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STUDIES ON SOME NEW COORDINATION COMPOUNDS OF LEAD WITH SEMICARBAZONES AND THIOSEMICARBAZONES

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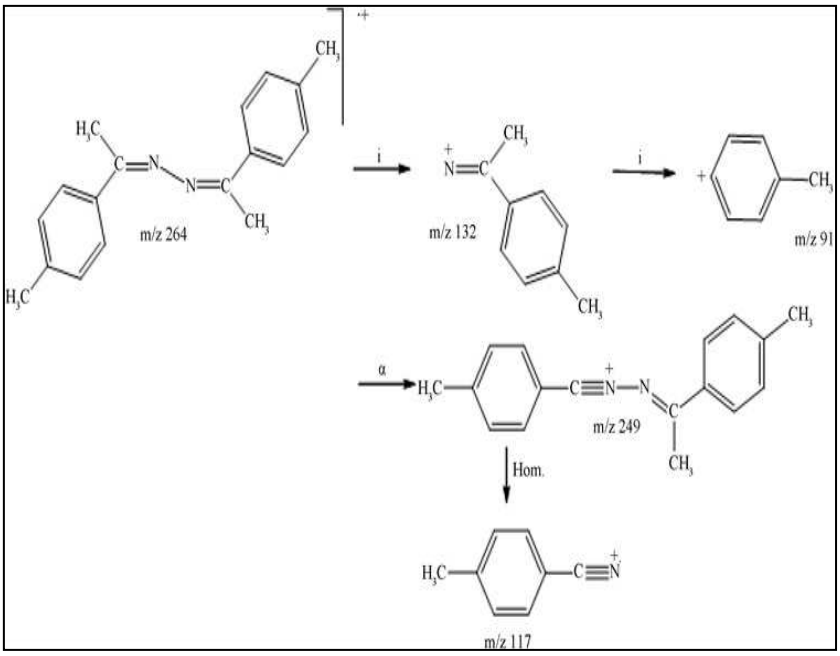


ABSTRACT:

The present paper is a report on the synthesis of some new lead complexes by the reaction of diphenyl lead dichloride with semicarbazones and thiosemi carbazones. Semicarbazones and thiosemicarbazones used in these studies are synthesized by the condensation of 1-acetyl-2-naphthol, 2-acetyl-1-naphathol, 2-acetyl-5-methyl furan, 2-acetyl-4-methyl thiophene and 2-acetyl-naphthalene with semicarbazide/ thiosemi carbazide. The bonding pattern and geometry of lead complexes are characterized by spectroscopic evidences. The ligands and their metal complexes have been screened for antitubercular, antibacterial and antifungal activities and are found quite active in this respect. Plant growth activity has also been evaluated and is found negative growth.

KEYWORDS: *Semicarbazones, Thiosemicarbazones, Antibacterial activities, Spectroscopic evidence*

INTRODUCTION

Schiff base ligands are able to coordinate metals through imine nitrogen or another group, usually linked to aldehyde or ketone. These ligands represent the most widely utilized classes of ligand in metal coordination chemistry. Their complexes find many important catalytic applications to various types of polymerization. The real impetus towards developing the coordination chemistry of these potential ligands was probably provided by the remarkable, antitumer, antiviral and antimalarial activity observed for some of these derivatives which have been shown to be related to their complexing ability. Semicarbazones and thiosemicarbazones are also similar and most important nitrogen oxygen / sulphur donor ligands because of them act as neutral or charged ligand moieties. Therefore, the present paper is an effort to describe structural characterization of some new compounds lead (II) & (IV) with semicarbazones and thiosemicarbazones.

EXPERIMENTAL

Analytical methods and physical measurements

Lead was estimated gravimetrically as EDTA. Nitrogen and sulphur were estimated by Kjeldahl's method and Messenger's method, respectively. The IR spectra were recorded on FTIR spectrophotometer using a model A-8400 S, Shimadzu in KBr pellets. The electronic spectra were taken with a Toshniwal spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded on

JEOL AL-300 spectrometer. Molar conductance measurements were made in anhydrous dimethyl formamide at 36±1°C using a model 305 systronics conductivity bridge. Molecular weight determinations were carried out by the Rast Method.

