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### Synthesis and Spectroscopic Properties of the First Series of Heteronuclear Derivatives Containing Organosilicon/Organotin (IV) and Oxovanadium (V) Moieties

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## Synthesis and Spectroscopic Properties of the First Series of Heteronuclear Derivatives Containing Organosilicon/Organotin (IV) and Oxovanadium (V) Moieties

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*Me<sub>3</sub>SiCl reacts with [VO(OGO)(GOH)] (1a) or [VO(L)(LH)] (1b) (where OGO = OCMe<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CMe<sub>2</sub>O and L = OC<sub>6</sub>H<sub>4</sub>CH=NCH<sub>2</sub>-CH<sub>2</sub>O) in the presence of Et<sub>3</sub>N as a proton acceptor (1:1:1 stoichiometric ratio) in benzene to form heteronuclear derivatives [VO(OGO)(OGOSiMe<sub>3</sub>)]<sub>2</sub> (2) and [VO(L)(LSiMe<sub>3</sub>)] (3), respectively. The reactions of Bu<sub>3</sub>Sn(OPr<sup>i</sup>) with a variety of oxovanadium(V) complexes (1a), (1b), and [VO(PhDEA)(PhDEAH)] (1c) (in a 1:1 stoichiometric ratio, where PhDEA = C<sub>6</sub>H<sub>6</sub>N(CH<sub>2</sub>CH<sub>2</sub>O)<sub>2</sub>) afford derivatives [VO(OGO)(OGOSnBu<sub>3</sub>)]<sub>2</sub> (4), [VO(L)(LSnBu<sub>3</sub>)] (5), and [VO(PhDEA)(PhDEASnBu<sub>3</sub>)] (6), respectively. Derivatives [(Ph<sub>3</sub>SiO)VO(OGO)]<sub>2</sub> (7) and [(Ph<sub>3</sub>SiO)VO(L)] (8) have been prepared by the equimolar reactions of Ph<sub>3</sub>SiOH with [VO(OGO)(OPr<sup>i</sup>)] (1d) and [VO(L)(OPr<sup>i</sup>)] (1e), respectively. All these new derivatives have been characterized by elemental analyses, molecular weight measurements, and spectroscopic [IR, NMR (<sup>1</sup>H, <sup>29</sup>Si, <sup>51</sup>V, and <sup>119</sup>Sn)] studies.*

**Keywords** Heteronuclear complexes; organosilicon complexes; organotin complexes; oxovanadium(V) complexes

There has been considerable development during the last four decades in the chemistry of organosilicon and organotin derivatives derived from glycols,<sup>1–3</sup> Schiff bases,<sup>4–10</sup> and diethanolamines<sup>11,12</sup> resulting in derivatives with interesting structural and chemical features. Furthermore, in spite of considerable progress in the heterometallic alkoxide

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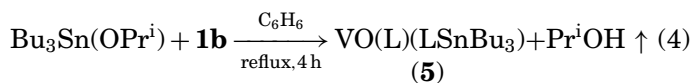
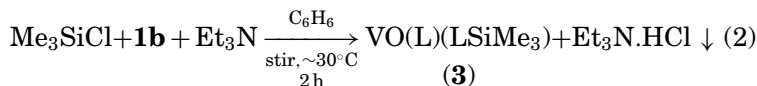
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chemistry of 3d transition elements,<sup>13</sup> there appears no reported example in the literature on derivatives incorporating both oxovanadium(V) and organosilicon/organotin(IV) groups. Recently, we have initiated an investigation to explore the ability of homometal chelate complexes having at least one non-deprotonated hydroxy group to bind different metal atoms, which led to the formation of many interesting heterometallic alkoxide coordination compounds, an account of which has been recently reviewed.<sup>2</sup> The studies in this direction now have been extended to incorporate organosilicon/organotin(IV) and oxovanadium(V) groups within the heterometallic species. We therefore report in this article, synthesis and spectroscopic properties of novel heteronuclear systems containing organosilicon(IV)/organotin(IV) and oxovanadium(V) groups.

