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Synthesis and spectroscopic investigation of diclofenac-Hg(II) complexes with amines as co-ligands

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ABSTRACT

New mixed ligand Hg(II) complexes of the anti-inflammatory drug (diclofenac (diclo^{-})) and amines have been synthesized and fully characterized. The IR data showed that diclo^{-} bonded as a monodentate ligand with Hg(II) ion through the deprotonated carboxylategroup, and the diamine {**Phen** (1,10-phenanthroline), **Bipy**(2,2'-bipyridine), and **en**(ethylenediamine)} bonded as bidentate. Whereas the primary amine {**Py**(Pyridine), **2-Apy**(2-aminopyridine), **3-Apy**(3-aminopyridine), and **2-Apmy**(2-aminopyrimidine)} bonded as monodentate mode through the nitrogen atom of the heterocyclic ring to afford tetrahedral Hg(II) complexes.

Introduction

"It is well-known that most anti-inflammatory drugs are carboxylic acids in which the carboxylate group is presented for metal–ligand interaction [1]. Sodium diclofenac [2-[(2,6-dichlorophenyl) amino]-phenyl acetate (**Fig. 1 a**) is an effective non-steroidal anti-inflammatory drug (NSAID), therapeutically used in inflammatory and painful diseases of rheumatic and non-rheumatic origin[1]. The anti-inflammatory activity of diclofenac and most of its other pharmacological effects are thought to be related to the inhibition of the conversion of arachidonic acid to prostaglandins, which are the mediators of the inflammatory process" [2-4]. The carboxylate ligands can coordinated in different coordination fashions, being able to linkage as monodentate fashion through the negatively charged oxygen atom of the carboxylate groups or as a bidentate or polydentate modes through oxygen donor atoms of the carboxylate groups[1-4] (**Fig. 1b**).

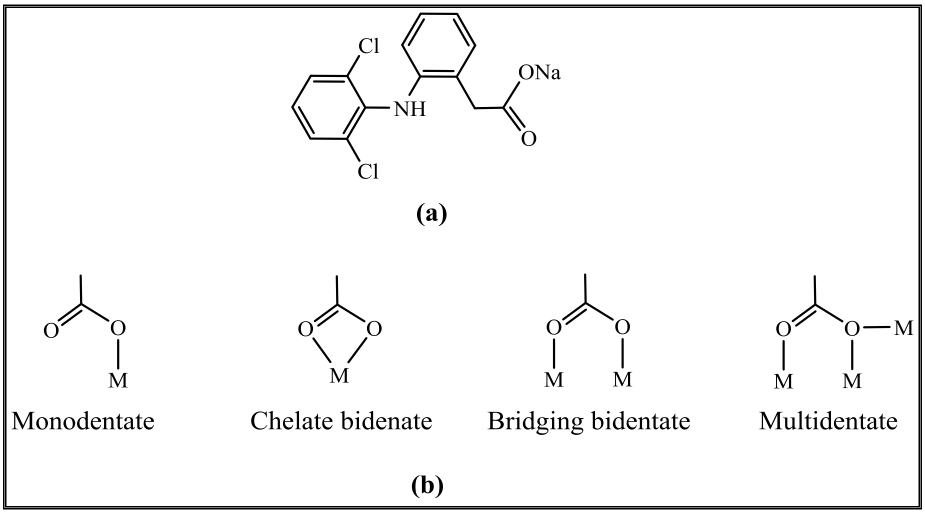


Fig. 1: (a) sodium diclofenacate (b) Coordination fashions of carboxylate groups

In the literature, many reports on diclofenac as ligands with different metal ions such as Co(II), Ni(II), Mn(II), Hg(II), Cd(II) ...etc. can be found[3-19], the reports on mixed ligand-Metal complexes of diclofenac and amines are scanty in the literature. In continuation of our previous studies[3-19]. We reported her new diclofenac Hg(II) complexes were synthesis and characterization, with some amine ligands (Bipy, Phen, en, Py, 3-Apy, 2-Apy and 2-Apmy).

