Electronic Supplementary Information (ESI)

2-Bromo-6-(chlorodiisopropylsilyl)phenyl Tosylate as Efficient Platform for Intramolecular Benzyne–Diene [4+2] Cycloaddition

Arata Nishii, Hiroshi Takikawa, and Keisuke Suzuki*

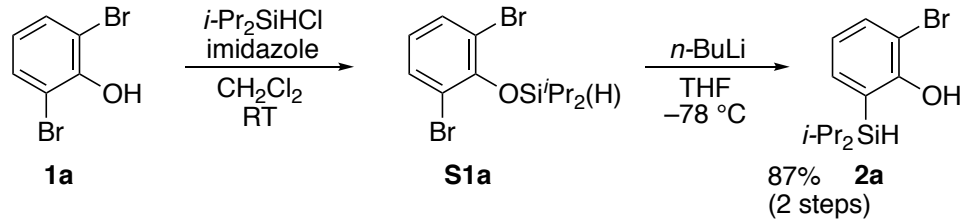
Contents of Supplementary Information

1. General Experimental Procedure

All reactions utilizing air- or moisture-sensitive reagents were performed in dried glassware under an atmosphere of dry argon. THF, Et_2O, and dichloromethane (anhydrous; Kanto Chemical Co., Inc.) were purified under argon using a solvent purification unit (Wako Pure Chemical Industries, Ltd.). THF solution of PhMgBr was prepared from bromobenzene and magnesium turning according to the standard protocols. Other reagents were used without further purification. For thin-layer chromatography (TLC) analysis, Merck pre-coated plates (silica gel 60 F254, 0.25 mm) were used. Silica-gel preparative thin-layer chromatography (PTLC) was performed using plates prepared from Merck Silica gel 60 PF254 (Art7747). For flash column chromatography, silica gel 60N (Spherical, neutral, 63–210 μm) from Kanto Chemical was used. Higher-accuracy purifications were performed by Smart Flash EPCLC W-Prep 2XY system (YAMAZEN SCIENCE, inc.). Melting point (mp) determinations were performed by using a Yanaco MP-500 instrument or a METTLER TOLEDO MP70 melting point system, and are uncorrected. ^{1}H- and ^{13}C-NMR were measured on a Bruker Avance III 600 (600 MHz) spectrometer in the solvent indicated; Chemical shifts (δ) are expressed in parts per million (ppm) downfield from internal standard (tetramethylsilane, 0.00 ppm), and coupling constants are reported as hertz (Hz). Splitting patterns are indicated as follows: br, broad; s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Infrared (IR) spectra were recorded on a Thermo Scientific Nicolet iS5 FT-IR spectrometer. Attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectra were recorded by using a Thermo Scientific Nicolet iS5 FT-IR spectrometer equipped with a universal ATR sampling accessory (iD5 ATR). Elemental analyses were recorded on an Elementar vario MICRO cube analyzer. High-resolution mass spectra (HRMS) were obtained with Bruker Daltonics micrOTOF-Q II.

2. Preparations of cycloaddition precursors

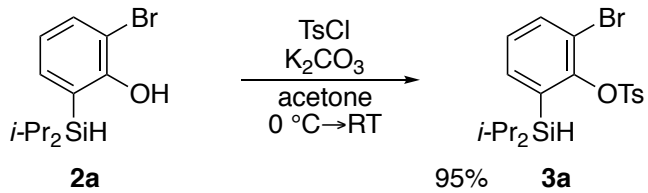
*Synthesis of phenol **2a***



To a solution of 2,6-dibromophenol (**1a**, purchased from Tokyo Chemical Industry Co., Ltd., 7.60 g, 30.2 mmol) in CH_2Cl_2 (90 mL) were added imidazole (4.07 g, 59.8 mmol) and *i*-Pr_2SiHCl (6.10 mL, 36.0 mmol). After stirring for 2.5 h at room temperature, the reaction was quenched by adding phenol (562 mg, 5.98 mmol) at 0 °C and stirred for 30 min, to which was hexane (60 mL) to form white precipitates. CH_2Cl_2 was removed by concentration in vacuo (ca. 300 hPa). The resulting suspension was filtered through a Celite@ pad (washed with hexane), and the filtrate was concentrated in vacuo to afford silyl ether **S1a**. The crude material was dissolved in THF (90 mL), to which was added dropwise *n*-BuLi (1.51 M in hexane, 30.0 mL, 45.3 mmol) 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 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 = 99/1) to afford phenol **2a** (7.55 g, 87%) as colorless oil.

2a: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.98 (d, 6H, *J* = 7.2 Hz), 1.08 (d, 6H, *J* = 7.2 Hz), 1.34 (qqd, 2H, *J* = 7.2, 7.2, 3.6 Hz), 3.93 (t, 1H, *J* = 3.6 Hz), 5.70 (s, 1H), 6.78 (dd, 1H, *J* = 7.8, 6.0 Hz), 7.35 (dd, 1H, *J* = 6.0, 1.5 Hz), 7.47 (dd, 1H, *J* = 7.8, 1.5 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 10.9, 18.89, 18.92, 110.3, 121.6, 122.0, 133.4, 137.4, 155.9; **IR** (neat) 3518, 2941, 2096, 1584, 1425, 1235, 1082 cm^{-1}; **HRMS** (APCI): Calcd. for $C_{12}H_{18}BrOSi$ $[M–H]^+$: 285.0305; Found: 285.0310.

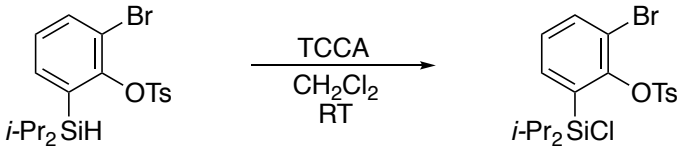
*Synthesis of tosylate **3a***



To a solution of phenol **2a** (1.44 g, 5.01 mmol) in acetone (10 mL) were added K_2CO_3 (1.73 g, 12.5 mmol) and TsCl (1.24 g, 6.50 mmol). After stirring for 2.5 h at 0 °C, the reaction was warmed to room temperature. After stirring for 1.5 h at this temperature, 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/EtOAc = 95/5) to afford aryl tosylate **3a** (2.09 g, 95%) as a white solid.

3a: **mp** 53–54 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.98 (d, 6H, *J* = 7.8 Hz), 1.09 (d, 6H, *J* = 7.2 Hz), 1.31 (qqd, 2H, *J* = 7.8, 7.2, 3.6 Hz), 2.47 (s, 3H), 4.15 (t, 1H, *J* = 3.6 Hz), 7.13 (dd, 1H, *J* = 7.8, 7.2 Hz), 7.34 (d, 2H, *J* = 8.4 Hz), 7.42 (dd, 1H, *J* = 7.2, 1.5 Hz), 7.55 (dd, 1H, *J* = 7.8, 1.5 Hz), 7.85 (d, 2H, *J* = 8.4 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 11.2, 18.7, 18.9, 21.7, 117.5, 127.6, 128.8, 129.6, 134.0, 134.6, 135.2, 135.4, 145.2, 151.2; **IR** (ATR) 2938, 2100, 1596, 1399, 1196, 1077 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{19}H_{25}BrNaO_3SSi$ $[M+Na]^+$: 463.0369; Found: 463.0371.

*Synthesis of silyl chloride **4a***



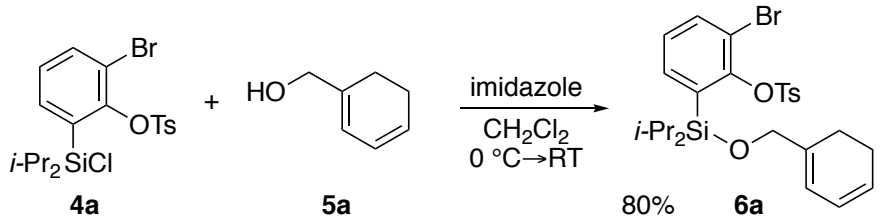
3a quant. **4a**

To a solution of hydrosilane **3a** (2.20 g, 4.98 mmol) in CH_2Cl_2 (26 mL) was added trichloroisocyanuric acid (TCCA, 464 mg, 2.00 mmol).[1] After stirring for 2 h at room temperature, the reaction mixture was filtered through a Celite@ pad (washed with CH_2Cl_2), and the filtrate was concentrated in vacuo to afford silyl chloride **4a** (2.38 g, quant.) as a white solid.

