

## Synthesis and characterization of novel metal complexes of (pentulose- $\gamma$ -lactone-2,3-enedibenzoate barbituric acid) with some metal ions

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### Abstract:

New (pentulose- $\gamma$ -lactone-2,3-enedibenzoate barbituric acid) (L) have been synthesized by reaction of (5-C-dimethyl malonyl-pentulose- $\gamma$ -lactone-2,3-enedibenzoate) with urea in alkaline media (sodium methoxide). ( $\text{Ca}^{+2}$ ,  $\text{Co}^{+2}$ ,  $\text{Ni}^{+2}$ ,  $\text{Cu}^{+2}$ ,  $\text{Zn}^{+2}$ ,  $\text{Cd}^{+2}$  and  $\text{Hg}^{+2}$ ) complexes of (pentulose- $\gamma$ -lactone-2,3-enedibenzoate barbituric acid) (L) have been prepared and characterized by ( $^1\text{H}$  and  $^{13}\text{CNMR}$ ), FTIR, (U.V-Vis) spectroscopy, Atomic absorption spectrophotometer (A.A.S), Molar conductivity measurements and Magnetic moment measurements, and the following general formula has been given for the prepared complexes  $[\text{MCl}_2(\text{H}_2\text{O})].\text{XH}_2\text{O}$ , where  $\text{M} = (\text{Ca}^{+2}, \text{Co}^{+2}, \text{Ni}^{+2}, \text{Cu}^{+2}, \text{Zn}^{+2}, \text{Cd}^{+2}, \text{Hg}^{+2})$ ,  $\text{X} =$  five molecules with ( $\text{Cd}^{+2}$ ) complex,  $\text{L} =$  (pentulose- $\gamma$ -lactone-2,3-enedibenzoate barbituric acid).

**Key words:** dimethyl malonyl, pentulose, lactone, benzoate, barbituric acid, complexes.

### Introduction

A variety of transition metal complexes with barbiturates have been prepared owing to their important roles in clinical detection and identification of drug[1-5]. Masoud[6] reported the synthesis of copper complexes with azobarbituric and azothiobarbituric acids. Also Mazharul[7] reported the synthesis of new ligands of macrocycle systems from the reaction of thiobarbituric acid with diethylenetriamine and diethyl oxalate. In this paper, properties of some new metal ions complexes with (pentulose- $\gamma$ -lactone-2,3-enedibenzoate barbituric acid) (L) have been prepared.

### Materials and Methods:

Metal salts ( $\text{CaCl}_2.6\text{H}_2\text{O}$ ,  $\text{CoCl}_2.6\text{H}_2\text{O}$ ,  $\text{NiCl}_2.6\text{H}_2\text{O}$ ,  $\text{CuCl}_2.2\text{H}_2\text{O}$ ,  $\text{ZnCl}_2$ ,  $\text{CdCl}_2.\text{H}_2\text{O}$  and  $\text{HgCl}_2$ ) were obtained from Fluka,

Merck in high purity, Urea (Fluka), Sodium metal, Dimethyl malonate (BDH). ( $^1\text{H}$  and  $^{13}\text{CNMR}$ ) spectra were recorded using Ultra Shield 300 MHz, Bruker, Switzerland, at University of Al al-Bayt, Jordan. IR spectra (in KBr discs) were recorded on Shimadzu FT Infrared spectrophotometer. The (U.V-Vis) were recorded using (Shimadzu U.V-Vis 160A), (U.V-Vis) spectrophotometer in dimethyl sulphoxide solution ( $10^{-3}$  M). Metal contents of the complexes were determined by Atomic absorption technique by using Shimadzu (AA680G) Atomic absorption spectrophotometer. The magnetic moments ( $\mu_{\text{eff}}$  B.M) were calculated on Faraday method by using (Balance Magnetic Susceptibility Model MSBMKT). Melting points were determined by using (Stuart-melting point apparatus). Conductivity

