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Lithiated dimethylaminomethyl ferrocenes and ruthenocenes

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Abstract

Dimethylaminomethylferrocenyl lithium and -ruthenocenyl lithium were generated using tin/lithium exchange reactions. The four different metallocenyl lithium compounds were analysed using NMR spectroscopy. The metallocenyl lithium reagents are useful reagents and have been shown to react with MeOD, $ClSiMe_3$ and DMF to give air-stable derivatives.

Keywords: Lithium; Ferrocene; Ruthenocene; Tin/lithium exchange

1. Introduction

Lithiated ferrocenes and ruthenocenes are useful synthetic reagents in organometallic chemistry. We have recently utilised 2- and 1′-(dimethylaminomethyl)-1-(lithium)-ferrocenides **3a** and **6a** [1] and the corresponding ruthenocenides **3b** and **6b** [2] in the synthesis of organometallic chloroquine analogues (see Scheme 1). A monomeric deprotonating agent seems to be essential for the metallation of ferrocenes to be carried out smoothly and in high yield [3]. The lithiation of *N*,*N*-dimethylaminomethylferrocene (**1a**) [4] is fast (ca. 1 h with *n*-BuLi in diethyl ether) and clean [5]. No X-ray structural analysis has so far been carried out, but the structure probably involves chelation of the carbon-2-bound lithium with the amine nitrogen as related structures show this chelation [6]. In contrast to the lithiation of **1a**, the ruthenium analogue **1b** gives under similar conditions a mixture of 1,2- and 1,1′-(dimethylaminomethyl)-(lithium)-ruthenocenides [2]. This spurred us on to find a methodology to cleanly generate either the 2- or the 1′-(dimethylaminomethyl)-1-(lithium)-metallocenides. Previously, we have isolated polylithiated carbosilanes of the type $Si[(CH_2)_3SiMe_2CH_2Li]_4$ by treating $Si[(CH_2)_3SiMe_2CH_2SnBu_3]_4$ with *n*-BuLi in tetrahydrofuran (thf) at −78 °C; the Bu_4Sn byproduct was removed by successive washes with pentane [7]. We have now adopted this methodology to isolate **3a**, **3b** and **6a**, **6b**, since the ferrocenyl or ruthenocenyl stannanes are readily prepared and the C–Sn bond can be cleaved quantitatively to yield the appropriate lithiated metallocene.

2. Results and discussion

2.1. Isolation of *3a*, *3b* and *6a*, *6b*

Though 2-(dimethylaminomethyl)-1-(lithium)-ferrocenide, **3a**, has previously been isolated [8], it has not been characterised by ^1H and ^{13}C NMR before. We have now used a modified version of the Rausch procedure to isolate **3a** directly from the parent compound *N*,*N*-dimethylaminomethylferrocene, **1a**. In addition we have isolated lithium ferrocenide **3a** from the corresponding stannane **2a**. Interestingly, ferrocenide **3a** precipitated out of solution when commercial samples of *N*,*N*-dimethylaminomethylferrocene **1a** were used but not when "home-made" samples of either **1a** or **2a** were utilised. Since commercial samples of **1a** contain trace quantities of ferrocene (TLC

