

**Synthesis And Characterization Of Some New Metals Complexes Of
[N-(3-acetylphenylcarbamothioyl)-4-methoxybenzamide]
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Abstract

A new ligand [N-(3-acetylphenylcarbamothioyl)-4-methoxybenzamide](MAA) was synthesized by reaction of 4-methoxybenzoylisothiocyanate with 3-aminoacetophenone, The ligand was characterized by elemental microanalysis C.H.N.S, FT-IR, UV-Vis and ^1H , ^{13}C -NMR spectra, some transition metals complexes of this ligand were prepared and characterized by FT-IR, UV-Vis spectra, conductivity measurements, magnetic susceptibility and atomic absorption, From obtained results the molecular formula of all prepared complexes were $[\text{M}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$ ($\text{M}^{+2} = \text{Mn, Co, Ni, Cu, Zn, Cd}$ and Hg), the proposed geometrical structure for all complexes were octahedral.

Key Word: 3-Aminoacetophenon, 4-methoxybenzoylisothiocyanate, complexes.

تحضير وتشخيص بعض المعقدات الفلزية الجديدة مع [N-(3-اسيتايل فنييل كارباموثايويل)-4-

ميثوكسيبنزمايد

باسمة محسن سرحان، ساجد محمود لطيف، ايناس جاسم وحي
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الخلاصة

حضر الليكاند الجديد [N-(3-اسيتايل فنييل كارباموثايويل)-4-ميثوكسيبنزمايد] (MAA) وذلك من مفاعله (4-كلوروبنزويلايزوثايوسيانات) مع 3-امينو اسيتوفينون وبنسبة (1:1) وشخص بوساطة التحليل الدقيق للعناصر (S.N.H.C) والأشعة تحت الحمراء والأشعة فوق البنفسجية- المرئية وطيف الرنين النووي المغناطيسي، كما حضرت وشخصت معقدات أملاح بعض ايونات العناصر الانتقالية الثنائية التكافؤ (Mn, Co, Ni, Cu, Zn, Cd, Hg) مع الليكاند (MAA) وشخصت المعقدات المحضرة باستخدام 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 الليكاند (MAA) ثنائي السن.

الكلمات المفتاحية: 3-امينو اسيتوفينون، 4-كلوروبنزويل ايزوثايوسيانيت، معقدات

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Introduction

3-Aminoacetophenone derived from aromatic amines have a wide variety and an important class of ligands in coordination chemistry and find extensive applications in different fields, e.g., biological, inorganic and analytical chemistry. Many biologically important acetophenone derivative have been reported in the literature possessing, antimicrobial antibacterial, antifungal, anti inflammatory antitumor and anti HIV activities[1]. Transition metals and their complexes are used in various industries such as leather tanning, electroplating, metal finishing, chemicals manufacturing etc. Thus these metals enter in water supply through industrial waste [2]. The Schiff bases derived from thiazole and substituted acetophenone have been widely used as ligand for the synthesis of transition metal complexes[3]. Metal complexes have been prepared by interacting acetophenone derivative with metal ions Ni(II), Cu(II) 1:2 (metal:ligand) ratio, also it was prepared by conventional as well as microwave methods [4]. A series of metal complexes of Co(II), Ni(II), Cu(II) and Zn(II) with 3-aminoacetophenone derivative have been synthesized[5]. New ligand was prepared by the reaction of 3-aminoacetophenone with salicylaldehyde. Also its complexes were prepared which have molecular formula $[ML_2(H_2O)_2]$, (where $M^{2+} = Mn, Fe, Co, Ni$ and Cu)[6]. The aim of this work to prepare new ligand [N-(3-acetyl phenyl carbamothioyl)-4-methoxybenzamide] (MAA), and its 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 purchased from Merk and Fluka com., and were used without purification.

Instruments : 1H and ^{13}C -NMR was recorded using Ultra Shield 300 MHz Switzerl and 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 $25^\circ C$ for $10^{-3}M$ solution DMSO with 1.000 ± 0.001 cm matched quartz cell. Molar Conductivity was measured at $25^\circ C$ for $10^{-3}M$ solution of DMSO by using Philips

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PW.Digital.Elemental micro analyses(C.H.N.S) were performed using Acro Erba 1106elemental analyzer. Magnetic susceptibility measurements were obtained by balance magnetic susceptibilityby model MSB-MKI. Metal contents of the complexes were determined by atomic absorption technique by using Shimadzu (AA680G).

