Electronic Supplementary Information (ESI)

2-(Chlorodiisopropylsilyl)-6-(trimethylsilyl)phenyl triflate: a modified platform for intramolecular benzyne cycloadditions

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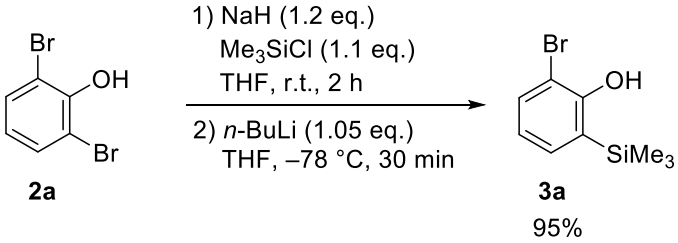
1. General experimental procedure

All non-aqueous reactions were carried out in dried glassware under an atmosphere of dry argon unless otherwise noted. THF, Et_2O, toluene and CH_2Cl_2 (anhydrous; Kanto Chemical Co., Inc.) were used as receive. CH_3CN was distilled prior to use according to the standard protocols. KF, CsF, 18-crown-6, Cs_2CO_3, and K_2CO_3 were dried with heating under reduced pressure prior to use. The other reagents were purchased and used without further purifications. Analytical TLC was performed on pre-coated silica gel plate (Wako Silicagel 70 F254). Flash column chromatography was performed on Wakogel 60N. Preparative thin-layer chromatography (PTLC) was performed using plates prepared from Wakogel® B5-F. HPLC was performed on a YMC LC-forte/R using YMC-GPC T4000® and YMC-GPC T2000® columns. Melting points (mp) were determined on YANACO micro melting point apparatus. ^1H and ^{13}C NMR spectra were recorded on a JEOL JNM-LA 500 or a JEOL JNM ECZ 600R in the solvent indicated; Chemical shifts (δ) are expressed in parts per million (ppm), and coupling constants are reported as hertz (Hz). Tetramethylsilane (δ 0.00 ppm) was used as an internal standard for ^1H NMR. Chloroform-D (δ 77.0 ppm) was used as an internal standard for ^{13}C NMR. Multiplicities are indicated as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet) and br (broad). Attenuated total reflectance Fourier transform infrared (ATR-IR) spectra were recorded on a Shimadzu IRAffinity-1 with a ATR measurement attachment (MIRacle 10), and the wave numbers of maximum absorption peaks are reported in cm^{-1}. High-resolution mass spectra (HRMS) were recorded on a JEOL MS700 spectrometer for FAB mass spectrometry or a SHIMADZU LCMS-IT-TOF for ESI mass spectrometry.

2. Experimental procedure and characterization data

2-1. Preparation of cycloaddition precursors

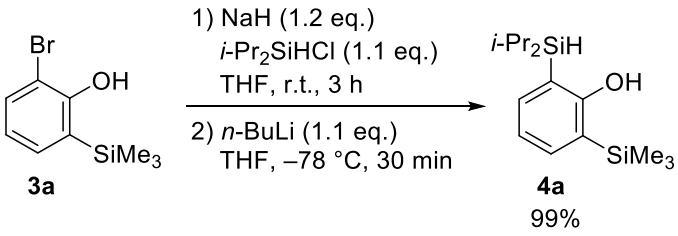
Synthesis of phenol **3a**



To a solution of 2,6-dibromophenol (**2a**) (7.60 g, 30.2 mmol) in THF (90 mL) was added NaH (60% dispersion in mineral oil, 1.45 g, 36.2 mmol) at 0 °C. After stirring for 30 min at this temperature, Me_3SiCl (4.19 mL, 33.2 mmol) was added dropwise and the mixture was stirred for 2 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.60 M) in hexane (19.8 mL, 31.7 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford phenol **3a** (6.99 g, 95%) as colorless oil.

3a: $\boldsymbol{R_f}$ 0.44 (hexane); Spectral data matched those reported in the literature.[1]

Synthesis of phenol **4a**

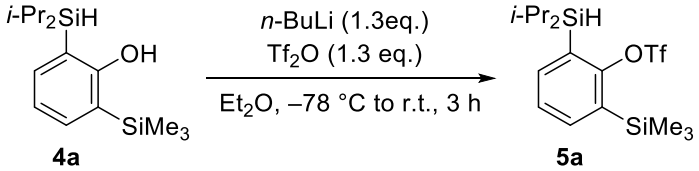


To a solution of **3a** (6.99 g, 28.6 mmol) in THF (85 mL) was added NaH (60% dispersion in mineral oil, 1.37 g, 34.3 mmol) at 0 °C. After stirring for 30 min at this temperature, *i*-Pr_2SiHCl (5.33 mL, 31.5 mmol) was added, and the mixture was stirred for 3 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.60 M) in hexane (19.7 mL, 31.5 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford phenol **4a** (8.03 g, 99%) as colorless oil.

4a: $\boldsymbol{R_f}$ 0.52 (hexane); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.30 (s, 9H), 1.00 (d, 6H, J = 7.5 Hz), 1.10 (d, 6H, J =

7.5 Hz), 1.29 (qqd, 2H, J = 7.5, 7.5, 3.4 Hz), 4.13 (t, 1H, J = 3.4 Hz), 5.50 (s, 1H), 6.92 (dd, 1H, J = 7.5, 7.2 Hz), 7.31 (dd, 1H, J = 7.5, 1.7 Hz), 7.40 (dd, 1H J = 7.2, 1.7 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ –1.4, 10.3, 18.1, 18.4, 116.8, 120.2, 125.0, 137.4, 137.6, 166.2; **IR** (neat): 3549, 2951, 2866, 2059, 1392, 906, 840, 732 cm^{-1}; **HRMS** (ESI): calcd. for $C_{15}H_{27}OSi_2$ $[M–H]^-$: 279.1606; found: 279.1602.

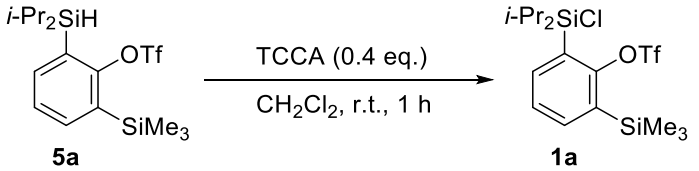
Synthesis of triflate **5a**



To a solution of **4a** (17.5 g, 62.5 mmol) in Et_2O (400 mL) was added was added *n*-BuLi (2.3 M) in hexane (35.3 mL, 81.3 mmol) at –78 °C. After stirring for 30 min at this temperature, Tf_2O (13.7 mL, 81.3 mmol) was added dropwise to the mixture. The mixture was warmed to room temperature, and the stirring was continued for 3 h. The reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford triflate **5a** (22.0 g, 85%) as colorless oil.

5a: R_f 0.50 (hexane); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.37 (s, 9H), 0.95 (d, 6H, J = 7.5 Hz), 1.06 (d, 6H, J = 7.5 Hz), 1.21–1.29 (m, 2H), 4.19–4.21 (m, 1H), 7.36 (dd, 1H, J = 7.2, 7.2 Hz), 7.49 (dd, 1H, J = 7.2, 1.5 Hz), 7.61 (dd, 1H J = 7.2, 1.5 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ –0.2, 10.5, 18.2, 118.8 (q, J_{CF} = 320 Hz), 127.5, 130.4, 135.3, 138.0, 138.7, 156.3; **IR** (neat): 2954, 2866, 2179, 1400, 1219, 1138, 910, 875, 848, 736 cm^{-1}; **HRMS** (ESI): calcd. for $C_{16}H_{26}F_3O_3SSi_2$ $[M–H]^-$: 411.1099; found: 411.1087.

Synthesis of silyl chloride **1a**

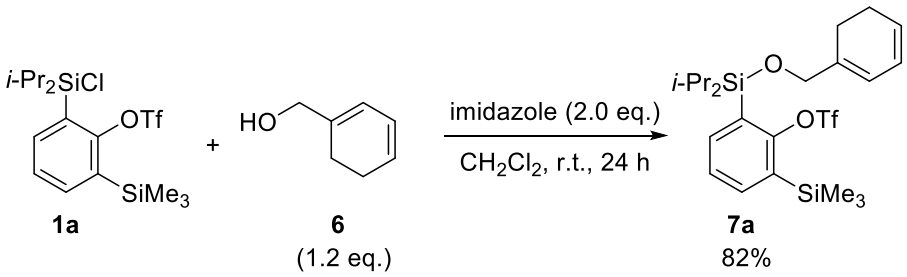


To a solution of **5a** (22.0 g, 53.3 mmol) in CH_2Cl_2 (400 mL) was added trichloroisocyanuric acid[2] (TCCA, 4.95 g, 21.3 mmol). After stirring for 1 h at room temperature, the reaction mixture was filtered through a Celite® pad (washed with hexane). The filtrate was concentrated in vacuo to afford silyl chloride **1a** (23.7 g, 99%) as colorless oil, which solidified at 5 °C to give colorless crystals.

1a: **mp**: 45–51 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.36 (s, 9H), 0.97 (d, 6H, J = 7.5 Hz), 1.18 (d, 6H, J = 7.2 Hz), 1.64 (qq, 2H, J = 7.5, 7.2 Hz), 7.43 (dd, 1H, J = 7.5, 7.5 Hz), 7.66 (dd, 1H, J = 7.5, 2.0 Hz), 7.88 (dd, 1H, J = 7.5, 2.0 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 0.2, 15.1, 17.9, 118.4 (q, J_{CF} = 318 Hz), 127.4, 128.9, 135.0, 139.4, 140.4, 153.9; **IR** (neat): 2954, 2870, 2252, 1396, 1219, 906, 729 cm^{-1}; **HRMS** (FAB):

calcd. for $C_{16}H_{27}ClF_3O_3SSi_2$ $[M+H]^+$: 447.0855; found: 447.0849.

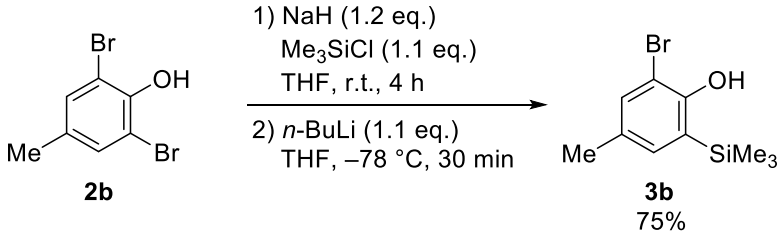
Synthesis of silyl ether **7a**



To a solution of **1a** (1.88 g, 4.20 mmol) in CH_2Cl_2 (42 mL) were added imidazole (572 mg, 8.40 mmol) and alcohol **6**[3] (555 mg, 5.04 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/50) to afford silyl ether **7a** (1.80 g, 82%) as colorless oil.

7a: $\boldsymbol{R_f}$ 0.48 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.36 (s, 9H), 1.01 (d, 6H, J = 7.6 Hz), 1.15 (d, 6H, J = 6.9 Hz), 1.40 (qq, 2H, J = 7.6, 6.9 Hz), 2.09 (brt, 2H, J = 9.7 Hz), 2.18–2.23 (m, 2H), 4.14 (s, 2H), 5.71–5.75 (m, 1H), 5.93–5.97 (m, 2H), 7.38 (dd, 1H, J = 6.9, 6.8 Hz), 7.61 (dd, 1H, J = 6.8, 2.1 Hz), 7.65 (dd, 1H, J = 6.9, 2.1 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.2, 13.4, 17.5, 18.0, 22.3, 22.9, 66.4, 118.0, 118.6 (q, J_{CF} = 319 Hz), 124.6, 125.2, 127.4, 130.4, 135.1, 138.2, 139.0, 139.3, 155.7; **IR** (neat): 2951, 2866, 2252, 1396, 1215, 1141, 1095, 910, 721 cm^{-1}; **HRMS** (ESI): calcd. for $C_{23}H_{35}F_3NaO_4SSi_2$ $[M+Na]^+$: 543.1639; found: 543.1641.

Synthesis of phenol **3b**

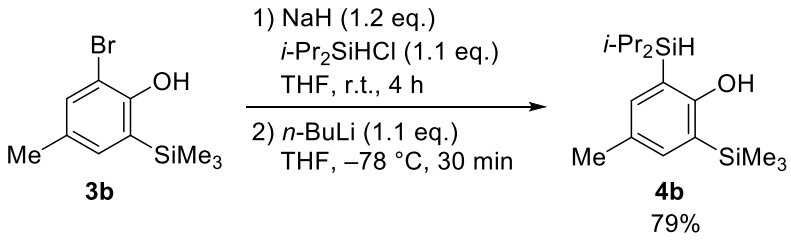


To a solution of 2,6-dibromo-4-methylphenol (**2b**) (726 mg, 2.73 mmol) in THF (15 mL) was added NaH (60% dispersion in mineral oil, 131 mg, 3.28 mmol) at 0 °C. After stirring for 30 min at this temperature, Me_3SiCl (379 μL, 3.00 mmol) was added and the mixture was stirred for 4 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.6 M) in hexane (1.88 mL, 3.00 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column

chromatography (silica gel, EtOAc/hexane = 1/100) to afford phenol **3b** (531 mg, 75%) as colorless oil.

3b: $\boldsymbol{R_f}$ 0.71 (EtOAc/hexane = 1/10); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.29 (s, 9H), 2.66 (dd, 3H, J = 0.9, 0.9 Hz), 5.53 (s, 1H), 7.07 (dq, 1H, J = 2.0, 0.9 Hz), 7.28 (dq, 1H, J = 2.0, 0.9 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ –1.1, 20.2, 110.1, 126.6, 130.9, 133.1, 135.0, 153.9; **IR** (neat): 3522, 2954, 1450, 1392, 1246, 1176, 906, 837, 744 cm^{-1}; **HRMS** (ESI): calcd. for $C_{10}H_{14}BrOSi$ $[M–H]^–$: 257.0003; found: 257.0003.

Synthesis of phenol **4b**



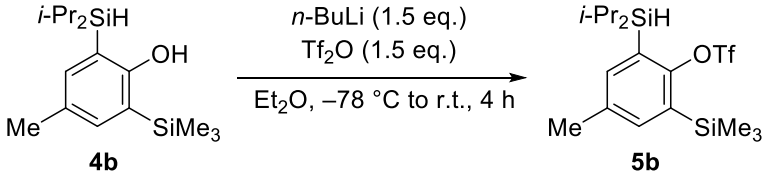
3b **4b**

79%

To a solution of **3b** (444 mg, 1.71 mmol) in THF (10 mL) was added NaH (60% dispersion in mineral oil, 82.0 mg, 2.05 mmol) at 0 °C. After stirring for 30 min at this temperature, *i*-Pr_2SiHCl (318 μL, 1.88 mmol) was added and the mixture was stirred for 4 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.6 M) in hexane (1.17 mL, 31.5 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford phenol **4b** (395 mg, 79%) as colorless oil.

4b: $\boldsymbol{R_f}$ 0.45 (hexane); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.30 (s, 9H), 1.00 (d, 6H, J = 6.8 Hz), 1.09 (d, 6H, J = 6.9 Hz), 1.28 (qqd, 2H, J = 6.9, 6.8, 3.5 Hz), 2.27 (s, 3H), 4.09 (t, 1H, J = 3.5 Hz), 5.33 (s, 1H), 7.08 (d, 1H, J = 2.1 Hz), 7.18 (d, 1H, J = 2.1 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –1.4, 10.3, 18.2, 18.4, 20.3, 116.7, 124.7, 128.8, 137.9, 164.2 (several signals overlapped); **IR** (neat): 3549, 2947, 2862, 2059, 1573, 1246, 1176, 906, 837, 729 cm^{-1}; **HRMS** (ESI): calcd. For $C_{16}H_{29}Osi_2$ $[M–H]^–$: 293.1762; found: 293.1760.

Synthesis of triflate **5b**



4b **5b**

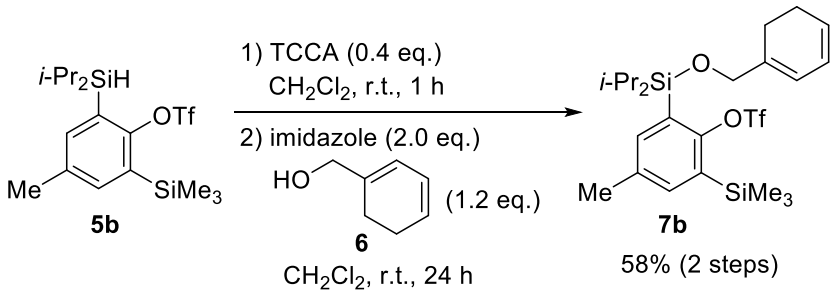
74%

To a solution of **4b** (191 mg, 0.649 mmol) in Et_2O (6.5 mL) was added was added *n*-BuLi (1.6 M) in hexane (610 μL, 0.974 mmol) at –78 °C. After stirring for 30 min at this temperature, Tf_2O (165 μL, 0.974 mmol) was added dropwise to the mixture. The mixture was warmed to room temperature, and the stirring was continued for 4 h. The reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and

concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford triflate **5b** (206 mg, 74%) as colorless oil.

5b: R_f 0.34 (hexane); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.36 (s, 9H), 0.95 (d, 6H, J = 7.2 Hz), 1.06 (d, 6H, J = 7.5 Hz), 1.19–1.29 (m, 2H), 2.37 (s, 3H), 4.14–4.18 (m, 1H), 7.25 (d, 1H, J = 2.3 Hz), 7.36 (d, 1H, J = 2.3 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 0.2, 10.9, 18.5, 18.6, 20.9, 118.5 (q, J_{CF} = 318 Hz), 129.6, 134.4, 136.6, 138.2, 138.9, 153.8; **IR** (neat): 2947, 2866, 2175, 1396, 1249, 1138, 1053, 910, 844 cm^{-1}; **HRMS** (FAB): calcd. for $C_{17}H_{30}F_3O_3SSi_2$ $[M+H]^+$: 427.1401; found: 427.1403.

Synthesis of silyl ether **7b**

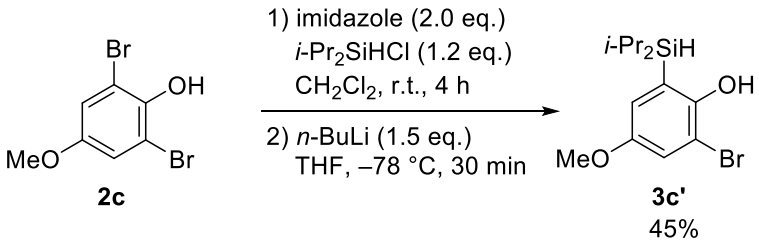


1) To a solution of **5b** (145 mg, 0.339 mmol) in CH_2Cl_2 (3.5 mL) was added trichloroisocyanuric acid[2] (TCCA, 31.5 mg, 0.136 mmol). After stirring for 1 h at room temperature, the reaction mixture was filtered through a Celite® pad (washed with hexane), and the filtrate was concentrated in vacuo to afford the corresponding silyl chloride as colorless oil. This material was employed in the next reaction without further purification.

2) To a solution of the crude material (*vide supra*) in CH_2Cl_2 (3.5 mL) were added imidazole (46.4 mg, 0.678 mmol) and alcohol **6**[3] (44.8 mg, 0.407 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford silyl ether **7b** (104 mg, 58% in 2 steps) as colorless oil.

7b: R_f 0.52; (EtOAc/hexane = 1/20); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.35 (s, 9H), 1.01 (d, 6H, J = 7.6 Hz), 1.15 (d, 6H, J = 7.6 Hz), 1.39 (qq, 2H, J = 7.6, 7.6 Hz), 2.09 (brt, 2H, J = 8.9 Hz), 2.18−2.23 (m, 2H), 2.34 (s, 3H), 4.13 (s, 2H), 5.72–5.76 (m, 1H), 5.93–5.98 (m, 2H), 7.37 (d, 1H, J = 2.3 Hz), 7.43 (d, 1H, J = 2.3 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.2, 13.5, 17.6, 18.1, 20.5, 22.3, 22.9, 66.4, 118.1, 118.6 (q, J_{CF} = 321 Hz), 124.6, 125.2, 129.9, 134.6, 136.9, 138.3, 139.6, 139.9, 153.7; **IR** (neat): 2947, 2866, 2360, 1396, 1249, 1211, 1138, 1045, 914, 810 cm^{-1}; **HRMS** (ESI): calcd. for $C_{24}H_{37}F_3NaO_4SSi_2$ $[M+Na]^+$: 557.1795; found: 557.1798.

Synthesis of phenol **3c'**

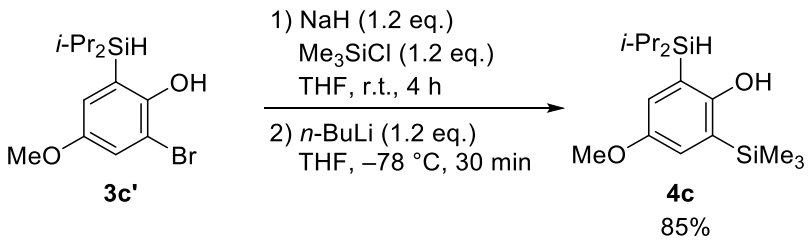


2c **3c'** 45%

To a solution of 2,6-dibromo-4-methoxyphenol (**2c**) (2.53 g, 8.98 mmol) in CH_2Cl_2 (30 ml) were added imidazole (1.22 g, 18.0 mmol) and *i*-Pr_2SiHCl (1.83 mL, 10.8 mmol). After stirring for 3 h at room temperature, the reaction was quenched by adding phenol (169 mg, 1.80 mmol) and hexane (20 mL) was added. CH_2Cl_2 was removed by concentration (ca. 300 mbar, 30 °C). The resulting suspension was filtered through a Celite® pad (wash with hexane), and the filtrate was concentrated in vacuo. The crude material was dissolved in THF (30 mL), to which was added *n*-BuLi (1.6 M in hexane, 8.42 mL, 13.5 mmol) dropwise at –78 °C, and the stirring was continued for 30 min at this temperature. The reaction was quenched by adding saturated aqueous NH_4Cl, and the reaction mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 1/30) to afford phenol **3c'** (1.28 g, 45%) as red oil.

3c': $\boldsymbol{R_f}$ 0.53 (EtOAc/hexane = 1/5); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.98 (d, 6H, *J* = 7.2 Hz), 1.07 (d, 6H, *J* = 7.2 Hz), 1.33 (qqd, 2H, *J* = 7.2, 7.2, 3.8 Hz), 3.76 (s, 3H), 3.89 (t, 1H, *J* = 3.8 Hz), 5.33 (s, 1H), 6.93 (d, 1H, *J* = 3.2 Hz), 7.02 (d, 1H, *J* = 3.2 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 10.9, 18.9, 55.9, 109.7, 117.9, 122.5, 123.0, 150.0, 153.1; **IR** (neat): 3529, 2043, 2098, 1562, 1458, 1400, 1284, 1068, 1045, 1002, 906, 790 cm^{-1}; **HRMS** (ESI): calcd. for $C_{13}H_{20}BrO_2Si$ $[M–H]^-$: 315.0421; found: 315.0410.

Synthesis of phenol **4c**

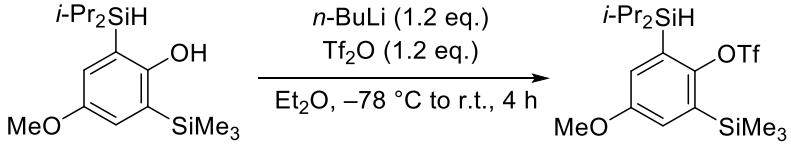


3c' **4c** 85%

To a solution of **3c'** (1.26 g, 3.96 mmol) in THF (12 mL) was added NaH (60% dispersion in mineral oil, 190 mg, 4.75 mmol) at 0 °C. After stirring for 30 min at this temperature, Me_3SiCl (600 μL, 4.75 mmol) was added and the mixture was stirred for 4 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.6 M) in hexane (2.97 mL, 4.75 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/30) to afford phenol **4c** (1.04 g, 85%) as colorless oil.

4c: R_f 0.50 (EtOAc/hexane = 1/10); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.30 (s, 9H), 1.01 (d, 6H, *J* = 7.5 Hz), 1.10 (d, 6H, *J* = 7.2 Hz), 1.28 (qqd, 2H, *J* = 7.5, 7.2, 3.5 Hz), 3.77 (s, 3H), 4.08 (t, 1H, *J* = 3.5 Hz), 5.13 (s, 1H), 6.83 (d, 1H, *J* = 3.2 Hz), 6.94 (d, 1H, *J* = 3.2 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ –1.0, 10.7, 18.5, 18.7, 55.7, 118.0, 121.6, 122.2, 126.2, 152.7, 159.7; **IR** (neat): 3521, 2947, 2063, 2098, 1577, 1396, 1246, 1207, 1049, 1006, 906, 837, 791 cm^{-1}; **HRMS** (ESI): calcd. For $C_{13}H_{29}O_2Si_2$ $[M–H]^–$: 309.1712; found: 309.1715.

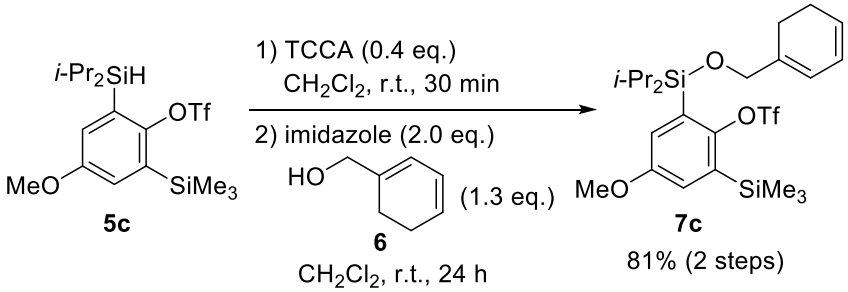
Synthesis of triflate **5c**



To a solution of phenol **4c** (780 mg, 2.51 mmol) in Et_2O (25 mL) was added was added *n*-BuLi (1.6 M) in hexane (1.89 mL, 3.01 mmol) at –78 °C. After stirring for 30 min at this temperature, Tf_2O (507 μL, 3.01 mmol) was added dropwise to the mixture. The mixture was warmed to room temperature, and the stirring was continued for 4 h. The reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/50) to afford triflate **5c** (955 mg, 86%) as colorless oil.

5c: R_f 0.64 (EtOAc/hexane = 1/5); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.37 (s, 9H), 0.96 (d, 6H, *J* = 7.5 Hz), 1.07 (d, 6H, *J* = 7.2 Hz), 1.21–1.28 (m, 2H), 3.83 (s, 3H), 4.17 (brs, 1H), 6.95 (d, 1H, *J* = 3.2 Hz), 7.04 (d, 1H, *J* = 3.2 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 0.2, 10.9, 18.5, 18.6, 55.5, 118.5 (q, J_{CF} = 320 Hz), 122.2, 122.5. 131.5, 136.3, 148.7 157.5; **IR** (neat) 2951, 1570, 1396, 1246, 1215, 1145, 1037, 910, 844, 732 cm^{-1}; **HRMS** (FAB): calcd. For $C_{17}H_{30}F_3O_4Ssi_2$ $[M+H]^+$: 443.1350; found 443.1351.

