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A preparation of *N,N*-bis(trimethylsilyl)allenamines

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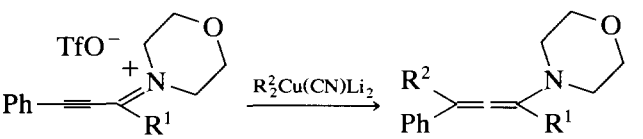
Abstract

The lithium diisopropylamide-induced elimination of {2-[*N,N*-bis(trimethylsilyl)amino]alkenyl}(diethyl)phosphates affords 1-[*N,N*-bis(trimethylsilyl)amino]-1,2-alkadienes ("allenamines"). Conjugatively-substituted allenamines undergo further metalation under these eliminative conditions to form allenyl carbanions which may be trapped in situ by chlorotrimethylsilane to give 3-trimethylsilyl- or 3,3-bis(trimethylsilyl)-*N,N*-bis(trimethylsilyl)allenamines.

Keywords: Silicon; Carbanions; Allene; Silyl; Amine; Trimethylsilyl

1. Introduction

While interesting reactivity patterns are suggested by the allenic and electron-rich molecular format of allenamines (**1**), they constitute a little-explored class of compounds [1]. One reason for this neglect is undoubtably due to the availability of only a few and structurally limited synthetic approaches to these species. Traditional methods have involved base-catalyzed isomerization of tertiary propargylamines [2–6] or metalation-protonation of these substrates [7] (Scheme 1). Recently, a novel approach to morpholinoallenes has been reported by Maas [8] (Eq. 1). For reasons detailed below, our interest focussed on the preparation of *N,N*-bis(trimethylsilyl)allenamines **5**. These allenamines are unique in their incorporation of the bis(TMS)amino function, and chemistry paralleling and expanding that known for bis(trimethylsilyl)enamines [9,10] appeared possible. In addition, if derivatives of these allenamines could be prepared that contained an (sp^3) C–N(TMS)$_2$ bonding arrangement [11], hydrolytic demasking of the *N,N*-bis(TMS)amino function would generate a primary amine which could be utilized for further elaboration.



(1)

2. Results and discussion

As a consequence of some recent investigations [12,13], we had access to the lithium enolates **3**, generated by tandem 1,3(C → N) and 1,4(O → N) silyl group migrations initiated by the addition of organolithium reagents to the O-TMS cyanohydrins (**2**) of acylsilanes (Scheme 2). Since it appeared that **3** could be derivatized by phosphorylation into enol esters **4**, these latter were viewed as candidates for eliminative transformation into allenamines of structure **5**.

In the event, treatment of **3a–i** with diethylchlorophosphite afforded the enol phosphates **4a–i** which, by NMR assay, were cleanly obtained after a simple anhydrous workup. However, these phosphates could not be completely freed from dissolved inorganic

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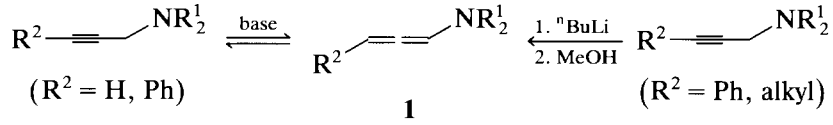
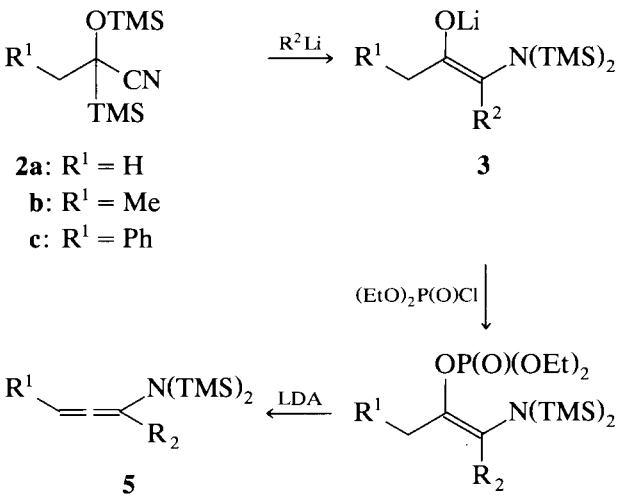