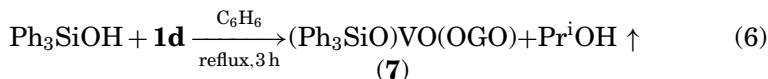
## RESULTS AND DISCUSSION

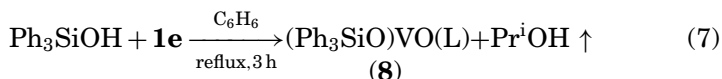
### Synthesis of Heteronuclear Derivatives

The heteronuclear derivatives (2)–(8) were prepared readily according to the reactions illustrated in Eqs. (1)–(7):



Reactions of  $\text{Ph}_3\text{SiOH}$  with (1d) and (1e) afford heteronuclear derivatives (7) and (8), respectively.



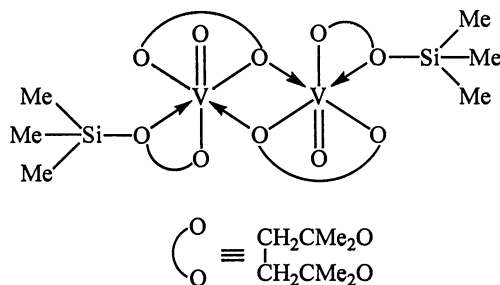


All these derivatives are colorless (**2**, **4**, and **7**) or colored (**3**, **5**, and **8** are red brown, **6** is black) solids, soluble in organic (e.g., benzene, toluene, dichloromethane, chloroform, and THF) solvents. Derivatives (**2**), (**4**), and (**7**) are dimeric (cryoscopically) in benzene, whereas (**3**), (**5**), (**6**), and (**8**) appear to be monomeric.

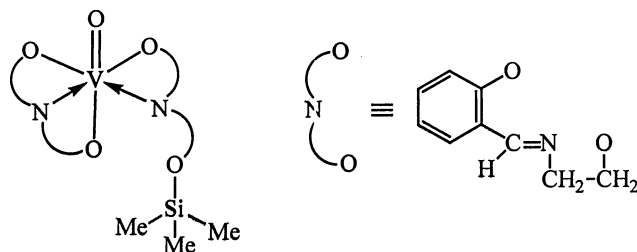
### Spectroscopic Studies

Infrared spectrum of (**2**) exhibits absorption bands ( $\text{cm}^{-1}$ ) at (1) 1250 due to  $\nu(\text{Si}-\text{CH}_3)$  symmetric deformation,<sup>14,15</sup> (2) 1080 for  $\nu(\text{C}-\text{O})$ ; (3) 967 for  $\nu(\text{V}=\text{O})$ , an absorption characteristic of six-coordinate<sup>16–19</sup> oxovanadium(V) complexes, (4) 930 due to  $\nu(\text{Si}-\text{O})$  stretching vibration; (5) 830 and 750 for asymmetric and symmetric  $\nu(\text{Si}-\text{C})$  vibrations of  $\text{Si}(\text{CH}_3)_3$  group,<sup>15,20</sup> respectively; and (6) 565 due to a  $\nu(\text{V}-\text{O})$  stretching vibration.<sup>21</sup> The  $^1\text{H}$  NMR spectrum of (**2**) shows signals at  $\delta$  0.10 (s, 9H,  $\text{OSiMe}_3$ ); 1.20 (s, 24H,  $\text{OCMe}_2$ ); and 1.55 (s, 8H,  $\text{CH}_2$ ). The  $^{29}\text{Si}$  NMR signal for (**2**) appears at  $\delta$  -8.77 ppm, which is consistent with four-coordinate<sup>22–24</sup> silicon complexes (Figure 1). Appearance of  $^{51}\text{V}$  NMR signal at  $\delta$  -679 ppm provides an evidence<sup>25,26</sup> for the presence of vanadium in six-coordinate environment (Figure 1).

Infrared absorption bands ( $\text{cm}^{-1}$ ) for the derivative (**3**) appear at (1) 1630 due to  $\nu(\text{C}=\text{N})$  stretching vibration, with a lowering of  $\sim 10$   $\text{cm}^{-1}$  wave numbers compared to that found in the free ligand, which is interpretable in terms of coordination of azomethine nitrogen<sup>29,30</sup> to vanadium; (2) 1266 for phenolic<sup>31</sup>  $\nu(\text{C}-\text{O})$ ; (3) 1240 due to  $\nu(\text{Si}-\text{CH}_3)$  symmetric deformation;<sup>14,15</sup> (4) 1042 for alcoholic<sup>31</sup>  $\nu(\text{C}-\text{O})$ ; (5) 958 for  $\nu(\text{V}=\text{O})$ ;<sup>16–19</sup> (6) 917 for  $\nu(\text{Si}-\text{O})$ , and (7) 833 and 733 for asymmetric