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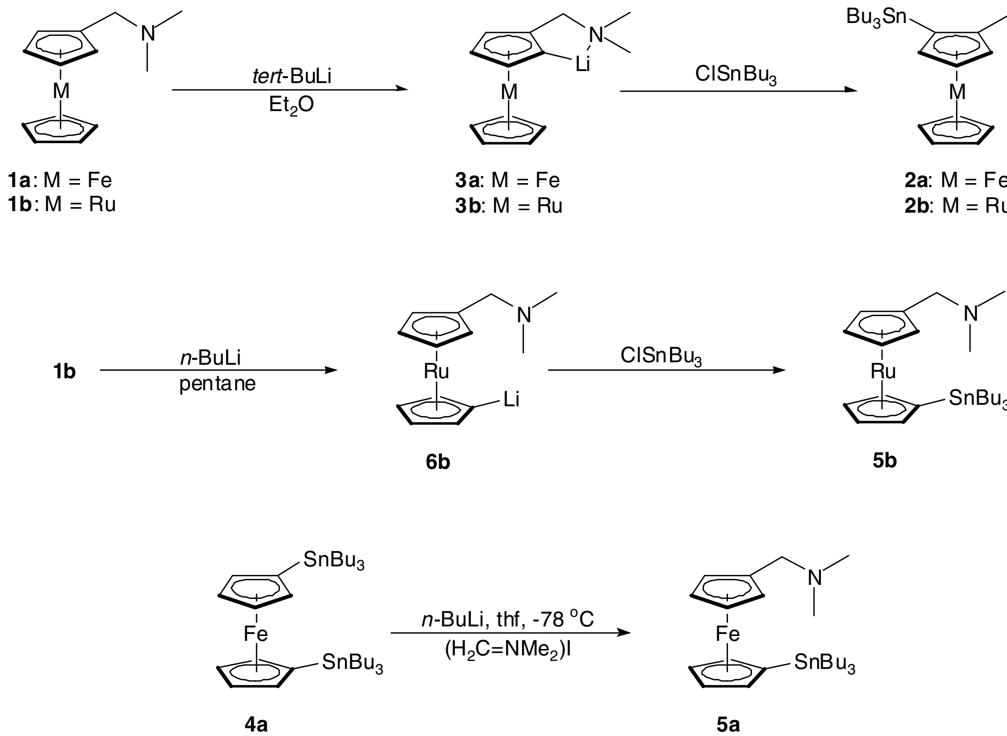


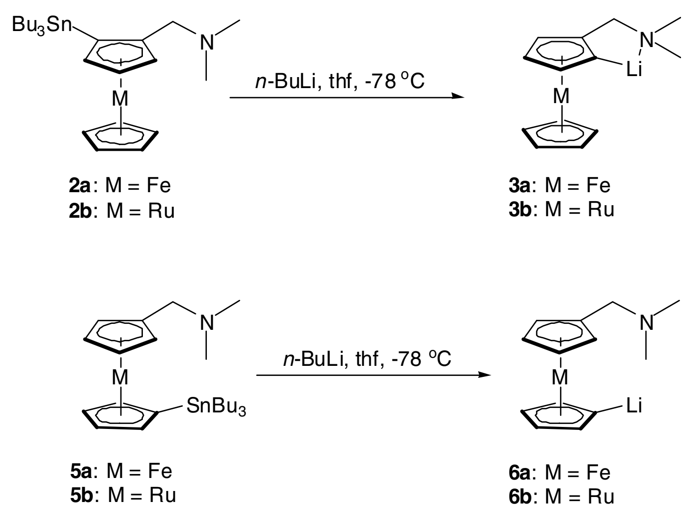
Scheme 1. Synthesis of 2- and 1′-(dimethylaminomethyl)-1-ferrocene and -ruthenocene.

and [1]H NMR), we suspect that lithium-ferrocenide provides a nucleation site for the co-precipitation of **3a**.

The new compounds **3b**, **6a** and **6b** were isolated from the corresponding tri-*n*-butylstannanes **2b**, **5a** and **5b**. The preparations of the tri-*n*-butylstannane starting materials are shown in Scheme 1. The ruthenocene complex **1b** was selectively lithiated and reacted with tri-*n*-butylstannylchloride to give the new compounds **2b** or **5b**, depending on the deprotonation reagent and solvent. Compound **5a** [1,9], was prepared from 1,1′-*bis*(tri-*n*-butylstannyl)ferrocene, **4a** (see Scheme 1) using a tin/lithium exchange reaction using one molar equivalent of *n*-butyllithium, followed by reaction with Eschenmoser's salt.

