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Reactivity of Silicon tetrachloride and Trimethylsilyl chloride with *O, O'*-Bis (α-Naphthyl, β-Naphthyl or 2, 3, 5-trimethylphenyl) dithiophosphate ligands

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Abstract: Reactions of silicon tetrachloride, $SiCl_4$, or trimethylsilyl chloride, Me_3SiCl, with sodium salt of O,O'-Bis(α-Naphthyl, β-Naphthyl or 2,3,5-trimethylphenyl) dithiophosphate ligands, [{α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, or $(CH_3)_3C_6H_2O$}$_2PS_2Na$], in different molar ratio in chloroform under anhydrous conditions results in the formation of complexes corresponding to [{(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, or $Me_3C_6H_2O)_2PS_2$}$_nSiCl_{4-n}$] (n = 1 or 2) or [(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, or $Me_3C_6H_2O)_2PS_2Si\ Me_3$] in 84-97% yield. These colorless oily liquid complexes were characterized by elemental analysis, molecular weight determinations, IR and NMR (^1H, ^{13}C and ^{31}P) spectroscopic studies, which revealed monomeric nature of the complexes and monodentate mode of bonding of dithio moiety with silicon atom, leading to a tetrahedral geometry.

Key words: Bis(α-Naphthyl, β-Naphthyl or 2,3,5-trimethylphenyl) dithiophosphate ligands, Silicon Dithiophosphates*.*

I. INTRODUCTION

Acylic dithiophosphate, $(RO)_2PS_2X$, and cyclic dithiophosphate ligands, $OGOPS_2X$, (R = Me, Et, Pr^n, Pr^i or Bu^t, G = -$CH_2CMe_2CH_2$-, -$CH_2CEt_2CH_2$-, -CMe_2CH_2CHMe- or -CMe_2CMe_2- ; X = H, Na or NH_4) occupies an unique position as versatile chelating ligands [1-3]. These ligands show bidentate [4-10], monodentate [11, 13] and also bridging mode of bonding with metals and metalloids [14]. Various dithiophosphato derivatives finds extensive applications in agriculture[15], industries such as extreme pressure oil additives [16], heat stabilizers for polymers [17], hydraulic fluid additives [18], extraction[19], analytical [20] and also show biologiocal activities [21] Literature survey revealed that substantial amount of work has been done with the dialkyl and alkylene ligands with silicon and no information are available on the derivatives of *O,O'*-Bis(α-Naphthyl, β-Naphthyl or 2,3,5-trimethylphenyl) dithiophosphate ligands, [22-25]. Recently, some metal complexes with the ditolyl dithiophosphate ligands have been synthesized and characterized [26-29]. Utilities of some derivatives of ditolyl dithiophosphates in industries [28-30] and agriculture [31] have also been described. Keeping in view of the above interesting facets of dithiophosphate chemistry, we report herein the synthesis and characterization of some new complexes of silicon(IV)
with *O,O'*-Bis(α-Naphthyl, β-Naphthyl or 2,3,5-trimethylphenyl) dithiophosphate ligands, by using silicon tetrachloride, $SiCl_4$, or trimethylsilyl chloride, Me_3SiCl.

II. RESULTS AND DISCUSSION

Silicon the first among the group 14 elements shows its inclination for making more number of bonds than predicated by its valance state. This was perhaps the most compelling reason to distinguish it from its predecessor element, carbon. Consequently the chemistry of silicon becomes the thrust area of research for silicon chemists. The *O,O'*-dialkyl dithiophosphates and *O,O'*-alkylene dithiophosphates complexes of silicon(IV) are well known[11,13]. In these complexes the dithio ligands can bind with silicon(IV) in a monodentate or bidentate manner……..In the present research work we have reacted sodium salt of *O,O'*-Bis(α-Naphthyl, β-Naphthyl or 2,3,5-trimethylphenyl) dithiophosphate ligands, [{α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, or $(CH_3)_3C_6H_2O$}$_2PS_2Na$] with $SiCl_4$ or Me_3SiCl in different stoichiometry and the binding modes of dithio ligands are studied.