1

Scheme 1.

salts without causing attendant decomposition upon distillation, or partial hydrolysis during chromatography. Nevertheless, rapid chromatography on Florisil afforded analytically pure samples of **4a–i** which were used for characterization purposes. Only one stereoisomer of each **4** was isolated [14], and these have been assumed to possess *Z* geometry by extrapolation from previous considerations relating to the structure of **3** [15]. All ^{13}C NMR spectra of **4** displayed significant ^{31}P-^{13}C ^{2}J and ^{3}J values for the olefinic and ethoxy carbon atoms [16]. For preparative purposes, the crude phosphates **4a–c** were directly treated with LDA at 25°C to afford the corresponding allenamines **5a–c**. These were very hydrolytically labile, but could be obtained pure without significant loss by kugelrohr distillation. The more hindered **4d** was more resistant to elimination, but LDA treatment at 65°C effected conversion to **5d**. Attempted extension of this methodology to alkenyl- or phenyl-substituted substrates was largely unsuccessful, leading to low yields of impure



2a: R^1 = H
b: R^1 = Me
c: R^1 = Ph

3

5

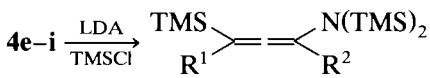
4

3, **4**, **5a**, R^1 = H, R^2 = Me
b, R^1 = H, R^2 = ^{n}Bu
c, R^1 = H, R^2 = cPr
d, R^1 = Me, R^2 = Me
e, R^1 = H, R^2 = CH=CH_2
f, R^1 = H, R^2 = CH(Me)=CH_2
g, R^1 = H, R^2 = CH=CMe_2
h, R^1 = H, R^2 = Ph
i, R^1 = Ph, R^2 = Me

Scheme 2.

allenamines. However, small quantities of **5e**, **5h** and **5i** were isolated by preparative gas chromatography and characterized. The allenic structures of **5** display very weak absorption in the expected IR region (1930–1940 cm^{-1}), but are confirmed by the typical ^{13}C NMR chemical shift distribution which includes the characteristic low-field (∂ 205–215) resonance of the central allenic carbon atom [8b,17].

Pursuant to an assumption that competitive LDA metalation of alkenyl- or phenyl-substituted allene products was occurring during the elimination process owing to conjugative stabilization of the resulting organolithium species, the latter was carried out in the presence of chlorotrimethylsilane (Eq. (2)). This led smoothly to good overall (**2** → **6**) yields of the C-silylated allenamines **6a–e** which proved to be more hydrolytically stable than **5** and could be purified by column chromatography. Although the phenyl-substituted species **6d,e** displayed the very weak IR absorption (1936 and 1926 cm^{-1}, respectively) characteristic of the C-silyl allenamines reported here, this region was dramatically different for the alkenyl-substituted species **6a–c**. For example, comparison of the IR spectra of **5e** with that of its bissilylated analogue **6a** showed that in the latter, the weak band at 1932 cm^{-1} had been replaced by a strong absorption at 1879 cm^{-1}. Changes and similarities between the NMR spectra of **5** and **6** were consistent with expectations.



(2)

6a: R^1 = TMS, R^2 = CH=CH_2
b: R^1 = TMS, R^2 = CH(Me)=CH_2
c: R^1 = TMS, R^2 = CH=CMe_2
d: R^1 = TMS, R^2 = Ph
e: R^1 = Ph, R^2 = Me

3. Experimental details

3.1. General

NMR spectra were obtained at 4.697T using $CDCl_3$ solutions ($CHCl_3$ taken as ∂ 7.24). IR spectra were obtained on neat films. Gas chromatographic (GC) analyses utilized a 2 ft × 0.25 in. 20% SE-30 column which was preconditioned with bis(trimethylsilyl)acet-

amide before allenamine injections. Column chromatography of C-silylated allenamines employed 100-mesh Florisil which was preconditioned with triethylamine (5% in hexane) before use. Kugelrohr distillations reflect oven temperatures and are listed as "Kd °C (torr)". All reactions were carried out under positive argon pressure. THF and ether were distilled from sodium benzophenone ketyl immediately before use. *n*-Butyllithium (^{n}BuLi), 1.6 M in hexane, *tert*-butyllithium (^{t}BuLi), 1.7M in pentane, and methyllithium (MeLi) 1.5 M in ether were obtained from Aldrich Chemical Co. The term "anhydrous workup" consisted of removing volatiles under vacuum (25°C, 1 torr), adding dry pentane, and filtering the suspension through Celite (glass frit) under Ar.