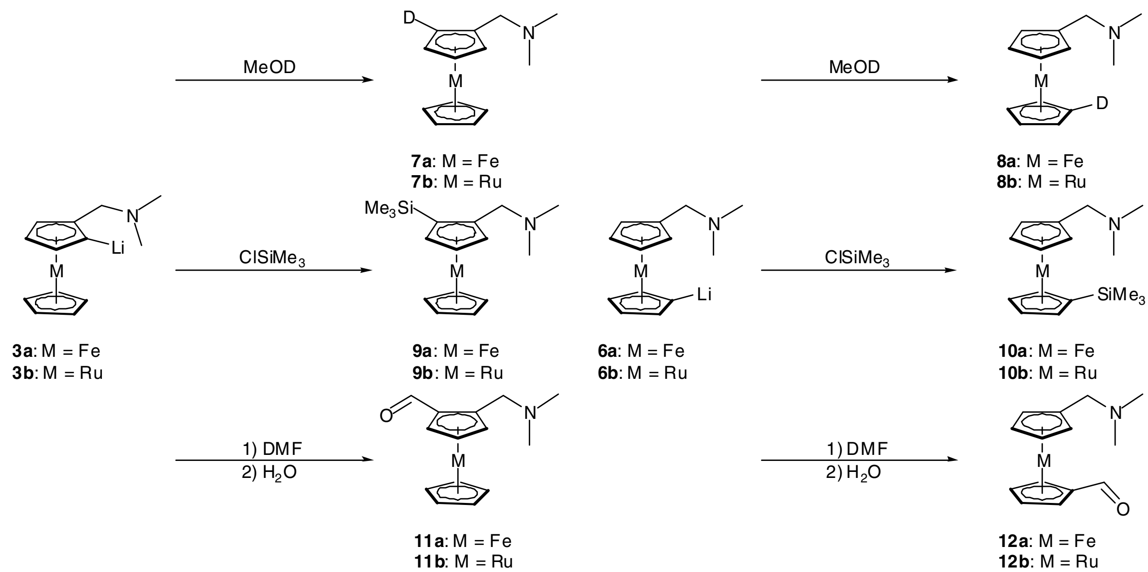
The corresponding 2- and 1′-(dimethylaminomethyl)-1-(lithium)-metallocenides (**3** and **6**) were prepared and isolated from the corresponding tri-*n*-butyltin complexes **2** and **5**. The tin/lithium exchange reaction proceeds fast and quantitative at −78 °C in thf. The reaction had to be performed in the relatively strongly coordinating, and reactive solvent thf, in order to stabilise the penta-substituted stannate that is proposed as an intermediate for this type of reaction [10]. The complexes **6a** and **6b** can be isolated as air- and moisture sensitive solids by successive washes with pentane. The compounds **3a** and **3b** appeared to be soluble in pentane. They could not be separated from the Bu_4Sn byproduct and were analysed as a mixture of **3** and Bu_4Sn. Unfortunately, we were unable to grow crystals suitable for X-ray diffraction analysis (see Scheme 2).

As far as we are aware, the synthesis and characterisation of 1′-(dimethylaminomethyl)-1-(lithium)-ferrocenide (**6a**) and -ruthenocenide (**6b**) have not been reported



Scheme 2. Isolation of 2- and 1′-(dimethylaminomethyl)-1-(lithium) metallocenides of iron and ruthenium.

before. The lithiation of metallocenes using tin/lithium exchange reactions is not a new route [9], but it has not been fully explored yet. The tin/lithium exchange reaction appears to be a powerful tool to functionalise metallocenes and, despite the incorporation of strongly *ortho*-directing groups such as $–CH_2NMe_2$, this functionalisation can be performed at any predetermined position. The presence of the $–CH_2NMe_2$ functionality in the starting material suggests that the here described route might also be successful in the presence of other coordinating functionalities, such as ethers, thioethers, etc.



Scheme 3. Reactions of **3** to form **7**, **9** and **11** and **6** to form **8**, **10** and **12**.

Complete lithiation of the metallocenes **3a**, **3b**, **6a** and **6b** was confirmed by quenching the complex in CH_3OD and analyses of the products by NMR spectroscopy (**7a**, **7b**, **8a** and **8b**). In addition, the lithiated metallocenes were reacted with $ClSiMe_3$ to give the corresponding silanes (**9a**, **9b**, **10a** and **10b**). The lithiated metallocenes were also reacted with DMF to give the corresponding carbaldehydes (after quenching the reaction with H_2O) **11a**, **11b**, **12a** and **12b**. The latter compounds were reported previously (**11a**: [11], **12a**: [1], **11b** and **12b**: [2]), though they were isolated using a different synthetic route. The analytical data of the latter compounds were identical to the previously reported data (see Scheme 3).