2. Experimental

2.1. Methods and Materials:

Sodium diclofenacate, amine ligands and mercuric chloride(II) were provided from Aldrich, or BDH companies, and used without further purification. melting points were measured on a SMP40 / Stuart melting point apparatus and were uncorrected. Element analysis were determined by CHN-analyzer type 1106 Carlo-Erba in Ibn Al-Haitham college Lab. / University of Baghdad. Molar conductivities of freshly prepared ($1x10^{-3}$) M of DMSO –solutions of complexes were measured at 25^{o}C using Digital conductivity meter. Infrared spectra (KBr disc) were recorded in the 4000 – 400 cm^{-1} range on Shimadzu 8400S FTIR Spectrophotometer. NMR spectra of the prepared complexes were recorded on Varian Unity spectrometer (400MHz) in DMSO-d^6 solvent and TMS as an internal reference.

2.2 Preparation of [Hg(Diclo)$_2$].2H$_2$O (1)

Aqueous solution of sodium diclofenate (NaDiclo) (0.469g, 1.473mmol) in (15ml) was added to an aqueous of $HgCl_2$ (0.200g, 0.7366mmol) in (10ml) with stirring, a white solid precipitate was formed. The mixture was stirred for 3h at 50°C. The white solid was filtered off, washed with distal water and dried in oven under vacuum.(Yield: 0.519 g, 85%).

2.3 Preparation of [Hg(Diclo)$_2$(Phen)] (2)

To a suspensionof [Hg(Diclo)$_2$].2H$_2$O (0.0200g, 0.253mmol) in acetone (10ml), a hot solution of 1,10-phenanthroline (Phen) (0.045g, 0.253mmol) in acetone (15ml) was added with stirring, color changed to yellow. The mixture was stirred at room temperature for 3h. A yellow ppt. was formed which was filtered off, washed with acetone, and dried in oven under vacuum.(Yield: 0.200 g, 81%).

The **[Hg(Diclo)$_2$(Bipy)] (3)** and **[Hg(Diclo)$_2$(en)] (4)**; complexes were prepared and isolated in a similar method.

2.4 Preparation of [Hg(Diclo)$_2$(2-Apy)$_2$] (7)

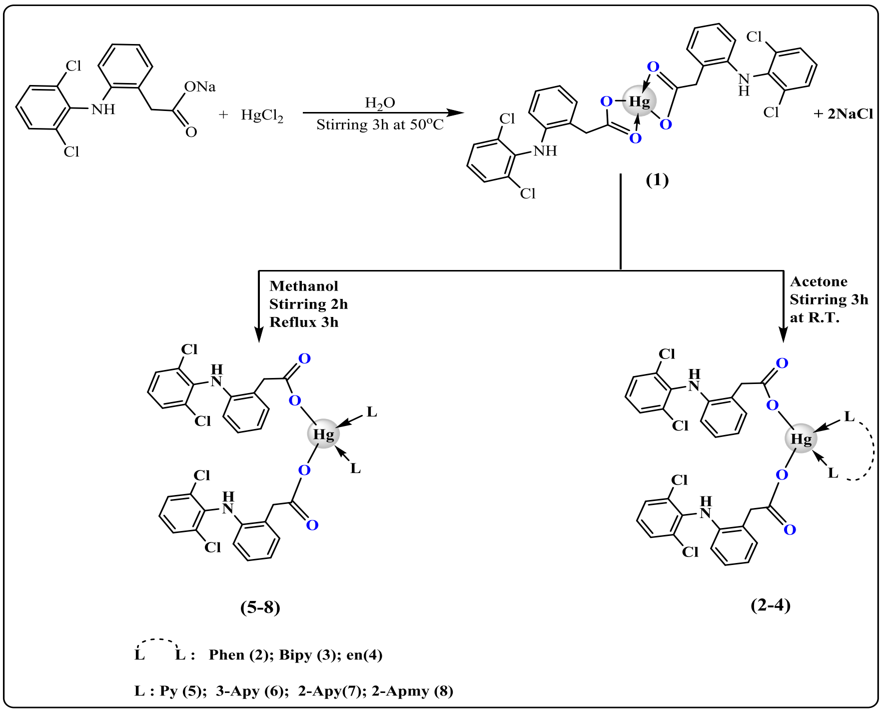
Ethanolic solution of 2-aminopyridine **(2-Apy)** (0.023g, 0.242mmol) in (5ml) was added to a suspensionof **(1)** (0.100g, 0.121mmol) in methanol (10ml), with stirring. The resulted creamy mixture was stirred for 3h at room temperature, then reflux for another 3h. The ppt. formed was filtered off, washed with EtOH, and dried in oven under vacuum. (Yield: 0.045 g, 38%).