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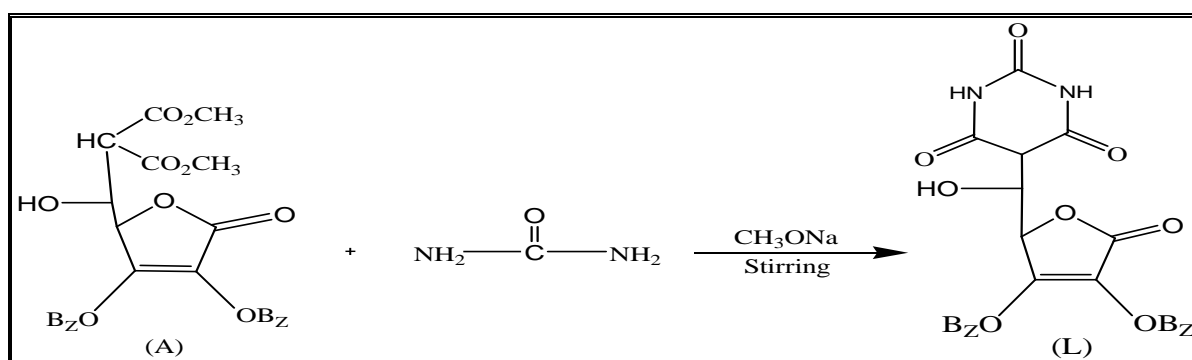
measurements were carried out using Philips PW.Digital.

### Synthesis of ligand, (pentulose- $\gamma$ -lactone-2,3-enedibenzoate barbituric acid)

1- Dry urea (15.5mmol) was added to solution of sodium methoxide (30.9mmol of sodium metal in absolute

methanol (20ml)), stirring at room temperature for 1 hour.

2- Solution of (5-C-dimethyl malonyl-pentulose- $\gamma$ -lactone-2,3-enedibenzoate)[8] (A) (10.3mmol) in absolute methanol (30ml) was added, stirring was continued at room temperature for 48 hours. The solvent was evaporated; the combined residue was washed with hot absolute ethanol to give the white crystals, scheme (1).



Scheme (1) the synthesis route of ligand (L)

### Synthesis of complexes

Addition of ethanol solution of the metal salts ( $\text{Ca}^{+2}$ ,  $\text{Co}^{+2}$ ,  $\text{Ni}^{+2}$ ,  $\text{Cu}^{+2}$ ,  $\text{Z}^{+2}$ ,  $\text{Cd}^{+2}$  and  $\text{Hg}^{+2}$ ) (1mmol) to solution of (L) (1mmol) distilled water. The mixture was stirring for 3 hours at room temperature, the resulting precipitates were filtered, washed and recrystallized from ethanol and dried in an oven at  $50^\circ\text{C}$ .

### Results and Discussion:

The isolated complexes were crystalline solids, soluble in some of common solvents such as dimethyl formamide (DMF), dimethyl sulphoxide (DMSO). The conductivity measurements in DMSO indicated the non-electrolyte behavior; Table (1) includes the physical properties. The analytical data confirmed the (1:1) (Metal:Ligand) composition of the complexes. The magnetic measurements ( $\mu_{\text{eff}}$  B.M) for the complexes are also listed in Table (1).

Table (1) Physical properties of ligand and its complexes

Formula	Colour	M.p. <sup>o</sup> C or dec.	Metal % Found (Calc.)	Molar conductivity (S.cm <sup>2</sup> mole <sup>-1</sup> ) in DMSO (10 <sup>-3</sup> M)	μ <sub>eff</sub> (B.M)
C <sub>23</sub> H <sub>16</sub> O <sub>10</sub> N <sub>2</sub> [L]	White	210(dec.)	-	3.42	-
[CaCl <sub>2</sub> (H <sub>2</sub> O)]	Yellow	185(dec.)	7.81 (6.58)	20.1	0.00
[CoCl <sub>2</sub> (H <sub>2</sub> O)]	Brown	230(dec.)	10.44 (9.38)	14.1	4.35
[NiCl <sub>2</sub> (H <sub>2</sub> O)]	Green	220(dec.)	10.15 (9.35)	14.37	3.03
[CuCl <sub>2</sub> (H <sub>2</sub> O)]	Blue	190(dec.)	10.12 (10.04)	21	1.72
[ZnCl <sub>2</sub> (H <sub>2</sub> O)]	Yellow	220(dec.)	10.22 (10.30)	18.02	0.00
[CdCl <sub>2</sub> (H <sub>2</sub> O)].5H <sub>2</sub> O	Yellow	240(dec.)	14.80 (14.57)	13.02	0.00
[HgCl <sub>2</sub> (H <sub>2</sub> O)]	Yellow	200(dec.)	26.75 (26.06)	16.88	0.00

### Spectral studies

<sup>1</sup>H and <sup>13</sup>CNMR spectra studies of the ligand (L):-

#### A) <sup>1</sup>HNMR spectrum for the ligand (L)

The <sup>1</sup>HNMR spectrum of (L), Fig. (1) showed the following signals: doublet (d) at δ(3) ppm for (1H, CH malonate), doublet (d) at δ(4.70-4.78) ppm for (1H, lactone ring H<sub>4</sub>), and multiplet (m) at δ(7.25-7.73) ppm for aromatic protons.

