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SYNTHESIS AND CHARACTERIZATION OF NEW COMPLEXES *O, O'*-BIS (α-NAPHTHYL, β-NAPHTHYL AND 2, 3, 5- TRIMETHYLPHENYL) DITHIOPHOSPHATE OF ZINC (II), CADMIUM (II) AND MERCURY (II)

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ABSTRACT
O,O'-BIS(α-Naphthyl, β-Naphthyl and 2,3,5- trimethylphenyl) Dithiophosphate complexes of Zinc(II), Cadmium (II) and Mercury(II) coressponding to$[(ArO)_2PS_2]_2M$,Where ArO =(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, OR $(CH_3)_3C_6H_2O)_2PS_2]_2$ M and M= zinc(II), cadmium (II) and mercury(II), have been synthesized by the reaction of MCl_2 in equimolar ratio 2:1 with sodium salts of O,O' α-naphthyl, β-naphthyl and 2,3,5-trimethylphenyl dithiophosphate in refluxing toulene results in the formation of complexes of the type [(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, OR $(CH_3)_3C_6H_2O)_2PS_2]_2$M. These complexes have been characterized by elemental analyses, C,H,S, Zn, Cd, Hg.and have been further characterized by some spectroscopical data IR and NMR (^1H,C^{13} and ^{31}P) and found the monomeric nature of these derivatives and metal atom was four coordinate bonded to two bidentate dithiophosphate ligands leading to a square planar geometry around the metal atom.

KEYWORDS: α-naphthyl, β-naphthyl and 2, 3, 5- trimethylphenyl, Dithiophosphates *etc*.

INTRODUCTION

Acylic dithiophosphate, $(RO)_2PS_2X$, and cyclic dithiophosphate ligands, $OGPS_2X$, (R = Me, Et, Pr^n, Pr^i or Bu^i, G = -CH_2 CM_2CH_2-, - $CH_2CEt_2CH_2$ -$CH_2CEt_2CH_2$-, CMe_2CH_2CHMe- or -CMe_2CMe_2-; X = H, Na or NH_4) occupy a unique position as versatile chelating ligands[1-3]. These ligands show monodentate[4-6], bidentate [7-13], and also bridging mode of bonding with metals[14]. Various dithiophosphato derivatives find extensive applications in industries such as extreme pressure oil additives[15], agriculture[16], hydraulic fluid additives [17], heat stabilizers for polymers [18], analytical[19], extraction[20], and also found biological activities[21] . A literature survey revealed that a substantial amount of work has been done with the dialkyl- and alkylenedithiophosphate ligands, but scanty information is available on the derivatives of (*O,O'*-bis(α-Naphthyl, β-Naphthyl and 2,3,5- Trimethylphenyl) ligands

[{(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, OR $(CH_3)_3C_6H_2O)_2PS_2Na$] [22-25], However, some metal complexes with the bis (α-naphthyl, β-naphthyl and 2,3,5- trimethylphenyl) dithiophosphate ligands have been synthesized and characterized [26-29]. Some utilizations of the derivatives of bis (α-naphthyl, β-naphthyl and 2, 3, 5- trimethylphenyl) dithiophosphate in agriculture[30], industry[31-33]. we reported the synthesis and characterization of new complexes *O,O'*-bis(α-naphthyl, β-naphthyl and 2,3,5-trimethylphenyl) dithiophosphate complexes of Zinc(II), Cadmium (II) and Mercury(II) by the reaction of MCl_2 in equimolar ratio 2:1 with sodium salts of O,O' α-naphthyl, β-naphthyl and 2,3,5-trimethylphenyl dithiophosphate in refluxing toulene results in the formation of complexes.

MATERIALS AND METHODS

Moisture was carefully excluded throughout the experimental work using standard schlenk's technique. Solvents were dried by standard methods prior to their use. Sulfur was estimated as $BaSO_4$ (Messenger's method) and, Zn, Cd, Hg were estimated gravimetrically. α-naphthyl, β-naphthyl and 2,3,5- trimethylphenyl dithiophosphates ligands were prepared by literature methods[32-33]. Molecular weights were determined cryscopically in freezing benzene. IR spectra were recorded in KBr mulls in the range 4000-200 cm^{-1} on a Perkin Elmer= 377 spectrophotometer. The ^1H, ^{13}C and ^{31}P NMR spectra were recorded on a Bruker DRX 300 (120 MHz) spectrometer using TMS as the internal reference for ^1H NMR and 85% H_3PO_4 as an external reference for ^{31}P NMR.

Preparation of the compounds

These complexes were prepared by the methods as reported in the literature[34]-[37].