Preparation of ligand(MAA)

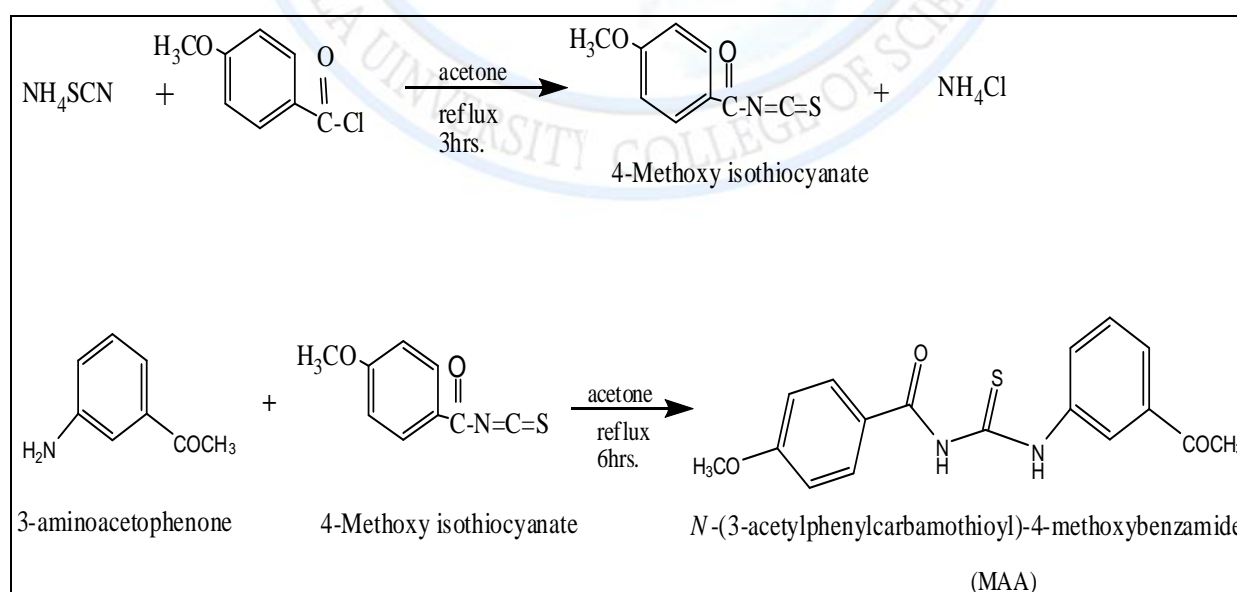
The ligand was prepared by two steps(scheme-1)

(A)- Preparation of the (4-methoxybenzoyl isothiocyanate)

Mixture of 4-methoxybenzoylchloride (3.55ml,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[7].

(B)- Preparation of N-(3-acetylphenylcarbamoithioyl)-4-methoxy benzamide (MAA)

(3.60g, 1mmol) of 3-aminoacetophenon in (20ml) acetone was rapidly added to 4-methoxybenzoyl isothiocyanate and maintaining reflux. After refluxing for 6hrs, the resulting solid was collected, washed with acetone and recrystallization from ethanol, Yield(81%), (m.p=163-165)^oC, C% found (61.93) calc.(62.20), H% found (4.53), calc.(4.88),N%found (8.50), calc.(8.54),S% found (10.47), calc.(9.76).



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Synthesis ligand (MAA) complexes

Synthesis of the $[\text{Ni}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$ complex

A solution of (0.237g, 1mmol) $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ in (10ml) ethanol was added to solution of (0.66g, 2mmol) (MAA) in (10ml) ethanol. The mixture was stirred for 6 hours at room temperature, the green 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 $[\text{Mn}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$, $[\text{Co}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$, $[\text{Cu}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$, $[\text{Zn}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$, $[\text{Cd}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$, $[\text{Hg}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$ complexes

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

Results and Discussion

Ligand (MAA)

The FT-IR spectrum of the free ligand (MAA), fig.(3) showed bands at $(1681)\text{ cm}^{-1}$, $(1602)\text{ cm}^{-1}$ and $(1340)\text{ cm}^{-1}$ due to $\nu\text{C}=\text{O}$ (ketonic), $\nu\text{C}=\text{O}$ (amidic) and $\nu\text{C}=\text{S}$ respectively. While another absorption band at $(3441)\text{ cm}^{-1}$ could be explained as $\nu\text{ N-H}$ [8-10], The FT-IR spectral data of the free ligand were listed in table(2).