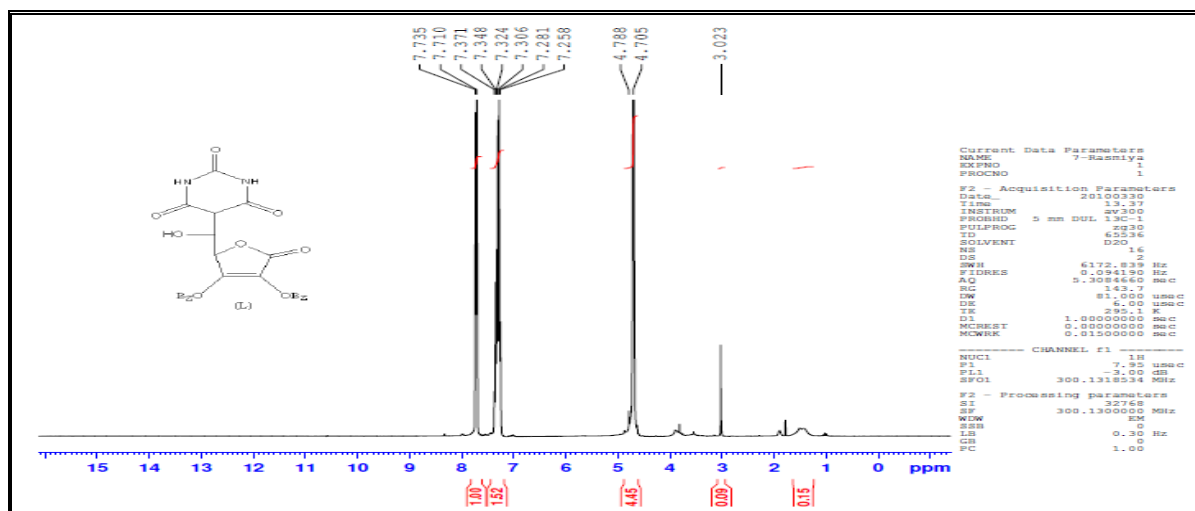


Fig. (1) <sup>1</sup>HNMR spectrum of ligand (L)

#### B) <sup>13</sup>CNMR spectrum for the ligand (L)

The <sup>13</sup>CNMR spectrum of (L), Fig. (2) showed the following signals:

signal at δ(177.53) ppm for carbon (C=O) in (HNCONH), signal at δ(175.65) ppm for carbon (C=O) in (CHCONH), signal at δ(164.53) ppm for carbon (C=O) of the ester, lactone

ring, signal at  $\delta(136.14)$  ppm for C-3, signal at  $\delta(131.63)$  ppm for C-2, signals at  $\delta(131.24-127.85)$  ppm for

aromatic carbons, signal at  $\delta(61.24)$  ppm for C-4, and signal at  $\delta(47.85)$  ppm for C-5 and C-6.