4a: **mp** 122–123 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.00 (d, 6H, *J* = 7.8 Hz), 1.22 (d, 6H, *J* = 7.2 Hz), 1.93 (qq, 2H, *J* = 7.8, 7.2 Hz), 2.48 (s, 3H), 7.19 (dd, 1H, *J* = 7.8, 7.2 Hz), 7.36 (d, 2H, *J* = 7.8 Hz), 7.59 (dd, 1H, *J* = 7.8, 1.8 Hz), 7.84 (dd, 1H, *J* = 7.2, 1.8 Hz), 7.86 (d, 2H, *J* = 7.8 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 15.0, 18.0, 18.1, 21.8, 117.3, 127.8, 128.8, 129.6, 132.4, 134.0, 136.1, 137.8, 145.6, 149.8; **IR** (ATR) 2948, 1596, 1398, 1197 cm^{-1}; **HRMS** (APCI): Calcd. for $C_{19}H_{25}BrClO_3SSi$ $[M+H]^+$: 475.0160; Found: 475.0140.

[1] Note that the both TCCA and its byproduct were almost insoluble to CH_2Cl_2 at room temperature, and those could be removed by filtration. For the detail, see ref [9b] in manuscript.

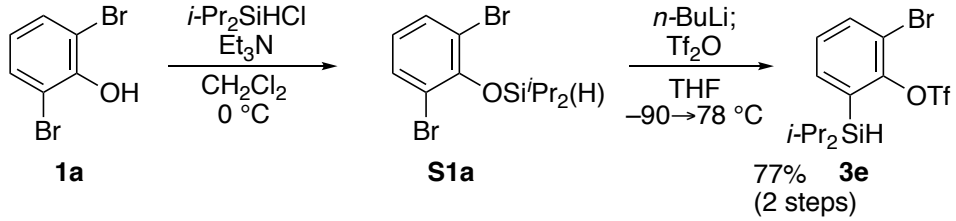
*Synthesis of silyl ether **6a***



To a solution of alcohol **5a** (prepared according to the reported procedure, [2] 39.9 mg, 0.362 mmol) in CH_2Cl_2 (1.9 mL) were added imidazole (50.0 mg, 0.734 mmol) and silyl chloride **4a** (176 mg, 0.370 mmol) at 0 °C. After stirring for 20 min at this temperature, the reaction mixture was warmed to room temperature, and stirred for 3 h at this temperature. The reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 95/5) to afford silyl ether **6a** (159 mg, 80%) as a white solid.

6a: **mp** 85–86 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.16 (d, 6H, *J* = 7.2 Hz), 1.19 (d, 6H, *J* = 7.8 Hz), 1.62 (qq, 2H, *J* = 7.8, 7.2 Hz), 2.10 (brt, 2H, *J* = 9.3 Hz), 2.18–2.25 (m, 2H), 2.47 (s, 3H), 4.22 (s, 2H), 5.72–5.78 (m, 1H), 5.94–6.00 (m, 2H), 7.15 (dd, 1H, *J* = 7.8, 7.5 Hz), 7.34 (d, 2H, *J* = 8.4 Hz), 7.56 (dd, 1H, *J* = 7.8, 1.7 Hz), 7.61 (dd, 1H, *J* = 7.5, 1.7 Hz), 7.85 (d, 2H, *J* = 8.4 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 13.9, 18.0, 18.6, 21.8, 22.7, 23.3, 66.7, 117.6, 117.7, 124.3, 124.9, 127.7, 128.6, 129.5, 134.1, 134.5, 135.4, 136.5, 138.0, 145.2, 150.3; **IR** (ATR) 2941, 1397, 1362, 1169, 858 cm^{-1}; **HRMS** (APCI): Calcd. for $C_{26}H_{33}BrNaO_4SSi$ $[M+Na]^+$: 571.0944; Found: 571.0947.

*Synthesis of triflate **3e***

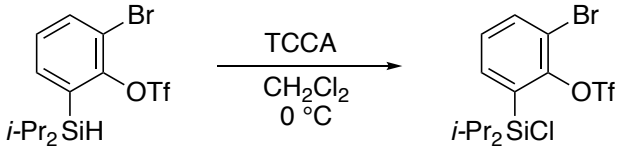


[2] a) F. Bohlmann and C. Zdero, *Chem. Ber.,* 1973, **106**, 3779–3787; b) K. E. Harding, J. B. Strickland and J. Pommerville, *J. Org. Chem.,* 1988, **53**, 4877–4883; c) H. Gradén, J. Hallberg, N. Kann and T. Olsson, *J. Comb. Chem.* 2004, **6**, 783–788.

To a solution of 2,6-dibromophenol (**1a**, 1.27 g, 5.05 mmol) in CH_2Cl_2 (15 mL) were added triethylamine (1.40 mL, 10.1 mmol) and *i*-Pr_2SiHCl (0.930 mL, 5.49 mmol). After stirring for 1 h at 0 °C, the reaction mixture was concentrated in vacuo. The resulting suspension was filtered through a Celite@ pad (washed with hexane), and the filtrate was concentrated in vacuo to afford silyl ether **S1a** The crude material was dissolved in THF (15 mL), to which was added dropwise *n*-BuLi (1.55 M in hexane, 3.90 mL, 6.05 mmol) at –90 °C, and the stirring was continued for 25 min at this temperature. Tf_2O (0.90 mL, 5.5 mmol) was added to the reaction mixture, which was then warmed to –78 °C and was stirred for 20 min. 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 **3e** (1.61 g, 77%) as colorless oil.

3e: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.97 (d, 6H, *J* = 7.8 Hz), 1.10 (d, 6H, *J* = 7.2 Hz), 1.29 (qqd, 2H, *J* = 7.8, 7.2, 3.6 Hz), 4.28 (t, 1H, *J* = 3.6 Hz), 7.24 (dd, 1H, *J* = 7.8, 7.2 Hz), 7.44 (dd, 1H, *J* = 7.2, 1.8 Hz), 7.70 (dd, 1H, *J* = 7.8, 1.8 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 11.0, 18.5, 18.6, 116.4, 118.6 (q, J_{CF} = 319 Hz), 128.8, 133.0, 135.5, 136.0, 150.5; **IR** (neat) 2946, 2165, 1425, 1208 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{13}H_{17}BrF_3O_3SSi$ $[M–H]^+$: 416.9798; Found: 416.9793.

*Synthesis of silyl chloride **4e***



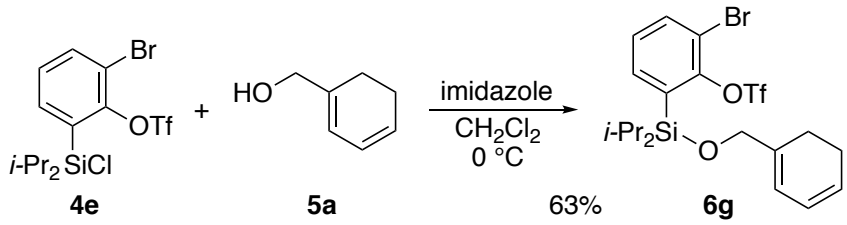
3e quant. **4e**

To a solution of hydrosilane **3e** (588 mg, 1.40 mmol) in CH_2Cl_2 (7.0 mL) was added trichloroisocyanuric acid (TCCA, 130 mg, 0.559 mmol). After stirring for 2 h at 0 °C, the reaction mixture was concentrated in vacuo. The resulting suspension was filtered through a Celite@ pad (washed with hexane), and the filtrate was concentrated in vacuo to afford silyl chloride **4e** (640 mg, quant.) as colorless oil.

