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Synthesis of new imines and amines containing organosilicon groups

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ABSTRACT

The Peterson olefination reaction of terephthalaldehyde with tris(trimethylsilyl)methyl lithium, $(Me_3Si)_3CLi$, in THF at 0 °C gives 4-[2,2-bis(trimethylsilyl)ethenyl]benzaldehyde (**1**) and 4,4-bis[2,2-bis(trimethylsilyl)ethenyl]benzene (**2**). The new aldehyde (**1**) reacts with variety of amines in ethanol to afford the corresponding imines (**3**) containing vinylbis(trimethylsilyl) group. The newly synthesized imines (**3**) can be completely converted into amines containing vinylbis(trimethylsilyl) group with an excess amount of $NaBH_4$. In the case of N-[4-(2,2-bis(trimethylsilyl)ethenyl)benzyl]-2,6-dimethylaniline $LiAlH_4$ was used as a reducing agent in THF.

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1. Introduction

Vinylsilanes are useful reagents in organic synthesis because the C (sp^2)-Si bonds undergo numerous transformation [1]. Vinylbis(silanes), which show a number of similar properties, have received significant attention as potential synthons in organic and organosilicon synthesis [2]. The potential of the vinylbis(silanes) as precursors for the preparation of ketones and isoxazoline derivatives, as well as a variety of important organosilicon reagents, such as acylsilanes, epoxy silanes, silanols, has stimulated their development. In contrast to vinyl silanes however, vinylbis(silanes) are relatively unexplored [3]. We have recently reported [4] new derivatives of vinylbis(silanes) via Peterson-type olefination reaction. However, no vinylbis(silanes) containing imine and amine group have hitherto been reported.

Imine, also known as azomethine, is formed by the reversible condensation between amine and carbonyl group. This reaction was discovered by the German chemist Hugo Schiff in 1864 and since then imines are also called as Schiff base [5]. An imine is a chemical compound containing carbon–nitrogen double bond with a general structure $R_1R_2C{=}NR_3$ used as a key intermediate for the synthesis of nitrogen hetrocycles [6]. From the fundamental point of view, reduction of imines to the corresponding amines represent one of the most widely used and valuable functional group transformations in synthetic organic chemistry, since amines constitute important precursors to compounds that are of such interest in pharmaceutical and agricultural industries [7,8].

In the present work, we describe the synthesis of some vinylbis(silane) derivatives containing imine and amine groups. The newly synthesized vinylbis(silanes) containing imine and amine group might be used for the preparation of hetrocycles and other organic compounds containing organosilicon group, which cannot be achieved via other methods.

2. Results and discussion

Tris(trimethylsilyl)methane, $(Me_3Si)_3CH$, has been conveniently prepared by the reaction of $CHCl_3$ and Li with Me_3SiCl in THF. The generation of $(Me_3Si)_3CLi$ was easily accomplished via deprotonation of $(Me_3Si)_3CH$ by MeLi in THF [9,10].

The precursor 4-[2,2-bis(trimethylsilyl)ethenyl]benzaldehyde (**1**) was prepared from the reaction of terephthalaldehyde with [(trimethylsilyl)methyl]lithium, $(Me_3Si)_3CLi$, (Scheme 1).

In order to optimize the conditions for the formation of **1**, we decided to investigate the reaction of $(Me_3Si)_3CLi$ (1 equiv.) with various amounts of terephthalaldehyde at different temperatures (Table 1). When 5 equivalent of terephthalaldehyde was used at 0 °C, **1** was the major product.

The newly synthesized 4-[2,2-bis(trimethylsilyl)ethenyl]benzaldehyde (**1**) was treated with an excess of various aromatic amines, the formation of the corresponding imines took place quite smoothly (Scheme 2). The reaction was performed in alcohol (methanol or ethanol) and was monitored via FTIR, actually, the infrared spectra of imines showed the expected absorptions in the