(1) Synthesis of complexes of type [(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, OR $(CH_3)_3C_6H_2O)_2PS_2]_2$ Zn.

For the synthesis of [(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, OR $(CH_3)_3C_6H_2O)_2PS_2]_2$ Zn. (1-3), (~ 40 ml) toluene solution of $ZnCl_2$(0.34 g or 2.5 mmol) was taken in a 100 ml round bottom flask. To this solution, was added (~ 40ml) toluene suspension of sodium salt of *O,O'*-bis (α-naphthyl, β-naphthyl and 2,3,5-trimethylphenyl)dithiophosphates ligand, (α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, or $(CH_3)_3C_6H_2O)_2PS_2Na$ (2.02 g or 5.0 mmol) in a drop wise manner, through a dropping funnel. During the course of the addition of $ZnCl_2$ solution and precipitation of sodium chloride took place. The contents were further refluxed for 4 hours. Then the contents were brought to room temperature and sodium chloride formed during the course of the reaction was filtered off through Sintered G4 glass disc. Finally the excess of solvent was removed from the filtrate under reduced pressure, which

yielded [(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, OR $(CH_3)_3C_6H_2O)_2PS_2]_2$ Zn.(1-3), as colourless sticky solid in 75 % yield. The synthetic and analytical details are given in Table 1. Similar methodology was applied for the synthesis of complexes (4-9).

TABLE 1. Synthetic and analytical data of ZINC(II), CADMIUM (II) and MERCURY(II): *O,O'*- α-Naphthyl, β-Naphthyl and 2,3,5-Trimethylphenyl) dithiophosphates.[M= Zn^{2+},Cd^{2+},Hg^{2+}].

SN	. Ligand (gm) mmol	Metal salt (gm) mmol	Molar ratio	Ref. Time (hrs.)	Product/ (Color/M.pt)	Analyses Calc. (Found)	
	[(α-$C_{10}H_7O$-)$_2PS_2Na$]	$ZnCl_2$	2:1	3	[(α-$C_{10}H_7O$-)$_2PS_2]_2Zn$	M	S
1	2.02gm,5.0mmol	0.34gm,2.5mmol			828.27	7.8 (7.6)	15.4 15.3)
2	[(β-$C_{10}H_7O$-)$_2PS_2Na$] 2.02gm,5.0mmol	$ZnCl_2$ 0.34gm,2.5mmol	2:1	3	[(β-$C_{10}H_7O$-)$_2PS_2]_2Zn$ 828.27	7.8 (7.7)	15.4 (15.2)
3	[$((CH_3)_3C_6H_2O$-)$_2PS_2Na$] 1.94gm,5.0mmol	$ZnCl_2$ 0.34gm,2.5mmol	2:1	3	[$((CH_3)_3C_6H_2O$-)$_2PS_2]_2$ Zn 796.35	8.2 (8.1)	16.1 (16.0)
4	[(α-$C_{10}H_7O$-)$_2PS_2Na$] 2.02gm,5.0mmol	$CdCl_2$. 0.45gm,2.5mmol	2:1	3	[(α-$C_{10}H_7O$-)$_2PS_2]_2Cd$ 875.28	12.8 (2.6)	14.6 (14.4)
5	[(β-$C_{10}H_7O$-)$_2PS_2Na$] 2.02gm,5.0mmol	$CdCl_2$. 0.45gm,2.5mmol	2:1	3	[(β-$C_{10}H_7O$-)$_2PS_2]_2Cd$ 875.28	12.8 (12.6)	14.6 (14.4)
6	[$((CH_3)_3C_6H_2O$-)$_2PS_2Na$] 1.94gm,5.0mmol	$CdCl_2$. 0.45gm,2.5mmol	2:1	3	[$((CH_3)_3C_6H_2O$-)$_2PS_2]_2Cd$ 843.37	13.3 (13.0)	15.2 (15.1)
7	[(α-$C_{10}H_7O$-)$_2PS_2Na$] 2.02gm,5.0mmol	$HgCl_2$ 0.34gm,2.5mmol	2:1	3	[(α-$C_{10}H_7O$-)$_2PS_2]_2Hg$ 963.47	20.8 (20.6)	13.3 (13.2)
8	[(β-$C_{10}H_7O$-)$_2PS_2Na$] 2.02gm,5.0mmol	$HgCl_2$ 0.34gm,2.5mmol	2:1	3	[(β-$C_{10}H_7O$-)$_2PS_2]_2Hg$ 963.47	20.8 (20.7)	13.3 (13.2)
9	[$((CH_3)_3C_6H_2O$-)$_2PS_2Na$] 1.94gm,5.0mmol	$HgCl_2$ 0.34gm,2.5mmol	2:1	3	[$((CH_3)_3C_6H_2O$-)$_2PS_2]_2Hg$ 931.55	21.5 (21.1)	13.7 (13.5)