**FIGURE 1** Proposed structure for the derivative (**2**).

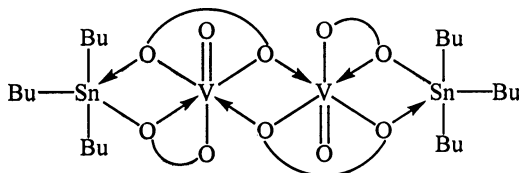


**FIGURE 2** Plausible structure for (3).

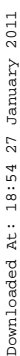
and symmetric  $\nu(\text{Si}-\text{C})$  stretching vibrations,<sup>15,20</sup> respectively. The  $^1\text{H}$  NMR spectrum of (3) shows signals at  $\delta$  0.08 (s, 9H,  $\text{OSiMe}_3$ ); 3.72 (m, 4H,  $\text{NCH}_2$ ); 3.95 (m, 4H,  $\text{CH}_2\text{O}$ ); 6.90–7.36 (m, 8H, aromatic-H); and 8.46 (s, 2H,  $\text{HC}=\text{N}$ ). The  $^{29}\text{Si}$  and  $^{51}\text{V}$  NMR signals at  $\delta$  –9.87 ppm and  $\delta$  –560 ppm, respectively, are characteristic of four-coordinate<sup>22–24</sup> silicon and six-coordinate<sup>30,32</sup> vanadium in derivative (3) (Figure 2).

Derivative (4) exhibits infrared absorption bands ( $\text{cm}^{-1}$ ) due to glycolate moieties,  $\text{Bu}_3\text{Sn}$  groups, and metal–oxygen bonds at 1090; 1040  $\nu(\text{C}-\text{O})$ ; 972  $\nu(\text{V}=\text{O})$ ;<sup>16–19</sup> 599  $\nu_{\text{asym}}(\text{Sn}-\text{C})$ ;<sup>27</sup> 566  $\nu(\text{V}-\text{O})$ ; 532  $\nu(\text{Sn}-\text{O})$ ;<sup>27</sup> and 500  $\nu_{\text{sym}}(\text{Sn}-\text{C})$ .<sup>27</sup>  $^1\text{H}$  NMR signals in (4) appear at  $\delta$  0.95 (t, 3H,  $\text{Sn}(\text{CH}_2)_3\text{CH}_3$ ); 1.22 (s, 24H,  $\text{OCMe}_2$ ); 1.46–1.77 (m, 14H,  $\text{Sn}(\text{CH}_2)_3 + \text{CH}_2\text{CMe}_2\text{O}$ ). Appearance of a  $^{51}\text{V}$  NMR signal at  $\delta$  –677 ppm and a  $^{119}\text{Sn}$  NMR signal at  $\delta$  –14 ppm is consistent with a six- and five-coordinate vanadium<sup>25,26</sup> and tin,<sup>28</sup> respectively (Figure 3).

The derivative (5) shows infrared absorption bands ( $\text{cm}^{-1}$ ) at 1632  $\nu(\text{C}=\text{N})$ ; 1266  $\nu(\text{C}-\text{O})$  phenolic; 1066  $\nu(\text{C}-\text{O})$  alcoholic; 970  $\nu(\text{V}=\text{O})$ ; 584  $\nu_{\text{asym}}(\text{Sn}-\text{C})$ ; 550  $\nu(\text{V}-\text{O})$ ; 540  $\nu(\text{Sn}-\text{O})$ ; 516  $\nu_{\text{sym}}(\text{Sn}-\text{C})$ ; and 475  $\nu(\text{V} \leftarrow \text{N})$ . Infrared absorptions (in  $\text{cm}^{-1}$ ) for (6) are observed at 1316  $\nu(\text{C}-\text{N})$  aromatic amino group and 1233  $\nu(\text{C}-\text{N})$  aliphatic amino group; 1066, 1024  $\nu(\text{C}-\text{O})$ ; 984  $\nu(\text{V}=\text{O})$ ; 591  $\nu_{\text{asym}}(\text{Sn}-\text{C})$ ; 556  $\nu(\text{V}-\text{O})$ ; 547  $\nu(\text{Sn}-\text{O})$ ; 510  $\nu_{\text{sym}}(\text{Sn}-\text{C})$ ; 485  $\nu(\text{V} \leftarrow \text{N})$ .  $^1\text{H}$  NMR spectrum of (5) shows signals at  $\delta$  0.93 (t, 3H,  $\text{Sn}(\text{CH}_2)_3\text{CH}_3$ ); 1.37–1.74 (m, 6H,  $\text{Sn}(\text{CH}_2)_3\text{CH}_3$ ); 3.75 (m, 4H,  $\text{NCH}_2$ ); 3.93 (m, 4H,