Reactions of sodium salt of *O,O'*-Bis(α-Naphthyl, β-Naphthyl or 2,3,5-trimethylphenyl) dithiophosphate ligands, [{α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, or $(CH_3)_3C_6H_2O$}$_2PS_2Na$] with $SiCl_4$ in 1:1 and 1:2 molar ratio in refluxing chloroform, are bit sluggish and proceeds with the elimination of sodium chloride. The reactions get completed in 3-4 hours and results in the formation of complexes of the $[\{(\alpha\text{-}C_{10}H_7O\text{-}, \beta\text{-}C_{10}H_7O\text{-}, \text{or } Me_3C_6H_2O)_2PS_2\}_nSiCl_{4-n}]$ (n = 1 or 2)

$$(RO)_2PS_2Na \quad + \quad SiCl_4 \quad \xrightarrow{\text{Chloroform}} \quad \underset{\mathbf{(1\text{-}3)}}{(RO)_2PS_2SiCl_3} \quad + \quad NaCl$$

$$2(RO)_2PS_2Na \quad + \quad SiCl_4 \quad \xrightarrow{\text{Chloroform}} \quad \underset{\mathbf{(4\text{-}6)}}{[\{(RO)_2PS_2\}_2SiCl_2]} \quad + \quad 2\,NaCl$$

(where R = $\alpha\text{-}C_{10}H_7O\text{-}$, $\beta\text{-}C_{10}H_7O\text{-}$, and $(CH_3)_3C_6H_2O\text{-}$).

A. *Scheme 1*

Reactions of sodium salt of *O,O'*-Bis(α-Naphthyl, β-Naphthyl or 2,3,5-trimethylphenyl) dithiophosphate ligands, $[\{\alpha\text{-}C_{10}H_7O\text{-}, \beta\text{-}C_{10}H_7O\text{-}, \text{or } (CH_3)_3C_6H_2O\}_2PS_2Na]$ with Me_3SiCl in equimolar ratio in refluxing chloroform results in the formation of complexes of the type $[(\alpha\text{-}C_{10}H_7O\text{-}, \beta\text{-}C_{10}H_7O\text{-}, \text{or } Me_3C_6H_2O)_2PS_2Si\,Me_3]$.These reactions are bit sluggish and proceed with the precipitation of Sodium chloride**.**

$$(RO)_2PS_2Na \quad + \; Me_3SiCl \quad \xrightarrow{\text{Chloroform}} \quad \underset{\mathbf{(7\text{-}9)}}{(RO)_2PS_2SiMe_3} \quad + \quad NaCl$$

B. *Scheme 2*

All these complexes were obtained as colorless oily liquids and are characterized by various physico-chemical techniques *viz.* elemental analyses, molecular weight determinations and spectral studies including IR, NMR (^{1}H , ^{13}C and ^{31}P). The micro elemental analyses, particularly C, H and S 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. These compounds are soluble in common organic solvents like benzene, toluene, chloroform, acetone, methylene chloride but slightly soluble in carbon tetrachloride. They appears to be susceptible to the moisture but can be kept unbothered for long time under dried and nitrogen atmosphere.

IR spectra were recorded in the range 4000-200 cm^{-1} and the tentative assignments were made on the basis of relevant literature reports[1-5, 32]. In all these complexes appearance of a new sharp band of medium intensity for ν Si-S bond in the region 536-509 cm^{-1}indicating formation of new bond between ligand and silicon atom. Appearance of a new bands in the region 704-698 cm^{-1} have been assigned for ν P=S vibrations. This value does not show any noticeable shift compared to parent dithio ligand, which indicates monodentate nature of all these ligands. The absorption band observed in the region 681-666Cm is due to ν P-S vibrations with a slight shift towards lower wave number compared to parent dithio moiety, indicating monodentate type of bonding in these complexes. The absorption bands of medium intenisity in the reagion504-498 may be assign to Si-Cl vibrations in complexes**1-6**. The νCH stretching vibrations due to aromatic group were found in the region 2958–2943 cm^{-1}. Bands assigned to ν(P)-O-C and νP-O-(C) stretching vibrations in both type of complexes were appeared in the region 1120-1060 and 835-818 cm^{-1} The relevant IR spectral values are given in Table I.