4e: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.95 (d, 6H, *J* = 7.2 Hz), 1.19 (d, 6H, *J* = 7.2 Hz), 1.72 (qq, 2H, *J* = 7.2, 7.2 Hz), 7.31 (dd, 1H, *J* = 7.8, 7.8 Hz), 7.76 (dd, 1H, *J* = 7.8, 1.8 Hz), 7.85 (dd, 1H, *J* = 7.8, 1.8 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 15.0, 17.8, 17.9, 116.6, 118.6 (q, J_{CF} = 320

Hz), 129.2, 131.7, 137.1, 138.0, 147.7; **IR** (neat) 2954, 1412, 1216, 1133 cm^{-1}; **HRMS** (APCI): Calcd. for $C_{13}H_{17}BrF_3O_3SSi$ $[M–Cl]^+$: 416.9798; Found: 416.9804.

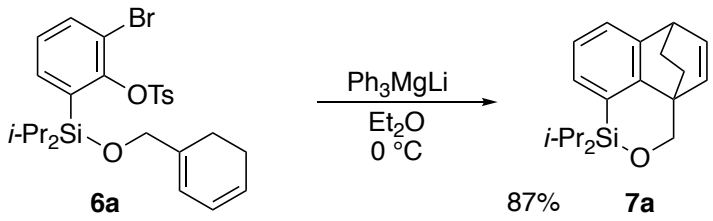
Synthesis of silyl ether ***6g***



To a solution of alcohol **5a** (158 mg, 1.43 mmol) in CH_2Cl_2 (7.0 mL) were added imidazole (194 mg, 2.85 mmol) and silyl chloride **4e** (640 mg, 1.41 mmol). After stirring for 11 h at 0 °C, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 98/2) to afford silyl ether **6g** (477 mg, 63%) as colorless oil.

6g: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.02 (d, 6H, J = 7.2 Hz), 1.14 (d, 6H, J = 7.8 Hz), 1.46 (qq, 2H, J = 7.8, 7.2 Hz), 2.13 (brt, 2H, J = 9.5 Hz), 2.19–2.24 (m, 2H), 4.26 (s, 2H), 5.72–5.78 (m, 1H), 5.92–5.97 (m, 2H), 7.25 (dd, 1H, J = 7.7, 7.2 Hz), 7.57 (dd, 1H, J = 7.2, 1.7 Hz), 7.71 (dd, 1H, J = 7.7, 1.7 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 13.6, 17.7, 18.2, 22.6, 23.3, 66.9, 116.5, 118.3, 118.7 (q, J_{CF} = 320 Hz), 124.2, 125.2, 128.9, 133.6, 136.2, 136.3, 137.5, 149.1; **IR** (neat) 2947, 1410, 1214, 1135 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{20}H_{26}BrF_3NaO_4SSi$ $[M+Na]^+$: 549.0349; Found: 549.0370.

3. Synthesis of cycloadduct 7a: Typical procedure

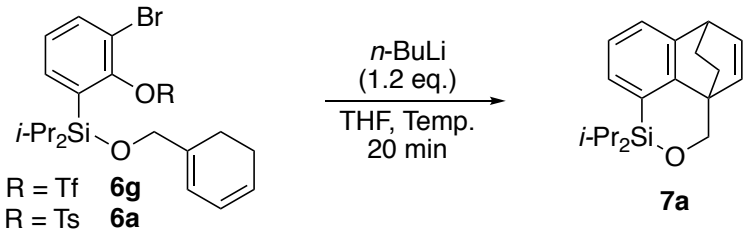


To a solution of PhLi (0.67 M in cyclohexane and Et_2O, 1.1 mL, 0.74 mmol) in Et_2O (1.5 mL) was added PhMgBr (0.94 M in THF, 0.41 mL, 0.39 mmol) at 0 °C, and the mixture was stirred for 30 min. The resulting solution of Ph_3MgLi was used in the following experiment.

To a solution of bromoaryl tosylate **6a** (165 mg, 0.300 mmol) in Et_2O (6.0 mL) was added dropwise Ph_3MgLi (*vide supra*) via cannula at 0 °C. After stirring for 10 min, the reaction was stopped 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 PTLC (hexane/EtOAc = 98/2 x2) to afford cycloadduct **7a** (77.8 mg, 87%) as colorless oil.

7a: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.96 (d, 3H, *J* = 7.2 Hz), 1.00 (d, 3H, *J* = 7.2 Hz), 1.11 (d, 3H, *J* = 7.8 Hz), 1.13 (d, 3H, *J* = 7.2 Hz), 1.17 (qq, 1H, *J* = 7.2, 7.2 Hz), 1.30 (qq, 1H, *J* = 7.8, 7.2 Hz), 1.37 (ddd, 1H, *J* = 10.2, 10.2, 3.6 Hz), 1.45–1.50 (m, 1H), 1.63–1.73 (m, 2H), 3.90–3.92 (m, 1H), 4.30 (d, 1H, *J* = 11.7 Hz), 4.60 (d, 1H, *J* = 11.7 Hz), 6.06 (dd, 1H, *J* = 7.6, 0.6 Hz), 6.57 (dd, 1H, *J* = 7.6, 6.3 Hz), 7.07 (dd, 1H, *J* = 7.7, 7.7 Hz), 7.19 (dd, 1H, *J* = 7.7, 1.2 Hz), 7.20 (dd, 1H, *J* = 7.7, 1.2 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 12.7, 13.3, 17.3, 17.4, 17.5, 17.9, 27.5, 30.1, 41.0, 45.1, 68.3, 123.5, 124.1, 126.4, 129.7, 135.6, 135.9, 143.9, 150.9; **IR** (neat) 2943, 1463, 1139, 1057 cm^{-1}; **HRMS** (APCI) Calcd. for $C_{19}H_{27}OSi$ $[M+H]^+$: 299.1826; Found: 299.1832.

Supplementation: Optimization of leaving group and temperature.

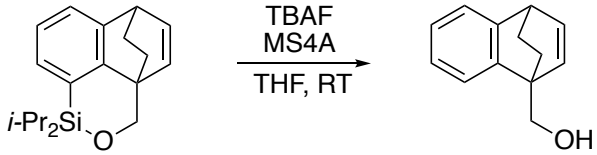


Entry	**6**	Temp. [°C]	Yield [%][a]
1	**6g**	–78	21
2	**6a**	–78	29
3	**6g**	0	48
4	**6a**	0	53

[a] Isolated yield of **7a**

4. Transformations of 7a

*Synthesis of alcohol **9***

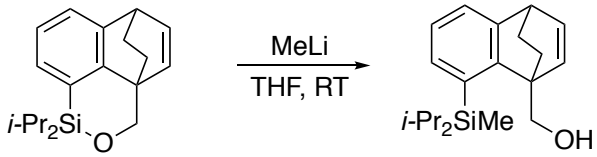


7a 92% **9**

To a solution of silyl ether **7a** (189 mg, 0.633 mmol) and MS4A (636 mg) in THF (3.2 mL) was added tetrabutylammonium fluoride (TBAF, 1.0 M in THF, 1.40 mL, 1.4 mmol). After stirring for 30 min at room temperature, 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, toluene/Et_2O = 9/1→4/1) and PTLC (toluene/Et_2O = 4/1) to afford alcohol **9** (109 mg, 92%) as a white solid.

9: **mp** 100–101 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.29 (ddd, 1H, *J* = 11.0, 10.2, 4.2 Hz), 1.49 (ddd, 1H, *J* = 11.0, 10.5, 4.0 Hz), 1.52–1.58 (m, 1H), 1.64–1.71 (m, 2H), 3.93–3.95 (m, 1H), 4.28 (d, 1H, *J* = 10.8 Hz), 4.43 (d, 1H, *J* = 10.8 Hz), 6.42 (d, 1H, *J* = 7.5 Hz), 6.61 (dd, 1H, *J* = 7.5, 6.6 Hz), 7.09 (ddd, 1H, *J* = 7.4, 7.2, 0.8 Hz), 7.13 (ddd, 1H, *J* = 7.4, 7.2, 1.2 Hz), 7.19 (brd, 1H, *J* = 7.2 Hz), 7.25 (brd, 1H, *J* = 7.2 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 27.2, 28.4, 40.8, 47.5, 65.2, 120.1, 122.8, 125.01, 125.02, 135.3, 136.2, 143.8, 145.1; **IR** (ATR) 3218, 2951, 1475, 1043 cm^{-1}; **Anal.** Calcd. for $C_{13}H_{14}O$: C, 83.83; H, 7.58; Found: C, 83.56; H, 7.52.

