

Synthesis and Characterisation of Oxovanadium(V) Derivatives of *O,O'*-Bis(α -naphthyl, β -naphthyl and 2, 3, 5-trimethylphenyl)dithiophosphates

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Abstract: Reactions of VOCl_3 with sodium salts of *O,O'*-bis(α -naphthyl, β -naphthyl or 2,3,5-trimethylphenyl) dithiophosphate ligands, [$\{\alpha\text{-C}_{10}\text{H}_7\text{O-}, \beta\text{-C}_{10}\text{H}_7\text{O-}, \text{or } (\text{CH}_3)_3\text{C}_6\text{H}_2\text{O}\}_2\text{PS}_2\text{Na}$], in 1:3 molar ratio in toluene yielded new seven coordinated vanadium derivatives of the type [$\{(\text{RO})_2\text{PS}_2\}_3\text{VO}$] (where $\text{R} = \alpha\text{-C}_{10}\text{H}_7\text{O-}, \beta\text{-C}_{10}\text{H}_7\text{O-}$ and $(\text{CH}_3)_3\text{C}_6\text{H}_2\text{O-}$). The reactions of [GVOVOCl] (where $\text{G} = \text{-CH}_2\text{CH}_2\text{-}$ or $\text{-CH}_2\text{CH}_2\text{CH}_2\text{-}$), with sodium salts of *O,O'*-bis(α -naphthyl, β -naphthyl or 2,3,5-trimethylphenyl) dithiophosphate ligands, [$\{\alpha\text{-C}_{10}\text{H}_7\text{O-}, \beta\text{-C}_{10}\text{H}_7\text{O-}$ and $(\text{CH}_3)_3\text{C}_6\text{H}_2\text{O}\}_2\text{PS}_2\text{Na}$], in 1:1 molar ratio in toluene were quite facile which afforded 5-coordinated soluble complexes corresponded to formula [$\text{GVOVOS}_2\text{P}(\text{OR})_2$]. These compounds have been characterized by elemental analysis (C, H, V and S), mass, IR and NMR (^1H , ^{13}C and ^{31}P) spectroscopic studies which indicates bidentate linkages of dithiophosphate moieties leading to pentagonal bipyramidal and square pyramidal geometry around the vanadium(V) atom.

Keywords: α -Naphthyl, β -Naphthyl, 2,3,5-Trimethylphenyl, Dithiophosphates, Vanadium dithiophosphates, Vanadiumoxytrichloride

Introduction

Dithiophosphate ligands occupies an unique position as versatile chelating ligands¹⁻³. These ligands are known to form a variety of complexes with transition and non-transition elements⁴⁻¹⁰. In general, a bi-dentate nature of these ligands have been established¹¹⁻¹³. However, they have also depicted other bonding modes *e.g.* monodentate¹⁴, anisobidentate¹⁵ and bridging¹⁶. Various dithiophosphate complexes finds broad applications in medicines¹⁷, as catalyst in asymmetric synthesis¹⁸, agriculture¹⁹, sol-gel processing²⁰, anti-wear additive to gear and motor oils²¹, as heat stabilizers for polymers²², extraction²³ and analytical processes²⁴.

Vanadium and its compounds also find applications in biological and industrial processes²⁵⁻²⁹. The $V^{IV}O$ and V^VO complexes act as catalysts for the conversion of sulfides to sulfoxides^{30,31}. Vanadate acts as an inhibitor³² towards a number of enzymes such as ATP. Phosphohydrolases, ribonucleases and phosphotyrosyl protein phosphates undergoes intracellular reduction to vanadyl (VO^{2+}) by NADH, ascorbate or glutathione. In this paper, we are reporting synthesis and characterization of some new complexes of vanadium(V) with *O,O*-bis(α -naphthyl, β -naphthyl and 2, 3, 5-trimethylphenyl) dithiophosphate ligands.

Experimental

All the experimental work was carried out strictly under anhydrous conditions, nitrogen atmosphere and by means of adapted Schlenk techniques. Triethylamine (Sd Fine Chem., b.p. 88.8 °C) was freshly distilled prior to use; chloroform and toluene were dried by refluxing over phosphorus pentoxide and sodium metal, respectively and kept under a nitrogen atmosphere. Naphth-1-ol, naphth-2-ol (Sd Fine Chem.), 2,3,5-trimethylphenol (Aldrich), phosphorus pentasulfide (Merck) were procured commercially and were used as such.