SYNTHESIS OF LIGANDS

Semicarbazones and thiosemicarbazones were synthesized by the condensation of aldehydes/ketones viz. 1-acetyl-2-naphthol, 2-acetyl-1-naphathol, 2-acetyl-5-methyl furan, 2-acetyl-4-methyl thiophene and 2-acetyl-naphthalene with semicarbazide/thiosemicarbazide in 1:2 molar ratio using absolute alcohol as the reaction medium. The mixture was heated on a water bath for about half an hour and then allowed to cool at room temperature. The crystals that separated out were recrystallized from the same solvent. Their physical properties and analysis have been recorded in Table-1.

SYNTHESIS OF LEAD (IV) COMPLEXES

Lead (IV) complexes were prepared by the reaction of diphenyl lead dichloride with above mentioned semicarbazones and thiosemicarbazones in 1:2 molar ratio in sodium salt. The mixture was refluxed on refluxing column for one hour. The solvent was removed and the product was finally dried in vacuo at 40-50°C. Their physical properties and analysis have been recorded in Table-2.

RESULTS AND DISCUSSION

The reactions of diphenyl lead dichloride with, these ligands are as follows:

$$(C_6H_5)_2PbCl_2 + 2NaSCZ \longrightarrow (C_6H_5)_2Pb(SCZ)_2 + 2NaCl$$

$$(C_6H_5)_2PbCl_2 + 2NaTSCZ \longrightarrow (C_6H_5)_2Pb(TSCZ)_2 + 2NaCl$$

Where SCZ = Semicarbazones, TSCZ = Thiosemicarbazones

The resulting complexes are obtained as coloured solids which are soluble in DMF and DMSO. These complexes are sensitive to moisture.

ELECTRONIC SPECTRA

The electronic spectra of ligands and their coordination compounds with lead have been recorded in methanol. The spectra of ligands show maximum at 350 nm due to $n \rightarrow \pi^*$ transition of the non bonding electrons present of the nitrogen atom of the azomethine group. In the spectra of the corresponding coordination compounds, this band undergoes hypsochromic shift due to coordination through nitrogen atom. Two other bands in the spectra of ligands at 264 nm and 300 nm due to $\pi \rightarrow \pi^*$ transition of electrons remain unaltered on coordination.

IR Spectra

(i) All the ligands display a strong band in the region 1600-1620 cm^{-1} which is due to ν(>C=N–) stretching frequency in the free ligands. This band gets shifted in lower frequency region (1590 cm^{-1}) showing the coordination of nitrogen to lead atom.

(ii) The infrared spectra of ligands exhibit broad peak in the region 3100-3400 cm^{-1} due to ν(OH). The absence of ν(OH) band in the IR spectra of the compounds provide an evidence that the ligand is coordinated to lead atom, in its deprotonated form. The shifting of ν(C-O) band at 1260 cm^{-1} towards higher frequency in the compounds show chelation through oxygen atom.

(iii) The ν(N-N) at ~970 cm^{-1} in the ligand spectra also gets shifted towards higher frequency region as a result of complex formation and further support the above mode of coordination.

(iv) New medium intensity bands in the far IR region 470-485 cm^{-1} in the lead compounds may be attributed to *v*(Pb←N) vibrations. Band in the region 518-525 cm^{-1} is attributed to *v*(Pb-O) stretching vibrations indicating the coordination of metal through the oxygen atom. The IR spectral data were recorded in Table 5.