*Synthesis of alcohol **10***



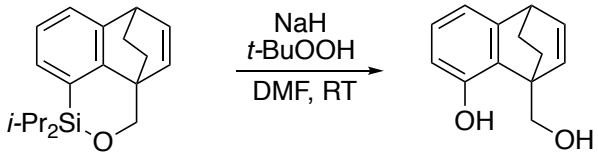
7a 89% **10**

To a solution of silyl ether **7a** (21.2 mg, 0.0710 mmol) in THF (0.36 mL) was added methyllithium (1.12 M in Et_2O, 125 μL, 0.140 mmol). After stirring for 15 min 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 PTLC (hexane/EtOAc = 9/1) to afford alcohol **10** (19.9 mg, 89%) as a white solid.

10: **mp** 77–79 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.33 (s, 3H), 0.80–0.85 (m, 6H), 1.07 (d, 3H, *J* = 7.2 Hz), 1.10 (d, 3H, *J* = 7.2 Hz), 1.23 (ddd, 1H, *J* = 11.5, 10.1, 4.7 Hz), 1.35–1.43 (m, 2H), 1.55–1.61 (m, 1H), 1.62–1.70 (m, 2H), 1.89 (ddd, 1H, *J* = 11.5, 10.8, 4.2 Hz), 3.88–3.91 (m, 1H), 4.43 (d, 1H, *J* = 9.6 Hz), 4.65 (d, 1H, *J* = 9.6 Hz), 6.48 (dd, 1H, *J* = 7.8, 0.6 Hz), 6.63 (dd, 1H, *J* = 7.8, 6.6 Hz), 7.02 (dd, 1H, *J* = 7.3, 7.3 Hz), 7.18 (dd, 1H, *J* = 7.3, 1.2 Hz), 7.36 (dd, 1H, *J* = 7.3, 1.2 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ –6.6, 14.9, 15.1, 19.0, 19.2, 19.3, 19.4, 26.6, 28.4, 42.6, 50.1, 66.2, 123.7, 124.5, 129.2, 134.1, 136.3, 136.5, 145.9, 150.0; **IR** (ATR) 3279, 2954, 1462, 1254 cm^{-1}; **HRMS** (ESI) Calcd. for $C_{20}H_{30}NaOSi$ $[M+Na]^+$: 337.1958; Found: 337.1955.

*Synthesis of phenol **11***



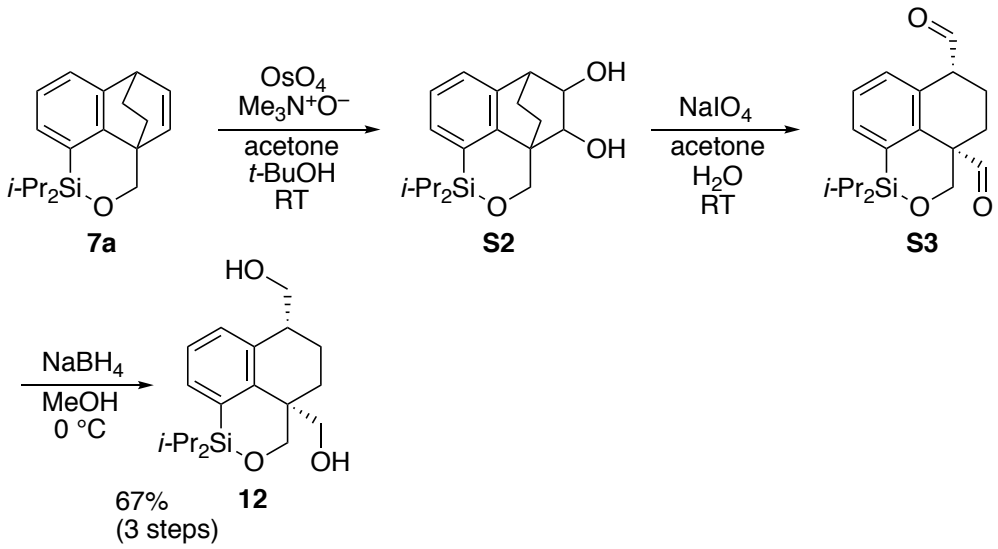
7a 79% **11**

To a solution of silyl ether **7a** (26.0 mg, 0.0871 mmol) in DMF (0.44 mL) were added *t*-BuOOH (ca. 5.0 M in decane, 175 μL, ca. 0.88 mmol) and NaH (63% dispersion in mineral oil, 8.9 mg, 0.23 mmol). After stirring for 23 h at room temperature, additional portion of *t*-BuOOH (ca. 5.0 M in decane, 174 μL, ca. 0.87 mmol) was added to the reaction mixture, which was stirred for 4 h at room temperature. Additional portion of NaH (63% dispersion in mineral oil, 7.5 mg, 0.20 mmol) was added, and the stirring was continued for 6 h at this temperature. The reaction was quenched by adding 10% aqueous $Na_2S_2O_3$ and 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 PTLC (hexane/EtOAc = 4/1) to afford phenol **11** (14.0 mg, 79%) as a white solid.

11: **mp** 157–161 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.26 (ddd, 1H, *J* = 10.8, 10.2, 3.4 Hz), 1.52–1.63 (m, 2H), 1.66 (dddd, 1H, *J* = 10.5, 10.2, 3.2, 2.6 Hz), 3.65 (brs, 1H), 3.88–3.91 (m, 1H), 4.29 (d, 1H, *J* = 11.1 Hz), 4.38 (d, 1H, *J* = 11.1 Hz), 6.25 (d, 1H, *J* = 7.2 Hz), 6.59 (dd, 1H, *J* = 7.2, 6.6 Hz), 6.68 (d, 1H, *J* = 7.9 Hz), 6.78 (d, 1H, *J* = 7.1 Hz), 6.97 (dd, 1H, *J* = 7.9, 7.1 Hz), 8.42 (brs, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 26.8, 29.1, 41.4, 47.5, 66.6, 115.4, 115.8,

126.4, 128.6, 135.8, 136.2, 147.8, 150.0; **IR** (ATR) 3369, 3048, 2941, 1585, 1456, 1300 cm^{-1}; **Anal.** Calcd. for $C_{13}H_{14}O_2$: C, 77.20; H, 6.98; Found: C, 77.39; H, 6.91.

*Synthesis of diol **12***



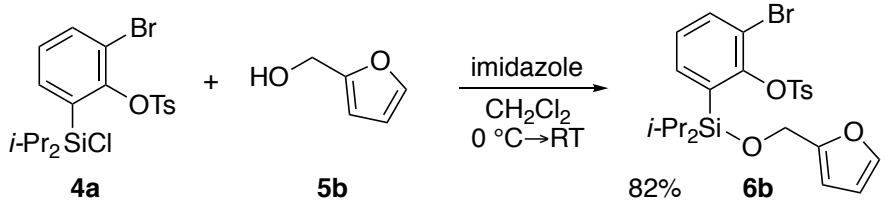
To a solution of silyl ether **7a** (28.6 mg, 0.0958 mmol) in acetone (0.48 mL) were added trimethylamine *N*-oxide (43.5 mg, 0.579 mmol) and OsO_4 (0.03 M in *t*-BuOH, 0.32 mL, 0.01 mmol). After stirring for 5 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHSO_3$, and was stirred for 11 h. The reaction mixture was extracted with EtOAc (x4). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo to afford diol **S2** (d.r. = 2:1). The crude material was dissolved in acetone (0.96 mL) and H_2O (0.96 mL), to which was added $NaIO_4$ (41.6 mg, 0.192 mmol). After stirring for 30 min at room temperature, the reaction was diluted with water, and the mixture was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo to afford aldehyde **S3**. The crude material was dissolved in MeOH (0.96 mL), to which was added $NaBH_4$ (12.3 mg, 0.325 mmol). After stirring for 15 min at 0 °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 PTLC (hexane/acetone = 3/2) to afford diol **12** (21.4 mg, 67%) as a white solid.

