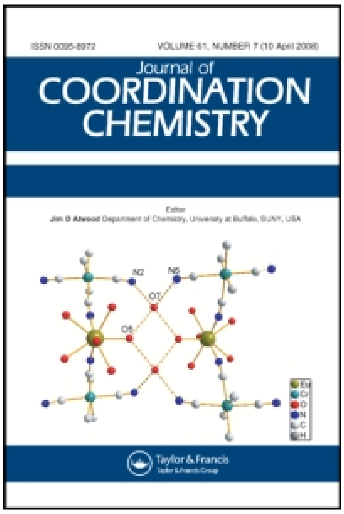

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Synthesis and Characterization of Some Unique Heterocyclic Derivatives Containing Aluminium(III) Atoms in 4- and 6-Co-Ordination States: Reaction of Bis(β-Diketonato) Aluminium (III)-DI-μ-Isopropoxo-DI-Isopropoxo Aluminium(III) with Triphenylsilanol and Diphenylsilanediol

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SYNTHESIS AND CHARACTERIZATION OF SOME UNIQUE HETEROCYCLIC DERIVATIVES CONTAINING ALUMINIUM(III) ATOMS IN 4- AND 6-CO-ORDINATION STATES: REACTION OF BIS(β-DIKETONATO) ALUMINIUM(III)-DI-μ-ISOPROPOXO-DI-ISOPROPOXO ALUMINIUM(III) WITH TRIPHENYLSILANOL AND DIPHENYLSILANEDIOL

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Reactions of bis (β-diketonato) aluminium(III)-di-μ-isopropoxo-di-isopropoxo-aluminium (III), $[CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OPr^i)_2]$, with triphenylsilanol, Ph_3SiOH, in 1:1 and 1:2 molar ratios and with diphenylsilanediol, $Ph_2Si(OH)_2$, in a 1:1 molar ratio, have resulted in the synthesis of $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$, $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$ and $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2\overline{Al(OSiPh_2O})]$, respectively. These are soluble in a variety of organic solvents (*e.g.*, benzene, chloroform and dimethylsulfoxide) and show dinuclear behaviour in chloroform. These derivatives have been characterized by elemental analyses, molecular weight measurements, IR and NMR (^1H, ^{13}C and ^{27}Al) studies.

Keywords: Bis(β-diketonato)aluminium(III)-di-μ-isopropoxo-isopropoxo-triphenylsilanolato aluminium(III); Bis(β-diketonato)aluminium(III)-di-μ-isopropoxo-bis(triphenylsilanolato) aluminium(III); Bis(β-diketonato)aluminium(III)-di-μ-isopropoxo-diphenylsilanediolato aluminium(III); Structure; NMR

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INTRODUCTION

The facile reactivity of metal-alkoxy bond(s) in metal alkoxides with a variety of reagents has been utilized for the synthesis of a number of interesting derivatives, *e.g.*, β-diketonates [1], carboxylates [2], silyloxides and even heterometallic alkoxide [3]. The feasibility [1–4] of carrying out such reactions in the desired molar ratio with continuous fractionation of alcohol (generally ethyl or isopropyl) liberated in the reaction(s) azeotropically with benzene has resulted even in further interesting mixed-alkoxy ligand derivatives with novel structural features.

Reactions of aluminium isopropoxide with β-diketones (β-dik) in different molar ratios yield derivatives of the types $[Al(OPr^i)_2(\beta\text{-dik})]_2$, $[Al(OPr^i)(\beta\text{-dik})_2]_2$ and $[Al(\beta\text{-dik})_3]$. Of these, the 1:1 derivative generally shows unsymmetrical behaviour as represented [5] by **(1)** $[(\beta\text{-dik})_2Al(\mu\text{-}OPr^i)_2Al(OPr^i)_2]$, instead of the symmetrical product $[(\beta\text{-dik})(OPr^i)Al(\mu\text{-}OPr^i)_2Al(OPr^i)(\beta\text{-dik})]$. Reactions of this interesting (1:1) derivative **(1)** with a variety of reagents *e.g.*, glycols [6], 2-mercaptoethanol, 2-2′ dithioethanol [7] and 8-hydroxyquinoline [8] have already been reported from these laboratories. In view of the interesting results obtained, we describe in this paper reactions of **(1)** with Ph_3SiOH and $Ph_2Si(OH)_2$.