The UV-Vis spectrum of the free ligand (MAA). Fig.(5) exhibits a high intense absorption peak at $(34364)\text{ cm}^{-1}$ which was attributed to electronic transition type $\pi \longrightarrow \pi^*$ [11,12]. The data of electronic spectrum of the free ligand (MAA) were listed in table (3).

The $^1\text{H-NMR}$ spectrum of free ligand (MAA), Fig.(1) which was recorded in DMSO-d_6 solvent showed the following signals: The singlet at $\delta(2.50)$ ppm refer to DMSO, singlet signal at $\delta(2.60)$ ppm due to (3H, CH_3CO), the singlet at $\delta(3.33)$ ppm refer to (3H, CH_3O), the singlet signal at $\delta(3.86)$ ppm due to (1H, NH sec amine), the multiplet signals at $\delta(6.81-8.27)$ ppm were attributed to aromatic protons, the singlet signals at $\delta(11.65)$ ppm and at $\delta(11.46)$ ppm refer to (2H, NH sec amide).

$^{13}\text{C-NMR}$ spectrum of the free ligand (MAA), Fig.(2) showed chemical shift at $\delta(26.37)$ ppm refers (CH_3) for (CH_3CO) group. Chemical shifts at $\delta(38.74-41.34)$ ppm refer to DMSO.

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signal at δ (55.59)ppm was attributed to (CH₃O).The chemical shifts at range δ (113.83-163.29)ppm due to aromatic carbons. while the signals at Fig. (2) showed the following signals: signal at for(CH₃CO), signals at δ (167.41)ppm, δ (179.69)ppm and δ (197.27)ppm were attributed to (CONH),(C=S) and CH₃for (CH₃CO) group [13,14].

Complexes of the ligand (MAA)

The solidcomplexessoluble in some common solvent such as dimethylformamide, dimethylsulphoxide and relatively thermally stable. The molar conductivity values of allcomplexes in DMSO solvent in 10⁻³M at 25°C(table-1)indicated electrolyte nature with 1:2 ratio [15].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 ν (C=O amido) in the range between (1570-1529)cm⁻¹ shifted lower frequencies by(73-32)cm⁻¹ suggesting of the possibility of the coordination of ligand through the oxygen atom at the carbonyl group[16]while the band caused by ν (C=S) appeared between (1435-1400)cm⁻¹ shifted to higher frequencies by (95-60)cm⁻¹ which indicates to the coordination of ligand through the sulfur atom at the thiol group to the central ion[17]. The stretching vibration band ν (C=O ketone) and ν (N-H) either show no change or very little in their frequencies (1689-1670) cm⁻¹ and (3450-3271) 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 vibration of ν (M-O) and ν (M-S) around (509-439)cm⁻¹ and (425-405)cm⁻¹ respectively the spectra of complexes showed the appearance of bands in the range(840-804) cm⁻¹ attributed to ν (OH),These bands confirm the coordination of the water with metal[16],Table (2) describe the important bands and assignment for all prepared complexes.

Magnetic moment

The values of measured magnetic susceptibility and effective magnetic moment (μ_{eff}) 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 μ_{eff} (5.92, 4.82, 2.95, 1.79) B.M respectively ,which can be a normal values for high spin octahedral complexes[18].

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Electronic spectra of complexes

-[Mn(MAA)₂(H₂O)₂]Cl₂

The Brown complex of Mn(II) Fig. (6) shows band at (35211)cm⁻¹ due to ligand field and another bands at (11299) cm⁻¹ and (10384)cm⁻¹ which are caused by the electronic transfer ${}^6A_{1g} \longrightarrow {}^4T_{1g(G)}$ and ${}^6A_{1g} \longrightarrow {}^4T_{2g(G)}$ respectively, suggesting octahedral geometry around Mn(II) ion[19].

-[Co(MAA)₂(H₂O)₂]Cl₂

The spectrum of the green complex gave four bands at (33898)cm⁻¹, (26455)cm⁻¹, (16286)cm⁻¹ and (12360)cm⁻¹ attributed to (L.F),C.T 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 Racah parameter (B⁻) was found to be (377.4)cm⁻¹, from the relation $\beta = B^- / B^{\circ}$, was found to be equal (0.40), these parameter are accepted to Co(II) octahedral complex[20].

