

*Supporting Information for*

**Simple Routes to Silyl Substituted Acetylide Ligands and Examples of V(III), Fe(II) and Mn(II) Complexes**

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**Experimental Physical Measurements.** Elemental analyses were performed by Columbia Analytical.  $^1\text{H}$  NMR spectra were recorded at ambient temperature using a Varian 300, 400, and 600 MHz spectrometer. Chemical shifts were referenced to residual solvent. Electrochemical measurements were recorded in a glovebox under a dinitrogen atmosphere using a CH Instruments Electrochemical Analyzer, a glassy carbon working electrode, a platinum wire auxiliary-electrode, and an  $\text{Ag}/\text{AgNO}_3$  non-aqueous reference electrode. Reported potentials are all referenced to the SCE couple, and were determined using decamethylferrocene as an internal standard. The number of electrons passed in a given redox process was estimated by comparison of the peak current with the peak current of decamethylferrocene included as an internal standard. Magnetic measurements were recorded using a Quantum Designs MPMS XL magnetometer at 0.1 T. The sample was contained under nitrogen in a gelcap and suspended in the magnetometer in a plastic straw. The magnetic susceptibility was adjusted for diamagnetic contributions using the constitutive corrections of Pascal's constants.

**X-ray Structure determinations.** X-ray diffraction studies were carried out on a Bruker SMART APEXII and a Bruker SMART APEX Duo diffractometer equipped with a CCD detector.<sup>1a</sup> Measurements were carried out at  $-175\text{ }^\circ\text{C}$  using both Cu and Mo  $K\alpha$  ( $\lambda = 1.54178$  and  $\lambda = 0.71073\text{ \AA}$ , respectively) radiation. Crystals were mounted on a glass capillary or Kapton Loop with Paratone-N oil. Initial lattice parameters were obtained from a least-squares analysis of more than 100 centered reflections; these parameters were later refined against all data. Data were integrated and corrected for Lorentz polarization effects using SAINT<sup>1b</sup> and were corrected for absorption effects using SADABS 2.3.<sup>1c</sup>

Space group assignments were based upon systematic absences,  $E$  statistics, and successful refinement of the structures. Structures were solved by direct methods with the aid of successive difference Fourier maps and were refined against all data using the SHELXTL 5.0 software package.<sup>1d</sup> Thermal parameters for all non-hydrogen atoms were refined anisotropically. Hydrogen atoms, where added, were assigned to ideal positions and refined using a riding model with an isotropic thermal parameter 1.2 times that of the attached carbon atom (1.5 times for methyl hydrogens).

**Preparation of Compounds.** All procedures were performed under a dinitrogen atmosphere using standard Schlenk or glove box techniques unless otherwise noted. Solvents

were thoroughly sparged with argon and dried over activated alumina prior to use. Solvents were stored over 3 Å molecular sieves when not used immediately. Trimethylsilylchloride reagent was distilled and stored over 3 Å molecular sieves. Triethylamine was stirred in NaOH overnight and distilled prior to usage. Deuterated solvents for NMR spectroscopy were obtained from Cambridge Isotopes Laboratories, Inc., degassed with dinitrogen and stored over 3 Å molecular sieves. 2-trimethylsilylphenylacetylene and (2-Bromophenylethynyl)trimethylsilane were prepared according to a previously reported procedure.<sup>2,3</sup> All other starting reagents were used as received.