Synthesis of silyl ether **7c**



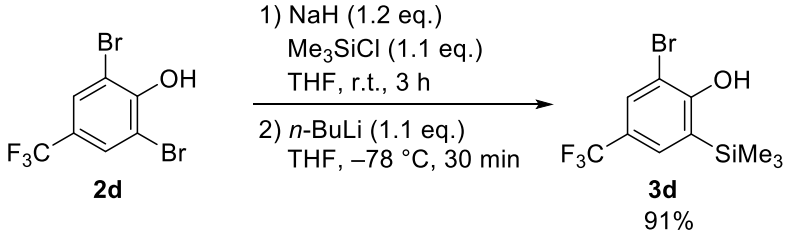
1) To a solution of **5c** (164 mg, 0.371 mmol) in CH_2Cl_2 (4 mL) was added trichloroisocyanuric acid[2] (TCCA, 34.5 mg, 0.148 mmol). After stirring for 30 min at room temperature, the reaction mixture was filtered through a Celite® pad (washed with hexane), and the filtrate was concentrated in vacuo to afford the corresponding silyl chloride as colorless oil. This material was employed in the next reaction without further

purification.

2) To a solution of the crude material (*vide supra*) in CH_2Cl_2 (4 mL) were added imidazole (50.5 mg, 0.742 mmol) and alcohol **6**[3] (53.1 mg, 0.482 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/100) to afford silyl ether **7c** (143 mg, 81% in 2 steps) as colorless oil.

7c: R_f 0.67 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.35 (s, 9H), 1.03 (d, 6H, J = 6.8 Hz), 1.17 (d, 6H, J = 6.9 Hz), 1.40 (qq, 2H, J = 6.9, 6.8 Hz), 2.07 (brt, 2H, J = 9.7 Hz), 2.16–2.23 (m, 2H), 3.77 (s, 3H), 4.15 (s, 2H), 5.71–5.74 (m, 1H), 5.93–5.96 (m, 1H), 5.99–6.01 (m, 1H), 7.07 (d, 1H, J = 2.8 Hz), 7.12 (d, 1H, J = 2.8 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.3, 13.5, 17.6, 18.2, 22.2, 22.9, 55.4, 66.3, 117.9, 118.6 (q, J_{CF} = 319 Hz), 122.3, 124.5, 124.6, 125.2, 131.8, 136.6, 138.2, 148.4, 158.2 ; **IR** (neat) 2947, 1566, 1396, 1246, 1215, 1145, 906, 844, 732 cm^{-1}; **HRMS** (ESI): calcd. for $C_{24}H_{37}F_3NaO_5SSi_2$ $[M+Na]^+$: 573.1745; found: 573.1753.

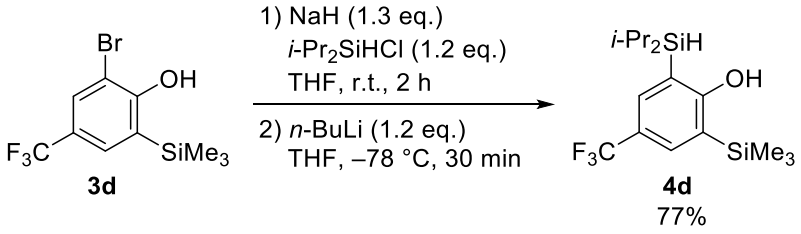
Synthesis of phenol **3d**



To a solution of 2,6-dibromo-4-trifluoromethylphenol (**2d**) (2.05 g, 6.41 mmol) in THF (30 mL) was added NaH (60% dispersion in mineral oil, 308 mg, 7.70 mmol) at 0 °C. After stirring for 30 min at this temperature, Me_3SiCl (891 μL, 7.06 mmol) was added and the mixture was stirred for 3 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.6 M) in hexane (4.41 mL, 7.06 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/20) to afford phenol **3d** (1.82 g, 91%) as colorless oil.

3d: R_f 0.57 (EtOAc/hexane = 1/5); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.33 (s, 9H), 6.03 (s, 1H), 7.51 (d, 1H, J = 2.1 Hz), 7.73 (d, 1H, J = 2.1 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ –1.5, 110.1, 123.6 (q, J_{CF} = 271 Hz), 123.9 (q, J_{CF} = 32 Hz), 128.0, 130.2 (q, J_{CF} = 3.6 Hz), 131.5 (q, J_{CF} = 3.6 Hz), 158.7; **IR** (neat): 3514, 2958, 1597, 1311, 1246, 1122, 906, 840, 732 cm^{-1}; **HRMS** (ESI): calcd. for $C_{10}H_{11}BrF_3OSi$ $[M–H]^–$: 310.9720; found: 310.9726.

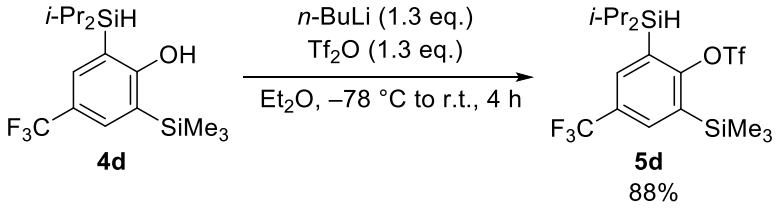
Synthesis of phenol **4d**



To a solution of **3d** (578 mg, 1.84 mmol) in THF (10 mL) was added NaH (60% dispersion in mineral oil, 96 mg, 2.40 mmol) at 0 °C. After stirring for 30 min at this temperature, *i*-Pr_2SiHCl (375 µL, 2.21 mmol) was added and the mixture was stirred for 2 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.60 M) in hexane (1.38 mL, 2.21 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/100) to afford phenol **4d** (496 mg, 77%) as colorless oil.

4d: $\boldsymbol{R_f}$ 0.64 (EtOAc/hexane = 1/20); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.33 (s, 9H), 1.00 (d, 6H, J = 7.5 Hz), 1.10 (d, 6H, J = 7.5 Hz), 1.32 (qqd, 2H, J = 7.5, 7.5, 3.4 Hz), 4.15 (t, 1H, J = 3.4 Hz), 5.86 (s, 1H), 7.52 (d, 1H, J = 2.3 Hz), 7.60 (d, 1H J = 2.3 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ −1.7, 10.1, 18.0, 18.2, 117.4, 122.5 (q, J_{CF} = 31 Hz), 125.1 (q, J_{CF} = 272 Hz), 125.9, 134.34 (q, J_{CF} = 2.4 Hz), 134.43 (q, J_{CF} = 3.6 Hz), 168.6; **IR** (neat): 3529, 2954, 2866, 2013, 1577, 1315, 1149, 1122, 940, 844, 736 cm^{-1}; **HRMS** (ESI): calcd. for $C_{16}H_{26}F_3OSi_2$ $[M–H]^-$: 347.1480; found: 347.1488.

Synthesis of triflate **5d**

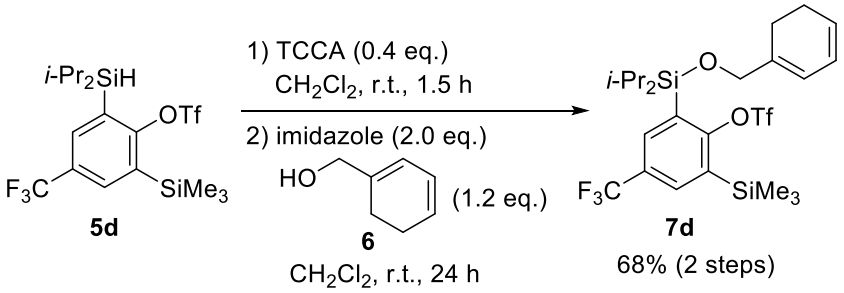


To a solution of **4d** (446 mg, 1.28 mmol) in Et_2O (13 mL) was added was added *n*-BuLi (1.6 M) in hexane (1.04 mL, 1.66 mmol) at –78 °C. After stirring for 30 min at this temperature, Tf_2O (280 µL, 1.66 mmol) was added dropwise to the mixture. The mixture was warmed to room temperature, and the stirring was continued for 4 h. The reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford triflate **5d** (540 mg, 88%) as colorless oil.

5d: $\boldsymbol{R_f}$ 0.48 (hexane); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.41 (s, 9H), 0.95 (d, 6H, J = 7.2 Hz), 1.07 (d, 6H, J = 7.4 Hz), 1.24–1.34 (m, 2H), 4.21–4.24 (m, 1H), 7.70 (d, 1H, J = 2.0 Hz), 7.82 (d, 1H, J = 2.0 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 0.1, 10.7, 18.4, 118.5 (q, J_{CF} = 318 Hz), 123.7 (q, J_{CF} = 272 Hz), 129.4 (q, J_{CF} = 32 Hz),

131.9, 134.3 (q, J_{CF} = 3.6 Hz), 135.1 (q, J_{CF} = 3.6 Hz), 136.6, 157.6; **IR** (neat): 2951, 2866, 2183, 1400, 1319, 1215, 1161, 1134, 1099, 1060, 883, 833, 725 cm^{-1}; **HRMS** (FAB): calcd. for $C_{17}H_{27}F_6O_3SSi_2$ $[M+H]^+$: 481.1118; found: 481.1121.

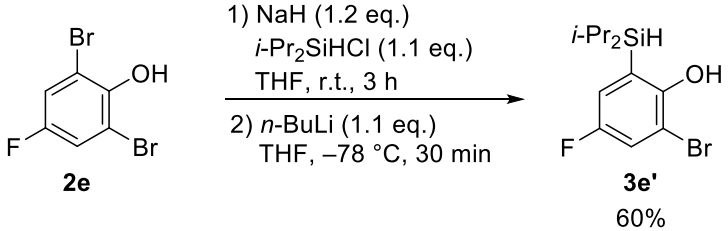
Synthesis of silyl ether **7d**



1) To a solution of **5d** (282 mg, 0.586 mmol) in CH_2Cl_2 (6 mL) was added trichloroisocyanuric acid[2] (TCCA, 54.4 mg, 0.234 mmol). After stirring for 1.5 h at room temperature, the reaction mixture was filtered through a Celite® pad (washed with hexane), and the filtrate was concentrated in vacuo to afford the corresponding silyl chloride as colorless oil. This material was employed in the next reaction without further purification.
2) To a solution of the crude material (*vide supra*) in CH_2Cl_2 (6 mL) were added imidazole (79.8 mg, 1.17 mmol) and alcohol **6**[3] (77.5 mg, 0.703 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford silyl ether **7d** (236 mg, 68% in 2 steps) as colorless oil.

7d: $\boldsymbol{R_f}$ 0.45 (EtOAc/hexane = 1/20); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.40 (s, 9H), 1.01 (d, 6H, *J* = 7.6 Hz), 1.16 (d, 6H, *J* = 7.6 Hz), 1.42 (qq, 2H, *J* = 7.6, 7.6 Hz), 2.09 (brt, 2H, *J* = 8.9 Hz), 2.18–2.24 (m, 2H), 4.15 (s, 2H), 5.73–5.76 (m, 1H), 5.92–5.97 (m, 2H), 7.82 (d, 1H, *J* = 2.1 Hz), 7.91 (d, 1H, *J* = 2.1 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.4, 13.3, 17.4, 17.9, 22.2, 22.8, 66.7, 118.57, 118.60 (q, J_{CF} = 321 Hz), 124.1 (q, J_{CF} = 273 Hz), 124.4, 125.5, 129.7 (q, J_{CF} = 32 Hz), 132.3, 135.7 (q, J_{CF} = 2.9 Hz), 136.3 (q, J_{CF} = 2.9 Hz), 136.7, 137.5, 157.6; **IR** (neat) 2951, 2870, 1404, 1319, 1215, 1134, 1099, 883, 833, 729 cm^{-1}; **HRMS** (ESI): calcd. for $C_{24}H_{34}F_6NaO_4SSi_2$ $[M+Na]^+$: 611.1513; found: 611.1524.

Synthesis of phenol **3e'**

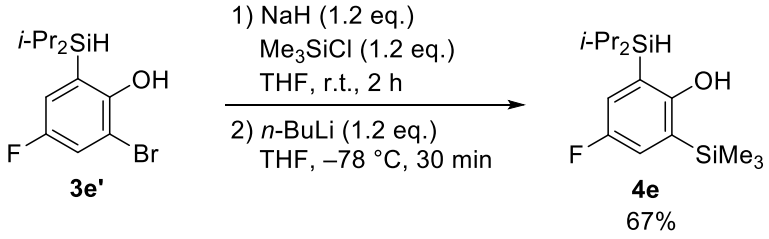


To a solution of 2,6-dibromo-4-fluorophenol (**2e**) (1.56 g, 5.78 mmol) in THF (20 mL) was added NaH (60%

dispersion in mineral oil, 277 mg, 6.93 mmol) at 0 °C. After stirring for 30 min at this temperature, *i*-Pr_2SiHCl (1.08 mL, 6.35 mmol) was added and the mixture was stirred for 3 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.6 M) in hexane (3.97 mL, 6.35 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford phenol **3e'** (1.06 g, 60%) as colorless oil.

3e': R_f 0.63 (EtOAc/hexane = 1/10); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.98 (d, 6H, J = 7.4 Hz), 1.07 (d, 6H, J = 7.5 Hz), 1.33 (qqd, 2H, J = 7.5, 7.4, 3.7 Hz), 3.89 (t, 1H, J = 3.7 Hz), 5.52 (s, 1H), 7.08 (dd, 1H, J_{HF} = 7.7, J = 3.2 Hz), 7.22 (dd, 1H, J_{HF} = 7.5, J = 3.2 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 10.8, 18.8, 109.3 (d, J_{CF} = 8.4 Hz), 119.8 (d, J_{CF} = 26 Hz), 123.1 (d, J_{CF} = 20 Hz), 152.2, 155.8 (d, J_{CF} = 244 Hz) (several signals overlapped); **IR** (neat): 3525, 2943, 2862, 2102, 1446, 1400, 1195, 906 cm^{-1}; **HRMS** (ESI): calcd. for $C_{12}H_{17}BrFOSi$ $[M–H]^-$: 303.0222; found 303.0219.

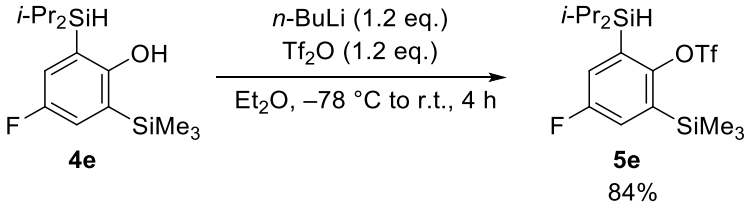
Synthesis of phenol **4e**



To a solution of **3e'** (665 mg, 2.18 mmol) in THF (8 mL) was added NaH (60% dispersion in mineral oil, 105 mg, 2.61 mmol) at 0 °C. After stirring for 30 min at this temperature, Me_3SiCl (330 µL, 2.61 mmol) was added and the mixture was stirred for 2 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.60 M) in hexane (1.63 mL, 2.61 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford phenol **4e** (435 mg, 67%) as colorless oil.

4e: R_f 0.39 (hexane); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.30 (s, 9H), 1.00 (d, 6H, J = 7.5 Hz), 1.10 (d, 6H, J = 7.2 Hz), 1.28 (qqd, 2H, J = 7.5, 7.2, 3.2 Hz), 4.08 (t, 1H, J = 3.2 Hz), 5.32 (s, 1H), 6.94 (dd, 1H, J_{HF} = 8.0, J = 2.9 Hz), 7.04 (dd, 1H, J_{HF} = 8.3, J = 2.9 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ –1.1, 10.5, 18.4, 18.6, 118.7 (d, J_{CF} = 2.4 Hz), 122.2 (d, J_{CF} = 20 Hz), 122.8 (d, J_{CF} = 20 Hz), 127.3 (d, J_{CF} = 3.6 Hz), 156.9 (d, J_{CF} = 240 Hz), 161.3; **IR** (neat): 3552, 2951, 2866, 2067, 1396, 1195, 906, 840 cm^{-1}; **HRMS** (ESI): calcd. for $C_{15}H_{26}FOSi_2$ $[M–H]^-$: 297.1512; found: 297.1509.

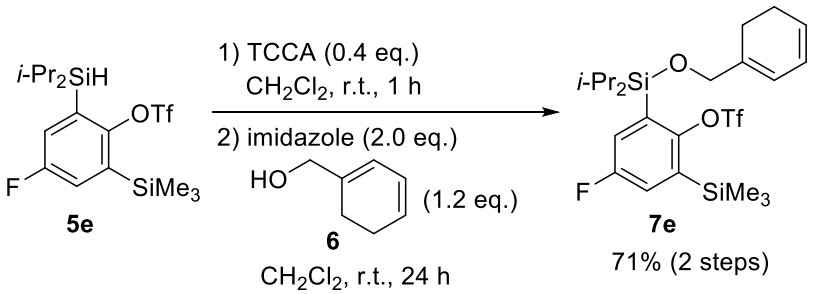
Synthesis of triflate **5e**



To a solution of **4e** (552 mg, 1.85 mmol) in Et_2O (20 mL) was added was added *n*-BuLi (1.6 M) in hexane (1.38 mL, 2.22 mmol) at –78 °C. After stirring for 30 min at this temperature, Tf_2O (373 µL, 2.22 mmol) was added dropwise to the mixture. The mixture was warmed to room temperature, and the stirring was continued for 4 h. The reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford triflate **5e** (671 mg, 84%) as colorless oil.

5e: R_f 0.47 (hexane); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.38 (s, 9H), 0.96 (d, 6H, J = 7.5 Hz), 1.07 (d, 6H, J = 7.2 Hz), 1.20–1.28 (m, 2H), 4.17 (brs, 1H), 7.13 (dd, 1H, J_{HF} = 7.5, J = 3.2 Hz), 7.24 (dd, 1H, J_{HF} = 8.1, J = 3.2 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 0.1, 10.8, 18.5, 118.5 (q, J_{CF} = 317 Hz), 123.5 (d, J_{CF} = 22 Hz), 124.2 (d, J_{CF} = 22 Hz), 133.4 (d, J_{CF} = 4.8 Hz), 138.3 (d, J_{CF} = 3.6 Hz), 150.5, 160.9 (d, J_{CF} = 251 Hz); **IR** (neat): 2954, 2183, 1400, 1219, 1141, 906, 848 cm^{-1}; **HRMS** (FAB): calcd. for $C_{16}H_{26}F_4NaO_3Si_2$ $[M+Na]^+$: 453.0970; found: 453.0969.

Synthesis of silyl ether **7e**

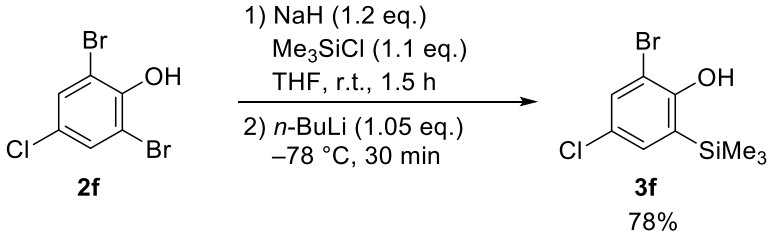


1) To a solution of **5e** (420 mg, 0.975 mmol) in CH_2Cl_2 (10 mL) was added trichloroisocyanuric acid[2] (TCCA, 90.7 mg, 0.390 mmol). After stirring for 1 h at room temperature, the reaction mixture was filtered through a Celite® pad (washed with hexane), and the filtrate was concentrated in vacuo to afford the corresponding silyl chloride as colorless oil. This material was employed in the next reaction without further purification.

2) To a solution of the crude material (*vide supra*) in CH_2Cl_2 (10 mL) were added imidazole (133 mg, 1.95 mmol) and alcohol **6**[3] (129 mg, 1.17 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford silyl ether **7e** (372 mg, 71% in 2 steps) as colorless oil.

7e: $\boldsymbol{R}_f$ 0.30 (hexane); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.36 (s, 9H), 1.02 (d, 6H, J = 7.6 Hz), 1.15 (d, 6H, J = 7.6 Hz), 1.39 (qq, 2H, J = 7.6, 7.6 Hz), 2.11 (brt, 2H, J = 8.9 Hz), 2.19–2.24 (m, 2H), 4.18 (s, 2H), 5.73–5.77 (m, 1H), 5.93–5.97 (m, 2H), 7.25 (dd, 1H, J_{HF} = 7.6, J = 2.8 Hz), 7.33 (dd, 1H, J_{HF} = 7.6, J = 2.8 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.4, 13.3, 17.5, 17.9, 22.3, 22.9, 66.7, 118.4, 118.6 (q, J_{CF} = 319 Hz), 124.5, 124.8 (d, J_{CF} = 22 Hz), 125.2 (d, J_{CF} = 22 Hz), 125.4, 134.1 (d, J_{CF} = 2.9 Hz), 137.8, 138.5 (d, J_{CF} = 2.9 Hz), 150.3, 161.6 (d, J_{CF} = 253 Hz); **IR** (neat): 2951, 2870, 1566, 1400, 1365, 1215, 1141, 1053, 906, 840, 732 cm^{-1}; **HRMS** (ESI): calcd. for $C_{23}H_{34}F_4NaO_4SSi_2$ $[M+Na]^+$: 561.1545; found: 561.1552.

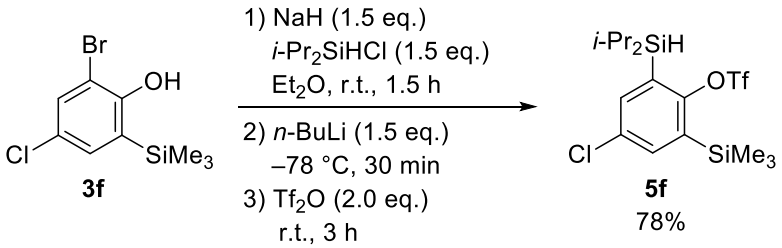
Synthesis of phenol **3f**



To a solution of 2,6-dibromo-4-chlorophenol (**2f**) (2.99 g, 10.4 mmol) in THF (30 mL) was added NaH (60% dispersion in mineral oil, 501 mg, 12.5 mmol) at 0 °C. After stirring for 30 min at this temperature, Me_3SiCl (1.45 mL, 11.49 mmol) was added dropwise and the mixture was stirred for 1.5 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (2.30 M) in hexane (4.77 mL, 11.0 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford phenol **3f** (2.29 g, 78%) as a white solid.

3f: $\boldsymbol{R}_f$ 0.50 (hexane); Spectral data matched those reported in the literature.[4]

Synthesis of triflate **5f**

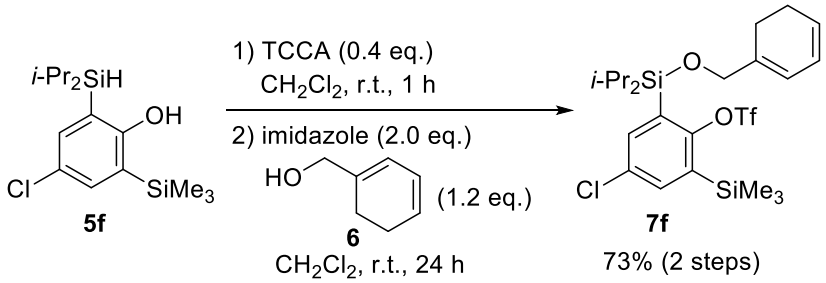


To a solution of **3f** (537 mg, 1.92 mmol) in Et_2O (20 mL) was added NaH (60% dispersion in mineral oil, 115 mg, 2.88 mmol) at 0 °C. After stirring for 30 min at this temperature, *i*-Pr_2SiHCl (488 µL, 2.88 mmol) was added and the mixture was stirred for 1.5 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.60 M) in hexane (1.80 mL, 2.88 mmol) over 5 min. After stirring for 30 min at –78 °C, Tf_2O (647 µl, 3.84 mmol) was added and the the mixture was warmed to room temperature and stirred for 3 h. The reaction was quenched by adding saturated aqueous $NaHCO_3$,

and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford triflate **5f** (672 mg, 78%) as colorless oil.

5f: $\boldsymbol{R}_f$ 0.50 (hexane); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.38 (s, 9H), 0.96 (d, 6H, J = 7.5 Hz), 1.07 (d, 6H, J = 7.2 Hz), 1.21–1.29 (m, 2H), 4.15–4.18 (m, 1H), 7.40 (d, 1H, J = 2.9 Hz), 7.51 (d, 1H, J = 2.9 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 0.1, 10.8, 18.4, 118.5 (q, J_{CF} = 318 Hz), 130.0, 133.9, 136.8, 137.69, 137.73, 153.6; **IR** (neat): 2951, 2866, 2183, 1400, 1365, 1211, 1138, 1060, 879, 732 cm^{-1}; **HRMS** (FAB): calcd. for $C_{16}H_{27}ClF_3O_3SSi_2$ $[M+H]^+$: 447.0855; found: 447.0865.

Synthesis of silyl ether **7f**

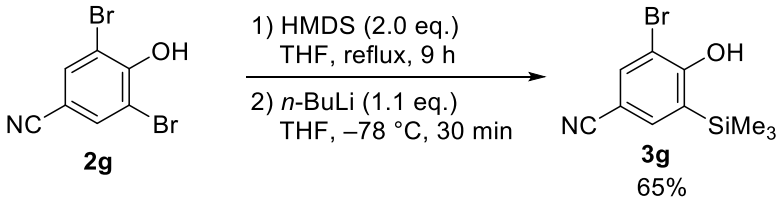


1) To a solution of **5f** (127 mg, 0.284 mmol) in CH_2Cl_2 (3 mL) was added trichloroisocyanuric acid[2] (TCCA, 26.4 mg, 0.113 mmol). After stirring for 1 h at room temperature, the reaction mixture was filtered through a Celite® pad (washed with hexane), and the filtrate was concentrated in vacuo to afford the corresponding silyl chloride as colorless oil. This material was employed in the next reaction without further purification.

2) To a solution of the crude material (*vide supra*) in CH_2Cl_2 (3 mL) were added imidazole (38.7 mg, 0.568 mmol) and alcohol **6**[3] (37.5 mg, 0.341 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford silyl ether **7f** (114 mg, 73% in 2 steps) as colorless oil.