O,O'-Bis(α -naphthyl, β -naphthyl and 2, 3, 5-trimethylphenyl) dithiophosphate ligands [$(\alpha-C_{10}H_7O-$, $\beta-C_{10}H_7O-$ and $(CH_3)_3C_6H_2O)_2PS_2Na$] were prepared³³ by the reaction of substituted phenols with P_2S_5 in presence of Et_3N in 4:1:2 molar ratio in toluene under anhydrous conditions. $VOCl_3$ was prepared³⁴ by the reaction of V_2O_5 (Spectrochem) and $SOCl_2$ (spectrochem, b.p. 79 °C). 2-Chloro-2-oxo-1,3-dioxavanadacyclopentane and 2-chloro-2-oxo-1,3-dioxavanadacyclohexane[OGOVOC] where ($G=CH_2CH_2-$, $CH_2CH_2CH_2-$) were prepared by the reactions of ethylene (Thomas Baker) and propylene glycol (Thomas Baker) with $VOCl_3$ in presence of Et_3N as reported³⁵. Micro elemental analysis (C, H and S) was done on Vario EL III elemental analyzer in IIM Jammu while sulfur was estimated as $BaSO_4$ by messenger's method and vanadium was determined gravimetrically as vanadium pentaoxide. Infrared spectra were recorded in KBr on FTIR Perkin Elmer-377 spectrophotometer. The 1H and ^{13}C NMR spectra were measured on Jeol FX 90Q 90MHz using TMS as external reference. The ^{31}P NMR spectra were recorded by means of a Bruker DRX 300 (120 MHz) using 85% H_3PO_4 as external reference. The mass spectrophotometric analysis (EI) was carried out on ESQUIRE-300 (Bruker-Daltonics).

Synthesis of new oxo-vanadium(V) dithiophosphates

Synthesis of $\{(\alpha-C_{10}H_7O)_2PS_2\}_3VO$ (1)

For the synthesis of compound (1), 0.17 g or 1.0 mmol of $VOCl_3$ was dissolved in 40 mL of toluene in a 100 mL round bottom flask. To this solution 1.21 g (3.0 mmol) toluene suspension of sodium salt of $(\alpha-C_{10}H_7O)_2PS_2Na$ was added stoichiometrically in a drop wise manner with constant stirring and green color appeared in the flask, which further deepened and the reaction mixture was refluxed for 4 hours to ensure the completion of reaction. The reaction mixture was filtered through Sintered G4 glass disc to remove precipitates of NaCl and subsequently the excess of solvent was evaporated under reduced pressure. The green color solid was obtained in 90% yield. Similar procedure was applied for the synthesis of compounds (2-3). The synthetic and analytical details for all these complexes are being listed in Table 1.

Synthesis of compounds $[(\alpha-C_{10}H_7O)_2PS_2\overline{V(O)OCH_2CH_2O}]$ (4)

To a toluene suspension of $\alpha-C_{10}H_7O)_2PS_2Na$ (0.40 g, 1.0 mmol) in a round bottom flask (100 mL) toluene (40 mL) solution of $OCH_2CH_2\overline{OVOC}$ (0.16 g or 1.0 mmol) was added. As soon as the greenish color appeared in the flask with constant stirring and the reaction mixture was refluxed for 4 hours to ensure the completion of reaction. The reaction mixture

was filtered through sintered G4 glass disc to remove precipitates of NaCl and subsequently the excess of solvent was evaporated under reduced pressure. The green color solid was obtained in 90% yield. Similar procedure was applied for the synthesis of compound (5-9). The synthetic and analytical data are given in Table 1.

