Synthesis of 2-aryl-1,1-bis(silyl)alkenes containing alkyl(aryl)amine groups

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4-Alkyl-(aryl)aminobenzaldehydes have been generated via the nucleophilic aromatic substitution reaction of 4-fluorobenzaldehyde with appropriated amines and *N*-heterocycles using hexadecyltrimethylammonium bromide as catalyst and converted to 1,1-bis(silyl)-1-alkene derivatives via the Peterson olefination reaction. The reaction of $(HMe_2Si)_3CLi$ with these aromatic aldehydes led to new 2-aryl-1,1-bis(silyl)alkenes.

Keywords: nucleophilic aromatic substitution, hexadecyltrimethylammonium bromide, *N*-heterocycles, 2-aryl-1,1-bis(silyl) alkenes, Peterson olefination reaction

1,1-Disilylated-1-alkenes are useful starting materials in organic synthesis, and their electrophilic chemistry has attracted attention.[1] 2-Aryl-1,1-bis(silyl)alkenes are organometallic compounds that are widely recognised as valuable reagents in organic chemistry for their ability to participate in large number of synthetically useful reactions. They are also stable to many reagents (*e.g.* mild acids, strong bases, hydride reducing agents) and can therefore be carried through a number of steps in a synthetic sequence.[2]

2-Aryl-1,1-bis(silyl)alkenes are precursors for the preparation of ketones and isoxazoline derivatives, as well as a variety of important organosilicon reagents, such as acyl silanes, epoxy silanes and silanols.[3] The low cost, stability and non-toxic nature of many silicon-containing functional groups has increased their application as synthetic intermediates.[4]

We have recently reported the synthesis of new 2-aryl-1,1-bis(silyl)alkene derivatives *via* Peterson olefination reaction.[5–7] In the present work, we describe the synthesis of some 2-aryl-1,1-bis(silyl)alkene derivatives containing an amine group and *N*-heterocycles.

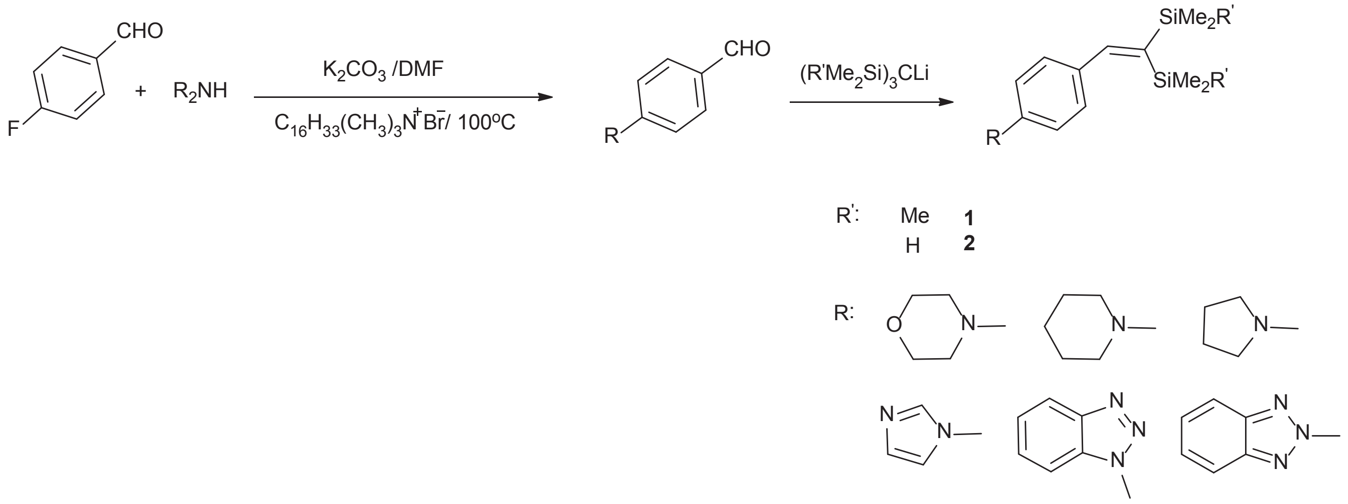
Results and discussion

Recently, we have embarked on a program directed towards the development for the generation of synthetically useful organosilicon compounds.[8,9] We paid particular attention to *N*-aryl amines, specially imidazole and benzotriazoles because of the frequent occurrence of these structural units in biologically active inhibitors.[10–12]