The **[Hg(Diclo)$_2$(Py)$_2$] (5)**; **[Hg(Diclo)$_2$(3-Apy)$_2$] (6)** and **[Hg(Diclo)$_2$(2-Apmy)$_2$] (8)**; complexes were prepared and isolated in a similar method.

3. Results and Discussions

3.1 Synthesis of complexes

Reaction of sodium diclofenac with mercuric chloride ($HgCl_2$) resulted in the product of [Hg(diclo)$_2$].2H$_2$O **(1)** by chloride exchange under normal conditions as showed in Scheme 1. Complex **(1)** was reacted with amine ligand {Phen, Bipy, and en} in 1:1 molar ratio or {Py,2-Apy, 3-Apy, and 2-Apmy} in 1:2 molar ratio (complex: ligand) (Scheme 1).



Scheme 1: preparation of 1-8 complexes

The prepared complexes are air stable in the solid state, insoluble common solvents (Such as MeOH, EtOH, Acetone, $CHCl_3$) but soluble in DMSO or DMF. The elemental analysis are listed in **Table 1** and they agreed well with the suggested structure the complexes. The molar conductance values of complexes **(1-8)** are measured in DMSO at room temperature and formed to be in the range of non-electrolytes[20].

Table 1: Color, yield, m.p, and elemental analysis of prepared complexes(1-8)

Seq.	Complexes	Chemical formula	Color	M.P. °C	Yield% Wt.(g)	Λ (ohm^{-1}. cm^2. mol^{-1}) 10^{-3} M / DMSO	Elemental Analysis Calc. (Found) %		
							C	H	N
1	[Hg(diclo)$_2$].2H_2O	$C_{28}H_{20}Cl_4HgN_2O_4$	White	147-150	91 0.531	20.18	42.52 (42.78)	2.55 (2.76)	3.54 (3.72)
2	[Hg(diclo)$_2$(Phen)]	$C_{40}H_{28}Cl_4HgN_4O_4$	Yellow	192-194	81 0.200	0.02	49.47 (49.66)	2.91 (2.98)	5.77 (5.87)
3	[Hg(diclo)$_2$(Bipy)]	$C_{38}H_{28}Cl_4HgN_4O_4$	white	220-224	63 0.190	4.17	48.19 (48.41)	2.28 (2.32)	5.92 (5.73)
4	[Hg(diclo)$_2$(en)]	$C_{30}H_{26}Cl_4HgN_4O_4$	Gray	130-132	82 0.220	1.20	42.44 (41.89)	3.09 (3.31)	6.60 (6.71)
5	[Hg(diclo)$_2$(Py)]	$C_{38}H_{30}Cl_4HgN_4O_4$	White	116-117	75 0.180	2.42	48.09 (48.19)	3.19 (3.28)	5.90 (5.72)
6	Hg(diclo)$_2$(3-Apy)$_2$]	$C_{38}H_{32}Cl_4HgN_6O_4$	White	160-164	78 0.092	1.78	46.62 (46.74)	3.29 (3.40)	8.58 (8.61)
7	[Hg(diclo)$_2$(2-Apy)$_2$]	$C_{38}H_{32}Cl_4HgN_6O_4$	Off white	209-210	65 0.200	1.47	46.62 (46.69)	3.29 (3.33)	8.58 (8.70)
8	[Hg(diclo)$_2$(2-Apmy)]	$C_{36}H_{30}Cl_4HgN_8O_4$	Off white	212-213	77 0.0,95	2.98	44.07 (44.2)	3.08 (3.3)	11.42 (11.50)

3.2 Infrared spectra

The infrared data of sodium diclofenac and its complexes **(1-8)** are listed in **Table 2**. and **Fig. 1-3.** IR spectrum of Nadiclo, showed bands at 3260cm^{-1}, 1572cm^{-1} and 1402cm^{-1}, 1510cm^{-1} due to ν(N-H), ν(COO^-)$_{asym.}$ ν(COO^-)$_{sym.}$, and ν(C-N) respectively. The IR spectrum of [Hg(diclo)$_2$].2H_2O complex showed ν(COO^-)$_{asym.}$ and ν(COO^-)$_{sym.}$, at 1573 cm^{-1} and 1425 cm^{-1}, respectively, providing a Δ value of 148 cm^{-1} that is indicative of a bidentate bridging coordination mode [21] for the carboxylato group of the ligand. The Δ ν(COO^-) value shifted to a lower wave number from that of the free diclo$^-$ ligand (170 cm^{-1}). The parameter usually used to determine the coordination mode of the carboxylato ligands is Δ ν(COO^-) which is defined as the difference ν(COO^-)$_{asym.}$ and ν(COO^-)$_{sym}$. For complexes **2-8** the Δ ν(COO^-) value are within (190-252)cm^{-1} range is higher than that of the Δν(COO^-) of [Hg(diclo)$_2$].2H_2O complex (151 cm^{-1}), suggesting a monodentate bonding[22].