7f: $\boldsymbol{R}_f$ 0.27 (EtOAc/hexane = 1/100); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.37 (s, 9H), 1.01 (d, 6H, J = 7.6 Hz), 1.15 (d, 6H, J = 7.6 Hz), 1.39 (qq, 2H, J = 7.6, 7.6 Hz), 2.11 (brt, 2H, J = 9.6 Hz), 2.20–2.24 (m, 2H), 4.16 (s, 2H), 5.73–5.77 (m, 1H), 5.92–5.97 (m, 2H), 7.51 (d, 1H, J = 2.8 Hz), 7.59 (d, 1H, J = 2.8 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.4, 13.4, 17.4, 18.0, 22.3, 22.9, 66.7, 118.58, 118.59 (q, J_{CF} = 319 Hz), 124.5, 125.4, 133.4, 134.3, 137.7, 137.9, 138.3, 138.6, 153.6; **IR** (neat) 2951, 2870, 1400, 1211, 1138, 1049, 875, 840, 732 cm^{-1}; **HRMS** (ESI): calcd. for $C_{23}H_{34}ClF_3KO_4Ssi_2$ $[M+K]^+$: 593.0989; found: 593.0987.

Synthesis of phenol **3g**

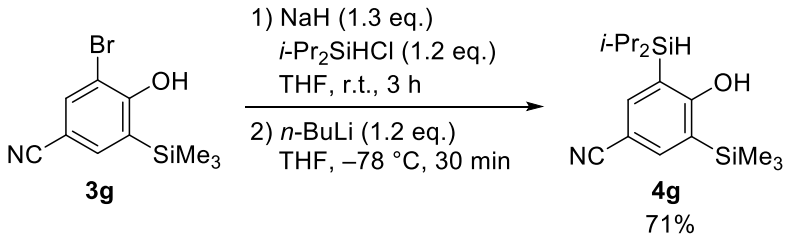


1) To a solution of 2,6-dibromo-4-cyanophenol (**2g**) (1.48 g, 5.45 mmol) in THF (6 mL) was added 1,1,1,3,3,3-hexamethyldisilazane (HMDS, 2.28 mL, 10.9 mmol) at room temperature and the mixture was refluxed for 9 h. After cooling to room temperature, the solvent and HMDS were removed under reduced pressure to afford the corresponding silyl ether as colorless oil. This material was employed in the next reaction without further purification.

2) To a stirred solution of the crude material (*vide supra*) in THF (27 mL) was added dropwise *n*-BuLi (1.60 M in hexanes, 3.74 mL, 5.99 mmol) at –78 °C. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/10) to afford phenol **3g** (1.01 g, 65%) as a white solid.

3g: **mp**: 113–120 °C; $\boldsymbol{R_f}$ 0.38 (EtOAc/hexane = 1/5); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.32 (s, 9H), 6.20 (s, 1H), 7.57 (d, 1H, J = 1.6 Hz), 7.78 (d, 1H, J = 1.6 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ –1.5, 105.4, 110.4, 118.0, 128.9, 136.4, 138.7, 159.6; **IR** (neat): 3402, 2958, 2225, 1577, 1442, 1242, 1138, 1080, 902, 840, 729 cm^{-1}; **HRMS** (ESI): calcd. for $C_{10}H_{11}BrNOSi$ $[M–H]^–$: 267.9799; found: 267.9795.

Synthesis of phenol **4g**

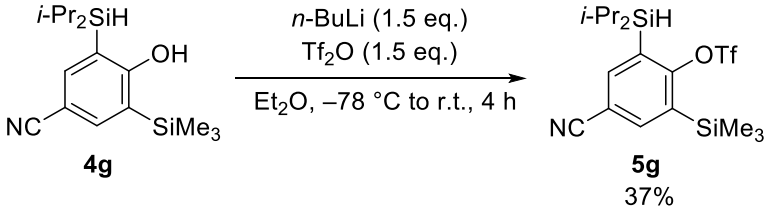


To a solution of **3g** (502 mg, 1.86 mmol) in THF (10 mL) was added NaH (60% dispersion in mineral oil, 97 mg, 2.42 mmol) at 0 °C. After stirring for 30 min at this temperature, *i*-Pr_2SiHCl (390 µL, 2.23 mmol) was added and the mixture was stirred for 3 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.60 M) in hexane (1.39 mL, 2.23 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/50) to afford phenol **4g** (402 mg, 71%) as colorless oil.

4g: $\boldsymbol{R_f}$ 0.58 (EtOAc/hexane = 1/20); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.32 (s, 9H), 0.99 (d, 6H, J = 7.5 Hz), 1.10 (d, 6H, J = 7.4 Hz), 1.31 (qqd, 2H, J = 7.5, 7.4, 3.5 Hz), 4.14 (t, 1H, J = 3.5 Hz), 6.04 (s, 1H), 7.58 (d,

1H, J = 2.1 Hz), 7.65 (d, 1H J = 2.1 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ –1.8, 9.9, 17.8, 18.1, 104.2, 118.6, 120.1, 127.1, 141.3, 141.5, 169.0; **IR** (neat): 3525, 2951, 2866, 2222, 2067, 1562, 1404, 1246, 1006, 906, 840, 729 cm^{-1}; **HRMS** (ESI): calcd. for $C_{16}H_{26}NOSi_2$ [M–H]$^-$: 304.1558; found: 304.1567.

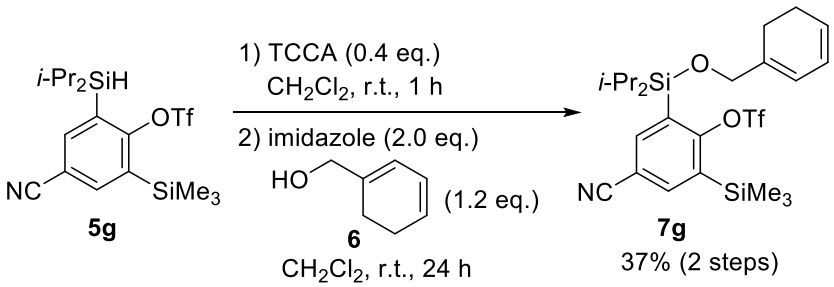
Synthesis of triflate **5g**



To a solution of **4g** (222 mg, 0.725 mmol) in Et_2O (8 mL) was added was added *n*-BuLi (1.6 M) in hexane (473 µL, 1.09 mmol) at –78 °C. After stirring for 30 min at this temperature, Tf_2O (183 µL, 1.09 mmol) was added dropwise to the mixture. The mixture was warmed to room temperature, and the stirring was continued for 4 h. The reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/30) and gel-permeation chromatography [YMC-GPC T4000® (2.0 cm φ×60 cm) + T2000® (2.0 cm φ × 60 cm), $CHCl_3$, flow rate 8.0 mL/min] to afford triflate **5g** (117 mg, 37%) as colorless oil.

5g: $\boldsymbol{R_f}$ 0.19 (EtOAc/hexane = 1/20); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.40 (s, 9H), 0.95 (d, 6H, J = 7.6 Hz), 1.07 (d, 6H, J = 7.6 Hz), 1.24–1.30 (m, 2H), 4.19–4.22 (m, 1H), 7.74 (d, 1H, J = 2.8 Hz), 7.87 (d, 1H, J = 2.8 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.5, 10.2, 17.9, 18.0, 112.5, 118.2, 118.6 (q, J_{CF} = 319 Hz), 133.1, 137.9, 141.1, 142.2, 158.2; **IR** (neat): 2954, 2866, 2233, 1400, 1219, 1138, 1064, 906, 844, 732 cm^{-1}; **HRMS** (FAB): calcd. for $C_{17}H_{27}F_3NO_3SSi_2$ [M+H]$^+$: 438.1202; found: 438.1211.

Synthesis of silyl ether **7g**



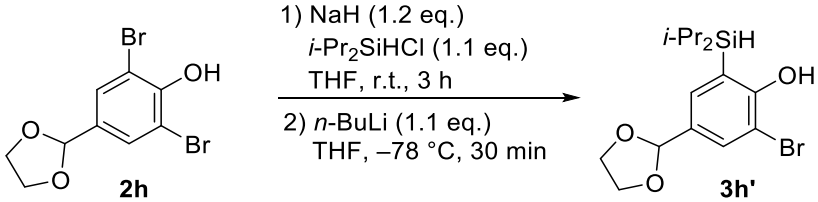
1) To a solution of **5g** (79.5 mg, 0.182 mmol) in CH_2Cl_2 (2 mL) was added trichloroisocyanuric acid[2] (TCCA, 16.9 mg, 0.0727 mmol). After stirring for 1 h at room temperature, the reaction mixture was filtered through a Celite® pad (washed with hexane), and the filtrate was concentrated in vacuo to afford the corresponding silyl chloride as colorless oil. This material was employed in the next reaction without further purification.

2) To a solution of the crude material (*vide supra*) in CH_2Cl_2 (2 mL) were added imidazole (24.8 mg, 0.364 mmol) and alcohol **6**[3] (24.1 mg, 0.218 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction

was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/30) and further purified by PTLC (hexane/EtOAc = 1/50 x2) to afford silyl ether **7g** (37.0 mg, 37% in 2 steps) as colorless oil.

7g: $\boldsymbol{R_f}$ 0.63 (EtOAc/hexane = 1/5); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.39 (s, 9H), 1.00 (d, 6H, J = 7.6 Hz), 1.15 (d, 6H, J = 7.6 Hz), 1.40 (qq, 2H, J = 7.6, 7.6 Hz), 2.12 (brt, 2H, J = 8.9 Hz), 2.21–2.25 (m, 2H), 4.18 (s, 2H), 5.74–5.78 (m, 1H), 5.90–5.97 (m, 2H), 7.87 (d, 1H, J = 2.7 Hz), 7.93 (d, 1H, J = 2.7 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.4, 13.2, 17.3, 17.8, 22.2, 22.9, 66.9, 112.4, 118.2, 118.6 (q, J_{CF} = 319 Hz), 118.9, 124.4, 125.7, 133.3, 137.3, 137.6, 142.4, 142.6, 157.9; **IR** (neat) 2951, 2870, 2233, 1404, 1369, 1215, 1138, 1053, 906, 841, 733 cm^{-1}; **HRMS** (ESI): calcd. for $C_{24}H_{34}F_3KNO_4SSi_2$ $[M+K]^+$: 584.1331; found 584.1357.

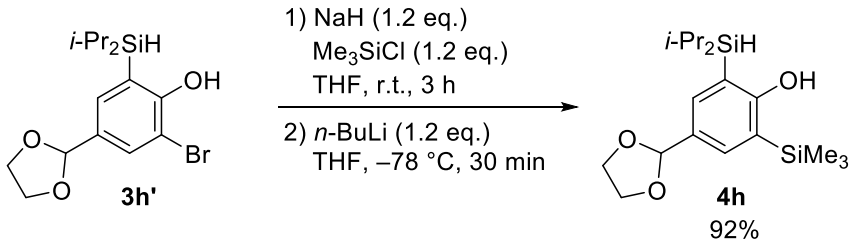
Synthesis of phenol **3h'**



To a solution of **2h** (1.51 g, 4.67 mmol) in THF (12 mL) was added NaH (60% dispersion in mineral oil, 244 mg, 5.60 mmol) at 0 °C. After stirring for 30 min at this temperature, i-Pr_2SiHCl (1.08 mL, 6.35 mmol) was added and the mixture was stirred for 3 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of n-BuLi (1.6 M) in hexane (3.19 mL, 5.11 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/10) to afford phenol **3h'** (1.45 g, 85%) as a white solid.

3h': **mp**: 61–63 °C; $\boldsymbol{R_f}$ 0.35 (EtOAc/hexane = 1/5); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.97 (d, 6H, J = 7.5 Hz), 1.07 (d, 6H, J = 7.5 Hz), 1.33 (qqd, 2H, J = 7.5, 7.5, 3.7 Hz), 3.92 (t, 1H, J = 3.7 Hz), 4.01–4.04 (m, 2H), 4.12–4.15 (m, 2H), 5.71 (s, 1H), 5.79 (s, 1H), 7.43 (d, 1H, J = 2.0 Hz), 7.63 (d, 1H, J = 2.0 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 10.8, 18.9, 65.2, 102.9, 110.3, 121.8, 131.2, 131.6, 135.9, 156.6; **IR** (neat): 3514, 2943, 2862, 2012, 1361, 1238, 1087, 906, 790 cm^{-1}; **HRMS** (ESI): calcd. for $C_{15}H_{22}BrO_3Si$ $[M–H]^-$: 357.0522; found: 357.0520.

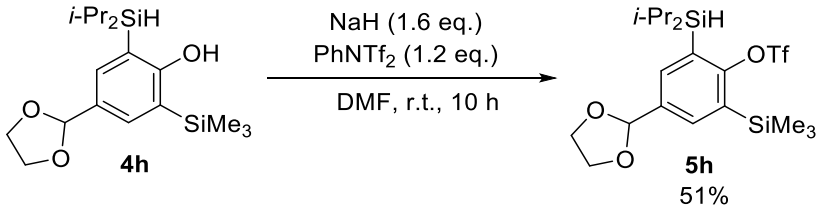
Synthesis of phenol **4h**



To a solution of **3h'** (1.41 g, 3.93 mmol) in THF (12 mL) was added NaH (60% dispersion in mineral oil, 105 mg, 2.61 mmol) at 0 °C. After stirring for 30 min at this temperature, Me_3SiCl (596 µL, 4.71 mmol) was added and the mixture was stirred for 3 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.6 M) in hexane (2.95 mL, 4.71 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/20) to afford phenol **4h** (1.28 g, 92%) as colorless oil.

4h: R_f 0.44 (EtOAc/hexane = 1/5); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.30 (s, 9H), 0.99 (d, 6H, *J* = 7.5 Hz), 1.09 (d, 6H, *J* = 7.2 Hz), 1.25‑1.32 (m, 2H), 4.01–4.03 (m, 2H), 4.11–4.16 (m, 3H), 5.60 (s, 1H), 5.72 (s, 1H), 7.38 (d, 1H, *J* = 2.3 Hz), 7.50 (d, 1H, *J* = 2.3 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ –1.0, 10.6, 18.5, 18.7, 65.2, 104.1, 116.4, 124.9, 128.6, 135.3, 135.8, 166.6; **IR** (neat): 3537, 2951, 2866, 2059, 1577, 1411, 1365, 1246, 1087, 906, 837, 790 cm^{-1}; **HRMS** (ESI): calcd. for $C_{18}H_{31}O_3Si_2$ $[M-H]^-$: 351.1812; found: 351.1809.

Synthesis of triflate **5h**

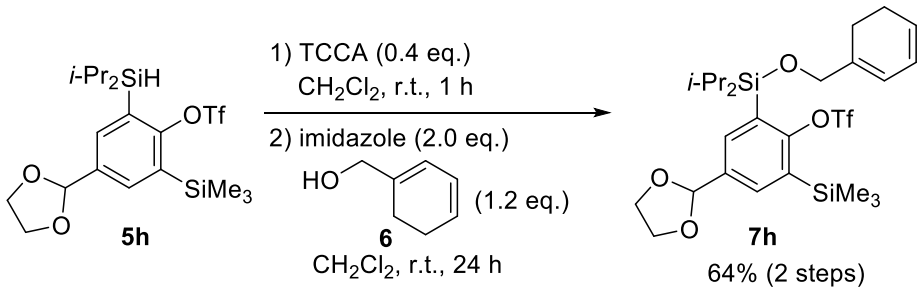


To a solution of **4h** (652 mg, 1.85 mmol) in DMF (12 ml) was added NaH (60% dispersion in mineral oil, 118 mg, 2.96 mmol) at 0 °C and the mixture was then stirred for 30 min. $PhNTf_2$ (793 mg, 2.22 mmol) was added and the mixture was warmed to room temperature. After stirring for 10 h, the reaction was quenched by adding water, and the reaction mixture was extracted with EtOAc/Hexane = 1/4 (x3). The combined organic layer was washed with water (x3), brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 1/50) to afford triflate **5h** (454 mg, 51%) as colorless oil.

5h: R_f 0.44 (EtOAc/hexane = 1/5); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.38 (s, 9H), 0.95 (d, 6H, *J* = 7.5 Hz), 1.06 (d, 6H, *J* = 7.2 Hz), 1.23–1.29 (m, 2H), 4.04–4.14 (m, 4H), 4.20 (brs, 1H), 5.84 (s, 1H), 7.57 (d, 1H, *J* = 2.0 Hz), 7.69 (d, 1H, *J* = 2.0 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 0.2, 10.9, 18.5, 18.6, 63.1, 102.8, 118.5

(q, J_{CF} = 318 Hz), 130.2, 135.0, 135.7, 136.4, 136.5, 156.1; **IR** (neat): 2954, 2886, 1396, 1215, 1138, 906, 844 cm^{-1}; **HRMS** (FAB): calcd. for $C_{19}H_{32}F_3O_5SSi_2$ $[M+H]^+$: 485.1456; found: 485.1467.

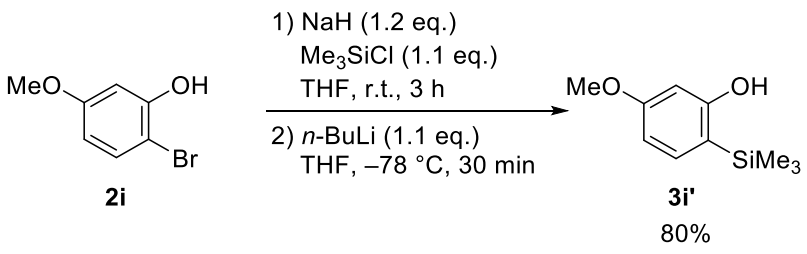
Synthesis of silyl ether **7h**



1) To a solution of **5h** (246 mg, 0.508 mmol) in CH_2Cl_2 (5 mL) was added trichloroisocyanuric acid[2] (TCCA, 47.2 mg, 0.203 mmol). After stirring for 1 h at room temperature, the reaction mixture was filtered through a Celite® pad (washed with hexane), and the filtrate was concentrated in vacuo to afford the corresponding silyl chloride as colorless oil. This material was employed in the next reaction without further purification.
2) To a solution of the crude material (*vide supra*) in CH_2Cl_2 (5 mL) were added imidazole (67.2 mg, 1.02 mmol) and alcohol **6**[3] (69.2 mg, 0.610 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/20) to afford silyl ether **7h** (192 mg, 64% in 2 steps) as colorless oil.

7h: R_f 0.38 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.36 (s, 9H), 1.01 (d, 6H, J = 7.6 Hz), 1.16 (d, 6H, J = 7.6 Hz), 1.40 (qq, 2H, J = 7.6, 7.6 Hz), 2.08 (brt, 2H, J = 10.3 Hz), 2.18–2.23 (m, 2H), 4.00–4.07 (m, 2H), 4.10–4.14 (m, 4H), 5.72–5.75 (m, 1H), 5.80 (s, 1H), 5.94–6.00 (m, 2H), 7.69 (d, 1H, J = 2.8 Hz), 7.76 (d, 1H, J = 2.8 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.3, 13.5, 17.6, 18.1, 22.3, 22.9, 65.4, 66.4, 103.2, 118.0, 118.6 (q, J_{CF} = 321 Hz), 124.6, 125.1, 130.6, 135.3, 136.9, 137.2, 137.8, 138.2, 156.2; **IR** (neat) 2951, 2870, 1573, 1400, 1357, 1249, 1215, 1138, 1091, 906, 840, 732 cm^{-1}; **HRMS** (ESI): calcd. for $C_{26}H_{39}F_3KO_6SSi_2$ $[M+K]^+$: 631.1590; found: 631.1604.

Synthesis of phenol **3i'**

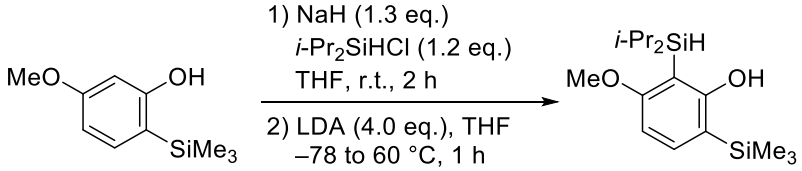


To a solution of 2-bromo-5-methoxyphenol (**2i**) (1.53 g, 7.53 mmol) in THF (23 mL) was added NaH (60% dispersion in mineral oil, 391 mg, 9.78 mmol) at 0 °C. After stirring for 30 min at this temperature, Me_3SiCl

(1.14 mL, 9.03 mmol) was added and the mixture was stirred for 3 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.60 M) in hexane (5.64 mL, 9.03 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/4) to afford phenol **3i'** (1.18 g, 80%) as a white solid.

3i': **mp**: 81–85 °C; $\boldsymbol{R_f}$ 0.45 (EtOAc/hexane = 1/3); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.28 (s, 9H), 3.78 (s, 3H), 4.86 (s, 1H), 6.27 (d, 1H, J = 2.0 Hz), 6.50 (dd, 1H, J = 8.3, 2.0 Hz), 7.25 (d, 1H J = 8.3 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –1.3, 55.0, 101.3, 106.0, 116.9, 136.5, 162.1, 162.5; **IR** (neat): 3375, 2954, 1600, 1504, 1404, 1269, 1199, 1161, 1080, 906, 840, 732 cm^{-1}; **HRMS** (FAB): calcd. for $C_{10}H_{17}O_2Si$ $[M+H]^+$: 197.0992; found: 197.0994.

Synthesis of phenol **4i**

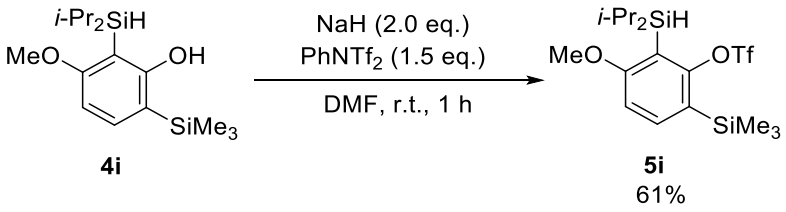


3i' **4i** 62%

To a solution of **3i'** (496 mg, 2.53 mmol) in THF (13 mL) was added NaH (60% dispersion in mineral oil, 131 mg, 3.28 mmol) at 0 °C. After stirring for 30 min at this temperature, *i*-Pr_2SiHCl (513 μL, 3.03 mmol) was added and the mixture was stirred for 2 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of LDA (1.00 M) in THF (10.1 mL, 10.1 mmol) over 5 min. The mixture was warmed to 60 °C and stirred for 1 h. The reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/100) to afford phenol **4i** (487 mg, 62%) as colorless oil.

4i: $\boldsymbol{R_f}$ 0.72 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.26 (s, 9H), 0.95 (d, 6H, J = 7.6 Hz), 1.08 (d, 6H, J = 7.6 Hz), 1.35 (qqd, 2H, J = 7.6, 7.6, 3.4 Hz), 3.75 (s, 3H), 4.04 (t, 1H, J = 3.4 Hz), 6.17 (s, 1H), 6.39 (d, 1H, J = 8.2 Hz), 7.34 (d, 1H, J = 8.2 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –1.3, 10.6, 18.7, 19.0, 54.7,101.4, 104.8, 117.4, 138.6, 166.6, 167.8; **IR** (neat): 3518, 2943, 2862, 2040, 1562, 1462, 1373, 1273, 1203, 1118, 1080, 1010, 906, 837, 794, 732 cm^{-1}; **HRMS** (ESI): calcd. for $C_{16}H_{29}O_2Si_2$ $[M–H]^–$: 309.1712; found: 309.1706.

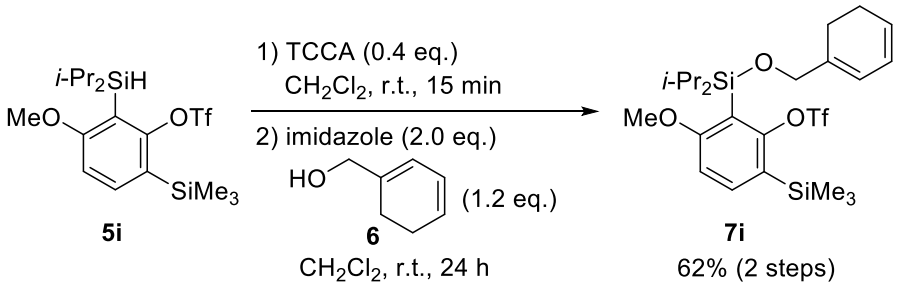
Synthesis of triflate **5i**



To a solution of **4i** (487 mg, 1.57 mmol) in DMF (16 ml) was added NaH (60% dispersion in mineral oil, 125 mg, 3.13 mmol) at 0 °C and the mixture was then stirred for 30 min. $PhNTf_2$ (840 mg, 2.35 mmol) was added and the mixture was warmed to room temperature. After stirring for 1 h. The reaction was quenched by adding water, and the reaction mixture was extracted with EtOAc/Hexane = 1/4 (x3). The combined organic layer was washed with water (x3), brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 1/30) to afford triflate **5i** (419 mg 61%) as a white solid.

5i: **mp**: 34–37 °C; $\boldsymbol{R}_f$ 0.45 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.33 (s, 9H), 0.89 (d, 6H, J = 7.6 Hz), 1.08 (d, 6H, J = 7.6 Hz), 1.25–1.32 (m, 2H), 3.82 (s, 3H), 4.02–4.04 (m, 1H), 6.86 (d, 1H, J = 8.2), 7.54 (d, 1H, J = 8.2 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.1, 11.1, 18.97, 19.05, 55.2, 109.4, 118.8 (q, J_{CF} = 319 Hz), 120.1, 126.2, 139.9, 156.6, 166.3; **IR** (neat): 2947, 2164, 1581, 1458, 1396, 1350, 1249, 1211, 1138, 1045, 916, 844, 736 cm^{-1}; **HRMS** (FAB): calcd. for $C_{17}H_{30}F_3O_4SSi_2$ $[M+H]^+$: 443.1350; found: 443.1354.

Synthesis of silyl ether **7i**



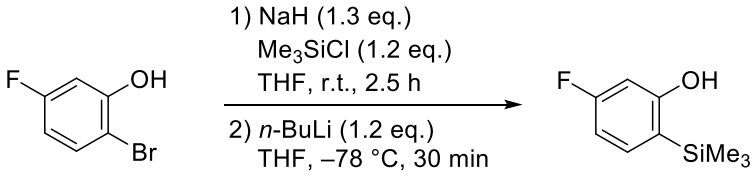
1) To a solution of **5i** (173 mg, 0.392 mmol) in CH_2Cl_2 (4 mL) was added trichloroisocyanuric acid[2] (TCCA, 36.4 mg, 0.157 mmol). After stirring for 15 min at room temperature, the reaction mixture was filtered through a Celite® pad (washed with hexane), and the filtrate was concentrated in vacuo to afford the corresponding silyl chloride as colorless oil. This material was employed in the next reaction without further purification.

2) To a solution of the crude material (*vide supra*) in CH_2Cl_2 (4 mL) were added imidazole (53.3 mg, 0.784 mmol) and alcohol **6**[3] (52.0 mg, 0.470 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/30) to afford silyl ether 7**i** (134 mg,

62% in 2 steps) as colorless oil.