The ^{1}H NMR spectra was recorded in $CDCl_3$. The chemical shift for $–CH_3$ protons attached to aromatic ring was observed as singlet at δ2.30-2.33 ppm whereas the chemical shift for the aromatic ring protons appeared at δ7.0-7.9 ppm as a multiplet for all the complexes (**1-9**) in the ^{1}H NMR spectra. An integral of these shifts also supports the formation of 1:1 and 1:2 complexes between silicon(IV) chloride and dithiophosphate ligands[32]. The chemical shifts for $–CH_3$ group attached to silicon atom were found at δ0.10-0.11 ppm which favours complexation between ligand and trimethylsilyl substrate. The detailed ^{1}H NMR spectral data of all the complexes are given in Table II.

The^{31}P NMR spectra (proton-decoupled) have shown the chemical shift as doublet 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 doublet in each case is indicating the monodentate mode of binding for the dithiophosphate ligand in all these molecules[3,12,32].The chemical shift values of ^{31}P NMR spectra are given in the Table II.

The ^{13}C NMR spectra of the compounds at ambident temperature donot show any appreciable change from parent dithiophosphate ligands and $(CH_3)_3Si$- moity[32]. The chemical shifts for various carbon atoms are given in the Table III.

III. EXPERIMENTAL

All the experimental work was carried out strictly under anhydrous conditions, nitrogen atmosphere and by 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 [(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, and $(CH_3)_3C_6H_2O)_2PS_2Na$] were prepared[32] 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. Silicon Tetrachloride and Trimethylsilyl Chloride were obtained(Merck) commercially and used as such. Micro elemental analysis (C, H and S) was done on Vario EL III elemental analyzer in IIIM Jammu while sulfur was estimated as $BaSO_4$ by messenger's method. Infrared spectra were recorded in KBr on FTIR Perkin Elmer-377 spectrophotometer. The ^{1}H 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 Brucker DRX 300 (120 MHz) using 85% H_3PO_4 as external reference. The mass spectrophotometeric analysis (EI) was carried out on ESQUIRE-300 (Brucker-Daltonics).

A. Synthesis of complexes of type [{(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, or $Me_3C_6H_2O)_2PS_2\}_nSiCl_{4-n}$] *(Where n = 1 or 2) :*

For the synthesis of [(α-$C_{10}H_7O$-$)_2PS_2SiCl_3$] (**1**), (~ 40 ml) chloroform solution of $SiCl_4$ (0.31g or 0.18mmol) was taken in a 100 ml round bottom flask. To this solution, was added (~ 40ml) ice cold suspension of sodium salt of *O,O'*-Bis(α-Naphthyl)dithiophosphate ligands, [(α-$C_{10}H_7O$-$)_2PS_2Na$], (0.61 g or 0.18 mmol) in a drop wise manner, through a dropping funnel. After the complete addition of $SiCl_4$ solution, the reaction mixture was refluxed for 3 hours. Sodium chloride formed during the course of the reaction was filtered off through Sintered G4 glass disc and subsequently the excess of solvent was evaporated under reduced pressure. Final drying of the product in *vacuo* resulted [(α-$C_{10}H_7O)_2PS_2SiCl_3$] (**1**), as colorless oily liquid product in 84 % yield. Similar methodology was applied for the synthesis of complexes **2-6**. The synthetic and analytical details for these complexes are listed in **Table 1.**

B. Synthesis of complexes of complexes of type [(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, or $Me_3C_6H_2O)_2PS_2SiMe_3$] *(Where n = 1 or 2) :*

For the synthesis of [(α-$C_{10}H_7O)_2PS_2SiMe_3$] (**7**), (~ 40 ml) chloroform solution of Me_3SiCl (0.20 g or 0.19 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 (*ortho*- tolyl) dithiophosphates ligand [(α-$C_{10}H_7O$-$)_2PS_2Na$], (0.64 g or 0.19 mmol) in a drop wise manner, through a dropping funnel. After the complete addition of Me_3SiCl solution, the reaction mixture was refluxed for 3 hours. Sodium chloride formed during the course of the reaction was filtered off through Sintered G4 glass disc and subsequently the excess of solvent was evaporated under reduced pressure. Final drying of the product in *vacuo* resulted [(α-$C_{10}H_7O$-$)_2PS_2SiMe_3$] (**7**) as colorless oily product in 89 % yield. Similar methodology was applied for the synthesis of complexes **8-9**. The synthetic and analytical details for all the these complexes are being listed in Table 1.