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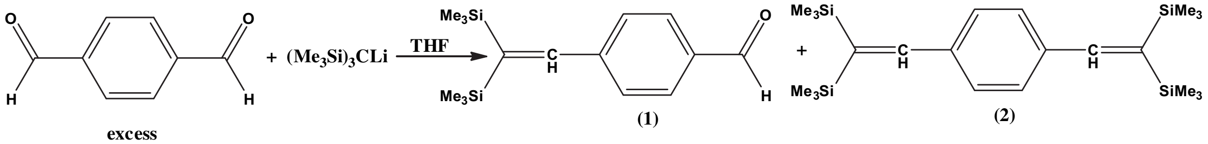
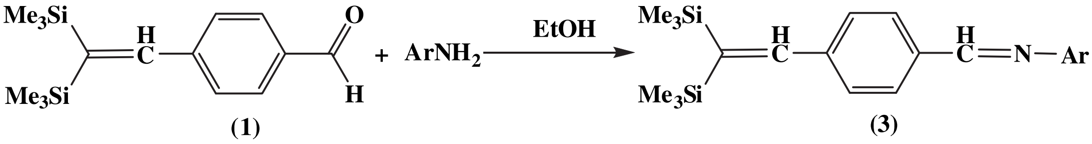


Scheme 1. The preparation of 4-[2,2-bis(trimethylsilyl)ethenyl]benzaldehyde via Peterson protocol.

Table 1
Summary of experimental conditions and yields obtained in the reaction of terephthalaldehyde with $(Me_3Si)_3CLi$.

Entry	Terephthalaldehyde (equiv.)	$(Me_3Si)_3CLi$ (equiv.)	Temperature	Condition	1^a (%)	2^a (%)
1	5	1	r.t	$(Me_3Si)_3CLi$ was added dropwise over terephthalaldehyde and the reaction mixture was mixed for 15 h.	43	45
2	5	1	reflux	$(Me_3Si)_3CLi$ was added quickly over terephthalaldehyde and the reaction mixture was mixed for 15 h.	33	55
3	5	1	r.t	$(Me_3Si)_3CLi$ was added quickly over terephthalaldehyde and the reaction mixture was mixed for 15 h.	56	33
4	5	1	0 °C	$(Me_3Si)_3CLi$ was added quickly over terephthalaldehyde and the reaction mixture was mixed for 30 min.	65	25

[a] Isolated yields.



Scheme 2. The synthesis of imines containing vinylbis(trimethylsilyl) group.

range of 1550–1690 cm^{-1}usually attributed to C=N stretching. In addition, the ^1H NMR spectrum of the raw product of the imines showed a complete disappearance of –CHO at 9.99 ppm.

All the new synthesized imines (Table 2) were identified by GC-Mass method. In order to separate pure imines containing vinylbis (trimethylsilyl) group from the reaction mixture, preparative TLC was used. But in the presence of silica gel, the newly synthesized imines were converted to the precursor **1**.

So, we decided to apply direct reductive amination of imines which are attractive methods for the preparation of amine derivatives in organic synthesis [6].

Sodium borohydride, as a kind of common reducing agent, has been used widely in organic synthesis and there are several reports in the literature for reducing the imines by sodium borohydride [11–13].

We initially studied the reductive amination of the newly synthesized aldehyde (**1**) with aniline in ethanol, which afforded the corresponding imine (Scheme 3) that was then reduced with $NaBH_4$ to give the corresponding amine in 65%, isolated yield.

With the first successful result in hand, reduction of other imines with $NaBH_4$ was carried out under similar reduction conditions. The obtained results are presented in Table 3.

As shown in Table 3, high yield in the reduction of the imines was achieved in the presence of halogen (flourine) functional groups. In the case of 2,6-dimethylaniline, reaction was not completed in the presence of $NaBH_4$, so $LiAlH_4$ was used as the reducing agent in THF.

3. Conclusion

We demonstrated the convenient one-pot preparation of 4-[2,2-bis(trimethylsilyl)ethenyl]benzaldehyde (**1**) via Peterson protocol. When 5-fold the mol ratio of terephthaldehyde was used at 0 °C, **1** was the major product.

The newly synthesized imines containing vinylbis(trimethylsilyl) group were obtained from the reaction of **1** with variety of amines. These imines might be used as intermediate for the synthesis of attractive molecules containing organosilicon group. The reaction of imines containing organosilicon groups with $NaBH_4$ gives the corresponding secondary amines containing organosilicon groups, which cannot be achieved via other methods.

