Electronic Supplementary Information

Silylium Ion-promoted Dehydrogenative Cyclization: Synthesis of Silicon-containing Compounds Derived from Alkynes

Contents

Preparation of compounds 4-6. .................................................. S1

Table S1 Crystallographic data for 4ab and 4ae. ......................... S8

Fig. S1 Crystal structure of 4ae, showing 50% probability thermal ellipsoids. ........................................... S9

Fig. S2-15 $^1$H and $^{13}$C NMR spectra of 4-6. ...................... S10-23
**General Procedure.** All the reagents were of the highest grade available and were used without further purification. Benzyldimethylsilane (1a) and trityl tetrakis(pentafluorophenyl)borate were synthesized according to the previously reported method. Benzyldiisopropylsilane (1b) was prepared by the same method as 1a except for use of chlorodiisopropylsilane as a starting material. The NMR spectral measurements were performed on a Varian 400-MR NMR spectrometers. The 1H and 13C chemical shifts are reported relative to the residual protonated solvent and the solvent, respectively, according to the literature. High-resolution mass spectroscopy was measured by a JEOL GCMATE II corrected by perfluorokerosene. Elemental analysis was performed on a Thermo Scientific FLASH 2000 corrected by acetoanilide. Gel permeation column chromatography (GPC) was performed by a Japan Analytical Industry LC-918 using chloroform as an eluent.

**Preparation of Compounds.**

**Stoichiometric Cyclization**

**Method A.** To a hydrosilane (0.20 mmol), an alkyne (0.30 mmol) and a base (0.30 mmol) in benzene (3 mL) was slowly added a benzene solution (5 mL) of trityl tetrakis(pentafluorophenyl)borate (203 mg, 0.22 mmol) at room temperature under Ar atmosphere, and the resulting solution was stirred for 15 min for 1a or 30 min for 1b. The reaction was quenched by 1 M HCl (or water in the cases of trimethylsilylacetylene), and then the organic layer was extracted. After extraction by hexane, the organic layers were combined and dried over anhydrous sodium sulfate. After filtration, the filtrate was concentrated under reduced pressure to remove volatiles, and the residue was purified by silica gel column (eluent: hexane). Further purification was carried out by GPLC to obtain each of products.

**Method B.** To trityl tetrakis(pentafluorophenyl)borate (203 mg, 0.22 mmol), an alkyne (0.60 mmol) and a base (0.30 mmol) in benzene (5 mL) was slowly added a benzene solution (2 mL) of a hydrosilane (0.20 mmol) at room temperature under Ar atmosphere, and the resulting solution was
stirred for 15 min for 1a or 30 min for 1b. The following work-up was done according to Method A.

**Catalytic Cyclization.** To trityl tetrakis(pentafluoro-phenyl)borate (3.7 mg, 4.0 µmol) in benzene-\(d_6\) (0.2 mL) was added a benzene-\(d_6\) solution of hydrosilane 1a or 1b (0.22 mmol) and 1-hexyne (3c) (0.20 mmol) at room temperature under Ar atmosphere for 30 min for 1a and for 90 min for 1b monitored by \(^1\)H NMR. The reaction was quenched by 2,6-lutidine (5 µL), and then all volatiles were removed under reduced pressure. Purification was achieved by GPLC to afford the sila-cyclic products 5ac and 5bc in 33% and 50% isolated yields, respectively.

3-Trimethylsilyl-2,2-dimethyl-1,2-dihydro-2-silanaphthalene (4aa). 4aa was obtained as a colorless oil from the reaction using 1a and trimethylsilylacetylene (3a) in 73% yield. \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.41 (s, 1H, C=CH–Ar), 7.2-7.1 (m, 4H, Ph), 2.06 (s, 2H, SiCH\(_2\)), 0.17 (s, 9H, SiMe\(_3\)), 0.13 (s, 6H, SiMe\(_2\)). \(^{13}\)C NMR (CDCl\(_3\), 100 MHz): \(\delta\) 153.0, 140.3, 136.3, 136.0, 131.7, 130.8, 127.6, 125.6, 19.6, –0.44, –1.7. HRMS: Calcd for C\(_{14}\)H\(_{22}\)Si\(_2\) (M), 246.1260; Found, 246.1220.

