

Synthesis and Characterization of Mixed Ligands Cadmium (II) Complexes with N-Hydroxymethylsaccharinate and diphosphines

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Abstract

Reaction of cadmium(II) chloride $\text{CdCl}_2 \cdot 6\text{H}_2\text{O}$ with two equivalents of N-hydroxymethylsaccharin ($\text{Sac-CH}_2\text{OH}$) (1) in the presence of NEt_3 afforded the presumably tetrahedral complex $[\text{Cd}(\text{K}^2\text{-Sac-CH}_2\text{O})_2]$ (2). The $\text{Sac-CH}_2\text{O}$ ligand behaves as a bidentate chelate. Reaction of (2) with one equivalent of the bidentate ligands (dipos); $\text{Ph}_2\text{P}(\text{CH}_2)_n\text{PPh}_2$, ($n = 1$ dppm; 2 dppe; 3 dppp; 4 dppb), $\text{Ph}_2\text{P}(\text{S})(\text{CH}_2)\text{P}(\text{S})\text{Ph}_2$ (dppmS₂) afforded tetrahedral complexes of the type $[\text{Cd}(\text{K}^1\text{-Sac-CH}_2\text{O})_2(\text{dipos})]$ (3-7). The reaction with two equivalents of the monodentate ligands (phos); Ph_3P or Ph_3PS gave $[\text{Cd}(\text{K}^1\text{-Sac-CH}_2\text{O})_2(\text{phos})_2]$ (8,9). The N-hydroxymethylsaccharinate anion acts as a monodentate ligand to the cadmium center through the hydroxymethyl oxygen atom. The complexes were characterized by physico-chemical and spectroscopic methods.

Keywords: Cadmium, saccharine derivative, phosphine.

1. Introduction

Saccharin, 1,2-benzisothiazole-3-one-1,1-dioxide, is a well-known heterocyclic compound and has been used as a sweetener in the form of its sodium salt since 1885. It is also a heterocyclic molecule of pharmaceutical importance and one of the key structural elements of certain CNS-active drugs [1].

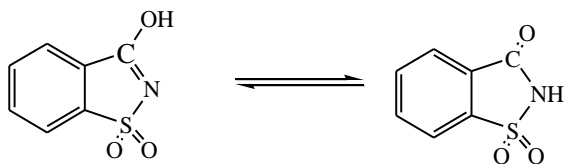


Fig 1: Enol - keto form of saccharin

Benzothiazine derivatives such as, piroxicam, meloxicam, droxicam, ampiroxicam, sudoxicam, and cinoxicam [2-9], are the well known oxicam drugs which possess potent biological activities [5,10-14]. Complexes of piroxicam, and droxicam with metal ions such as Cu(II), Cd(II), Pd(II) and other ions, have been recently been synthesized and characterized [15-17].

Although complexes of saccharin with transition and non transition metals have been well studied, complexes of saccharine derivatives such as N-hydroxymethylsaccharin are rare. In this study, we report synthesis, characterization and biological studies of Cadmium(II) mixed ligands complexes with N-hydroxymethylsaccharin and phosphines as co-ligands.

2. Experimental

2.1. General

All the reagents, starting materials as well as solvents were purchased commercially and used without any further purification. Melting points were recorded on a hot stage Gallen Kamp melting point apparatus. Elemental CHN, analysis were carried out on a 1106 Carlo-Erba analyzer. Infrared spectra were recorded in the 4000 – 200 cm^{-1} range using a Bruker Tensor

28 spectrometer with a Platinum ATR unit. Conductivity measurements were carried out by using a WTW conductivity meter. The ¹H- NMR spectra were recorded on Varian unity 400 spectrometers with DMSO-d₆ as solvent and Me₄Si as internal reference. ³¹P NMR spectra were recorded on Gemini 200 spectrometer with DMSO-d₆ as solvent and H₃PO₄(85%) as external reference. The NMR spectra and element analysis were determined in the Institute für Anorganische Chemie, Martin-Luther-Universität, Halle, Germany.