3. Conclusions

We describe a new and versatile route to the synthesis and characterisation of lithiated metallocenes of iron and ruthenium. Key to this route is the tin/lithium exchange reaction in tetrahydrofuran. The tin/lithium exchange allows lithiation of metallocenes at predetermined sites, in the presence of the strongly *ortho*-directing dimethylaminomethyl functionality. We expect that this synthetic route will also be successful in the presence of other coordinating groups. Several derivatives of the lithiated metallocenes have been isolated and characterised in high (>90%) yield.

4. Experimental

4.1. General remarks

Lithiated metallocenes are extremely air-sensitive compounds. Manipulations were carried out under purified nitrogen using glovebox (MBraun Unilab) or standard Schlenk-line techniques [12]. Solvents were dried by passage through a column containing alumina (neutral, Brockmann grade I) and distilled from sodium/benzophenone ketyl prior to use [13]. All reagents were stored under argon. All reagents were purchased from Sigma–Aldrich. *N*,*N*-Dimethylaminomethylferrocene [2] and 1′-dimethylaminomethyl-1-tri-*n*-butylstannyl ferrocene [1] (**5a**) were prepared according to the literature methods.

NMR spectra were recorded on either a Varian Unity-400 (^{1}H: 400 MHz; ^{13}C: 100.6 MHz; ^{29}Si: 79.5 MHz) spectrometer or a Varian Mercury-300 (^{1}H: 300 MHz; ^{13}C: 75.5 MHz) spectrometer at ambient temperature. Chemical shifts were referenced to TMS using either the residual proton impurities in the solvent (^{1}H NMR), the solvent resonances (^{13}C NMR) or external TMS (^{29}Si NMR). Infrared spectra were recorded on a Perkin–Elmer Paragon 1000 FT-IR spectrometer in the range 450–4400 cm^{-1}. Spectra were recorded on neat samples between NaCl plates. Mass spectra were determined by Dr. P. Boshoff of the mass spectrometry unit at the Cape Technikon. The selected *m*/*z* values given refer to the isotopes ^{1}H, ^{12}C, ^{14}N, ^{28}Si, ^{102}Ru and ^{120}Sn. In all cases, the isotopic distribution pattern was checked against the theoretical distribution. Elemental analyses were performed using a Carlo Erba EA1108 elemental analyser in the microanalytical laboratory of the University of Cape Town.

4.2. Dimethylaminomethyl-tri-n-butylstannyl metallocenes

4.2.1. 2-Dimethylaminomethyl-1-tri-n-butylstannyl ferrocene (2a)

t-BuLi (4.4 cm^3, of a 1.4 M solution in pentane, 6.2 mmol) was added to a solution of *N*,*N*-dimethylaminomethylferrocene (1 g, 4.1 mmol) in diethyl ether (75 cm^3) and stirred for 0.5 h. *n*-Bu_3SnCl (1.68 cm^3, 6.2 mmol) was added and the mixture was stirred for 3 h. Water (20 cm^3) was added and the organic phase removed, the aqueous phase was washed with diethyl ether

($2 \times 15\ cm^3$), the organic fractions were combined, dried over Na_2SO_4, filtered and the solvent removed in vacuo. The crude product was purified by column chromatography on alumina (Brockman V) eluting with hexane–diethyl ether–triethylamine 70:29:1; as an orange oil (1.94 g, 89%) (Found: C, 56.20; H, 8.02; N, 2.58%. $FeSnC_{25}H_{43}N$ calculated C, 56.42; H, 8.14; N, 2.63%); $\tilde{\nu}_{max}/cm^{-1}$ 3094m, 2955s, 2926s, 2852s, 2812s, 2762s, 1457s, 1375m, 1342m, 1260m, 1174m, 1130m, 1106m, 1069m, 1023s, 1000m, 960m, 845m, 816m, 665m, 597m, 597m, 487m, 458w, 414m neat (NaCl); $\delta_H(C_6D_6$; 400 MHz) 4.15 (1H, m), 4.13 (1H, m), 4.00 (5H, s), 3.99 (1H, m), 3.53 [1H, d, ^2J(HH) 12 Hz], 2.69 [1H, d, ^2J(HH) 12 Hz], 2.01 (6H, s), 1.64–1.75 (6H, m), 1.37–1.48 (6H, m), 1.13–1.19 (6H, m), 0.95 [9H, t, ^3J(HH) 7 Hz]; $\delta_C(C_6D_6$; 100 MHz) 91.0, 80.3 (*ipso*-Cp), 75.4, 72.7, 69.8, 68.8 (Cp), 60.8 (CH_2N), 44.9 (NMe_2), 29.7, [$^3J(CCC^{117/119}Sn)$ 18 Hz], 27.8 [$^2J(CC^{117/119}Sn)$ 59 Hz], 13.8, 10.8 [$^1J(C^{117/119}Sn)$ 349/334 Hz].