RESULTS AND DISCUSSION

literature survey[38-44] revealed that O,O'-*dialkyl dithiophosphates and* O,O'-*alkylene dithiophosphates of* zinc(II), cadmium (II) and mercury(II): *are well known. but no*. *O,O'*-bis(α-naphthyl, β-naphthyl and 2,3,5-trimethylphenyl) dithiophosphate of zinc(II), cadmium (II) and mercury(II) *has been so far reported. So, it was thought valueable to incorporate* zinc(II), cadmium (II) and mercury(II) as *O,O'*-bis(α-naphthyl,β-naphthyl and 2,3,5-trimethylphenyl) dithiophosphate complexes.These complexes have been prepared in good yield by the reaction of sodium salt of O,O' α-naphthyl, β-naphthyl and 2,3,5-trimethylphenyl) dithiophosphate ligands with $ZnCl_2$ in equimolar ratio 2:1 in refluxing chloroform results in the formation of complexes of the type [(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, OR $(CH_3)_3C_6H_2O)_2PS_2]_2$ Zn. These reactions are bit sluggish and proceed removal of sodium chloride, (NaCl) and the evaporation of solvent under reduced pressure results the formation of the compounds as colorless solid

$$ZnCl_2 + 2[(\alpha\text{-}C_{10}H_7O\text{-}, \beta\text{-}C_{10}H_7O\text{-}, \text{or } (CH_3)_3C_6H_2O)_2PS_2Na] \xrightarrow[-2NaCl]{\text{Chloroform}} [(\alpha\text{-}C_{10}H_7O\text{-},\beta\text{-}C_{10}H_7O\text{-},\text{or } (CH_3)_3C_6H_2O)_2PS_2]_2Zn \quad (1\text{-}3)$$

Similar methodology was applied for the synthesis of complexes (4-6) and(7-9). as colorless solid. (4-6)

$$CdCl_2 + [(\alpha\text{-}C_{10}H_7O\text{-}, \beta\text{-}C_{10}H_7O\text{-}, \text{or } (CH_3)_3C_6H_2O)_2PS_2Na] \xrightarrow[-2NaCl]{\text{Chloroform}} [(\alpha\text{-}C_{10}H_7O\text{-},\beta\text{-}C_{10}H_7O\text{-},\text{or } (CH_3)_3C_6H_2O)_2PS_2]_2Cd \quad (4\text{-}6)$$

as white sticky solid. (7-9).

$$HgCl_2 + 2[(\alpha\text{-}C_{10}H_7O\text{-}, \beta\text{-}C_{10}H_7O\text{-}, \text{or } (CH_3)_3C_6H_2O)_2PS_2Na] \xrightarrow[-2NaCl]{\text{Chloroform}} [(\alpha\text{-}C_{10}H_7O\text{-},\beta\text{-}C_{10}H_7O\text{-},\text{or } (CH_3)_3C_6H_2O)_2PS_2]_2Hg \quad (7\text{-}9)$$

These compounds were obtained as colorless sticky solids in 80-90% yield after the separation of sodium chloride. The elemental analyses, particularly,C,H,S, Zn,Cd,Hg of all the complexes were found reliable to the molecular formula of the complexes. The molecular weight determination of the few represented compounds indicated the monomeric nature of these derivatives. They have been further characterized by some other spectroscopical data like IR and (^{1}H NMR and ^{31}P, C^{13}). These compounds were obtained just about in quantitative yield, soluble in common organic solvents like benzene, toluene, chloroform, acetone and also in coordinating solvents like DMSO and DMF but they are sparingly soluble in the non-polar organic solvents *viz*. carbon tetrachloride. The

compounds obtained were sufficiently pure but for the sake of extra purity, these were further washed with dried *n*-hexane or diethyl ether.

Infrared spectra

IR spectra were recorded in the range 4000-200 cm^{-1} and the tentative assignments were made on the basis of relevant literature reports. The IR spectra of these complexes shows formation of a new sharp peak for ν Zn-S bond in the region 468-466 cm^{-1} for complexes (1-3). ,a peak for ν Cd-S bond in the region 466--462cm^{-1} for complexes (4-6).and a peak for ν Hg-S bond in the region 440-434cm^{-1} for complexes (7-9). But no such peak was present in either of the reactants. Peak for ν P-S was found in the region 468-434cm.$^{-1}$ Peaks for ν(P)-O-C, ν P-O-(C) and Aromatic ν (C-H) were observed in the region 1154-1152, 942-937 and 2950-2945 cm^{-1} for these complexes. The detailed data are given in Table 2.