**2,6-Bis(trimethylsilyl)phenylacetylene (1).** A solution of *n*BuLi (130.1 mL, 2.5M in hexane, 325 mmol) was transferred to a 3-neck, round-bottom flask. The hexane was removed under vacuum, and the flask was subsequently cooled to -78°C, before 100 mL of THF was added. A solution of 2-trimethylsilylphenylacetylene (27.7 g, 158 mmol) in 100 mL of THF was added dropwise over 0.25 h, resulting in the formation of a golden brown colored solution. The solution was warmed to -25°C and stirred for 1 hour, then cooled to -78°C before KO<sup>t</sup>Bu (36.5 g, 325 mmol) was added slowly with vigorous stirring. The solution was warmed to -43°C for 90 minutes, and then again cooled to -78°C before TMSCl (41.3 mL, 325 mmol) was added via syringe. The reaction mixture was stirred for 16 h before the THF was removed in vacuo. A solution of NaOH (1.34 M in 95% ethanol, 400 mL) was added to the resulting golden-brown gel and the mixture was heated at reflux for 1 hour in air. Upon cooling to room temperature, 400 mL of cold water was added and the mixture was extracted with hexanes (3 × 500 mL, 3 × 300 mL, 3 × 100 mL). The combined organic layers were washed with water (2 × 200 mL) and brine (2 × 100 mL), and dried over MgSO<sub>4</sub>. The hexane was removed in vacuo to give a viscous, red-orange oil. GC-MS analysis revealed a mixture of 2-trimethylsilylphenylacetylene, **1** and an undetermined isomer of tris(trimethylsilyl)phenylacetylene in an approximate ratio of 32:77:1. The 2-trimethylsilylphenylacetylene was collected by vacuum distillation (41°C, 0.1 mmHg) over 16 h. The remaining mixture was redistilled (60°C, 0.1 mmHg) to yield **1** as a white solid which was recrystallized from methanol (19.0 g, 49%). <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>): 7.50 (d, 2H, *m*-H), 7.29 (t, 1H, *p*-H), 3.38 (s, 1H, CCH), 0.38 (s, 18H, Si(CH<sub>3</sub>)<sub>3</sub>) ppm. <sup>13</sup>C{<sup>1</sup>H} (600 MHz, C<sub>6</sub>D<sub>6</sub>): 143 (s, 1C, C(CCH)), 135 (s, 2C, *m*-C(CCH)), 133 (s, 1C, *p*-C(CCH)), 127 (s, 2C, C-TMS), 86.9 (s, 1C, C(CH)), 84.4 (s, 1C, CH), -0.67 ppm (s, 6C, TMS). <sup>29</sup>Si (600 MHz, C<sub>6</sub>D<sub>6</sub>): -

3.52 ppm (s, 2Si). IR (KBr):  $\nu_{\text{CC}}$  2094  $\text{cm}^{-1}$  (w),  $\nu_{\text{CH}}$  3266  $\text{cm}^{-1}$  (m), 3280  $\text{cm}^{-1}$  (s), 3291  $\text{cm}^{-1}$  (s), 3312  $\text{cm}^{-1}$  (w). Anal. Calcd. for  $\text{C}_{14}\text{H}_{22}\text{Si}_2$ : C, 67.94; H, 8.96. Found: C, 68.18; H, 8.57 %.

**2-(bromophenylethynyl)trimethylsilane (2a).** This procedure is slightly modified from the reported procedure.<sup>3</sup> A 350 mL flame-dried reaction tube was charged with  $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$  (1.240 g, 1.76 mmol) and CuI (0.96 g, 3.53 mmol) dissolved in 250 mL of triethylamine and then 2-bromoiodobenzene (10.0 g, 35.35 mmol) and trimethylsilyl acetylene (5.2 mL, 36.79 mmol) were added. The mixture was stirred vigorously for 24 hours. The resulting darkly colored solution was acidified using 2M HCl (~800 mL), extracted with hexane (400 mL) and neutralized with sat.  $\text{NaHCO}_3$  solution (200 mL). The organic layer was collected, filtered through silica gel, dried over  $\text{Na}_2\text{SO}_4$  and subsequently concentrated to afford 2-(bromophenylethynyl)trimethylsilane (8.2 g, 91%) as an orange-brown oil which was used without further purification. Compound **2a** was identified by comparison with spectra reported in the literature.<sup>3</sup>  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ): 7.55 (d, 1H, *o*-H), 7.48 (d, 1H, *m*-H), 7.22 (t, 1H, *m*-H), 7.14 (t, 1H, *p*-H), 0.35 – 0.20 (m, 9H,  $\text{Si}(\text{CH}_3)_3$ ).  $^{13}\text{C}\{^1\text{H}\}$  (300 MHz,  $\text{CDCl}_3$ ): 133.0 (s, 1C, C(CCSi)), 132.6 (s, 1C, *o*-CBr), 129.8 (s, 1C, *o*-CH), 127.1 (s, 1C, *m*-CH), 125.9 (s, 1C, *p*-CH), 125.5 (s, 1C, *m*-C(CBr)), 103.3 (s, 1C, C(CSi( $\text{CH}_3$ )<sub>3</sub>)), 99.9 (s, 1C, C(Si( $\text{CH}_3$ )<sub>3</sub>)), 0.26 (s, 3C, C( $\text{H}_3$ )<sub>3</sub>). GC-MS 254.94 *m/z*.