2,2-Dimethyl-4-phenyl-1,2-dihydro-2-silanaphthalene (4ab). 4ab was obtained as a white solid from the reaction using 1a and phenylacetylene (3b) in 34% yield

4ab: Mp: 77-78 °C. \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.4-7.3 (m, 5H, Ph), 7.20 (d, 1H, \(J = 7.2\) Hz, ArH), 7.11 (dt, 1H, \(J = 7.2\) Hz, \(J = 1.6\) Hz, ArH), 7.02 (t, 1H, \(J = 7.2\) Hz, ArH), 6.97 (dd, 1H, \(J = 7.6\) Hz, \(J = 1.2\) Hz, ArH), 6.10 (s, 1H, Si–CH=C), 2.19 (s, 2H, SiCH\(_2\)), 0.14 (s, 6H, SiMe\(_2\)). \(^{13}\)C NMR (CDCl\(_3\), 100 MHz): \(\delta\) 156.8, 145.1, 137.5, 137.4, 131.2, 129.4, 128.2, 128.0 (overlapped two signals), 127.3, 127.1, 125.0, 21.4, –3.7. HRMS: Calcd for C\(_{17}\)H\(_{18}\)Si (M),
250.1178; Found, 250.1179. Anal. Calcd for 4ab (C_{17}H_{18}Si): C, 81.54; H, 7.25. Found: C, 81.75; H, 7.49.

4-n-Butyl-2,2-dimethyl-1,2-dihydro-2-silanaphthalene (4ac): 4ac was obtained as a colorless oil from the reaction using 1a and 1-hexyne (3c) in 33% yield. \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.38 (d, 1H, \(J = 7.6\) Hz, ArH), 7.2-7.0 (m, 3H, ArH), 5.86 (s, 1H, Si–CH=C), 2.60 (t, 2H, \(J = 7.6\) Hz, \(CH_2(\text{CH}_2)\)), 2.05 (s, 2H, SiCH\(_2\)), 1.6-1.3 (m, 4H, \(CH_2(\text{CH}_2)\)), 0.92 (t, 3H, \(J = 7.6\) Hz, \((\text{CH}_2)_3\)). \(^1\)C NMR (CDCl\(_3\), 100 MHz): \(\delta\) 155.2, 137.3, 137.1, 131.5, 126.8, 125.7, 125.2, 124.2, 38.3, 30.8, 22.6, 21.7, 14.0, -3.5. HRMS: Calcd for C\(_{15}\)H\(_{22}\)Si (M), 230.1491; Found, 230.1475.

2,2-Dimethyl-3,4-diphenyl-1,2-dihydro-2-silanaphthalene (4ae): 4ae was obtained as a white solid from the reaction using 1a and diphenylacetylene (3e) in 77% yield. Mp: 128-130 °C. \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.38 (d, 1H, \(J = 7.2\) Hz, ArH), 7.15-7.05 (m, 6H, ArH), 7.0-6.9 (m, 4H, ArH), 6.8-6.7 (m, 3H, ArH), 2.30 (s, 2H, SiCH\(_2\)), 0.71 (s, 6H, SiMe\(_2\)). \(^1\)C NMR (CDCl\(_3\), 100 MHz): \(\delta\) 150.3, 143.0, 141.8, 140.5, 138.9, 136.3, 131.0, 130.6, 130.1, 128.0, 127.6, 127.5, 127.0, 126.1, 125.1, 124.7, 21.3, -3.7. HRMS: Calcd for C\(_{23}\)H\(_{22}\)Si (M), 236.1491; Found, 236.1525. Anal. Calcd for 4ae (C\(_{23}\)H\(_{22}\)Si): C, 84.61; H, 6.79. Found: C, 84.64; H, 6.52.

3,4-Diethyl-2,2-dimethyl-1,2-dihydro-2-silanaphthalene (4af): 4af was obtained as a colorless oil from the reaction using 1a and 3-hexyne (3f) in 34% yield. \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.38 (d, 1H, \(J = 7.6\) Hz, ArH), 7.15-7.05 (m, 2H, ArH), 7.04 (m, 1H, \(J = 7.6\) Hz, ArH), 2.66 (q, 2H, \(J = 7.6\) Hz, \(CH_2\text{CH}_3\)), 2.39 (q, 2H, \(J = 7.6\) Hz, \(CH_2\text{CH}_3\)), 2.01 (s, 2H, SiCH\(_2\)),
1.09 (t, 3H, J = 7.6 Hz, CH₂CH₃), 1.03 (t, 3H, J = 7.6 Hz, CH₂CH₃), 0.06 (s, 6H, SiMe₂). ¹³C NMR (CDCl₃, 100 MHz): δ 148.3, 138.2, 137.8, 136.5, 131.0, 125.84, 125.82, 125.2, 24.2, 22.9, 21.7, 14.9, 14.6, –4.0. HRMS: Calcd for C₁₅H₂₂Si (M), 230.1491; Found, 230.1496.