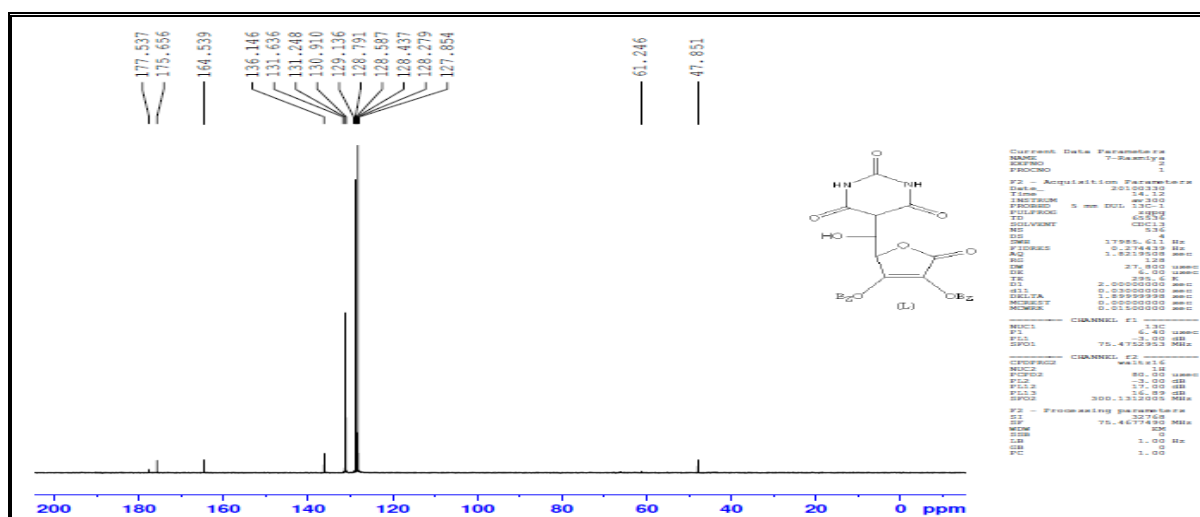


Fig. (2)  $^{13}\text{C}$ NMR spectrum of ligand (L)

### Infrared spectra

The important infrared spectra data of ligand (L) and its complexes are given in Table (2). The strong absorption band at  $(1662)\text{ cm}^{-1}$  and  $(1627)\text{ cm}^{-1}$  in the free ligand, Fig. (3) due to  $\nu(\text{C}=\text{O})$ [9],[10], in the complexes spectra, this band has been found in the range between  $(1600-1593)\text{ cm}^{-1}$  and  $(1585-1550)\text{ cm}^{-1}$  shifted to lower frequencies by  $(62-69)\text{ cm}^{-1}$  and  $(42-77)\text{ cm}^{-1}$ , which indicates the coordination of the oxygen atom at the  $\nu(\text{C}=\text{O})$  group[11],[12].

In the free ligand, the band at  $(3471)\text{ cm}^{-1}$  due to  $\nu(\text{N-H})$ , the spectra of the complexes show this band about  $(3444-3390)\text{ cm}^{-1}$  shifted to lower frequencies by  $(27-81)\text{ cm}^{-1}$ [13],[14].

The stretching vibration band  $\nu(\text{C-N})$  observed at  $(1346)\text{ cm}^{-1}$  in the spectrum of the free ligand, has been

found in the range  $(1454-1400)\text{ cm}^{-1}$  shifted to higher frequencies on complexes formation by  $(108-54)\text{ cm}^{-1}$ , which indicates the coordination of the ligand (L) through the nitrogen atom of the aliphatic amine[15],[16]. New bands appeared in the spectra of metal complexes at  $(678-470)\text{ cm}^{-1}$  and  $(520-435)\text{ cm}^{-1}$  attributed to the (M-N) and (M-O) respectively[17],[18]. The bands are due to coordinate chloride ion in complexes formed with metal chloride were fall bellow the limits of our infrared spectrophotometer and could not be observed.

The spectra of complexes showed the appearance of bands in the range  $(871-833)\text{ cm}^{-1}$  attributed to  $\delta(\text{OH})$ , these bands confirm the coordination of the water with metal[19]. Fig. (4) showed the infrared spectrum of nickel complex.

Table (2) the characteristic infrared of the ligand (L) and its metal complexes

Compound	$\nu(\text{N-H})$	$\nu(\text{C=O})$	$\nu(\text{C-N})$	$\delta(\text{OH})$	M-N	M-O
$\text{C}_{23}\text{H}_{16}\text{O}_{10}\text{N}_2$ [L]	3471(s)	1662(s) 1627(m)	1346(w)	-	-	-
$[\text{CaLCl}_2(\text{H}_2\text{O})]$	3414(s)	1597(s) 1550(s)	1415(s)	871(s)	482(m)	455(w)
$[\text{CoLCl}_2(\text{H}_2\text{O})]$	3425(br)	1593(s) 1554(s)	1415(s)	837(m)	474(m)	435(m)
$[\text{NiLCl}_2(\text{H}_2\text{O})]$	3390(m.b)	1597(s) 1550(s)	1400(s)	840(s)	478(m)	451(w)
$[\text{CuLCl}_2(\text{H}_2\text{O})]$	3444(s)	1600(s) 1585(s)	1435(m)	833(s)	509(w)	489(m)
$[\text{ZnLCl}_2(\text{H}_2\text{O})]$	3410(br)	1597(s) 1554(s)	1454(m)	844(s)	532(w)	509(w)
$[\text{CdLCl}_2(\text{H}_2\text{O})].5\text{H}_2\text{O}$	3433(br)	1593(s) 1554(s)	1415(m)	856(s)	470(w)	447(w)
$[\text{HgLCl}_2(\text{H}_2\text{O})]$	3433(br)	1597(s) 1550(s)	1411(s)	844(s)	678(m)	520(w)