^{1}H NMR SPECTRA

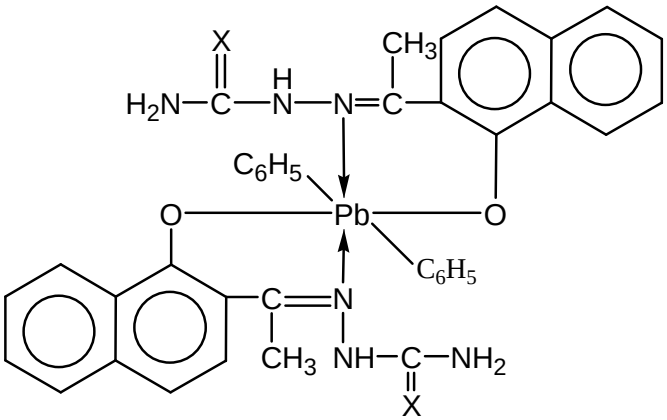
In proton magnetic resonance spectra a hydrogen bonded phenolic proton signal observed at 12.20±5 ppm in the ligand, is absent in the lead compounds showing the complexation of the lead by the phenolic oxygen after deprotonation.

The signal due to methyl proton [-C(CH_3)=N-] at δ1.83-1.88 ppm in ligands is shifted downfield in the compounds and appears at δ 1.94-1.99 ppm. This is probably due to the donation of the lone pair of electrons by the nitrogen to the lead atom to form coordinate linkage. A broad signal at δ 3.50 ppm due to NH_2 protons remain almost unchanged in the spectra of lead complexes which clearly indicates the non involvement of this group in complexation .The ligands also exhibit NH proton signal at δ 10.15 ppm, remain on same position due to non involvement of this group in complexation. The ^{1}H NMR spectral data were recorded in Table 6.

^{13}C NMR SPECTRA

^{13}C NMR spectra of the ligands and their lead compounds have been recorded and the data are given in Table 8. The ^{13}C chemical shifts of the spectra of compounds compared to the ligands clearly show the coordination of the azomethine nitrogen and oxygen to the lead atom.

Thus on the basis of the above discussion, it is evident that the ligands coordinate through the azomethine nitrogen and attach oxygen of phenolic group after deprotanation to the lead ion. The Pb(IV) compounds may be represented by the following structure (Fig 1).



X= O or S

Fig. 1. Lead (IV) complex

ANTIBACTERIAL ACTIVITY:

All the synthesized ligand and their correspond Lead complexes were screened in vitro for their antibacterial activity against Gram-negative (*E.coli* and *P. milamilis*) and Gram-positive (*B.thuringiensis* and *S.aureus*) bacterial strains using paper disc plate method. The nutrient agar medium (peptone, beef extract, NaCl and agar-agar) and 5mm diameter papar disc of Whatman filter paper No.1 were used. The compounds under investigation were dissolved in methanol to give concentration of 500 and 1,000 ppm. The plates were

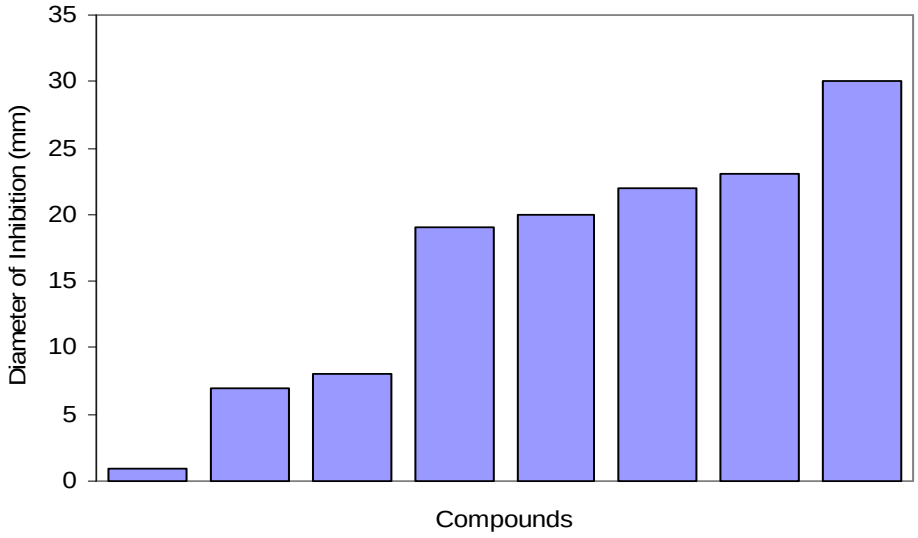
incubated for 48 h at 28±2°c and inhibition zone around each disc was measured. The antibacterial activity displayed by various compound is shown in Table-3.