The vibration frequency of the υ(C=N) appeared within the 1552-1627 cm^{-1} range for the complexes**(1-8)**, the υ(C=N) stretching was shifted to low frequency wave number compared to that of the free amine ligands, this is in agreement with coordination of the amine ligands with the Hg(II) through nitrogen atoms[22-25].

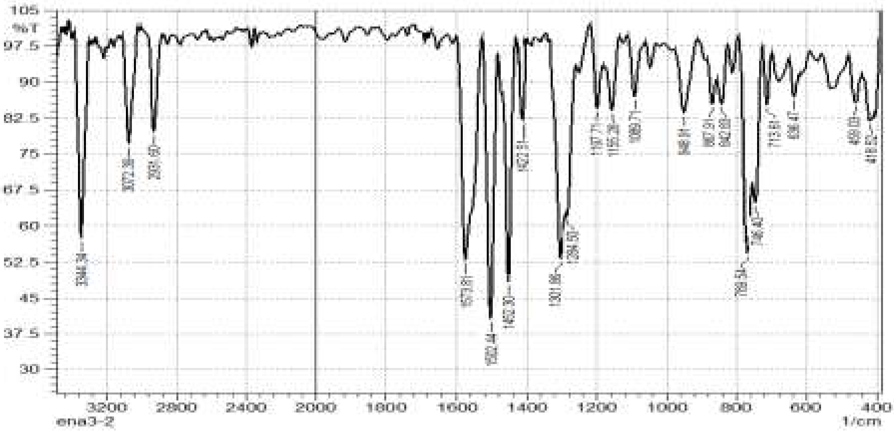


Fig. 1: IR spectrum of [Hg(diclo)$_2$].2H_2O(1)