-[Ni(MAA)₂(H₂O)₂]Cl₂

The spectrum of green complex of Ni(II) has revealed the following electronic transfer (L.F),C.T with ${}^3A_{2g} \longrightarrow {}^3T_{1g(P)}$, ${}^3A_{2g} \longrightarrow {}^3T_{1g(F)}$, and ${}^3A_{2g} \longrightarrow {}^3T_{2g(F)}$, transition at (35587)cm⁻¹, (28089)cm⁻¹, (14388)cm⁻¹ and (12376)cm⁻¹ respectively, the (B⁻) value found to be (356.6)cm⁻¹, while β was equal to (0.34) these are the characteristics for octahedral complexes of Ni(II)[21,22].

-[Cu(MAA)₂(H₂O)₂]Cl₂

The spectrum of brown complex of Cu(II) show two bands at (35842)cm⁻¹, (14347)cm⁻¹ caused to (C.T), ${}^2E_g \longrightarrow {}^2T_{2g}$ transition respectively, which was a good agreement for distorted octahedral complex for Cu(II) ion[23,24].

-[Zn(MAA)₂(H₂O)₂]Cl₂, [Cd(MAA)₂(H₂O)₂]Cl₂ and [Hg(MAA)₂(H₂O)₂]Cl₂

Shows only charge transfer of (M→L) in range (35714-34246)cm⁻¹[25,26]. 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-¹³C NMR for ligand(MAA) only) for the ligand and all prepared complexes, we suggested that the ligand

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(MAA) 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 sulfur atom of (C=S) group, suggesting octahedral geometry around metal ions for all prepared complexes.

Conclusions

The new ligand in this work has been readily prepared by reaction from 4-methoxybenzoyl isothiocyanate with 3-aminoacetophenone. The ligand was characterized by elemental micro analysis C.H.N.S, FT-IR, UV-Vis and ^1H , ^{13}C -NMR spectra. The metal complexes of this ligand were prepared and characterized by FT-IR, UV-Vis spectra, conductivity measurements, magnetic susceptibility and atomic absorption, the proposed geometrical structure for all complexes were octahedral.

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

Compound	M.wt (gm/mole)	Color	M.p °C or dec.	M% Calculation (Found)	Molar Cond. Ohm ⁻¹ cm ² Mol ⁻¹ in DMSO	μ _{eff} (B.M)
Ligand (MAA)	328	White	163-165 °C	–	3.6	–
[Mn(MAA) ₂ (H ₂ O) ₂]Cl ₂	816.94	Brown	175-177 °C	6.72 (6.86)	68.69	5.92
[Co(MAA) ₂ (H ₂ O) ₂]Cl ₂	820.93	Green	139-141 °C	7.13 (6.95)	81.65	4.82
[Ni(MAA) ₂ (H ₂ O) ₂]Cl ₂	820.71	Green	164-166 °C	7.15 (7.36)	77.03	2.95
[Cu(MAA) ₂ (H ₂ O) ₂]Cl ₂	825.54	Brown	136 dec	7.69 (7.28)	78.70	1.79
[Zn(MAA) ₂ (H ₂ O) ₂]Cl ₂	827.38	Brown	164-166 °C	7.90 (8.01)	69.72	0
[Cd(MAA) ₂ (H ₂ O) ₂]Cl ₂	874.41	Brown	230 dec	12.86 (12.56)	83.42	0
[Hg(MAA) ₂ (H ₂ O) ₂]Cl ₂	962.59	Brown	158-160 °C	20.84 (20.23)	71.5	0

dec =decomp

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

Com.	$\nu(\text{N-H})$	$\nu(\text{C=O})$ Ketone	$\nu(\text{C=O})$ Amide	$\nu(\text{C=S})$	$\nu(\text{O-H})$	$\nu(\text{M-O})$	$\nu(\text{M-S})$
Ligand MAA	3441 (s)	1681 (s)	1602(s)	1340(s)	–	–	–
$[\text{Mn}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$	3415(s)	1681(s)	1529(s)	1433(m)	840(s)	470(w)	425(w)
$[\text{Co}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$	3317(s)	1681(s)	1566(m)	1434(m)	840(s)	470(w)	420(w)
$[\text{Ni}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$	3423(m)	1681(w)	1570(w)	1400(w)	840(m)	484(w)	405(w)
$[\text{Cu}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$	3450(s)	1670(w)	1535(m)	1400(m)	824(m)	447(w)	416(w)
$[\text{Zn}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$	3317(s)	1680(s)	1566(S)	1434(s)	840(s)	470(w)	420(w)
$[\text{Cd}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$	3271(S)	1689(S)	1566(S)	1434(S)	820(s)	439(w)	408(w)
$[\text{Hg}(\text{MAA})_2(\text{H}_2\text{O})_2]\text{Cl}_2$	3342(w)	1681(S)	1552(s)	1435(w)	804(s)	509(w)	425(w)