4. Experimental

4.1. Solvent and reagents

Reactions involving organolithium reagents were carried out under dry argon. Solvents were dried by standard methods. Substrates for preparation of $(Me_3Si)_3CLi$, viz., Me_3SiCl, Li, $CHCl_3$ and terephthaldehyde, and also all amines used in synthesis of imines, $NaBH_4$ and $LiAlH_4$ were purchased from Merck and Fluka and used without further purification.

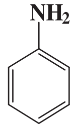
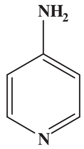
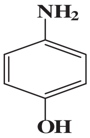
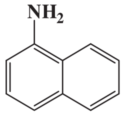
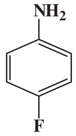
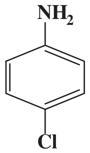
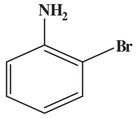
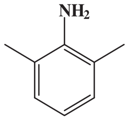
4.2. Spectra

The ^1H NMR and ^{13}C NMR spectra were recorded with a Bruker FT-400 MHz spectrometer at room temperature and with $CDCl_3$ as the solvent. The Mass spectra were obtained with a GC-Mass Agilent, quadrupole model 5973 N instrument, operating at 70 eV. The FTIR spectra were recorded on a Bruker-Tensor 270 spectrometer. Elemental analysis were carried out with an elemental Vario EL III instrument.

4.3. Preparation of tris(trimethylsilyl)methyllithium, $(Me_3Si)_3CLi$, in THF

The reagent was prepared as described by Grobel and coworkers [10].

Table 2
Reaction of 4-[2,2-bis(trimethylsilyl)ethenyl]benzaldehyde with various amines.

Entry	Amine	Condition	Time (h)	Yield[a] (%)
1		Reflux	44	**91**
2		Reflux	85	**70**
3		Reflux	1	**99**
4		r.t	30	**98**
5		r.t	2	**98**
6		r.t	12	**98**
7		r.t	16	**71**
8		Reflux	86	**82**

[a] Yields obtained by GC-Mass.

4.4. *Preparation of 4-[2,2-bis(trimethylsilyl)ethenyl]benzaldehyde*

Tris(trimethylsilyl)methyllithium (22.4 mmol) was poured quickly onto terephthaldehyde 18.7 g (139.4 mmol) in THF at 0 °C under argon and was mixed for 30 min, then the reaction mixture was poured into 200 ml of water and extracted into ether (3×100 ml). The organic layer was dried (Na_2SO_4) and the solvent was evaporated and the residue was purified by preparative column chromatography (n-hexane/ethyl acetate, 10:1, $R_f = 0.7$) to give highly viscose yellow oil (65%). FTIR (KBr, cm^{-1}): 840, 1251 (Si–CH_3), 1558, 1603 (Ph), 1703 (C═O); ^1H NMR (400 MHz, $CDCl_3$, ppm): δ −0.05 (s, 9H, $SiMe_3$), 0.2 (s, 9H, $SiMe_3$), 7.32–7.34 (d, 2H, $J = 7.9$ Hz, Ar), 7.7 (s, 1H, HC =), 7.8–7.82 (d, 2H, $J = 8.7$ Hz, Ar), 9.99 (s, 1H, HC═O); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ −0.6, 0.9 ($SiMe_3$), 127.4, 128.3, 133.9, 148.2, 148.6, 151.9, 190.9; *m*/*z* (EI): 276 (45%, $[M]^+$), 261 (100%, $[M - Me]^+$), 73 (73%, $[SiMe_3]^+$); Anal. Calcd for $C_{15}H_{24}OSi_2$ (276.14): C, 65.15; H, 8.75. Found: C, 65.00; H, 8.61%.

