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Note

New ferrocenyl amine derivatives: *N*-silyl, *N*-stannyl and *N*-boryl ferrocenyl amines

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Abstract

N-Ferrocenyl amine, Fc-NH_2 (**1**), reacts with chlorosilanes in the presence of Et_3N to give *N*-silylated derivatives such as Fc-NH-$SiMe_3$ (**2**), Fc-NH-$SiMe_2H$ (**3**) and $(Fc\text{-}NH)_2SiMe_2$ (**6**). *N*-Lithiation of **1**, followed by reaction with Me_3SnCl, gives Fc-NH-$SnMe_3$ (**4**) which is in equilibrium with Fc-*N*$(SnMe_3)_2$ (**5**). Lithiated **1**, Fc-NH-Li, reacts with Me_2SnCl_2 (1:1) to give the trimer $(Fc\text{-}N\text{-}SnMe_2)_3$ (**7**). The tin analogue of **6** was not found. Lithiation of **2**, followed by reaction with Me_3SnCl, affords Fc-*N*$(SiMe_3)$-$SnMe_3$ (**8**). The silylamine **2** reacts with the 9-borabicyclo[3.3.1]nonane dimer $(H\text{-}9\text{-}BBN)_2$ and with tetraethyldiborane(6), either by elimination of H_2 to give Fc-*N*$(SiMe_3)$-(9-BBN) (**9**) and Fc-*N*$(SiMe_3)$-BEt_2 (**10**), or by elimination of Me_3SiH to give Fc-NH-(9-BBN) (**11**) and Fc-NH-BEt_2 (**12**), respectively, depending on the reaction conditions. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Aminoborane; Aminosilane; Aminostannane; Ferrocene; NMR

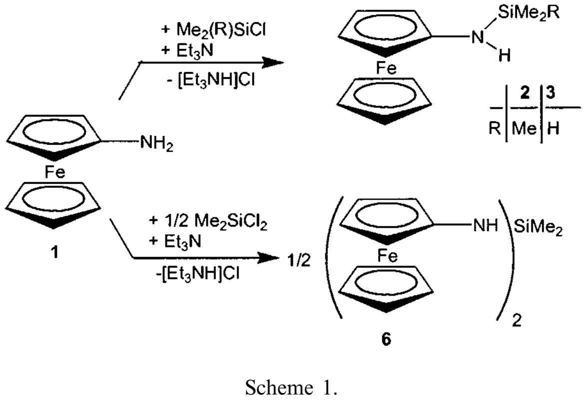
1. Introduction

N-Ferrocenyl amine, Fc-NH_2 **1**, has been known for a long time [1], and its chemistry has been studied in some detail [2,3]. Recently, improved synthetic procedures have been reported for **1** [4], and there is also a recent study on the limited reactivity of *N,N*-diferrocenyl amine [5]. Aiming for a systematic investigation of organometallic *N*-ferrocenyl derivatives, the transformation of **1** into *N*-functionally substituted derivatives was studied. To the best of our knowledge, only a single *N*-silyl derivative has been described, Fc-NH-$SiMe_3$ [3b], and *N*-stannyl- or *N*-boryl-ferrocenyl amines are unknown so far. Considering the well known reactivity of N–Si, N–Sn and N–B bonds for further transformations, we have tried to prepare such *N*-substituted ferrocenyl amines.

2. Results and discussion

2.1. Syntheses

N-Ferrocenyl amine **1** reacts with chloro(trimethyl)-, chloro(dimethyl)- or dichloro(dimethyl)silane in the presence of triethylamine to give the respective *N*-silyl



Scheme 1.