Where: s = strong, m = medium, w = weak, br = broad

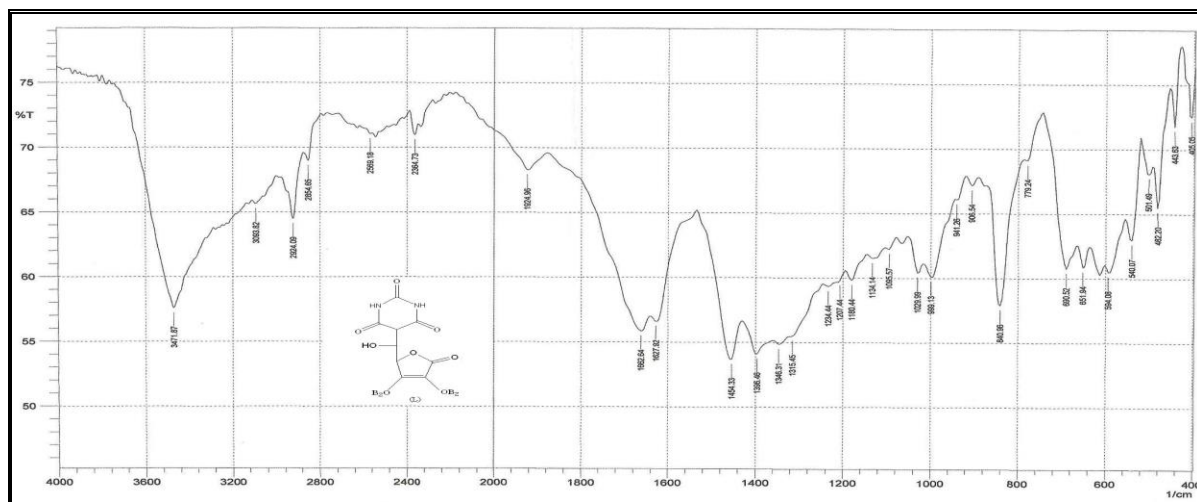


Fig. (3) Infrared spectrum of ligand (L)

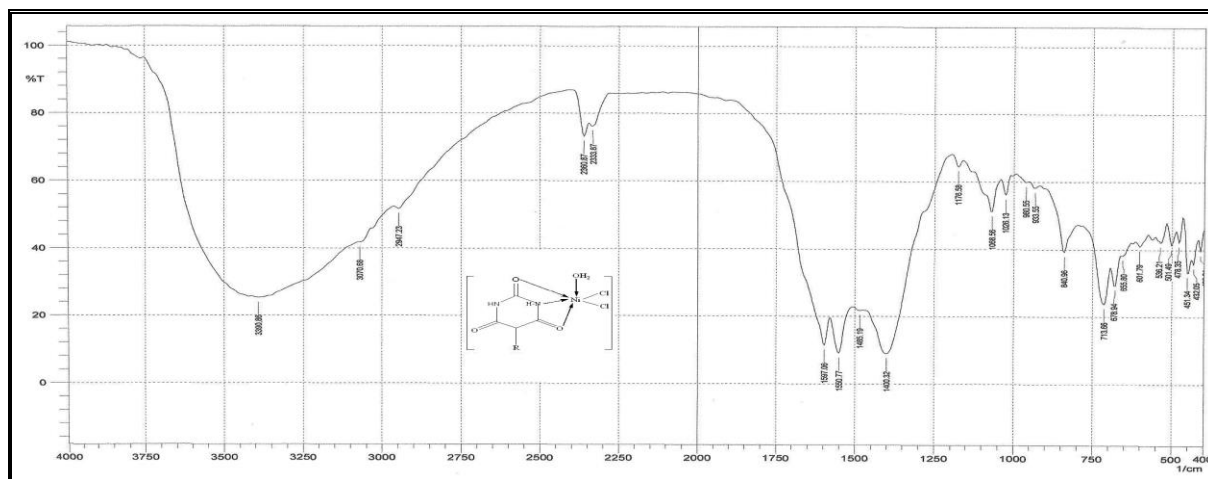


Fig. (4) Infrared spectrum of [NiLCl<sub>2</sub>(H<sub>2</sub>O)]

**Electronic spectra**

The absorptions and assignments related to the ligand and their complexes are listed in Table (3). The ligand (L), Fig. (5) exhibited an

absorption band in (U.V) region at (294) nm (34013) cm<sup>-1</sup> which may be attributed to (n → π\*) transition[9],[10].

