Supplementary Materials

Isolation of two Bis(silyl)nickel Complexes with Si–Si Bond Formation in Single-Crystal-to-Single-Crystal Fashion

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1. Copies of NMR spectra of the product

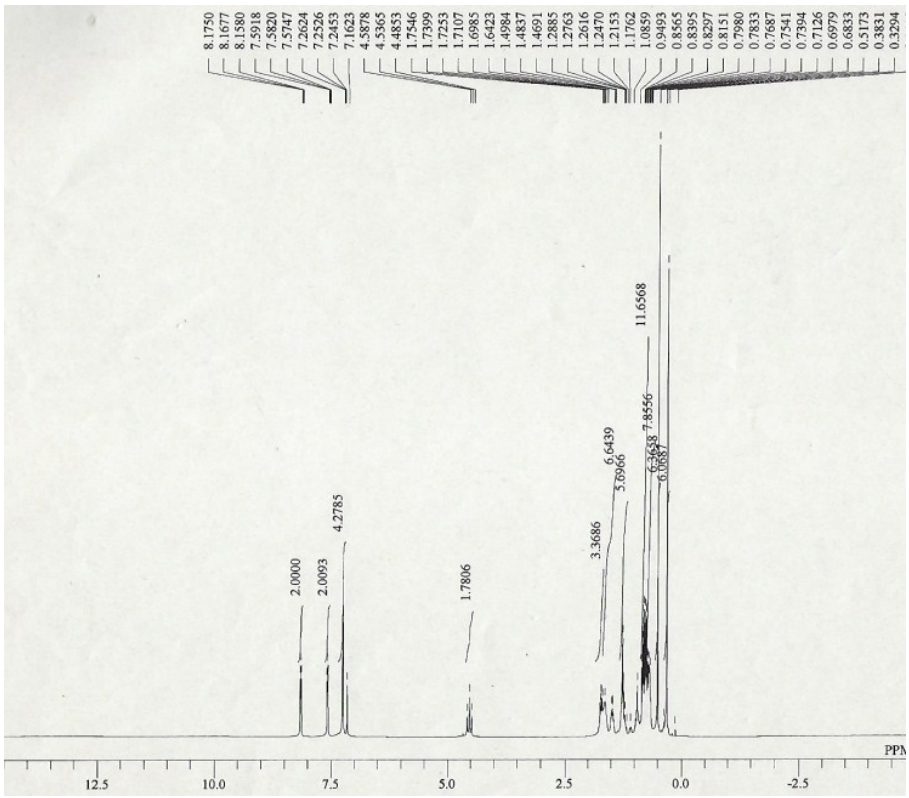