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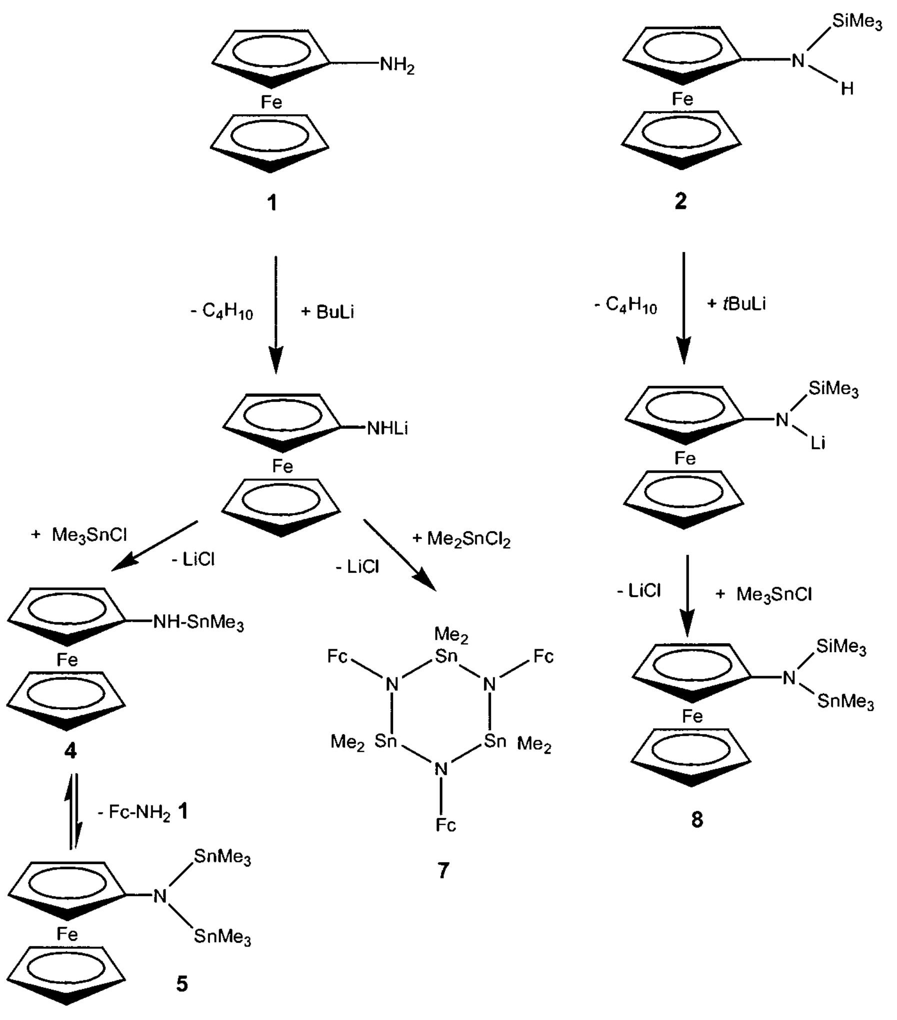
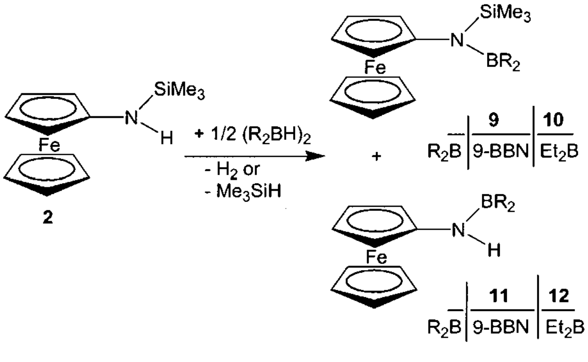


Scheme 2.

derivatives **2**, **3** and **6** in high yield (Scheme 1). The products are isolated as orange, moisture-sensitive solids, soluble in hydrocarbons and chloroform. If Fc-NH-SiMe$_2$H **3** was kept in hexane for a prolonged time (with the intention to isolate crystalline material), complex **1** was obtained as a solid, while the supernatant liquid phase still contained **3**. The formation of another conceivable product, *N*-ferrocenyl-bis(dimethylsilyl) amine, Fc-*N*(SiMe$_2$H)$_2$, was not observed. Thus, a hydrogen from the SiMe$_2$H group in **3** must have been transferred to the nitrogen atom to give **1** and undefined polysilanes.