2.2 Preparation of N-hydroxymethylsaccharin (Sac-CH₂OH) (1)

A mixture of saccharin (5.00 g, 27 mmol) and formalin 37% (5 ml, 67 mmol) in water (20 ml) was heated under reflux for 1 h., then cooled at room temperature, the white crystalline solid formed was filtered off and dried in oven. (Yield: 5.1g, 88%. m.p.: 134-136 °C (lit. 135-137)[18].

2.3 Synthesis of [Cd(Sac-CH₂O)₂] (2)

An aqueous solution of Cadmium chloride hydrate ($\text{CdCl}_2 \cdot 6\text{H}_2\text{O}$) (0.099 g, 0.340 mmole) (10ml) was added to a hot aqueous solution of $\text{Sac-CH}_2\text{OH}$ (0.145 g, 0.680 mmole) (10 ml) containing few drops of Et_3N as a base. The mixture was stirred at room temperature for 2h. The white precipitate formed was filtered, washed with water and dried in oven. (Yield: 0.158g, 86%. m.p.: 270 °C decom.)

2.4 Synthesis of [Cd(Sac-CH₂O)₂(dppm)] (3)

A solution of dppm (0.050 g, 0.130 mmole) in CHCl_3 (10 ml) was added to a suspension of $[\text{Cd}(\text{Sac-CH}_2\text{O})_2]$ (0.069 g, 0.130 mmole) in CHCl_3 (10 ml) to give a clear solution. The solution was refluxed for 4h at 50°C, and the resulting mixture was left to cool at room temperature. The white precipitate formed was filtered, washed with ethanol, chloroform (10 ml each) and dried in oven. Recrystallization from DMSO/EtOH (3:1 v/v) gave the product as a white powder (Yield: 0.088g, 74%. m.p.: >300 °C (decom.).

The following complexes $[\text{Cd}(\text{Sac-CH}_2\text{O})_2(\text{dppe})]$ (4), $[\text{Cd}(\text{Sac-CH}_2\text{O})_2(\text{dppp})]$ (5), $[\text{Cd}(\text{Sac-CH}_2\text{O})_2(\text{dppb})]$ (6) and $[\text{Cd}(\text{Sac-CH}_2\text{O})_2(\text{dppmS}_2)]$ (7) were prepared and isolated in a similar method.

2.5 Synthesis of $[\text{Cd}(\text{Sac-CH}_2\text{O})_2(\text{PPh}_3)_2]$ (8)

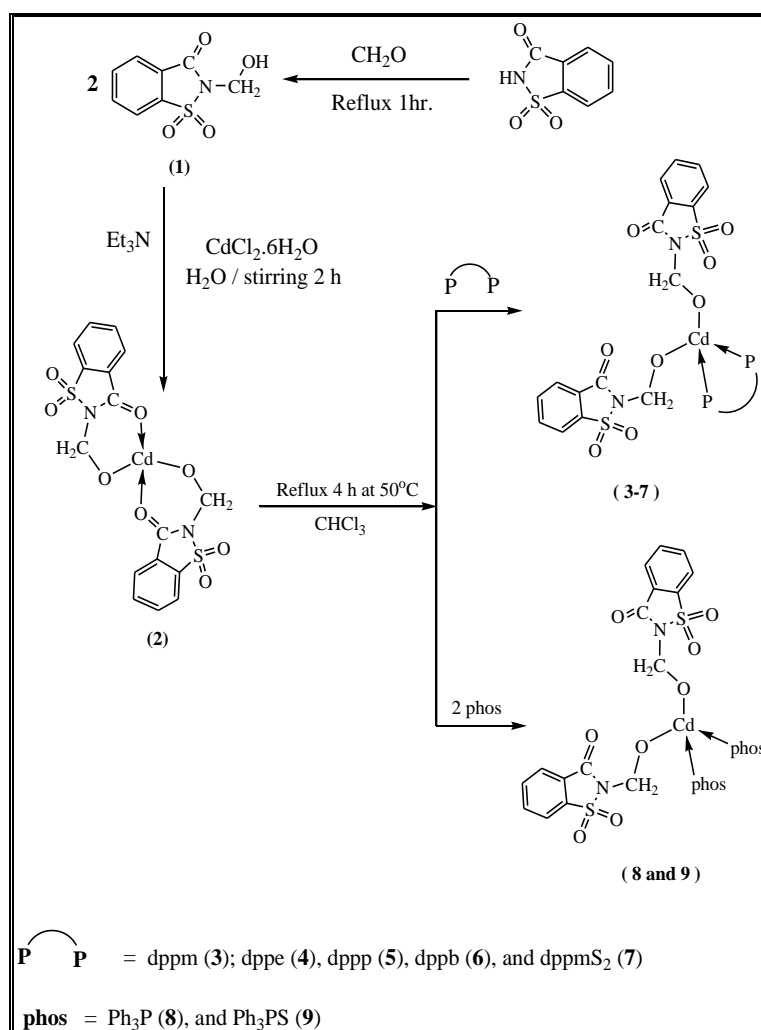
A solution of PPh_3 (0.068 g, 0.230 mmol) in CHCl_3 (15 ml) was added to a suspension of (2) (0.069 g, 0.130 mmol) in CHCl_3 (15 ml) with stirring. The clear solution formed was refluxed for 4 h and then left to cool at room temperature. The white solid formed was filtered, washed with diethyl ether, CHCl_3 and dried under vacuum (Yield: 0.112g, 82%. m.p.: 223-225°C).