12: **mp** 137–138 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.99 (d, 3H, *J* = 7.2 Hz), 1.08–1.15 (m, 9H), 1.18–1.27 (m, 3H), 1.87 (ddd, 1H, *J* = 13.2, 3.6, 3.6 Hz), 1.92–2.16 (m, 3H), 3.09 (dddd, 1H, *J* = 9.9, 8.2, 4.2, 4.2 Hz), 3.69 (d, 1H, *J* = 11.7 Hz), 3.72 (d, 1H, *J* = 11.1 Hz), 3.89 (d, 1H, *J* =

11.7 Hz), 3.91 (dd, 1H, *J* = 10.5, 4.2 Hz), 4.00 (d, 1H, *J* = 11.1 Hz), 4.01 (dd, 1H, *J* = 10.5, 4.2 Hz), 7.23 (dd, 1H, *J* = 7.2, 6.6 Hz), 7.26 (d, 1H, *J* = 6.6 Hz), 7.38 (d, 1H, *J* = 7.2 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 13.5, 14.3, 17.4, 17.6, 17.9, 18.3, 21.3, 25.8, 40.5, 42.2, 64.4, 67.7, 69.2, 126.1, 129.3, 131.6, 131.9, 136.2, 148.0; **IR** (ATR) 3334, 2940, 1463, 1024 cm^{-1}; **Anal.** Calcd. for $C_{19}H_{30}OSi$: C, 68.22; H, 9.04; Found: C, 68.31; H, 9.07.

5. Substrate scope

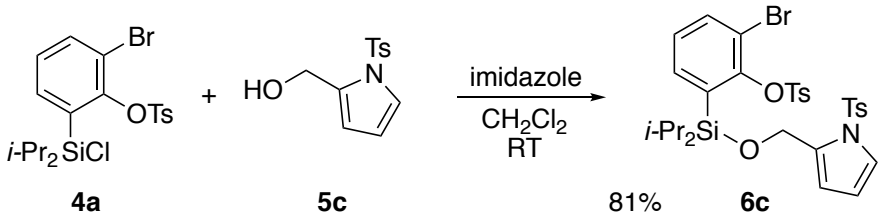
*Synthesis of silyl ether **6b***



To a solution of alcohol **5b** (purchased from Tokyo Chemical Industry Co., Ltd., 65 μL, 0.75 mmol) in CH_2Cl_2 (2.5 mL) were added imidazole (84.9 mg, 1.25 mmol) and silyl chloride **4a** (238 mg, 0.500 mmol). After stirring for 1 h at 0 °C, the reaction mixture was warmed to room temperature and stirred for 4 h at this temperature. The reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 93/7) to afford silyl ether **6b** (221 mg, 82%) as colorless oil.

6b: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.07 (d, 6H, *J* = 7.5 Hz), 1.17 (d, 6H, *J* = 7.5 Hz), 1.63 (qq, 2H, *J* = 7.5, 7.5 Hz), 2.47 (s, 3H), 4.75 (s, 2H), 6.28 (dd, 1H, *J* = 3.3, 0.5 Hz), 6.34 (dd, 1H, *J* = 3.3, 2.0 Hz), 7.15 (dd, 1H, *J* = 7.8, 7.2 Hz), 7.33 (d, 2H, *J* = 8.1 Hz), 7.39 (dd, 1H, *J* = 2.0, 0.5 Hz), 7.56 (dd, 1H, *J* = 7.8, 1.8 Hz), 7.64 (dd, 1H, *J* = 7.2, 1.8 Hz), 7.85 (d, 2H, *J* = 8.1 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 13.7, 17.9, 18.3, 21.8, 59.0, 107.2, 110.2, 117.5, 127.7, 128.6, 129.5, 133.9, 134.6, 135.5, 136.5, 142.0, 145.2, 150.4, 154.2; **IR** (neat) 2945, 1396, 1198, 1075 cm^{-1}; **HRMS** (APCI): Calcd. for $C_{24}H_{30}BrO_5SSi$ $[M+H]^+$: 537.0761; Found: 537.0741.

Synthesis of silyl ether ***6c***

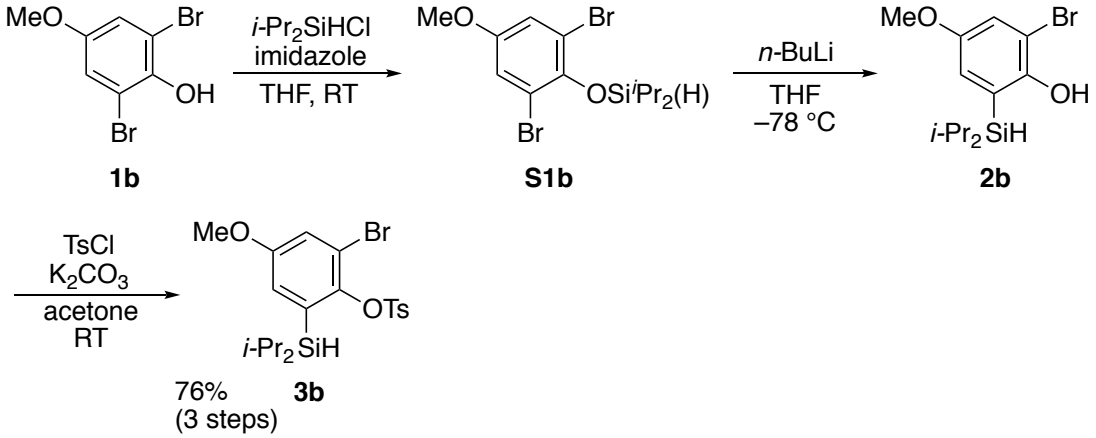


To a solution of alcohol **5c** (prepared according to the reported procedure,[3] 154 mg, 0.613 mmol) in CH_2Cl_2 (2.5 mL) were added imidazole (86.1 mg, 1.26 mmol) and silyl chloride **4a** (240 mg, 0.504 mmol). After stirring for 1.5 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 5/1) to afford silyl ether **6c** (282 mg, 81%) as a white solid.

6c: **mp** 137 °C (decomp.); **^{1}H NMR** (600 MHz, acetone-d_6) δ 1.03 (d, 6H, J = 7.5 Hz), 1.15 (d, 6H, J = 7.5 Hz), 1.65 (qq, 2H, J = 7.5, 7.5 Hz), 2.40 (s, 3H), 2.49 (s, 3H), 4.93 (s, 2H), 6.35 (dd, 1H, J = 3.3, 3.3 Hz), 6.44 (brs, 1H), 7.21 (dd, 1H, J = 7.8, 7.5 Hz), 7.33–7.39 (m, 3H), 7.50 (d, 2H, J = 8.4 Hz), 7.59 (dd, 1H, J = 7.5, 1.5 Hz), 7.68–7.73 (m, 3H), 7.86 (d, 2H, J = 8.4 Hz); **^{13}C NMR** (150 MHz, acetone-d_6) δ 14.4, 18.2, 18.8, 21.5, 21.7, 60.3, 112.7, 113.7, 118.4, 123.6, 127.6, 129.0, 129.4, 130.7, 131.1, 134.0, 135.1, 136.7, 137.0, 137.3, 146.3, 146.8, 151.1 (several signals overlapped); **IR** (ATR) 2948, 1595, 1361, 1169 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{31}H_{36}BrNNaO_6S_2Si$ $[M+Na]^+$: 714.0814; Found: 714.0803.

[3] H. Prinzbach, H. Bingmann, H. Fritz, J. Markert, L. Knothe, W. Eberbach, J. Brokatzky-Geiger, J. C. Sekutowskib and C. Krüger, *Chem. Ber.,* 1986, **119**, 616–644.

*Synthesis of tosylate **3b***



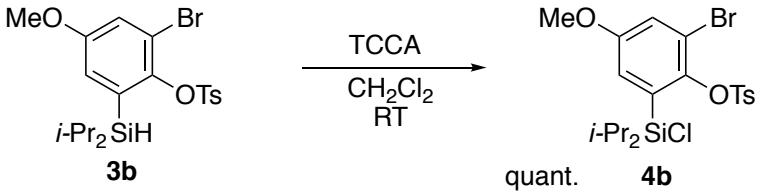
To a solution of dibromophenol **1b** (prepared according to the reported procedure,[4] 282 mg, 1.00 mmol) in THF (5.0 mL) were added imidazole (103 mg, 1.51 mmol) and *i*-Pr_2SiHCl (254 μL, 1.50 mmol). After stirring for 2 h at room temperature, hexane (10 mL) was added to the mixture to form white precipitates. The resulting suspension was filtered through a Celite@ pad (washed with Et_2O), and the filtrate was concentrated in vacuo to afford silyl ether **S1b**. The crude material was dissolved in THF (3.0 mL), to which was added dropwise *n*-BuLi (1.55 M in hexane, 0.90 mL, 1.4 mmol) at –78 °C. After stirring for 20 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 to afford phenol **2b**. The crude material was dissolved in acetone (5.0 mL), to which were added K_2CO_3 (208 mg, 1.50 mmol) and TsCl (247 mg, 1.30 mmol). After stirring for 18 h at room temperature, 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/EtOAc = 95/5) to afford aryl tosylate **3b** (358 mg, 76%) as colorless oil.