3.2. Preparation of {2-[N,N,-bis(trimethylsilyl)amino]alkenyl} phosphates (4)

3.2.1. {2-[N,N-Bis(trimethylsilyl)amino]-1-methyl-1-propenyl} diethyl phosphate (4a)

This procedure is representative of all preparations of **4**. A solution of **2a** [12] (1.1 g, 5 mmol) in 50 cm^3 of ether at 0°C was treated with MeLi (3.7 cm^3, 5.5 mmol). After 1 h at 25°C, the mixture was cooled to −78°C and diethylchlorophosphate (1.0 g, 5.5 mmol) was added. After 18 h at 25°C, an anhydrous workup gave 2.1 g of crude product used for subsequent elimination. The product from a separate run was chromatographed on Florisil (hexane to 5% ether-hexane) to give a sample for spectral and elemental analysis. Anal. Found: C, 45.62; H, 9.51; N, 3.72. $C_{14}H_{34}NO_4PSi_2$ calc.: C, 45.75; H, 9.32; N, 3.81. ^{1}H NMR: ∂ 0.00 (s, 18H); 1.23 (t, 6H); 1.57 (s, 3H); 1.92 (s, 3H); 4.02 (m, 4H). ^{13}C NMR: ∂ 2.0, 16.0, 16.3 (J = 6.0 Hz), 22.5, 63.6 (J = 6.0 Hz), 125.2 (J = 9.4 Hz), 136.5 (J = 5.8 Hz).

3.2.2. {2-[N,N-Bis(trimethylsilyl)amino]-1-methyl-1-hexenyl} diethyl phosphate (4b)

Anal. Found: C, 49.82; H, 9.84; N, 3.45. $C_{17}H_{40}NO_4PSi_2$ calc.: C, 49.84; H, 9.84; N, 3.42. ^{1}H NMR: ∂ 0.10 (s, 18H); 0.89 (t, 3H); 1.31 (t, 6H); 1.2–1.5 (m, 4H); 1.9–2.1 (m, 2H); 2.01 (s, 3H); 4.11 (m, 4H). ^{13}C NMR: ∂ 2.4, 13.9, 16.1 (J = 7.1 Hz), 16.2, 23.1, 29.1, 34.9, 63.5 (J = 5.5 Hz), 130.2 (J = 9.1 Hz), 135.5 (J = 6.3 Hz).

3.2.3. {2-[N,N-Bis(trimethylsilyl)amino]-2-cyclopropyl-1-methylethenyl} diethyl phosphate (4c)

Cyclopropyllithium was prepared by treating bromocyclopropane in ether at −78°C with 2 equivalents of ^{t}BuLi. After warming to 25°C, the solution was cooled to 0°C, **2a** added, and the general procedure followed. Anal. Found: C, 48.87; H, 9.41; N, 3.41. $C_{16}H_{36}NO_4PSi_2$ calc.: C, 48.82; H, 9.22; N, 3.56. ^{1}H NMR: ∂ 0.08 (s, 18H); 0.47 (m, 2H); 0.63 (m, 2H); 1.32 (t, 6H); cyclopropyl C*H* hidden: 2.16 (s, 3H); 4.12 (m, 4H). ^{13}C NMR: ∂ 1.8, 2.1, 6.4, 15.0, 16.0 (J = 4.0 Hz), 63.5 (J = 6.0 Hz), 128.2 (J = 10.1 Hz), 138.9 (J = 6.1 Hz).