C. Structural features

It is interesting to note that in ^{31}P spectra of complexes show a doublet in the range δ90.64-94.43 ppm. Which shows that ligand has behaved in monodentate manner and only one sulphur is attached to silicon atom. Monodentate behaviour of the ligands and tetrahedral structure around silicon atom is further supported by IR spectra which shows separate peaks for P-S and P=S. On the basis of literature survey[11,12,13] and above spectral studies, the following type of the structure can be proposed for these two type of the complexes:

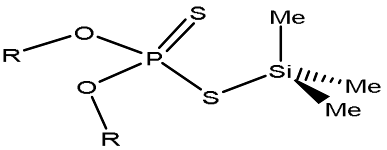

Figure 1: Proposed tetrahedral structure around silicon in [{(α-$C_{10}H_7O$-, β-$C_{10}H_7O$-, or $Me_3C_6H_2O)_2PS_2\}_nSiX_3$] (Where X = Cl or $-CH_3$)

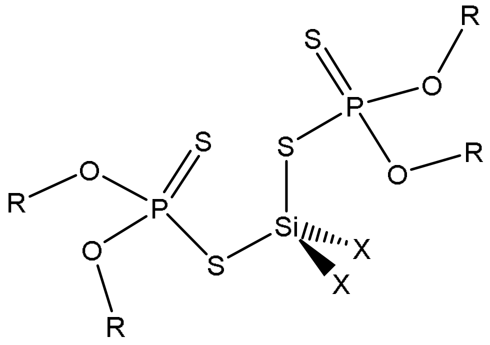

Figure 2: Proposed tetrahedral structure around silicon in $[\{(\alpha\text{-}C_{10}H_7O\text{-}, \beta\text{-}C_{10}H_7O\text{-}, \text{or } Me_3C_6H_2O)_2PS_2\}_2SiCl_2]$

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Table I. IR spectral data of Silicon(IV) O,O'-Bis(α-Naphthyl, β-Naphthyl and 2,3,5-Trimethylphenyl)dithiophosphates (in cm^{-1}).

S. NO.	Compound	Aromatic(νC-H)	ν(P)-O-C	νP-O-(C)	νP=S	νP—S	νSi-C	νSi-Cl	νSi-S
1	$[(\alpha\text{-}C_{10}H_7O)_2PS_2\,SiCl_3]$	2958 b	1115s	829 s	970 m	655m	----	650m	595m
2	$[(\beta\text{-}C_{10}H_7O)_2PS_2\,SiCl_3]$	2957 b	1051s	820 s	975 m	659m	----	654m	587m
3	$[\{(CH_3)_3C_6H_2O\}_2PS_2SiCl_3]$	2944 b	1117s	830 s	950m	670 m	----	651m	599m
4	$[\{(\alpha\text{-}C_{10}H_7O)_2PS_2\}_2SiCl_2]$	2950 b	1060s	834 s	920 m	664m	----	648m	584m
5	$[\{(\alpha\text{-}C_{10}H_7O)_2PS_2\}_2SiCl_2]$	2948b	1058s	831s	978 m	669 m	----	658	578m
6	$[\{(Me_3C_6H_2O)_2PS_2\}_2SiCl_2$	2943b	1120s	832 s	960 m	672 m	----	662	587m
7	$[(\alpha\text{-}C_{10}H_7O)_2PS_2Si(CH_3)_3]$	2950b	1115s	834 s	920 m	664 m	630m	---	581m
8	$[(\beta\text{-}C_{10}H_7O)_2PS_2\,Si(CH_3)_3]$	2952b	1112s	832 s	954 m	659 m	631m	---	598m
9	$[\{(CH_3)_3C_6H_2O\}_2PS_2\,Si(CH_3)_3]$	2945b	1120s	835s	978m	670 m	636m	---	581m

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

Table II.1H and 31P NMR spectral data of Silicon(IV) O,O'-Bis(α-Naphthyl, β-Naphthyl and 2,3,5-Trimethylphenyldithiophosphates(δ ppm).