3b: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.99 (d, 6H, *J* = 7.2 Hz), 1.09 (d, 6H, *J* = 7.2 Hz), 1.30 (qqd, 2H, *J* = 7.2, 7.2, 3.8 Hz), 2.46 (s, 3H), 3.78 (s, 3H), 4.11 (t, 1H, *J* = 3.8 Hz), 6.92 (d, 1H, *J* = 3.0 Hz), 7.05 (d, 1H, *J* = 3.0 Hz), 7.34 (d, 2H, *J* = 8.1 Hz), 7.84 (d, 2H, *J* = 8.1 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 11.2, 18.7, 19.0, 21.7, 55.8, 117.7, 119.3, 121.1, 128.8, 129.6, 134.3, 134.6,

[4] S. Kajigaeshi, T. Kakinami, H. Tokiyama, T. Hirakawa and T. Okamoto, *Chem. Lett.,* 1987, 627–630.

144.8, 145.1, 157.4; **IR** (neat) 2941, 2162, 1580, 1378, 1167 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{20}H_{27}BrNaO_4SSi$ $[M+Na]^+$: 493.0475; Found: 493.0464.

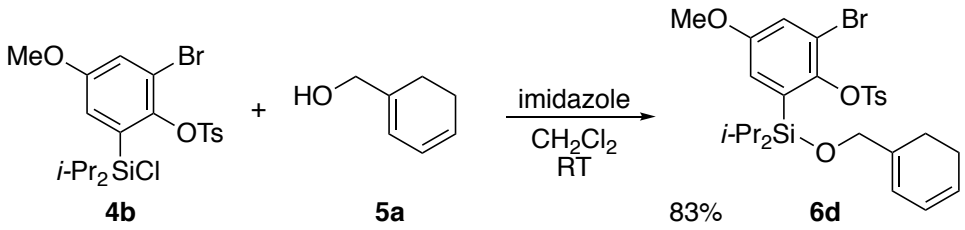
*Synthesis of silyl chloride **4b***



To a solution of hydrosilane **3b** (299 mg, 0.634 mmol) in CH_2Cl_2 (3.1 mL) was added trichloroisocyanuric acid (TCCA, 59.3 mg, 0.255 mmol). After stirring for 3 h at room temperature, the reaction mixture was filtered through a Celite@ pad (washed with CH_2Cl_2), and the filtrate was concentrated in vacuo to afford silyl chloride **4b** (329 mg, quant.) as a pale yellow solid.

4b: **mp** 91–92 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.00 (d, 6H, *J* = 7.2 Hz), 1.21 (d, 6H, *J* = 7.2 Hz), 1.92 (qq, 2H, *J* = 7.2, 7.2 Hz), 2.48 (s, 3H), 3.81 (s, 3H), 7.09 (d, 1H, *J* = 3.0 Hz), 7.33–7.37 (m, 3H), 7.85 (d, 2H, *J* = 8.4 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 15.0, 18.0, 18.1, 21.8, 55.8, 117.6, 120.8, 122.9, 128.7, 129.6, 132.6, 134.0, 143.3, 145.5, 157.6; **IR** (ATR) 2951, 1557, 1370, 1162 cm^{-1}; **HRMS** (APCI): Calcd. for $C_{20}H_{27}BrClO_4SSi$ $[M+H]^+$: 505.0266; Found: 505.0263.

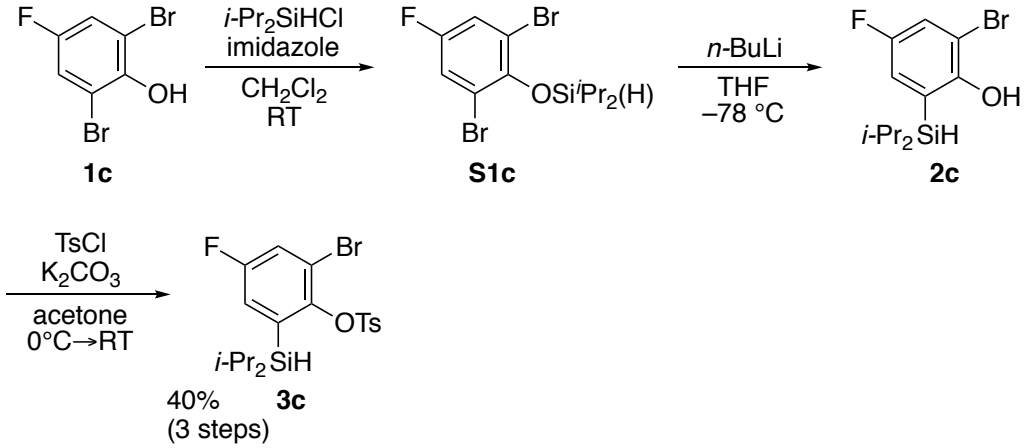
*Synthesis of silyl ether **6d***



To a solution of alcohol **5a** (74.8 mg, 0.679 mmol) in CH_2Cl_2 (3.8 mL) were added imidazole (91.8 mg, 1.35 mmol) and silyl chloride **4b** (378 mg, 0.747 mmol). After stirring for 2.5 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 93/7) to afford silyl ether **6d** (327 mg, 83%) as a white solid.

6d: **mp** 80 °C (decomp.); **^{1}H NMR** (600 MHz, acetone-d_6) δ 1.07 (d, 6H, J = 7.2 Hz), 1.21 (d, 6H, J = 7.2 Hz), 1.65 (qq, 2H, J = 7.2, 7.2 Hz), 2.13 (brtd, 2H, J = 9.6, 1.2 Hz), 2.17–2.23 (m, 2H), 2.49 (s, 3H), 3.82 (s, 3H), 4.30 (s, 2H), 5.74 (dt, 1H, J = 9.4, 4.4 Hz), 5.94–5.99 (m, 1H), 6.04 (brd, 1H, J = 4.2 Hz), 7.19 (d, 1H, J = 3.3 Hz), 7.21 (d, 1H, J = 3.3 Hz), 7.51 (d, 2H, J = 8.1 Hz), 7.88 (d, 2H, J = 8.1 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 13.9, 18.0, 18.6, 21.8, 22.6, 23.3, 55.8, 66.6, 117.67, 117.71, 120.6, 120.8, 124.3, 124.9, 128.6, 129.5, 134.3, 134.5, 138.0, 143.8, 145.1, 157.7; **IR** (ATR) 2950, 1555, 1364, 1198 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{27}H_{35}BrKO_5SSi$ $[M+K]^+$: 617.0789; Found: 617.0769.