4.2.2. 2-Dimethylaminomethyl-1-tri-n-butylstannyl ruthenocene (2b)

Prepared by an analogous method to **2a** and obtained as a yellow oil. Yield: 85%; (Found: C, 52.21; H, 7.79; N, 2.21%. $RuSnC_{25}H_{43}N$ calculated C, 52.00; H, 7.51; N, 2.42%); $\tilde{\nu}_{max}/cm^{-1}$ 3094m, 2950s, 2918s, 2856s, 2848s, 2810, 2761s, 1455s, 1376m, 1341m, 1258m, 1171m, 1128m, 1101m, 1070m, 1020m, 996m, 842m, 805s, 686m, 668m, 593m, 504m, 414m neat (NaCl); $\delta_H(C_6D_6$; 400 MHz) 4.65 (1H, m), 4.55 (1H, m), 4.45 (5H, s), 4.39 (1H, m), 3.36 [1H, d, ^2J(HH) 12 Hz], 2.63 [1H, d, ^2J(HH) 12 Hz], 2.06 (6H, s), 1.55–1.75 (6H, m), 1.30–1.48 (6H, m), 1.10–1.18 (6H, m), 0.94 [9H, t, ^3J(HH) 7 Hz]; $\delta_C(C_6D_6$; 100 MHz) 94.8, 77.2 (*ipso*-Cp), 75.1, 72.0, 70.8 (Cp), 60.6 (CH_2N), 44.9 (NMe_2), 29.7 [$^3J(CCC^{117/119}Sn)$ 18 Hz], 27.8 [$^2J(CC^{117/119}Sn)$ 59 Hz], 13.8, 11.0 [$^1J(C^{117/119}Sn)$ 349/334 Hz]; MS (FAB): *m/z* 578 (23%, $M^+ - H$), 535 (44, $M^+ - NMe_2$), 522 (65, $M^+ - {}^nBu$), 478 (46, $M^+ - {}^nBu - NMe_2$), 407 (52, $M^+ - 3{}^nBu$), 364 (85, $M^+ - 3{}^nBu - NMe_2$), 288 (34, $M^+ - Sn{}^nBu_3$), 244 (100, $M^+ - NMe_2 - Sn{}^nBu_3$).

4.2.3. 1′-Dimethylaminomethyl-1-tri-n-butylstannyl ruthenocene (5b)