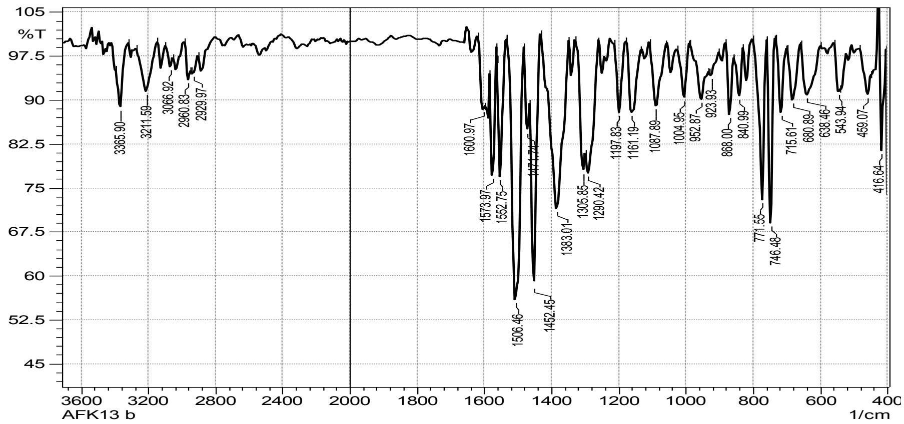


Fig. 2: IR spectrum of $[Hg(diclo)_2(en)](4)$

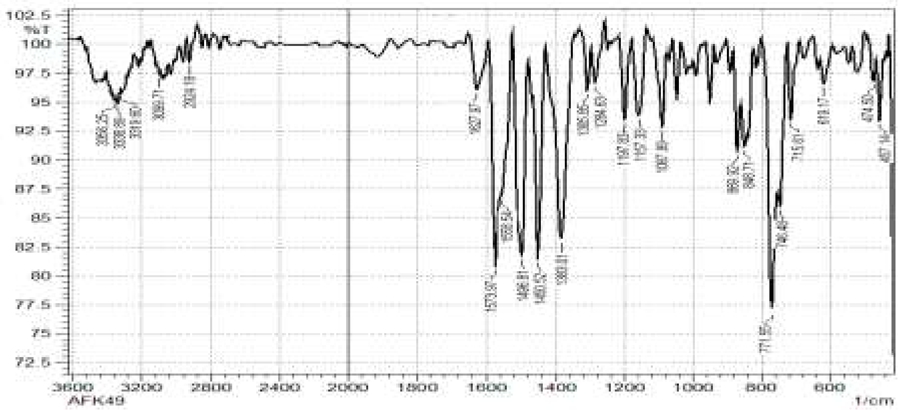


Fig. 3: IR spectrum of $[Hg(diclo)_2(2\text{-}Apmy)_2](8)$

Table 2: : Selected IR Stretching vibration bonds (cm^{-1}) of the prepared complexes (1-8)

Assignments	**Compounds**								
	Sodium diclofenac	**(1)**	**(2)**	**(3)**	**(4)**	**(5)**	**(6)**	**(7)**	**(8)**
υ(N-H)	3260w	3309s	3286m	3307w	3365m 3211m	3259m	3338 3219	3379m 3288m	3338m 3155m
$\upsilon(COO^-)_{asym}$	1572s	1573s	1571s	1581s	1573s	1575s	1573s	1575s	1564s
$\upsilon(COO^-)_{sym.}$	1402s	1422m	1357s	1329m	1383s	1379s	1383s	1382s	1374s
$\Delta\upsilon(COO^-)$	170	151	214	252	190	196	190	193	190
υ(C=N) **or** υ(C-N)	1510s	1502s	1558s 1496s	1560s 1502s	1552s 1506s	1554s 1504m	1627m 1496s	1602s 1498s	1609s 1508s
υ(Hg-O)	-	418w	420w	420	416w	445w	457w	404w	420w
υ(Hg-N)	-	-	441w	443w	459w	493w	474w	453w	474w

w= weak , m=medium , s= strong

3.2 ^{1}H NMR spectra

The chemical shift data of diclofenac complexes with amines ligands are listed in **Table 3**. The ^{1}H-NMR spectrum of $[Hg(diclo)_2].2H_2O$(**1**) complex (**Fig. 4**) shown seven peaks, two singlets at δ8.05ppm, and δ3.39ppm due to NH and CH_2 groups respectively, and two doublets at δ7.44ppm, and δ6.22ppm with coupling constant ($^3J_{H\text{-}H}$: 7.68Hz and 7.55Hz)assign to the protons in position (**5,7**) and (**1**) respectively. Whereas the other peak are listed in **Table 3**.

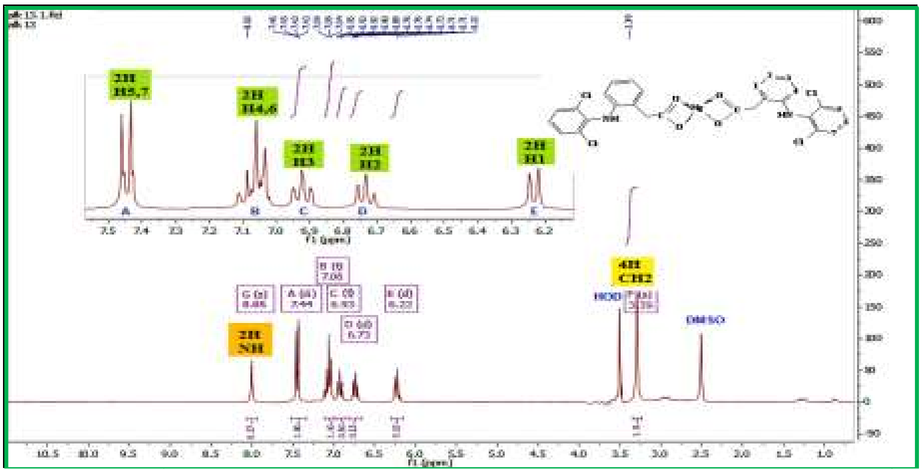


Fig. 4: The ^{1}H-NMR spectrum of [Hg(diclo)$_2$].2H$_2$O(1) complex in DMSO-d^6

The ^{1}H-NMR spectra of **[Hg(diclo)$_2$(Phen)](2)**(**Fig. 5**), **[Hg(diclo)$_2$(Bipy)] (3)**, and **[Hg(diclo)$_2$(en)](4)** displayed the expected signals for the diclofenac ligand as well as and diamines ligands.