Initially we synthesised 4-alkyl-(aryl)aminobenzaldehydes *via* the nucleophilic aromatic substitution reaction of 4-fluorobenzaldehyde with an appropriate amines and *N*-heterocycles, using *N*,*N*-dimethylformamide (DMF) as the solvent and hexadecyltrimethylammonium bromide as a catalyst. The reaction was performed by heating at 100 °C for 24 hours. Among different *para*-substituted benzaldehydes, 4-fluorobenzaldehyde is the more than the corresponding bromo and chloro analogues.[13] The 4-alkyl-(aryl)aminobenzaldehydes that were synthesised were then reacted with $(Me_3Si)_3CLi$ or $(HMe_2Si)_3CLi$ to afford the title compounds in good overall yields (Scheme 1).

Tris(trimethylsilyl)methane, $(Me_3Si)_3CH$, has been 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.[14] Tris(dimethylsilyl)methane has been prepared from the reaction between $CHBr_3$, Mg and HMe_2SiCl in THF. $(HMe_2Si)_3CLi$ was then metallated by treatment of $(HMe_2Si)_3CH$ with LDA at room temperature.[15]

By using the Peterson olefination reaction, alkoxide intermediate **A** forms when $(Me_3Si)_3CLi$ is treated with a compound containing a carbonyl group. An intramolecular alkoxide attack takes place and then gives **1** (Scheme 2). Thus, synthetically useful 2,2-bis(trimethylsilyl)ethenyl groups can be prepared by the reaction of $(Me_3Si)_3CLi$ with variety of 4-dialkyl(aryl)aminobenzaldehydes (Table 1).



Scheme 1 Synthesis of some 2-aryl-1,1-bis(silyl)alkene derivatives containing amine group and *N*-heterocycles.

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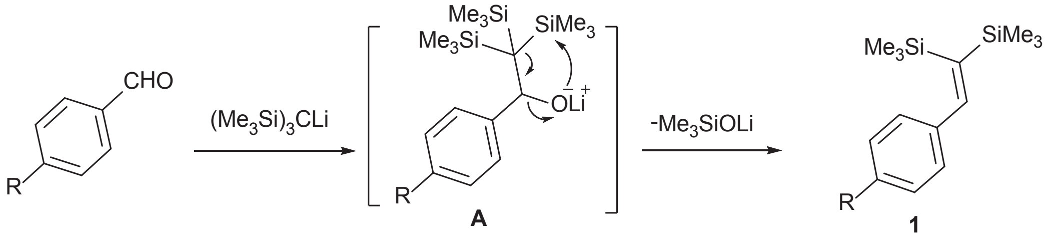


Scheme 2 Synthesis of some 2,2-bis(trimethylsilyl)ethenyl derivatives containing amine group and *N*-heterocycles.

Table 1 The results of reaction of 4-alkyl(aryl)aminobenzaldehydes with (Me$_3$Si)$_3$CLi

R	Product	Time/min	Yield/%[a]
Morpholine-4-yl	**1a**	10	92
Piperidine-1-yl	**1b**	10	93
Pyrrolidine-1-yl	**1c**	10	90
Imidazole-1-yl	**1d**	30	70
Benzotriazole-1-yl	**1e**	15	80
Benzotriazol-2-yl	**1f**	15	85

[a] Isolated yield.

Table 2 The results of reaction of 4-alkyl(aryl)aminobenzaldehydes with (HMe$_2$Si)$_3$CLi

R	product	Time/min	Yield/%[a]
Morpholine-4-yl	**2a**	15	90
Piperidine-1-yl	**2b**	15	88
Pyrrolidine-1-yl	**2c**	15	86
Imidazole-1-yl	**2d**	90	70
Benzotriazole-1-yl	**2e**	30	77
Benzotriazol-2-yl	**2f**	30	73

[a] Isolated yield.

The reaction of (HMe$_2$Si)$_3$CLi and 4-alkyl(aryl) aminobenzaldehydes give the corresponding 2-aryl-1,1-bis(silyl)alkenes (Table 2). We postulated that when (HMe$_2$Si)$_3$CLi reacts with a carbonyl group, it initially forms the alkoxide intermediate **B**, then because of the presence of the Si–H bond, an intramolecular alkoxide attack takes place and gives **2**. It seems likely that the cyclic intermediate **B** is unstable and swiftly fragments to 1,1-bis(silyl)-1-alkene with the elimination of Me$_2$SiO (Scheme 3).