**FIGURE 3** Plausible structure for (4).



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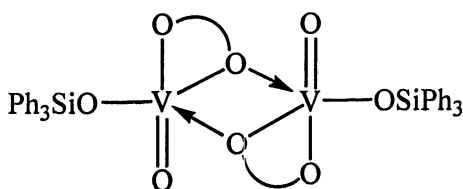
$\delta$  – 600 ppm, respectively is consistent with four-coordinate silicon<sup>22–24</sup> and five-coordinate vanadium<sup>26,33</sup> (Figure 6).

## EXPERIMENTAL

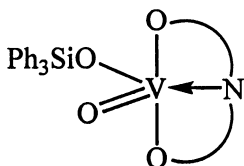
All experimental manipulations were performed under a moisture-free environment using oven-dried ( $\sim 160^\circ\text{C}$ ) glassware, fitted with quick-fit interchangeable joints. Solvents (Merck, India) were dried and purified by the standard methods.<sup>30</sup>  $\text{Me}_3\text{SiCl}$  (Fluka) was distilled (b.p.  $57^\circ\text{C}/758$  mm) prior to use and  $\text{Ph}_3\text{SiOH}$  (Fluka) was used as supplied.  $\text{Bu}_3\text{Sn}(\text{OPr}^i)^{13}$  and  $\text{VO}(\text{OPr}^i)_3^{34}$  were prepared according to the literature methods. 2,5-dimethyl-2,5-hexanediol (Aldrich) and *n*-phenyldiethanolamine (Aldrich) were distilled at  $81^\circ\text{C}/0.2$  mmHg and  $158^\circ\text{C}/0.2$  mmHg, respectively, prior to use. *N*-(1-hydroxyethyl)salicylalimine ( $\text{LH}_2$ ) was prepared<sup>35</sup> by the condensation reaction of salicylaldehyde with monoethanolamine (Merck) in benzene and purified by distillation ( $200^\circ\text{C}/2.1$  mmHg).

Silicon and tin were determined gravimetrically<sup>36</sup> as their oxides. Vanadium was estimated by the redox-titration<sup>37</sup> method. Nitrogen was determined by the Kjeldhal's method.<sup>36</sup>

IR spectra ( $4000$ – $200$   $\text{cm}^{-1}$ ) were recorded on a Nicolet Magna 550 spectrophotometer as Nujol mulls.  $^1\text{H}$  (89.55 MHz),  $^{29}\text{Si}$  (17.75 MHz),  $^{51}\text{V}$  (23.51 MHz), and  $^{119}\text{Sn}$  (33.35 MHz) NMR in  $\text{CDCl}_3$  solutions were recorded on a JEOL FX 90Q FTNMR Spectrometer. Microelemental (C, H, and N) analyses were performed on a Perkin Elemer Series II CHNS/O 2400 analyser.



**FIGURE 5** Plausible structure for (7).



**FIGURE 6** Proposed structure for (8)

## Preparation of Homonuclear Complexes (1a)–(1e)

Due to similarity in the procedures adopted for the preparation of complexes (1a)–(1e), synthetic details of only one typical complex is given below.