The complex $[\text{Cd}(\text{Sac-CH}_2\text{O})_2(\text{S=PPh}_3)_2]$ (9) was prepared and isolated in a similar method.

3. Results and Discussion

3.1 Synthesis of complexes (2-9)

The cadmium complex $[\text{Cd}(\text{Sac-CH}_2\text{O})_2]$ (2) was synthesized by treatment of two moles equivalent of the ligand $\text{Sac-CH}_2\text{OH}$ with one mole equivalent of cadmium chloride hydrate in aqueous solution, in the presence of Et_3N as a base. The reaction of sac derivative complex (2) with different diphosphines in an equivalent molar ratio afforded $[\text{Cd}(\text{Sac-CH}_2\text{O})_2(\text{diphos})]$ (diphos: dppm, dppe, dppp, dppb and dppmS_2) (3-7) in 67–87 % yields, as illustrated in Scheme 3. The same reaction with two mole equivalents of the monophosphines (phos) gave rise to $[\text{Cd}(\text{Sac-CH}_2\text{O})_2(\text{phos})_2]$ (8 and 9) (phos: PPh_3 and S=PPh_3) as white solids in 82-79 % yields (Scheme 1).



Scheme 1: Preparation of complexes 2-9

All the cadmium(II) complexes are solids and stable towards air and moisture at room temperature. They decompose on heating at high temperature (Up to 350°C). The physical properties data of the compounds are given in Table 1.

3.2 Infrared spectra of compounds (1-9)

The i.r. data of the ligand (1) and its complexes are listed in Table 3. The IR spectra of the complexes

were compared with the spectra of the free ligands ($\text{Sac-CH}_2\text{OH}$ and phosphines) in order to determine the involvement of the coordination sites.

IR spectrum of the $\text{Sac-CH}_2\text{OH}$ (1) ligand exhibit the most characteristic bands at 3290 cm^{-1} , $\nu(\text{O-H})$, 3095 cm^{-1} , $\nu(\text{C-H})$, 2975 cm^{-1} , $\nu(\text{C-H})$, 1747 cm^{-1} , $\nu(\text{C=O})$, 1559 cm^{-1} , $\nu(\text{C=C})$, and 1340 cm^{-1} , $\nu(\text{SO}_2)_{\text{asy}}$; 1184 cm^{-1} , $\nu(\text{SO}_2)_{\text{sy}}$ [18]. In the IR