3.2.4. {2-[N,N-Bis(trimethylsilyl)amino]-1-ethyl-1-propenyl} diethyl phosphate (4d)

The preparation of 2-(trimethylsilyl)-2-[(trimethylsilyl)oxy]butanenitrile (**2b**) was carried out following the procedure of Ref. [12]. A solution of 60 mmol of LDA in 150 cm^3 of THF at −78°C was treated dropwise with a solution of 2-[(trimethylsilyl)oxy]butanenitrile (7.85 g, 50 mmol) and chlorotrimethylsilane (6.5 g, 60 mmol) in 20 cm^3 of THF. After 4 h at −78°C, the mixture was stirred at 25°C for 7 days. An anhydrous workup followed by distillation gave 5.3 g (46%) of **2a** admixed with 15% of its *N*-trimethylsilylketenimine isomer, Kd 55–58°C (1 torr). Anal. Found: C, 52.08; H, 10.25; N, 6.18. $C_{10}H_{23}NOSi_2$ calc.: C, 52.34; H, 10.10; N, 6.10. IR 2238 cm^{-1}. ^{1}H NMR: ∂ 0.15 (s, 9H); 0.20 (s, 9H); 1.06 (t, 3H); 1.80 (q, 2H). ^{13}C NMR: ∂ −4.1, 1.5, 9.4, 30.4, 66.0, 121.8.

4d was obtained from **2b** and MeLi by the general procedure. Anal. Found: C, 46.99; H, 9.77; N, 3.94. $C_{15}H_{36}NO_4PSi_2$ calc.: C, 47.21; H, 9.51; N, 3.67. ^{1}H NMR: ∂ 0.01 (s, 18H); 0.98 (t, 3H); 1.22 (t, 6H); 1.58 (s, 3H); 2.30 (q, 2H); 4.02 (m, 4H). ^{13}C NMR: ∂ 2.0, 11.8, 15.9 (J = 7.0 Hz), 22.0, 23.7, 63.4 (J = 5.9 Hz), 125.3 (J = 9.1 Hz), 141.9 (J = 6.1 Hz).

3.2.5. {2-[N,N-Bis(trimethylsilyl)amino]-1-methyl-1,3-butadienyl} diethyl phosphate (4e)

Vinyllithium was prepared by treating vinyl bromide in ether at −120°C with 2.2 equivalents of ^{t}BuLi. After warming to 25°C, the solution was cooled to 0°C, **2a** added, and the general procedure followed. Anal. Found: C, 47.27; H, 8.84; N, 3.83. $C_{15}H_{34}NO_4PSi_2$ calc.: C, 47.46; H, 9.03; N, 3.69. ^{1}H NMR: ∂ 0.09 (s, 18H); 1.33 (t, 6H); 2.08 (s, 3H); 4.14 (m, 4H); 5.05 (d, J = 11 Hz, 1H); 5.23 (d, J = 18 Hz, 1H); 6.86 (dd, J = 11 Hz, 18 Hz, 1H). ^{13}C NMR: ∂ 2.0, 16.0 (J = 6.8 Hz), 17.6, 64.0 (J = 5.9 Hz), 114.8, 132.4 (J = 9.2 Hz), 132.7, 143.6 (J = 9.6 Hz).

3.2.6. {2-[N,N-Bis(trimethylsilyl)amino]-1,3-dimethyl-1,3-butadienyl} diethyl phosphate (4f)

Prepared from 2-bromopropene and ^{t}BuLi as for **4e**. Anal. Found: C, 48.96; H, 9.25; N, 3.85. $C_{16}H_{36}NO_4PSi_2$ calc.: C, 48.82; H, 9.22; N, 3.56. ^{1}H NMR: ∂ 0.06 (s, 18H); 1.28 (t, 6H); 1.71 (s, 3H); 2.08 (s, 3H); 4.09 (m, 4H); 4.82 (s, 1H); 5.03 (s, 1H). ^{13}C NMR: ∂ 1.9, 16.0 (J = 6.7 Hz), 17.7, 20.9, 63.6 (J = 5.5 Hz), 117.8, 132.6 (J = 10.4 Hz), 138.0 (J = 5.6 Hz), 143.3.

