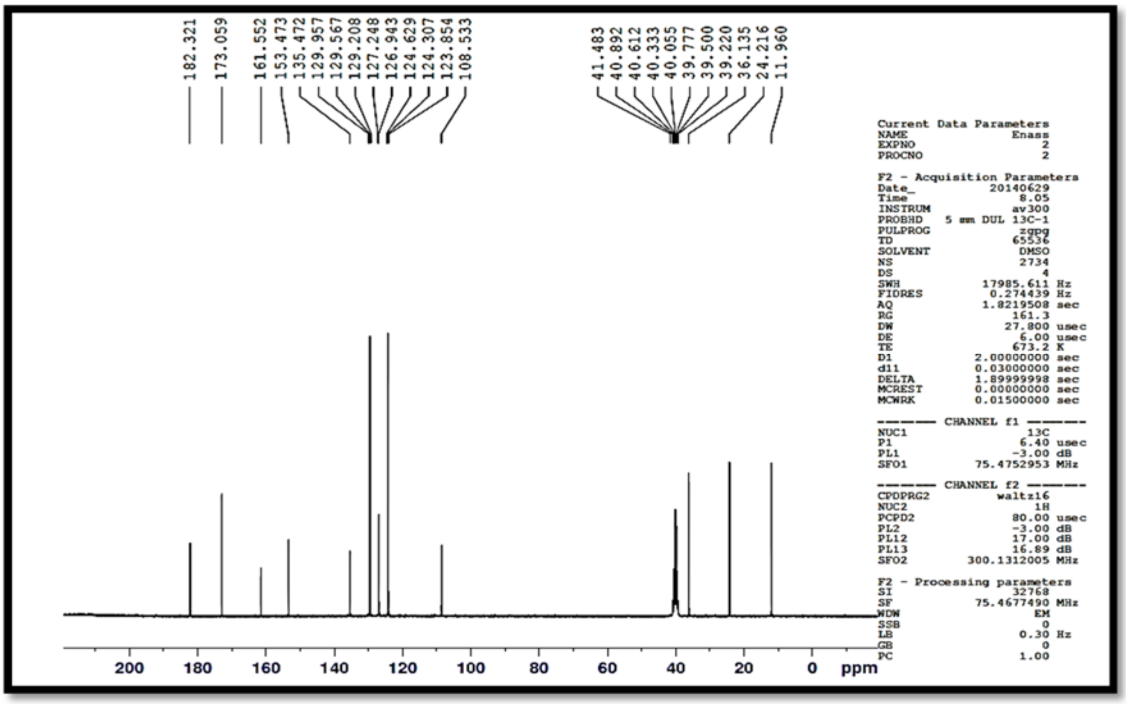
Figure No. (1): ^{1}H-NMR spectrum of ligand (AAD)



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SFO1           75.4752953 MHz

======== CHANNEL f2 ========
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Figure No. (2): ^{13}C-NMR spectrum of ligand (AAD)