spectrum of the complex $[\text{Cd}(\text{Sac-CH}_2\text{O})_2]$ (**2**) (Fig. 2), the $\nu(\text{O-H})$ 3298 cm^{-1} stretching vibration found in the spectrum of the free ligand (**1**) is not observed, suggesting a deprotonation of the hydroxyl group [18]. The asymmetric (ν_{as}) and symmetric (ν_{s}) stretching of the SO_2 group are observed as strong IR bands at 1290 cm^{-1} and 1184 cm^{-1} respectively [19-22]. The carbonyl group C=O stretching vibration that appeared at 1747 cm^{-1} in the spectrum of **Sac-CH₂OH** (**1**) ligand shifted towards a lower frequency (1696 cm^{-1}) in complex (**2**). This shift confirms the participation of the oxygen atom in the $\text{C=O} \rightarrow \text{M}$ bond. This indicating coordination of the ligand to the cadmium as a bidentate chelating ligand ($K^2\text{-O,O}$), via the oxygen atoms of the carbonyl and the deprotonated hydroxyl groups respectively [23,24]. Other bands are listed in the Table 2

The IR spectra of complexes **3-9** (Fig. 1 and 2), displayed the $\nu(\text{C=O})$ band at $1709\text{-}1731\text{ cm}^{-1}$ indicating a un coordination of the carbonyl group and the $\text{Sac-CH}_2\text{O}$ anion ligand behaves as a monodentate bonded with cadmium through the oxygen atom of the CH_2O^- group only [23,24]. The strong bands at $1433\text{-}1436\text{ cm}^{-1}$, assigned to the $\nu(\text{C}_6\text{H}_5\text{-P})$ grouping. It is thought that this vibration arises by the deformation of the planarity of the phenyl ring bonded to a heavy atom (phosphorus) [25-27]. The new bands appeared $493\text{ - }513\text{ cm}^{-1}$ respectively, assigned to the $\nu(\text{P-C})$ [24]. The IR spectrum of complex **6** displayed the $\nu(\text{S=P})$ group at 664 cm^{-1} , and complex **7** at 658 cm^{-1} [28].

3.3 NMR spectra of compounds (2, 6-8)

The ^1H NMR spectrum of complex **2** measured in dmsO-d_6 showed a singlet at $\delta 5.23$ ppm assigned to the protons of the methylene groups. The phenyl protons in position 1 and 4 appeared as a doublet each at $\delta 8.18$ and 8.08 ppm, with $^3J_{\text{H-H}} = 7.88$ Hz respectively, whereas the protons in position 2 and 3 appeared as a doublet of doublets at $\delta 7.66$ and 7.98 ppm ($^3J_{\text{H-H}} = 7.88\text{ Hz}$; $^3J_{\text{H-H}} = 1.48\text{ Hz}$). In the ^{13}C nmr spectrum the carbonyl carbon atom appeared at 172.35 and the methylene group at 62.58 ppm [29, 30].

The $^{31}\text{P}\{-^1\text{H}\}$ NMR spectra of the complexes (**6-8**) displayed a singlet at 37.38 ppm for complex (**6**), at 26.29 ppm for complex (**7**) and at 24.76 ppm for complex (**8**) (Fig. 3,4). The singlet indicates the presence of a single product for each complex.

The $^1\text{H}\{-^{31}\text{P}\}$ NMR spectrum of the $[\text{Cd}(\text{Sac-CH}_2\text{O})\text{dppb}]$ (**6**) complex (Fig. 5), displayed two broad singlets at $\delta 1.53$ ppm and $\delta 2.71$ ppm, each represent two protons, assigned for the two CH_2 groups of phosphine ligand and a singlet at $\delta 5.25$ ppm assigned to $-\text{OCH}_2^-$. While the $^1\text{H}\{-^{31}\text{P}\}$ NMR spectrum of the $[\text{Cd}(\text{Sac-CH}_2\text{O})\text{dppmS}_2]$ (**7**) showed two singlets at $\delta 2.95$ ppm and $\delta 5.10$ ppm, assigned to the methylene groups of the dppmS_2 and $-\text{OCH}_2^-$ groups respectively. The $^1\text{H}\{-^{31}\text{P}\}$ NMR spectrum of the $[\text{Cd}(\text{Sac-CH}_2\text{O})(\text{PPh}_3)_2]$ (**8**), showed a singlet at $\delta 5.10$ ppm assigned to the $-\text{OCH}_2^-$ group while the phenyl protons of PPh_3 and $\text{Sac-CH}_2\text{O}^-$ ligands appeared within $\delta 6.96\text{ - }8.22$ ppm range as unresolved multiplets.