**2-(Triphenylsilyl)phenylacetylene (2).** A mixture of sodium metal (3.45 g, 150.07 mmol) and toluene (250 mL) was heated at reflux under dinitrogen for 1 hr. A solution of **2a** (8.50 g, 33.35 mmol) dissolved in toluene (350 mL) was added via canula to the mixture along with solid triphenylsilyl chloride (9.83 g, 33.34 mmol) and the reaction mixture heated at reflux for an additional 3 hours. The resulting darkly colored solution was filtered, concentrated to ca. 50 mL and then stored at  $-10^\circ\text{C}$  for 16 h to afford 5.90 g of 2-(bromophenylethynyl)trimethylsilane as a white crystalline solid. 2-(bromophenylethynyl)trimethylsilane (3.0 g, 6.94 mmol) was added to a 1.34 M solution of KOH in EtOH (50 mL) in a round bottomed flask and the mixture was heated at reflux for 45 min. Ice-cold deionized water was added to the reaction mixture and was subsequently extracted with ether (150 mL) and hexanes (300 mL). The combined organic layer was dried over  $\text{Na}_2\text{SO}_4$  and the solvent removed in vacuo to afford **2** as a white powder (2.30 g, 92%).  $^1\text{H}$  NMR (600 MHz,  $\text{C}_6\text{D}_6$ ): 7.73 (dd, 6H, *o*-H), 7.47 (dd, 2H, *o*-H), 7.20-7.12 (m, 9H, *m*-H, *p*-H), 6.94 (dt, 2H,

*m*-H), 2.28 (s, 1H, CCH).  $^{13}\text{C}\{^1\text{H}\}$  (600 MHz,  $\text{C}_6\text{D}_6$ ): 138.1 (s, 1C, C(CCH)), 137.5 (s, 1C, *o*-C(CCH)), 136.7 (s, 1C, *m*-C(CCH)), 134.4 (s, 1C, *p*-C(CCH)), 133.8 (s, 1C, *m*-C(CCH)), 129.9 (s, 6C, *o*-C(SiC)), 129.6 (s, 6C, *m*-C(CSi)), 129.4 (s, 3C, *p*-C(CSi)), 128.3 (s, 1C, *o*-C(SiC)), 127.9 (s, 1C, *o*-C(SiC)) 84.7 (s, 1C, C(CH)), 82.4 (s, 1C, CH).  $^{29}\text{Si}$  (600 MHz,  $\text{C}_6\text{D}_6$ ): -13.38 ppm (s, 1Si). GC-MS 360.12 *m/z*. IR (KBr):  $\nu_{\text{CC}}$  2104 (w),  $\nu_{\text{CH}}$  3286 (s), 3132 (w), 3062 (s), 3044 (s), 3007 (m), 2956 (w), 2923 (w)  $\text{cm}^{-1}$ . Anal. Calcd. for  $\text{C}_{14}\text{H}_{22}\text{Si}_2$ : C, 86.62; H, 5.59. Found: C, 86.75; H, 5.80 %.

**[Li·THF][(2,6-(Me<sub>3</sub>Si)<sub>2</sub>PhC≡C)<sub>4</sub>V(III)] (3).** A solution of **1** (10.0g, 40.4 mmol) dissolved in 100 mL of benzene was cooled to -78°C. After addition of Li[N(*i*Pr)<sub>2</sub>] (21.2 mL, 2.0M THF solution, 42.4 mmol) to the thawing solution at room temperature, the reaction mixture was stirred for 30 min at room temperature. After chilling at -35°C, a solution of VCl<sub>3</sub>·THF (3.77 g, 10.1 mmol) in 60 mL of benzene was added to the thawing solution which was then stirred at room temperature for 3 days. After removal of the benzene in vacuo, the dark olive green residue was dissolved in 10 mL of THF, 50 mL of hexanes were added and the resulting solution was stored at -35°C for 24 h. The blue powder which precipitated was collected and washed with cold pentane (15 mL). Dark blue needle-shaped crystals suitable for X-ray diffraction analysis were obtained by diffusion of pentane into a THF solution of the blue powder at room temperature to yield **3** (5.70 g, 42%).  $^1\text{H}$  NMR (400 MHz,  $\text{C}_6\text{D}_6$ ): 21.81 (br), 3.36 (br), 1.31 (br). IR (KBr):  $\nu_{\text{CC}}$  2006  $\text{cm}^{-1}$  (w), 2033  $\text{cm}^{-1}$  (m), 2049  $\text{cm}^{-1}$  (s). UV-Vis  $\lambda(\epsilon)$ : 216 (105,300), 240 (47,600), 278 (28,000), 328 (33,400), 342 (35,100), 612 (2,700) nm ( $\text{Lmol}^{-1}\cdot\text{cm}^{-1}$ ). Anal. Calcd. for  $\text{C}_{14}\text{H}_{22}\text{Si}_2$ : C, 65.02; H, 8.67. Found: C, 65.37; H, 8.91 %.  $\mu_{\text{eff}} = 2.78 \mu_{\text{B}}$  at 300 K.