Table 1. Synthetic and analytical data of V(V)-*O,O'*- α -naphthyl, β -naphthyl and 2,3,5-trimethylphenyl) dithiophosphates

No.	Ligand	OGOVOCl/ VOCl ₃	Ref. Time	Product (M.Wt/Color/M.pt °C/ yield %)	Analyses Calcd. (Found)			
					C	H	V	S
1	[(α -C ₁₀ H ₇ O) ₂ PS ₂ Na] 1.21 g, 3.0 mmol	VOCl ₃ , 0.17 g, 1.0 mmol	4-6	[(α -C ₁₀ H ₇ O) ₂ PS ₂] ₃ V(O)] 1211.25/dark green/170/90%	59.5 (59.0)	3.5 (3.2)	4.2 (4.0)	15.8 (15.5)
2	[(β -C ₁₀ H ₇ O) ₂ PS ₂ Na] 1.21 g, 3.0 mmol	VOCl ₃ , 0.17 g, 1.0 mmol	4-6	[(β -C ₁₀ H ₇ O) ₂ PS ₂] ₃ V(O)] 1211.25/ dark green/178/90%	59.5 (59.1)	3.5 (3.4)	4.1 (4.0)	15.5 (15.3)
3	[{(CH ₃) ₃ C ₆ H ₂ O) ₂ PS ₂ Na] 1.16 g, 3.0 mmol	VOCl ₃ , 0.17 g, 1.0 mmol	4-6	[{(CH ₃) ₃ C ₆ H ₂ O) ₂ PS ₂] ₃ V(O)] 1163.38/dark green /195/93.5%	55.7 (55.3)	5.7 (5.4)	4.3 (4.0)	16.5 (16.3)
4	[(α -C ₁₀ H ₇ O) ₂ PS ₂ Na] 0.40 g, 1.0 mmol	OCH ₂ CH ₂ OV(O)Cl 0.16 g, 1.0 mmol	4-6	[(α -C ₁₀ H ₇ O) ₂ PS ₂ V(O) O(CH ₂) ₂ O] 508.43/ green/155/90%	51.9 (51.0)	3.5 (3.0)	10.0 (9.8)	12.6 (12.5)
5	[(β -C ₁₀ H ₇ O) ₂ PS ₂ Na] 0.40 g, 1.0 mmol	OCH ₂ CH ₂ OV(O)Cl 0.16 g, 1.0 mmol	4-6	[(β -C ₁₀ H ₇ O) ₂ PS ₂ V(O)O(CH ₂) ₂ O] 508.43/ green/159/90%	51.9 (51.1)	3.5 (3.1)	10.0 (9.9)	12.6 (12.5)
6	[{(CH ₃) ₃ C ₆ H ₂ O) ₂ PS ₂ Na] 0.38 g, 1.0 mmol	OCH ₂ CH ₂ OV(O)Cl 0.16 g, 1.0 mmol	4-6	[{(CH ₃) ₃ C ₆ H ₂ O) ₂ PS ₂ V(O)O(C H ₂) ₂ O] 492.47/ green/163/87.7%	48.7 (48.2)	5.3 (5.1)	10.3 (10.0)	13.0 (12.8)
7	[(α -C ₁₀ H ₇ O) ₂ PS ₂ Na] 0.40 g, 1.0 mmol	OCH ₂ CH ₂ CH ₂ OV (O)Cl 0.17 g, 1.0 mmol	4-6	[(α -C ₁₀ H ₇ O) ₂ PS ₂ V(O)O (CH ₂) ₃ O] 522.45 / green/160/90%	52.8 (52.2)	3.8 (3.2)	9.7 (9.5)	12.2 (12.0)
8	[(β -C ₁₀ H ₇ O) ₂ PS ₂ Na] 0.40 g, 1.0 mmol	OCH ₂ CH ₂ CH ₂ OV (O)Cl 0.17 g, 1.0 mmol	4-6	[(β -C ₁₀ H ₇ O) ₂ PS ₂ V(O)O (CH ₂) ₃ O] 522.45/ green/162/90%	52.8 (52.0)	3.8 (3.0)	9.6 (9.4)	12.2 (12.0)
9	[{(CH ₃) ₃ C ₆ H ₂ O) ₂ PS ₂ Na] 0.38 g, 1.0 mmol	OCH ₂ CH ₂ CH ₂ OV (O)Cl 0.17 g, 1.0 mmol	4-6	[{(CH ₃) ₃ C ₆ H ₂ O) ₂ PS ₂ V(O)O(C H ₂) ₃ O]506.5 / green/165/88%	49.8 (49.2)	5.5 (5.4)	10.0 (9.8)	12.6 (12.3)