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

S.NO	Compound	^{1}H NMR	^{31}PNMR
1	$[(\alpha\text{-}C_{10}H_7O)_2PS_2\ SiCl_3]$	7.36-7.50, m, 14H $(\text{-}C_{10}H_7)$	90.90,d
2	$[(\beta\text{-}C_{10}H_7O)_2PS_2\ SiCl_3]$	7.37-7.48, m, 14H $(\text{-}C_{10}H_7)$	91.64,d
3	$[\{(CH_3)_3C_6H_2O\}_2PS_2SiCl_3]$	2.34, s, 18H $(\text{-}CH_3)$; 7.35-7.49, m, 4H $(\text{-}C_6H_2)$	93.60,d
4	$[\{(\alpha\text{-}C_{10}H_7O)_2PS_2\}_2SiCl_2]$	7.35-7.49, m, 28H $(\text{-}C_{10}H_7)$	90.64,d
5	$[\{(\alpha\text{-}C_{10}H_7O)_2PS_2\}_2SiCl_2]$	7.35-7.45, m, 28H $(\text{-}C_{10}H_7)$	92.90,d
6	$[\{(Me_3C_6H_2O)_2PS_2\}_2SiCl_2$	2.32, s, 36H $(\text{-}CH_3)$; 7.35-7.48, m, 8H $(\text{-}C_6H_2)$	93.41,d
7	$[(\alpha\text{-}C_{10}H_7O)_2PS_2Si(CH_3)_3]$	7.35-7.46, m, 14H $(\text{-}C_{10}H_7)$; 0.98,t, 9H, $[\text{-}Si(CH_3)_3]$	92.74,d
8	$[(\beta\text{-}C_{10}H_7O)_2PS_2\ Si(CH_3)_3]$	7.35-7.46, m, 14H $(\text{-}C_{10}H_7)$; 0.96,t, 9H, $[\text{-}Si(CH_3)_3]$	92.32,d
9	$[\{(CH_3)_3C_6H_2O\}_2PS_2Si(CH_3)_3]$	2.33, s, 18H $(\text{-}CH_3)$; 7.35-7.46, m, 4H $(\text{-}C_6H_2\text{-})$; 0.96,t, 9H, $[\text{-}Si(CH_3)_3]$	94.43,d

Table III. ^{13}C NMR spectral data of Silicon(IV) O,O’-Bis (α-naphthyl, β-naphthyl and 2,3,5- trimethylphenyl)dithiophosphates (δ ppm).

S. NO	Compound	C-O	-CH_3 (at C2)	-CH_3 (at C3)	-CH_3 (at C5)	Aromatic carbons									-Si$(CH_3)_3$
						C2	C3	C4	C5	C6	C7	C8	C-9	C-10	
1	1	151.9	---	---	---	131.6	123.1	115.6	126.7	120.2	125.3	121.5	128.2	133.8	----
2	2	151.8	---	---	---	134	123.2	116	126.9	120.6	125.8	121.7	128.4	133.4	----
3	3	151.7	9.9m	18.3m	24.1m	125	123.3	121	126.8	120.9	----	-----	-----	----	----
4	4	151.5	---	---	----	126	123.1	117	126.2	120.1	125.7	121.6	128.5	133.6	----
5	5	151.3	---	---	---	127	123.3	116	126.4	120.4	125.6	121.4	128.1	133.9	----
6	6	151.2	9.8m	18.4m	24.8m	128	123.2	118	126.1	120.3	----	-----	----	----	----
7	7	151.4	---	---	---	133	123.5	119	126.3	120.5	125.5	121.2	128.3	133.7	31.7s
8	8	151.6	---	---	---	132	123.6	120	126.5	120.8	125.4	121.4	128.6	133.5	30.9s
9	9	151.1	9.6m	18.7m	24.3m	127	123.7	121.4	126.6	120.7	-----	----	----	----	27.9s

Table IV. Synthetic and analytical data of Silicon(1V) -*O,O'*- α-Naphthyl, β-Naphthyl and 2,3,5-Trimethylphenyl) dithiophosphates.