**[Li·THF]<sub>2</sub>[(2,6-(Me<sub>3</sub>Si)<sub>2</sub>PhC≡C)<sub>4</sub>Fe(II)] (4).** A solution of **1** (5.0g, 20.2 mmol) dissolved in 20 mL of THF was cooled to -35°C. After addition of Li[N(*i*Pr)<sub>2</sub>] (10.6 mL, 2.0M THF solution, 21.2 mmol) the solution was stirred at -35°C for 2 hours before being added, dropwise at room temperature, to a stirred 20 mL THF suspension of FeCl<sub>2</sub> (0.64 g, 5.05 mmol) which had been chilled at -35°C. The resulting dark orange-brown solution was cooled to -35°C for one hour and then stirred at room temperature for 3 days. THF was removed in vacuo to yield a dark orange-brown, oily residue. The oily residue was stirred in 50 mL of hexanes to afford **2** as a yellow powder which was collected by filtration. The yellow powder was extracted into benzene (50 mL), the resulting solution filtered through celite, and the benzene removed in

vacuo. The resulting powder was washed with hexanes (15 mL) and collected by filtration. Yellow block-shaped crystals of **4** were obtained by diffusion of pentane into a THF solution of **4** (2.04g, 34%).  $^1\text{H}$  NMR (400 MHz,  $\text{C}_6\text{D}_6$ ): 22.7 (s), 2.97 (br), -2.40 (s), -3.56 (b), -4.33 (s) ppm. IR (KBr):  $\nu_{\text{CC}}$  2031  $\text{cm}^{-1}$  (m). UV-Vis  $\lambda(\epsilon)$ : 216 (59,300), 234 (47,000), 262 (16,100), 294 (30,000), 310 (23,600) nm ( $\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$ ). Anal. Calcd. for  $\text{C}_{14}\text{H}_{22}\text{Si}_2$ : C, 64.28; H, 8.43. Found: C, 63.96; H, 8.17 %.  $\mu_{\text{eff}} = 5.08 \mu_{\text{B}}$  at 300 K.

**[Li·THF]<sub>2</sub>[(2,6-(Me<sub>3</sub>Si)<sub>2</sub>PhC≡C)<sub>4</sub>Mn(II)] (5).** Compound **3** was prepared from  $\text{MnI}_2$  (1.56 g, 5.05 mmol) and **1** (5.0g, 20.2 mmol) by a method analogous to that described for the preparation of **3** except that solutions were cooled to  $-78^\circ\text{C}$  instead of  $-35^\circ\text{C}$  wherever indicated. Colorless block-shaped crystals suitable for X-ray diffraction were obtained by diffusion of pentane into a THF solution of **5** at room temperature (2.69 g, 45%).  $^1\text{H}$  NMR (400 MHz,  $\text{C}_6\text{D}_6$ ): 3.28 (br), 1.59 (m) ppm. IR (KBr):  $\nu_{\text{CC}}$  2049  $\text{cm}^{-1}$  (w). UV-Vis  $\lambda(\epsilon)$ : 216 (49,500), 236 (37,800), 262 (10,900), 284 (25,800), 294 (31,400)  $\text{nm}$  ( $\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$ ). Anal. Calcd. for  $\text{C}_{14}\text{H}_{22}\text{Si}_2$ : C, 64.33; H, 8.43. Found: C, 64.64; H, 8.56 %.  $\mu_{\text{eff}} = 6.01 \mu_{\text{B}}$  at 300 K.