**Benzyltrimethyl(2-phenylethynyl)silane (6ag):** 6ag was also obtained as a colorless oil from the reaction using 1a and phenyl(trimethylsilyl)acetylene (3g) in 54% yield. The spectroscopic data of 6ag is consistent with the previous reported data.¹ H NMR (CDCl₃, 400 MHz): δ 7.5-7.4 (m, 2H, ArH), 7.4-7.2 (m, 5H, ArH), 7.2-7.1 (m, 3H, ArH), 2.21 (s, 2H, SiC₂H₂Ph), 0.23 (s, 6H, SiMe₂).

¹³C NMR (CDCl₃, 100 MHz): δ 139.0, 131.9, 128.6, 128.4, 128.21, 128.16, 124.4, 123.0, 106.4, 92.5, 26.3, –2.1. HRMS: Calcd for C₁₇H₁₈Si (M), 250.1178; Found, 250.1151.

**2,2-Dimethyl-3-trimethylsilyl-1,2,3,4-tetrahydro-2-silanaphthalene** (5aa): 5aa was obtained as a colorless oil from the reaction using 1a and trimethylsilylacetylene (3a) in 12% yield. ¹H NMR (CDCl₃, 400 MHz): δ 7.15-7.0 (m, 4H, ArH), 2.80 (dd, 1H, J = 13.6 Hz, J = 4.0 Hz, CHCH₂Ar), 2.59 (dd, 1H, J = 14.0 Hz, J = 11.6 Hz, CHCH₂Ar), 2.02 (d, 1H, J = 14.4 Hz, SiCH₂Ar), 1.88 (d, 1H, J = 14.4 Hz, SiCH₂Ar), 0.17 (s, 3H, SiMe₂), 0.052 (s, 9H, SiMe₃), –0.036 (s, 3H, SiMe₂), –0.15 (dd, 1H, J = 11.6 Hz, J = 4.0 Hz, CHCH₂Ar). ¹³C NMR (CDCl₃, 100 MHz): δ 142.8, 138.2, 129.5, 127.4, 126.2, 124.7, 31.1, 21.6, 13.0, –0.57, –0.91, –1.3. HRMS: Calcd for C₁₄H₂₄Si₂ (M), 248.1417; Found, 248.1418.

**4-n-Butyl-2,2-Dimethyl-1,2,3,4-tetrahydro-2-silanaphthalene** (5ac): 5ac was obtained as a colorless oil from the catalytic reaction using 1a and 3c in 33% yield. ¹H NMR (CDCl₃, 400 MHz): δ 7.15-7.05 (m, 4H, ArH), 2.8-2.7 (m, 1H, CH₄(₆Bu)), 2.04 (d, 1H, J = 14.8 Hz, SiCH₂Ar),
1.97 (d, 1H, \(J = 14.8\) Hz, SiCH\(_2\)Ar), 1.9-1.8 (m, 1H, \(^7\)Bu), 1.65-1.5 (m, 1H, \(^7\)Bu), 1.5-1.2 (m, 4H, \(^7\)Bu), 0.99 (dd, 1H, \(J = 14.0\) Hz, \(J = 4.8\) Hz, SiCH\(_2\)CH), 0.93 (t, 3H, \(J = 7.2\) Hz, \(^7\)Bu), 0.51 (dd, 1H, \(J = 14.4\) Hz, \(J = 8.8\) Hz, SiCH\(_2\)CH), 0.10 (s, 3H, SiMe\(_2\)), 0.001 (s, 3H, SiMe\(_2\)). \(^{13}\)C NMR (CDCl\(_3\), 100 MHz): \(\delta\) 144.3, 138.0, 130.2, 126.3, 125.9, 124.7, 40.0, 35.0, 30.4, 22.9, 20.8, 18.0, 14.1, -1.2, -1.6. HRMS: Calcd for C\(_{15}\)H\(_{24}\)Si (M), 232.1647; Found, 232.1621.