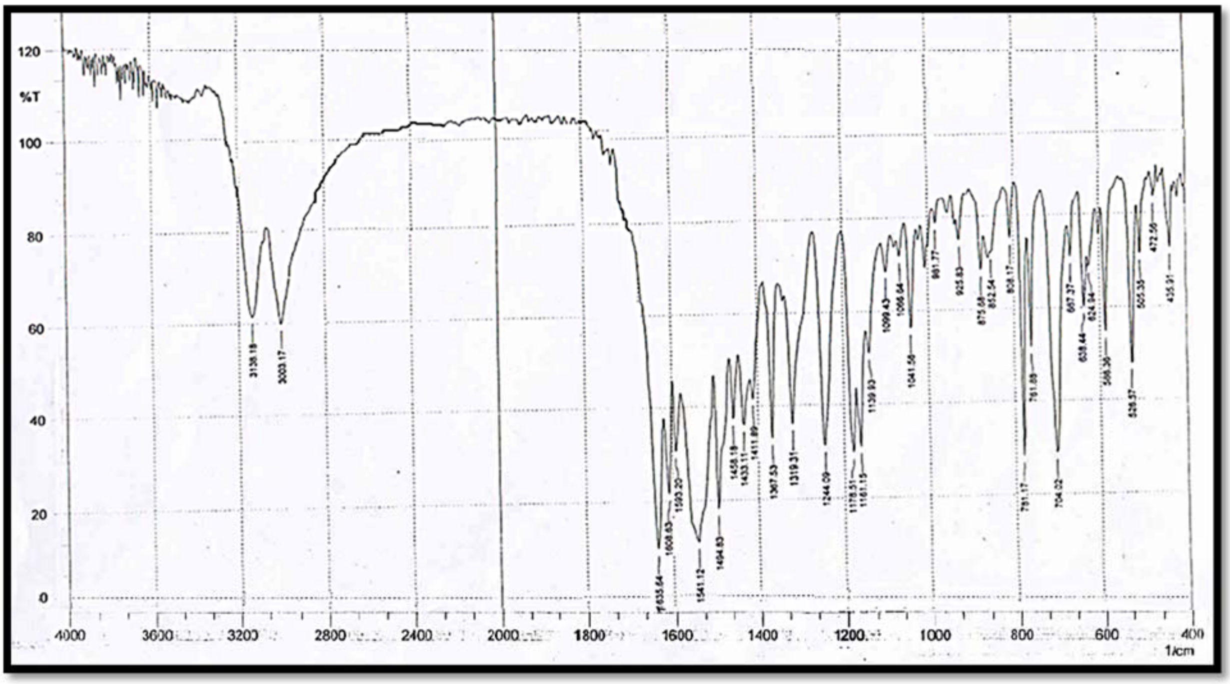


Figure No. (3): Infrared spectrum of ligand (AAD)