TABLE 2. *IR spectral data of ZINC (II), CADMIUM (II) AND MERCURY (II): O,O'- α-Naphthyl, β-Naphthyl and 2,3,5-Trimethylphenyl) dithiophosphates. (in cm^{-1})*

M=[ZINC(II), CADMIUM (II) AND MERCURY(II):]

S.NO.	COMPOUND NO.	Aromatic(νC-H)	ν(P)-O-C	νP-O-(C)	νP—S	νM-S
1	$[(\alpha\text{-}C_{10}H_7O\text{-})_2PS_2]_2Zn$	2950	1154S	937s	671m	468
2	$[(\beta\text{-}C_{10}H_7O\text{-})_2PS_2]_2$ Zn	2950	1154S	937s	672 m	468
3	$[((CH_3)_3C_6H_2O\text{-})_2PS_2]_2Zn$	2945	1154S	937s	672 m	466
4	$[(\alpha\text{-}C_{10}H_7O\text{-})_2PS_2]_2Cd$	2947	1154S	937s	671 m	462
5	$[(\beta\text{-}C_{10}H_7O\text{-})_2PS_2]_2Cd$	2948	1152S	940s	669 m	464
6	$[((CH_3)_3C_6H_2O\text{-})_2PS_2]_2Cd$	2950	1152S	942s	670 m	466
7.	$[(\alpha\text{-}C_{10}H_7O\text{-})_2PS_2]_2Hg$	2950	1154S	942s	671 m	440
8.	$[(\beta\text{-}C_{10}H_7O\text{-})_2PS_2]_2$ Hg	2948	1152S	942s	672 m	442
9.	$[((CH_3)_3C_6H_2O\text{-})_2PS_2]_2$ Hg	2948	1152S	942s	672 m	434

^{1}H NMR spectra

The chemical shifts of $-CH_3$ (attached to benzene ring) for compounds (3, 6 and 9) of the ligand moiety was recorded at δ 2.12-2.14. A multiplet characteristic for ring protons were observed at δ 6.93-7.45 for compounds (1-9). The detailed ^{1}H NMR spectral data are given in Table 3.

TABLE 3. ^{1}H and ^{31}P NMR spectral data zinc (II), cadmium (II) and mercury (II):*O,O'*- α-Naphthyl, β-Naphthyl and 2,3,5-Trimethylphenyl) dithiophosphates.in $CDCl_3$ (δ ppm)

S.NO	Compound	^{1}H NMR	^{31}PNMR
1	$[(\alpha\text{-}C_{10}H_7O\text{-})_2PS_2]_2Zn$	6.94-7.44, m, 28H ($-C_{10}H_7$);	84.4s
2	$[(\beta\text{-}C_{10}H_7O\text{-})_2PS_2]_2$ Zn	6.95-7.45, m, 28H ($-C_{10}H_7$);	82.8s
3	$[((CH_3)_3C_6H_2O\text{-})_2PS_2]_2Zn$	2.32, s, 36H ($-CH_3$); 6.94-7.43, m, 8H ($-C_6H_2$);	83.5s
4	$[(\alpha\text{-}C_{10}H_7O\text{-})_2PS_2]_2Cd$	6.93-7.44, m, 28H ($-C_{10}H_7$);	85.5s
5	$[(\beta\text{-}C_{10}H_7O\text{-})_2PS_2]_2Cd$	6.97-7.42, m, 28H ($-C_{10}H_7$);	82.6s
6	$[((CH_3)_3C_6H_2O\text{-})_2PS_2]_2Cd$	2.33, s, 36H ($-CH_3$); 6.93-7.42, m, 8H ($-C_6H_2$-)	83.6s
7	$[(\alpha\text{-}C_{10}H_7O\text{-})_2PS_2]_2Hg$	6.96-7.41, m, 28H ($-C_{10}H_7$)	82.3s
8	$[(\beta\text{-}C_{10}H_7O\text{-})_2PS_2]_2$ Hg	6.97-7.43, m, 28H ($-C_{10}H_7$)	84.7s
9	$[((CH_3)_3C_6H_2O\text{-})_2PS_2]_2$ Hg	2.33, s, 36H ($-CH_3$); 6.93-7.45, m, 8H ($-C_6H_2$)	84.6s