EXPERIMENTAL

Aluminium isopropoxide was prepared as described in the literature [9]. Solvents were made anhydrous by reported methods. Acetylacetone and ethylacetoacetate were distilled before use; benzoyl acetone, triphenylsilanol and diphenylsilanediol were dried under reduced pressure before use. Aluminium was estimated gravimetrically as the oxinate. Silicon was estimated gravimetrically as silicon oxide [10]. Isopropanol was estimated by oxidimetric method [11]. The starting materials $[Al(CH_3COCHCOR)(OPr^i)_2]_2$ were prepared as reported earlier [6–8].

IR spectra were recorded as Nujol mulls using KBr and CsI plates in the range $4000-200\,cm^{-1}$ on a Nicolet Magna-550 spectrophotometer. ^1H NMR spectra were recorded on a Jeol FX 90 Q spectrometer in $CDCl_3$ using TMS ($\delta = 0$) as internal reference. ^{13}C NMR studies have been carried out in chloroform. ^{27}Al NMR spectra were recorded on a Varian 300 MHz at 78.16 MHz in toluene. Molecular weight measurements were carried out on a Knauer Vapour Pressure Osmometer in chloroform at 45°C.

In view of the similar nature of the reactions, only a typical case is described below and all synthetic and analytical data have been summarized in Tables I and II.

TABLE I Synthetic and analytical data and analytical data for $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$ and $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$ complexes

	Reactants (g)			Pr^i *OH (g)*	*Molecular*	*Analysis %* *found*		*Mol. wt.*
	(*a*)	(*b*)	*Molar*	*found*	*formula*	(*calcd.*)		*found*
	$[Al(CH_3COCHCOR(OPr^i)_2]_2$	Ph_3SiOH	*ratio*	(*calcd.*)	(*Yield %*)	*Al*	*Si*	(*calcd.*)
1	$R = -CH_3$ 4.67	2.65	1:1	0.57 (0.51)	$C_{37}H_{50}O_8SiAl_2$ (97)	7.63 (7.66)	3.95 (3.99)	710 (705)
2	$R = -CH_3$ 3.80	4.31	1:2	0.91 (0.94)	$C_{32}H_{58}O_8Si_2Al_2$ (96)	5.82 (5.86)	6.06 (6.10)	900 (921)
3	$R = -OC_2H_5$ 3.02	1.52	1:1	0.33 (0.33)	$C_{39}H_{54}O_{10}SiAl_2$ (98)	6.98 (7.05)	3.63 (3.67)	753 (765)
4	$R = -OC_2H_5$ 3.24	3.28	1:2	0.67 (0.71)	$C_{54}H_{62}O_{10}Si_2Al_2$ (95)	5.46 (5.50)	5.67 (5.73)	958 (981)
5	$R = -C_6H_5$ 2.55	1.20	1:1	0.23 (0.25)	$C_{47}H_{54}O_8SiAl_2$ (99)	6.51 (6.52)	3.60 (3.38)	802 (828)
6	$R = -C_6H_5$ 2.23	2.10	1:2	0.42 (0.43)	$C_{62}H_{64}O_9Si_2Al_2$ (100)	5.17 (5.11)	5.20 (5.36)	1042 (1044)

TABLE II Synthetic and analytical data for $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2\overline{Al(OSiPh_2O)}]$ complexes