**Table S1.** Crystallographic data<sup>a</sup> for the complexes [Li·4THF][(2,6-(Me<sub>3</sub>Si)<sub>2</sub>PhC≡C)<sub>4</sub>V(III)] (**3**), [Li·THF]<sub>2</sub>[(2,6-(Me<sub>3</sub>Si)<sub>2</sub>PhC≡C)<sub>4</sub>Fe(II)] (**4**), and [Li·THF]<sub>2</sub>[(2,6-(Me<sub>3</sub>Si)<sub>2</sub>PhC≡C)<sub>4</sub>Mn(II)] (**5**).

	<b>3</b>	<b>4</b>	<b>5</b>
Formula	C <sub>72</sub> H <sub>116</sub> LiO <sub>4</sub> Si <sub>8</sub> V	C <sub>69</sub> H <sub>112</sub> FeLi <sub>2</sub> O <sub>2</sub> Si <sub>8</sub>	C <sub>69</sub> H <sub>114</sub> Li <sub>2</sub> O <sub>2</sub> MnSi <sub>8</sub>
Crystal size	0.15 × 0.07 × 0.06	0.07 × 0.05 × 0.03	0.21 × 0.17 × 0.13
Formula weight, g·mol <sup>-1</sup>	1328.25	1268.04	1269.14
Space group	<i>P</i> 4 <sub>1</sub> 2 <sub>1</sub> 2	<i>P</i> 1	<i>I</i> 2
<i>a</i> , Å	14.788(3)	12.393(6)	15.202(8)
<i>b</i> , Å	14.788(3)	13.481(6)	12.406(6)
<i>c</i> , Å	38.081(9)	14.580(7)	20.766(1)
<i>a</i> , deg	90	93.651(2)	90
<i>b</i> , deg	90	114.647(2)	94.668(1)
<i>g</i> , deg	90	114.067(2)	90
<i>V</i> , Å <sup>3</sup>	8328.3(3)	1939.8(2)	3903.2(3)
<i>Z</i>	4	1	2
<i>T</i> , K	90(2)	90(2)	90(2)
ρ, calcd, g cm <sup>-3</sup>	1.059	1.085	1.080
Refl. collected/2θ <sub>max</sub>	7380/137.14	11875/50.64	11838/61.00
Unique refl./ <i>I</i> > 2σ ( <i>I</i> )	6824	11820	11460
No. parameters/restraints	389/0	785/35	410/19
λ, Å/μ (Ka), cm <sup>-1</sup>	1.54178/2.403	0.71073/0.356	0.71073/0.330
R <sub>1</sub> /GOF <sup>b</sup>	0.0374/1.048	0.0449/1.094	0.0310/1.044
wR <sub>2</sub> ( <i>I</i> > 2σ( <i>I</i> )) <sup>b</sup>	0.0973	0.1217	0.0867
Residual density, e Å <sup>-3</sup>	+0.43/-0.18	+0.60/-0.31	+0.50/-0.53

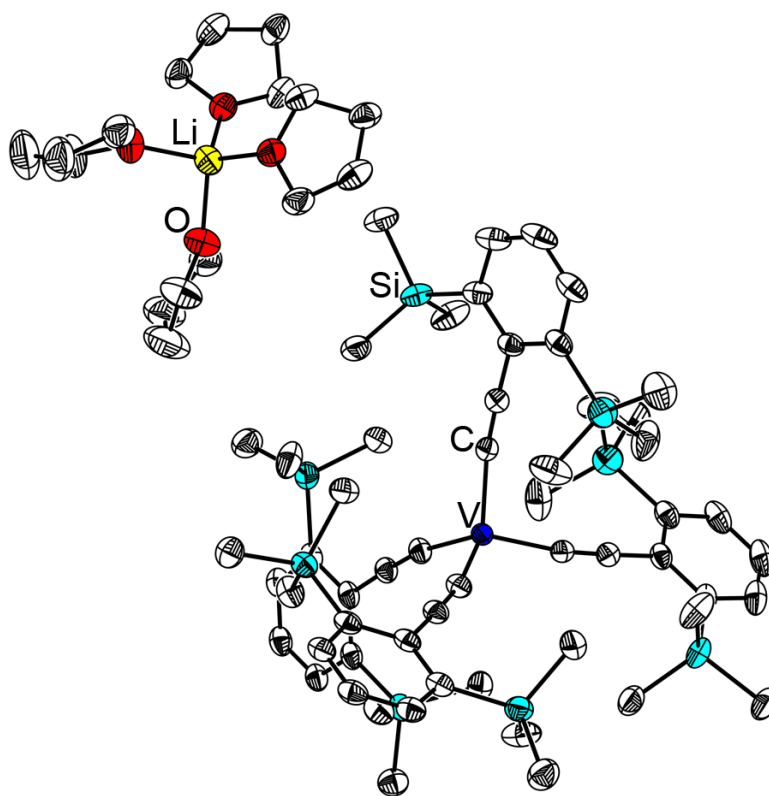
<sup>a</sup>Obtained with graphite-monochromated Cu and Mo Kα (λ = 1.54178 Å and 0.71073 Å, respectively) radiation.