**Table (3) Electronic spectra data of (L) complexes in DMSO solvent**

Compound	λ <sub>max</sub> nm	Wave number cm <sup>-1</sup>	ε <sub>max</sub> molar <sup>-1</sup> cm <sup>-1</sup>	Assignment
[L]	294	34013	66	n → π*
[CaLCl <sub>2</sub> (H <sub>2</sub> O)]	296	33783	253	C.T
[CoLCl <sub>2</sub> (H <sub>2</sub> O)]	292	34246	83	C.T
	350	28571	18	${}^4T_{1g}(F) \xrightarrow{V_3} {}^4T_{1g}(P)$
	551	18148	14	${}^4T_{1g}(F) \xrightarrow{V_2} {}^4A_{2g}$
	804	12437	9	${}^4T_{1g}(F) \xrightarrow{V_1} {}^4T_{2g}$
[NiLCl <sub>2</sub> (H <sub>2</sub> O)]	290	34482	156	C.T
	340	29411	25	${}^3A_{2g} \xrightarrow{V_3} {}^3T_{1g}(P)$
	542	18450	21	${}^3A_{2g} \xrightarrow{V_2} {}^3T_{1g}(F)$
	750	13333	10	${}^3A_{2g} \xrightarrow{V_1} {}^3T_{2g}$
[CuLCl <sub>2</sub> (H <sub>2</sub> O)]	211	47393	89	C.T
	295	33898	598	C.T
	671	14903	89	${}^2E_g \rightarrow {}^2T_{2g}$
[ZnLCl <sub>2</sub> (H <sub>2</sub> O)]	299	33444	127	I.L.C.T
[CdLCl <sub>2</sub> (H <sub>2</sub> O)].5H <sub>2</sub> O	298	33557	217	I.L.C.T
[HgLCl <sub>2</sub> (H <sub>2</sub> O)]	252	39682	54	I.L.C.T

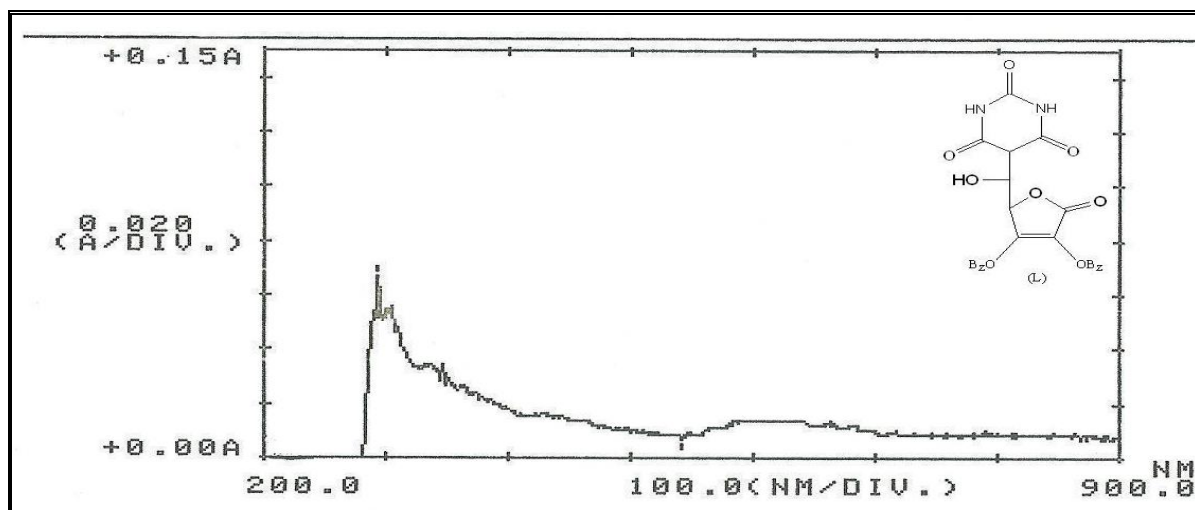


Fig. (5) Electronic spectrum of ligand (L)

**The spectra of complexes**

**- [CoLCl<sub>2</sub>(H<sub>2</sub>O)] complex**

The spectrum of the brown complex of Co(II), Fig. (6) exhibited the following bands at (34246) cm<sup>-1</sup>, (28571) cm<sup>-1</sup>, (18148) cm<sup>-1</sup> and (12437) cm<sup>-1</sup> which have been assigned to (C.T),  $^4T_{1g}(F) \xrightarrow{V_3} ^4T_{1g}(p)$ ,  $^4T_{1g}(F) \xrightarrow{V_2} ^4A_{2g}$

and  $^4T_{1g}(F) \xrightarrow{V_1} ^4T_{2g}$  respectively[20].