### VO(OGO)(OGO) (1a)

A colorless benzene solution containing VO(OPr<sup>i</sup>)<sub>3</sub> (2.39 g, 9.78 mmol) and 2,5-dimethyl-2,5-hexanediol, HOGO (2.86 g, 19.55 mmol) was refluxed under a fractionating column with continuous removal of the liberated isopropyl alcohol as an azeotrope with benzene, and determined by oxidimetric method. When required, an amount of isopropyl alcohol (1.76 g) was distilled out and the distillate showed a negligible presence of an oxidizable material; refluxing was stopped and the reaction mixture was allowed to cool to room temperature. Volatile components from the reaction mixture were removed under reduced pressure (10 mmHg) to obtain a white waxy solid in a quantitative (99%) yield. Recrystallization from the 1:3 mixture of dichloromethane and *n*-hexane afforded an analytically pure compound (1a) as a white waxy solid (2.79 g, 80%). Anal. calcd. for C<sub>16</sub>H<sub>33</sub>O<sub>5</sub>V (356): C, 53.92; H, 9.33; V, 14.29%. Found: C, 53.72; H, 9.27; V, 14.20%. M.wt., 733. IR (Nujol, cm<sup>-1</sup>): 1071, 1036 ν(C–O); 957 ν(V=O); 529 ν(V–O); 443 ν(V←O). <sup>1</sup>H NMR (CDCl<sub>3</sub>; δ, ppm): 1.24 (s, 24H, OCM<sub>2</sub>); 1.60 (s, 8H, CH<sub>2</sub>); 2.21 (br, 1H, OH). <sup>51</sup>V NMR (C<sub>6</sub>H<sub>6</sub>; δ, ppm) –679.

Using the appropriate reactants in the desired molar ratios, complexes (1b)–(1e) were prepared by the procedure similar to that previously described. The actual amounts of the reactants used are given in parentheses against each complex:

(1b): VO(OPr<sup>i</sup>)<sub>3</sub> (2.22 g, 9.09 mmol) and LH<sub>2</sub> (3.00 g, 18.16 mmol).

(1c): VO(OPr<sup>i</sup>)<sub>3</sub> (4.24 g, 17.36 mmol) and PhDEAH<sub>2</sub> (6.30 g, 34.76 mmol).

(1d): VO(OPr<sup>i</sup>)<sub>3</sub> (4.28 g, 17.52 mmol) and HOGO (2.56 g, 17.50 mmol).

(1e): VO(OPr<sup>i</sup>)<sub>3</sub> (2.10 g, 8.59 mmol) and LH<sub>2</sub> (1.42 g, 8.59 mmol).

All these homometallic complexes of (1b)–(1e) have been satisfactorily characterized by elemental analyses and spectroscopic studies:

(1b): Brown solid; m.p., 140°C. Anal. calcd. for C<sub>18</sub>H<sub>19</sub>N<sub>2</sub>O<sub>5</sub>V (394): C, 54.82; H, 4.86; N, 7.10; V, 12.92%. Found: C, 54.79; H, 4.72; N, 7.05; V, 12.80%. IR (Nujol, cm<sup>-1</sup>): 1630 ν(C=N); 1217, 1050 ν(C–O); 967 ν(V=O); 550 ν(V–O); 450 ν(V←N). <sup>1</sup>H NMR (CDCl<sub>3</sub>; δ, ppm): 3.82 (t, J = 2.70 Hz, 4H, NCH<sub>2</sub>); 3.97 (t, J = 2.70 Hz, 4H, CH<sub>2</sub>O); 4.10 (br, 1H, CH<sub>2</sub>OH);



6.93–7.40 (m, 8H, aromatic-**H**); 8.50 (s, 2H, **HC=N**).  $^{51}\text{V}$  NMR ( $\text{CDCl}_3$ ;  $\delta$ , ppm): –554.

(**1c**): Orange solid; m.p., 120°C. Anal. calcd. for  $\text{C}_{20}\text{H}_{27}\text{N}_2\text{O}_5\text{V}$  (426): C, 56.33; H, 6.38; N, 6.57; V, 11.94%. Found: C, 56.37; H, 6.44; N, 6.51; V, 11.90%. IR (Nujol,  $\text{cm}^{-1}$ ): 1365, 1210  $\nu(\text{C-N})$ ; 1047  $\nu(\text{C-O})$ ; 976  $\nu(\text{V=O})$ ; 569  $\nu(\text{V-O})$ ; 484,  $\nu(\text{V}\leftarrow\text{N})$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ;  $\delta$ , ppm): 3.62 (t,  $J = 4.90$  Hz, 8H,  $\text{NCH}_2$ ); 3.90 (t,  $J = 4.90$  Hz, 8H,  $\text{CH}_2\text{O}$ ); 5.07 (br, 1H,  $\text{CH}_2\text{OH}$ ); 6.80–7.40 (m, 10H, aromatic-**H**).  $^{51}\text{V}$  NMR ( $\text{CDCl}_3$ ;  $\delta$ , ppm): –514.