7i: $\boldsymbol{R}_f$ 0.52 (EtOAc/hexane = 1/20); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.33 (s, 9H), 0.98 (d, 6H, J = 7.6 Hz), 1.06 (d, 6H, J = 7.6 Hz), 1.42 (qq, 2H, J = 7.6, 7.6 Hz), 2.11 (brt, 2H, J = 9.0 Hz), 2.17–2.22 (m, 2H), 3.79 (s, 3H), 4.22 (s, 2H), 5.69–5.72 (m, 1H), 5.89–5.94 (m, 2H), 6.85 (d, 1H, J = 8.3 Hz), 7.53 (d, 1H, J = 8.3 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 0.0, 14.0, 17.6, 18.2, 22.3, 22.8, 55.0, 66.3, 109.1, 117.9, 118.7 (q, J_{CF} = 321 Hz), 119.5, 124.7, 124.8, 126.2, 138.4, 139.7, 157.4, 166.3; **IR** (neat) 2947, 2866, 1577, 1396, 1203, 1138, 1091, 1045, 906, 837, 810, 779 cm^{-1}; **HRMS** (ESI): calcd. for $C_{24}H_{37}F_3NaO_5SSi_2$ $[M+Na]^+$: 573.1745; found: 573.1731.

Synthesis of phenol **3j'**

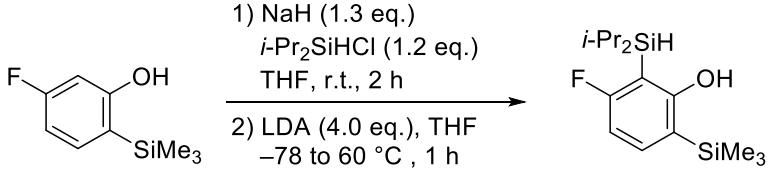


2j **3j'** 97%

To a solution of 2-bromo-5-fluorophenol (**2j**) (2.0 ml, 18.0 mmol) in THF (54 mL) was added NaH (60% dispersion in mineral oil, 935 mg, 23.4 mmol) at 0 °C. After stirring for 30 min at this temperature, Me_3SiCl (2.73 mL, 21.6 mmol) was added and the mixture was stirred for 2.5 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of *n*-BuLi (1.60 M) in hexane (13.5 mL, 21.6 mmol) over 5 min. After stirring for 30 min at –78 °C, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/10) to afford phenol **3j'** (3.20 g, 97%) as colorless oil.

3j': $\boldsymbol{R}_f$ 0.43 (EtOAc/hexane = 1/5); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.29 (s, 9H), 5.07 (s, 1H), 6.41 (dd, 1H, J_{HF} = 10.3, J = 2.0 Hz), 6.63 (ddd, 1H, J_{HF} = 8.2, J = 8.2, 2.0 Hz), 6.41 (dd, 1H, J = 8.2 , J_{HF} = 7.6 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –1.5, 102.4 (d, J_{CF} = 23 Hz), 107.6 (d, J_{CF} = 19 Hz), 121.3, 136.8 (d, J_{CF} = 8.7 Hz), 162.1 (d, J_{CF} = 10 Hz), 165.1 (d, J_{CF} = 247 Hz); **IR** (neat): 3595, 2954, 1593, 1500, 1400, 1284, 1246, 1184, 1068, 972, 833, 759 cm^{-1}; **HRMS** (FAB): calcd. for $C_9H_{14}FOSi$ $[M+H]^+$: 185.0792; found: 185.0801.

Synthesis of phenol **4j**



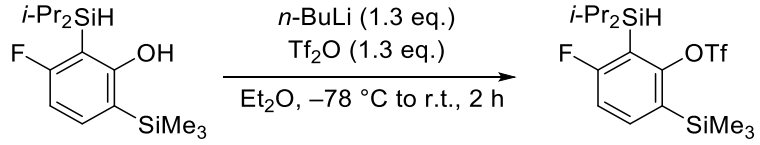
3j' **4j** 51%

To a solution of **3j'** (783 mg, 4.25 mmol) in THF (20 mL) was added NaH (60% dispersion in mineral oil,

221 mg, 5.52 mmol) at 0 °C. After stirring for 30 min at this temperature, *i*-Pr_2SiHCl (899 μL, 5.31 mmol) was added and the mixture was stirred for 2 h at room temperature. The reaction mixture was cooled to –78 °C, to which was added dropwise a solution of LDA (1.00 M) in THF (17.0 mL, 17.0 mmol) over 5 min. The mixture was warmed to 60 °C and stirred for 1 h. The reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/100) to afford phenol **4j** (647 mg, 51%) as colorless oil.

4j: R_f 0.57 (EtOAc/hexane = 1/100); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.28 (s, 9H), 1.00 (d, 6H, J = 7.6 Hz), 1.10 (d, 6H, J = 6.8 Hz), 1.35–1.43 (m, 2H), 4.10 (dt, J_{HF} = 3.4, J = 3.4 Hz), 6.00 (s, 1H), 6.58 (dd, J_{HF} = 7.6, J = 7.6 Hz), 7.35 (dd, J_{HF} = 7.6, J = 7.6 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ −1.5, 10.4, 18.2, 18.6, 104.0 (d, J_{CF} = 35 Hz), 106.5 (d, J_{CF} = 25 Hz), 120.9, 138.9 (d, J_{CF} = 10 Hz), 167.3 (d, J_{CF} = 13 Hz), 169.1 (d, J_{CF} = 241 Hz); **IR** (neat): 3529, 2947, 2866, 2506, 1593, 1558, 1462, 1365, 1246, 1188, 991, 837, 736 cm^{-1}; **HRMS** (ESI): calcd. for $C_{15}H_{26}FOSi_2$ $[M–H]^-$: 297.1512; found: 297.1507.

Synthesis of triflate **5j**

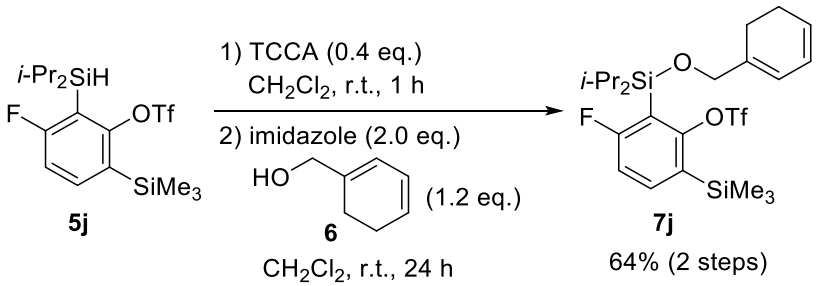


4j → **5j** 79%

To a solution of **4j** (554 mg, 1.86 mmol) in Et_2O (20 mL) was added was added *n*-BuLi (1.6 M) in hexane (1.51 mL, 2.41 mmol) at –78 °C. After stirring for 30 min at this temperature, Tf_2O (406 μL, 2.41 mmol) was added dropwise to the mixture. The mixture was warmed to room temperature, and the stirring was continued for 2 h. The reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford triflate **5j** (632 mg, 79%) as colorless oil.

5j: R_f 0.50 (hexane); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.36 (s, 9H), 0.95 (d, 6H, J = 6.8 Hz), 1.09 (d, 6H, J = 6.8 Hz), 1.31–1.37 (m, 2H), 4.10–4.13 (m, 1H), 7.08 (dd, 1H, J_{HF} = 7.6, J = 7.6 Hz), 7.60 (dd, 1H, J_{HF} = 7.6, J = 7.6 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.2, 10.8, 18.6, 115.0 (d, J_{CF} = 25 Hz), 118.7 (q, J_{CF} = 321 Hz), 118.9 (d, J_{CF} = 36 Hz), 131.2, 140.0 (d, J_{CF} = 8.7 Hz), 155.6 (d, J_{CF} = 14 Hz), 168.1 (d, J_{CF} = 244 Hz); **IR** (neat): 2954, 1400, 1219, 1138, 945, 910, 844, 732 cm^{-1}; **HRMS** (FAB): calcd. for $C_{16}H_{27}F_4O_3SSi_2$ $[M+H]^+$: 431.1150; found: 431.1164.

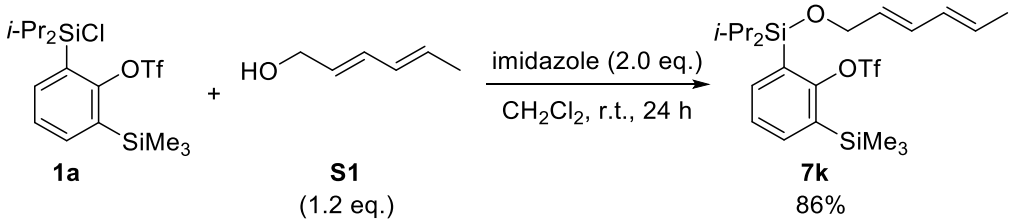
Synthesis of silyl ether **7j**



1) To a solution of **5j** (135 mg, 0.314 mmol) in CH_2Cl_2 (3 mL) was added trichloroisocyanuric acid[2] (TCCA, 29.2 mg, 0.126 mmol). After stirring for 1 h at room temperature, the reaction mixture was filtered through a Celite® pad (washed with hexane), and the filtrate was concentrated in vacuo to afford the corresponding silyl chloride as colorless oil. This material was employed in the next reaction without further purification.
2) To a solution of the crude material (*vide supra*) in CH_2Cl_2 (3 mL) were added imidazole (42.8 mg, 0.628 mmol) and alcohol **6**[3] (41.5 mg, 0.377 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane) to afford silyl ether **7j** (109 mg, 64% in 2 steps) as colorless oil.

7j: $\boldsymbol{R}_f$ 0.26 (EtOAc/hexane = 1/100); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.36 (s, 9H), 1.03 (d, 6H, J = 7.6 Hz), 1.09 (d, 6H, J = 7.6 Hz), 1.45 (qqd, 2H, J = 7.6, 7.6, J_{HF} = 2.1 Hz), 2.12 (brt, 2H, J = 10.3 Hz), 2.18–2.22 (m, 2H), 4.24 (s, 2H), 5.70–5.73 (m, 1H), 5.88–5.94 (m, 2H), 7.07 (dd, 1H, J_{HF} = 7.6, J = 7.6 Hz), 7.59 (dd, 1H, J_{HF} = 7.6, J = 7.6 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.1, 13.8, 17.1, 17.8, 22.3, 22.8, 66.6, 115.0 (d, J_{CF} = 26 Hz), 118.4, 118.68 (q, J_{CF} = 321 Hz), 118.72 (d, J_{CF} = 35 Hz), 124.6, 125.1, 131.1 (d, J_{CF} = 4.3 Hz), 137.8, 139.9 (d, J_{CF} = 8.9 Hz), 156.2 (d, J_{CF} = 14 Hz), 168.0 (d, J_{CF} = 246 Hz); **IR** (neat) 2951, 2870, 1518, 1400, 1270, 1134, 1099, 937, 840, 733 cm^{-1}; **HRMS** (ESI): calcd. for $C_{23}H_{34}F_4NaO_4SSi_2$ $[M+Na]^+$: 561.1545; found: 561.1539.

Synthesis of silyl ether **7k**

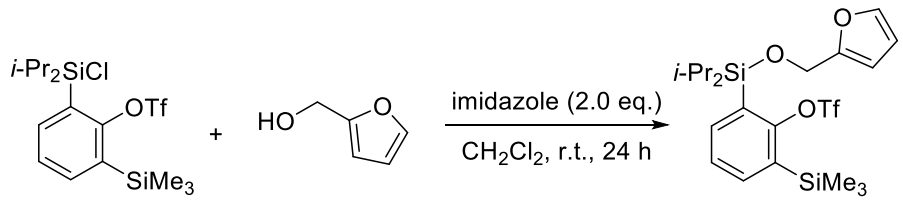


To a solution of **1a** (270 mg, 0.604 mmol) in CH_2Cl_2 (6 mL) were added imidazole (82.2 mg, 1.21 mmol) and alcohol **S1**[5] (71.2 mg, 0.726 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/100) to afford silyl ether **7k** (264

mg, 86%) as colorless oil.

7k: $\boldsymbol{R}_\mathbf{f}$ 0.64 (EtOAc/hexane = 1/20); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.36 (s, 9H), 1.01 (d, 6H, J = 7.5 Hz), 1.12 (d, 6H, J = 7.5 Hz), 1.39 (qq, 2H, J = 7.5, 7.5 Hz), 1.76 (d, 3H, J = 6.6 Hz), 4.24 (d, 2H, J = 4.9 Hz), 5.62–5.72 (m, 2H), 6.05–6.11 (m, 1H), 6.24 (dd, 1H, J = 15.2, 10.6 Hz), 7.37 (dd, 1H, J = 7.5, 7.2 Hz), 7.60–7.63 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 0.3, 13.7, 17.8, 18.1, 18.3, 64.2, 118.5 (q, J_{CF} = 318 Hz), 127.0, 129.0, 129.4, 130.1, 131.0, 134.8, 138.6, 138.7, 155.5 (several signals overlapped); **IR** (neat) 2951, 2870, 1400, 1215, 1138, 991, 871, 844, 736 cm^{-1}; **HRMS** (ESI): calcd. for $C_{22}H_{35}F_3NaO_4SSi_2$ $[M+Na]^+$: 531.1639; found: 531.1642.

Synthesis of silyl ether **7l**

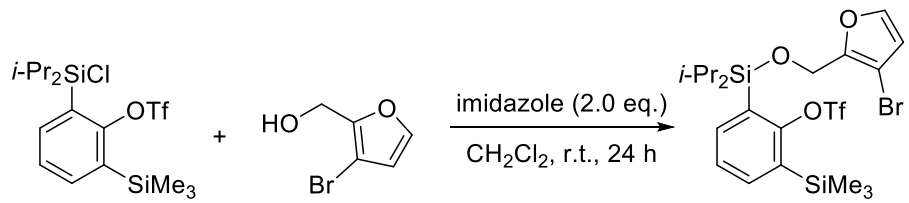


1a **S2** (1.2 eq.) **7l** 80%

To a solution of **1a** (226 mg, 0.505 mmol) in CH_2Cl_2 (5 mL) were added imidazole (68.8 mg, 1.01 mmol) and 2-furfuryl alcohol (**S2**) (52.5 μL, 0.606 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/50) to afford silyl ether **7l** (205 mg, 80%) as colorless oil.

7l: $\boldsymbol{R}_\mathbf{f}$ 0.50 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.36 (s, 9H), 1.02 (d, 6H, J = 6.9 Hz), 1.11 (d, 6H, J = 7.6 Hz), 1.42 (qq, 2H, J = 7.6, 6.9 Hz), 4.68 (s, 2H), 6.26 (d, 1H, J = 3.3 Hz), 6.33 (dd, 1H, J = 3.3, 2.0 Hz), 7.36–7.39 (m, 2H), 7.62 (dd, 1H, J = 7.6, 2.1 Hz), 7.66 (dd, 1H, J = 6.8, 2.1 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.2, 13.2, 17.3, 17.8, 58.6, 107.4, 110.3, 118.2 (q, J_{CF} = 319 Hz), 127.4, 130.1, 135.2, 139.1, 139.2, 142.4, 154.4, 155.9; **IR** (neat) 2951, 2870, 1396, 1215, 910, 844, 733 cm^{-1}; **HRMS** (ESI): calcd. for $C_{21}H_{31}F_3NaO_5SSi_2$ $[M+Na]^+$: 531.1275; found: 531.1284.

Synthesis of silyl ether **7m**

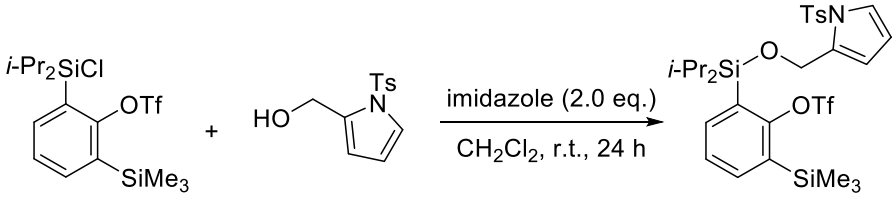


1a **S3** (1.3 eq.) **7m** 79%

To a solution of **1a** (232 mg, 0.518 mmol) in CH_2Cl_2 (5 mL) were added imidazole (70.5 mg, 1.04 mmol) and alcohol **S3**[6] (119 mg, 0.673 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/30) to afford silyl ether **7m** (241 mg, 79%) as colorless oil.

7m: $\boldsymbol{R_f}$ 0.48 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.36 (s, 9H), 1.03 (d, 6H, J = 7.6 Hz), 1.12 (d, 6H, J = 6.9 Hz), 1.43 (qq, 2H, J = 7.6, 6.9 Hz), 4.69 (s, 2H), 6.40 (d, 1H, J = 2.0 Hz), 7.37 (d, 1H, J = 2.0 Hz), 7.39 (dd, 1H, J = 7.6, 7.6 Hz), 7.62 (dd, 1H, J = 7.6, 2.1 Hz), 7.68 (dd, 1H, J = 7.6, 2.1 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.2, 13.2, 17.3, 17.8, 56.2, 98.4, 114.0, 118.7 (q, J_{CF} = 321 Hz), 127.5, 129.9, 135.2, 139.1, 139.2, 142.9, 150.9, 155.8; **IR** (neat) 2951, 2870, 1396, 1215, 1138, 867, 844, 733 cm^{-1}; **HRMS** (ESI): calcd. for $C_{21}H_{30}BrF_3KO_5SSi_2^+$ $[M+K]^+$: 625.0120; found: 625.0124.

Synthesis of silyl ether **7n**

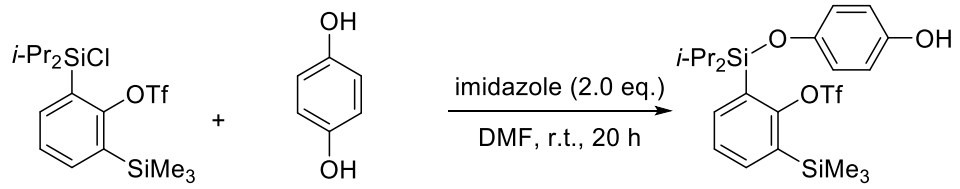


1a **S4** (1.2 eq.) **7n** 99%

To a solution of **1a** (234 mg, 0.524 mmol) in CH_2Cl_2 (5 mL) were added imidazole (71.3 mg, 1.05 mmol) and alcohol **S4**[7] (158.1 mg, 0.629 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/20) to afford silyl ether **7n** (343 mg, 99%) as colorless oil.

7n: $\boldsymbol{R_f}$ 0.31 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.38 (s, 9H), 0.98 (d, 6H, J = 7.6 Hz), 1.08 (d, 6H, J = 7.6 Hz), 1.39 (qq, 2H, J = 7.6, 7.6 Hz), 2.39 (s, 3H), 4.82 (s, 2H), 6.27 (dd, 1H, J = 3.4, 3.4 Hz), 6.37 (brs, 1H), 7.20–7.23 (m, 3H), 7.30 (dd, 1H, J = 7.6, 7.6 Hz), 7.56–7.64 (m, 4H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ –0.2, 13.3, 17.4, 17.9, 21.2, 59.4, 112.1, 112.8, 118.6 (q, J_{CF} = 321 Hz), 122.7, 126.9, 127.5, 129.9, 130.2, 134.7, 135.3, 136.4, 139.1, 139.2, 145.2, 155.5; **IR** (neat) 2951, 2870, 1396, 1361, 1215, 1138, 1076, 867, 840, 729 cm^{-1}; **HRMS** (ESI): calcd. for $C_{28}H_{38}F_3NNaO_6S_2Si_2$ $[M+Na]^+$: 684.1523; found: 684.1526.

Synthesis of silyl ether **7o**

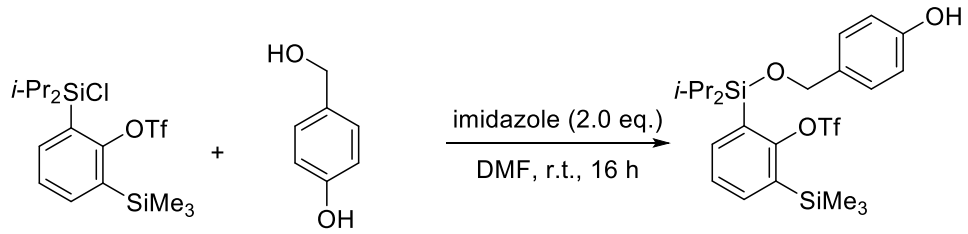


1a **S5** (4.0 eq.) **7o** 67%

To a solution of **1a** (643 mg, 1.46 mmol) in DMF (15 mL) were added imidazole (199 mg, 2.92 mmol) and hydroquinone (**S5**) (643 mg, 5.84 mmol) at 0 °C. After stirring for 20 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with EtOAc/hexane =1/2 (x3). The combined organic layer was washed with water (x3), brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/5) to afford silyl ether **7o** (507 mg, 67%) as pale yellow oil.

7o: $\boldsymbol{R_f}$ 0.34 (EtOAc/hexane = 1/5); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.37 (s, 9H), 1.02 (d, 6H, J = 7.8 Hz), 1.09 (d, 6H, J = 7.5 Hz), 1.49 (qq, 2H, J = 7.8, 7.5Hz), 4.36 (s, 1H, OH), 6.62–6.69 (m, 4H), 7.40 (dd, 1H, J = 7.5, 7.2 Hz), 7.65–7.70 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 0.3, 13.9, 17.6, 18.1, 115.7, 118.4 (q, J_{CF} = 318 Hz), 120.6, 127.1, 130.0, 135.2, 138.6, 138.9, 149.2, 149.7, 155.0; **IR** (neat): 3387, 2951, 1508, 1215, 1138, 906, 729 cm^{-1}; **HRMS** (ESI): calcd. for $C_{22}H_{31}F_3NaO_5SSi_2$ $[M+Na]^+$: 543.1275; found: 543.1271.

Synthesis of silyl ether **19a**



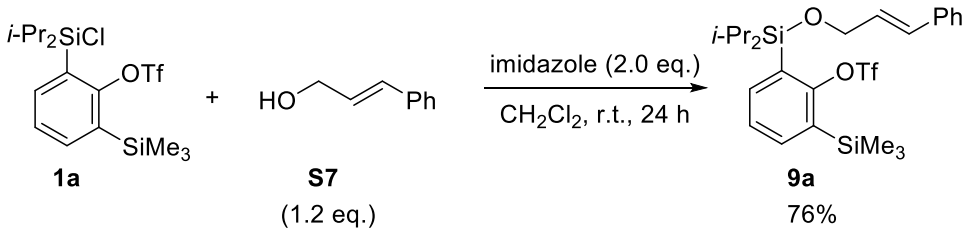
1a **S6** (2.0 eq.) **7p** 57%

To a solution of **1a** (246 mg, 0.550 mmol) in DMF (5.5 mL) were added imidazole (75 mg, 1.10 mmol) and *p*-hydroxy benzylalcohol (**S6**) (133 mg, 1.10 mmol) at 0 °C. After stirring for 16 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with EtOAc/hexane =1/2 (x3). The combined organic layer was washed with water (x3), brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/10) to afford silyl ether **7p** (166 mg, 57%) as pale yellow oil.

7p: $\boldsymbol{R_f}$ 0.25 (EtOAc/hexane = 1/5); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.37 (s, 9H), 1.03 (d, 6H, J = 7.5 Hz), 1.13 (d, 6H, J = 7.5 Hz), 1.43 (qq, 2H, J = 7.5, 7.5 Hz), 3.75 (s, 1H), 4.71 (s, 2H), 6.82 (d, 2H, J = 8.6 Hz), 7.23 (d, 2H, J = 8.6 Hz), 7.35 (dd, 1H, J = 7.5, 7.2 Hz), 7.61–7.63 (m, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ –0.2, 13.3, 17.5, 18.0, 65.0, 115.2, 118.6 (q, J_{CF} = 319 Hz), 127.4, 127.7, 130.3, 133.6, 135.2, 139.0, 139.2, 155.0, 155.8; **IR** (neat): 3394, 2951, 2870, 1512, 1396, 1215, 1138, 910, 871, 844, 736 cm^{-1}; **HRMS** (ESI):

calcd. for $C_{23}H_{33}F_3NaO_5SSi_2$ $[M+Na]^+$: 557.1432; found: 557.1433.

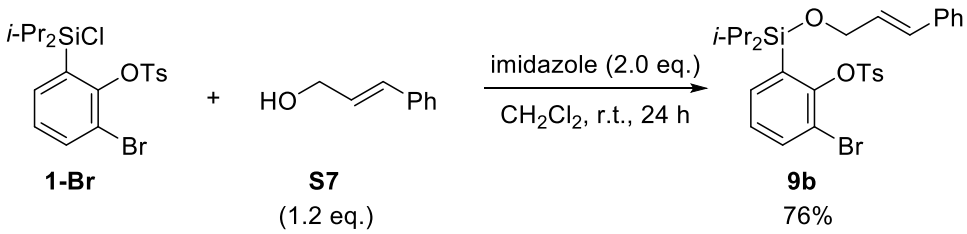
Synthesis of silyl ether **9a**



To a solution of **1a** (432 mg, 0.966 mmol) in CH_2Cl_2 (10 mL) were added imidazole (132 mg, 1.93 mmol) and cinnamyl alcohol (**S7**) (156 mg, 1.16 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/50) to afford silyl ether **9a** (402 mg, 76%) as colorless oil.

9a: $\boldsymbol{R_f}$ 0.59 (EtOAc/hexane = 1/10); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.37 (s, 9H), 1.04 (d, 6H, J = 7.8 Hz), 1.16 (d, 6H, J = 7.5 Hz), 1.43 (qq, 2H, J = 7.8, 7.5 Hz), 4.40 (dd, 2H, J = 4.9, 1.7 Hz), 6.31 (dt, 1H, J = 15.8, 4.9 Hz), 6.66 (dt, 1H, J = 15.8, 1.7 Hz), 7.23 (tt, 1H, J = 7.5, 2.0 Hz), 7.30–7.34 (m, 2H), 7.37–7.40 (m, 3H), 7.63 (dd, 1H, J = 7.2, 2.0 Hz), 7.65 (dd, 1H, J = 7.2, 2.0Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 0.3, 13.7, 17.9, 18.3, 64.4, 118.5 (q, J_{CF} = 318 Hz), 126.4, 127.1, 127.3, 128.5, 128.7, 129.4, 130.0, 134.9, 137.1, 138.66, 138.67, 155.5; **IR** (neat): 2951, 1396, 1215, 1138, 964, 906, 871, 841, 779 cm^{-1}; **HRMS** (ESI): calcd. for $C_{25}H_{35}F_3NaO_4SSi_2$ $[M+Na]^+$: 567.1639; found: 567.1657.