Table 1: Color, yield %, m.p. and elemental analysis for the prepared complexes

Seq.	Complexes	Color	m.p (°C)	Yield %	Elemental analysis Calc. (Found) %		
					C	H	N
1	Sac-CH ₂ OH	White	134-136	88	45.07 (45.11)	3.31 (3.40)	6.57 (6.62)
2	$[\text{Cd}(\text{Sac-CH}_2\text{O})_2]$	White	270 ^a	69	35.80 (36.11)	2.25 (2.54)	5.22 (5.18)
3	$[\text{Cd}(\text{Sac-CH}_2\text{O})_2\text{dppm}]$	White	>300 ^a	74	53.46 (53.77)	3.72 (3.87)	3.04 (2.97)
4	$[\text{Cd}(\text{Sac-CH}_2\text{O})_2\text{dppe}]$	White	243-245	87	53.94 (54.20)	3.88 (4.01)	3.00 (3.24)
5	$[\text{Cd}(\text{Sac-CH}_2\text{O})_2\text{dppp}]$	White	286-289	82	54.41 (54.57)	4.03 (3.98)	2.95 (3.11)
6	$[\text{Cd}(\text{Sac-CH}_2\text{O})_2\text{dppb}]$	White	253-255 ^a	67	54.84 (54.62)	4.19 (4.10)	2.91 (3.04)
7	$[\text{Cd}(\text{Sac-CH}_2\text{O})_2\text{dppmS}_2]$	White	181-183	72	49.98 (50.02)	3.48 (3.56)	2.84 (3.08)
8	$[\text{Cd}(\text{Sac-CH}_2\text{O})_2(\text{PPh}_3)_2]$	White	223-225	82	58.84 (59.11)	3.99 (3.67)	2.64 (2.53)
9	$[\text{Cd}(\text{Sac-CH}_2\text{O})_2(\text{SPPH}_3)_2]$	White	189-192	79	55.49 (55.81)	3.76 (4.05)	2.49 (2.38)