Structural features

O,O'-Alkylenedithiophosphate ligands mostly bind with the transition metal centers in bidentate mode as illustrated by the literature survey^{9,10}. Though, it would not be appropriate to predict a precise structure for these complexes until single crystal x-ray analysis. However, in conjunction with the literature and observations based on elemental analysis, molecular weight determinations, IR and NMR (¹H, ¹³C and ³¹P) spectra, a pentagonal bipyramidal geometry akin²⁷ to [VO(O₂)(H₂O)₂(L-L')] ⁿ⁻ may be proposed for complexes VO[(RO)₂PS₂]₃ (Figure 1) whereas square pyramidal geometry, akin to exo{*N*-(2-oxidonaphthal)-His}²⁸ may be proposed for the complexes[OGOVOPS₂(OR)₂] Figure 2.

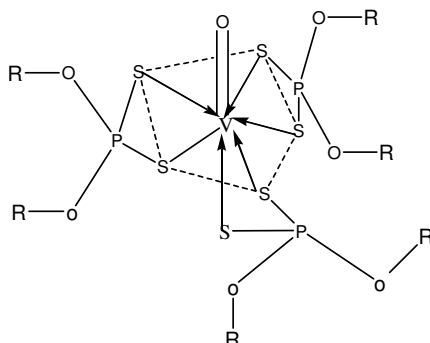


Figure 1. Proposed geometry for the complexes of type, $[\text{VO}\{\text{S}_2\text{P}(\text{OR})_2\}_3]$ Where $\text{R} = \alpha\text{-naphthyl}$ (1), $\beta\text{-naphthyl}$ (2) or 2,3,5-trimethylphenyl (3)

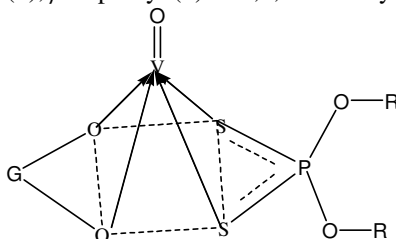
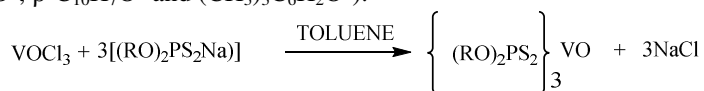


Figure 2. Proposed geometry for the complexes of the type, $[\text{OGOV}(\text{O})\text{S}_2\text{P}(\text{RO})_2]$ Where $\text{R} = \alpha\text{-naphthyl}$, $\beta\text{-naphthyl}$ or 2,3,5-trimethylphenyl and $\text{G} = -\text{CH}_2\text{CH}_2-$ for (4, 5, 6) and $\text{G} = -\text{CH}_2\text{CH}_2\text{CH}_2-$ for (7, 8 and 9)

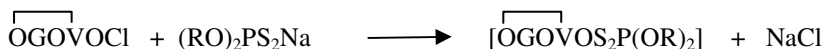
Results and Discussion

Vanadium has capacity to form complexes in higher coordination number as it is a d^0 system and has vacant 4s and 3d orbitals, which is also evident by the formation of the complexes like $\text{K}_4[\text{V}(\text{CN})_7] \cdot 2\text{H}_2\text{O}$, $\text{VO}(\text{NO}_3)_3 \cdot \text{CH}_3\text{CN}$, $[\text{VO}(\text{Et}_2\text{NCS}_2)_3]$ and $[\text{VO}(\text{O}_2)(\text{H}_2\text{O})_2(\text{L-L}')^n]$ where vanadium attained 7-coordinated and possesses pentagonal bipyramidal geometry²⁷. Here we have carried out the reaction of vanadium oxychloride (VOCl_3) with *O,O*-bis(α -naphthyl, β -naphthyl and 2, 3, 5-trimethylphenyl) dithiophosphate ligands, $[(\alpha\text{-C}_{10}\text{H}_7\text{O}-, \beta\text{-C}_{10}\text{H}_7\text{O}-, \text{or } (\text{CH}_3)_3\text{C}_6\text{H}_2\text{O})_2\text{PS}_2\text{Na}]$, in 1:3 molar ratio in toluene (Scheme 1). The reaction appeared quite facile as immediate appearance of green color took place, however the reaction mixture was refluxed for 6 hours to ensure completion of reaction. This reaction resulted in seven coordinated complexes of the type $[\{(\text{RO})_2\text{PS}_2\}_3\text{VO}]$. (Where $\text{R} = \alpha\text{-C}_{10}\text{H}_7\text{O}-, \beta\text{-C}_{10}\text{H}_7\text{O}-$ and $(\text{CH}_3)_3\text{C}_6\text{H}_2\text{O}-$).