Synthesis of silyl ether **9b**

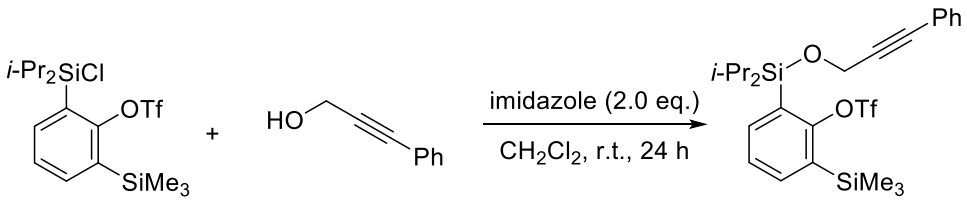


To a solution of **1-Br**[8] (309 mg, 0.651 mmol) in CH_2Cl_2 (6.5 mL) were added imidazole (88.6 mg, 1.30 mmol) and cinnamyl alcohol (**S7**) (105 mg, 0.781 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/20) to afford silyl ether **9b** (263 mg, 70%) as a white solid.

9b: **mp**: 74–79 °C; $\boldsymbol{R_f}$ 0.53 (EtOAc/hexane = 1/5); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 1.09 (d, 6H, J = 7.6 Hz), 1.21 (d, 6H, J = 6.9 Hz), 1.65 (qq, 2H, J = 7.6, 6.9 Hz), 2.46 (s, 3H), 4.45 (dd, 2H, J = 4.1, 1.4 Hz), 6.33 (dt,

1H, *J* = 15.8, 4.1 Hz), 6.69 (brd, 1H, *J* = 15.8 Hz), 7.15 (dd, 1H, *J* = 7.6, 7.6 Hz), 7.23 (t, 1H, *J* = 6.9 Hz), 7.30–7.35 (m, 4H), 7.40 (d, 2H, *J* = 7.6 Hz), 7.56 (dd, 1H, *J* = 7.6, 1.4 Hz), 7.62 (dd, 1H, *J* = 7.6, 1.4 Hz), 7.85 (d, 2H, *J* = 7.6 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 13.4, 17.6, 18,1, 21.4, 64.4, 117.8, 126.7, 127.6, 128.0, 128.8, 128.9, 129.2, 129.5, 129.8, 134.3, 134.8, 135.8, 136.7, 137.4,145.6, 150.7; **IR** (neat): 2943, 2866, 1373, 1168, 1072, 964, 856, 718 cm^{-1}; **HRMS** (ESI): calcd. for $C_{28}H_{33}BrNaO_4SSi$ $[M+Na]^+$: 595.0944; found: 595.0958.

Synthesis of silyl ether **11a**

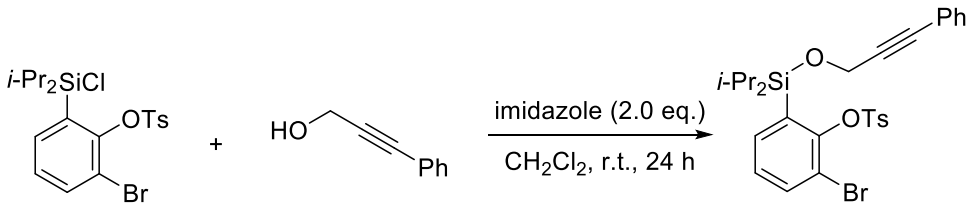


1a **S8** (1.2 eq.) **11a** 63%

To a solution of **1a** (240 mg, 0.537 mmol) in CH_2Cl_2 (5 mL) were added imidazole (73.1 mg, 1.07 mmol) and 3-phenyl-2-propyn-1-ol (**S8**) (80.3 μL, 0.644 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/50) to afford silyl ether **11a** (184 mg, 63%) as colorless oil.

11a: R_f 0.57 (EtOAc/hexane = 1/10); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.35 (s, 9H), 1.07 (d, 6H, *J* = 7.5 Hz), 1.17 (d, 6H, *J* = 7.5 Hz), 1.46 (qq, 2H, *J* = 7.5, 7.5 Hz), 4.60 (s, 2H), 7.28–7.32 (m, 3H), 7.35–7.41 (m, 3H), 7.62 (dd, 1H, *J* = 7.2, 1.8 Hz), 7.69 (dd, 1H, *J* = 7.5, 1.8 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 0.3, 13.6, 17.7, 18.1, 52.9, 85.1, 87.5, 118.5 (q, J_{CF} = 318 Hz), 122.9, 127.1, 128.2, 129.7, 131.5, 134.9, 138.6, 138.7, 155.6 (several signals overlapped); **IR** (neat): 2951, 2870, 1396, 1219, 1138, 906, 732 cm^{-1}; **HRMS** (ESI): calcd. for $C_{25}H_{35}F_3NaO_4SSi_2$ $[M+Na]^+$: 565.1482; found: 565.1493.

Synthesis of silyl ether **11b**



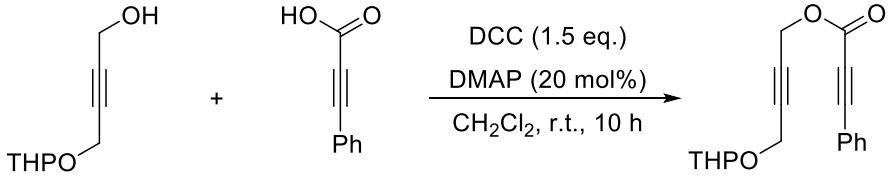
1-Br **S8** (1.2 eq.) **11b** 94%

To a solution of **1-Br**[8] (192 mg, 0.404 mmol) in CH_2Cl_2 (4 mL) were added imidazole (55.0 mg, 0.808 mmol) and 3-phenyl-2-propyn-1-ol (**S8**) (60.5 μL, 0.485 mmol) at 0 °C. After stirring for 24 h at room temperature,

the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/20) to afford **11b** (217 mg, 94%) as a white solid.

11b: **mp**: 110–113 ℃; $\boldsymbol{R_f}$ 0.42 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 1.11 (d, 6H, *J* = 7.6 Hz), 1.22 (d, 6H, *J* = 7.6 Hz), 1.65 (qq, 2H, *J* = 7.6, 7.6 Hz), 2.47 (s, 3H), 4.64 (s, 2H), 7.17 (dd, 1H, *J* = 7.6, 7.6 Hz), 7.28–7.32 (m, 3H), 7.33 (d, 2H, *J* = 8.3 Hz), 7.38–7.42 (m, 2H), 7.55 (dd, 1H, *J* = 7.6, 2.1 Hz), 7.70 (dd, 1H, *J* = 7.6, 2.1 Hz), 7.85 (d, 2H, *J* = 8.3 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 13.3, 17.5, 17.9, 21.4, 53.0, 85.0, 87.8, 117.8, 123.2, 128.0, 128.5, 128.9, 129.8, 131.9, 134.0, 134.8, 135.9, 136.8, 145.6, 150.8 (several signals overlapped); **IR** (neat): 2947, 2866, 1369, 1168, 1072, 910, 856, 713 cm^{-1}; **HRMS** (ESI): calcd. for $C_{28}H_{31}BrNaO_4SSi$ $[M+Na]^+$: 593.0788; found: 593.0800.

Synthesis of ester **S11**

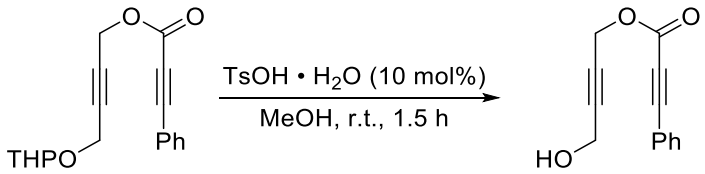


S9 (1.5 eq.) **S10** **S11** 76%

To a solution of **S9** (2.01 g, 11.8 mmol) and phenylpropiolic acid (**S10**) (1.15g, 7.87 mmol) in CH_2Cl_2 (30 mL) were added DCC (2.43 g, 11.8 mmol) and DMAP (192 mg, 1.57 mmol) at 0 °C. After stirring for 10 h at room temperature, the reaction was quenched by water, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with 5% HCl (x1), saturated aqueous $NaHCO_3$ (x1) and brine (x1), dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/10) to afford ester **S11** (1.79 g, 76%) as colorless oil.

S11: $\boldsymbol{R_f}$ 0.31 (EtOAc/hexane = 1/5); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 1.50–1.67 (m, 4H), 1.73–1.78 (m, 1H), 1.80–1.87 (m, 1H), 3.52–3.57 (m, 1H), 3.81–3.86 (m, 1H), 4.28 (dt, 1H, *J* = 16.5, 2.0 Hz), 4.36 (dt, 1H, *J* = 16.5, 2.0 Hz), 4.81 (t, 1H, *J* = 3.4 Hz), 4.88 (t, 2H, *J* = 1.4 Hz), 7.38 (dd, 2H, *J* = 7.6, 7.6 Hz), 7.46 (t, 1H, *J* = 7.6 Hz), 7.59 (d, 2H, *J* = 7.6 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 18.5, 24.9, 29.8, 53.4, 53.9, 61.7, 78.9, 79.8, 83.8, 87.4, 96.9, 119.4, 128.8, 131.1, 133.3, 153.5; **IR** (neat): 2943, 2854, 2218, 1712, 1280, 1161, 1022, 902, 744 cm^{-1}; **HRMS** (ESI): calcd. for $C_{18}H_{18}NaO_4$ $[M+Na]^+$: 321.1097; found: 321.1082.

Synthesis of alcohol **13**

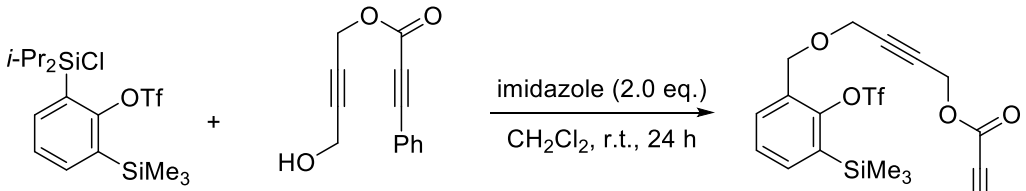


S11 **13** 95%

To a solution of **13** (1.34 g, 4.69 mmol) in MeOH (10 mL) was added TsOH・H_2O (89.2 mg, 0.469 mmol) at 0 °C. After stirring for 1.5 h at room temperature, the reaction was quenched by saturated aqueous $NaHCO_3$, and the mixture was extracted with EtOAc(x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/2) to afford alcohol **13** (0.956 g, 95%) as colorless oil.

13: $\boldsymbol{R}_f$: 0.45 (EtOAc/hexane = 1/1); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 1.65 (brs, 1H), 4.34 (d, 2H, J = 5.6 Hz), 4.87 (s, 2H), 7.38 (dd, 2H, J = 7.6, 7.6 Hz), 7.47 (t, 1H, J = 7.6 Hz), 7.59 (d, 2H, J = 7.6 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 50.4, 54.4, 78.4, 79.7, 86.0, 87.6, 119.2, 128.8, 131.1, 133.2, 153.6; **IR** (neat): 3383, 2218, 1708, 1280, 1165, 1018, 968, 906, 729 cm^{-1}; **HRMS** (ESI): calcd. for $C_{13}H_{10}NaO_3$ $[M+Na]^+$: 237.0522; found: 237.0525.

Synthesis of silyl ether **14**



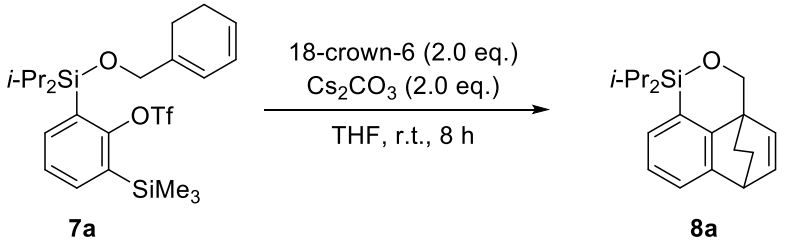
1a **13** (1.1 eq.) **14** 85%

To a solution of **1a** (401 mg, 0.899 mmol) in CH_2Cl_2 (9 mL) were added imidazole (122 mg, 1.80 mmol) and alcohol **13** (212 mg, 0.989 mmol) at 0 °C. After stirring for 24 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with $CHCl_3$ (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/100) to afford **14** (478 mg, 85%) as colorless oil.

14: $\boldsymbol{R}_f$ 0.48 (EtOAc/hexane = 1/5); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.36 (s, 9H), 1.04 (d, 6H, J = 7.5 Hz), 1.14 (d, 6H, J = 7.5 Hz), 1.43 (qq, 2H, J = 7.5, 7.5 Hz), 4.12 (t, 2H, J = 1.7 Hz), 4.85 (t, 2H, J = 1.7 Hz), 7.37–7.43 (m, 3H), 7.47 (tt, 1H, J = 7.5, 1.5 Hz), 7.58–7.66 (m, 4H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ –0.2, 13.1, 17.3, 17.7, 52.2, 53.4, 78.1, 79.9, 86.0, 87.4, 118.7 (q, J_{CF} = 320 Hz), 119.6, 127.5, 128.9, 129.7, 131.2, 133.4, 135.3, 138.9, 139.2, 153.6, 155.9; **IR** (neat) 2966, 1716, 1396, 1219, 1138, 910, 871, 844, 733 cm^{-1}; **HRMS** (ESI): calcd. for $C_{29}H_{35}F_3NaO_6SSi_2$ $[M+Na]^+$: 647.1543; found: 647.1545.

2-2. Synthesis of cycloadducts

Typical procedure: Synthesis of cycloadduct **8a**

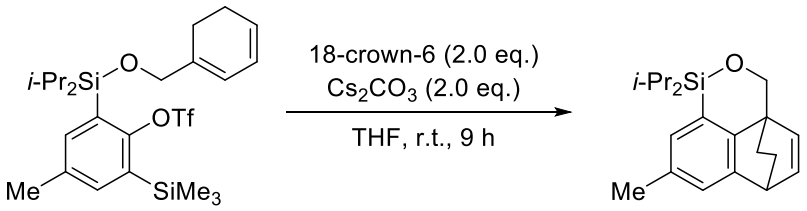


7a **8a** 80%

A flame-dried two-necked flask charged with 18-crown-6 (89.1 mg, 0.337 mmol) was evacuated (1 mmHg) at room temperature for 1 h, to which were added **6a** (87.8 mg, 0.168 mmol) and THF (3 mL). To the mixture was added Cs_2CO_3 (110 mg, 0.337 mmol) and stirred for 8 h at room temperature. The reaction was quenched by adding saturated aqueous NH_4Cl and the mixture was extracted with EtOAc (x3). The combined organic layers were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/100) to afford cycloadduct **8a** (40.0 mg, 80%) as colorless oil.

8a: $\boldsymbol{R_f}$ 0.43 (EtOAc/hexane = 1/10); Spectral data matched those reported in the literature.[8]

Synthesis of cycloadduct **8b**

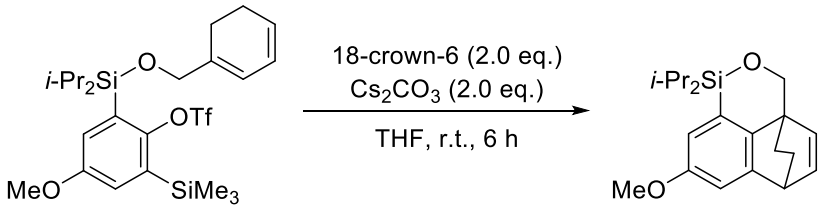


7b **8b** 81%

According to the typical procedure, **8b** was prepared from the reaction of 18-crown-6 (96.2 mg, 0.364 mmol), **7b** (97.3 mg, 0.182 mmol) in THF (3 mL) and Cs_2CO_3 (119 mg, 0.364 mmol) at room temperature for 9 h. Purification by flash column chromatography (silica gel, EtOAc/hexane = 1/100) afforded cycloadduct **8b** (46.2 mg, 81%) as colorless oil.

8b: $\boldsymbol{R_f}$ 0.48 (EtOAc/hexane = 1/20); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.96 (d, 3H, J = 7.6 Hz), 1.00 (d, 3H, J = 7.6 Hz), 1.10 (d, 3H, J = 7.6 Hz), 1.13 (d, 3H, J = 7.6 Hz), 1.15–1.20 (m, 1H), 1.29 (qq, 1H, J = 7.6, 7.6 Hz), 1.36(ddd, 1H, J = 10.3, 10.3, 4.9 Hz), 1.43–1.50 (m, 1H), 1.62–1.72 (m, 2H), 2.31 (s, 3H), 3.83–3.86 (m, 1H), 4.28 (d, 1H, J = 11.7 Hz), 4.57 (d, 1H, J = 11.7 Hz), 6.05 (d, 1H, J = 7.6 Hz), 6.55 (dd, 1H, J = 7.6, 7.6 Hz), 7.00 (s, 1H), 7.02 (s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 12.2, 12.9, 16.9, 17.0, 17.2, 17.6, 21.0, 27.2, 29.9, 40.7, 44.6, 68.2, 124.9, 126.4, 130.2, 133.6, 136.1, 136.2, 144.4, 148.5; **IR** (neat): 2947, 2866, 2337, 1462, 1207, 906, 779, 733 cm^{-1}; **HRMS** (ESI): calcd. for $C_{20}H_{29}OSi$ $[M+H]^+$: 313.1982; found: 313.1963.

Synthesis of cycloadduct **8c**



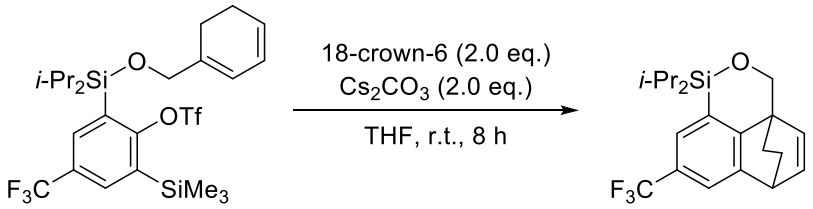
7c **8c**

83%

According to the typical procedure, **8c** was prepared from the reaction of 18-crown-6 (131 mg, 0.495 mmol), **7c** (136 mg, 0.247 mmol) in THF (4 mL) and Cs_2CO_3 (161 mg, 0.495 mmol) at room temperature for 6 h. Purification by flash column chromatography (EtOAc/hexane = 1/50) afforded cycloadduct **8c** (67.4 mg, 83%) as colorless oil.

8c: $\boldsymbol{R}_f$ 0.52 (EtOAc/hexane = 1/10); Spectral data matched those reported in the literature.[8]

Synthesis of cycloadduct **8d**



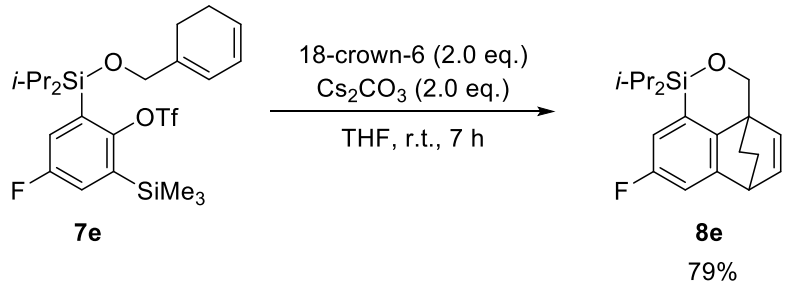
7d **8d**

74%

According to the typical procedure, **8d** was prepared from the reaction of 18-crown-6 (129 mg, 0.489 mmol), **7d** (144 mg, 0.244 mmol) in THF (4 mL) and Cs_2CO_3 (159 mg, 0.489 mmol) at room temperature for 8 h. Purification by flash column chromatography (silica gel, EtOAc/hexane = 1/50) afforded cycloadduct **8d** (66.1 mg, 74%) as colorless oil.

8d: $\boldsymbol{R}_f$ 0.47 (EtOAc/hexane = 1/20); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.95 (d, 3H, J = 6.9 Hz), 1.01 (d, 3H, J = 7.6 Hz), 1.10 (d, 3H, J = 7.6 Hz), 1.14 (d, 3H, J = 7.6 Hz), 1.20 (qq, 1H, J = 7.6, 6.9 Hz), 1.32 (qq, 1H, J = 7.6, 7.6 Hz), 1.42 (ddd, 1H, J = 10.3, 10.3, 4.1 Hz), 1.46–1.52 (m, 1H), 1.66–1.73 (m, 2H), 3.97–4.01 (m, 1H), 4.32 (d, 1H, J = 11.6 Hz), 4.60 (d, 1H, J = 11.6 Hz), 6.06 (d, 1H, J = 6.9 Hz), 6.59 (dd, 1H, J = 6.9, 6.2 Hz), 7.42 (s, 1H), 7.43 (s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 12.1, 12.8, 16.8, 16.9, 17.1, 17.5, 26.8, 29.4, 40.7, 45.1, 67.9, 120.3 (q, J_{CF} = 4.3 Hz), 125.0 (q, J_{CF} = 273 Hz), 126.5 (q, J_{CF} = 2.9 Hz), 126.6 (q, J_{CF} = 32 Hz), 127.7, 135.5, 136.1, 144.8, 155.3; **IR** (neat): 2947, 2866, 2360, 1334, 1311, 1122, 1053, 906, 729 cm^{-1}; **HRMS** (ESI): calcd. for $C_{20}H_{26}F_3OSi$ $[M+H]^+$: 367.1700; found: 367.1713.

Synthesis of cycloadduct **8e**

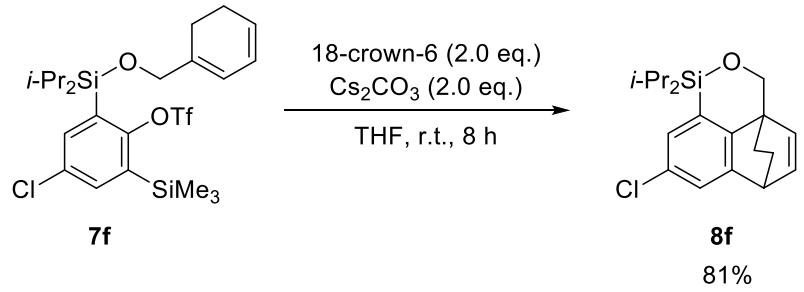


7e **8e** 79%

According to the typical procedure, **8e** was prepared from the reaction of 18-crown-6 (235 mg, 0.889 mmol), **7e** (240 mg, 0.445 mmol) in THF (7.5 mL) and Cs_2CO_3 (290 mg, 0.889 mmol) at room temperature for 7 h. Purification by flash column chromatography (EtOAc/hexane = 1/50) afforded cycloadduct **8e** (111 mg, 79%) as colorless oil.

8e: $\boldsymbol{R}_\mathbf{f}$ 0.60 (EtOAc/hexane = 1/20); Spectral data matched those reported in the literature.[8]

Synthesis of cycloadduct **8f**

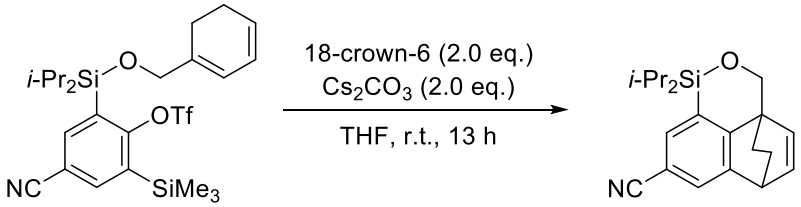


7f **8f** 81%

According to the typical procedure, **8f** was prepared from the reaction of 18-crown-6 (86.3 mg, 0.326 mmol), **7f** (90.6 mg, 0.163 mmol) in THF (3 mL) and Cs_2CO_3 (106.2 mg, 0.326 mmol) at room temperature for 8 h. Purification by flash column chromatography (silica gel, EtOAc/hexane = 1/50) to afford cycloadduct **8f** (44.1 mg, 81%) as colorless oil.

8f: $\boldsymbol{R}_\mathbf{f}$ 0.59 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.95 (d, 3H, J = 7.6 Hz), 1.01 (d, 3H, J = 7.6 Hz), 1.10 (d, 3H, J = 7.6 Hz), 1.13 (d, 3H, J = 6.8 Hz), 1.17 (qq, 1H, J = 7.6, 7.6 Hz), 1.29 (qq, 1H, J = 7.6, 6.8 Hz), 1.37 (ddd, 1H, J = 10.3, 10.3, 5.5 Hz), 1.44–1.50 (m, 1H), 1.64–1.70 (m, 2H), 3.87–3.90 (m, 1H), 4.28 (d, 1H, J = 11.7 Hz), 4.56 (d, 1H, J = 11.7 Hz), 6.04 (d, 1H, J = 6.9 Hz), 6.55 (dd, 1H, J = 6.9, 6.2 Hz), 7.14 (d, 1H, J = 2.0 Hz), 7.17 (d, 1H, J = 2.0 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 12.1, 12.8, 16.8, 16.9, 17.1, 17.5, 26.9, 29.6, 40.5, 44.7, 68.0, 124.1, 128.97, 129.01, 130.7, 135.8, 136.0, 146.5, 149.6; **IR** (neat): 2943, 2866, 1462, 1196, 1138, 1056, 991, 883, 779 cm^{-1}; **HRMS** (ESI): calcd. for $C_{19}H_{26}ClOSi$ $[M+H]^+$: 333.1436; found: 333.1450.

Synthesis of cycloadduct **8g**

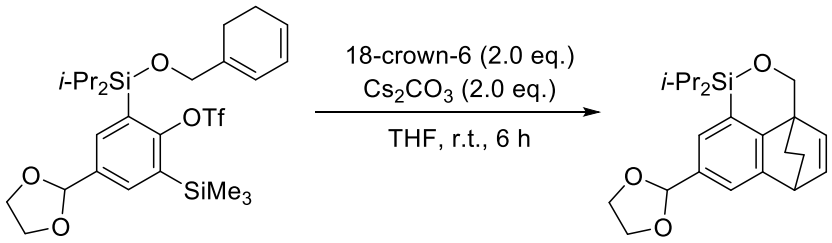


7g **8g** 33%

According to the typical procedure, **8g** was prepared from the reaction of 18-crown-6 ether (34.9 mg, 0.132 mmol), **7g** (36.0 mg, 0.0660 mmol) in THF (1.1 mL) and Cs_2CO_3 (43.0 mg, 0.132 mmol) at room temperature for 13 h. Purification by PTLC (EtOAc/hexane = 1/30 x3) afforded cycloadduct **8g** (7.0 mg, 33%) as colorless oil.