**2,2-Diisopropyl-3-trimethylsilyl-1,2-dihydro-2-silanaphthalene (4ba)**

(4ba) was obtained as a colorless oil from the reaction using 1b and 3a in 71% yield. \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.59 (s, 1H, C=CH–Ar), 7.2-7.1 (m, 4H, ArH), 2.09 (s, 2H, SiCH\(_2\)), 1.2-1.0 (m, 2H, CH(CH\(_3\))\(_2\)), 0.98 (d, 6H, \(J = 7.6\) Hz, CH(CH\(_3\))\(_2\)), 0.94 (d, 6H, \(J = 6.8\) Hz, CH(CH\(_3\))\(_2\)), 0.22 (s, 6H, SiMe\(_2\)). \(^{13}\)C NMR (CDCl\(_3\), 100 MHz): \(\delta\) 156.7, 136.8, 136.6, 136.5, 131.6, 131.0, 127.8, 125.4, 18.9, 18.1, 12.3, 11.6, 0.38. HRMS: Calcd for C\(_{18}\)H\(_{30}\)Si\(_2\) (M), 302.1886; Found, 302.1884.

**2,2-Diisopropyl-4-phenyl-1,2-dihydro-2-silanaphthalene (4bb)**

(4bb) was obtained as a colorless oil from the reaction using 1b and 3b in 67% yield. \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.4-7.3 (m, 5H, ArH), 7.21 (d, 1H, \(J = 7.6\) Hz, ArH), 7.10 (td, 1H, \(J = 7.6\) Hz, \(J = 1.6\) Hz, ArH), 6.94 (d, 1H, \(J = 6.4\) Hz, ArH), 6.08 (s, 1H, Si–CH=Si), 2.23 (s, 2H, SiCH\(_2\)), 1.1-0.9 (m, 14H, CH(CH\(_3\))\(_2\)). \(^{13}\)C NMR (CDCl\(_3\), 100 MHz): \(\delta\) 158.5, 145.5, 137.79, 137.75, 131.2, 129.5, 128.3, 128.0, 127.4, 127.1, 124.9, 124.6, 18.3, 18.2, 15.4, 11.1. HRMS: Calcd for C\(_{21}\)H\(_{26}\)Si (M), 306.1804; Found, 306.1845.

**2,2-Diisopropyl-4-n-butyl-1,2-dihydro-2-silanaphthalene (4bc)**

(4bc) was obtained as a colorless oil from the reaction using 1b with 3c in 66% yield. \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.38 (d, 1H, \(J = 7.6\) Hz, ArH), 7.2-7.0 (m, 3H, ArH), 5.85 (s, 1H, Si–CH=Si), 2.66 (t, 2H, \(J = 7.6\) Hz, CH\(_2\)(CH\(_2\))\(_2\)CH\(_3\)), 2.09 (s, 2H, SiCH\(_2\)), 1.6-1.3 (m, 4H, CH\(_2\)(CH\(_2\))\(_2\)CH\(_3\)), 1.1-0.9 (m, 17H, CH(CH\(_3\))\(_2\) and
CH₂(CH₂)₂CH₃. ¹³C NMR (CDCl₃, 100 MHz): δ 156.8, 137.6, 137.4, 131.5, 126.9, 125.7, 125.1, 120.6, 38.6, 31.0, 22.5, 18.2, 18.1, 15.3, 14.0, 11.1. HRMS: Calcd for C₁₉H₃₀Si (M), 286.2117; Found, 286.2112.