Scheme 1. Reactions of VOCl_3 with $(\text{RO})_2\text{PS}_2\text{Na}$
(Where $\text{R} = \alpha\text{-naphthyl}$ (1), $\beta\text{-naphthyl}$ (2) or 2,3,5-trimethylphenyl (3))

Reactions of $[\text{OGOV}(\text{O})\text{Cl}]$, with sodium salts of *O,O'*-bis(α -naphthyl, β -naphthyl or 2,3,5-trimethylphenyl) dithiophosphate ligands, $(\alpha\text{-C}_{10}\text{H}_7\text{O}-, \beta\text{-C}_{10}\text{H}_7\text{O}-$ and $(\text{CH}_3)_3\text{C}_6\text{H}_2\text{O})_2\text{PS}_2\text{Na}$, in 1:1 molar ratio in toluene were quite facile which afforded 5-coordinated soluble complexes corresponded to formula $[\text{OGOVOS}_2\text{P}(\text{OR})_2]$.



Scheme 2. Reactions of $\overline{\text{OGO}}\text{VOCl}$ with $[(\alpha\text{-C}_{10}\text{H}_7\text{O}-, \beta\text{-C}_{10}\text{H}_7\text{O}- \text{ and } (\text{CH}_3)_3\text{C}_6\text{H}_2\text{O})_2\text{PS}_2\text{Na}]$ (Where $G = -\text{CH}_2\text{CH}_2-$ for (4), (5), (6) and $G = -\text{CH}_2\text{CH}_2\text{CH}_2-$ for (7), (8) and (9)).

These complexes of vanadium are obtained as dark green solids and are fairly soluble in common organic solvents like benzene, toluene, chloroform, ethanol *etc.*

Monomeric nature of these complexes (**1-9**) has been confirmed by their molecular weight determinations. Elemental analysis (H, C, S and V) also favored the composition to their general formula $[(\alpha\text{-C}_{10}\text{H}_7\text{O}-, \beta\text{-C}_{10}\text{H}_7\text{O}- \text{ and } (\text{CH}_3)_3\text{C}_6\text{H}_2\text{O})_2\text{PS}_2\text{VO}]$ or $(\text{OR})_2\text{PS}_2\text{V}(\text{O})\overline{\text{OGO}}$. Though the above complexes are moisture sensitive but they can be stored unchanged for a long time under dry and inert conditions.

IR spectra were recorded in the range $4000\text{-}200\text{ cm}^{-1}$ and the tentative assignments were done on the basis of relevant literature reports⁷⁻¹¹. The νCH stretching vibrations due to aromatic group were found in the region $2958\text{-}2943\text{ cm}^{-1}$. Bands assigned to $\nu(\text{P})\text{-O-C}$ and $\nu\text{P-O}(\text{C})$ stretching vibrations in both type of complexes were appeared in the region $1120\text{-}1060$ and $835\text{-}818\text{ cm}^{-1}$. Appearance of new bands in the region $490\text{-}450\text{ cm}^{-1}$ indicates the formation of V-S bond. The bands for $\nu\text{V=O}$ and $\nu\text{P-S}$ stretching vibrations were found in the region $970\text{-}920\text{ cm}^{-1}$ and $675\text{-}655\text{ cm}^{-1}$, respectively. The relevant IR spectral values are given in Table 2.