3.2.7. {2-[N,N-Bis(trimethylsilyl)amino]-1,4-dimethyl-1,3-pentadienyl} diethyl phosphate (4g)

Prepared from 1-bromo-2-methylpropene and ^{t}BuLi as for **4e**. Anal. Found (best analysis): C, 51.04; H, 9.62; N, 3.21. $C_{17}H_{38}NO_4PSi_2$ calc.: C, 50.09; H, 9.40; N, 3.44. ^{1}H NMR: ∂ 0.03 (s, 18H); 1.28 (t, 6H); 1.56 (s, 3H); 1.69 (s, 3H); 1.86 (s, 3H), 4.10 (m, 4H); 5.19 (s, 1H). ^{13}C NMR: ∂ 2.1, 16.0 (J = 6.8 Hz), 17.7, 20.7, 25.5, 63.6 (J = 5.2 Hz), 125.9, 128.1 (J = 9.4 Hz), 134.8, 137.4 (J = 6.6 Hz).

3.2.8. {2-[N,N-Bis(trimethylsilyl)amino]-1-methyl-2-phenylethenyl} diethyl phosphate (4h)

Prepared from bromobenzene and ^{t}BuLi at −78°C as for **4e**. Anal. Found: C, 52.91; H, 8.49; N, 3.23. $C_{19}H_{36}NO_4PSi_2$ calc.: C, 53.12; H, 8.45; N, 3.26. ^{1}H NMR: ∂ 0.06 (s, 18H); 1.36 (t, 6H); 2.10 (s, 3H); 4.18 (m, 4H); 7.26 (m, 5H). ^{13}C NMR: ∂ 2.1, 16.1 (J = 7.2 Hz), 18.1, 63.8 (J = 5.6 Hz), 127.2, 127.4, 130.2 (J = 10.4 Hz); 132.0, 138.4 (J = 5.9 Hz), 140.4.

3.2.9. {2-[N,N-Bis(trimethylsilyl)amino]-1-(phenylmethyl)-1-propenyl} diethyl phosphate (4i)

Prepared from **2c** [12] and MeLi. Anal. Found: C, 54.23; H, 8.92; N, 3.10. $C_{20}H_{38}NO_4PSi_2$ calc.: C, 54.14; H, 8.63; N, 3.16. ^{1}H NMR: ∂ 0.10 (s, 18H); 1.19 (t, 6H); 1.80 (s, 3H); 3.8 (s, 2H); 3.94 (m, 4H); 7.25 (m, 5H). ^{13}C NMR: ∂ 2.2, 15.8 (J = 7.4 Hz), 22.8, 36.3, 63.4 (J = 5.3 Hz), 126.1, 128.0, 128.3 (J obscured), 128.7, 138.3, 139.2 (J = 6.4 Hz).

3.3. Preparation of 3-[N,N-Bis(trimethylsilyl)amino]-1,2-alkadienes (5)

3.3.1. 3-[N,N-Bis(trimethylsilyl)amino]-1,2-butadiene (5a)

Except as noted, this procedure is representative of all preparations of **5**. A sample of **4a** obtained from 2.2 g (10 mmol) of **2a** after anhydrous workup was added to a solution of LDA in 25 cm^3 of THF prepared from 1.5 g (15 mmol) of diisopropylamine and ^{n}BuLi (15 mmol). After 18 h at 25°C, an anhydrous workup was followed by kugelrohr distillation to give 1.5 g (70% overall) of **5a**, Kd 60–65°C (6 torr). An analytical sample was obtained from preparative GC after conditioning the column with bis(trimethylsilyl)acetamide. Anal. Found: C, 56.46; H, 10.92; N, 6.79. $C_{10}H_{23}NSi_2$ calc.: C, 56.26; H, 10.86; N, 6.56. ^{1}H NMR: ∂ 0.10 (s, 18H); 1.68 (t, J = 3 Hz, 3H); 4.66 (t, J = 3 Hz, 2H). ^{13}C NMR: ∂ 1.9, 24.6, 75.5, 110.0, 210.4. IR: 1940 cm^{-1}.