All of the newly synthesised compounds were characterised by spectroscopic techniques. The ^{1}H NMR spectra of the **1a** show the complete disappearance of aldehydic proton resonance and the concomitant appearance of signals assigned to HC=C at 7.68 ppm and –SiMe$_3$ protons at 0.02 and 0.19 ppm. Similar results were observed for **1b**–**f**. The Si–H IR stretching frequency for **2a** was at 2110 cm^{-1}. The ^{1}H NMR spectrum of **2a** showed two doublets at 0.25 and 0.3 ppm assigned to the two methyl groups on each silicon atom, one singlet at 7.72 ppm assigned to a proton on the β-carbon, and one septet in the range 1.72–1.73 ppm for the Si–H protons, resulting from vicinal coupling. All these results clearly show the formation of 2-aryl-1,1-bis(silyl)alkenes **2a**–**f**.

Conclusion

The new 2-aryl-1,1-bis(silyl)alkenes were obtained from the reaction of 4-alkyl(aryl)aminobenzaldehydes with (Me$_3$Si)$_3$CLi or (HMe$_2$Si)$_3$CLi. These compounds are potential intermediates for the functionalisation of *N*-aryl amine which cannot be achieved *via* other methods.

Experimental

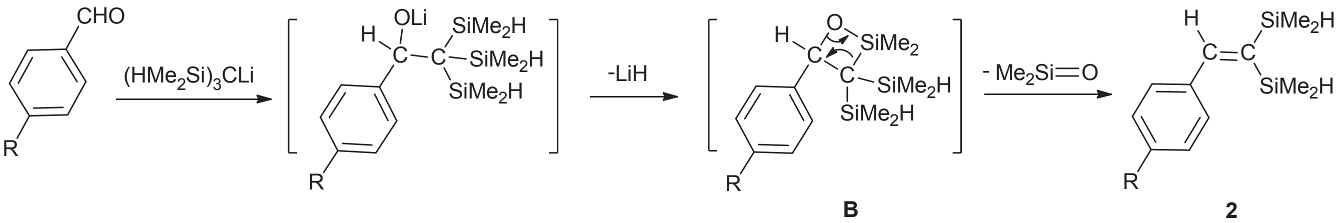
All chemicals were used as purchased and 4-alkyl(aryl) aminobenzaldehydes was synthesised by literature methods. The ^{1}H and ^{13}C NMR spectra were recorded with a Bruker FT-400 MHz spectrometer at room temperature and CDCl$_3$ as a solvent. The FTIR spectra (KBr discs) were recorded on a Bruker-Tensor 270 spectrometer. Elemental analyses (C, H and N) were performed with a Heraeus CHN-O-Rapid analyser. The products were purified by PTLC on silica gel with hexane/ethyl acetate as eluent. All compounds were characterised by spectroscopic data and elemental analysis.

Synthesis of new 2-aryl-1,1-bis(silyl)alkenes containing amine and heterocycles groups; general procedure

The 4-alkyl(aryl)aminobenzaldehyde (1 mmol) was added to (RMe$_2$Si)$_3$CLi, R=H,Me (1 mmol) in THF under argon. The mixture was reacted according to Tables 1 and 2. The reaction was quenched with H$_2$O, extracted with CH$_2$Cl$_2$ and dried over Na$_2$SO$_4$. The solvent was evaporated under reduced pressure and the residue was purified by preparative column chromatography (n-hexane/ethylacetate) and the corresponding products were obtained.