ANTIFUNGAL ACTIVITY:

The antifungal activity was evaluated against *A .flavus, F.oxysporum, A.niger and R.phaseoli* by the agar palate technique. Solutions of the compounds in different concentrations in DMF were then mixed with the medium. The linear growth of the fungus was recorded by measuring the diameters of the fungus colony in the control and test plates, respectively as shown in Table-4.

ANTITUBERCULAR ACTIVITY:

The YT agar medium was prepared using 1% yeast extract, 2% trypton, 1.5% agar, 1% NaC1 in 250 mL distilled water by maintaining the pH of the medium at 7 using 10% NaOH solution This medium was then sterilized by autoclaving at 120°C for 15 mm. After cooling to 50°C the medium was poured into 85 mm diameter Petri dishes (approx. 25 mL each) and setting aside at 37°C. After a few hours, Petri dishes were stored in the cold room at 4°C. Freshly prepared 100 mL of inoculum of Micobacterium smegmatis was spread in each dish and 20 mL (100 mg) solution of the test compound was poured in each well. 20 mL DMSO was used as negative control. The plates were kept at 37°C for 24 h after which the diameter of the inhibition zones was measured (Bar diagram). Ciprofloxacin was used as a standard reference drug for comparison.



PLANT GROWTH ACTIVITY:

Lead (IV) complexes were tested for their plant growth regulating activity against gram plant. The observations for percent germination and normal seedling were recorded on the 4th and 8th days. The seedlings, which possessed the ability to develop into fully normal and healthy plants, were considered as normal seedlings.

The seeds were treated with physiologically active concentration of the plant growth regulators solution for six h at room temperature and drying them to the original moisture level by a hot air circulating oven. After that, uniform size seeds were placed on Whatman no. 1 filter paper lying in the glass petri plates. Each petri plate has 15 seeds placed at equidistance. The filter papers were moistened with fresh solutions of required concentrations. The concentrations of the plant growth regulators used were 1,5,10, and 25 ppm.

Table - Effect of ligands and lead (II) and (IV) complexes on the concentrations of the plant growth regulators used was 1, 5, 10, 20 and 25 ppm.

Group	Treatment	Plant Growth Regulators (ppm)	PGIZ (mm)		PGAI(mm)		Plant Growth %
A	Control	0	0	0	0	0	0
B	L^1H	1	0.0	0.0	0.00	0.0	0.0
C	L^2H	5	32	0.0	0.62	0.0	62 (–)
D	L^3H	10	31	0.0	0.68	0.0	78 (–)
E	L^4H	20	29	0.0	0.56	0.0	53 (–)
F	L^5H	25	18	0.0	0.33	0.0	41 (–)
A	Control	0	0	0	0	0	0
B	$(C_6H_5)_2PbCl_2$	1	11	0.0	0.91	0.0	86 (–)
C	$(C_6H_5)_2PbCl_2$	5	15	0.0	0.63	0.0	79 (–)
D	$(C_6H_5)_2PbCl_2$	10	13	0.0	0.74	0.0	92 (–)
E	$Pb(CH_3COO)_2.3H_2O$	20	19	0.0	0.93	0.0	74 (–)
F	$Pb(CH_3COO)_2.3H_2O$	25	21	0.0	0.85	0.0	89 (–)

Dose.1, 5,10,20,25 ppm

PGIZ= Plant Growth Inhibition Zone (diameter in mm)