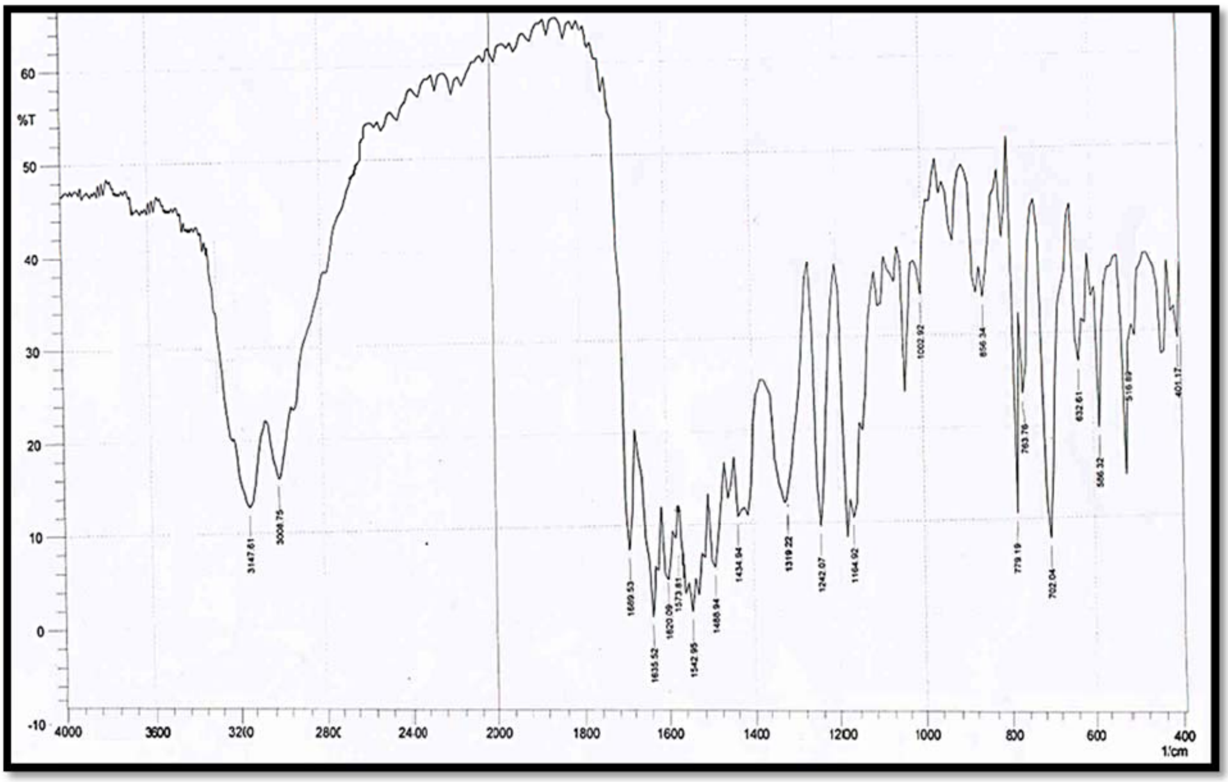


Figure No. (4): Infrared spectrum of complex [Mn $(AAD)_2(H_2O)_2]Cl_2$

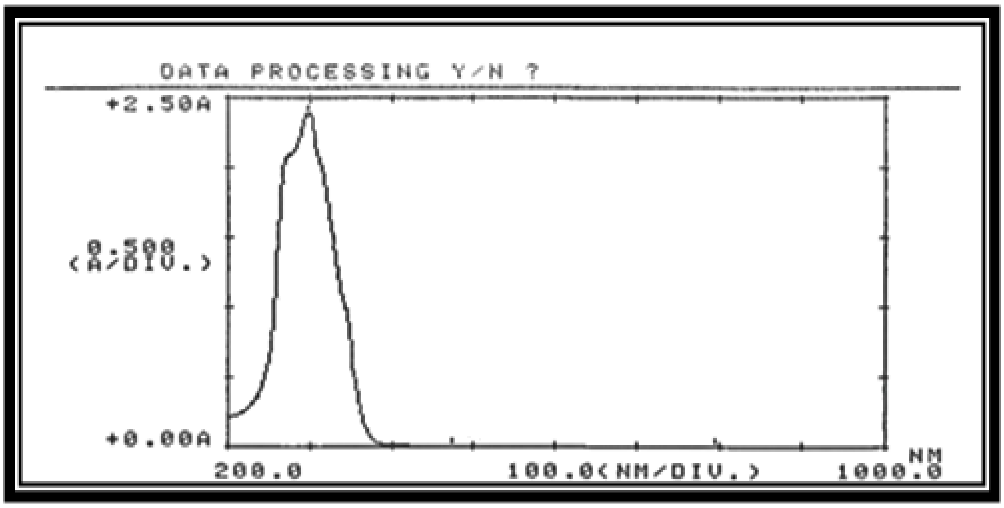


Figure No. (5): U.V. spectrum of ligand (AAD)