4-(4-(2,2-Bis(trimethylsilyl)vinyl)phenyl)morpholine (**1a**): Yellow oil; yield 92%, (silica gel, n-hexane/ethyl acetate, 10 : 4, R_f=0.5); FTIR (KBr, cm^{-1}): 2847 (HC=), 1244 and 843 (Si–C), 1120 (C–N), 1055 (C–O); ^{1}H NMR (400 MHz, CDCl$_3$): δ 0.02 (s, 9H, SiMe$_3$), 0.19 (s, 9H, SiMe$_3$), 3.17 (t, 4H, J=4.7 Hz, CH$_2$–N–CH$_2$), 3.87 (t, 4H, J=4.7 Hz, CH$_2$–O–CH$_2$), 6.85 (d, 2H, J=8.5 Hz, Ar), 7.14 (d, 2H, J=8.5 Hz, Ar), 7.68 (s, 1H, HC=); ^{13}C NMR (100 MHz, CDCl$_3$): δ –0.36, 1.13 (SiMe$_3$), 48.13, 65.79, 113.56, 128.03, 133.11, 143.11, 149.27, 153.72; *m/z* (EI): 333 (100%, [M]$^+$), 260 (37%, [M–SiMe$_3$]$^+$), 73 (33%, [SiMe$_3$]$^+$). Anal. calcd for C$_{18}$H$_{31}$NOSi$_2$: C, 64.80; H, 9.37; N, 4.20; found: C, 64.87; H, 9.42; N, 4.12%.

4-(4-(2,2-Bis(dimethylsilyl)vinyl)phenyl)morpholine (**2a**): Yellow oil; yield 90%, (silica gel, n-hexane/ethyl acetate, 10 : 4, R_f=0.55); FTIR (KBr, cm^{-1}): 2842 (HC=), 2110 (Si–H), 1245 and 830 (Si–C), 1120 (C–N), 1052 (C–O); ^{1}H NMR (400 MHz, CDCl$_3$): δ 0.25 (d, 6H, J=3.7 Hz, SiMe$_2$), 0.30 (d, 6H, J=3.7 Hz, SiMe$_2$), 3.24 (t, 4H, J=4.7 Hz, 2 CH$_2$–N–CH$_2$), 3.90 (t, 4H, J=4.7 Hz, 2 CH$_2$–C–OH$_2$), 4.29–4.32 (m, 1H, Si–H), 4.39–4.42 (m, 1H, Si–H), 6.90 (d, 2H, J=8.5 Hz, Ar),



Scheme 3 Synthesis of some 2,2-bis(dimethylsilyl)ethenyl derivatives containing amine group and *N*-heterocycles.

7.31 (d, 2H, $J=8.5$ Hz, Ar), 7.72 (s, 1H, HC=); ^{13}C NMR (100 MHz, $CDCl_3$): δ –3.82, –3.26 ($SiMe_2$), 47.76, 65.79, 113.38, 128.71, 131.12, 136.15, 149.56, 154.54; *m/z* (EI): 306 (30%, $[M+1]^+$), 305 (100%, $[M]^+$), 246 (79%, $[M–SiMe_2H]^+$), 59 (18%, $[SiM_2H]^+$). Anal. calcd for $C_{16}H_{27}NOSi_2$: C, 62.89; H, 8.91; N, 4.58; found: C, 62.87; H, 9.06; N, 4.40%.

1-(4-(2,2-Bis(trimethylsilyl)vinyl)phenyl)piperidine (**1b**): Red oil; yield 93%, (silica gel, n-hexane/ethyl acetate, 15 : 3, $R_f=0.64$); FTIR (KBr, cm^{-1}): 2803 (HC=), 1240 and 846 (Si–C), 1127 (C–N); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.06 (s, 9H, $SiMe_3$), 0.22 (s, 9H, $SiMe_3$), 1.61–1.62 (m, 2H, CH_2), 1.73–1.74 (m, 4H, $2CH_2$), 3.2 (t, 4H, $J=4.9$ Hz, CH_2–N–CH_2), 6.9 (d, 2H, $J=8.2$ Hz, Ar), 7.14 (d, 2H, $J=8.2$ Hz, Ar), 7.71 (s, 1H, HC=); ^{13}C NMR (100 MHz, $CDCl_3$): δ –0.34, 1.15 ($SiMe_3$), 23.24, 24.62, 49.44, 114.34, 114.95, 126.17, 127.96, 142.26, 154.01; *m/z* (EI): 332 (31%, $[M+1]^+$), 331 (100%, $[M]^+$),258 (18%, $[M–SiMe_3]^+$), 73 (19%, $[SiMe_3]^+$). Anal. calcd for $C_{19}H_{33}NSi_2$: C, 68.81; H, 10.03; N, 4.22; found: C, 68.93; H, 10.16; N, 4.27%.