S. no.	Ligand	$SiCl_4/(CH_3)_3SiCl$	*Ref. Time*	*Product (M.Wt/Color/ yield %)*	Analyses Calc. (Found)			
					C	H	Cl	S
1	$[(\alpha\text{-}C_{10}H_7O)_2PS_2Na]$ 0.40gm, 1.0 mmol	$SiCl_4$, 0.17gm,1.0mmol	3-4	$[(\alpha\text{-}C_{10}H_7O)_2PS_2\,SiCl_3]$ 513.90/oily liquid/84	46.57 (46.51)	2.74 (2.65)	20.62 (20.58)	12.43 (12.38)
2	$[(\beta\text{-}C_{10}H_7O)_2PS_2Na]$ 0.40gm, 1.0 mmol	$SiCl_4$, 0.17gm,1.0mmol	3-4	$[(\beta\text{-}C_{10}H_7O)_2PS_2\,SiCl_3]$ 513.90/oily liquid/85	46.57 (46.48)	2.74 (2.66)	20.62 (20.55)	12.43 (12.38)
3	$[\{(CH_3)_3C_6H_2O\}_2PS_2Na]$ 0.38gm,1.0 mmol	$SiCl_{43}$, 0.17gm,1.0mmol	3-4	$[\{(CH_3)_3C_6H_2O\}_2PS_2SiCl_3]$ 497.96/oily liquid/84	43.25 (43.18)	4.44 (4.39)	21.27 (21.21)	12.83 (12.76)
4	$[(\alpha\text{-}C_{10}H_7O)_2PS_2Na$ 0.80gm, 2.0 mmol	$SiCl_4$ 0.17gm,1.0mmol	4-5	$[\{(\alpha\text{-}C_{10}H_7O)_2PS_2\}_2SiCl_2]$ 859.95/oily liquid/88	55.57 (55.51)	3.27 (3.24)	8.23 (8.10)	14.88 (14.73)
5	$[(\beta\text{-}C_{10}H_7O)_2PS_2Na]$ 0.80gm,2.0 mmol	$SiCl_4$ 0.17gm,1.0mmol	4-5	$[\{(\alpha\text{-}C_{10}H_7O)_2PS_2\}_2SiCl_2]$ 859.95/oily liquid/87	55.57 (55.50)	3.27 (3.24)	8.23 (8.07)	14.88 (14.78)
6	$[\{(CH_3)_3C_6H_2O\}_2PS_2Na]$ 0.76gm 2.0 mmol	$SiCl_4$, 0.17gm,1.0mmol	4-5	$[\{(Me_3C_6H_2O)_2PS_2\}_2SiCl_2$ 859.95/oily liquid/87	54.19 (54.09)	5.56 (5.50)	8.89 (8.68)	16.07 (15.99)
7	$[(\alpha\text{-}C_{10}H_7O)_2PS_2Na]$ 0.40gm,1.0 mmol	$(CH_3)_3SiCl$ 0.11gm,1.0mmol	3-4	$[(\alpha\text{-}C_{10}H_7O)_2PS_2Si(CH_3)_3]$ 454.06/oily liquid/93	60.77 (60.58)	5.10 (4.99)	…	14.10 (14.00)
8	$[(\beta\text{-}C_{10}H_7O)_2PS_2Na]$ 0.40gm,1.0 mmol	$(CH_3)_3SiCl$ 0.11gm,1.0mmol	3-4	$[(\beta\text{-}C_{10}H_7O)_2PS_2\,Si(CH_3)_3]$ 454.06/oily liquid/93	60.77 (60.60)	5.10 (5.03)	…	14.10 (14.01)
9	$[\{(CH_3)_3C_6H_2O\}_2PS_2Na]$ 0.38gm,1.0 mmol	$(CH_3)_3SiCl$ 0.11gm,1.0mmol	3-4	$[\{(CH_3)_3C_6H_2O\}_2PS_2\,Si(CH_3)_3]$ 438.13/oily liquid/95	57.50 (57.25)	7.12 (7.05)	…	14.62 (14.57)