3.3.2. 3-[N,N-Bis(trimethylsilyl)amino]-1,2-heptadiene (5b)

Yield: 80%, Kd 55°C (0.3 torr). Anal. Found; C, 61.26; H, 11.68; N, 5.54. $C_{13}H_{29}NSi_2$ calc.: C, 61.10; H, 11.44; N, 5.48. ^{1}H NMR: ∂ 0.09 (s, 18H); 0.87 (m, 3H); 1.28 (m, 4H); 1.84 (m, 2H); 4.78 (t, J = 4 Hz, 2H). ^{13}C NMR: ∂ 2.0, 14.1, 22.6, 28.9, 36.9, 77.9, 115.0, 209.7.

3.3.3. 1-[N,N-Bis(trimethylsilyl)amino]-1-cyclopropyl-1,2-propadiene (5c)

Yield: 57%, Kd 50–60°C (0.2 torr). Anal. Found: C, 60.19; H, 10.77; N, 5.96. $C_{12}H_{25}NSi_2$ calc.: C, 60.18; H, 10.52; N, 5.85. ^{1}H NMR (C_6D_6): ∂ 0.33 (s, 18H); 0.62 (m, 4H); 1.0 (m, 1H); 4.80 (d, J = 1 Hz, 2H). ^{13}C NMR (C_6D_6): ∂ 2.8, 8.8, 16.7, 79.9, 118.5, 208.9.

3.3.4. 2-[N,N-Bis(trimethylsilyl)amino]-2,3-pentadiene (5d)

Yield: 72%, Kd 55–60°C (0.1 torr). Anal. Found: C, 57.98; H, 10.81; N, 6.09. $C_{11}H_{25}NSi_2$ calc.: C, 58.08; H, 11.08; N, 6.16. ^{1}H NMR: ∂ 0.10 (s, 18H); 1.61 (d, J = 7 Hz, 3H); 1.67 (d, J = 3 Hz, 3H); 5.01 (m, 1H). ^{13}C NMR: ∂ 1.8, 15.2, 25.1, 85.9, 109.1, 204.8.

3.3.5. 3-[N,N-Bis(trimethylsilyl)amino]-1,2,4-pentatriene (5e)

As in the preparation of **5a**, but starting with **4e**. After kugelrohr distillation (Kd 50–70°C, 1 torr), preparative GC gave **5e**. Anal. Found: C, 58.47; H, 10.49; N, 6.51. $C_{11}H_{23}NSi_2$ calc.: C, 58.60; H, 10.28; N, 6.21. ^{1}H NMR: ∂ 0.09 (s, 18H); 4.92 (broad s, J = 2 Hz, 2H); 4.97 (apparent dq, J = 10 Hz, 2 Hz, 2 Hz, 1H).; 5.28 (apparent dq, J = 17 Hz, 2 Hz, 2 Hz, 1H); 6.22 (dd, J = 17 Hz, 10 Hz, 1H). ^{13}C NMR (CD_2Cl_2): ∂ 1.8, 77.1, 114.0, 115.0, 137.1, 214.6. IR: 1932, 1609, 1571 cm^{-1}.

3.3.6. 1-[N,N-Bis(trimethylsilyl)amino]-1-phenyl-1,2-propadiene (5h)

Only the following spectral data were obtained: ^{1}H NMR: ∂ 0.09 (s, 18H); 5.17 (s, 2H); 7.1–7.5 (m, 5H). ^{13}C NMR: ∂ 1.8, 79.9, 115.5, 126.0, 126.6, 127.9, 134.2, 211.7.

3.3.7. 3-[N,N-Bis(trimethylsilyl)amino]-1-phenyl-1,2-butadiene (5i)

Only the following spectral data were obtained: ^{1}H NMR: ∂ 0.17 (s, 18H); 1.85 (d, J = 3 Hz, 3H); 6.12 (q, J = 3 Hz, 1H); 7.30 (m, 5H). ^{13}C NMR: ∂ 2.0, 25.1, 94.9, 114.1, 126.6, 127.2, 128.4, 135.9, 206.2.