	Reactants (g)		Pr^i *OH (g)*	*Molecular*	*Analysis %* *found*		*Mol. wt.*
	(a)	*(b)*	*found*	*formula*	*(calcd.)*		*found*
	$[(CH_3COCHCOR)\ (OPr^i)_2]_2$	$HOSiPh_2OH$	*(calcd.)*	*(Yield %)*	*Al*	*Si*	*(calcd.)*
1	R = $-CH_3$ 3.46	1.54	0.82 (0.85)	$C_{28}H_{38}O_8SiAl_2$ (97)	9.20 (9.23)	4.77 (4.80)	579 (585)
2	R = $-OC_2H_5$ 4.91	1.94	0.98 (1.08)	$C_{38}H_{42}O_8SiAl_2$ (98)	8.32 (8.31)	4.31 (4.36)	633 (645)
3	R = $-C_6H_5$ 3.67	1.31	0.70 (0.72)	$C_{30}H_{42}O_{10}SiAl_2$ (95)	7.59 (7.61)	3.89 (3.96)	712 (709)

Synthesis of $[(CH_3COCHCOCH_3)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$

To a benzene solution of $[Al(CH_3COCHCOCH_3)(OPr^i)_2]_2$ (4.67 g, 9.56 mmol) an appropriate amount of Ph_3SiOH (2.63 g, 9.60 mmol) in benzene ($\sim 70\,cm^3$) was refluxed on a fractionating column for $\sim$4 hours. The liberated isopropanol was fractionated out azeotropically with benzene. Progress as well as the completion of the reaction was checked by the estimation of isopropanol in the azeotrope by the oxidimetric method [11]. After stripping of the solvent under reduced pressure, the product was isolated as a white solid and purified by recrystallization from a mixture of dichloromethane and *n*-hexane.

RESULTS AND DISCUSSION

The reactions of $[Al(CH_3COCHCOR)(OPr^i)_2]_2$ with triphenylsilanol (PH_3SiOH) in 1:1 and 1:2 molar ratios in refluxing benzene yielded $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$ and $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$, (1) and (2), respectively. The reaction of $[Al(CH_3COCHCOR)(OPr^i)_2]_2$ with diphenylsilanediol ($HOSiPh_2OH$) in equimolar ratio in refluxing benzene yields, $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2\,\overline{Al(OSiPh_2O}\,)]$ (3).

$$[Al(CH_3COCHCOR)(OPr^i)_2]_2 + Ph_3SiOH \xrightarrow[\text{reflux}]{\text{Benzene}} [(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)] + Pr^iOH\uparrow \quad (1)$$

$$[Al(CH_3COCHCOR)(OPr^i)_2]_2 + 2Ph_3SiOH \xrightarrow[\text{reflux}]{\text{Benzene}} [(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2] + 2Pr^iOH\uparrow \quad (2)$$

$$[Al(CH_3COCHCOR)(OPr^i)_2]_2 + HOSiPh_2OH \xrightarrow[\text{reflux}]{\text{Benzene}} [(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2\,\overline{Al(OSiPh_2O}\,)] + 2Pr^iOH\uparrow \quad (3)$$

$$[R = -CH_3(\text{acac}), -OC_2H_5(\text{etacac}) \text{ and } -C_6H_5(\text{bzac})]$$

All the above reactions are quantitative and can be pushed to completion by fractionating out the liberated isopropanol azeotropically with benzene. Final removal of solvent under reduced pressure yielded white to pale yellow solids, soluble in benzene, chloroform and DMSO. These derivatives show dinuclear behaviour in chloroform and can be recrystallized from a mixture of dichloromethane and *n*-hexane.