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

Compounds	λ (nm)	ν (cm^{-1})	ABC	ϵ max	Transitions
Ligand MAA	291	34364	2.399	2399	$\pi \longrightarrow \pi^*$
[Mn(MAA) ₂ (H ₂ O) ₂]Cl ₂	284	35211	1.949	1949	L.F
	885	11299	0.024	24	${}^6A_{1g} \longrightarrow {}^4T_{1g}(G)$
	963	10384	0.024	24	${}^6A_{1g} \longrightarrow {}^4T_{2g}(G)$
[Co(MAA) ₂ (H ₂ O) ₂]Cl ₂	295	33898	2.448	2448	L.F
	378	26455	1.739	1739	C.T mixed with ${}^4T_{1g}(F) \longrightarrow {}^4T_{1g}(P)$
	614	16286	0.757	757	${}^4T_{1g}(F) \longrightarrow {}^4A_{2g}$
	809	12360	0.021	21	${}^4T_{1g}(F) \longrightarrow {}^4T_{2g}(F)$
[Ni(MAA) ₂ (H ₂ O) ₂]Cl ₂	281	35587	2.253	2253	L.F
	356	28089	0.018	18	C.T mixed with ${}^3A_{2g} \longrightarrow {}^3T_{1g}(P)$
	695	14388	0.015	15	${}^3A_{2g} \longrightarrow {}^3T_{1g}(F)$
	808	12376	0.010	10	${}^3A_{2g} \longrightarrow {}^3T_{2g}(F)$
[Cu(MAA) ₂ (H ₂ O) ₂]Cl ₂	279	35842	2.012	2012	C.T
	697	14347	0.017	17	${}^2E_g \longrightarrow {}^2T_{2g}$
[Zn(MAA) ₂ (H ₂ O) ₂]Cl ₂	292	34246	2.477	2477	C.T
[Cd(MAA) ₂ (H ₂ O) ₂]Cl ₂	281	35587	2.327	2327	C.T
[Hg(MAA) ₂ (H ₂ O) ₂]Cl ₂	280	35714	2.249	2249	C.T

C.T = Charge transfer

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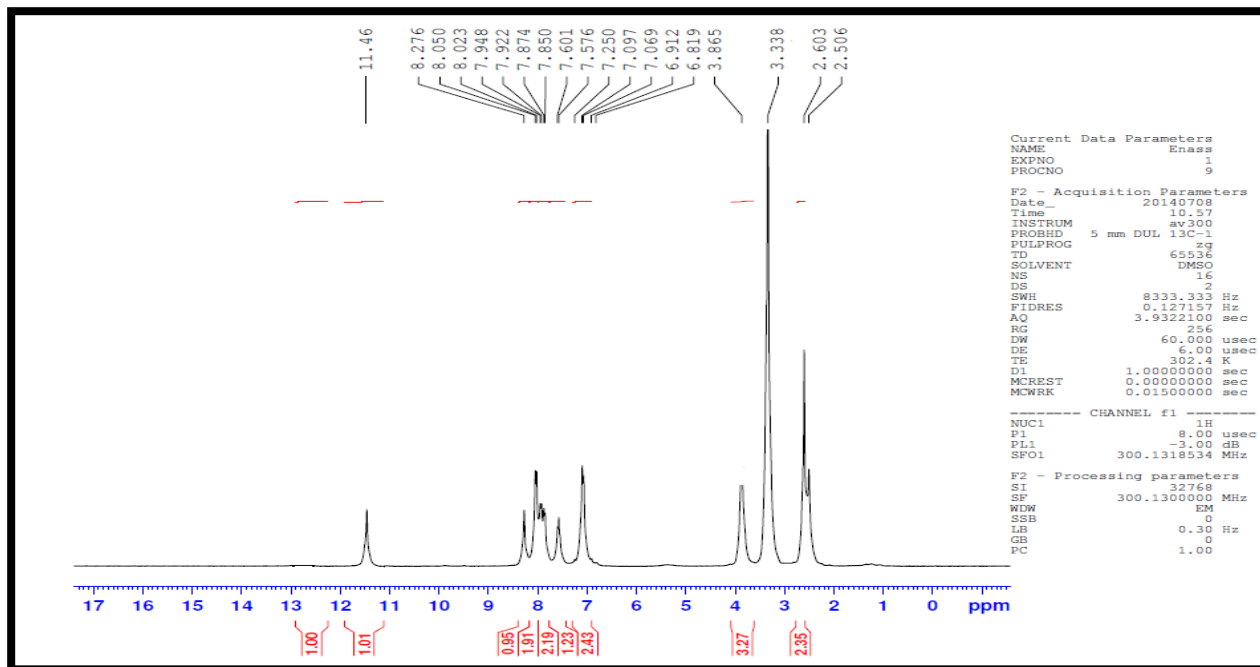