1-(4-(2,2-Bis(dimethylsilyl)vinyl)phenyl)piperidine (**2b**): Red oil; yield 88%, (silica gel, n-hexane/ethyl acetate, 15 : 3, $R_f=0.64$); FTIR (KBr, cm^{-1}): 2801 (HC=), 2110 (Si–H), 1235 and 891 (Si–C), 1126 (C–N); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.13 (d, 6H, $J=3.7$ Hz, $SiMe_2$), 0.16 (d, 6H, $J=3.7$ Hz, $SiMe_2$), 1.51–1.54 (m, 2H, CH_2), 1.60–1.65 (m, 4H, $2CH_2$), 3.13 (t, 4H, $J=5.4$ Hz, CH_2–N–CH_2), 4.16–4.19 (m, 1H, Si–H), 4.27–4.31 (m, 1H, Si–H), 6.80 (d, 2H, $J=8.7$ Hz, Ar), 7.16 (d, 2H, $J=8.7$ Hz, Ar), 7.57 (s, 1H, HC=); ^{13}C NMR (100 MHz, $CDCl_3$): δ –3.77, –3.25 ($SiMe_2$), 23.27, 24.63, 48.91, 113.93, 128.69, 130.0, 134.81, 150.41, 154.88; *m/z* (EI): 304 (27%, $[M+1]^+$), 303 (94%, $[M]^+$), 244 (100%, $[M–SiMe_2H]^+$), 59 (18%, $[SiM_2H]^+$). Anal. calcd for $C_{17}H_{29}NSi_2$: C, 67.26; H, 9.63; N, 4.61; found: C, 67.31; H, 9.97; N, 4.37%.

1-(4-(2,2-Bis(trimethylsilyl)vinyl)phenyl)pyrrolidine (**1c**): Red oil; yield 90%, (silica gel, n-hexane/ethyl acetate, 15 : 3, $R_f=0.63$); FTIR (KBr, cm^{-1}): 2803 (HC=), 1245 and 837 (Si–C), 1126 (C–N); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.03 (s, 9H, $SiMe_3$), 0.19 (s, 9H, $SiMe_3$), 1.72 (m, 4H, $2CH_2$), 3.18 (d, 4H, $J=4.5$ Hz, CH_2–N–CH_2), 6.88 (d, 2H, $J=8.1$ Hz, Ar), 7.12 (d, 2H, $J=8.1$ Hz, Ar), 7.68 (s, 1H, HC=); ^{13}C NMR (100 MHz, $CDCl_3$): δ –0.32, 1.13 ($SiMe_3$), 24.62, 49.26, 114.33, 124.07, 125.11, 127.93, 148.01, 154.20; *m/z* (EI): 317 (100%, $[M]^+$), 244 (32%, $[M–SiMe_3]^+$), 73 (24%, $[SiMe_3]^+$). Anal. calcd for $C_{18}H_{31}NSi_2$: C, 68.07; H, 9.84; N, 4.41; found: C, 68.15; H, 9.87; N, 4.32%.

1-(4-(2,2-Bis(dimethylsilyl)vinyl)phenyl)pyrrolidine (**2c**): Red oil; yield 86%, (silica gel, n-hexane/ethyl acetate, 15 : 3, $R_f=0.62$); FTIR (KBr, cm^{-1}): 2808 (HC=), 2107 (Si–H), 1245 and 891 (Si–C), 1128 (C–N); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.28 (d, 6H, $J=3.7$ Hz, $SiMe_2$), 0.3 (d, 6H, $J=3.7$ Hz, $SiMe_2$), 1.72–1.73 (m, 4H, $2CH_2$), 3.90 (t, 4H, $J=5.4$ Hz, CH_2–N–CH_2), 4.29–4.33 (m, 1H, Si–H), 4.39–4.43 (m, 1H, Si–H), 6.90 (d, 2H, $J=8.5$ Hz, Ar), 7.14 (d, 2H, $J=8.5$ Hz, Ar), 7.46 (s, 1H, HC=); ^{13}C NMR (100 MHz, $CDCl_3$): δ –3.90, –3.28 ($SiMe_2$), 29.79, 47.75, 113.36, 129.0, 131.12, 136.15, 150.47, 154.63; *m/z* (EI): 289 (100%, $[M]^+$), 230 (76%, $[M–SiMe_2H]^+$), 59 (18%, $[SiM_2H]^+$). Anal. calcd for $C_{16}H_{27}NSi_2$: C, 66.37; H, 9.40; N, 4.84; found: C, 66.40; H, 9.48; N, 4.77%.