Where s = singlet, d = doublet, t = triplet, q = quartet and m = multiplet

^{13}C NMR spectral

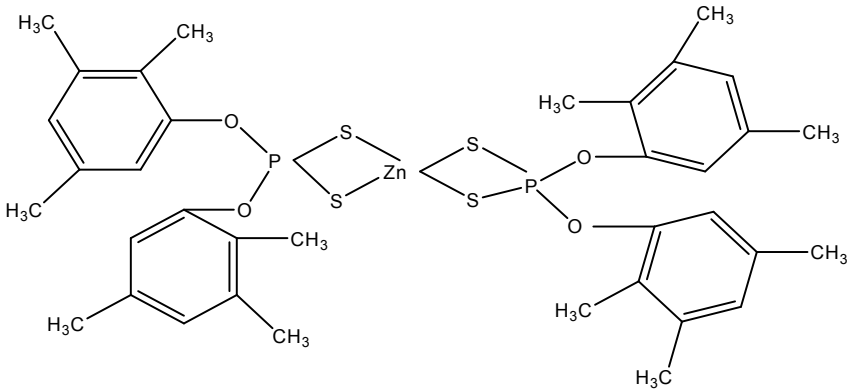
^{13}C NMR spectral analysis of above compounds, however, chemical shifts were observed for each carbon atom in these complexes has been shown in (Table 4.)

TABLE 4.^{13}C NMR spectral data of zinc (II), cadmium (II) and mercury (II) O, O'- α-Naphthyl, β-Naphthyl and 2,3,5-Trimethylphenyl) dithiophosphates in $CDCl_3$ (δ ppm).

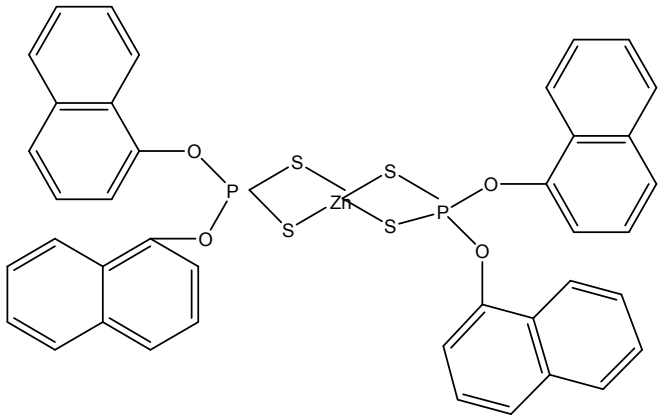
SNO	Compound	C-O	CH_3 (at C_2)	CH_3 (at C_3)	CH_3 (at C_5)	Aromatic carbons			
						C2&C7	C3&C6	C4&C5	C-9&C-10
1	$[(\alpha\text{-}C_{10}H_7O\text{-})_2PS_2]_2Zn$	151.1	---	---	----	126.2&124	121&121.5	115&120	135.1&133.0
2	$[(\beta\text{-}C_{10}H_7O\text{-})_2PS_2]_2$ Zn	151.3	---	---	---	128&125	121.2&122.4	116&120	136.5&133.0
3	$[((CH_3)_3C_6H_2O\text{-})_2PS_2]_2Zn$	151.4	16.5m	18.5m	20.5m	129&---	--&116.5	120.3&--	---
4	$[(\alpha\text{-}C_{10}H_7O\text{-})_2PS_2]_2Cd$	151.2	---	---	---	134&125	121.2&121.5	117&120	134.4&133.0
5	$[(\beta\text{-}C_{10}H_7O\text{-})_2PS_2]_2Cd$	151.1	---	---	---	137&124	121.1&121.5	118&120	134.8&133.0
6	$[((CH_3)_3C_6H_2O\text{-})_2PS_2]_2Cd$	151.3	19.5m	20.5m	23.5m	130&---	--&114.5	120.2&--	----
7	$[(\alpha\text{-}C_{10}H_7O\text{-})_2PS_2]_2Hg$	151.4	---	---	---	142&126	121.2&121.4	115.4&120	135.8&133.0
8	$[(\beta\text{-}C_{10}H_7O\text{-})_2PS_2]_2$ Hg	151.3	---	---	---	140&124	121.3&121.4	116&120	136.7&133.0
9	$[((CH_3)_3C_6H_2O\text{-})_2PS_2]_2$ Hg	151.2	17.5m	22.5m	24.2m	145.2&--	--&116.4	120.1&--	----

Structural features

On the basis of the above observations and the comparison of relevant data from literature, which revealed monomeric nature of these complexes and the metal atom being four coordinate bonded to two bidentate dithiophosphato ligands leading to a square planar geometry around the metal atom may be proposed for these complexes.



Proposed squre planar structure of the complexes of the typ $[(CH_3)_3C_6H_2O)_2PS_2]_2M$, M=[Zn,Cd,Hg]



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