N-Lithiation of **1** can be conveniently carried out by the reaction with BuLi. In contrast, *N*-lithiation of **2** requires the use of *t*BuLi in order to avoid cleavage of the N–Si bond. Treatment of these *N*-lithio reagents with either Me$_3$SnCl or Me$_2$SnCl$_2$ leads to the *N*-stannyl derivatives **4**, **5**, **7** and **8** (Scheme 2). The tin analogue of **6** was not detected. Compound **4** is obtained almost pure, immediately after the reaction. However, it slowly loses ammonia to give the bis(stannyl) derivative **5**. The same behaviour is known for other secondary *N*-trimethylstannyl amines [6].

Various *N*-trimethylsilyl amines, including Ph-*N*(H)-SiMe$_3$ [7], have been shown to react with tetraalkyldiboranes(6), preferably by cleavage of the N–Si bond and liberation of trimethylsilane [8]. In contrast to the behaviour of most other *N*-trimethylsilyl amines, H$_2$ liber-



Scheme 3.

Table 1
^{11}B-, ^{13}C-, ^{15}N-, ^{29}Si-, and ^{119}Sn-NMR spectroscopic data [a] of **1** to **12**

	δ^{13}C [$^{1}J(^{29}$Si,^{13}C)]	δ^{15}N [$^{1}J(^{29}$Si,^{15}N)] ($^{1}J(^{15}$N,^{1}H))	δ^{11}B	δ^{29}Si/δ^{119}Sn
1	69.0 (Cp), 59.0 (2,5), 63.6 (3,4), 105.2 (1)	−345.7 (72.5)	–	–
2	68.8 (Cp), 57.3 (2,5) 62.6 (3,4), 108.0 (1), 0.2 [57.0] (Me_3Si)	−340.0 [17.0] (78.5)	–	3.2 (Si)
3	69.2 (Cp), 57.8 (2,5), 63.1 (3,4), 107.7 (1), −1.6 [57.1] (Me_2Si)	−344.3 [17.3] (78.2)	–	−12.6 (Si)
4	69.2 (Cp), 56.8 {18.2} (2,5), 62.5 (3,4), 116.0 {6.6} (1), −6.0 {392.0} (Me_3Sn)	−351.3 {17.2} (74.4)	–	50.8 (Sn)
5	69.3 (Cp), 60.1 {29.6} (2,5), 62.4 (3,4), 122.6 {13.4} (1), −2.6 [375.8] (Me_3Sn)	n.m.	–	57.9 {43.8} (Sn)
6	69.3 (Cp), 58.4 (2,5), 63.2 (3,4), 106.6 (1), −0.9 [61.0] (Me_2Si)	−333.6.[21.0] (78.1)	–	−11.0 (Si)
7	69.1 (Cp), 61.4 (2,5), 62.8 (3,4), 113.8 (1), 1.9 (Me_2Sn)	n.m.	–	35.3 {40.8} (Sn)
8	69.2 (Cp), 62.5 (2,5), 62.9 (3,4), 114.3 (1), 3.1 {4.8} [56.2] (Me_3Si), 1.4 {382.4} (Me_3Sn)	n.m.	–	4.2 {19.8} (Si); 56.5 [19.8] (Sn)
9 [b]	69.4 (Cp), 66.5 (2,5), 63.7 (3,4), 106.5 (1), 3.9 [58.0] (Me_3Si)	-284 ± 2 (^{14}N)	+58.5	+8.3 (Si)
10 [c]	69.4 (Cp), 66.8 (2,5), 63.7 (3,4), 106.5 (1), 3.6 [57.8] (Me_3Si)	-285 ± 2 (^{14}N)	+55.7	+8.4 (Si)
11 [d]	69.4 (Cp), 64.5 (2,5), 63.0 (3,4), 101.0 (1)	−280.4 (81.0)	+51.4	–
12	69.4 (Cp), 64.7 (2,5), 63.8 (3,4), 100.8 (1), 13.4 broad, 10.7 broad, 9.3, 9.7 (Et_2B).	−275.7 (81.0)	+49.2	–