Figure S1: ^{1}H NMR spectrum (THF-d_8) for compound **6**

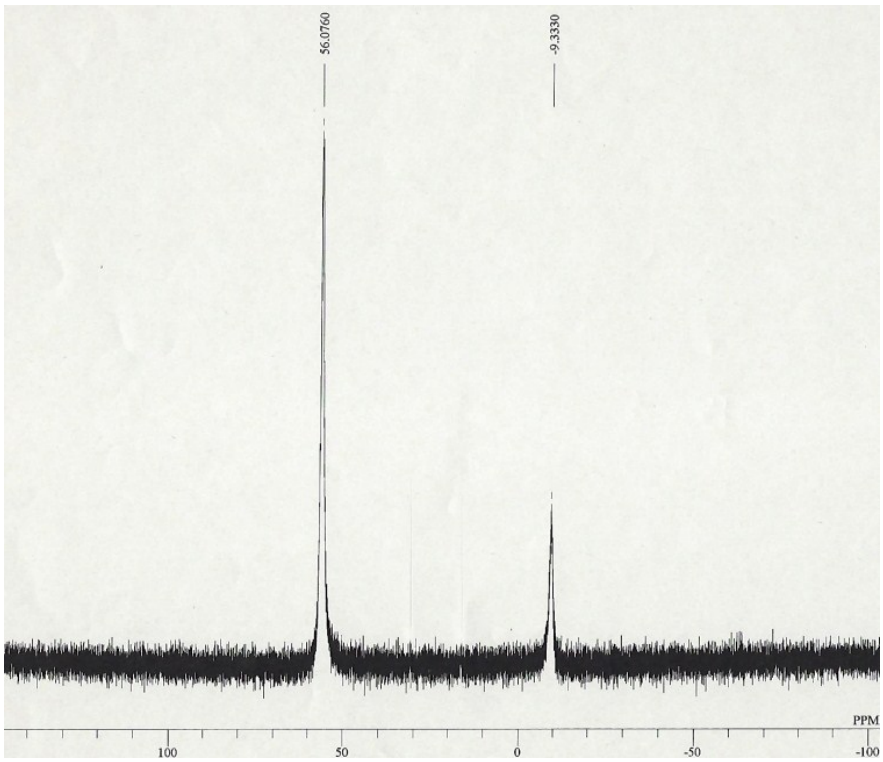

Figure S2: $^{31}P\{^{1}H\}$ NMR spectrum (THF-d_8) for compound **6**

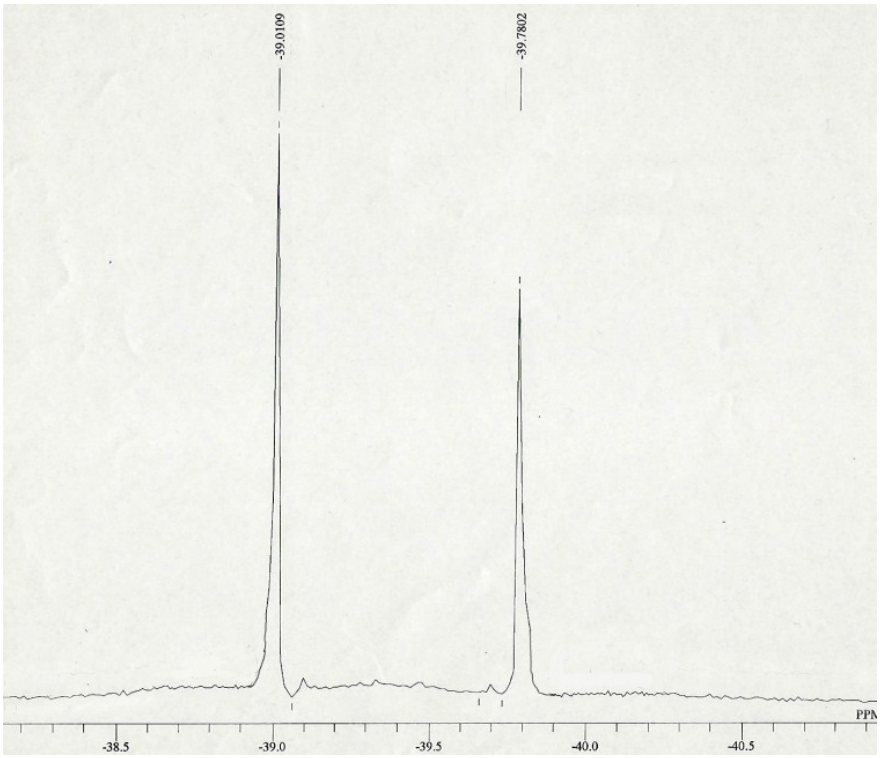

Figure S3: $^{29}Si\{^{1}H\}$ NMR spectrum (THF-$d_{8,}$ DEPT) for compound **6**