PGAI=Plant Growth Activity Index (diameter in mm)

CONCLUSION:

The synthesized derivatives were characterized and identified on the basis of physical and spectral data. These play a vital role as bioligands in biological systems. Nitrogen and sulfur/oxygen containing azomethine compounds are well recognized bioligands. It has been found that the activity of the metals is attained through the formation of complexes with different bioligands and the thermodynamic and kinetic properties of the complexes govern the mode of biological action. Antibacterial Activity, Antifungal Activity and Antitubercular Activity were found that metal complexes are much more active than the ligands. Plant growth Activity found negative because complex behave like toxin.

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Table -1: Analytical and Physical Properties of Ligands

Ligands	Colour & State	M.P.	Analysis (%)					M.Wt. Found (Calcd.)
			C Found (Calcd.)	H Found (Calcd.)	N Found (Calcd.)	O Found (Calcd.)	S Found (Calcd.)	
l-Acetyl-2-Naphtal Semicarbazone (L'H) ($C_{13}H_{13}N_3O_2$)	Yellow Solid	192	**64.19 (64.38)**	*5.39 (5.72)*	17.27 **(**17.91*)*	13.15 **(**13.78**)**	-	243.26 (245.71)
l-Acetyl-2-Naphtal Thio Semicarbazone (L^2H)	White Solid	**205**	**60.21 (61.19)**	5.05 (5.42)	16.20 **(**16.82*)*	6.17 **(**6.76**)**	12.36 (12.71**)**	**259.32 (260.91)**

($C_{13}H_{13}N_3OS$)								
2-Acetyl-l-Naphtal Semicarbazone (L^3H) ($C_{13}H_{13}N_3O_2$)	Pale Yellow Shiny	192	64.19 (64.38)	5.39 (5.72)	17.27 (17.91)	13.15 (13.56)	-	242.6 (243.71)
2-Acetyl-l-Naphtal Thio Semicarbazone (L^4H) ($C_{13}H_{13}N_3OS$)	Yellow Powder	205	60.21 (61.19)	5.5 (5.42)	16.20 (16.82)	6.17 (6.76)	12.36 (12.71)	259.32 (260.91)
2-Acetyl-5-methyl Furan Semi carbazone (L^5H) ($C_8H_{13}N_3O_2$)	Orange Shiny	182	53.03 (53.78)	6.12 (6.49)	23.19 (23.42)	17.66 (17.81)	-	181.19 (182.12)
2-Acetyl-5-methyl Furan Thio carbazone (L^6H) ($C_8H_{13}N_3OS$)	Yellowish Orange	190	48.71 (49.02)	5.62 (5.78)	21.30 (21.42)	8.11 (8.42)	16.26 (16.79)	197.26 (198.81)
2-Acetyl-4-methyi thiophene Semi carbazone (L^7H) ($C_7H_nN_3OS$)	Orange	178	48.71 (49.91)	5.62 (5.78)	21.30 (21.42)	8.11 (8.42)	16.26 (16.79)	197.26 (198.71)
2-Acetyl-4-methyl thiophene Thio Semi carbazone (L^8H) ($C_7HuN_3S_2$)	Yellow	191	45.04 (45.68)	5.20 (5.82)	19.70 (19.98)	-	21.02 (21.81)	213.33 (214.66)
2-Acetyl-naphtalene Semi carbazone (L^9H) ($Ci_3H_{23}N_30$)	White	188	69.29 (69.92)	5.77 (6.02)	18.49 (18.91)	7.04 (7.19)		227.26 (229.16)
2-Acetyl-naphtalene Thio Semi carbazone ($L^{10}H$) ($C_{13}H_{23}N_3S$)	White Crystal	196	65.58 (66.12)	6.48 (6.92)	18.21 (8.78)		18.21 (8.52)	243.33 (245.71)

Table 2: Analytical and physical data of Lead (IV) complexes of semicarbazones/ thiosemicarbazone