2,2-Diisopropyl-3,4-diphenyl-1,2-dihydro-2-silanaphthalene (4be):

4be was obtained as a colorless oil from the reaction of 1b and 3e in 82% yield. ¹H NMR (CDCl₃, 400 MHz): δ 7.22 (d, 1H, J = 6.8 Hz, ArH), 7.2-7.0 (m, 6H, ArH), 7.0-6.9 (m, 4H, ArH), 6.84 (d, 2H, J = 7.2 Hz, ArH), 6.70 (d, 1H, J = 7.6 Hz, ArH), 2.34 (s, 2H, SiC₂H₂), 1.1-1.0 (m, 2H, C₂H₂C₃), 0.98 (d, 6H, J = 6.8 Hz, CH₂(CH₃)₂), 0.83 (d, 6H, J = 7.2 Hz, CH₂(CH₃)₂). ¹³C NMR (CDCl₃, 100 MHz): δ 152.6, 143.4, 142.0, 139.2, 138.7, 136.9, 130.70, 130.65, 130.1, 128.6, 127.5 (overlapped two signals), 127.2, 126.1, 125.0, 124.7, 18.4, 18.0, 14.7, 11.6. HRMS: Calcd for C₂₄H₂₃Si (M–iPr), 339.1569; Found, 339.1636.

3,4-Diethyl-2,2-diisopropyl-1,2-dihydro-2-silanaphthalene (4bf): 4bf

was obtained as a colorless oil from the reaction of 1b and 3f in 39% yield. ¹H NMR (CDCl₃, 400 MHz): δ 7.34 (d, 1H, J = 8.0 Hz, ArH), 7.15-7.1 (m, 2H, ArH), 7.02 (t, 1H, J = 7.6 Hz, ArH), 2.69 (q, 2H, J = 7.6 Hz, CH₂CH₃), 2.38 (q, 2H, J = 7.6 Hz, CH₂CH₃), 2.04 (s, 2H, SiCH₂), 1.1-1.0 (m, 8H, CH₂CH₃ and CH₂(CH₃)₂), 0.97 (d, 6H, J = 6.4 Hz, CH₂(CH₃)₂), 0.89 (d, 6H, J = 7.2 Hz, CH₂(CH₃)₂). ¹³C NMR (CDCl₃, 100 MHz): δ 151.2, 138.6, 136.9, 135.5, 130.8, 126.0, 125.8, 125.1, 24.3, 22.6, 18.7, 18.2, 15.1, 14.8, 14.5, 11.3. HRMS: Calcd for C₂₄H₂₃Si (M–iPr), 339.1569; Found, 339.1634.

4-n-Butyl-2,2-Dimethyl-1,2,3,4-tetrahydro-2-silanaphthalene (5bc):

5bc was obtained as a colorless oil from the catalytic reaction using 1b and 3c in 50% yield. ¹H NMR (CDCl₃, 400 MHz): δ 7.15-7.05 (m, 4H, ArH), 2.7-2.6 (m, 1H, CH₂Bu), 2.05 (d, 1H, J = 14.8 Hz, SiCH₂Ar),
2.00 (d, 1H, J = 14.8 Hz, SiCH$_2$Ar), 1.95-1.85 (m, 1H, nBu), 1.7-1.3 (m, 5H, nBu and iPr), 1.06 (dd, 1H, J = 14.8 Hz, J = 4.8 Hz, SiCH$_2$), 1.01 (brs, 7H, iPr), 0.94 (t, 3H, J = 7.6 Hz, nBu), 0.9-0.8 (m, 7H, iPr), 0.37 (dd, 1H, J = 14.4 Hz, J = 10.0 Hz, SiCH$_2$CH), 1.01 (brs, 7H, iPr), 0.94 (t, 3H, J = 7.6 Hz, nBu), 0.9-0.8 (m, 7H, iPr), 0.37 (dd, 1H, J = 14.4 Hz, J = 10.0 Hz, SiCH$_2$CH). $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta$ 144.3, 138.6, 130.0, 125.9, 125.4, 124.6, 39.3, 35.3, 30.3, 23.0, 18.2, 18.14, 18.09, 17.9, 15.0, 14.2, 12.3, 11.8, 11.7. HRMS: Calcd for C$_{19}$H$_{32}$Si (M), 288.2273; Found, 288.2297.

X-ray Crystallography. Single crystals of 4ab and 4ae suitable for XRD analyses were obtained. Each crystal was mounted on a glass fiber, and the diffraction data was collected on a Bruker APEX II CCD detector using graphite monochromated Mo Ka radiation at 123 K.