Figure No(1) : ¹H-NMR spectrum of ligand (MAA)

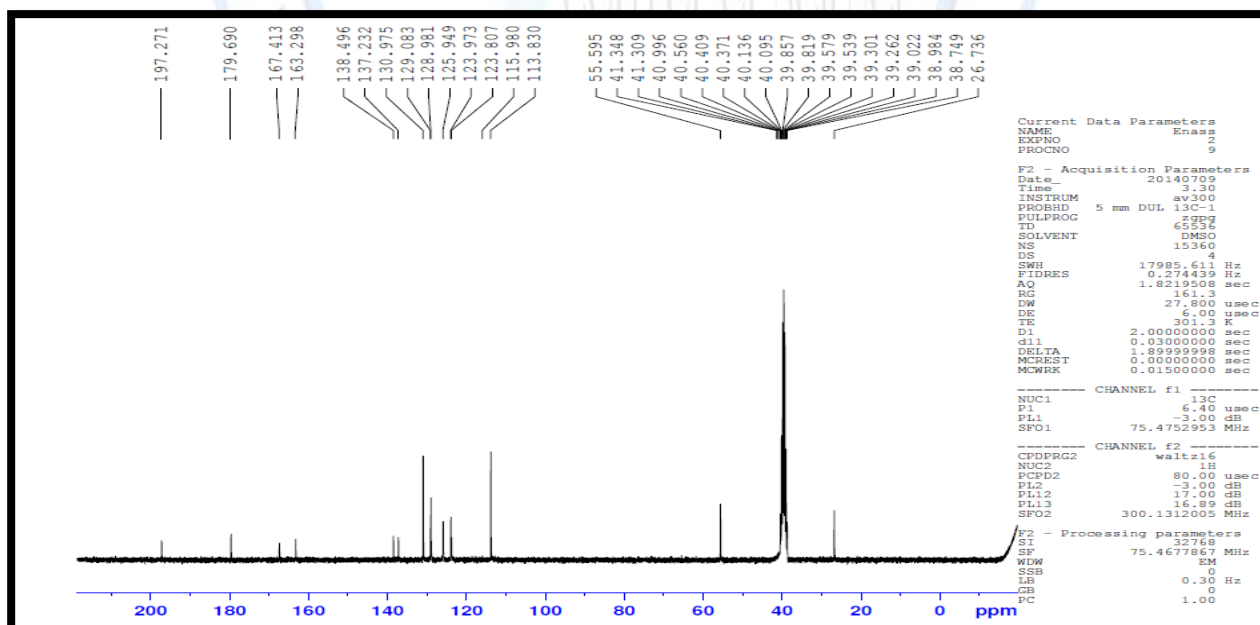


Figure No. (2): ¹³C-NMR spectrum of ligand (MAA)