S. No	Reactants		Molar ratio	Product and Characteristics (Colour & state)	M.P. (°C)	Analysis: Found (Calcd.)%			Molecular Wt. Found (Calcd.)
	Lead Compound	Ligand				N Found (Calcd.)	S Found (Calcd.)	Pb Found (Calcd.)	
1.	$(C_6H_5)_2PbCl_2$	$C_{13}H_{13}N_3O_2$	1 :2	$C_{38}H_{34}N_6O_4Pb$ Yellow Solid	198	10.26 (10.56)	-	21.52 (21.96)	845.28 (819.52)
2.	$(C_6H_5)_2PbCl_2$	$C_{13}H_{13}N_3OS$	1 :2	$C_{38}H_{34}N_6O_2S_2Pb$ Dark Brown Solid	210	08.96 (09.02)	04.92 (05.13)	21.06 (21.29)	877.89 (879.01)
3.	$(C_6H_5)_2PbCl_2$	$C_{13}H_{13}N_3O_2$	1 :2	$C_{38}H_{34}N_6O_4Pb$ Light Yellow solid	196	10.26 (10.56)	-	21.52 (21.96)	845.28 (819.52)
4.	$(C_6H_5)_2PbCl$	$C_{13}H_{13}N_3O$	1 :2	$C_{38}H_{34}N_6O_2S_2P$	225	08.96	04.92	21.06	877.89

	$_2$	S		b Light Brown solid		(09.02)	(05.13)	(21.29)	(879.01)
5.	$(C_6H_5)_2PbCl_2$	$C_8H_{11}N_3O_2$	1 :2	$C_{28}H_{30}N_6O_4Pb$ Yellow Solid	191	11.96 (12.09)	-	21.28 (21.39)	753.66 (753.92)
6.	$(C_6H_5)_2PbCl_2$	$C_8H_{11}N_3OS$	1 :2	$C_{28}H_{30}N_6O_2S_2Pb$ Light Yellow Solid	206	09.87 (08.96)	04.18 (04.25)	19.92 (20.06)	721.12 (721.42)
7.	$(C_6H_5)_2PbCl_2$	$C_8H_{11}N_3OS$	1 :2	$C_{28}H_{30}N_6O_2S_2Pb$ Yellow Solid	184	09.87 (08.96)	04.18 (04.25)	19.92 (20.06)	721.12 (721.42)
8.	$(C_6H_5)_2PbCl_2$	$C_8H_{11}N_3S_2$	1 :2	$C_{28}H_{30}N_6S_4Pb$ Yellow Solid	201	09.21 (09.48)	05.16 (05.39)	21.06 (21.15)	785.81 (785.95)

Table 3: Antibacterial screening data of semicarbazones/thiosemicarbazones and their lead (IV) complexes

Compounds	**Diameter (mm) of Inhibition Zone after 24h (conc. in ppm)**							
	***Staphylococcus aureus* (+)**		***Proteus mirabilis* (-)**		***Escherichia coli* (-)**		***Bacillus thuringiensis* (+)**	
	500 ppm	**1000 ppm**	**500 ppm**	**1000 ppm**	**500 ppm**	**1000 ppm**	**500 ppm**	**1000 ppm**
$C_{13}H_{13}N_3O_2$	6	7	4	5	5	6	5	6
$C_{13}H_{13}N_3OS$	7	7	5	6	6	7	6	8
$C_{13}H_{13}N_3O_2$	8	9	7	8	8	9	7	9
$C_{38}H_{34}N_6O_4Pb$	10	11	8	9	9	10	9	11
$C_{38}H_{34}N_6O_2S_2Pb$	9	12	9	10	11	12	8	10
$C_{38}H_{34}N_6O_4Pb$	11	13	9	10	11	13	7	8
Streptomycin	15	17	12	15	17	18	14	16

Table 4: Antifungal screening data of semicarbazones/thiosemicarbazones and their lead (IV) complexes