(**1d**): Colorless waxy solid. Anal. calcd. for  $\text{C}_{11}\text{H}_{23}\text{O}_4\text{V}$  (270): C, 48.88; H, 8.58; V, 18.85%. Found: C, 48.72; H, 8.49; V, 18.58%. M.wt., 553. IR (Nujol,  $\text{cm}^{-1}$ ): 1174, 1131  $\nu(\text{OPr}^i)$ ; 1070, 1040  $\nu(\text{C-O})$ ; 980  $\nu(\text{V=O})$ ; 540  $\nu(\text{V-O})$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ;  $\delta$ , ppm): 1.23 (d,  $J = 3.80$  Hz, 6H,  $\text{OCHMe}_2$ ); 1.27 (s, 12H,  $\text{OCMe}_2$ ); 1.60 (s, 4H,  $\text{CH}_2$ ); 4.12 (m, 1H,  $\text{OCHMe}_2$ ).  $^{51}\text{V}$  NMR ( $\text{CDCl}_3$ ;  $\delta$ , ppm): –657.

(**1e**): Brown solid; m.p., 110°C. Anal. calcd. for  $\text{C}_{12}\text{H}_{16}\text{NO}_4\text{V}$  (289): C, 49.83; H, 5.58; N, 4.84; V, 17.61%. Found: C, 49.71; H, 5.49; N, 4.79; V, 17.54%. M.wt., 595. IR (Nujol,  $\text{cm}^{-1}$ ): 1621  $\nu(\text{C=N})$ ; 1237, 1033, 950  $\nu(\text{C-O})$ ; 1151, 1117  $\nu(\text{OPr}^i)$ ; 975  $\nu(\text{V=O})$ ; 566  $\nu(\text{V-O})$ ; 424  $\nu(\text{V}\leftarrow\text{N})$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ;  $\delta$ , ppm): 1.21 (d,  $J = 5.65$  Hz, 6H,  $\text{OCHMe}_2$ ); 4.02 (m, 1H,  $\text{OCHMe}_2$ ); 4.65 (m, 4H,  $\text{CH}_2\text{O} + \text{NCH}_2$ ); 6.80–7.33 (m, 4H, aromatic-**H**); 8.55 (s, 1H, **HC=N**).  $^{51}\text{V}$  NMR ( $\text{CDCl}_3$ ;  $\delta$ , ppm): –535.

## Preparation of Heteronuclear Derivatives (2)–(8)

### **VO(OGO)(OGOSiMe<sub>3</sub>) (2)**

To a benzene (~15 mL) solution of **1a** (2.11 g, 5.93 mmol) was added successively  $\text{Me}_3\text{SiCl}$  (0.64 g, 5.93 mmol) and  $\text{Et}_3\text{N}$  (0.61 g, 5.94 mmol). The resulting reaction mixture was allowed to stir at room temperature for ~4 h. The precipitated  $\text{Et}_3\text{N}\cdot\text{HCl}$  (0.81 g) was removed by filtration. After removal of volatile components from the filtrate, a colorless waxy solid was obtained in a quantitative (~98%) yield. Recrystallisation from *n*-hexane at –20°C afforded an analytically pure derivative (**2**) as a white waxy solid 1.76 g, 69%. Analytical details are given in Table I.

Adopting the procedure described for (**2**), derivative (**3**) was prepared by the interaction of **1b** (2.36 g, 5.98 mmol) with  $\text{Me}_3\text{SiCl}$  (0.65 g, 5.98 mmol) in the presence of  $\text{Et}_3\text{N}$  (0.60 g, 5.93 mmol).

### **VO(OGO)(OGOSnBu<sub>3</sub>) (4)**

A colorless benzene (~40 mL) solution containing (**1a**) (1.40, 3.92) and  $\text{Bu}_3\text{Sn(OPr}^i)$  (1.37, 3.92) was refluxed for 4 h with continuous removal of the liberated isopropyl alcohol as an azeotrope with benzene. When the required amount (0.23 g) of isopropyl alcohol was distilled

TABLE I Analytical and Molecular Weight Data for Heteronuclear Derivatives (2)–(8)