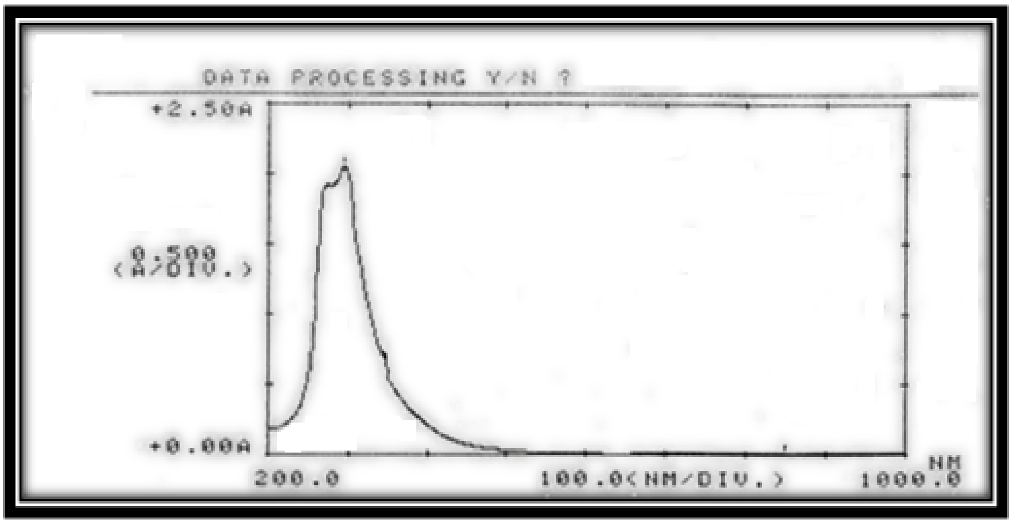
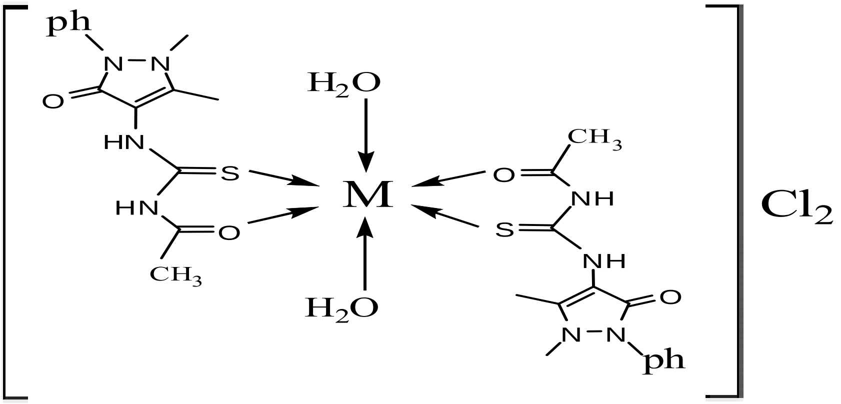


Figure No. (6): U.V. spectrum of complex $[Cu(AAD)_2(H_2O)_2]Cl_2$



M^{+2} =[Mn, Co, Ni, Cu, Zn, Cd and Hg]

Figure No. (7): The proposed chemical structure formula of the complexes

تحضير وتشخيص بعض المعقدات الفلزية مع N- (١،٥- ثنائي مثيل-٣- اوكسو-٢-فنيل-٢،٣- ثنائي هايدرو-١- بايرازول-٤- يل كاربامو ثايويل) استمايد

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استلم البحث في: ٢٠ كانون الثاني ٢٠١٥ ،قبل البحث: ١٥ اذار ٢٠١٥

الخلاصة

حضر الليكاند الجديد N- (١،٥- ثنائي مثيل-٣- اوكسو-٢- فنيل-٢،٣- ثنائي هايدرو-١- بايرازول-٤- يل كاربامو ثايويل) استمايد] (AAD) وذلك من مفاعله (استيل ايزوثايوسيانات) مع ٤- امينو أنتي بايرين وبنسبة (١:١) وشخص بوساطة التحليل الدقيق للعناصر (CHNS) والأشعة تحت الحمراء والأشعة فوق البنفسجية- المرئية وطيف الرنين النووي المغناطيسي ، كما حضرت وشخصت معقدات أملاح بعض ايونات العناصر الانتقالية الثنائية التكافؤ (Hg, Cd, Zn, Cu, Ni, Co, Mn) مع الليكاند (AAD) وشخصت المعقدات المحضرة باستعمال الأشعة تحت الحمراء والأشعة فوق البنفسجية - المرئية والتوصيلية المولارية والحساسية المغناطيسية والامتصاص الذري واستنتج من الدراسات والتشخيصات إن المعقدات لها شكل ثماني السطوح حول الايون الفلزي مع اللكياند (AAD) ثنائي السن.

الكلمات المفتاحية: ٤-امينو انتيبايرين، استيل ايزوثايوسيانات، معقدات.