*Synthesis of tosylate **3c***



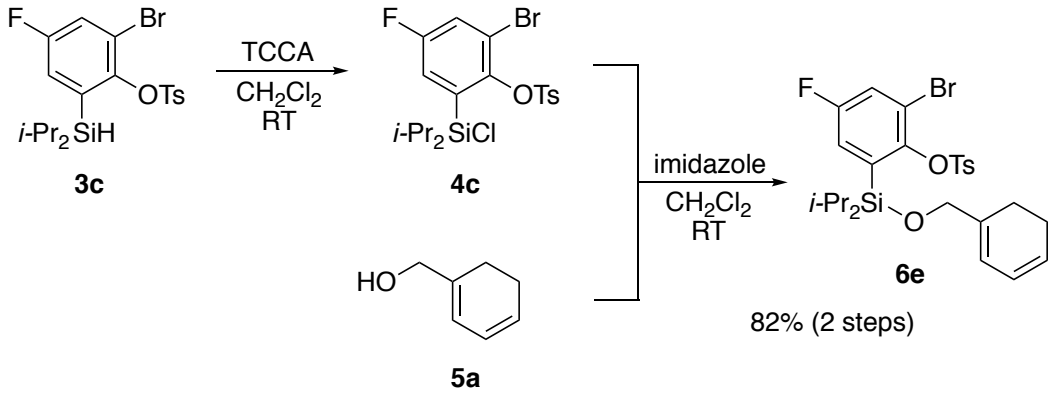
To a solution of dibromophenol **1c** (prepared according to the reported procedure,[5] 270 mg, 1.00 mmol) in CH_2Cl_2 (3.0 mL) were added imidazole (138 mg, 2.03 mmol) and *i*-Pr_2SiHCl (254 μL, 1.50 mmol). After stirring for 4 h at room temperature, hexane (6 mL) was added to the mixture to form white precipitates. CH_2Cl_2 was removed by concentration in vacuo (ca. 300 hPa). The resulting suspension was filtered through a Celite$^@$ pad (washed with hexane), and the filtrate was concentrated in vacuo to afford silyl ether **S1c**. The crude material was dissolved in THF (3.0 mL), to which was added dropwise *n*-BuLi (1.55 M in hexane, 0.90 mL, 1.4 mmol) at –78 °C. After stirring for 15 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 to afford phenol **2c**. The crude material was dissolved in acetone (2.0 mL), to which were added K_2CO_3 (345 mg, 2.50 mmol) and TsCl (247 mg, 1.30 mmol). After stirring for 1 h at 0 °C, the reaction

[5] R. E. Mewshaw, M. B. Webb, K. L. Marquis, G. B. McGaughey, X. Shi, T. Wasik, R. Scerni, J. A. Brennan and T. H. Andree, *J. Med. Chem.*, 1999, **42**, 2007–2020.

was warmed to room temperature. After stirring for 2.5 h at this temperature, 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/Et_2O = 95/5) and gel-permeation chromatography [YMC-GPC T4000® (2.0 cm φ×60 cm)+T2000® (2.0 cm φ×60 cm), EtOAc, flow rate 7.0 mL/min] to afford tosylate **3c** (184 mg, 40%) as colorless oil.

3c: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.99 (d, 6H, J = 7.2 Hz), 1.10 (d, 6H, J = 7.2 Hz), 1.31 (qqd, 2H, J = 7.2, 7.2, 3.6 Hz), 2.47 (s, 3H), 4.14 (t, 1H, J = 3.6 Hz), 7.12 (dd, 1H, J = 2.9 Hz, J_{HF} = 7.4 Hz), 7.27 (dd, 1H, J = 2.9 Hz, J_{HF} = 7.4 Hz), 7.35 (d, 2H, J = 8.1 Hz), 7.84 (d, 2H, J = 8.1 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 11.1, 18.6, 18.9, 21.8, 117.9 (d, J_{CF} = 7.5 Hz), 121.4 (d, J_{CF} = 21.0 Hz), 122.0 (d, J_{CF} = 25.5 Hz), 128.8, 129.7, 134.3, 135.9 (d, J_{CF} = 4.5 Hz), 145.4, 147.4 (d, J_{CF} = 3.0 Hz), 159.6 (d, J_{CF} = 253.5 Hz); **IR** (ATR) 2943, 2168, 1416, 1382, 1196 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{19}H_{23}BrFO_3SSi$ $[M–H]^+$: 457.0299; Found: 457.0302.

*Synthesis of silyl ether **6e***



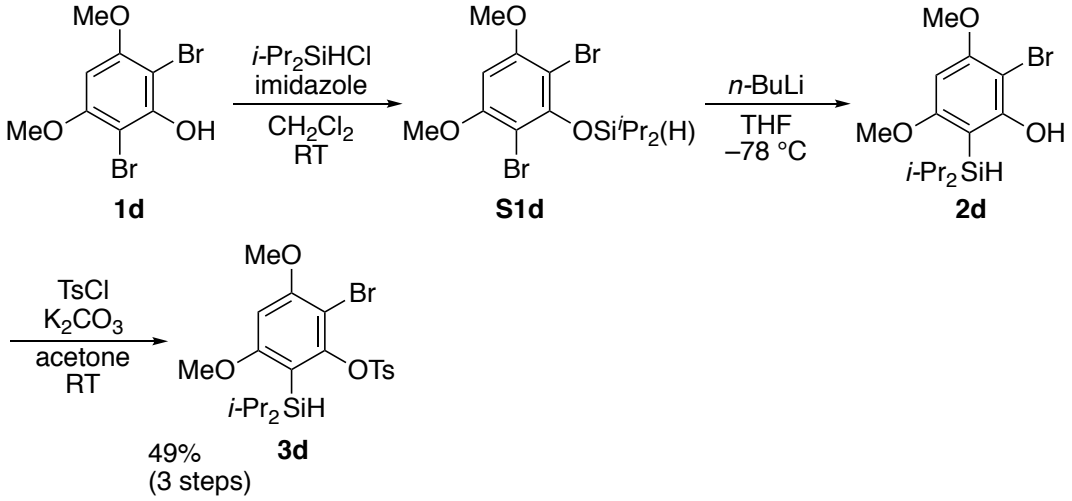
To a solution of hydrosilane **3c** (167 mg, 0.363 mmol) in CH_2Cl_2 (1.8 mL) was added trichloroisocyanuric acid (TCCA, 34.3 mg, 0.148 mmol). After stirring for 1 h at room temperature, the reaction mixture was filtered through a Celite@ pad (washed with CH_2Cl_2), and the filtrate was concentrated in vacuo to afford silyl chloride **4c**, which was used to the following reaction without further purification.

To a solution of alcohol **5a** (39.5 mg, 0.359 mmol) in CH_2Cl_2 (1.8 mL) were added imidazole (50.0 mg, 0.734 mmol) and **4c** (crude, *vide supra*). After stirring for 5 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 95/5) to afford silyl ether **6e** (169 mg, 82%) as a white solid.

6e: **mp** 84–86 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.06 (d, 6H, J = 7.7 Hz), 1.19 (d, 6H, J = 7.7 Hz), 1.62 (qq, 2H, J = 7.7, 7.7 Hz), 2.12 (brt, 2H, J = 9.9 Hz), 2.19–2.26 (m, 2H), 2.47 (s, 3H), 4.24 (s, 2H), 5.73–5.80 (m, 1H), 5.94–5.98 (m, 2H), 7.28 (dd, 1H, J = 3.0 Hz, J_{HF} = 6.6 Hz), 7.32–7.36 (m, 3H), 7.85 (d, 2H, J = 8.4 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 13.8, 17.9, 18.4, 21.8, 22.6, 23.3, 66.9, 117.8 (d, J_{CF} = 9.0 Hz), 118.1, 122.2 (d, J_{CF} = 27.0 Hz), 122.5 (d, J_{CF} = 19.5 Hz), 124.3, 125.1, 128.6, 129.6, 134.3, 136.3 (d, J_{CF} = 3.0 Hz), 137.6, 145.4, 146.4 (d, J_{CF} = 3.0 Hz), 159.9 (d, J_{CF} = 252.0 Hz); **IR** (ATR) 2932, 1565, 1368, 1153 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{26}H_{32}BrFNaO_4SSi$ $[M+Na]^+$: 589.0850; Found: 589.0822.