a: decompose temperature

Table 2: Selected IR stretching vibration bands (cm⁻¹) of prepared complexes

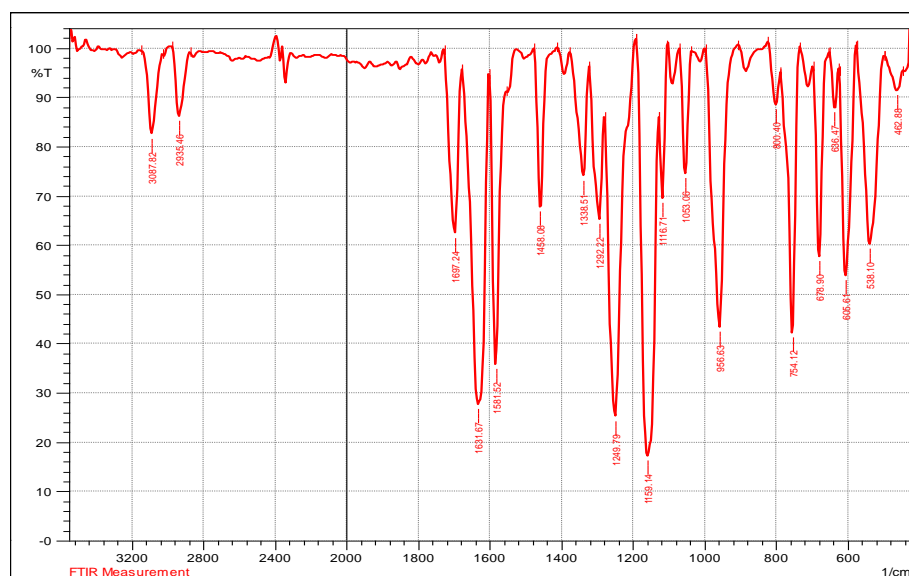
Seq.	Complexes	$\nu(\text{CH})$ arom.	$\nu(\text{CH})$ aliph.	$\nu(\text{C}=\text{O})$	$\nu(\text{SO}_2)$ asy/sy	$\nu(\text{P-Ph})$	$\nu(\text{P-C})$
1	Sac-CH ₂ OH	3093w	2975w	1747s	1340s 1184s		
2	[Cd(Sac-CH ₂ O) ₂]	3087w	2935w	1696s	1292s 1159s		
3	[Cd(Sac-CH ₂ O) ₂ dppm]	3067w	2939w 2889w	1724s	1294s 1159s	1436s	505s
4	[Cd(Sac-CH ₂ O) ₂ dppe]	3055w	2948w 2893w	1714s	1290s 1162s	1434s	513m
5	[Cd(Sac-CH ₂ O) ₂ dppp]	3094w	2939w 2870w	1731s	1288s 1159s	1433s	500s
6	[Cd(Sac-CH ₂ O) ₂ dppb]	3090w	2890w 2871w	1718s	1295s 1172s	1434s	505s
7	[Cd(Sac-CH ₂ O) ₂ dppmS ₂]	3078w	2932w 2893w	1709s	1304s 1163s	1434s	503s
8	[Cd(Sac-CH ₂ O) ₂ (PPh ₃) ₂]	3058w	2969w 2870w	1726s	1309s 1178s	1436s	493s
9	[Cd(Sac-CH ₂ O) ₂ (SPPH ₃) ₂]	3053w	2988w 2810w	1724s	1290s 1158s	1433s	506s

arom. = aromatic; aliph = aliphatic; w = weak; s = strong; m = medium

Table 3: ³¹P-¹H and ¹H NMR chemical shifts for the prepared compounds in (DMSO-d₆) solvent

Compounds	Chemical shift δ (ppm)	
	³¹ P- ¹ H NMR	¹ H - ³¹ P NMR
Sac-CH ₂ OH	-	8.28 (dd, 1H, H4, H1); 8.12 (dd, 1H, H4); 8.05 (td, 1H, H3); 7.99 (td, 1H, H1); 6.74 (bs, 1H, OH); 5.18 (s, 2H, OCH ₂).
[Cd(Sac-CH ₂ O) ₂]	-	8.18 (d, 1H, H1); 8.08 (d, 1H, H4); 7.98 (dd, 1H, H3); 7.66 (dd, 1H, H2); 5.23 (s, 2H, OCH ₂).
[Cd(Sac-CH ₂ O) ₂ dppb]	37.38	8.22–7.04 (m, 28H, phenyl), 5.25(s, 4H, OCH ₂), 2.71 (bs, 4H, CH ₂), 1.53 (bs, 4H, CH ₂).
[Cd(Sac-CH ₂ O) ₂ dppmS ₂]	26.29	8.22–6.96 (m, 28H, Ar), 5.10 (s, 4H, OCH ₂), 2.95 (s, 2H, CH ₂).
[Cd(Sac-CH ₂ O) ₂ (PPh ₃) ₂]	24.76	8.22–6.96 (m, 38H, phenyl), 5.10 (s, 4H, OCH ₂),

s: singlet; d: doublet; dd: doublet of doublets; td: triplet of doublets; m: multiplet; bs: broad singlet.