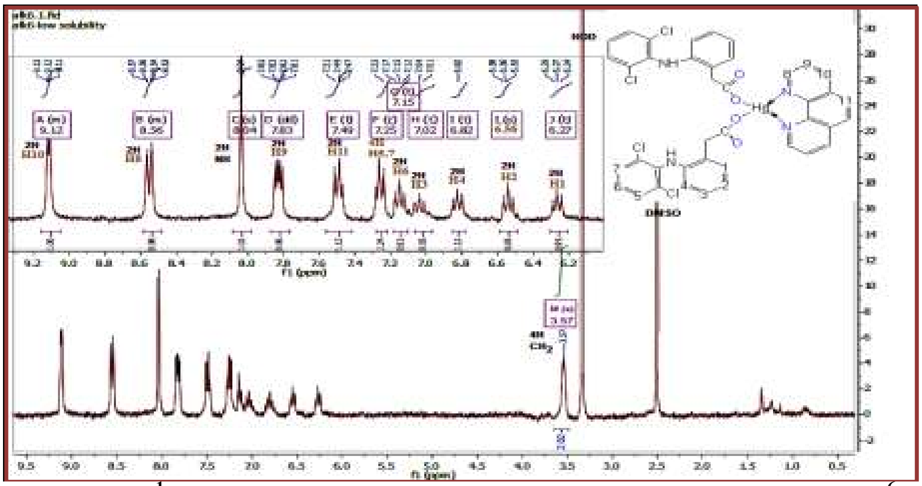


Fig. 5: The ^{1}H-NMR spectrum of [Hg(diclo)$_2$(Phen)](2) complex in DMSO-d^6

The ^{1}H-NMR spectrum of [Hg(diclo)$_2$(2-Apy)$_2$]**(7)** complex(**Fig. 6**) clearly displayed the expected peaks of diclofenac ligand as well as and 2-aminopyridine (2-Apy). The ^{1}H NMR spectrum showed a singlet peak at 5.87 ppm, which exchanged with D$_2$O and was assigned to the NH$_2$ group for **2-Apy**. The spectrum of **(7)** showed the protons of the amine ligand, at δ8.60ppm ($^3J_{H\text{-}H}$= 6.88Hz) as a doublet peaks, which assigned to the H9, and unresolved multiplets peaks withinδ(8.21-8.27)ppm for the H8, 10, 11.The other peak are listed in **Table 3**.

The ^{1}H-NMR spectra of [Hg(diclo)$_2$(Py)$_2$]**(5) (Fig. 7)**, [Hg(diclo)$_2$(3-Apy)$_2$]**(6)**, and [Hg(diclo)$_2$(2-Apmy)$_2$] **(8)** displayed the expected signals for the diclofenac ligand as well as and diamines ligands.