n-BuLi (1.9 cm^3, 3 mmol, of a 1.6 M solution in hexanes) was added to a mixture of dimethylaminomethylruthenocene (789 mg, 2.7 mmol) in pentane (50 cm^3) and stirred overnight *n*-Bu_3SnCl (0.82 cm^3, 3 mmol) was added and the mixture was stirred for 3 h. Water (20 cm^3) was added and the organic phase removed, the aqueous phase was washed with diethyl ether ($2 \times 15\ cm^3$), the organic fractions were combined, dried over Na_2SO_4, filtered and the solvent removed in vacuo. The crude product was purified by column chromatography on alumina (Brockman IV) eluting with hexane–diethyl ether–triethylamine 70:29:1; as a light yellow liquid (602 mg, 39%); (Found: C, 52.17; H, 7.45; N, 2.40%. $RuSnC_{25}H_{43}N$ calculated C, 52.00; H, 7.51; N, 2.42%); $\tilde{\nu}_{max}/cm^{-1}$ 3085m, 2953s, 2923s, 2856s, 2850s, 2810s, 2758s, 1457s, 1375m, 1344m, 1257m, 1168m, 1132m, 1018s, 959m, 841m, 806s, 688m, 668m, 592m, 492m, 444m neat (NaCl); $\delta_H(C_6D_6$; 300 MHz) 4.61–4.65 (4H, m), 4.47 (2H, m), 4.40 (2H, m), 3.15 (2H, s), 3.17 (6H, s), 1.56–1.69 (6H, m), 1.31–1.45 (6H, m) 0.90–1.05 (15H, m); $\delta_C(C_6D_6$; 75 MHz) 88.0 (*ipso*-Cp), 77.0, 73.3, 72.8 (Cp), 71.2 (*ipso*-Cp), 70.4 (Cp), 56.6 (CH_2N), 44.9 (NMe_2), 29.6 [$^3J(CCC^{117/119}Sn)$ 20 Hz], 27.8 [$^2J(CCSn^{117/119})$ 58 Hz], 13.9 (Me), 10.9 [$^1J(C^{117/119}Sn)$ 348/332 Hz]; MS (FAB): *m/z* 578 (23%, $M^+ - H$), 535 (75, $M^+ - NMe_2$), 522 (64, $M^+ - {}^nBu$), 478 (37, $M^+ - {}^nBu - NMe_2$), 407 (33, $M^+ - 3{}^nBu$), 364 (79, $M^+ - 3{}^nBu - NMe_2$), 288 (24, $M^+ - Sn{}^nBu_3$), 244 (100, $M^+ - NMe_2 - Sn{}^nBu_3$).

4.3. Dimethylaminomethyl lithium metallocenides

The synthesis of the lithium metallocenides is similar for all compounds. A representative example is given below.

4.3.1. NMR scale synthesis of 1′-dimethylaminomethyl-1-lithiumferrocenide (6a)

A Teflon valved NMR tube was loaded with of 1′-dimethylaminomethyl-1-tri-*n*-butylstannylferrocene (71.1 mg, 0.13 mmol). ^{n}BuLi in hexanes (86 mm^3, 1.6 M, 0.13 mmol) was added, followed by THF (1.0 cm^3). Immediately a yellow precipitate was observed to form. After shaking the NMR tube for several minutes the volatiles were removed in vacuo and the remaining solid was dissolved in C_6D_6. ^1H NMR confirmed the quantitative formation of the 1′-dimethylaminomethyl-1-lithiumferrocenide-THF adduct.

$\delta_H(C_6D_6$, 400 MHz): 4.55 (s, 2H, Cp), 4.17 (s, 2H, Cp), 4.10 (s, 2H, Cp), 3.85 (s, 2H, Cp), 3.55 (m, 4H, α-THF), 2.69 (s, 2H, CH_2N), 2.02 (s, 6H, $2CH_3$), 1.42 (s, 4H, β-THF), 1.52 (m, 8 H), 1.32 (m, 8H) and 0.88 (m, 16H) ($Sn{}^nBu_4$); $\delta_C(C_6D_6$; 75 MHz) 83.6, 80.6 (*ipso*-Cp), 72.1, 67.3, 67.1 (Cp), 58.6 (CH_2N), 47.0 (NMe_2).

4.3.2. NMR scale synthesis of 1′-dimethylaminomethyl-1-lithiumruthenocenide (6b)

A similar procedure to the NMR scale synthesis of **6a** was used. ^1H NMR confirmed the quantitative formation of the 1′-dimethylaminomethyl-1-lithiumruthenocenide-THF adduct.

$\delta_H(C_6D_6$, 400 MHz): 4.84 (s, 2H), 4.57 (m, 2H), 4.48 (s, 2H), 4.38 (s, 2H), 4.25 (s, 1H), 2.5 (m, 4H, α-THF), 2.70 (s, 2H, CH_2N), 2.05 (2, 6H, 2 CH_3), 1.43 (m, 4H, β-THF), 1.49 (m, 8H), 1.29 (m, 8H) and 0.86 (m, 16H) ($Sn{}^nBu_4$); $\delta_C(C_6D_6$; 100 MHz): 82.3, 77.0, 72.6, 71.5, 68.2 (Cp), 67.7 (THF), 57.0 (CH_2N), 46.1 (CH_3), 25.5 (THF), 29.5, 27.6, 13.6, 8.9 ($Sn{}^nBu_4$).