In the ^1H NMR spectra, the chemical shifts of $(\text{Ar})\text{CH}_3$ and ring protons were observed at δ 2.32-2.33 ppm and δ 7.35-7.46 ppm with usual splitting pattern. The chemical shifts for the $-\text{OCH}_2$ and CH_2 protons of the glycolate moiety of the compounds (4-9) were found in the region δ 3.51-3.54 and 4.0-4.4 respectively. The relevant ^1H NMR spectral values are given in Table 2.

The ^{31}P NMR spectra (proton-decoupled) have shown the chemical shift as singlet in each case in the down field region. The chemical shifts for the compounds (**1-9**) were found in the region δ 82.3-85.7 ppm, respectively. Occurrence of a singlet in each case is indicating the equivalent nature of phosphorus nucleus and bidentate mode of binding for the dithiophosphate ligand in these molecules¹⁰. The chemical shift values of ^{31}P NMR spectra are given in the Table 3.

Table 2. IR spectral data of oxovanadium(V) *O,O'*- α -naphthyl, β -naphthyl and 2,3,5-trimethylphenyl) dithiophosphates (in cm^{-1})

S.N°	Compound	Aromatic ($\nu\text{C-H}$)	ν ($\text{P})\text{-O-C}$	ν $\text{P-O}(\text{C})$	$\nu\text{V=O}$	$\nu\text{P-S}$	$\nu\text{V-O}$	$\nu\text{V-S}$
1	$[\{(\alpha\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2\}_3\text{V}(\text{O})]$	2958 b	1115s	829 s	970 m	655m	-	450m
2	$[\{(\beta\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2\}_3\text{V}(\text{O})]$	2957 b	1051s	820 s	975 m	659m	-	464m
3	$[\{((\text{CH}_3)_3\text{C}_6\text{H}_2\text{O})_2\text{PS}_2\}_3\text{V}(\text{O})]$	2944 b	1117s	830 s	950m	670 m	-	471m
4	$[(\alpha\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2\text{V}(\text{O})\text{O}(\text{CH}_2)_2\text{O}]$	2950 b	1060s	834 s	920 m	664m	720s	484m
5	$[(\beta\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2\text{V}(\text{O})\text{O}(\text{CH}_2)_2\text{O}]$	2948b	1058s	831s	978 m	669 m	725s	487m
6	$[\{((\text{CH}_3)_3\text{C}_6\text{H}_2\text{O})_2\text{PS}_2\text{V}(\text{O})\text{O}(\text{CH}_2)_2\text{O}\}]$	2943b	1120s	832 s	960 m	672 m	727s	486m
7	$[(\alpha\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2\text{V}(\text{O})(\text{CH}_2)_3\text{O}]$	2950b	1115s	834 s	920 m	664 m	724s	489m
8	$[(\beta\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2\text{V}(\text{O})(\text{CH}_2)_3\text{O}]$	2952b	1112s	832 s	954 m	659 m	728s	490m
9	$[\{((\text{CH}_3)_3\text{C}_6\text{H}_2\text{O})_2\text{PS}_2\text{V}(\text{O})(\text{CH}_2)_3\text{O}\}]$	2945b	1120s	835s	978m	670 m	730s	488m

Where s = Strong, b = broad, m = medium

Table 3. ^1H and ^{31}P NMR spectral data of oxovanadium(V) *O,O'*-bis α -naphthyl, β -naphthyl and 2,3,5-trimethylphenyldithiophosphates