[a] The compounds **1**–**3**, **7** were dissolved in $CDCl_3$; compounds **4**, **5**, **6, 8**–**12** in [D_8]toluene or C_6D_6 (there were no appreciable shift differences for these solvents); coupling constants $J(^{119}$Sn,X) { ± 0.5 Hz} (X = ^{13}C, ^{15}N, ^{117}Sn) are given in braces, $^{1}J(^{29}$Si,^{13}C) [± 0.3 Hz], $^{1}J(^{29}$Si,^{15}N) [± 0.1 Hz] in brackets, and $^{1}J(^{15}$N,^{1}H) (± 0.5 Hz) in parentheses.

[b] δ^{13}C(9-BBN) = 25–26 broad (BCH), 33.7 (CH_2) broad due to a dynamic process; 23.1 (CH_2); rotation about the BN bond is slow on the NMR time scale.

[c] δ^{13}C(Et_2B) = 15.2 broad (BCH_2), 9.7 (CH_3).

[d] δ^{13}C(9-BBN) = 26.5–27.5 broad (BCH), 33.9, 33.2 (CH_2), 23.7 (CH_2); rotation about the BN bond is slow on the NMR time scale.

ation is the favoured course of the reactions shown in Scheme 3, leading to the *N*-dialkylboryl-*N*-ferrocenyl-*N*-trimethysilyl amines **9** and **10** and only small amounts (< 20%) of **11** and **12**, respectively. However, when the reaction of **2** with $(9\text{-BBN})_2$ was performed in a highly diluted toluene solution, the alternative route — liberation of Me_3SiH — competes effectively with liberation of H_2, and a mixture (≈ 40:60) of Fc-N($SiMe_3$)-(9-BBN) **9** and Fc-NH-(9-BBN) **11** was obtained. Similarly, when tetraethyldiborane(6) reacts with **3**, the formation of **12** by liberation of Me_2SiH_2 is favoured (> 80%). Compounds **11** and **12** can be prepared independently by the reaction of **1** with the diborane(6) derivatives, as has been described for other amines [9].

2.2. ^{1}H-, ^{11}B-, ^{13}C-, ^{14,15}N-, ^{29}Si- and ^{119}Sn-NMR measurements

The characterisation of the compounds **1**–**12** by routine ^{1}H-, ^{11}B-, ^{13}C- and ^{29}Si-NMR spectroscopy was straightforward (see Table 1 and Section 3). The ^{15}N-NMR spectra of **1**–**4**, **6** and **11**, **12** could be recorded using the INEPT pulse sequence [10] (based on $^{1}J(^{15}$N,^{1}H) with or without ^{1}H decoupling). In the cases of **2**, **3**, and **6**, conditions close to ultrahigh resolution [11] served for the detection of ^{29}Si satellites in the ^{1}H decoupled ^{15}N-NMR spectra. The ^{14}N-NMR spectra of **9** and **10** showed rather broad signals ($h_{1/2} \geq 2000$ Hz); the ferrocenyl group contributes to the increased line width of the ^{14}N-NMR signal by a factor of at least two, as compared to the influence of a phenyl group. The ^{15}N-NMR signals of **9** and **10** are not readily detected because of broadening due to partially relaxed scalar ^{15}N–^{11}B coupling. Therefore, the coupling constants $^{1}J(^{29}$Si,^{15}N) had to be determined from the ^{15}N satellites in the ^{29}Si-NMR spectra. This was achieved by application of the HEED-INEPT pulse sequence [12] which reduces the intensity of the parent signal to some extent and facilitates the assignment of the ^{15}N satellites (Fig. 1).