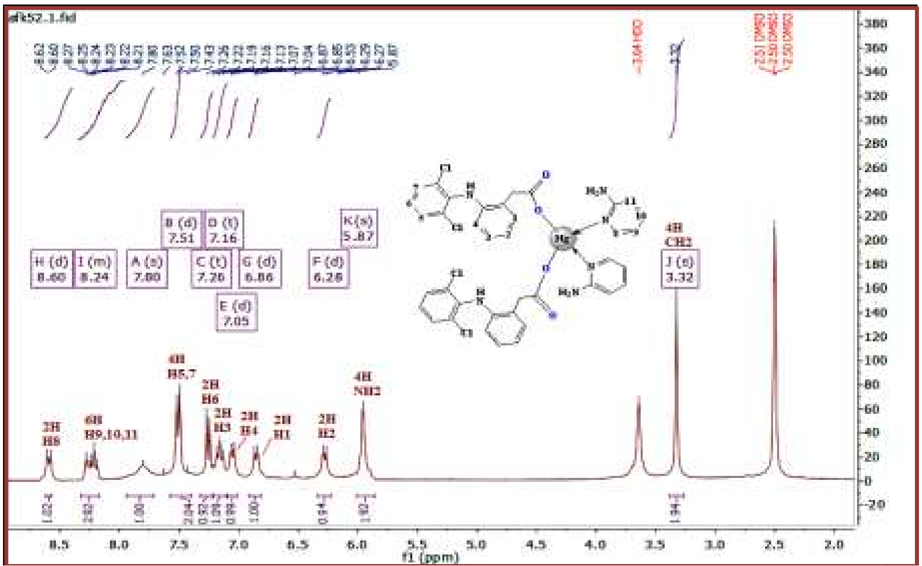


Fig. 6: The ^{1}H-NMR spectrum of [Hg(diclo)$_2$(2-Apy)$_2$]complex in DMSO-d^6

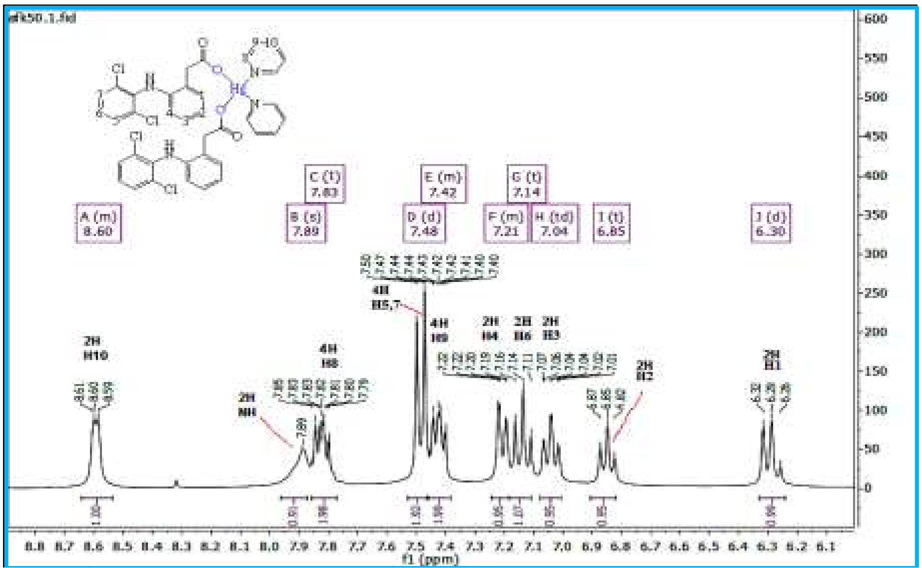
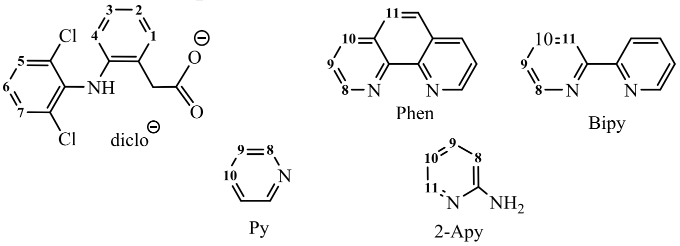


Fig. 7: The ^{1}H-NMR spectrum(6-8.7ppm range) of [Hg(diclo)$_2$(Py)$_2$]complex in DMSO-d^6

Table 3 : ^{1}H NMR data, chemical shifts (δppm) and coupling constants (Hz) for the some prepared complexes measured in DMSO-d^{6}