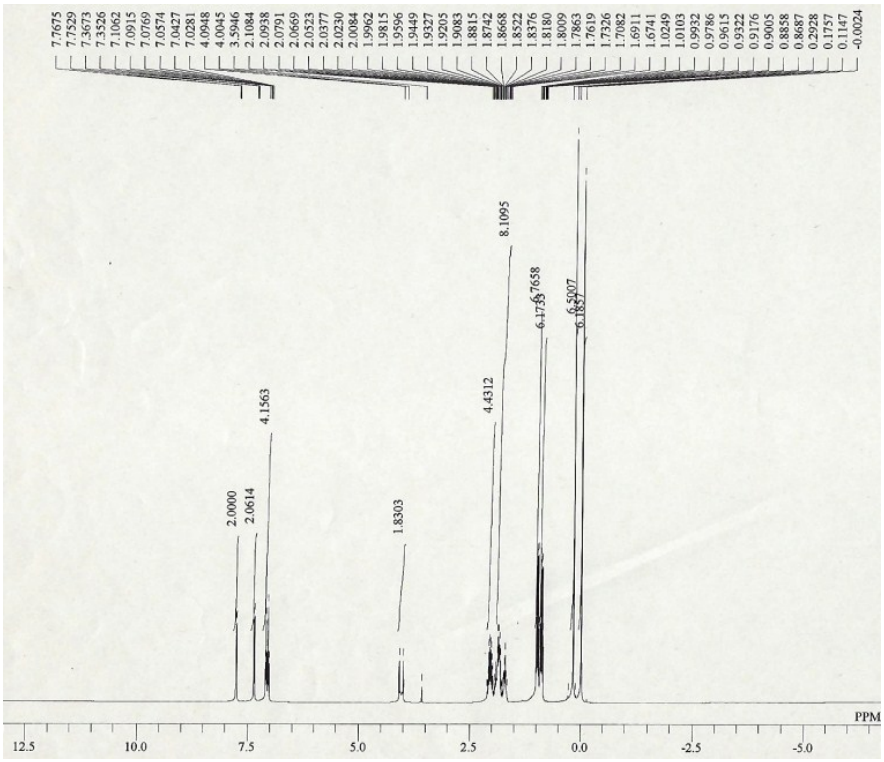

Figure S4: ^{1}H NMR spectrum (THF-d_8) for compound **6'**

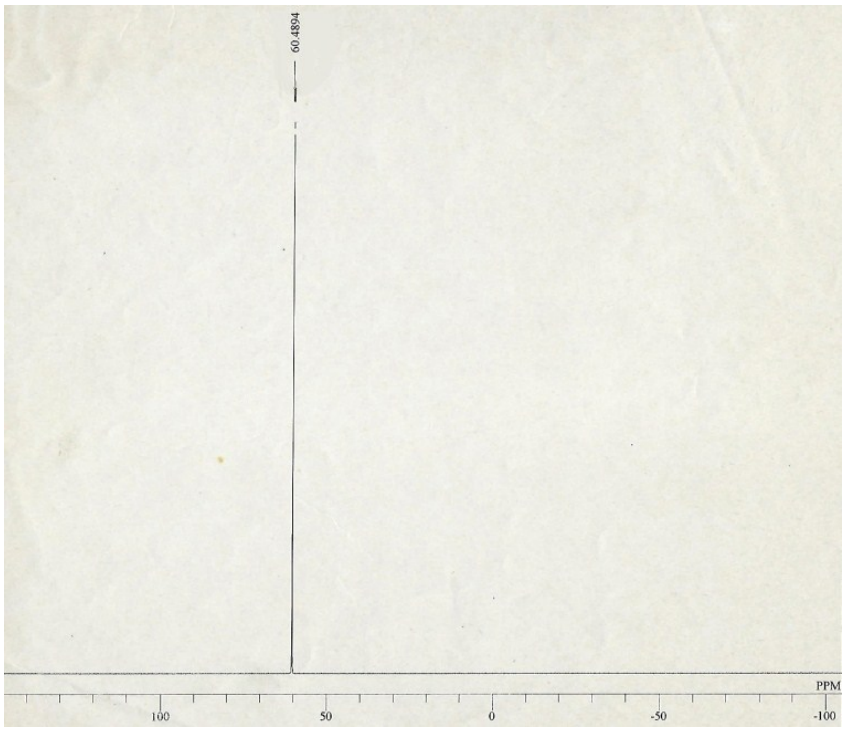

Figure S5: $^{31}P\{^{1}H\}$ NMR spectrum (THF-d_8) for compound **6'**