IR Spectra

IR data for these newly synthesized mixed ligand heterocyclic derivatives along with triphenylsilanol and diphenylsilanediol are summarized in Table III. Broad stretching vibrations at 3250 cm^{-1} due to the –OH groups of triphenylsilanol and diphenylsilanediol disappear in the IR spectra of the derivatives, suggesting the formation of an Al–O bond by the deprotonation of triphenylsilanol and diphenylsilanediol. Characteristic Al–O–Si bands of terminal siloxide groups are observed in the region 1066–1055 cm^{-1} but in diphenylsilanediol the Al–O–Si stretching vibrations are observed in the region 1045–1040 cm^{-1}. A bidentate chelating mode for the β-diketonate moiety is supported [12] by the presence of strong bands in the regions 1610–1600 and 1532–1524 cm^{-1}, corresponding to νC–O and νC–C, respectively. The medium intensity band observed in the region 995–989 cm^{-1} is assigned to νC–O of the bridging isopropoxy group. Al–O–Al vibrations have been observed [13] in the region 746–740 cm^{-1}.

^{1}H NMR Spectra

Important signals in the ^{1}H NMR spectra of the derivatives are summarized in Table IV. A comparison of the spectra of free ligands (triphenylsilanol and diphenylsilanediol) with spectra of the corresponding derivatives show the absence of –OH signals, indicating deprotonation of the hydroxy group. Aromatic protons are observed as a multiplet in the region δ7.15–8.05 ppm. The methine protons of the terminal and bridging isopropoxy groups are observed at δ3.92–4.00 and 4.02–4.16 ppm, respectively while methyl protons of the terminal and bridging isopropoxy groups merge to give a doublet δ1.16–1.18 ppm in the ^{1}H NMR spectra of 1:1 derivatives, $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$. The methyl and methine protons of the isopropoxy groups in $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$ appear at δ1.12–1.16 and δ4.02–4.07 ppm, respectively. Methyl and the methine signals of β-diketonate moiety appear at δ1.92–1.97 and δ4.96–5.51 ppm, respectively, and methyl and the methylene protons of the ethylacetoacetate moiety ($-OCH_2CH_3$) are observed at δ1.25 ppm and δ3.93–4.00 ppm, respectively.

TABLE III IR data (cm^{-1}) for the $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$, $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$ and $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2\overline{Al(OSiPh_2O)}]$ complexes

		β-diketonate moiety		*Isopropoxy group*	*νAl–O–Si or*	
	Compound	*νC–O*	*νC–C*	*νC–O*	*νSi–O–H*	*νAl–O–Al*
1	Ph_3SiOH				3250 br	
2	$HOSiPh_2OH$				3226 br	
3	$[(acac)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$	1610 s	1532 s	990 m	1060 m	745 w
4	$[(acac)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$	1602 s	1532 s	992 m	1060 m	746 w
5	$[(etacac)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$	1600 s	1525 s	989 m	1055 m	740 w
6	$[(etacac)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$	1604 s	1524 s	995 m	1055 m	742 w
7	$[(bzac)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$	1600 s	1525 s	980 m	1040 m	750 w
8	$[(bzac)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$	1600 s	1525 s	985 m	1030 m	755 w
9	$[(acac)_2Al(\mu\text{-}OPr^i)_2\overline{Al(OSiPh_2O)}]$	1596 s	1528 s	988 m	1040 m	750 w
10	$[(etacac)_2Al(\mu\text{-}OPr^i)_2\overline{Al(OSiPh_2O)}]$	1601 s	1530 s	995 m	1041 m	750 w
11	$[(bzac)_2Al(\mu\text{-}OPr^i)_2\overline{Al(OSiPh_2O)}]$	1600 s	1525 s	989 m	1045 m	747 w

s = strong; br = broad; m = medium; w = weak.

TABLE IV ^{1}H NMR data (δppm) for the $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$, $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$ and $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2\overline{Al(OSiPh_2O)}]$ complexes