Compound <sup>a</sup>	Color	Yield <sup>b</sup> g (%)	Et <sub>3</sub> N·HCl/ Pr <sup>i</sup> OH (g)		Analysis (%) Found (Calc.)					Mol. Wt. Found (Calc.)
			Found (Calc.)	Si/Sn	V	C	H	N		
C <sub>19</sub> H <sub>41</sub> O <sub>5</sub> SiV (2)	Colorless	1.76 (69)	0.81 (0.83)	6.31 (6.55)	11.63 (11.88)	53.57 (53.24)	9.41 (9.64)	—	866 (428)	
C <sub>21</sub> H <sub>27</sub> N <sub>2</sub> O <sub>5</sub> SiV (3)	Brown	2.01 (72)	0.80 (0.82)	5.91 (6.02)	10.78 (10.92)	54.00 (54.06)	5.91 (5.83)	5.90 (6.00)	471 (466)	
C <sub>28</sub> H <sub>59</sub> O <sub>5</sub> SnV (4)	White	1.69 (67)	0.22 (0.23)	18.11 (18.39)	7.81 (7.89)	52.22 (52.10)	9.09 (9.21)	—	1303 (645)	
C <sub>30</sub> H <sub>45</sub> N <sub>2</sub> O <sub>5</sub> SnV (5)	Red	2.11 (66)	0.26 (0.28)	17.29 (17.37)	7.11 (7.45)	52.61 (52.72)	6.69 (6.64)	4.11 (4.10)	699 (683)	
C <sub>32</sub> H <sub>53</sub> N <sub>2</sub> O <sub>5</sub> SnV (6)	Black	1.89 (76)	0.17 (0.18)	16.60 (16.59)	7.10 (7.12)	53.73 (53.71)	7.20 (7.47)	3.86 (3.91)	729 (715)	
C <sub>26</sub> H <sub>31</sub> O <sub>4</sub> SiV (7)	Colorless	1.54 (62)	0.30 (0.30)	5.64 (5.77)	10.40 (10.47)	64.11 (64.17)	6.10 (6.42)	—	980 (486)	
C <sub>27</sub> H <sub>24</sub> NO <sub>4</sub> SiV (8)	Brown	1.41 (70)	0.22 (0.23)	5.27 (5.55)	10.01 (10.07)	64.08 (64.14)	4.90 (4.78)	2.49 (2.77)	511 (505)	

<sup>a</sup>All are solids.

<sup>b</sup>Refers to recrystallised product.

out and further collected, azeotrope showed no evidence of the presence of an oxidizable material; the refluxing was stopped and the reaction mixture was allowed to cool to room temperature. Volatile components from the solution were removed under reduced (10 mmHg) pressure to obtain a quantitatively (>99%) colorless semisolid (**4**), which solidified as a white solid after ~10 h. Recrystallisation from *n*-hexane afforded (**4**) as a white waxy solid (1.69 g, 67%). Analytical details are listed in Table I.

Derivatives (**5**) and (**6**) were synthesized by a method similar to that used for the preparation of (**4**). The actual amounts of the reactants used are given in parentheses against each derivative:

**(5): 1b** (1.85 g, 4.69 mmol) and  $\text{Bu}_3\text{Sn}(\text{OPr}^i)$  (1.64 g, 4.69 mmol).

**(6): 1c** (1.32 g, 3.09 mmol) and  $\text{Bu}_3\text{Sn}(\text{OPr}^i)$  (1.08 g, 3.09 mmol).

Analytical details are summarized in Table I.

### **( $\text{Ph}_3\text{SiO}$ )VO( $\text{OGO}$ ) (**7**)**

To a colorless benzene (~30 mL) solution of **1d** (1.37 g, 5.07 mmol) was added  $\text{Ph}_3\text{SiOH}$  (1.40 g, 5.07 mmol) and the resulting reaction mixture was refluxed for ~3 h with continual removal of isopropyl alcohol. After the collection of the required (0.30 g) amount of isopropyl alcohol as an azeotrope, refluxing was stopped and the reaction mixture was allowed to cool to room temperature. Volatile components from the solution were removed under reduced pressure to obtain a colorless waxy solid in a quantitative (~99%) yield. Recrystallisation from *n*-hexane afforded analytically pure (**7**) as a colorless waxy solid (1.78 g, 72%). Analytical details are given in Table I.

Using the reactants **1e** (1.14 g, 3.94 mmol) and  $\text{Ph}_3\text{SiOH}$  (1.09 g, 3.94 mmol) derivative (**8**) was prepared by the method similar to that described for (**7**). Analytical details are listed in Table I.

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