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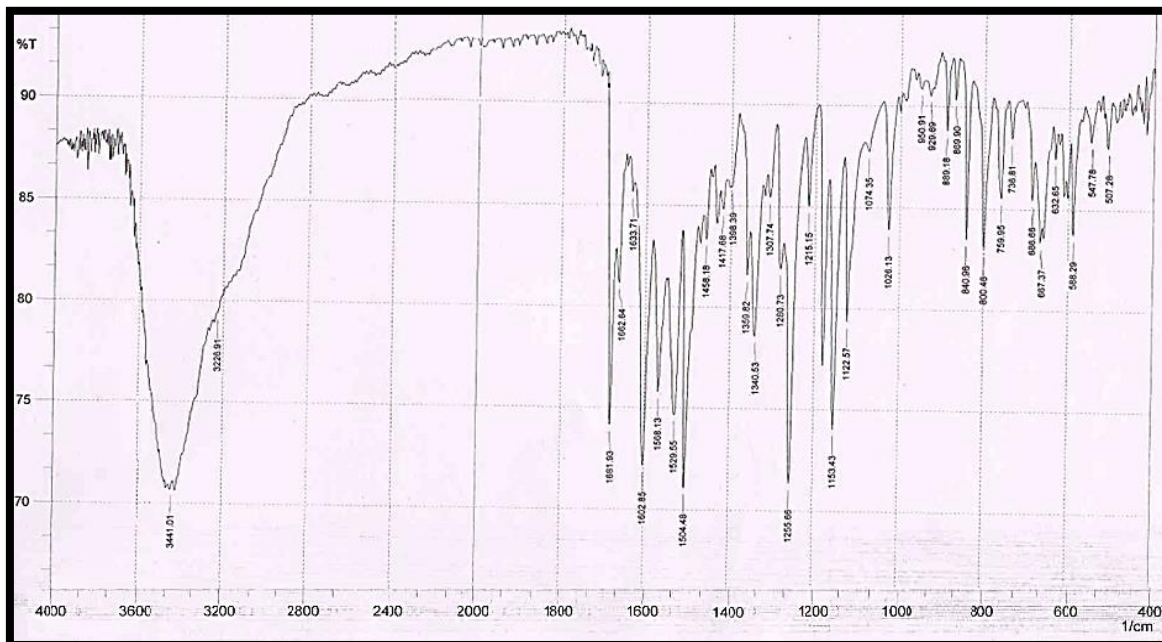


Figure No. (3): Infrared spectrum of ligand (MAA)