S.No	Compound	^1H NMR	^{31}P NMR
1	$[(\alpha\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2]_3\text{V}(\text{O})]$	7.36-7.46, m, 42H ($-\text{C}_{10}\text{H}_7$)	82.3s
2	$[(\beta\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2]_3\text{V}(\text{O})]$	7.37-7.46, m, 42H ($-\text{C}_{10}\text{H}_7$)	84.8s
3	$[(\text{CH}_3)_3\text{C}_6\text{H}_2\text{O})_2\text{PS}_2]_3\text{V}(\text{O})]$	2.33, s, 54H ($-\text{CH}_3$); 7.35-7.45, m, 12H ($-\text{C}_6\text{H}_2$)	84.6s
4	$[(\alpha\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2\text{V}(\text{O})\text{O}(\text{CH}_2)_2\text{O}]$	7.35-7.46, m, 14H ($-\text{C}_{10}\text{H}_7$); 3.52, s, 4H ($-\text{OCH}_2-$)	84.5s
5	$[(\beta\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2\text{V}(\text{O})\text{O}(\text{CH}_2)_2\text{O}]$	7.35-7.45, m, 14H ($-\text{C}_{10}\text{H}_7$); 3.53, s, 4H ($-\text{OCH}_2-$)	82.6s
6	$[(\text{CH}_3)_3\text{C}_6\text{H}_2\text{O})_2\text{PS}_2\text{V}(\text{O})\text{O}(\text{CH}_2)_2\text{O}]$	2.32, s, 18H ($-\text{CH}_3$); 7.35-7.44, m, 4H ($-\text{C}_6\text{H}_2$); 3.54, s, 4H ($-\text{OCH}_2-$)	83.4s
7	$[(\alpha\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2\text{V}(\text{O})\text{O}(\text{CH}_2)_3\text{O}]$	7.35-7.46, m, 14H ($-\text{C}_{10}\text{H}_7$); 4.0-4.4, m, 2H ($-\text{CH}_2-$); 3.53, s, 4H ($-\text{OCH}_2-$)	85.7s
8	$[(\beta\text{-C}_{10}\text{H}_7\text{O})_2\text{PS}_2\text{V}(\text{O})\text{O}(\text{CH}_2)_3\text{O}]$	7.36-7.46, m, 14H ($-\text{C}_{10}\text{H}_7$); 4.1-4.4, m, 2H ($-\text{CH}_2-$); 3.54, s, 4H ($-\text{OCH}_2-$)	82.3s
9	$[(\text{CH}_3)_3\text{C}_6\text{H}_2\text{O})_2\text{PS}_2\text{V}(\text{O})\text{O}(\text{CH}_2)_3\text{O}]$	2.33, s, 18H ($-\text{CH}_3$); 7.35-7.46, m, 4H ($-\text{C}_6\text{H}_2$); 4.2-4.4, m, 2H ($-\text{CH}_2-$); 3.54, s, 4H ($-\text{OCH}_2-$)	83.4s

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

The ^{13}C NMR spectra of the compounds at ambient temperature do not show any appreciable change from parent dithiophosphate ligands and glycolate moiety. The chemical shifts for various carbon atoms are given in the Table 4.

Table 4. ^{13}C NMR spectral data of oxovanadium(V) *O,O'*-bis α -naphthyl, β -naphthyl and 2,3,5-trimethylphenyldithiophosphates in CDCl_3 (δ ppm)

Compd.	C-O	-CH ₃ (at C2)	-CH ₃ (at C ₃)	-CH ₃ (at C ₅)	Aromatic carbons										Glycolate OCH ₂ CH ₂	
					C2	C3	C4	C5	C6	C7	C8	C-9	C-10			
1	151.9	-	-	-	131.6	123.1	115.6	126.7	120.2	125.3	121.5	128.2	133.8	-	-	
2	151.8	-	-	-	134	123.2	116	126.9	120.6	125.8	121.7	128.4	133.4	-	-	
3	151.7	9.9m	18.3m	24.1m	125	123.3	121	126.8	120.9	-	-	-	-	-	-	
4	151.5	-	-	-	126	123.1	117	126.2	120.1	125.7	121.6	128.5	133.6	26.7s	-	
5	151.3	-	-	-	127	123.3	116	126.4	120.4	125.6	121.4	128.1	133.9	26.6s	-	
6	151.2	9.8m	18.4m	24.8m	128	123.2	118	126.1	120.3	-	-	-	-	26.8s	-	
7	151.4	-	-	-	133	123.5	119	126.3	120.5	125.5	121.2	128.3	133.7	26.7s	19.8	
8	151.6	-	-	-	132	123.6	120	126.5	120.8	125.4	121.4	128.6	133.5	26.9s	19.9	
9	151.1	9.6m	18.7m	24.3m	127	123.7	121.4	126.6	120.7	-	-	-	-	26.5s	19.7	

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