4.5. *General procedure for the synthesis of new imines and amines containing vinylbis(trimethylsilyl) groups*

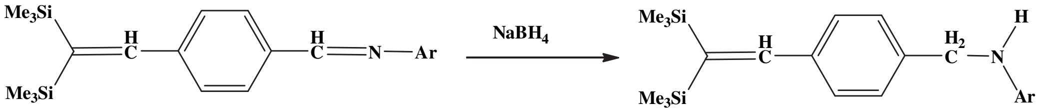
To a 50-ml round-bottom flask were added 0.72 mmol of 4-[2.2-bis(trimethylsilyl)ethenyl]benzaldehyde, 0.86 mmol of amine and 5 ml of ethanol. The mixture was performed according to Table 2. The reaction progress was monitored by FTIR spectroscopy, GC-Mass and TLC. After all the aldehyde precursor was consumed, 2.17 mmol of $NaBH_4$ was added and the mixture was stirred (Table 3) at room temperature. Then the reaction mixture poured into water (50 ml) and extracted into ether (3×15 ml). The organic layer was dried (Na_2SO_4) and the solvent was evaporated and the residue was purified by preparative TLC (n-hexane/ethyl acetate; 10:1) to give the corresponding amines.

4.5.1. *N-[4-(2,2,-bis(trimethylsilyl)ethenyl)benzyl]aniline*

A white solid (silica gel, n-hexane/ethyl acetate, 10:1, $R_f = 0.43$), m.p. 70–72 °C; FTIR (KBr, cm^{-1}): 837.9, 1257.9, (Si–CH_3), 1559.8, 1603.4 (Ph), 3423.1 (N–H); ^1H NMR (400 MHz, $CDCl_3$, ppm): δ −0.04 (s, 9H, $SiMe_3$), 0.18 (s, 9H, $SiMe_3$), 4.02 (bs, 1H, N–H), 4.3 (s, 2H, CH_2), 6.62–6.64 (d, 2H, J = 7.6 Hz, Ar), 6.69–6.73 (t, 1H, $J = 7.3$ Hz, Ar), 7.14–7.19 (m, 4H, Ar), 7.28–7.3(d, 2H, $J = 8.0$ Hz, Ar), 7.7(s, 1H, HC═); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ −0.48, 1.0 ($SiMe_3$), 47.13 (CH_2), 111.8, 116.5, 125.1, 127.1, 128.2, 137.1, 140.7, 145.4, 147.1, 153.5; *m*/*z* (EI): 352 (100%, $[M]^+$), 73 (38%, $[SiMe_3]^+$); Anal. Calcd for $C_{21}H_{31}NSi_2$ (353.2): C, 71.32; H, 8.84; N, 3.96. Found: C, 71.51; H, 8.80; N, 3.75%.

4.5.2. *N-[4-(2,2-bis(trimethylsilyl)ethenyl)]benzylpyridin-2-amine*

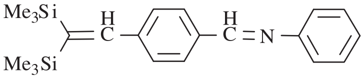
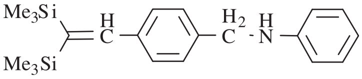
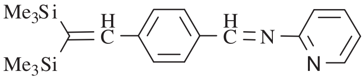
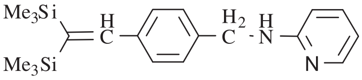
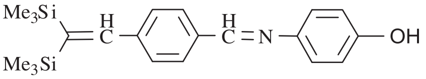
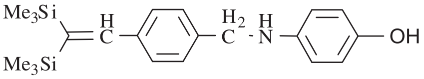
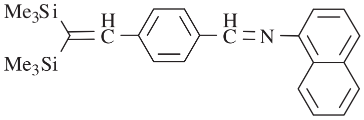
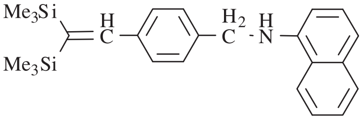
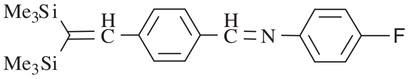
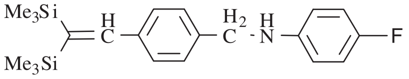
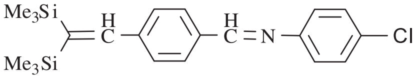
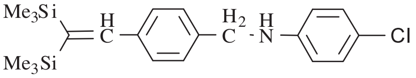
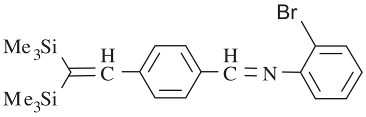
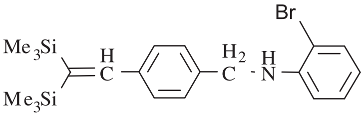
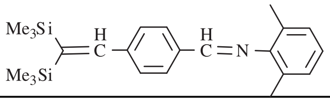
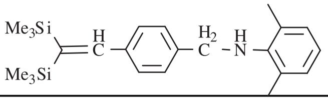
A yellow solid (silica gel, n-hexane/ethyl acetate, 10:1, $R_f = 0.3$), m.p. 75–77 °C; FTIR (KBr, cm^{-1}): 836.8, 1248.8 (Si–CH_3), 1522.8, 1602.8 (Ph), 3420.8 (N–H); ^1H NMR (400 MHz, $CDCl_3$, ppm): δ −0.05 (s, 9H, $SiMe_3$), 0.18 (s, 9H, $SiMe_3$), 4.4 (s, 2H, CH_2), 4.84 (bs, 1H, N–H), 6.35–6.37 (d, 1H, J = 8.4 Hz, Ar), 6.57–6.6 (m, 1H, Ar), 7.13–7.15 (d, 2H, J = 7.8 Hz, Ar), 7.27–7.29 (d, 2H, J = 7.9 Hz, Ar), 7.37–7.42 (m, 1H, Ar), 7.71 (s, 1H, HC═), 8.0–8.1 (m, 1H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ −0.5, 0.99 ($SiMe_3$), 45.1 (CH_2), 105.6,