The ^{15}N-NMR data of **1** indicate that the surroundings of the nitrogen atom are pyramidal. The magnitude of $|^{1}J(^{15}\text{N},^{1}\text{H})| = 72.5$ Hz (in C_6D_6) is relatively small when compared with that in aniline (78 Hz in $CDCl_3$ [13]). The magnitude of $|^{1}J(^{15}\text{N},^{1}\text{H})|$ in **2** (78.1 Hz), **3** (78.5 Hz) and **6** (78.2 Hz) is increased with respect to **1**, indicating that the *N*-silyl group causes a change from pyramidal to trigonal planar geometry at the nitrogen atom. In contrast, the magnitude of $|^{1}J(^{15}\text{N},^{1}\text{H})|$ in Ph-NH-$SiMe_3$ (76.5 Hz [13]) is slightly smaller than in aniline (78 Hz), which is the expected trend if there are no other significant structural changes.

Tin chemical shifts δ^{119}Sn of **4**, **5**, **7**, **8** are in the expected range [14]. The influence of the ferrocenyl group appears to be similar to that of a phenyl group (δ^{119}Sn (Ph-NH-SnMe$_3$) = +46.4, δ^{119}Sn (Ph-*N*(SnMe$_3$)$_2$) = +63.0, and δ^{119}Sn (Ph-*N*(SiMe$_3$)-SnMe$_3$) = +66.0). The value of the coupling constant $^1J(^{119}\text{Sn},^{15}\text{N}) = 74.4$ Hz for **4** is slightly larger than that for **1** but smaller than that for **2**, indicating a pyramidal geometry at the nitrogen atom, somewhat more flat than in **1**. The coupling constant $^1J(^{119}\text{Sn},^{15}\text{N})$ is small and may be of either sign, typical of many trimethylstannyl amines for which in most cases the coupling constants $^1J(^{119}\text{Sn},^{15}\text{N})$ are small and possess a negative sign [14–16].

The deshielding of the nitrogen nuclei in the aminoboranes **9**–**12** is in agreement with analogous effects in other aminoboranes and indicates BN(pp)π interactions [17]. The δ^{11}B values of **11** (51.4) and **12** (49.2) are similar to those of comparable *N*-phenyl aminoboranes (cf. Ph-NH-(9-BBN): δ^{11}B 51.5 [7] and Ph-NH-BMe$_2$: δ^{11}B 48.0 [18]). However, in the cases of **9** (δ^{11}B 58.5) and **10** (δ^{11}B 55.7), the ^{11}B nuclei are somewhat more deshielded (cf. Ph-*N*(SiMe$_3$)-BMe$_2$: δ^{11}B 51.7 [18]).

3. Experimental

3.1. General and starting materials

The starting complex, Fc-NH$_2$ (**1**) [4], and the diborane(6) derivatives [19,20] were prepared according to established procedures; the concentration of hydride in the borane mixture containing tetraethyldiborane(6) [20] was determined by ^{11}B-NMR spectroscopy [21]. The chlorosilanes and -stannanes were used as commercial products without further purification. The syntheses of **2**–**12** were carried out in an atmosphere of argon, observing all precautions to exclude oxygen and moisture. NMR measurements: Bruker ARX 250 and DRX 500 spectrometers, [chemical shifts are given with respect to CHCl$_3$/CDCl$_3$ ($\delta^1\text{H} = 7.24$; $\delta^{13}\text{C} = 77.0$), [D$_8$]toluene ($\delta^1$H(C$_6D_5CD_2$H) 2.03; δ^{13}C(C$_6D_5$*C*D$_3$) 20.4), C$_6D_6$ (δ^1H(C$_6$D$_5$H) 7.14; δ^{13}C 128.0), external Et$_2$O–BF$_3$ in CDCl$_3$ ($\delta^{11}\text{B} = 0$ for $\Xi(^{11}\text{B}) = 32.083971$ MHz), external neat MeNO$_2$ ($\delta^{15}\text{N} = 0$ for $\Xi(^{15}\text{N}) = 10.136767$ MHz; $\delta^{14}\text{N} = 0$ for $\Xi(^{14}\text{N}) = 7.226324$ MHz), external Me$_4$Si ($\delta^{29}\text{Si} = 0$ for $\Xi(^{29}\text{Si}) = 19.867184$ MHz) and external Me$_4$Sn ($\delta^{119}\text{Sn} = 0$ for $\Xi(^{119}\text{Sn}) = 37\,290\,665$ MHz)]. Mass spectra: VARIAN MAT CH7, EI-MS (70 eV), direct inlet.