<sup>b</sup>R<sub>1</sub> = Σ||F<sub>o</sub>| - |F<sub>c</sub>||/Σ|F<sub>o</sub>|, wR<sub>2</sub> = {Σ[w(F<sub>o</sub><sup>2</sup> - F<sub>c</sub><sup>2</sup>)<sup>2</sup>]/Σ[w(F<sub>o</sub><sup>2</sup>)<sup>2</sup>]}<sup>1/2</sup>

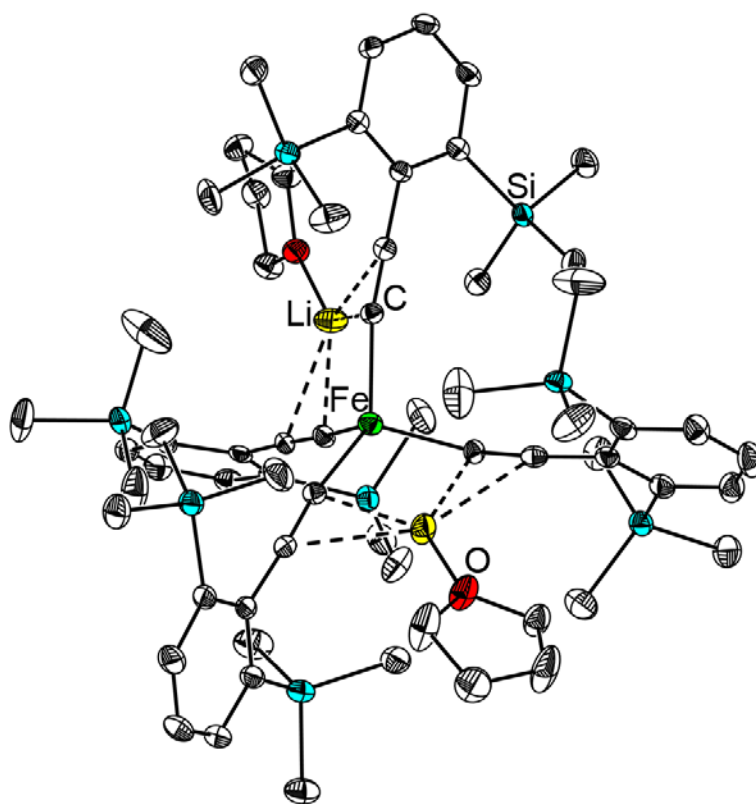
**Table S2.** Selected average interatomic distances (Å) and angles (deg) for the complexes [Li·4THF][(2,6-(Me<sub>3</sub>Si)<sub>2</sub>PhC≡C)<sub>4</sub>V(III)] (**3**), [Li·THF]<sub>2</sub>[(2,6-(Me<sub>3</sub>Si)<sub>2</sub>PhC≡C)<sub>4</sub>Fe(II)] (**4**), [Li·THF]<sub>2</sub>[(2,6-(Me<sub>3</sub>Si)<sub>2</sub>PhC≡C)<sub>4</sub>Mn(II)] (**5**).

	<b>3</b>	<b>4</b>	<b>5</b>
M-C	2.023(2)	2.043(3)	2.127(1)
C≡C	1.218(3)	1.219(5)	1.225(2)
C-Ar	1.437(3)	1.445(5)	1.441(2)
Si-Ar	1.883(2)	1.899(4)	1.893(2)
≡C-M-C≡ (no Li)	105.5(1), 106.8(1), 116.4(1)	103.4(1), 122.7(1)	106.0(1), 126.9(1)
≡C-M-C≡ (with Li)	—	103.2(1)	98.5(1)
M-C≡C	169.8(2)	165.3(3)	164.9(1)
C-C-Ar	177.3(2)	177.0(4)	177.0(1)