Complexes	δH * (ppm)
[Hg(diclo)$_2$].2H$_2$O	8.05(s, 2H, NH); 7.44(d,4H, $^{3}J_{HH}$=8Hz, H5,7); 7.06(m, 4H,H4,6); 6.93(t,2H, $^{3}J_{HH}$=8Hz,H3); 6.73(dd, 2H, H2); 6.22(d, 2H,H1).
[Hg(diclo)$_2$(Phen)]	9.12(d,2H, $^{3}J_{HH}$=8Hz, H10); 8.56(d,2H, $^{3}J_{HH}$=4Hz, H8); 8.04(s, 2H, NH);7.87(t,2H,H9); 7.49(d,2H,H11); 7.25(d, 4H, H5,7); 7.15(t,2H, $^{3}J_{HH}$=8Hz, H6); 7.02(t,2H, $^{3}J_{HH}$=4Hz, H3); 6.82(dd, 2H,H4); 6,56(t,2H, $^{3}J_{HH}$=8Hz, H2);6.27(dd, 2H,H1);3.57(s, 4H, CH$_2$).
[Hg(diclo)$_2$(Bipy)]	9.17(d,2H, $^{3}J_{HH}$=4Hz, H10); 8.82(d,2H, $^{3}J_{HH}$=8Hz, H8); 7.43(d, 2H, $^{3}J_{HH}$=8Hz, H11; 8.22(s, 2H, NH));7.84(m,2H,H9); 7.45(d,4H, $^{3}J_{HH}$=8Hz, H5,7); 7.06(m, 4H, H4,6); 6.93(d.d,2H, H3);6.75(d.d,2H,H2);6.25(d, 2H, $^{3}J_{HH}$=8Hz, H1); 3.58(s, 4H, CH$_2$).
[Hg(diclo)$_2$(en)]	8.24(s, 2H, NH(diclo)); 7.45(d,4H,H5,7); 7.06(m, 4H,H4,6); 6.93(t,2H, $^{3}J_{HH}$=8Hz,H3); 6.75(dd, 2H, H2); 6.25(d, 2H, $^{3}J_{HH}$=8Hz,H1); 4.38(s,4H,NH(amine)); 3.40(s, 4H, CH$_2$(diclo)); 2.88(s, 4H, CH$_2$(amine)).
[Hg(diclo)$_2$(Py)$_2$]	8.60(d,2H, $^{3}J_{HH}$=8Hz, H10); 7.89(b,2H,NH); 7.83(m, 4H,H8); 7.48(d,4H,$^{3}J_{HH}$=8Hz ,H5,7); 7.42(m,4H,H9);7.21(d,2H, $^{3}J_{HH}$=4Hz,H4); 7.14(t,2H, $^{3}J_{HH}$=8Hz,H6); 7.04(t,2H,$^{3}J_{HH}$=8Hz,H3); 6.85(dd, 2H,H2); 6.30(d,2H,H1); 3.67(s, 4H, CH$_2$).
[Hg(diclo)$_2$(2-Apy)$_2$]	8.60(d,2H, $^{3}J_{HH}$=8Hz, H8); 8.24(m,6H,H9,10,11); 7.80(b, 2H,NH(diclo)); 7.51(d,4H,$^{3}J_{HH}$=8Hz ,H5,7); 7.26(d,2H,H6);7.16(t,2H, $^{3}J_{HH}$=12Hz,H3); 7.05(d,2H,H4); 6.86(d,2H,$^{3}J_{HH}$=4Hz,H1); 6.28(d.2H, $^{3}J_{HH}$=8Hz,H2); 5.87(s,4H,NH(amine)); 3.32(s, 4H, CH$_2$).

s= singlet, d=doublet, m=multiple, b=broad, dd= doublet of doublets, t= triplet

Conclusions

In this work, the synthesis and characterization of Hg(II) metal complexes of diclofenac and amines as mixed ligand were investigated. In [Hg(diclo)$_2$] **(1)**, the diclofenate ligand behaves as a bidentate chelate through the oxygen atoms, whereas in complexes **(2-8)**, the **diclo** ligand behaves as a monodentate, to give of mononuclear complexes with a 1:1 (complex **1** : diamine) or 1:2(complex **1** : amine) stoichiometry

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تحضير وتشخيص معقدات الزئبق(II) – دايكلوفيناك مع الامينات كليكاندات مشاركة

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الملخص

حُضرت سلسلة من معقدات الزئبق (II) الحاوية على مزيج من ليكاندات الدايكلوفيناك (دواء مضاد للالتهابات) مع الامينات، وشخصت المعقدات المحضرة، وأظهرت نتائج الاشعة تحت الحمراء ان ليكاند الدايكلوفيناك يرتبط بشكل ثنائي السن من خلال مجموعة الكاربوكسيليت منقوصة الهيدروجين في المعقد (1) وبشكل احادي السن في المعقدات (2–8)، أما الامينات الثنائية ('1,1-بايبريدين ، 10,1– فينانثرولين، أثيلين ثنائي الامين) فقد ارتبطت بشكل ثنائي السن المخلبي مع ايون الزئبق الثنائي، في حين ارتبطت الامينات الاولية (بيريدين، 2–امينوبيريدين، 3–امينوبيريدين، 2–امينوبيرميدين،) بشكل احادي السن من خلال ذرة نيتروجين الحلقة غير المتجانسة معطية معقدات ذات شكل رباعي السطوح حول ايون الزئبق (II).