1-(4-(2,2-Bis(trimethylsilyl(vinyl)phenyl)-1H-imidazole (**1d**): Yellow oil, yield 70%, (silica gel, n-hexane/ethyl acetate, 10 : 15, $R_f=0.44$); FTIR (KBr, cm^{-1}): 2851 (HC=), 1255 and 838 (Si–C), 1115 (C–N); ^{1}H NMR (400 MHz, $CDCl_3$): δ –0.02 (s, 9H, $SiMe_3$), 0.21 (s, 9H, $SiMe_3$), 7.29–7.35 (m, 6H), 7.71 (s, 1H, HC=), 8.12 (s, 1H, N–CH=N); ^{13}C NMR (100 MHz, $CDCl_3$); δ –0.56, 1.01 ($SiMe_3$), 109.39, 119.83, 128.49, 130.08, 131.13, 136.83, 146.34, 148.56, 153.88; *m/z* (EI): 314 (87%, $[M]^+$), 313 (100%, $[M–1]^+$), 73 (63%, $[SiMe_3]^+$). Anal. calcd for $C_{17}H_{26}N_2Si_2$: C, 64.91; H, 8.33; N, 8.91; found: C, 65.05; H, 8.24; N, 8.84%.

1-(4-(2,2-Bis(dimethylsilyl(vinyl)phenyl)-1H-imidazole (**2d**): Red oil, yield 70%, (silica gel, n-hexane/ethyl acetate, 15 : 10, $R_f=0.37$); FTIR (KBr, cm^{-1}): 2836 (HC=), 2113 (Si–H), 1255 and 832 (Si–C), 1107 (C–N); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.18 (d, 6H, $J=3.4$ Hz, $SiMe_2$), 0.28 (d, 6H, $J=3.4$ Hz, $SiMe_2$), 4.26–4.28 (m, 2H, Si–H), 7.22 (bs, 1H), 7.31 (bs, 1H), 7.36 (d, 2H, $J=7.4$ Hz, Ar), 7.49 (d, 2H, $J=7.4$ Hz, Ar), 7.75 (s, 1H, CH=), 7.90 (s, 1H, N–CH=N); ^{13}C NMR (100 MHz, $CDCl_3$): δ –4.01, –3.38 ($SiMe_2$), 112.60, 119.70, 127.88, 128.83, 131.15, 137.89, 146.61, 149.92, 153.00; *m/z* (EI): 286 (53%, $[M]^+$), 227 (100%, $[M–SiMe_2H]^+$), 59 (17%, $[SiM_2H]^+$). Anal. calcd for $C_{15}H_{22}N_2Si_2$: C, 62.88; H, 7.74; N, 9.78; found: C, 63.00; H, 7.54; N, 9.72%.

1-(4-(2,2-Bis(trimethylsilyl)vinyl)phenyl)-1H-benzo[d][1,2,3] triazole (**1e**): Red oil, yield 80%, (silica gel, n-hexane/ethyl acetate, 15 : 3, $R_f=0.38$); FTIR (KBr, cm^{-1}): 2842 (HC=), 1251and 838 (Si–C), 1029 (C–N); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.01 (s, 9H, $SiMe_3$), 0.23 (s, 9H, $SiMe_3$), 7.41–7.46 (m, 3H, Ar), 7.56 (t, 1H, $J=7.6$ Hz, Ar), 7.73–7.78 (m, 4H), 8.15 (d, 1H, $J=8.3$ Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ –0.57, 0.99 ($SiMe_3$), 109.32, 119.23, 120.98, 123.32, 127.16, 128.23, 131.14, 134.61, 142.13, 145.40, 147.62, 151.90; *m/z* (EI): 366 (64%, $[M+1]^+$), 365 (84%, $[M]^+$), 73 (100%, $[SiMe_3]^+$). Anal. calcd for $C_{20}H_{27}N_3Si_2$: C, 65.70; H, 7.44; N, 11.49; found: C, 65.96; H, 7.53; N, 11.21%.