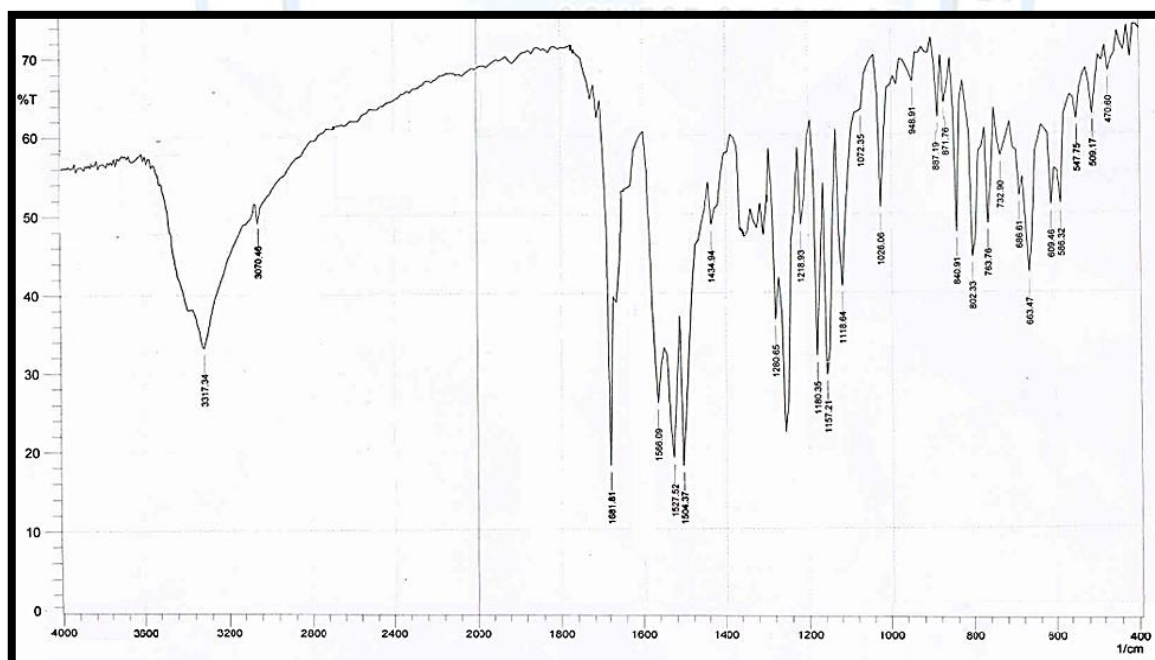


Figure No. (4): Infrared spectrum of complex $[Co(MAA)_2(H_2O)_2]Cl_2$

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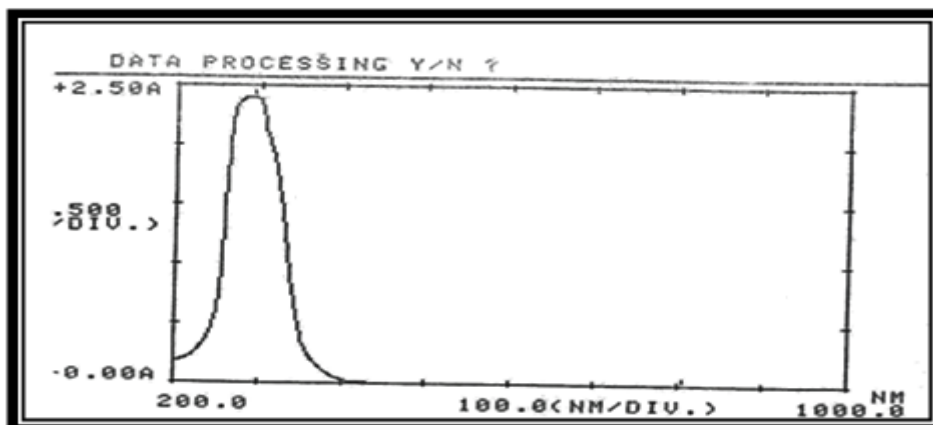


Figure No. (5): U.V spectrum of ligand (MAA)

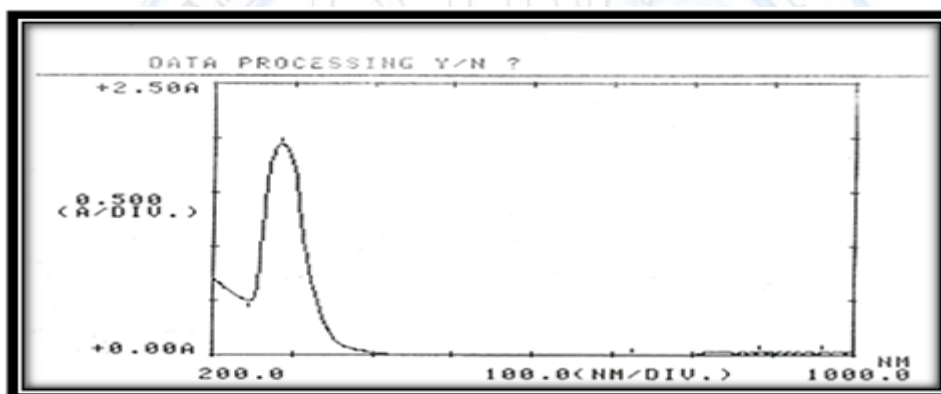


Figure No. (6): U.V spectrum of complex $[Mn(MAA)_2(H_2O)_2]Cl_2$

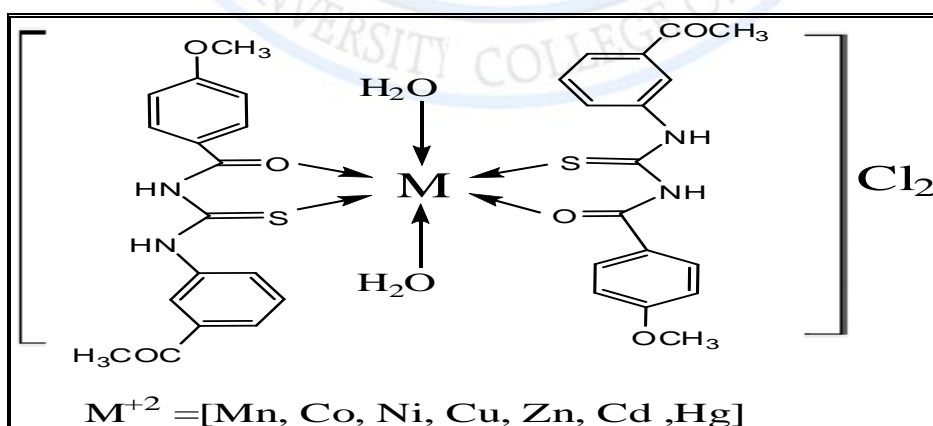


Figure No. (7): The proposed chemical structure formula of the complexes