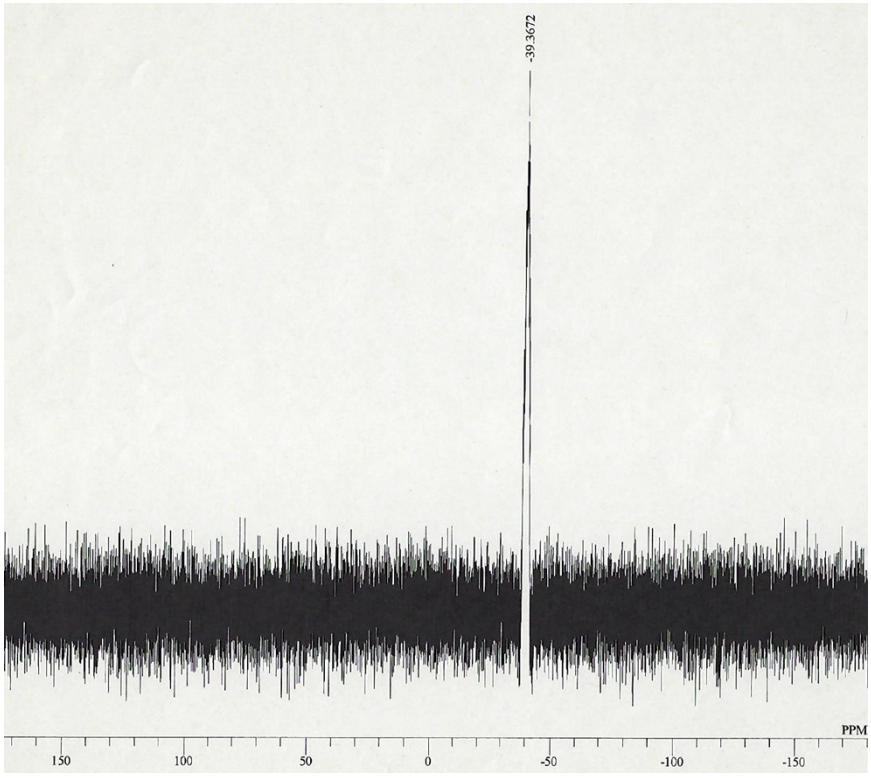

Figure S6: $^{29}Si\{^{1}H\}$ NMR spectrum (THF-$d_{8,}$ DEPT) for compound **6'**

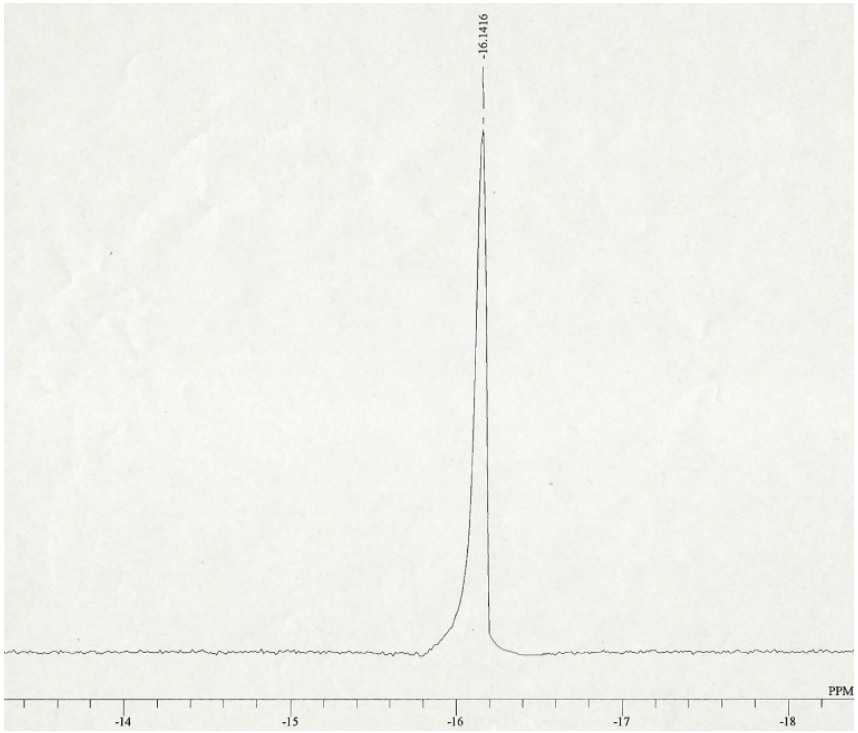

Figure S7: $^{29}Si\{^{1}H\}$ NMR spectrum (THF-$d_{8,}$ INEPT) for compound **6'**