3.2. N-Ferrocenyl-N-dimethylsilyl amine (3)

N-Ferrocenyl amine **1** (1.0 g, 5 mmol) was dissolved in hexane (100 ml) at room temperature (r.t.), and triethylamine (6 ml) was added to the yellow solution. Then chloro(dimethyl)silane (0.47 g; 5 mmol) was added dropwise as a solution in hexane (10 ml). The reaction mixture was stirred overnight, insoluble material was filtered off, and all volatile material was removed in vacuo. The pure product **3** is left as an orange powder (1.1 g; 85%; m.p. 28 °C).

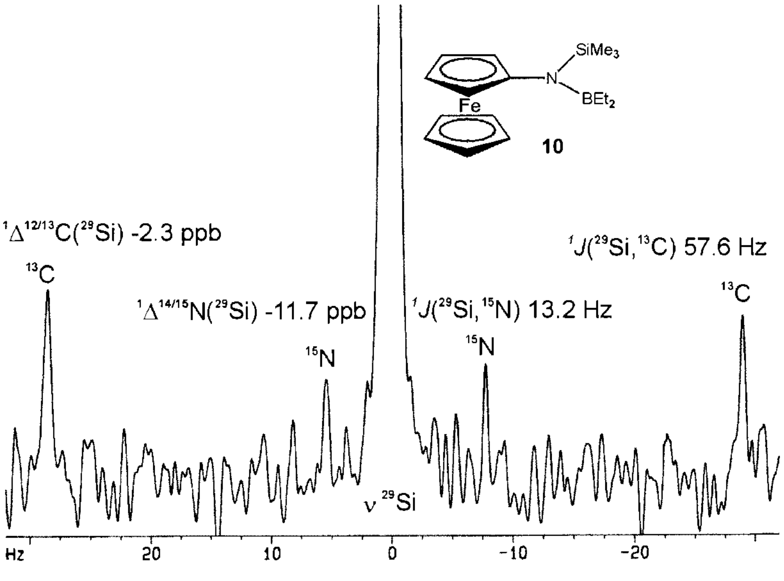


Fig. 1. 99.36 MHz ^{29}Si-NMR spectrum of Fc-*N*(SiMe$_3$)-BEt$_2$ (**10**), recorded by the refocused INEPT-HEED pulse sequence [12] (120 mg in 0.6 ml of [D$_8$]toluene; 20 °C; acquisition time 8 s; repetition time 10 s; 64 transients; Hahn-echo delay 0.4 s). The parent signal is sufficiently suppressed for the unambiguous identification of the ^{15}N satellites. (The ^{13}C satellites, which have the same line widths as the parent signal, are shown for comparison.)

3: ^{1}H-NMR (250 MHz, C_6D_6): δ^1H = 4.68 sp, ^3J(H,H) = 3.1 Hz, 1 H (SiH); 4.07 s, 5 H (Cp); 3.78 m, 4 H (CH(2,5,3,4)); 1.78 s, 1 H (NH); 0.08 d, ^3J(H,H) = 3.1 Hz, 6 H ($SiMe_2$). MS: m/e = 259 (18%, M^+), 201 (100%, Fc-NH_2).

Compound **2** was obtained in the same way (95%; m.p. 66 °C) [3b].