8g: R_f 0.50 (EtOAc/hexane = 1/5); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.94 (d, 3H, J = 7.6 Hz), 1.01 (d, 3H, J = 7.6 Hz), 1.09 (d, 3H, J = 7.6 Hz), 1.13 (d, 3H, J = 7.6 Hz), 1.20 (qq, 1H, J = 7.6, 7.6 Hz), 1.31 (qq, 1H, J = 7.6, 7.6 Hz), 1.40–1.50 (m, 2H), 1.64–1.73 (m, 2H), 3.96–3.99 (m, 1H), 4.30 (d, 1H, J = 12.4 Hz), 4.57 (d, 1H, J = 12.4 Hz), 6.05 (d, 1H, J = 6.9 Hz), 6.58 (dd, 1H, J = 6.9, 6.2 Hz), 7.44 (d, 1H, J = 1.4 Hz), 7.50 (d, 1H, J = 1.4 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 12.0, 12.7, 16.7, 16.9, 17.0, 17.4, 26.6, 29.2, 40.4, 45.2, 67.7, 108.2, 120.1, 126.5, 128.7,134.2, 135.4, 136.0, 145.1, 156.5; **IR** (neat): 2943, 2866, 2225, 1462, 1384, 1118, 1057, 991, 964, 883, 783 cm^{-1}; **HRMS** (ESI): calcd. for $C_{20}H_{25}NNaO_3Si$ $[M+Na]^+$: 346.1598; found: 346.1601.

Synthesis of cycloadduct **8h**



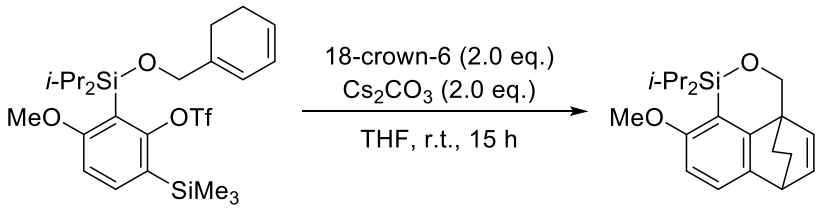
7h **8h** 73%

According to the typical procedure, **8h** was prepared from the reaction of 18-crown-6 (41.2 mg, 0.156 mmol), **7h** (46.2 mg, 0.0779 mmol) in THF (1.5 mL) and Cs_2CO_3 (50.8 mg, 0.156 mmol) at room temperature for 6 h. Purification by column chromatography (EtOAc/hexane = 1/20) afforded cycloadduct **8h** (20.9 mg, 73%) as colorless oil.

8h: R_f 0.35 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.95 (d, 3H, J = 6.9 Hz), 1.00 (d, 3H, J = 7.6 Hz), 1.09 (d, 3H, J = 7.6 Hz), 1.13 (d, 3H, J = 7.6 Hz), 1.18 (qq, 1H, J = 7.6, 6.9 Hz), 1.30 (qq, 1H, J = 7.6, 7.6 Hz), 1.37 (ddd, 1H, J = 12.1, 12.1, 6.9 Hz), 1.46–1.52 (m, 1H), 1.62–1.69 (m, 2H), 3.91–3.94 (m, 1H), 4.01–4.06 (m, 2H), 4.12–4.17 (m, 2H), 4.29 (d, 1H, J = 11.7 Hz), 4.58 (d, 1H, J = 11.7 Hz), 5.78 (s, 1H), 6.04 (d, 1H, J = 7.6 Hz), 6.56 (dd, 1H, J = 7.6, 6.2 Hz), 7.27 (d, 1H, J = 2.0 Hz), 7.34 (d, 1H, J = 2.0

Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 12.2, 12.8, 16.8, 17.0, 17.2, 17.6, 26.9, 29.7, 40.8, 44.9, 65.2, 68.2, 104.4, 121.6, 126.5, 128.3, 133.5, 135.7, 136.2, 144.6, 152.8; **IR** (neat): 2947, 2866, 1462, 1369, 1087, 906, 725 cm^{-1}; **HRMS** (ESI): calcd. for $C_{22}H_{31}O_3Si$ $[M+H]^+$: 371.2037; found: 371.2038.

Synthesis of cycloadduct **8i**



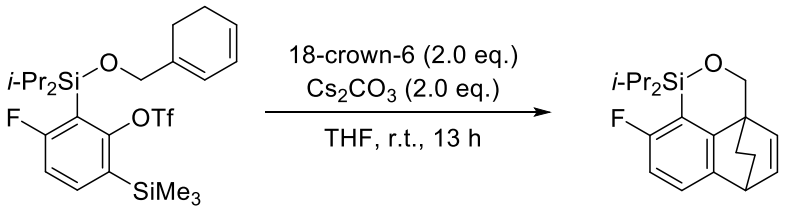
7i **8i**

72%

According to the typical procedure, **8i** was prepared from the reaction of 18-crown-6 ether (89.5 mg, 0.339 mmol), 7**i** (93.3 mg, 0.169 mmol) in THF (3 mL) and Cs_2CO_3 (111 mg, 0.339 mmol) at room temperature for 15 h. Purification by flash column chromatography (silica gel, EtOAc/hexane = 1/50) to afford cycloadduct **8i** (39.8 mg, 72%) as colorless oil.

8i: R_f 0.62 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.90 (d, 3H, J = 7.6 Hz), 1.02 (d, 3H, J = 7.6 Hz), 1.06 (d, 3H, J = 7.6 Hz), 1.12 (d, 3H, J = 7.6 Hz), 1.23 (qq, 1H, J = 7.6, 7.6 Hz), 1.30 (qq, 1H, J = 7.6, 7.6 Hz), 1.35 (ddd, 1H, J = 11.0, 11.0, 4.1 Hz), 1.44–1.50 (m, 1H), 1.60–1.70 (m, 2H), 3.73 (s, 3H), 3.84–3.87 (m, 1H), 4.24 (d, 1H, J = 11.7 Hz), 4.51 (d, 1H, J = 11.7 Hz), 6.03 (d, 1H, J = 7.6 Hz), 6.50 (d, 1H, J = 8.2 Hz), 6.57 (dd, 1H, J =7.6, 6.2 Hz), 7.11 (d, 1H, J = 8.2 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 12.2, 13.2, 17.0, 17.2, 17.6, 18.2, 27.4, 29.6, 39.9, 44.6, 54.7, 68.1, 104.8, 116.4, 124.5, 135.7, 136.8, 137.0, 152.9, 161.5; **IR** (neat): 2943, 2862, 1573, 1454, 1238, 1056, 991, 883, 771 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{20}H_{29}O_2Si$ $[M+H]^+$: 329.1931; found: 329.1922.

Synthesis of cycloadduct **8j**



7j **8j**

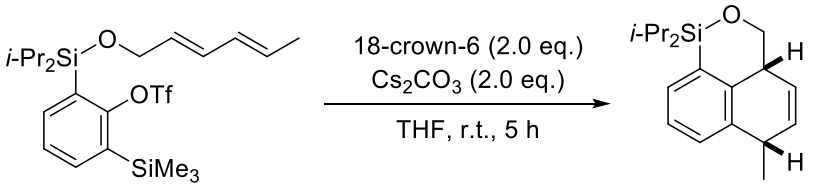
42%

According to the typical procedure, **8j** was prepared from the reaction of 18-crown-6 (104 mg, 0.395 mmol), **7j** (106 mg, 0.197 mmol) in THF (3.5 mL) and Cs_2CO_3 (129 mg, 0.395 mmol) at room temperature for 13 h. Purification by flash column chromatography (silica gel, EtOAc/hexane = 1/50) afford cycloadduct **8j** (26.2 mg, 42%) as colorless oil.

8j: R_f 0.47 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 0.93 (d, 3H, J = 7.6 Hz), 1.05 (d, 3H, J

= 7.6 Hz), 1.09 (d, 3H, J = 7.6 Hz), 1.14 (d, 3H, J = 7.6 Hz), 1.26 (qq, 1H, J = 7.6, 7.6 Hz), 1.31–1.39 (m, 2H) 1.44–1.49 (m, 1H), 1.61–1.69 (m, 2H), 3.90–3.93 (m, 1H), 4.27 (d, 1H, J = 11.7 Hz), 4.55 (d, 1H, J = 11.7 Hz), 6.04 (d, 1H, J = 8.2 Hz), 6.58 (dd, 1H, J = 8.2, 6.8 Hz), 6.71 (dd, 1H, J_{HF} = 8.2, J = 8.2 Hz), 7.13 (dd, 1H, J = 8.2, J_{HF} = 6.2 Hz) ; **^{13}C NMR** (150 MHz, $CDCl_3$): δ 12.1, 13.0, 16.8, 16.9, 17.1, 17.6, 27.1, 29.5, 40.1, 44.8, 67.9, 110.2 (d, J_{CF} = 26 Hz), 114.3 (d, J_{CF} = 38 Hz), 125.3 (d, J_{CF} = 8.7 Hz), 135.6, 136.6, 140.3, 152.8 (d, J_{CF} = 13 Hz), 164.4 (d, J_{CF} = 236 Hz); **IR** (neat): 2951, 2866, 1782, 1570, 1442, 1276, 1060, 964, 883, 736 cm^{-1}; **HRMS** (ESI): calcd. for $C_{19}H_{26}FOSi$ $[M+H]^+$: 317.1731; found: 317.1728.

Synthesis of cycloadduct **8k**

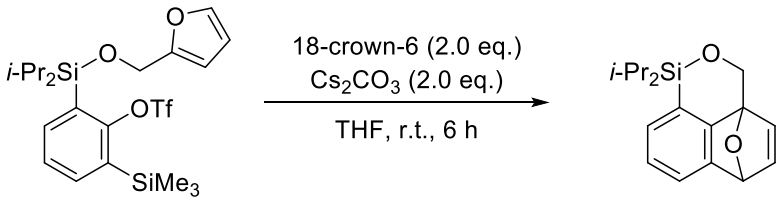


7k **8k** 68%

According to the typical procedure, **8k** was prepared from the reaction of 18-crown-6 (121 mg, 0.458 mmol), **7k** (117 mg, 0.229 mmol) in THF (4 mL) and Cs_2CO_3 (149 mg, 0.458 mmol) at room temperature for 5 h. Purification by column chromatography (EtOAc/hexane = 1/50) afforded cycloadduct **8k** (44.6 mg, 68%) as colorless oil.

8k: R_f 0.31 (EtOAc/hexane = 1/20); Spectral data matched those reported in the literature.[8]

Synthesis of cycloadduct **8l**

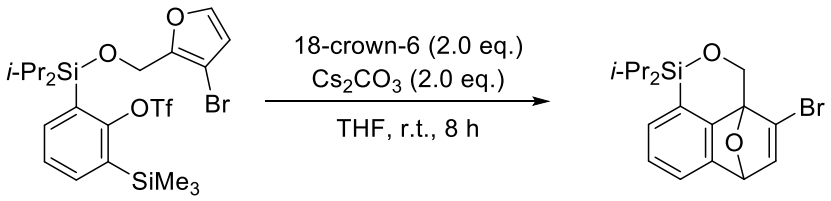


7l **8l** 86%

According to the typical procedure, **8l** was prepared from the reaction of 18-crown-6 (210 mg, 0.795 mmol), **7l** (202 mg, 0.397 mmol) in THF (7 mL) and Cs_2CO_3 (259 mg, 0.795 mmol) at room temperature for 6 h. Purification by flash column chromatography (EtOAc/hexane = 1/30) afforded cycloadduct **8l** (97.7 mg, 86%) as colorless oil.

12a: R_f: 0.33 (EtOAc/hexane = 1/10); Spectral data matched those reported in the literature.[8]

Synthesis of cycloadduct **8m**

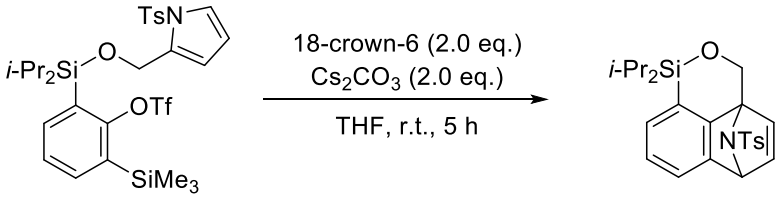


7m **8m** 80%

According to the typical procedure, **8m** was prepared from the reaction of 18-crown-6 (167 mg, 0.632 mmol), **7m** (186 mg, 0.316 mmol) in THF (5 mL) and Cs_2CO_3 (206 mg, 0.632 mmol) at room temperature for 8 h. Purification by flash column chromatography (EtOAc/hexane = 1/30) afforded cycloadduct **8m** (92.0 mg, 80%) as colorless oil.

8m: $\boldsymbol{R_f}$ 0.30 (EtOAc/hexane = 1/10); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 1.03–1.12 (m, 12H), 1.19–1.26 (m, 2H), 4.35 (d, 1H, J = 10.3 Hz), 4.70 (d, 1H, J = 10.3 Hz), 5.67 (d, 1H, J = 2.1 Hz), 7.01 (brs, 1H), 7.03 (dd, 1H, J = 7.6, 6.8 Hz), 7.11 (d, 1H, J = 7.6 Hz), 7.29 (d, 1H, J = 6.8 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 16.88, 16.98, 17.02, 17.4, 62.9, 83.3, 88.1, 121.4, 125.3, 126.4, 128.8, 138.7, 141.0, 146.5, 155.4; **IR** (neat): 2943, 2862, 1570, 1462, 1083, 1006, 968, 883, 756 cm^{-1}; **HRMS** (ESI): calcd. for $C_{17}H_{22}BrO_2Si$ $[M+H]^+$: 365.0567; found: 365.0583.

Synthesis of cycloadduct **8n**

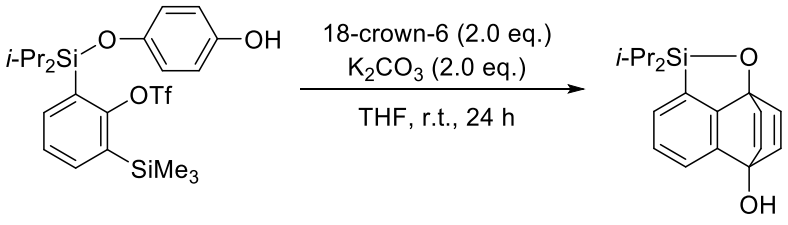


7n **8n** 77%

According to the typical procedure, **8n** was prepared from the reaction of 18-crown-6 (272 mg, 1.030 mmol), **7n** (341 mg, 0.515 mmol) in THF (9 mL) and Cs_2CO_3 (336 mg, 1.030 mmol) at room temperature for 5 h. Purification by flash column chromatography (EtOAc/hexane = 1/5) afforded cycloadduct **8n** (174 mg, 77%) as a white solid.

8n: $\boldsymbol{R_f}$ 0.50 (EtOAc/hexane = 1/3); Spectral data matched those reported in the literature.[8]

Synthesis of cycloadduct **8o**

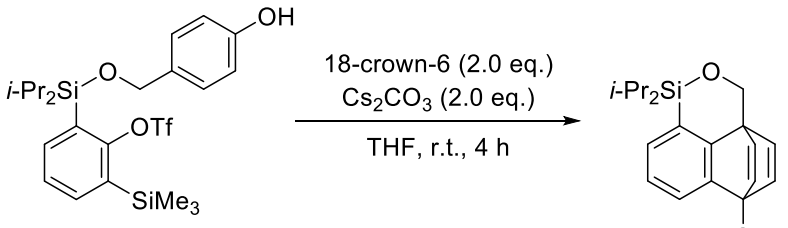


7o **8o** 32%

According to the typical procedure, **8o** was prepared from the reaction of 18-crown-6 (102 mg, 0.387 mmol), **7o** (101 mg, 0.193 mmol) in THF (3.5 mL) and K_2CO_3 (53.5 mg, 0.387 mmol) at room temperature for 24 h. Purification by column chromatography (EtOAc/hexane = 1/5) afforded cycloadduct **8o** (18.4 mg, 32%) as colorless oil along with starting material **7o** (15.9 mg, 16%).

8o: $\boldsymbol{R_f}$ 0.26 (EtOAc/hexane = 1/3); Spectral data matched those reported in the literature.[9]

Synthesis of cycloadduct **8p**

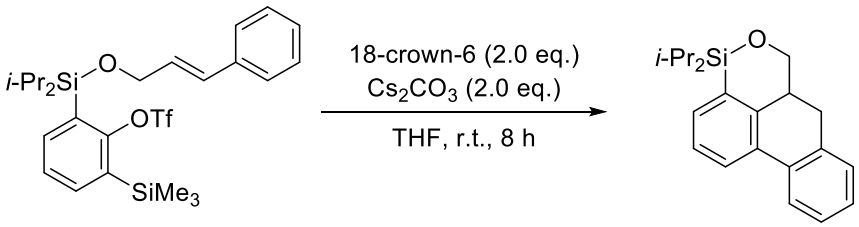


7p **8p** 48%

According to the typical procedure, **8p** was prepared from the reaction of 18-crown-6 (106 mg, 0.401 mmol), **7p** (107 mg, 0.200 mmol) in THF (3.5 mL) and Cs_2CO_3 (131 mg, 0.401 mmol) at room temperature for 4 h. Purification by column chromatography (EtOAc/hexane = 1/5) afforded cycloadduct **8p** (30.2 mg, 48%) as colorless oil.

8p: $\boldsymbol{R_f}$ 0.31 (EtOAc/hexane = 1/3); Spectral data matched those reported in the literature.[9]

Synthesis of dihydrophenanthrene **10**



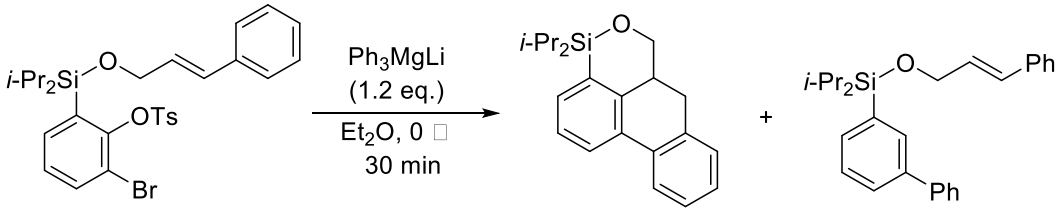
9a **10** 75%

According to the typical procedure, **10** was prepared from the reaction of 18-crown-6 (93.5 mg, 0.354 mmol), **9a** (96.4 mg, 0.177 mmol) in THF (3 mL) and Cs_2CO_3 (115 mg, 0.354 mmol) at room temperature for 8 h.

Purification by column chromatography (EtOAc/hexane = 1/50) afforded dihydrophenanthrene **10** (43.0 mg, 75%) as a white solid.

10: **mp**: 80–83 °C; $\boldsymbol{R_f}$ 0.43 (EtOAc/hexane = 1/20); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 0.89 (d, 3H, *J* = 7.5 Hz), 0.92 (d, 3H, *J* = 7.0 Hz), 1.11 (qq, 1H, *J* = 7.5, 7.0 Hz), 1.16 (d, 3H, *J* = 7.5 Hz), 1.20 (d, 3H, *J* = 7.5 Hz), 1.34 (qq, 1H, *J* = 7.5, 7.5 Hz), 2.48 (dd, 1H, *J* = 14.9, 14.6 Hz), 2.65 (dd, 1H, *J* = 14.6, 4.9 Hz), 3.18 (dddd, 1H, *J* = 14.9, 10.9, 4.9, 4.6 Hz), 3.93 (dd, 1H, *J* = 10.9, 10.9 Hz), 4.24 (dd, 1H, *J* = 10.9, 4.6 Hz), 7.21–7.25 (m, 2H), 7.31–7.40 (m, 3H), 7.72 (d, 1H, *J* = 7.8 Hz), 7.83 (dd, 1H, *J* = 7.8, 1.4 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 12.8, 13.3, 16.8, 17.1, 18.1, 31.2, 37.4, 68.5, 123.9, 125.0, 126.3, 127.1, 127.4, 127.8, 130.3, 132.8, 133.4, 134.4, 135.5, 146.2; **IR** (neat): 2943, 2862, 1462, 1099, 1080, 995, 910, 883, 736 cm^{-1}; **HRMS** (ESI): calcd. for $C_{21}H_{27}OSi$ $[M+H]^+$: 323.1826; found: 323.1831.

Reaction with *o*-bromoaryl tosylate **9b**



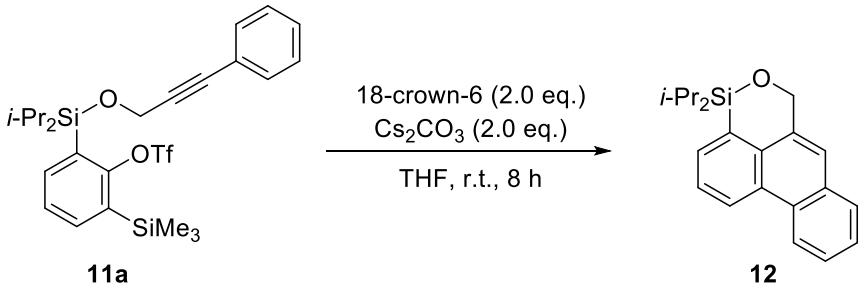
9b **10** 13% **17** 6%

To a solution of PhLi (1.76 M in Bu_2O, 424 µL, 0.746 mmol) in Et_2O (3 mL) was added PhMgBr (2.93 M in Et_2O, 138 µL, 0.404 mmol) at 0 °C, and the mixture was stirred for 30 min at this temperature. The resulting solution of Ph_3MgLi was used in the following experiment.

To a solution of bromoaryl tosylate **9b** (178 mg, 0.311 mmol) in Et_2O (6 mL) was added dropwise Ph_3MgLi (*vide supra*) at 0 °C. After stirring for 30 min at this temperature, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (EtOAc/hexane = 1/30 to 1/10) followed by PTLC ($CHCl_3$/hexane = 1/3 x2) to afford dihydrophenanthrene **10** (13.1 mg, 13%) and biaryl **17** (7.8 mg, 6%, colorless oil) along with **9b** (84.3 mg, 47%).

17: $\boldsymbol{R_f}$ 0.52 ($CHCl_3$/hexane = 1/2); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 1.08 (d, 6H, *J* = 7.6 Hz), 1.13 (d, 6H, *J* = 7.6 Hz), 1.38 (qq, 2H, *J* = 7.6, 7.6 Hz), 4.52 (dd, 2H, *J* = 4.8, 1.4 Hz), 6.36 (td, 1H, *J* = 15.8, 4.8 Hz), 6.72 (brd, 1H, *J* = 15.8 Hz), 7.23 (d, 1H, *J* = 6.8 Hz), 7.29–7.34 (m, 3H), 7.36–7.42 (m, 4H), 7.45 (dd, 1H, *J* = 7.6, 6.8 Hz), 7.54–7.60 (m, 3H), 7.62 (d, 1H, *J* = 7.6 Hz), 7.82 (s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 11.7, 17.0, 17.2, 64.4, 126.7, 127.4, 127.5, 127.6, 128.4, 128.5, 128.8, 129.0, 129.2, 129.8, 133.7, 134.0, 135.0, 137.4, 140.6, 141.8; **IR** (neat): 2943, 2866, 1462, 1384, 1122, 906, 733 cm^{-1}; **HRMS** (ESI): calcd. for $C_{27}H_{32}NaOSi$ $[M+Na]^+$: 423.2115; found: 423.2125.

Synthesis of phenanthrene **12**



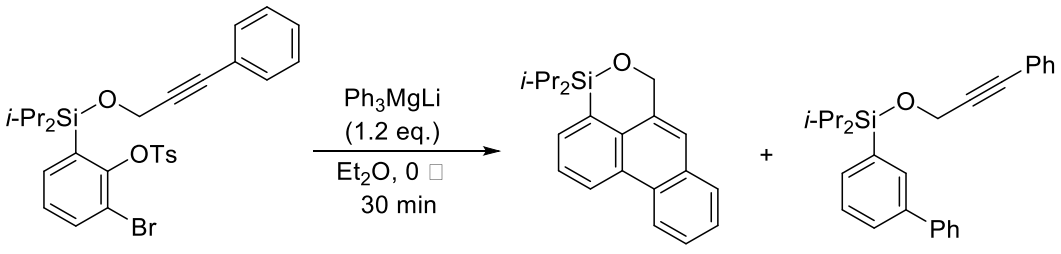
11a **12**

52%

According to the typical procedure, **12** was prepared from the reaction of 18-crown-6 (91.5 mg, 0.346 mmol), **11a** (93.8 mg, 0.173 mmol) in THF (3 mL) and Cs_2CO_3 (113 mg, 0.346 mmol) at room temperature for 8 h. Purification by column chromatography (EtOAc/hexane = 1/10) afforded phenanthrene **12** (28.8 mg, 52%) as a white solid.

12: **mp**: 48–51 °C; R_f 0.53 (EtOAc/hexane = 1/10); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 1.06 (d, 6H, *J* = 7.4 Hz), 1.08 (d, 6H, *J* = 7.5 Hz), 1.34 (qq, 2H, *J* = 7.5, 7.4 Hz), 5.32 (s, 2H), 7.53 (s, 1H), 7.59 (ddd, 1H *J* = 7.8, 7.8, 1.5 Hz), 7.64 (ddd, 1H, *J* = 7.8, 7.2, 1.5 Hz), 7.69 (dd, 1H *J* = 8.6, 8.0 Hz), 7.74 (dd, 1H, *J* = 7.2, 1.5 Hz), 7.84 (dd, 1H, *J* = 7.8, 1.5 Hz), 8.68 (brd, 1H, *J* = 8.0 Hz), 8.80 (dd, 1H *J* = 8.6, 1.5 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 13.3, 17.2, 17.7, 67.6, 122.5, 123.3, 124.4, 125.7, 126.4, 126.8, 128.3, 129.97, 130.00, 130.4, 131.2, 132.1, 133.9, 135.5; **IR** (neat): 2943, 2866, 1462, 1408, 1103, 1045, 906, 732 cm^{-1}; **HRMS** (ESI): calcd. for $C_{21}H_{25}OSi$ $[M+H]^+$: 321.1669; found: 321.1670.

Reaction with *o*-bromoaryl tosylate **11b**



11b **12** **18**

0% 29%

To a solution of PhLi (1.76 M in Bu_2O, 349 μL, 0.614 mmol) in Et_2O (2 mL) was added PhMgBr (2.93 M in Et_2O, 114 μL, 0.333 mmol) at 0 °C, and the mixture was stirred for 30 min at this temperature. The resulting solution of Ph_3MgLi was used in the following experiment.