1-(4-(2,2-Bis(dimethylsilyl)vinyl)phenyl)-1H-benzo[d][1,2,3]triazole (**2e**): Yellow solid, yield 77%, (silica gel, n-hexane/ethyl acetate, 15 : 4, $R_f=0.52$, m.p. 82–84 °C); FTIR (KBr, cm^{-1}): 2847 (HC=), 2113 (Si–H), 1250 and 894 (Si–C), 1112 (C–N); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.21 (d, 6H, $J=3.5$ Hz, $SiMe_2$), 0.29 (d, 6H, $J=3.5$ Hz, $SiMe_2$), 4.29–4.33 (m, 2H, Si–H), 7.43 (t, 1H, $J=7.6$ Hz, Ar), 7.51–7.57 (m, 3H, Ar), 7.77 (d, 3H, $J=8.2$ Hz, Ar), 7.81 (s, 1H, HC=), 8.14 (d, 1H, $J=8.2$ Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ –4.02, –3.38 ($SiMe_2$), 109.39, 119.34, 121.06, 123.39, 127.24, 128.67, 131.159, 135.06, 140.00, 142.67, 145.52, 153.08; *m/z* (EI): 338 (76%, $[M+1]^+$), 337 (66%, $[M]^+$), 278 (100%, $[M–SiMe_2H]^+$), 59 (29%, $[SiM_2H]^+$). Anal. calcd for $C_{18}H_{23}N_3Si_2$: C, 64.04; H, 6.87; N, 12.45; found: C, 64.32; H, 7.08; N, 12.22%.

2-(4-(2,2-Bis(trimethylsilyl)vinyl)phenyl)-2H-benzo[d][1,2,3] triazoles (**1f**): White solid, yield 85%, (silica gel, n-hexane/ethyl acetate, 15 : 3, $R_f=0.5$, m.p. 102–104 °C); FTIR (KBr, cm^{-1}): 2853 (HC=), 1248 and 839 (Si–C), 1111 (C–N); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.015 (s, 9H, $SiMe_3$), 0.25 (s, 9H, $SiMe_3$), 7.44 (d, 2H, $J=8.2$ Hz, Ar), 7.47–7.49 (m, 2H), 7.79 (s, 1H, HC=), 7.92–7.93 (dd, 2H, $J=2.5$ Hz, 6.3 Hz, Ar), 8.33 (d, 2H, $J=8.2$ Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ –0.56, 0.99 ($SiMe_3$), 109.33, 119.32, 121.02, 123.41, 127.15, 128.22, 130.99, 134.72, 142.22, 145.40, 146.87, 151.90; *m/z* (EI): 366 (19%, $[M+1]^+$), 365 (24%, $[M]^+$), 73 (100%, $[SiMe_3]^+$). Anal. calcd for $C_{20}H_{27}N_3Si_2$: C, 65.70; H, 7.44; N, 11.49; found: C, 65.89; H, 7.55; N, 11.12%.

2-(4-(2,2-Bis(dimethylsilyl)vinyl)phenyl)-2H-benzo[d][1,2,3]triazole (**2f**): Yellow solid, yield 73%, (silica gel, n-hexane/ethyl acetate, 15 : 4, $R_f=0.74$, m.p. 64–66 °C); FTIR (KBr, cm^{-1}): 2839 (HC=), 2121 (Si–H), 1253 and 891 (Si–C), 1105 (C–N); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.19 (d, 6H, $J=3.68$ Hz, $SiMe_2$), 0.29 (d, 6H, $J=3.68$ Hz, $SiMe_2$), 4.27–4.33 (m, 2H, Si–H), 7.41–7.43 (dd, 2H, $J=3.0$ Hz, 6.6 Hz, Ar), 7.47 (d, 2H, $J=8.5$ Hz, Ar), 7.80 (s, 1H, HC=), 7.92–7.94 (dd, 2H, $J=3.0$ Hz, 6.5 Hz, Ar), 8.33 (d, 2H, $J=8.5$ Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ –4.01, –3.36 ($SiMe_2$), 109.42, 119.36, 121.08, 123.43, 127.27, 128.69, 131.16, 135.04, 139.99, 142.66, 145.52, 153.07; *m/z* (EI): 337 (48%, $[M]^+$), 336 (56%, $[M–1]^+$), 278 (100%, $[M–SiMe_2H]^+$), 59 (26%, $[SiM_2H]^+$). Anal. calcd for $C_{18}H_{23}N_3Si_2$: C, 64.04; H, 6.87; N, 12.45; found: C, 64.26; H, 6.98; N, 12.29%.

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