*3.3. Bis(N-ferrocenylamino)dimethylsilane (**6**)*

Dichloro(dimethyl)silane (0.09 g; 0.77 mmol) was injected at r.t. through a syringe into a solution containing both *N*-ferrocenyl amine **1** (0.31 g; 1.54 mmol) and an excess of triethylamine (0.22 g; 2.2 mmol) in hexane (100 ml). The mixture was stirred overnight; insoluble material was filtered off, and after removing of the solvent in vacuo an orange powder was left. Recrystallisation from pentane/ether (10:1) gave the pure product **6** as orange crystals (0.33 g; 93%; m.p. 107 °C). ^{1}H-NMR (250 MHz, C_6D_6): δ^1H = 4.05 s, 10 H (Cp); 3.78 m, 3.84 m, 8 H (CH(2,5,3,4)); 2.14 s, 2 H (NH); 0.14 s, 6 H ($SiMe_2$). MS: m/e = 458 (15%; M^+), 201 (100%, Fc-NH_2^+).

*3.4. N-Ferrocenyl-N-trimethylstannyl amine **4** and N-ferrocenyl-N,N-bis(trimethylstannyl) amine **5***

A solution of BuLi (2 mmol) in hexane (10 ml) was cooled to − 30 °C, and a solution of Fc-NH_2 (**1**) (0.4 g; 2.0 mmol) in hexane/ether (25 ml; 4:1) was added dropwise. The reaction mixture was allowed to reach r.t., and after stirring for 30 min, solid material (Fc-NH-Li) was separated by centrifugation. After decanting the solvent and drying the orange solid for several hours at 10^{-2} Torr it can be used for further transformations.

Trimethyltin chloride (0.37 g; 1.9 mmol) was added at r.t. as a solid to a suspension of Fc-NH-Li (0.39 g; 1.9 mmol) in hexane (100 ml). The mixture was stirred at r.t. for 12 h, solid material was filtered off, and a brown oil remained after removing the solvent in vacuo. The oil was dissolved in hexane, filtrated again, and **4** (0.49 g; 70%) crystallized as a brown solid at − 20 °C.

4: ^{1}H-NMR (250 MHz, C_6D_6): δ^1H = 4.07 s, 5 H (Cp); 3.64 m, 3.79 m (CH(2,5,3,4)); 1.54 s, 1 H (NH); 0.13 s, 9 H ($SnMe_3$), $^2J(^{119}Sn,^1H_{Me})$ = 58.1 Hz. MS: m/e = 364 (8%, M^+), 201 (100%, Fc-NH_2^+).

Samples of **4** lost ammonia both in solution and in the solid state to give **5** which was characterised by NMR spectroscopy.

5: ^{1}H-NMR (250 MHz, C_6D_6): δ^1H = 4.06 s, 5 H (Cp); 3.70 m, 3.71 m (CH(2,5,3,4)); 0.20 s, 18 H ($SnMe_3$), $^2J(^{119}Sn,^1H_{Me})$ = 57.0 Hz. MS: m/e = 527 (5%, M^+), 201 (100%, Fc-NH_2^+).

*3.5. Trimeric N-ferrocenyl-N-dimethylstannyl amine **7***

Solid dimethyltin dichloride (0.26 g; 1.2 mmol) was added at r.t. to a suspension of Fc-NH-Li (0.5 g) in 100 ml of hexane (as described in Section 3.4), and the mixture was stirred for 12 h at r.t. After filtration, the product was extracted from the solid material using CH_2Cl_2. The cyclic trimer **7** was obtained after removing CH_2Cl_2 in vacuo as an orange solid (0.24 g; 80%; m.p. (dec.) above 155 °C).

7: ^{1}H-NMR (250 MHz, C_6D_6): δ^1H = 4.06 s, 15 H (Cp); 3.63 m, 3.77 m, 12 H (CH(2,5,3,4)); 0.71 s, 12 H ($SnMe_2$), $^2J(^{119}Sn,^1H_{Me})$ = 6.14 Hz. MS: m/e = 1044 (59%, M^+), 666 (100%, Fc-NH)$_2$SnMe$_2^+$).