The Racah interelectronic repulsion parameter ( $\hat{B}$ ) found to be (627.2) cm<sup>-1</sup>, the ratio  $\beta = \hat{B} / B_0$  comes out to be (0.650) which indicates present covalent bond between metal ion and donor atom in the complex and these parameters are accepted for cobalt(II) octahedral complexes[21].

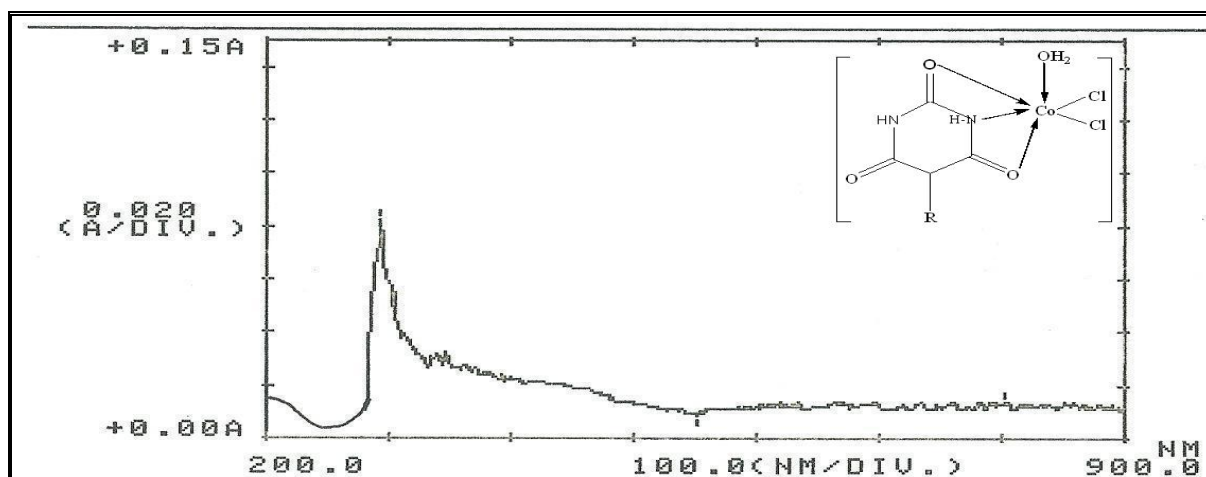


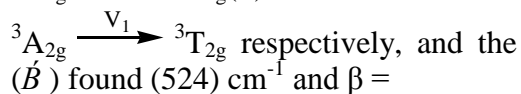
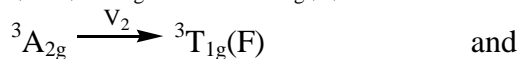
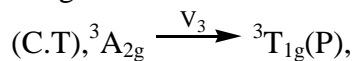
Fig. (6) Electronic spectrum of [CoLCl<sub>2</sub>(H<sub>2</sub>O)]

**- [NiLCl<sub>2</sub>(H<sub>2</sub>O)] complex**

The spectrum of the green complex exhibited the following absorptions at

(34482) cm<sup>-1</sup>, (29411) cm<sup>-1</sup>, (18450) cm<sup>-1</sup> and (13333) cm<sup>-1</sup>, these bands are characteristic of octahedral

nickel(II)[22] complex and were assigned to the transitions



$\dot{B} / B^o$  comes out to be (0.50) which indicates present covalent bond between metal ion and donor atom in the complex.

#### - [CuLCl<sub>2</sub>(H<sub>2</sub>O)] complex

The spectrum of the blue complex of Cu(II) gave two bands at (47393) cm<sup>-1</sup> and (33898) cm<sup>-1</sup> caused by (C.T) transitions and only one absorption band was observed at (14903) cm<sup>-1</sup> which assigned to  ${}^2E_g \longrightarrow {}^2T_{2g}$

transition in octahedral structurep[23],[24].