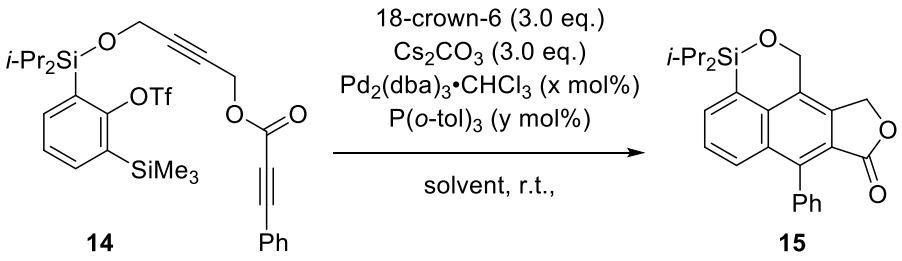
To a solution of bromoaryl tosylate **11b** (146 mg, 0.256 mmol) in Et_2O (5 mL) was added dropwise Ph_3MgLi (*vide supra*) at 0 °C. After stirring for 30 min at this temperature, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (EtOAc/hexane = 1/20) to afford biaryl **18** (29.1 mg, 29%) as colorless oil along with **11b**

(91.8 mg, 63%)

18: $\boldsymbol{R_f}$ 0.45 (EtOAc/hexane = 1/5); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 1.09 (d, 6H, J = 6.9 Hz), 1.15 (d, 6H, J = 7.6 Hz), 1.40 (qq, 2H, J = 7.6, 6.9 Hz), 4.69 (s, 2H), 7.27–7.34 (m, 4H), 7.37–7.40 (m, 4H), 7.46 (dd, 1H J = 7.6, 7.6 Hz), 7.57–7.64 (m, 4H), 7.86 (s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 11.7, 16.9, 17.0, 52.9, 85.1, 87.7, 123.2, 127.5, 127.6, 128.4, 128.5, 128.7, 129.0, 132.0, 133.8, 134.0, 134.5, 140.8, 141.8 (several signals overlapped); **IR** (neat): 2943, 2862, 1462, 1369, 1083, 756 cm^{-1}; **HRMS** (ESI): calcd. for $C_{27}H_{30}KOSi$ $[M+K]^+$: 437.1698; found: 437.1705.

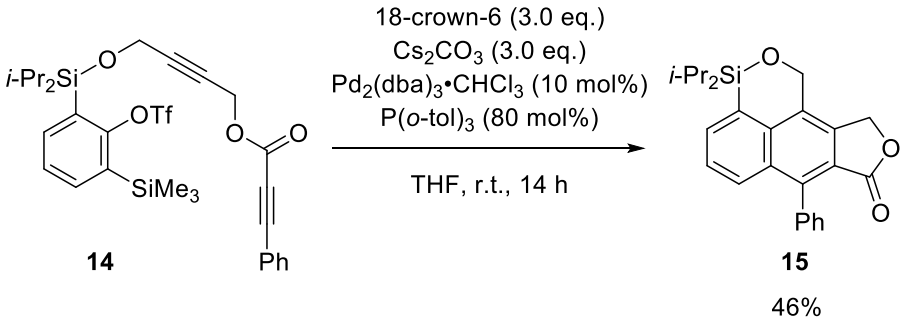
2-3. Pd-catalyzed (2+2+2) cycloaddition

Optimization study for (2+2+2) cycloaddition of **14**



entry	x	y	solvent	concentration	yield[a]
1	5[b]	40	THF	0.025 M	28%
2	5[b]	40	MeCN	0.025 M	15%
3	5	40	THF	0.025 M	38%
4	10	80	THF	0.025 M	46%
5	10	80	THF	0.125 M	24%
6	10	80	THF	0.01 M	44%

[a] isolated yield, [b] $Pd_2(dba)_3$ instead of $Pd_2(dba)_3$•$CHCl_3$

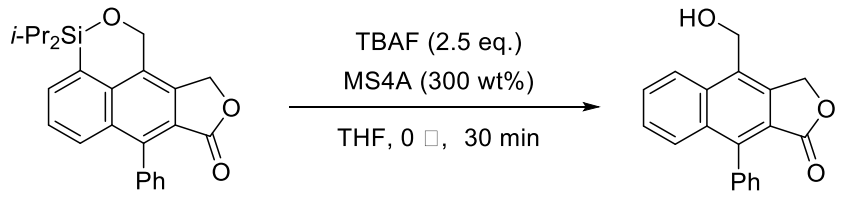


$Pd_2(dba)_3 \cdot CHCl_3$ (19.5 mg, 0.0188 mmol) and P(*o*-tol)$_3$ (45.8 mg, 0.150 mmol) were dissolved in THF (1.5 mL), and the mixture was stirred at room temperature for 15 min. The catalyst solution was added through a cannula to a solution of 18-crown-6 (149 mg, 0.563 mmol), Cs_2CO_3 (183 mg, 0.563 mmol), and **14** (117 mg, 0.188 mmol) in THF (4 mL) at room temperature. More THF (1.5 mL) was used to wash the catalyst through. After stirring for 14 h at room temperature, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/5) to afford naphthalene **15** (35.0 mg, 46%) as pale yellow oil.

15: R_f 0.38 (EtOAc/hexane = 1/3); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 1.07 (d, 6H, *J* = 7.6 Hz), 1.11 (d, 6H, *J* = 6.8 Hz), 1.36 (qq, 2H, *J* = 7.6, 6.8 Hz), 5.36 (s, 2H), 5.37 (s, 2H), 7.36‑7.40 (m, 2H), 7.49–7.56 (m, 4H), 7,76 (dd, 1H, *J* = 6.2, 1.4 Hz), 7,89 (dd, 1H, *J* = 8.2, 1.4 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 12.8, 16.7,

17.2, 63.1, 67.1, 119.2, 125.9, 128.3, 128.5, 129.1, 129.4, 130.41, 130.43, 133.0, 134.3, 134.8, 135.7, 138.2, 141.7, 170.2; **IR** (neat): 2966, 1716, 1396, 1219, 1138, 910, 871, 844, 733 cm^{-1}; **HRMS** (ESI): calcd. for $C_{25}H_{27}O_3Si$ $[M+H]^+$: 403.1724; found: 403.1733.

Protodesilylation of **15**



15 **16** 78%

To a solution of **15** (22.3 mg, 0.0554 mmol) and MS4A (66.9 mg) in THF (2 mL) was added tetrabutylammonium fluoride (TBAF, 1.0 M in THF, 138 µL, 0.138 mmol). After stirring for 30 min at 0 ℃, the reaction was quenched by adding saturated aqueous NH_4Cl. The resulting suspension was filtered through a Celite® pad (washed with EtOAc) and extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, EtOAc/hexane = 1/1) to afford alcohol **16** (12.5 mg, 78%) as a white solid.

16: **mp**: 150–155 °C; $\boldsymbol{R_f}$ 0.42 (EtOAc/hexane = 2/1); **^{1}H NMR** (600 MHz, $CDCl_3$): δ 2.26 (brs, 1H), 5.30 (s, 2H), 5.60 (s, 2H), 7.26–7.32 (m, 2H), 7.47–7.52 (m, 4H), 7,69 (t, 1H, J = 7.6 Hz), 7,80 (brd, 1H, J = 8.9 Hz), 8.16 (brd, 1H, J = 8.2 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 60.0, 68.6, 119.9, 123.7, 126.8, 128.3, 128.5, 129.2, 129.3, 129.4, 130.2, 133.4, 134.0, 134.8, 138.9, 142.5, 170.5; **IR** (neat): 3394, 1747, 1620, 1442, 1342, 1207, 1122, 1060, 1022, 910, 767, 732 cm^{-1}; **HRMS** (ESI): calcd. for $C_{19}H_{14}NaO_3$ $[M+H]^+$: 313.0835; found: 313.0841.

3. References

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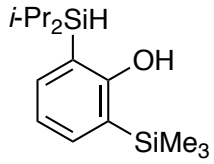

4a

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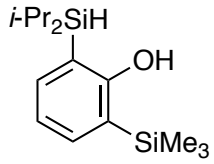

4a

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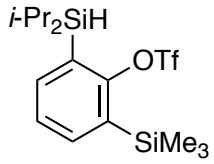

5a

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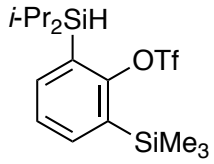
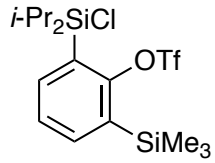
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31444.86	***Temperature (degree C)***	22.500							



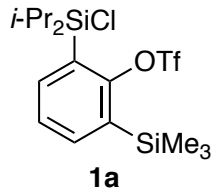
5a

Acquisition Time (sec)	3.4918	***Date***	25 Jun 2020 20:25:04		***File Name***	F:\NMR Œ t H \tawatari\TT0474-1Hretake-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	22.200							



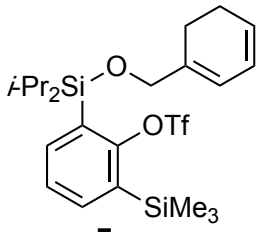
1a

Acquisition Time (sec)	0.8336	***Date***	25 Jun 2020 20:25:12		***File Name***	F:\NMR_OE_t_H_\tawatari\TT0474-13Cretake-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	22.700								



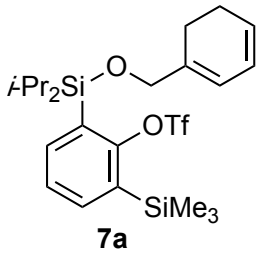
1a

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	18 Jun 2021 12:58:50			***File Name***	F:\NMR Œ t H \tawatari\TT0530-1H_proton-1-2.als						
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.300												



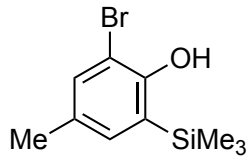
7a

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	08 Aug 2020 12:31:08		***File Name***	F:\NMR Œ t H \tawatari\TT0530-13C carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	401	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	21.500							



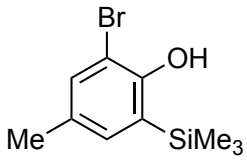
7a

Acquisition Time (sec)	3.4918	***Date***	15 Oct 2020 17:30:10		***File Name***	F:\NMR Œ t H \tawatari\TT0616column-1.als	***Frequency (MHz)***	500.16	***Nucleus*** 1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D	
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	18.200						



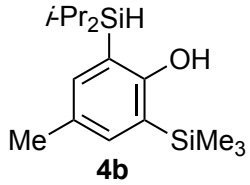
3b

Acquisition Time (sec)	0.8336	***Date***	18 Oct 2020 23:11:34		***File Name***	F:\NMR Œ t H \tawatari\TT0616-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	18.700							



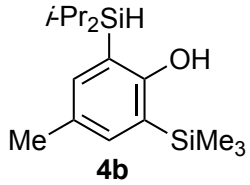
3b

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	18 Jun 2021 16:00:50			***File Name***	F:\NMR Œ t H \tawatari\TT0671-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	19.200								



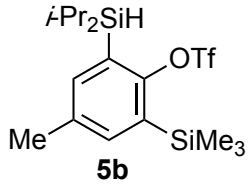
4b

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	25 Dec 2020 14:14:30		***File Name***	F:\NMR Œ t H \tawatari\TT0671-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	256	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	19.300							



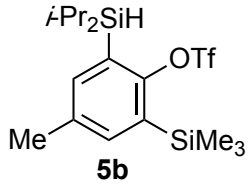
4b

Acquisition Time (sec)	3.4918	***Date***	20 Oct 2020 21:46:24			***File Name***	F:\NMR Œ t H \tawatari\TT0622column-fr7-9-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	20.500								



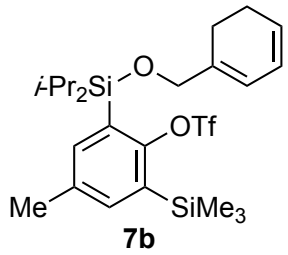
5b

Acquisition Time (sec)	0.8336	***Date***	20 Oct 2020 22:51:38		***File Name***	F:\NMR Œ t H \tawatari\TT0622-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	20.800							



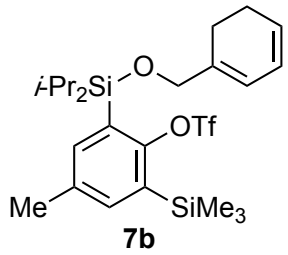
5b

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	06 Jan 2021 22:28:36			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0677-1H-retake_proton-1-1.als						
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	19.000												



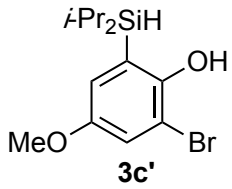
7b

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	06 Jan 2021 22:28:14		
File Name	F:\NMR Œ t H	\tawatari\TT0677-13C-retake_carbon-1.als	***Frequency (MHz)***	150.00		***Number of Transients***	256	***Original Points Count*** 26214	***Points Count*** 26214
Pulse Sequence	carbon_cool.jxp	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	37876.77	***Temperature (degree C)*** 19.000	



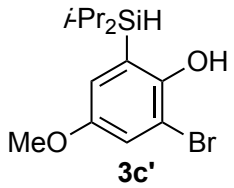
7b

Acquisition Time (sec)	3.4918	***Date***	03 Mar 2020 15:00:02		***File Name***	F:\NMR Œ t H \tawatari\TT0367column-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	19.700							



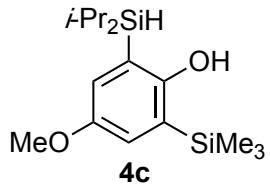
3c'

Acquisition Time (sec)	0.8336	***Date***	03 Mar 2020 15:03:30		***File Name***	F:\NMR Œ t H \tawatari\TT0367-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	20.000							



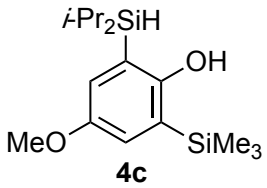
3c'

Acquisition Time (sec)	3.4918	***Date***	02 Mar 2020 23:13:08		***File Name***	F:\NMR Œ t H \tawatari\TT0375-1H-retake-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D	
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	19.400							



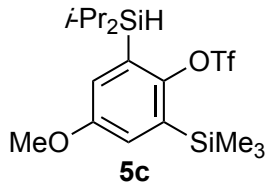
4c

Acquisition Time (sec)	0.8336	***Date***	02 Mar 2020 23:16:54		***File Name***	F:\NMR Œ t H \tawatari\TT0375-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	128	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	20.500							



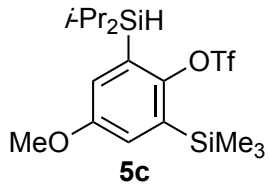
4c

Acquisition Time (sec)	3.4918	***Date***	16 Jan 2020 17:56:14		***File Name***	F:\NMR Œ t H \tawatari\TT0316ptlc1-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	19.500							



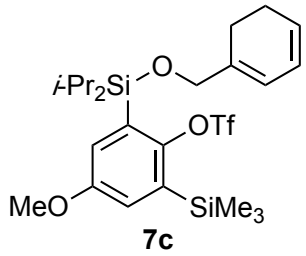
5c

Acquisition Time (sec)	0.8336	***Date***	16 Jan 2020 22:03:18			***File Name***	F:\NMR Œ t H \tawatari\TT0316-13C-1.als		***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	single_pulse_dec		***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	20.000									



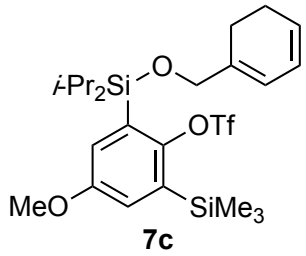
5c

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	04 Dec 2020 21:33:42			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0581-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.500								



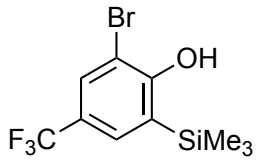
7c

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	04 Dec 2020 21:39:44		***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0581-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	257	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	20.600							



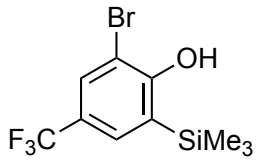
7c

Acquisition Time (sec)	3.4918	***Date***	18 Oct 2020 22:18:54		***File Name***	F:\NMR Œ t H \tawatari\TT0597-1H-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	18.500							



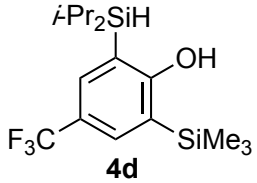
3d

Acquisition Time (sec)	0.8336	***Date***	18 Oct 2020 22:18:46		***File Name***	F:\NMR Œ t H \tawatari\TT0597-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	19.300							



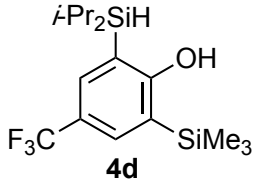
3d

Acquisition Time (sec)	3.4918	***Date***	03 Oct 2020 14:38:20			***File Name***	F:\NMR Œ t H \tawatari\TT0598column-1.als		***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	single_pulse.ex2		***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	18.600									



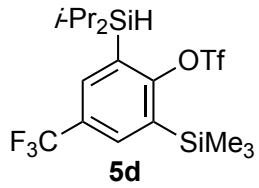
4d

Acquisition Time (sec)	0.8336	***Date***	14 Oct 2020 23:18:30		***File Name***	F:\NMR Œ t H \tawatari\TT0598-13C.als	***Frequency (MHz)***	125.00	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31444.86	***Temperature (degree C)***	19.200							



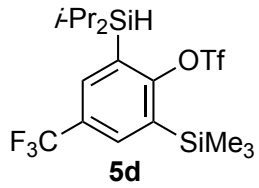
4d

Acquisition Time (sec)	3.4918	***Date***	14 Oct 2020 16:51:48		***File Name***	F:\NMR Œ t H \tawatari\TT0600ptlc1-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	18.400							



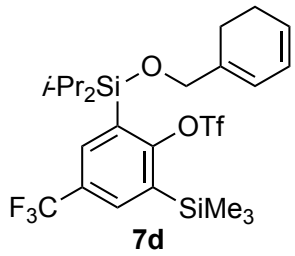
5d

Acquisition Time (sec)	0.8336	***Date***	05 Oct 2020 17:52:44		***File Name***	F:\NMR Œ t H \tawatari\TT0600-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	18.900							



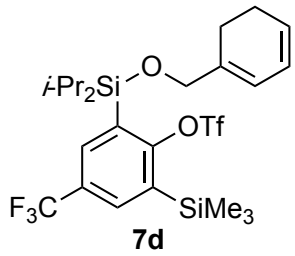
5d

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	25 Dec 2020 23:04:18			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0675-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	19.200								



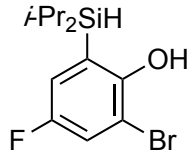
7d

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE		***Date***	25 Dec 2020 23:03:54		***File Name***	F:\NMR Œ t H \tawatari\TT0675-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	1024	***Original Points Count*** 26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	19.700						



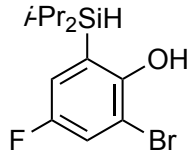
7d

Acquisition Time (sec)	3.4918	***Date***	02 Mar 2020 23:18:58		***File Name***	F:\NMR Œ t H \tawatari\TT0370-1H-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	19.300							



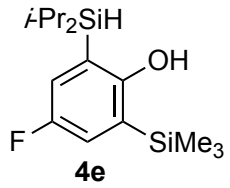
3e'

Acquisition Time (sec)	0.8336	***Date***	28 Feb 2020 21:03:34		***File Name***	F:\NMR Œ t H \tawatari\TT0370-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	128	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	19.800							



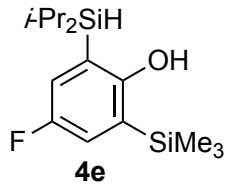
3e'

Acquisition Time (sec)	3.4918	***Date***	02 Mar 2020 22:48:52		***File Name***	F:\NMR Œ t H \tawatari\TT0376-1H-retake-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	19.700							



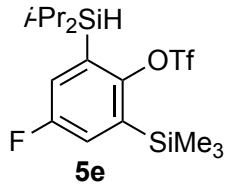
4e

Acquisition Time (sec)	0.8336	***Date***	28 Feb 2020 21:04:34		***File Name***	F:\NMR Œ t H \tawatari\TT0376-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	128	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	20.300							



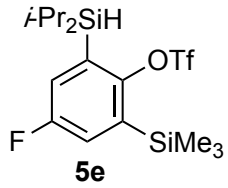
4e

Acquisition Time (sec)	3.4918	***Date***	07 Mar 2020 17:50:06		***File Name***	F:\NMR Œ t H \tawatari\TT0384-1H-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	18.900							



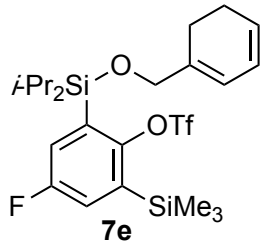
5e

Acquisition Time (sec)	0.8336	***Date***	07 Mar 2020 17:50:26		***File Name***	F:\NMR Œ t H \tawatari\TT0384-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	19.200							



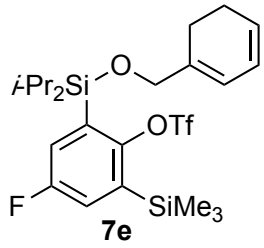
5e

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	22 Jun 2021 08:48:32			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0562-1H_proton-1-1.als						
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.500												



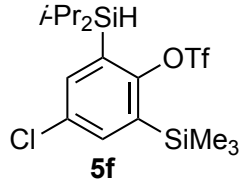
7e

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	12 Sep 2020 17:23:24		
File Name	F:\NMR Œ t H	\tawatari\TT0562-13Cretake_carbon-1.als	***Frequency (MHz)***	150.00		***Number of Transients***	254	***Original Points Count*** 26214	***Points Count*** 26214
Pulse Sequence	carbon_cool.jxp	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	37876.77	***Temperature (degree C)*** 21.600	



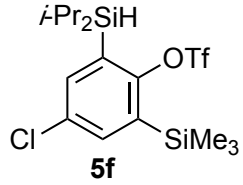
7e

Acquisition Time (sec)	3.4918	***Date***	23 Oct 2020 23:29:38		***File Name***	F:\NMR Œ t H \tawatari\TT0627-1H-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	20.900							



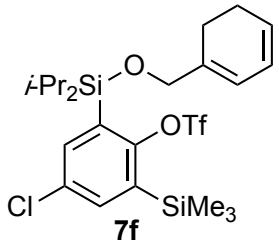
5f

Acquisition Time (sec)	0.8336	***Date***	23 Oct 2020 23:29:50		***File Name***	F:\NMR Œ t H \tawatari\TT0627-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	20.900							



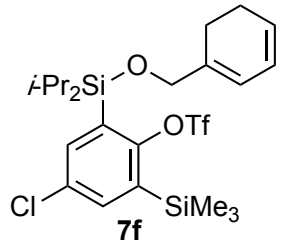
5f

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	16 Jan 2021 15:07:08			***File Name***	F:\NMR Œ t_H \tawatari\TT0689-1H_proton-1-1.als						
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	19.300												



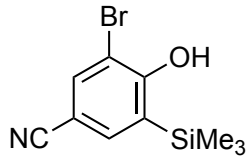
7f

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE		***Date***	16 Jan 2021 15:06:46		***File Name***	F:\NMR Œ t H \tawatari\TT0689-13C carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	400	***Original Points Count*** 26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	19.400						



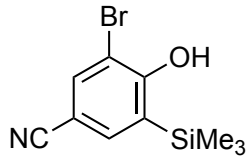
7f

Acquisition Time (sec)	3.4918	***Date***	23 Sep 2020 21:20:26		***File Name***	F:\NMR Œ t H \tawatari\TT0588column2-1.als	***Frequency (MHz)***	500.16	***Nucleus*** 1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D	
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	18.600						



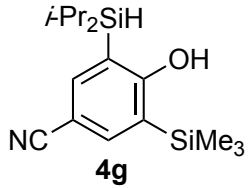
3g

Acquisition Time (sec)	0.8336	***Date***	04 Dec 2020 21:50:36		***File Name***	F:\NMR Œ t H \tawatari\TT0588-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	20.900							



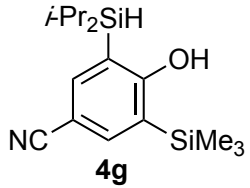
3g

Acquisition Time (sec)	3.4918	***Date***	25 Sep 2020 01:42:00		***File Name***	F:\NMR Œ t H \tawatari\TT0591-1H-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	18.500							



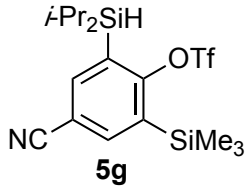
4g

Acquisition Time (sec)	0.8336	***Date***	14 Oct 2020 23:14:14		***File Name***	F:\NMR Œ t H \tawatari\TT0591-13C.als	***Frequency (MHz)***	125.00	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31444.86	***Temperature (degree C)***	18.900							



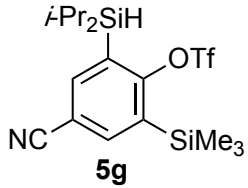
4g

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	04 Dec 2020 22:00:56			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0652-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.700								



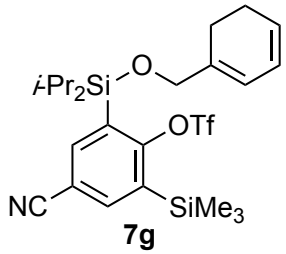
5g

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	04 Dec 2020 21:53:58		***File Name***	F:\NMR Œ t H \tawatari\TT0652-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	128	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	20.800							



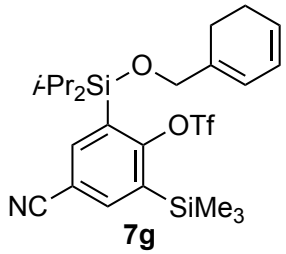
5g

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	11 Dec 2020 13:00:14			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0657-1Hretake_proton-1-1.als						
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.900												



7g

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	11 Dec 2020 12:59:08		
File Name	F:\NMR Œ t H	\tawatari\TT0657-13Cretake_carbon-1.als	***Frequency (MHz)***	150.00		***Number of Transients***	256	***Original Points Count*** 26214	***Points Count*** 26214
Pulse Sequence	carbon_cool.jxp	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	37876.77	***Temperature (degree C)*** 20.800	



7g

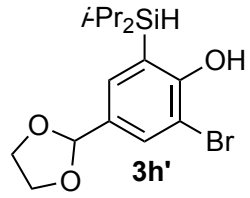
chloroform-d

77.2119
77.0000
76.7881
0.4114
17.7951
17.3327
13.2483
22.8718
22.2360
66.9142
125.6856
124.3658
118.8942
142.6494
142.4182
137.6017
137.2934
133.2764
157.8697
118.2488
112.4112
121.7553
119.6167
117.4878
115.3493

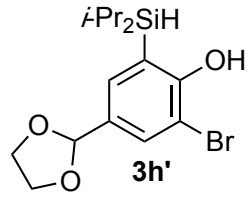
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

ppm

Acquisition Time (sec)	3.4918	***Date***	22 Jan 2020 11:35:38		***File Name***	F:\NMR Œ t H \tawatari\TT0327-1H-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	19.000							

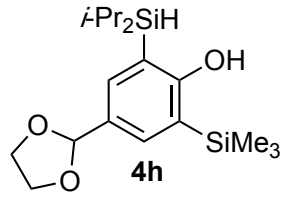


Acquisition Time (sec)	0.8336	***Date***	22 Jan 2020 11:39:58		***File Name***	F:\NMR Œ t H \tawatari\TT0327-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	20.200							



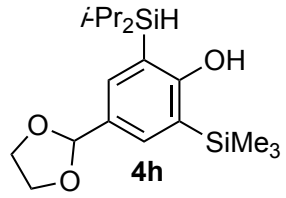
3h'