2. CCDC numbers for the two complexes: Crystallographic data for the structures have been deposited in the Cambridge Crystallographic Data Centre, CCDC-1879984 for the complex **6** and CCDC-1879981 for the complex **6'**. Copies of the data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Table S1 Crystallographic data for **6**

Compound	**6**
formula	$C_{32}H_{61}NiP_3Si_4$
formula weight	709.79
crystal system	monoclinic
Space group	P 21/n
a, Å	11.352(2)
b, Å	21.355(4)
c, Å	15.654(3)
α, deg	90.00
β, deg	100.59(3)
γ, deg	90.00
V, Å^3	3730.2(13)
Z	4
T/K	293(2)
D_{calcd}, g cm^{-3}	1.264
F(000)	1528
μ, mm^{-1}	0.798
Index ranges	$-13 \le h \le 14$, $-27 \le k \le 27$, $-20 \le l \le 20$
reflections collected	8580
unique reflections	6690
Data/restraints/parameters	8580/0/380
GOF (F^2)	0.955
R indexes [$I>2\sigma(I)$]	R_1=0.0444 wR_2=0.1083
R (all data)	R_1= 0.0556 wR_2=0.1118

$R_1=\sum||F_o|-|F_c||/\sum|F_o|$; $wR_2=[\sum w(F_o^2-F_c^2)^2/\sum w(F_o^2)^2]^{1/2}$

Table S2 Crystallographic data for **6'**

Compound	**6'**
formula	$C_{26}H_{46}NiP_2Si_4$
formula weight	591.64
crystal system	tetragonal
Space group	P -4c2
a, Å	12.4689(8)
b, Å	12.4689(8)
c, Å	20.552(2)
α, deg	90.00
β, deg	90.00
γ, deg	90.00
V, Å^3	3195.3(4)
Z	4
T/K	100(2)
D_{calcd}, g cm^{-3}	1.230
F(000)	1264
μ, mm^{-1}	0.871
Index ranges	$-16 \le h \le 16$, $-16 \le k \le 16$, $-26 \le l \le 26$
reflections collected	3705
unique reflections	3026
Data/restraints/parameters	3705/0/158
GOF (F^2)	0.891
R indexes [$I>2\sigma(I)$]	R_1=0.0256 wR_2=0.0535
R (all data)	R_1= 0.0326 wR_2=0.0542

$R_1=\sum||F_o|-|F_c||/\sum|F_o|$; $wR_2=[\sum w(F_o^2-F_c^2)^2/\sum w(F_o^2)^2]^{1/2}$

Table S3 Selected bond lengths (Å) and bond angles (º) for **6**

Ni1-P1	2.1792(8)	Ni1-P2	2.1832(7)
Ni1-P3	2.2630(8)	Ni1-Si1	2.2697(8)
Ni1-Si3	2.2789(9)	Si2-Si3	2.3744(10)
Si1-Si4	2.3472(10)	P1-Ni1-P2	87.58(3)
P1-Ni1-P3	100.87(3)	P2-Ni1-P3	111.02(3)
Si1-Ni1-Si3	76.06(4)	P1-Ni1-Si1	89.99(3)
P2-Ni1-Si1	145.13(3)	P3-Ni1-Si1	103.60(3)
P1-Ni1-Si3	152.18(3)	P2-Ni1-Si3	90.43(4)
P3-Ni1-Si3	105.71(3)	Si4-Si1-Ni1	118.45(4)
Si2-Si3-Ni1	119.47(4)	Ni1-P1-C17	105.78(8)
Si1-Si4-C14	110.75(8)	Si3-Si2-C6	108.85(8)

Table S4 Selected bond lengths (Å) and bond angles (º) for **6'**

Ni1-P1	2.1554(6)	Ni1-Si2	2.2440(6)
Si1-Si2A	2.3329(8)	P1-Ni1-P1A	90.19(3)
P1-Ni1-Si2	173.61(2)	P1-Ni1-Si2A	93.50(2)
Si2-Ni1-Si2A	83.32(3)	Si1-Si2A-Ni1	108.15(3)
Ni1-P1-C11	108.01(7)	SiA-Si2-C8	107.36(6)