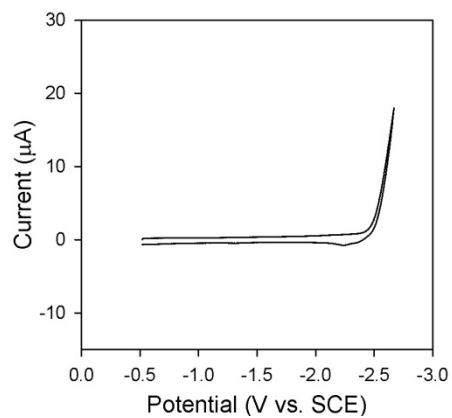




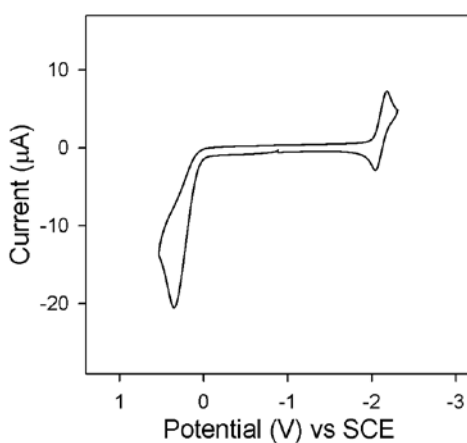
**Figure S1.** Solid state structure of  $[(2,6-(\text{Me}_3\text{Si})_2\text{PhC}\equiv\text{C})_4\text{V(III)}]^-$  in **3**. Dark blue, white, yellow, red, and light blue atoms represent vanadium, carbon, lithium, oxygen and silicon atoms, respectively. Thermal ellipsoids shown at 40% probability and H atoms omitted for clarity.



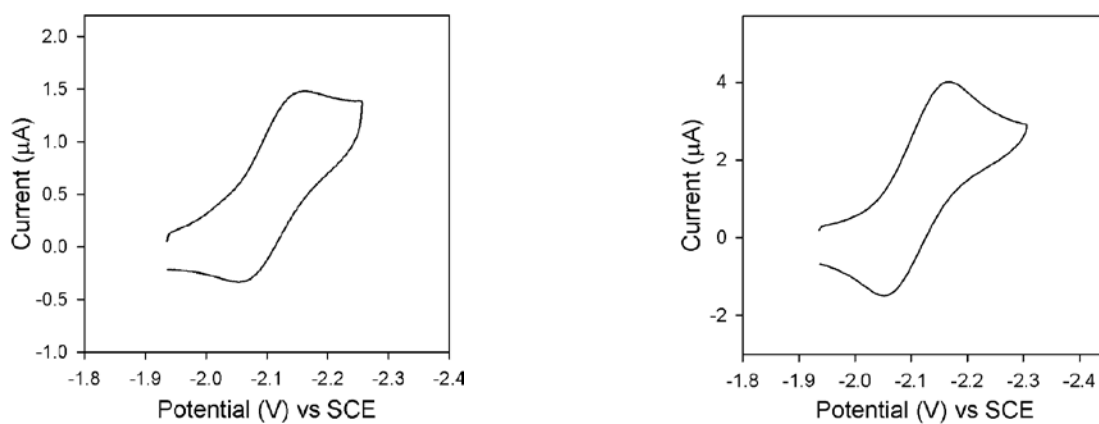
**Figure S2.** Solid state structure of **4** showing lithium ions coordinated between the ligands. Green, white, yellow, red and light blue atoms represent iron, carbon, lithium, oxygen, and silicon atoms, respectively. Thermal ellipsoids shown at 40% probability and H atoms omitted for clarity.



**Figure S3.** Cyclic voltammogram for the ligand, **1**.



**Figure S4.** Cyclic voltammogram for complex **5**.



**Figure S5.** Cyclic voltammogram for complexes **4** and **5** showing the reversible wave observable in the cathodic scan direction.

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- 1 a) SMART Software Users Guide, Version 5.1, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999. b) SAINT Software Users Guide, Version 7.0 Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999. c) G.M. Sheldrick SADABS, Version 2.03, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 2000. d) G.M. Sheldrick SHELXTL Version 6.12, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999.
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  - 3 G. Mao, A. Orita, D. Matsuo, T. Hirate, T. Iwanaga, S. Toyota, J. Otera, *Tet. Lett.*, 2009, **50**, 2860.