**Fig. 1: IR spectrum of [Cd(Sac-CH₂O)₂] complex**

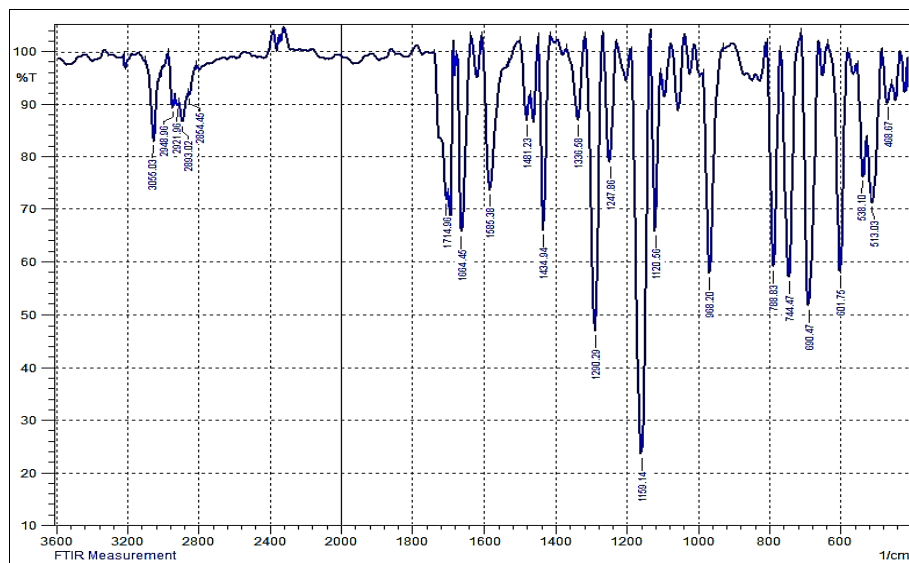


Fig 2: IR spectrum of [Cd(Sac-CH₂O)₂(dppp)] complex

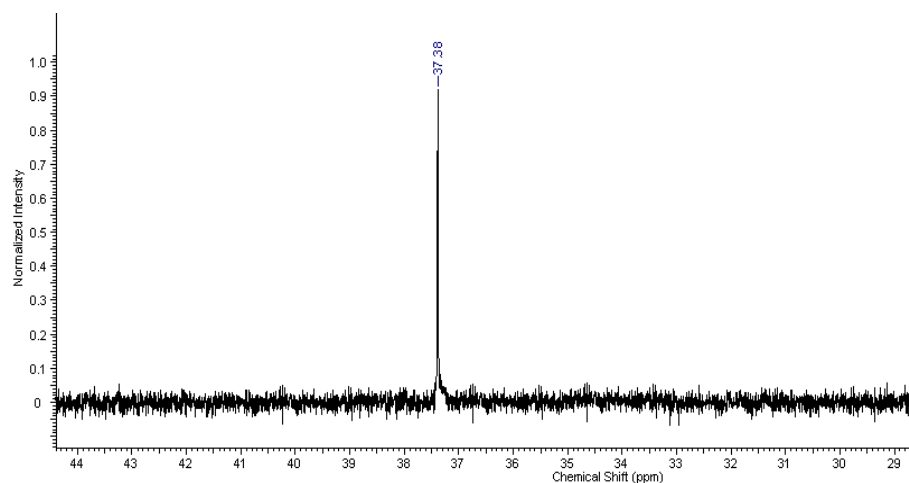


Fig. 3: ³¹P-{¹H} NMR spectrum of [Cd(Sac-CH₂O)₂(dppb)] complex

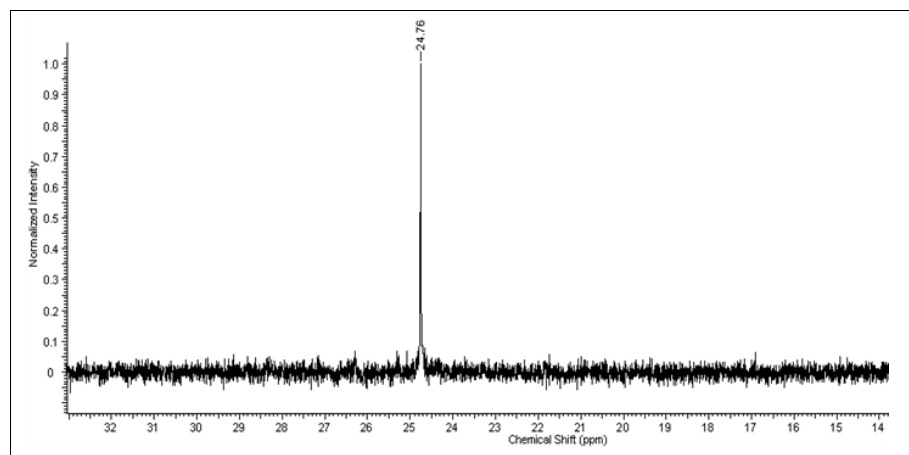


Fig. 4: ³¹P-{¹H} NMR spectrum of [Cd(Sac-CH₂O)₂(PPh₃)₂] complex

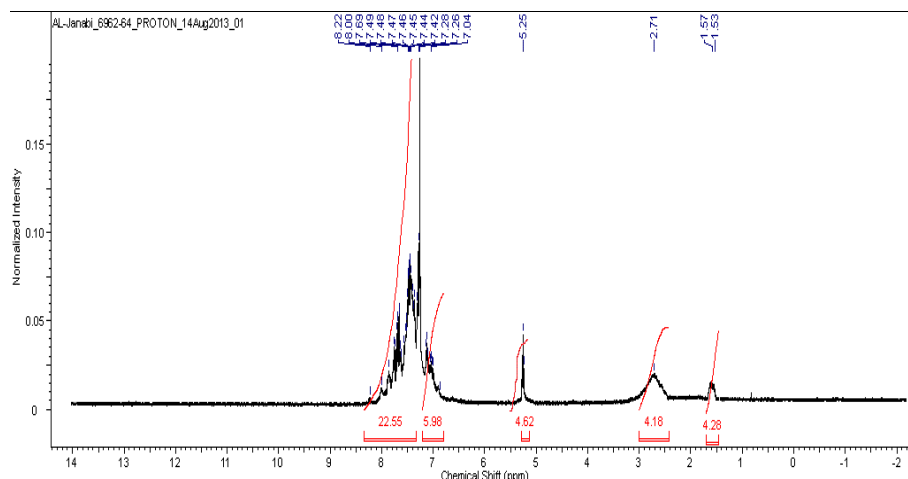


Fig. 5: $^1\text{H}\{-^{31}\text{P}\}$ NMR spectrum of $[\text{Cd}(\text{Sac-CH}_2\text{O})_2(\text{dppb})]$ complex

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تحضير وتشخيص معقدات الكادميوم (II) الحاوية على مزيج من ليكاندات N-هيدروكسي مثيل سكارينيت وثنائي الفوسفينات

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الملخص