(3) (4)

Scheme 3. The synthesis of secondary amine containing vinylbis(trimethylsilyl) group.

Table 3
Reducing various imines containing vinylbis(trimethylsilyl) group to the corresponding secondary amine by $NaBH_4$ at room temperature.

Entry	Substrate	Product	Time (h)	Yield[a] (%)
1			15	**65**
2			15	**60**
3			24	**75**
4			12	**80**
5			18	**95**
6			10	**78**
7			18	**67**
8[b]			48	**60**

[a] Isolated yields.
[b] $LiAlH_4$ was used as reducing agent in THF.

112.1, 125.7, 127.1, 136.4, 136.8, 140.8, 145.5, 147.2, 153.4, 157.6; *m/z* (EI): 354 (100%, $[M]^+$), 73 (37%, $[SiMe_3]^+$); Anal. for $C_{20}H_{30}N_2Si_2$ (354.19): C, 67.74; H, 8.53; N, 7.9. Found: C, 67.70; H, 8.31; N, 7.75%.

4.5.3. 4-[4-(2,2-bis(trimethylsilyl)ethenyl)benzylamino]phenol

A yellow solid (silica gel, n-hexane/ethyl acetate, 10:1, $R_f = 0.43$), m.p. 105–107 °C; FTIR (KBr, cm^{-1}): 835.9, 1251.0 (Si–CH_3), 1514.5, 1609.5 (Ph), 3297.9 (O–H), 3434.9 (N–H); ^1H NMR (400, MHz, $CDCl_3$): δ −0.05 (s, 9H, $SiMe_3$), 0.18 (s, 9H, $SiMe_3$), 3.8 (bs, 1H, N–H), 4.27 (s, 2H, CH_2), 5.3 (bs, 1H, OH), 6.53–6.56 (d, 2H, $J = 8.7$ Hz, Ar), 6.68–6.7 (d, 2H, $J = 8.7$ Hz, Ar), 7.13–7.15 (d, 2H, $J = 8$ Hz, Ar), 7.27–7.29 (d, 2H, $J = 7.9$ Hz, Ar), 7.72 (s, 1H, HC=); ^{13}C NMR (100 MHz $CDCl_3$, ppm): δ −0.47, 1.0 ($SiMe_3$), 48.1 (CH_2), 113.3, 115.1, 125.8, 127.1, 138, 140.7, 141.4, 146, 145, 153.5; *m/z* (EI): 369 (100%, $[M]^+$), 91 (39%, $[PhCH_2]^+$), 73 (35%, $[SiMe_3]^+$); Anal. Calcd For $C_{21}H_{31}NOSi_2$ (369.19): C, 68.23; H, 8.45; N, 3.79. Found: C, 68.49; H, 8.22; N, 3.81%.