All the structures were solved by the combination of the direct method and Fourier techniques, and all the non-hydrogen atoms were anisotropically refined by full-matrix least-squares calculations. The atomic scattering factors and anomalous dispersion terms were obtained from the International Tables for X-ray Crystallography IV.$^5$ The refinement of all structures was carried out by full-matrix least-squares method of SHELXL-97.$^6$

<table>
<thead>
<tr>
<th></th>
<th>4ab</th>
<th>4ae</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>formula</strong></td>
<td>C$<em>{34}$H$</em>{36}$Si$_2$</td>
<td>C$<em>{22}$H$</em>{20}$Si</td>
</tr>
<tr>
<td><strong>fw</strong></td>
<td>500.81</td>
<td>312.47</td>
</tr>
<tr>
<td><strong>crystal system</strong></td>
<td>triclinic</td>
<td>monoclinic</td>
</tr>
<tr>
<td><strong>space group</strong></td>
<td>P-1 (#2)</td>
<td>P$_2_1/n$ (#14)</td>
</tr>
<tr>
<td><strong>a, Å</strong></td>
<td>9.8603(13)</td>
<td>9.7459(10)</td>
</tr>
<tr>
<td><strong>b, Å</strong></td>
<td>10.5873(14)</td>
<td>10.4452(11)</td>
</tr>
<tr>
<td><strong>c, Å</strong></td>
<td>14.4723(19)</td>
<td>18.2147(19)</td>
</tr>
<tr>
<td><strong>α, deg</strong></td>
<td>72.748(2)</td>
<td>90</td>
</tr>
<tr>
<td><strong>β, deg</strong></td>
<td>86.325(2)</td>
<td>99.690(1)</td>
</tr>
<tr>
<td><strong>γ, deg</strong></td>
<td>84.298(2)</td>
<td>90</td>
</tr>
<tr>
<td><strong>V, Å$^3$</strong></td>
<td>1434.8(3)</td>
<td>1827.8(3)</td>
</tr>
<tr>
<td><strong>Z</strong></td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td><strong>ρ$\text{calcd}$, g cm$^{-3}$</strong></td>
<td>1.159</td>
<td>1.136</td>
</tr>
<tr>
<td><strong>μ(Mo Kα), mm$^{-1}$</strong></td>
<td>0.144</td>
<td>0.126</td>
</tr>
<tr>
<td><strong>total no. of data</strong></td>
<td>8189</td>
<td>10169</td>
</tr>
<tr>
<td><strong>no. of unique data ($R_{int}$)</strong></td>
<td>6222 (0.0171)</td>
<td>4132 (0.0174)</td>
</tr>
<tr>
<td><strong>no. of params</strong></td>
<td>329</td>
<td>219</td>
</tr>
<tr>
<td><strong>$R1^a$</strong></td>
<td>0.0449</td>
<td>0.0349</td>
</tr>
<tr>
<td><strong>wR2 (all data)$^b$</strong></td>
<td>0.0863</td>
<td>0.0764</td>
</tr>
</tbody>
</table>

$^a I > 2.00\sigma(I), R1 = \Sigma||F_o|| - |F_c||/\Sigma|F_o||.  
$^b wR2 = \{\Sigma(w|F_o|^2 - |F_c|^2)^2/\Sigma wF_o^2}\}^{1/2}.$
Fig. S1  Crystal structures of 4ae, showing 50% probability thermal ellipsoids.
**Fig. S2** $^1$H and $^{13}$C NMR spectra of 4aa.
Fig. S3  $^1$H and $^{13}$C NMR spectra of 4ab.
Fig. S4  $^1$H and $^{13}$C NMR spectra of 4ac.
Fig. S5  $^1$H and $^{13}$C NMR spectra of 4ae.
Fig. S6 $^1$H and $^{13}$C NMR spectra of 4af.
Fig. S7  $^1$H and $^{13}$C NMR spectra of 5aa.
Fig. S8  \(^1\)H and \(^{13}\)C NMR spectra of 5ac.
Fig. S9  $^1$H and $^{13}$C NMR spectra of 6ag.
Fig. S10  $^1$H and $^{13}$C NMR spectra of 4ba.
Fig. S11  $^1$H and $^{13}$C NMR spectra of 4bb.
Fig. S12  $^1$H and $^{13}$C NMR spectra of 4bc.
Fig. S13 $^1$H and $^{13}$C NMR spectra of 4be.
Fig. S14  \(^1\)H and \(^{13}\)C NMR spectra of 4bf.
Fig. S15 $^1$H and $^{13}$C NMR spectra of 5bc.