	β-diketonate moiety				*Isopropoxy group*		*Silanolate moiety*	
Compound	*$-CH_3$ att. to $-OCH_2-$*	*$-CH_3$*	*$-OCH_2-$*	*$-CH<$*	*$-CH_3$*	*$-OCH<$*	*$-C_6H_5$*	*$-OH$*
1							7.38 – 7.73, m(15H)	2.77, s(1H)
2							7.33 – 7.91, m(10H)	1.56, br(2H)
3		1.97, s(12H)		5.45, s(2H)	1.18, d(18H)	3.92 – 4.02, m(3H)	7.24 – 7.69, m(15H)	
4		1.97, s(12H)		5.51, s(2H)	1.12, d(12H)	4.02, m(2H)	7.15 – 7.69, m(30H)	
5	1.25, u(6H)	1.92, s(6H)	4.00 – 4(4H)	4.96, s(2H)	1.16, d(18H)	4.00 – 4.16, m(3H)	7.01 – 7.69, m(15H)	
6	1.25, u(6H)	1.92, s(6H)	3.93 – 4(4H)	4.96, s(2H)	1.16, d(12H)	4.07, m(2H)	7.06 – 7.64, m(30H)	
7		2.15, (6H)		6.46, s(2H)	1.26, d(18H)	3.70 – 4.27, m(3H)	7.67 – 8.05, m(10H)	
8		2.34, s(6H)		6.46, s(2H)	1.39, d(12H)	3.7 – 4.24, m(2H)	7.50 – 8.17, m(10H)	
9		2.03, s(12H)		5.57, s(2H)	1.21, d(12H)	4.05, m(2H)	7.22 – 7.33, m(10H)	
10	1.34, u(6H)	1.81, s(6H)	4.00,4(4H)	4.98, s(2H)	1.18, d(12H)	4.13, m(2H)	7.17 – 7.84, m(10H)	
11		2.17, s(6H)		6.30, s(2H)	1.16, d(12H)	4.00, m(2H)	7.15 – 8.05, m(20H)	

Compound numbers as in Table III; s = singlet; d = doublet; m = multiplet; br = broad; u = unresolved.

TABLE V ^{13}C and ^{27}Al NMR data (δppm) for the $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$, $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$ and $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2\overline{Al(OSiPh_2O)}]$ complexes

	^{13}C *Shifts*								
	β-diketonate moiety				*Isopropoxy group*				
	$-CH_3$*att. to* $-OCH_2-$	$-CH_3$	$-OCH_2-$	$-CH<$	$>C\overset{\cdots}{=}O$	$-CH_3$	$-OCH<$	*Aromatic Carbons*	^{27}*Al Shifts*
1								127.80, 129.97 134.95, 138.85	
2								126.55, 128.32 134.73, 142.12	
3		26.17		100.81	191.29	24.81,25.15	63.86, 64.04	127.04, 128.18 135.33, 138.90	2.67(o), 30.73(t)
4		26.66		101.04	191.30	25.03	64.05	127.15, 128.34 135.22, 138.96	0.80(o), 56.51(t)
5	14.20	25.95	60.58	85.12	174.12 187.72	24.60,25.25	63.69, 64.25	127.26, 128.67 134.95, 139.20	4.191(o), 56.57(t)
6	14.16	25.75	60.86	85.19	174.25 187.82	25.03	64.26	127.26, 128.67 134.89, 138.90	
7		27.52		97.50	182.90	25.20	64.24	126.55, 128.23 134.73, 142.49	
8		27.27		97.38	182.81 193.98	25.30	64.30	126.60, 128.32 134.78, 142.11	
9		26.28		100.98	191.34	24.81	66.04	126.55, 128.23 134.73, 142.49	
10	14.19	26.27	60.62	85.38	174.81 187.98	25.30	63.93	126.60, 128.32 134.78, 142.11	
11		27.47		97.41	182.35 193.24	25.19	64.31	126.55, 127.26 127.69, 131.37 131.37, 134.63 137.93, 142.13	

Compound numbers as in Table III.

^{13}C NMR Spectra

^{13}C NMR chemical shifts of the free ligands and their derivatives were recorded in chloroform at ambient temperature and are summarized in Table V. Both the number and positions of the various ^{13}C signals have been bound to be the same, as expected, with a slight shifting.