4.5.4. N-[4-(2,2-bis(trimethylsilyl)ethenyl)benzyl]naphtalen-1-amine

A yellow viscose oil (silica gel, n-hexane/ethyl acetate, 10:1, $R_f = 0.7$); FTIR (KBr, cm^{-1}): 837.9, 1248.8 (Si–CH_3), 1525.6, 1580.9 (Ph), 3444.7 (N–H); ^1H NMR (400 MHz, $CDCl_3$, ppm): δ 0.01 (s, 9H, $SiMe_3$), 0.2(s, 9H, $SiMe_3$), 4.52 (s, 2H, CH_2), 4.73 (bs, 1H, N–H), 6.61–6.63 (d, 2H, $J = 7.4$ Hz, Ar), 7.21–7.23 (d, 2H, $J = 7.9$ Hz, Ar), 7.27–7.29 (d, 1H, $J = 8.0$ Hz, Ar), 7.32–7.36 (t, 1H, $J = 7.4$ Hz, Ar), 7.39–7.41 (d, 2H, $J = 7.9$ Hz, Ar), 7.44–7.51 (m, 2H, Ar), 7.79 (s, 1H, HC=), 7.82–7.86 (t, 1H, $J = 7.7$ Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ −0.46, 1.03 ($SiMe_3$), 47 (CH_2), 103.8, 116.5, 118.8, 122.3, 123.7, 124.7, 125.5, 125.9, 127.2, 127.6, 133.2, 136.8, 140.9, 142.1, 145.5, 1534; *m/z* (EI): 403.4 (100%, $[M]^+$), 73 (22%, $[SiMe_3]^+$); Anal. Calcd for $C_{25}H_{33}NSi_2$ (403.22): C, 74.38; H, 8.24; N, 3.47. Found: C, 74.40; H, 8.5; N, 3.65%.

4.5.5. N-[4-(2,2-bis(trimethylsilyl)ethenyl)benzyl]-4-chloroaniline

A yellow viscose oil (silica gel, n-hexane/ethyl acetate, 10:1, $R_f = 0.56$); FTIR(KBr, cm^{-1}): 838.8, 1249.9 (Si–CH_3), 1501.9, 1601.7 (Ph), 3418.2 (N–H); ^1H NMR (400 MHz, $CDCl_3$, ppm): δ −0.04 (s, 9H, $SiMe_3$), 0.2 (s, 9H, $SiMe_3$), 4.06 (bs, 1H, N–H), 4.3 (s, 2H, CH_2), 6.53–6.55 (d, 2H, $J = 8.7$ Hz, Ar), 7.09–7.1 (d, 2H, $J = 7.9$ Hz, Ar), 7.15–7.17(d, 2H, $J = 8.01$ Hz, Ar), 7.26–7.28 (d, 2H, $J = 7.9$ Hz, Ar) 7.73 (s, 1H, CH=); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ −0.48, 1.0 ($SiMe_3$), 47.1 (CH_2), 112.9, 121.1, 125.7, 127.2, 128.0, 136.6, 140.9, 145.6, 145.7, 153.3; *m/z* (EI): 389.2 (44%, $[M + 2]^+$), 388.2 (34%, $[M + 1]^+$), 387.2

(100%, $[M]^+$), 73 (45%, $[SiMe_3]^+$); Anal. Calcd for $C_{21}H_{30}ClNSi_2$ (387.16): C, 64.99; H, 7.79; N, 3.61. Found: C, 64.97; H, 7.81; N, 3.60%.