*3.6. N-Ferrocenyl-N-trimethylsilyl-N-trimethylstannyl amine **8***

A solution of Fc-NH-$SiMe_3$ **2** (0.42 g; 1.5 mmol) in hexane (50 ml) was cooled to − 50 °C, and *t*BuLi (1.5 mmol; in pentane) was added slowly through a syringe. After warming to r.t., the suspension was stirred for 12 h, and then solid trimethyltin chloride (0.30 g; 1.5 mmol) was added. The reaction mixture was stirred again for 12 h at r.t., then all insoluble material was filtered off and the solvents removed in vacuo. A brown oil (0.68 g; 94%) remained and was identified as **8** (> 95% pure according to ^{1}H-NMR).

8: ^{1}H-NMR (250 MHz, C_6D_6): δ^1H = 4.06 s, 5 H (Cp); 3.72 m, 3.77 m (CH(2,5,3,4)); 0.09 s, 9 H ($SiMe_3$); 0.29 s, 9 H ($SnMe_3$), $^2J(^{119}Sn,^1H_{Me})$ = 58.8 Hz. MS: m/e = 437 (100%, $M^+ + 1$), 273 (40%, Fc-N(H)SiMe$_3^+$).

*3.7. Mixtures of the N-dialkylboryl-N-ferrocenyl amines (**9**/**11** and **10**/**12**)*

After dissolving *N*-ferrocenyl-*N*-trimethylsilyl amine **2** (160 mg; 0.6 mmol) in [D_8]toluene (0.6 ml), one equivalent of the respective borane was added in one portion at r.t. These mixtures were heated at 80 °C for 12 h. NMR spectra showed the presence of **9** and **11** (≈ 85:15), and of **10** and **12** (≈ 85:15). When the same reaction of **2** with 9-BBN was carried out in 100 ml of toluene, the ratio of **9**:**11** changed to ≈ 40:60. By using **3** instead of **2** in the reaction with tetraethyldiborane(6) in toluene (0.6 ml), dimethylsilane elimination took place, and **12** was formed almost exclusively.

9: ^{1}H-NMR (250.13 MHz, [D_8]toluene): δ^1H = 4.07 s, 5 H (Cp); 4.06 m, 3.78 m, 4 H (CH(2,5,3,4)); 0.37 s, 9 H ($SiMe_3$); 1.40 m, 1.57 m, 14 H (9-BBN).

10: ^{1}H-NMR (250.13 MHz, [D_8]toluene): δ^1H = 3.95 s, 5 H (Cp); 3.78 m, 4 H (CH(2,5,3,4)); 0.20 s, 9H ($SiMe_3$); 1.25 q, ^3J(H,H) = 7.6 Hz and 1.03 t, ^3J(H,H) = 7.6 Hz, 10 H (BEt_2).

11: ^{1}H-NMR (250.13 MHz, [D_8]toluene): δ^1H = 4.95 s, 1 H (NH); 3.96 s, 5 H (Cp); 3.96 m, 3.75 m, 4 H (CH(2,5,3,4)); 1.40 m, 1.57 m, 14 H (9-BBN).

12: ^{1}H-NMR (250.13 MHz, [D$_8$]toluene): δ^1H = 5.16 s, 1 H (NH); 4.01 s, 5 H (Cp); 4.02 m, 3.81 m, 4 H (C(2,5,3,4)); 1.01 m and 0.94 m, 10 H (BEt$_2$).

3.8. *N-Ferrocenyl-N-[9-(9-borabicyclo[3.3.1]nonyl)] amine* ***11***

N-Ferrocenyl amine **1** (0.41 g; 2.0 mmol) was dissolved in toluene (10 ml), and 9-BBN (0.25 g; 2.0 mmol) was added at r.t. This mixture was heated at 80 °C for 48 h. Then the solvent was removed in vacuo, the residue was taken up in hexane, and insoluble material was filtered off. Crystallization from hexane at −78 °C gave the pure product **11** as yellow-orange crystals (0.58 g; 90%; m.p. 64 °C). EI MS: $m/e = 321$ (100%, M$^+$), 309 (40%, M$^+$-BH), 213 (82%, M$^+$-C$_8$H$_{12}$).

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