^{27}Al NMR Spectra

The ^{27}Al NMR spectrum of a representative derivative, $[(CH_3COCHCOCH_3)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$, exhibits signals at δ2.67 and δ30.73 ppm, indicating the presence of hexa- and tetracoordination around aluminium atoms [6–8] (Fig. 1). The ^{27}Al NMR spectrum of $[(CH_3COCHCOCH_3)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$ also exhibits signals corresponding to hexa- and tetracoordinated aluminium atoms at δ0.80 and δ56.51 ppm, respectively.

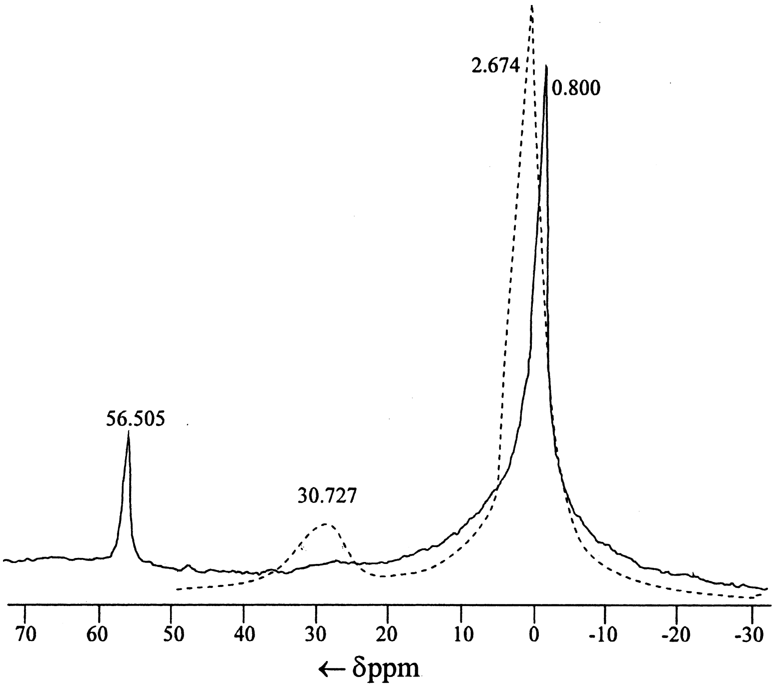

FIGURE 1 ^{27}Al spectra of $[(CH_3COCHCOCH_3)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(OPr^i)]$ (dotted line) and $[(CH_3COCHCOCH_3)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)_2]$ (full line).

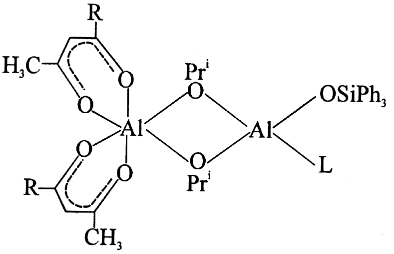

FIGURE 2 Structure of $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)(L)]$ (where, $L = OPr^i$ or $OSiPh_3$; $R = -CH_3$, $-OC_2H_5$ and $-C_6H_5$).

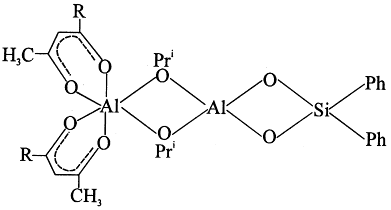

FIGURE 3 Structure of $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2\overline{Al(O\text{–}SiPh_2\text{–}O)}]$ (where $R = -CH_3$, $-OC_2H_5$ and $-C_6H_5$).

Although it is difficult to comment on the structures of these derivatives without single crystal X-ray analysis of at least one of the products, the above observations all indicate that these mixed ligand aluminium derivatives $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2Al(OSiPh_3)L]$ and $[(CH_3COCHCOR)_2Al(\mu\text{-}OPr^i)_2\overline{Al(OSiPh_2O)}]$ have structures containing both hexa and tetra-coordinated aluminium (III) atoms [6–8], as depicted in Figures 2 and 3, respectively.

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