4.5.6. N-[4-(2,2-bis(trimethylsilyl)ethenyl)benzyl]-2-bromoaniline

A colorless viscose oil (silica gel, n-hexane/ethyl acetate, 10:1, $R_f = 0.64$); FTIR (KBr, cm^{-1}): 838.6, 1248.3 ($Si{-}CH_3$), 1506.1, 1597.2 (Ph), 3422.4 (N–H); ^1H NMR (400 MHz, $CDCl_3$, ppm): δ 0.005 (s, 9H, $SiMe_3$), 0.24 (s, 9H, $SiMe_3$), 4.4 (s, 2H, CH_2), 4.8 (bs, 1H, N–H), 6.5–6.6 (m, 2H, Ar), 7.11–7.16 (m, 1H, Ar), 7.19–7.21 (d, 2H, $J = 7.9$ Hz, Ar), 7.31–7.33 (d, 2H, $J = 8.0$ Hz, Ar), 7.46–7.48 (q, 2H, Ar), 7.78 (s, 1H, HC=); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ −0.4, 1.0 ($SiMe_3$), 46.8 (CH_2), 108.6, 110.7, 116.9, 125.5, 127.2, 127.4, 131.3, 136.4, 140.8, 143.7, 145.6, 153.4; m/z (EI): 433.1 (100%, $[M+1]^+$), 431.1 (93%, $[M-1]^+$), 73 (52%, $[SiMe_3]^+$); Anal. Calcd for $C_{21}H_{30}BrNSi_2$ (431.11): C, 58.31; H, 6.99; N, 3.24. Found: C, 58.30; H, 7.1; N, 3.50%.

4.5.7. N-[4-(2,2-bis(trimethylsilyl)ethenyl)benzyl]-4-fluoroaniline

A yellow viscose oil (silica gel, n-hexane/ethyl acetate, 10:1, $R_f = 0.59$); FTIR (KBr, cm^{1-}): 839.8, 1250.2 ($Si{-}CH_3$), 1512.8, 1611.4 (Ph), 3427.6 (N–H); ^1H NMR (400 MHz, $CDCl_3$, ppm): δ −0.01 (s, 9H, $SiMe_3$), 0.2 (s, 9H, $SiMe_3$), 3.9 (bs, 1H, N–H), 4.3 (s, 2H, CH_2), 6.55–6.58 (q, 2H, Ar), 6.87–6.91 (t, 2H, $J = 8.6$ Hz, Ar), 7.17–7.19 (d, 2H, $J = 7.9$ Hz, Ar), 7.29–7.31 (d, 2H, $J = 8.0$ Hz, Ar), 7.79 (s, 1H, HC=); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ −0.47, 1.0 ($SiMe_3$), 47.7 (CH_2), 112.7, 114.7, 125.7, 127.2, 136.9, 140.8, 143.4, 145.6, 153.7, 156; m/z (EI): 372.2 (32%, $[M + 1]^+$), 371.2 (100%, $[M]^+$), 73 (31%, $[SiMe_3]^+$); Anal. Calcd for $C_{21}H_{30}FNSi_2$ (371.19): C, 67.87; H, 8.14; N, 3.77. Found: C, 67.98; H, 8.30; N, 3.56%.

4.5.8. N-[4-(2,2-bis(trimetylsilyl)ethenyl)benzyl]-2,6-dimetylaniline

A yellow viscose oil (silica gel, n-hexane/ethyl acetate, 10:1, $R_f = 0.58$); FTIR (KBr, cm^{-1}): 839.5, 1250.8 ($Si{-}CH_3$), 1552.6, 1596.9 (Ph), 3381.7 (N–H); ^1H NMR (400 MHz, $CDCl_3$, ppm): δ 0.03 (s, 9H, $SiMe_3$), 0.25 (s, 9H, $SiMe_3$), 2.3 (s, 6H, Me), 3.2 (bs, 1H, N–H), 4.1 (s, 2H, CH_2), 6.87–6.91 (t, 1H, $J = 7.45$ Hz, Ar), 7.04–7.06 (d, 2H, $J = 7.4$ Hz, Ar), 7.19–7.21 (d, 2H, $J = 7.8$ Hz, Ar), 7.3–7.32 (d, 2H, $J = 8$ Hz, Ar), 7.79 (s, 1H, HC=); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ −0.45, 0.94 ($SiMe_3$), 17.5 (CH_3), 51.65 (CH_2), 121.0, 126.3, 127.0, 127.8, 128.6, 138.2, 140.7, 144.8, 145.4, 153.5; m/z (EI): 381.2 (100%, $[M]^+$), 91 (72%, $[PhCH_2]^+$), 73 (57%, $[SiMe_3]^+$); Anal. Calcd for $C_{23}H_{35}NSi_2$ (381.23): C, 72.37; H, 9.24; N, 3.67. Found: C, 72.18; H, 9.14; N, 3.72%.

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