3.4. Preparation of 1-Trimethylsilylated-3-[N,N-Bis(trimethylsilyl)amino]-1,2-alkadienes (6)

3.4.1. 3-[N,N-Bis(trimethylsilyl)amino]-1,1-bis(trimethylsilyl)-1,2,4-pentatriene (6a)

This procedure was used in the preparations of all **6** from the respective precursors **4**. A sample of **4e** prepared from 2.2 g (10 mmol) of **2a** was dissolved in 20 cm^3 of THF and added dropwise to a mixture of LDA (33 mmol) and chlorotrimethylsilane (2.4 g, 22 mmol)

in 30 cm^3 of THF at −78°C. After 18 h at 25°C, an anhydrous workup was followed by chromatography on Florisil preconditioned with 5% triethylamine-hexane to give (5% ether–hexane eluent) 2.64 g (71% overall) of **6a**. Anal. Found: C, 55.32; H, 10.84; N, 4.00. $C_{17}H_{39}NSi_4$ calc.: C, 55.21; H, 10.63; N, 3.79. ^1H NMR: ∂ 0.13 (s, 18H); 0.16 (s, 18H); 4.76 (dd, $J = 10$ Hz, 2 Hz, 1H); 5.05 (dd, $J = 17$ Hz, 2 Hz, 1H); 6.14 (dd, $J = 17$ Hz, 10 Hz, 1H). ^{13}C NMR: ∂ 1.0, 2.6, 95.8, 105.7, 109.7, 138.7, 217.2. IR: 1879, 1609 cm^{-1}.

3.4.2. 3-[N,N-Bis(trimethylsilyl)amino]-4-methyl-1,1-bis(trimethylsilyl)-1,2,4-pentatriene (6b)

Yield: 74%. Anal. Found: C, 56.43; H, 10.92; N, 3.81. $C_{19}H_{43}NSi_4$ calc.: C, 56.32; H, 10.77; N, 3.65. ^1H NMR: ∂ 0.13 (s, 18H); 0.18 (s, 18H); 1.68 (s, 3H); 4.69 (s, 1H); 4.98 (s, 1H). ^{13}C NMR: ∂ 1.1, 2.5, 20.6, 97.3, 108.2, 109.7, 142.6, 215.8. IR: 1875, 1619 cm^{-1}.

3.4.3. 3-[N,N-Bis(trimethylsilyl)amino]-5-methyl-1,1-bis(trimethylsilyl)-1,2,4-hexatriene (6c)

Yield: 73%. Anal. Found: C, 57.22; H, 10.68; N, 3.60. $C_{19}H_{43}NSi_4$ calc.: C, 57.35; H, 10.89; N, 3.52. ^1H NMR: ∂ 0.12 (s, 18H); 0.16 (s, 18H); 1.66 (s 3H); 1.71 (s, 3H); 5.10 (s, 1H). ^{13}C NMR: ∂ 1.0, 2.7, 19.6, 27.3, 96.4, 103.7, 125.8, 128.6, 218.7. IR: 1880 cm^{-1}.

3.4.4. 1-[N,N-Bis(trimethylsilyl)amino]-1-phenyl-3,3-bis(trimethylsilyl)-1,2-propadiene (6d)

Yield: 71%. Anal. Found: C, 59.94; H, 9.95; N, 3.12. $C_{21}H_{41}NSi_4$ calc.: C, 60.07; H, 9.84; N, 3.34. ^1H NMR: ∂ 0.12 (s, 18H); 0.21 (s, 18H); 7.0–7.3 (m, 5H). ^{13}C NMR: ∂ 1.2, 2.5, 99.7, 105.4, 124.1, 124.6, 127.8, 140.7, 214.6. IR: 1876, 1936 cm^{-1}.

3.4.5. 3-[N,N-Bis(trimethylsilyl)amino]-1-phenyl-1-trimethylsilyl-1,2-butadiene (6e)

Yield: 70%. Anal. Found: C, 62.94; H, 9.74; N, 4.05. $C_{19}H_{35}NSi_3$ calc.: C, 63.08; H, 9.75; N, 3.87. ^1H NMR: ∂ 0.02 (s, 18H); 0.17 (s, 9H); 1.83 (s, 3H); 7.1–7.3 (m, 5H). ^{13}C NMR: ∂ 0.0, 2.1, 24.8, 103.7, 106.9, 125.7, 128.0, 128.1, 140.0, 211.5.

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References and notes

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