4.4. Reactions of the lithium salts

The NMR scale synthesis of derivatives of the dimethylaminomethyl-1-(lithium)-metallocenide reagents were all performed in a similar method. A representative example is given.

4.4.1. NMR scale synthesis of 1′-dimethylaminomethylferrocene-1-carboxaldehyde

A Teflon valved NMR tube was loaded with of 1′-(dimethylaminomethyl)-1-(tri-*n*-butylstannyl)ferrocene (71.1 mg). *n*-Butyllithium in hexanes (100 m^3, 1.6 M) was added, followed by THF (1.0 cm^3). Immediately a yellow precipitate was observed to form. After shaking the NMR tube for several minutes the volatiles were removed in vacuo and the remaining solid was dissolved in C_6D_6. ^{1}H NMR confirmed the quantitative formation of the 1′-(dimethylaminomethyl)-1-(lithium)ferrocenide-THF adduct.

δ_H(C_6D_6, 400 MHz): 4.55 (s, 2H, Cp), 4.17 (s, 2H, Cp), 4.10 (s, 2H, Cp), 3.85 (s, 2H, Cp), 3.55 (m, 4H, α-THF), 2.69 (s, 2H, CH_2N), 2.02 (s, 6H, 2 CH_3), 1.42 (s, 4H, β-THF), 1.52 (m, 8 H), 1.32 (m, 8H) and 0.88 (m, 16 H) (Sn^nBu_4).

N,*N*-dimethylformamide (30 mm^3) was condensed into the NMR tube and the mixture shaken. ^{1}H NMR revealed quantitative conversion. Water (5 mm^3) was added to the mixture and the yellow mixture turned red immediately. The resulting ^{1}H NMR revealed quantitative conversion of the lithium reagent to the carboxaldehyde. δ_H(C_6D_6, 400 MHz): 9.8 (s, 1H, CHO), 4.43 (s, 2H, Cp), 4.09 (s, 2H, Cp), 3.99 (s, 2H, Cp), 3.83 (s, 2H, Cp) 3.0 (s, 2H, CH_2N), 2.14 (s, 6H, NMe_2). In the mixture, the following compounds were also present: δ: 7.65, 2.38, 1.97 (DMF); δ: 1.55, 1.32, 0.88 (Sn^nBu_4); δ: 3.55, 1.41 (THF); δ; 1.35 (H_2O).

The preparative scale synthesis of derivatives of the dimethylaminomethyl-1-(lithium)-metallocenide reagents were all performed in a similar method. A representative example is given.

4.4.2. Synthesis of 1′-dimethylaminomethylferrocene-1-carboxaldehyde

A solution of *n*-BuLi in hexane (0.65 cm^3, 1.54 M, 1.0 mmol) was added to a solution of 1′-dimethylaminomethyl-1-tri-*n*-butylstannyl ferrocene (0.532 g, 1.00 mmol) in THF (5.0 cm^3) at −78 °C in an H-type Schlenk-tube [14]. The mixture was stirred for 30 min at −78 °C, during which time a precipitate was seen to form. Several drops of BuLi in hexane were added to the other leg of the H-type Schlenk-tube. The volatiles were evaporated in vacuo to leave a red solid and an oil. Pentane (2.0 cm^3) was brought into the vessel on the BuLi and distilled onto the reaction mixture (through this extra drying procedure pure solvents were guaranteed). The mixture was stripped with pentane (3 × 2 cm^3) by distilling the pentane onto the mixture and then evaporating in vacuo. Pentane (10 cm^3) was added and the mixture was washed 3 times with pentane by filtering to the 'butyllithium' leg and distilling the solvent onto the lithiated ferrocene. The volatiles were removed in vacuo to leave a red solid **6a** in the one leg and a red oil ($SnBu_4$ + BuLi + unreacted traces of starting material) in the other. A solution of *N*,*N*-dimethylformamide in THF was added to the **6a** at −78 °C and the mixture was allowed to attain room temperature while stirring. Evaporation of the solvent in vacuo and extraction into pentane gave **11a** in quantitative yield.

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