#### - [CaLCl<sub>2</sub>(H<sub>2</sub>O)], [ZnLCl<sub>2</sub>(H<sub>2</sub>O)],

#### [CdLCl<sub>2</sub>(H<sub>2</sub>O)].5H<sub>2</sub>O and

#### [HgLCl<sub>2</sub>(H<sub>2</sub>O)]

The yellow complexes [CaLCl<sub>2</sub>(H<sub>2</sub>O)], [ZnLCl<sub>2</sub>(H<sub>2</sub>O)], [CdLCl<sub>2</sub>(H<sub>2</sub>O)].5H<sub>2</sub>O and [HgLCl<sub>2</sub>(H<sub>2</sub>O)] showed absorption bands at (33783) cm<sup>-1</sup>, (33444) cm<sup>-1</sup>, (33557) cm<sup>-1</sup> and (39682) cm<sup>-1</sup> respectively[25-28] attributed to internal ligand charge transfer.

According to these results, the structural formula of prepared complexes may be proposed in Fig. (7).

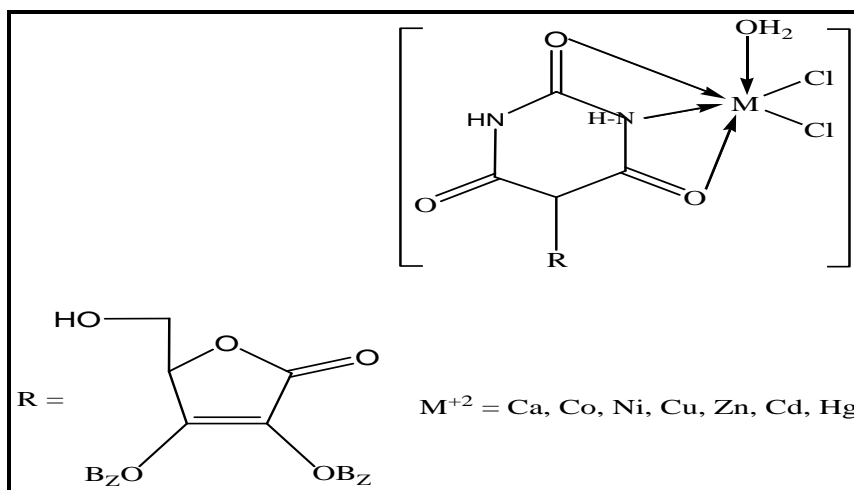


Fig. (7) the proposed structural formula of the complexes



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## تحضير وتشخيص بعض المعقدات الفلزية الجديدة لليكاند Pentulose- $\gamma$ -lactone-2,3-enedibenzoate barbituric acid مع بعض الايونات الفلزية

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### الخلاصة:

حضر الليكاند الجديد (L) (Pentulose- $\gamma$ -lactone-2,3-enedibenzoate barbituric acid) وذلك من خلال مفاعلة (5-C-dimethyl malonyl-pentulose- $\gamma$ -lactone-2,3-enedibenzoate) مع اليوريا وعند وسط قاعدي من ميثوكسيد الصوديوم. كما حضرت بعض المعقدات الفلزية الجديدة للايونات (الكالسيوم، الكوبلت، النيكل، النحاس، الخارصين، الكاديوم والزنك) الثنائية التكافؤ مع الليكاند (L)، شخص الليكاند مع معقداته الفلزية بالطرق الطيفية المتوفرة ومنها طيف الرنين النووي المغناطيسي ( $^1\text{H}$  and  $^{13}\text{CNMR}$ ) طيف الأشعة تحت الحمراء، طيف الأشعة فوق البنفسجية والمرئية إضافة الى تعيين نسبة الفلز في المعقدات بواسطة طيف الامتصاص الذري، قياس التوصيلية المولارية لمحاليل المعقدات في مذيب ثنائي مثيل سلفوكسايد (DMSO) وقياس العزم المغناطيسي المؤثر للمعقدات الصلبة. ومن نتائج هذه الدراسات التشخيصية امكن اعطاء الصيغة العامة لهذه المعقدات وكمالاتي  $[\text{MLCl}_2(\text{H}_2\text{O})].\text{XH}_2\text{O}$  حيث  $\text{M} =$  الايون الفلزي  $= \text{L}$  ،  $\text{X} =$  خمس جزيئات ماء مع معقد الكاديوم فقط،  $(\text{Hg}^{+2}$  ،  $\text{Cd}^{+2}$  ،  $\text{Zn}^{+2}$  ،  $\text{Cu}^{+2}$  ،  $\text{Ni}^{+2}$  ،  $\text{Co}^{+2}$  ،  $\text{Ca}^{+2}$ ) .(pentulose- $\gamma$ -lactone-2,3-enedibenzoate barbituric acid)