Acquisition Time (sec)	3.4918	***Date***	23 Jan 2020 11:55:50		***File Name***	F:\NMR Œ t H \tawatari\TT0329column2-1.als	***Frequency (MHz)***	500.16	***Nucleus*** 1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D	
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	19.300						



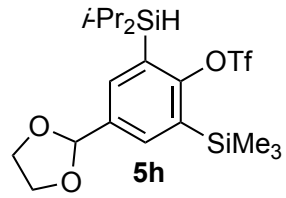
4h

Acquisition Time (sec)	0.8336	***Date***	02 Mar 2020 18:18:40		***File Name***	F:\NMR Œ t H \tawatari\TT0329-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	19.900							



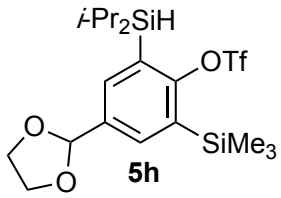
4h

Acquisition Time (sec)	3.4918	***Date***	05 Mar 2020 22:38:16		***File Name***	F:\NMR Œ t H \tawatari\TT0382-1H-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	18.800							



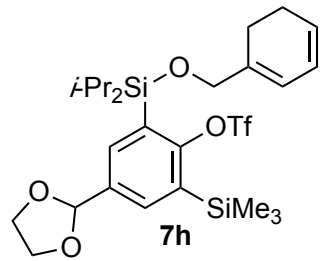
5h

Acquisition Time (sec)	0.8336	***Date***	05 Mar 2020 23:19:38		***File Name***	F:\NMR Œ t H \tawatari\TT0382-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	19.000							



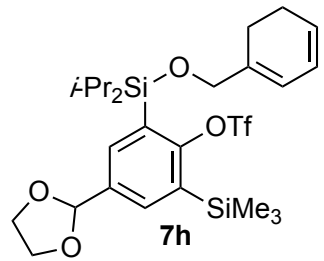
5h

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	22 Jun 2021 23:41:20			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0542-1H_proton-1-1.als						
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.300												



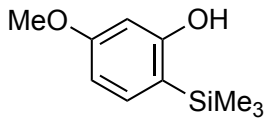
7h

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	26 Aug 2020 16:42:48		***File Name***	F:\NMR Œ t H \tawatari\TT0542-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	449	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	21.400							



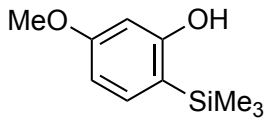
7h

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	04 Dec 2020 22:10:38			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0537-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.300								



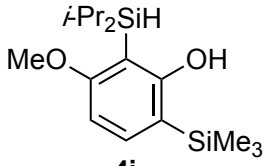
3i'

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	04 Dec 2020 22:14:06		***File Name***	F:\NMR Œ t H \tawatari\TT0537-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	421	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	21.400							



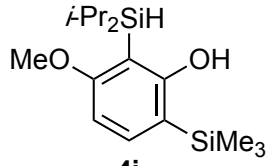
3i'

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	04 Dec 2020 22:20:00			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0538-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.100								



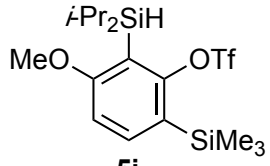
4i

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	04 Dec 2020 22:27:22	***File Name***	F:\NMR Œ t H \tawatari\TT0538-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	349	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	21.200						



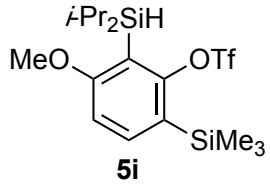
4i

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	23 Jun 2021 19:47:10			***File Name***	F:\NMR Œ t H \tawatari\TT0541-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.400								



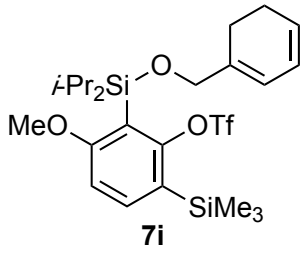
5i

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	04 Dec 2020 22:27:54			***File Name***	F:\NMR Œ t H \tawatari\TT0541-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	578	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	21.500								



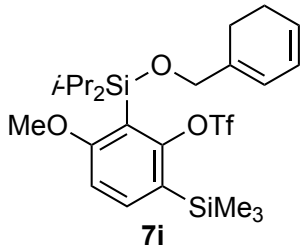
5i

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	23 Jun 2021 19:55:54			***File Name***	F:\NMR Œ t_H \tawatari\TT0546-1Hretake_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.600								



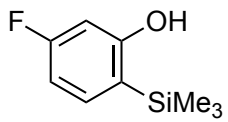
7i

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	05 Sep 2020 00:43:52		
File Name	F:\NMR Œ t H	\tawatari\TT0546-13C_carbon-1-1retake.als		***Frequency (MHz)***	150.00	***Number of Transients***	354	***Original Points Count***	26214	***Points Count***	26214
Pulse Sequence	carbon_cool.jxp	***Solvent***	CHLOROFORM-D	***Sweep Width (Hz)***	37876.77	***Temperature (degree C)***	21.700				



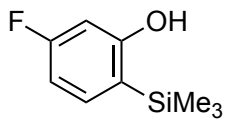
7i

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	23 Jun 2021 20:16:18			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0564-1H_proton-1-1.als						
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.600												



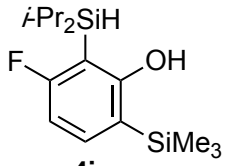
3j'

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	04 Dec 2020 22:35:12	***File Name***	F:\NMR Œ t H \tawatari\TT0564-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	101	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	21.500						



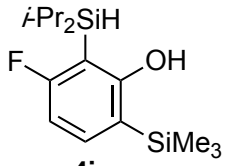
3j'

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	04 Dec 2020 22:42:50			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0566-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.400								



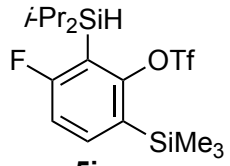
4j

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	04 Dec 2020 22:36:16			***File Name***	F:\NMR Œ t H \tawatari\TT0566-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	124	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	21.400								



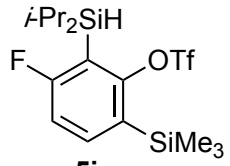
4j

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	23 Jun 2021 20:43:20			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0570-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.500								



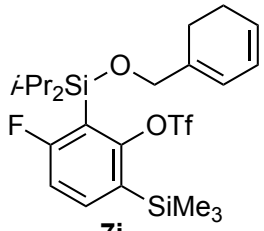
5j

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	04 Dec 2020 22:37:18			***File Name***	F:\NMR Œ t H \tawatari\TT0570-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	256	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	21.500								



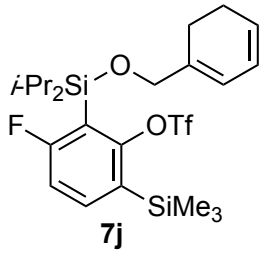
5j

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	23 Jun 2021 20:59:30			***File Name***	F:\NMR Œ t H \tawatari\TT0661-1H_proton-1-1.als						
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.900												



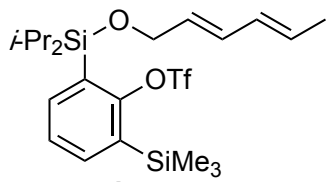
7j

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	11 Dec 2020 12:59:46		***File Name***	F:\NMR Œ t H \tawatari\TT0661-13C carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	512	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	21.000							



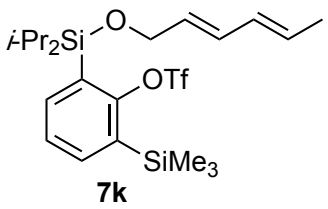
7j

Acquisition Time (sec)	3.4918	***Date***	07 Jul 2020 23:53:24		***File Name***	F:\NMR Œ t H \tawatari\TT0487-1H-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	22.600							



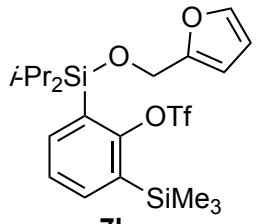
7k

Acquisition Time (sec)	0.8336	***Date***	07 Jul 2020 23:53:36		***File Name***	F:\NMR Œ t H \tawatari\TT0487-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	23.100							



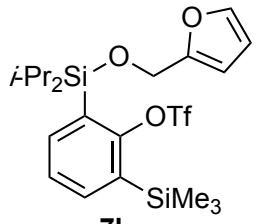
7k

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	22 Feb 2021 15:26:40			***File Name***	F:\NMR Œ t H \tawatari\TT0722-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.000								



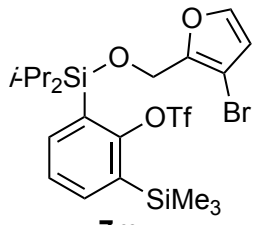
7l

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	22 Feb 2021 15:26:20		***File Name***	F:\NMR Œ t H \tawatari\TT0722-13C carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	256	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	20.300							



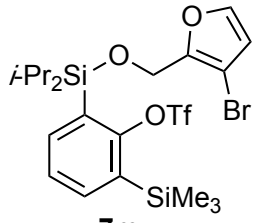
7l

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	22 Dec 2020 20:37:36			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0669-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	19.300								



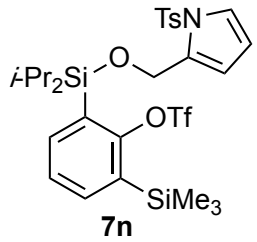
7m

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	22 Dec 2020 20:32:48			***File Name***	F:\NMR Œ t H \tawatari\TT0669-13C carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	256	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	19.400								



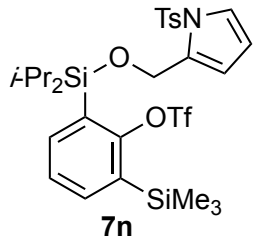
7m

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	24 Feb 2021 16:28:08			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0728-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.500								



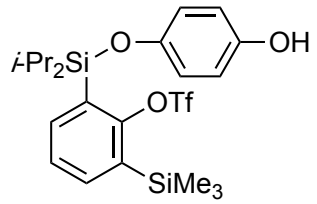
7n

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	24 Feb 2021 16:27:50			***File Name***	F:\NMR Œ t H \tawatari\TT0728-13C carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	257	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	20.800								



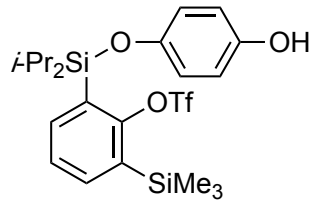
7n

Acquisition Time (sec)	3.4918	***Date***	24 Jun 2021 20:43:44		***File Name***	F:\NMR Œ t H \tawatari\compound10proton-2.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	20.200							



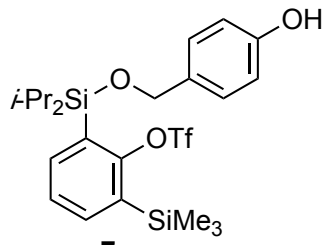
7o

Acquisition Time (sec)	0.8336	***Date***	24 Jun 2021 20:44:02		***File Name***	F:\NMR Œ t H \tawatari\compound10carbon-2.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	20.900							



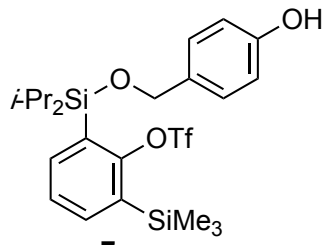
7o

Acquisition Time (sec)	3.4918	***Date***	24 Jun 2021 20:16:08		***File Name***	F:\NMR Œ t H \tawatari\TT0044-1H-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	22.200							



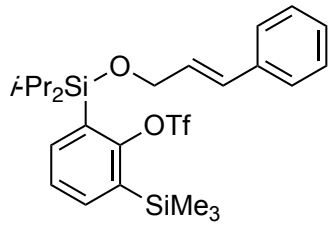
7p

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	07 Jul 2020 13:56:48	***File Name***	F:\NMR Œ t H \tawatari\TT0044-13C carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	333	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	21.200						



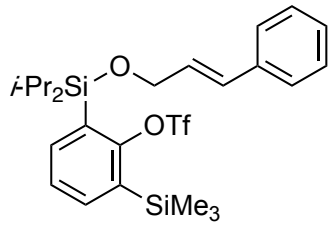
7p

Acquisition Time (sec)	3.4918	***Date***	27 Jun 2020 14:02:52			***File Name***	F:\NMR Œ t H \tawatari\TT0479-1H-1.als		***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	single_pulse.ex2		***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	22.400									



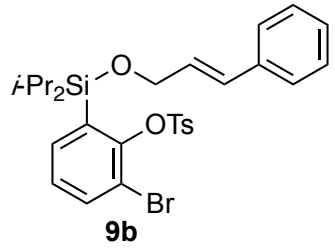
9a

Acquisition Time (sec)	0.8336	***Date***	27 Jun 2020 14:03:06		***File Name***	F:\NMR Œ t H \tawatari\TT0479-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	22.900							



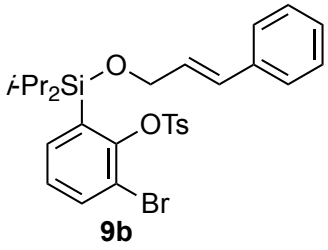
9a

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	14 Jan 2021 15:19:58			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0685-1H_proton-1-1.als						
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	18.400												



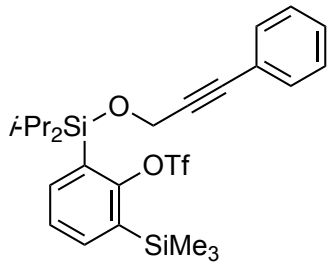
9b

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE		***Date***	14 Jan 2021 15:19:28		***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0685-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	256	***Original Points Count*** 26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	18.600						



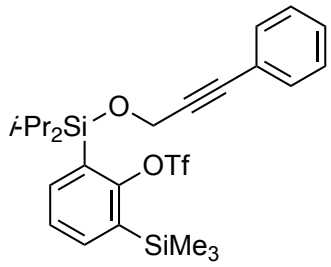
9b

Acquisition Time (sec)	3.4918	***Date***	30 Jun 2020 22:12:30		***File Name***	F:\NMR Œ t H \tawatari\TT0484column-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	22.800							



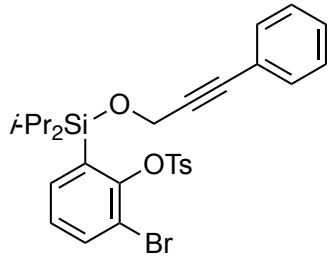
11a

Acquisition Time (sec)	0.8336	***Date***	30 Jun 2020 23:12:00		***File Name***	F:\NMR Œ t H \tawatari\TT0484-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	23.300							



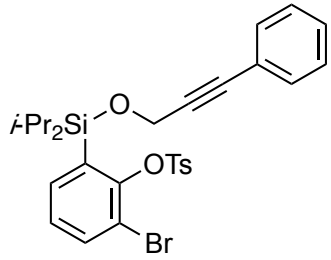
11a

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	17 Feb 2021 13:59:44			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0714-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.900								



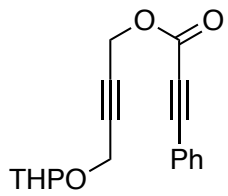
11b

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE		***Date***	17 Feb 2021 13:59:24		***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0714-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	256	***Original Points Count*** 26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	21.000						



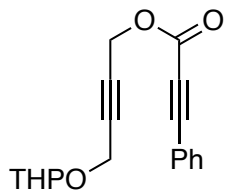
11b

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	10 Mar 2021 19:14:24			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0746-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.700								



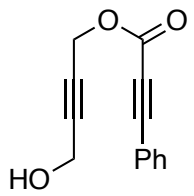
S11

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	10 Mar 2021 19:13:34	***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0746-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	256	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	20.900						



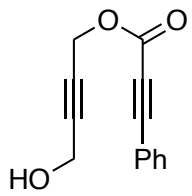
S11

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	13 Mar 2021 21:19:18			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0747-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.900								



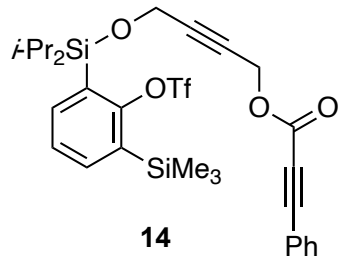
13

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	13 Mar 2021 21:19:00	***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0747-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	128	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent*** CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	20.700						



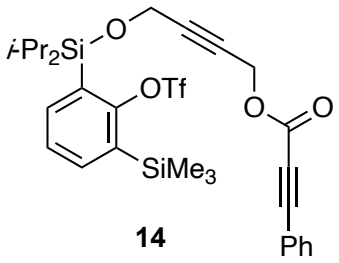
13

Acquisition Time (sec)	3.4919	***Date***	18 Jun 2020 22:49:08			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0467-1H.als	***Frequency (MHz)***	500.00	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.11	***Temperature (degree C)***	22.000								



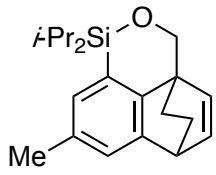
14

Acquisition Time (sec)	0.8336	***Date***	18 Jun 2020 22:49:38		***File Name***	F:\NMR Œ t H \tawatari\TT0467-13C.als	***Frequency (MHz)***	125.00	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31444.86	***Temperature (degree C)***	22.600							



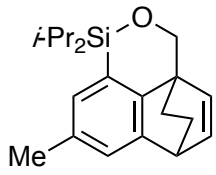
14

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	05 Feb 2021 21:14:56			***File Name***	F:\NMR Œ t H \tawatari\TT0705-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.100								



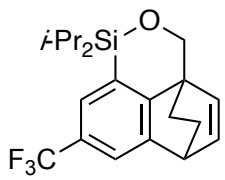
8b

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	05 Feb 2021 21:14:40		***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0705-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	256	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	20.200							



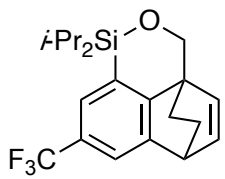
8b

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	26 Apr 2021 14:38:00			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0678-1H-retake_proton-1-1.als						
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.600												



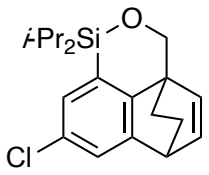
8d

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	26 Apr 2021 14:37:10		
File Name	F:\NMR Œ t H	\tawatari\TT0678-13C-retake_carbon_copy2-1.als				***Frequency (MHz)***	150.00	***Number of Transients*** 53	***Original Points Count*** 26214
Points Count	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D	***Sweep Width (Hz)***	37876.77	***Temperature (degree C)*** 20.700	



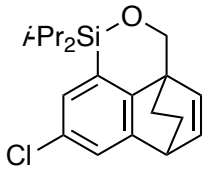
8d

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	17 Dec 2020 11:28:24			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0666-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	19.300								



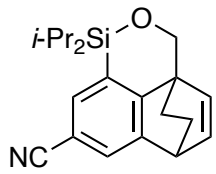
8f

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	17 Dec 2020 11:27:46	***File Name***	F:\NMR Œ t H \tawatari\TT0666-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	256	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	19.300						



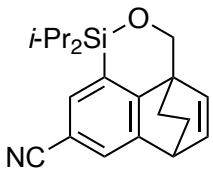
8f

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	15 Dec 2020 00:10:56			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0664_proton-1-1.als	***Frequency (MHz)***	600.17
Nucleus	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	19.800								



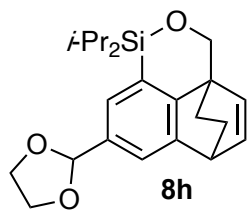
8g

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	15 Dec 2020 00:10:24	***File Name***	F:\NMR Œ t H \tawatari\TT0664_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	1024	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent*** CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	20.300						



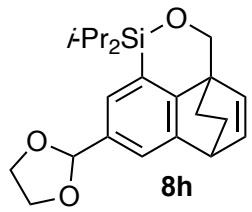
8g

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	16 Dec 2020 19:28:54			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0554-1H-retake_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.300								



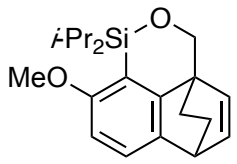
8h

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	11 Sep 2020 00:51:20		
File Name	F:\NMR Œ t H	\tawatari\TT0554-13C-retake2_carbon-1.als	***Frequency (MHz)***	150.00	***Number of Transients***	351	***Original Points Count***	26214	***Points Count***	26214
Pulse Sequence	carbon_cool.jxp	***Solvent***	CHLOROFORM-D	***Sweep Width (Hz)***	37876.77	***Temperature (degree C)***	21.400			



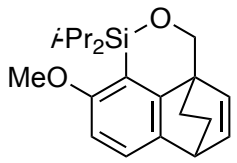
8h

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	23 Jun 2021 20:05:58			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0555-1Hretake_proton-1-1.als						
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	21.700												



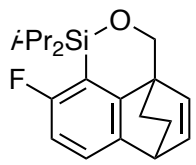
8i

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	05 Sep 2020 00:45:32		
File Name	F:\NMR Œ t H	\tawatari\TT0555-13Cretake_carbon-1.als	***Frequency (MHz)***	150.00		***Number of Transients***	364	***Original Points Count*** 26214	***Points Count*** 26214
Pulse Sequence	carbon_cool.jxp	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	37876.77	***Temperature (degree C)*** 21.600	



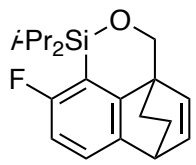
8i

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	23 Jun 2021 21:11:28			***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0665-retake_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	19.700								



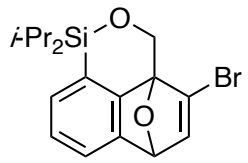
8j

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	15 Dec 2020 21:18:44		
File Name	F:\NMR Œ t H	\tawatari\TT0665-retake_carbon-1.als	***Frequency (MHz)***	150.00		***Number of Transients***	1024	***Original Points Count*** 26214	***Points Count*** 26214
Pulse Sequence	carbon_cool.jxp	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	37876.77	***Temperature (degree C)*** 20.100	



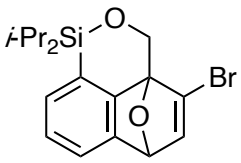
8j

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	24 Dec 2020 19:24:38			***File Name***	F:\NMR Œ t H \tawatari\TT0672-1H_proton-1-1.als		
Frequency (MHz)	600.17	***Nucleus***	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	19.400								



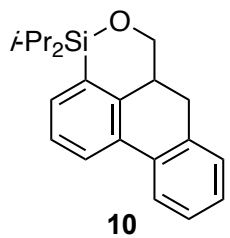
8m

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	24 Dec 2020 19:24:08		***File Name***	F:\NMR Œ t H \tawatari\TT0672-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	256	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	19.500							



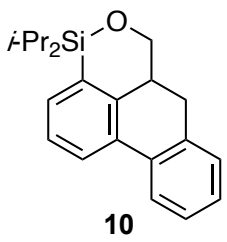
8m

Acquisition Time (sec)	3.4918	***Date***	03 Jul 2020 02:14:06			***File Name***	F:\NMR_OE_t_H_\tawatari\TT0485-1H-retake-1.als		***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	single_pulse.ex2		***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	22.500									



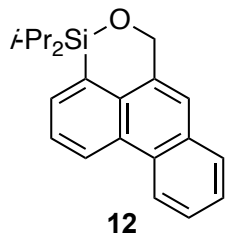
10

Acquisition Time (sec)	0.8336	***Date***	03 Jul 2020 02:26:58		***File Name***	F:\NMR Œ t H \tawatari\TT0485-13C-1.als	***Frequency (MHz)***	125.77	***Nucleus***	13C
Number of Transients	256	***Original Points Count***	26214	***Points Count*** 26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	22.900							



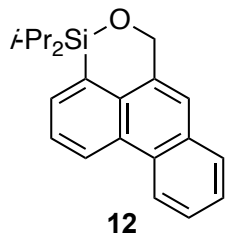
10

Acquisition Time (sec)	3.4918	***Date***	30 Jun 2020 13:36:02			***File Name***	F:\NMR Œ t H \tawatari\TT0051column1ptlc2-1.als	***Frequency (MHz)***	500.16	***Nucleus***	1H
Number of Transients	8	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	single_pulse.ex2	***Solvent***	CHLOROFORM-D		
Sweep Width (Hz)	7507.39	***Temperature (degree C)***	20.000								



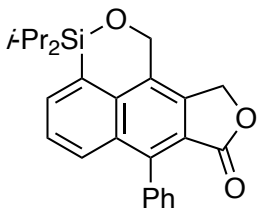
12

Acquisition Time (sec)	0.8336	***Date***	30 Jun 2020 13:35:30			***File Name***	F:\NMR Œ t H \tawatari\TT0051column1ptlc2carbon-1.als		***Frequency (MHz)***	125.77
Nucleus	13C	***Number of Transients***	256	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	single_pulse_dec	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	31446.06	***Temperature (degree C)***	20.600								



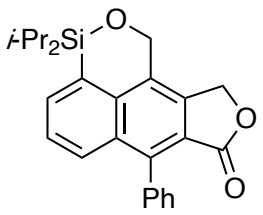
12

Acquisition Time (sec)	1.4524	***Comment***	single_pulse	***Date***	03 Jul 2020 02:29:02		***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0470-1H_proton-1.als	***Frequency (MHz)***	600.00
Nucleus	1H	***Number of Transients***	8	***Original Points Count***	13107	***Points Count*** 13107	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9024.44	***Temperature (degree C)***	20.000							



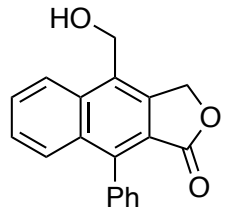
15

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	03 Jul 2020 02:29:26	***File Name***	F:\NMR_Œ_t_H_\tawatari\TT0470-13C_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	401	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence***	carbon_cool.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	20.000						



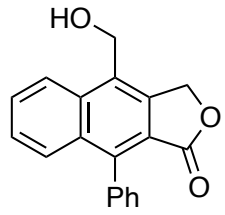
15

Acquisition Time (sec)	1.8153	***Comment***	single_pulse	***Date***	13 Sep 2021 22:28:40			***File Name***	F:\NMR_OE_t_H_\tawatari\TT0905_proton-1-1.als	***Frequency (MHz)***	600.17
Nucleus	1H	***Number of Transients***	8	***Original Points Count***	16384	***Points Count***	13120	***Pulse Sequence***	proton.jxp	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	9025.27	***Temperature (degree C)***	20.600								



16

Acquisition Time (sec)	0.6921	***Comment***	single pulse decoupled gated NOE			***Date***	13 Sep 2021 22:28:22	***File Name***	F:\NMR Œ t H \tawatari\TT0905_carbon-1.als
Frequency (MHz)	150.00	***Number of Transients***	1024	***Original Points Count***	26214	***Points Count***	26214	***Pulse Sequence*** carbon_cool.jxp	***Solvent*** CHLOROFORM-D
Sweep Width (Hz)	37876.77	***Temperature (degree C)***	20.500						



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