يتفاعل مول واحد من كلوريد الكادميوم (II) $CdCl_2 \cdot 6H_2O$ مع مولين مكافئين من ليكاند N-هيدروكسي مثيل سكارينيت (Sac-CH₂OH) (1) بوجود ثلاثي اثيل امين NEt_3 ليعطي معقد رباعي السطوح $[Cd(K^2-Sac-CH_2O)_2]$ (2) الذي يسلك فيه الليكاند $Sac-CH_2O^-$ سلوك ليكاند ثنائي السن المخليبي. يعطي تفاعل المركب (2) مع مكافئ واحد من ليكاندات ثنائي الفوسفينات (diphos) $Ph_2P(CH_2)_nPPh_2$ حيث (n=4 dppb; n=3 dppp; n=2 dppe; n=1 dppm) $Ph_2P(S)CH_2(S)PPh_2$ (dppmS₂) معقدات رباعية السطوح من النوع $[Cd(K^1-Sac-CH_2O)_2(diphos)]$ (3-7) اما تفاعل المركب (2) مع مولين مكافئين من ليكاندات احادي الفوسفين Ph_3P (phos) او Ph_3PS فانه يعطي المعقدات $[Cd(K^1-Sac-CH_2O)_2(phos)_2]$ (8 و9)، يسلك الليكاند N-هيدروكسي مثيل سكارينيت في هذه المعقدات سلوك ليكاند احادي السن يرتبط مع الكادميوم عن طريق ذرة اوكسجين مجموعة هيدروكسيل المثل CH_2O^- . شخصت المعقدات المحضرة باستخدام الطرق الطيفية والكيمووظيفية.

كلمات الدلالية: الكادميوم ، مشتق السكارين ، الفوسفينات.