Compounds	Percent Inhibition after 96h (conc in ppm)								
	Organism *Aspergillus flavus*			Organism Fusarium oxysporum			***Organism Aspergillus niger***		
	50 ppm	**100 ppm**	**200 ppm**	**50 ppm**	**100 ppm**	**200 ppm**	**50 ppm**	**100 ppm**	**200 ppm**
$C_{13}H_{13}N_3O_2$	40	60	72	42	63	79	49	63	81
$C_{13}H_{13}N_3OS$	49	58	71	50	68	78	52	69	84
$C_{13}H_{13}N_3O_2$	53	63	74	54	71	76	53	71	84
$C_{38}H_{34}N_6O_4Pb$	55	65	70	61	69	81	48	72	88
$C_{38}H_{34}N_6O_2S_2Pb$	44	66	78	48	72	76	65	82	86
$C_{38}H_{34}N_6O_4Pb$	45	71	79	54	73	79	61	84	87
Mycostatin	69	86	98	72	82	96	70	91	100

Table 5: IR spectral data (cm^{-1}) of ligands and their lead (IV) complexes

Compounds	***ν*(OH)**	***ν*(>C=N–)**	***ν*(Pb←N)**	***ν*(Pb←O)**
$C_{13}H_{13}N_3O_2$	3100-3400	1620	-	-
$C_{13}H_{13}N_3O\,S$	3100-3400	1610	-	-
$C_{13}H_{13}N_3O_2$	3100-3400	1615	-	-
$C_{38}H_{34}N_6O_4Pb$	-	1590	480	520
$C_{38}H_{34}N_6O_2S_2Pb$	-	1595	470	525

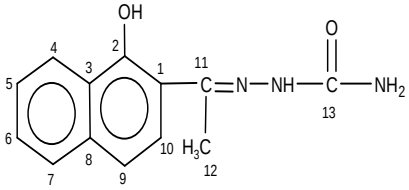
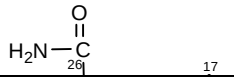
$C_{38}H_{34}N_6O_4Pb$	-	1590	485	518

Table 6: ^{1}H NMR spectral data (in δ, ppm) of ligands and their lead (IV) complexes

Compounds	–CH_3(s)	–OH(bs)	Aromatic protons (m)
$C_{13}H_{13}N_3O_2$	1.83	12.25	6.90-7.40
$C_{13}H_{13}N_3O$ S	1.84	12.18	6.95-7.80
$C_{13}H_{13}N_3O_2$	1.88	12.15	6.98-7.68
$C_{38}H_{34}N_6O_4Pb$	1.94	-	6.95-7.95
$C_{38}H_{34}N_6O_2S_2Pb$	1.95	-	6.80-7.50
$C_{38}H_{34}N_6O_4Pb$	1.99	-	6.95-7.80

s = strong; m = medium; bs = broad strong

Table 7: ^{13}C NMR spectral data of semicarbazones and its corresponding lead (IV) complex

Compounds	Chemical shift value in δ ppm										
	C-1	C-2	C-3	C-4	C-5	C-6	C-7	C-8	C-9	C-10	C-11
	C-12	C-13	C-14	C-15	C-16	C-17	C-18	C-19	C-20	C-21	C-22
	C-23	C-24	C-25	C-26	C-27	C-28	C-29	C-30	C-31	C-32	C-33
	C-34	C-35	C-36	C-37	C-38						
	112.1	126.9	126.3	126.4	123.5	127.7	129.0	132.1	127.7	132.7	155.4
	129.8	161.6									
	124.1	112.6	126.3	126.4	123.5	127.7	129.0	132.1	117.7	132.7	126.3

	129.8	161.2	112.6	121.9	126.3	126.4	123.5	127.7	129.0	132.1	117.7
	132.7	126.3	129.8	161.2	129.0	130.4	128.9	129.6	129.9	120.7	129.4
	129.6	129.9	120.7	129.4							

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