*Synthesis of tosylate **3d***



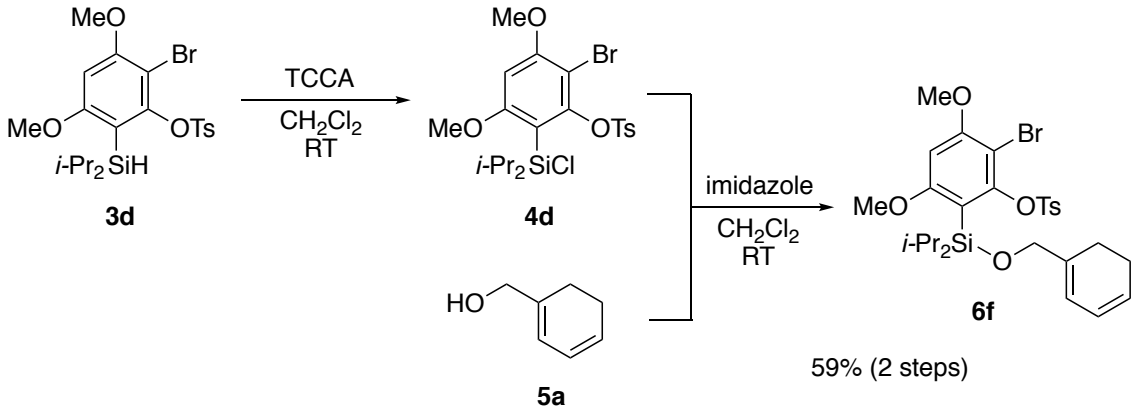
To a solution of dibromophenol **1d** (prepared according to the reported procedure,[6] 312 mg, 1.00 mmol) in CH_2Cl_2 (3.0 mL) were added imidazole (136 mg, 2.00 mmol) and *i*-Pr_2SiHCl (254 μL, 1.50 mmol). After stirring for 5 h at room temperature, hexane (6 mL) was added to the mixture to form white precipitates. CH_2Cl_2 was removed by concentration in vacuo (ca. 300 hPa). The resulting suspension was filtered through a Celite@ pad (washed with Et_2O), and the filtrate was concentrated in vacuo to afford silyl ether **S1d**. The crude material was dissolved in THF (3.0 mL), to which was added dropwise *n*-BuLi (1.55 M in hexane, 0.77 mL, 1.2 mmol) at –78 °C. After stirring for 25 min at this temperature, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x3). The combined

[6] E. Kiehlmann and R. W. Lauener, *Can. J. Chem.*, 1989, **67**, 335–344.

organic layer was washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was filtered through a short plug of silica gel (hexane/EtOAc = 2/1) to afford phenol **2d**. The crude material was dissolved in acetone (2.0 mL), to which were added K_2CO_3 (346 mg, 2.50 mmol) and TsCl (246 mg, 1.29 mmol). After stirring for 9 h at room temperature, 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/EtOAc = 4/1) to afford aryl tosylate **3d** (247 mg, 49%) as a white solid.

3d: **mp** 128.5–129.3 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.93 (d, 6H, *J* = 7.8 Hz), 1.09 (d, 6H, *J* = 7.2 Hz), 1.29 (qqd, 2H, *J* = 7.8, 7.2, 3.9 Hz), 2.45 (s, 3H), 3.82 (s, 3H), 3.90 (s, 3H), 4.12 (t, 1H, *J* = 3.9 Hz), 6.37 (s, 1H), 7.33 (d, 2H, *J* = 7.8 Hz), 7.86 (d, 2H, *J* = 7.8 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 11.7, 19.48, 19.54, 21.7, 55.4, 56.5, 93.8, 98.8, 113.7, 128.8, 129.6, 134.7, 145.0, 152.6, 158.9, 164.2; **IR** (ATR) 2941, 2143, 1583, 1353, 1176 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{21}H_{30}BrO_5SSi$ $[M+H]^+$: 501.0761; Found: 501.0753.

*Synthesis of silyl ether **6f***



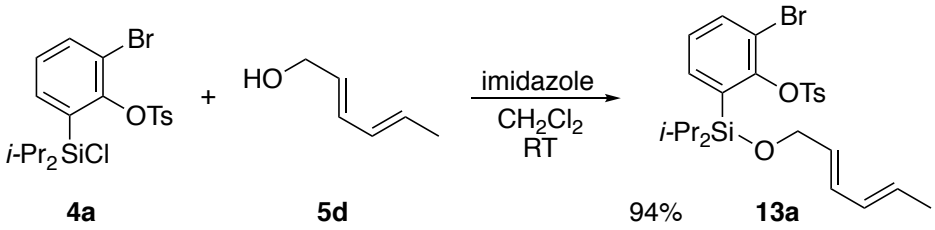
To a solution of hydrosilane **3d** (187 mg, 0.372 mmol) in CH_2Cl_2 (1.9 mL) was added trichloroisocyanuric acid (TCCA, 33.2 mg, 0.143 mmol). After stirring for 15 min at room temperature, the reaction mixture was filtered through a Celite$^{@}$ pad (washed with CH_2Cl_2), and the filtrate was concentrated in vacuo to afford silyl chloride **4d**, which was used to the following reaction without further purification.

To a solution of alcohol **5a** (41.3 mg, 0.375 mmol) in CH_2Cl_2 (1.9 mL) were added imidazole (50.7 mg, 0.745 mmol) and **4d** (crude, *vide supra*). After stirring for 5.5 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (x3). The combined organic layer was washed with brine, dried (Na_2SO_4), and

concentrated in vacuo. The residue was purified by PTLC (CH_2Cl_2) to afford silyl ether **6f** (134 mg, 59%) as white amorphous.

6f: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.02 (d, 6H, *J* = 7.8 Hz), 1.09 (d, 6H, *J* = 7.8 Hz), 1.46 (qq, 2H, *J* = 7.8, 7.8 Hz), 2.05 (brt, 2H, *J* = 9.6 Hz), 2.14–2.21 (m, 2H), 2.43 (s, 3H), 3.80 (s, 3H), 3.91 (s, 3H), 4.21 (s, 2H), 5.67–5.73 (m, 1H), 5.91–5.96 (m, 2H), 6.38 (s, 1H), 7.27 (d, 2H, *J* = 8.1 Hz), 7.85 (d, 2H, *J* = 8.1 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 14.5, 18.1, 18.8, 21.7, 22.7, 23.3, 55.2, 56.5, 66.3, 93.6, 99.7, 113.5, 117.3, 124.2, 124.7, 128.9, 129.4, 134.3, 138.8, 144.8, 151.6, 158.8, 164.4; **IR** (neat) 2941, 1583, 1364, 1069 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{28}H_{37}BrNaO_6SSi$ $[M+Na]^+$: 631.1156; Found: 631.1145.

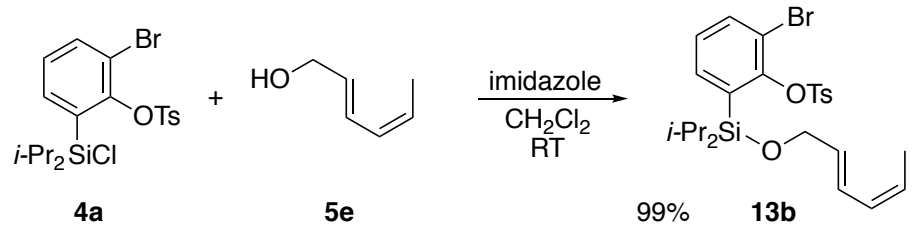
*Synthesis of silyl ether **13a***



To a solution of alcohol **5d** (purchased from Tokyo Chemical Industry Co., Ltd., 75.0 mg, 0.764 mmol) in CH_2Cl_2 (2.5 mL) were added imidazole (67.9 mg, 0.997 mmol) and silyl chloride **4a** (238 mg, 0.500 mmol). After stirring for 6 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 93/7) to afford silyl ether **13a** (235 mg, 94%) as a white solid.

13a: **mp** 100–103 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.05 (d, 6H, *J* = 7.6 Hz), 1.18 (d, 6H, *J* = 7.6 Hz), 1.61 (qq, 2H, *J* = 7.6, 7.6 Hz), 1.77 (d, 3H, *J* = 7.2 Hz), 2.47 (s, 3H), 4.29 (d, 2H, *J* = 4.8 Hz), 5.65–5.74 (m, 2H), 6.05–6.13 (m, 1H), 6.27 (brdd, 1H, *J* = 15.0, 10.8 Hz), 7.15 (dd, 1H, *J* = 7.8, 7.4 Hz), 7.34 (d, 2H, *J* = 8.4 Hz), 7.56 (dd, 1H, *J* = 7.8, 1.7 Hz), 7.60 (dd, 1H, *J* = 7.4, 1.7 Hz), 7.85 (d, 2H, *J* = 8.4 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 13.8, 18.0, 18.1, 18.5, 21.8, 64.3, 117.5, 127.7, 128.6, 129.0, 129.5, 129.6, 130.0, 131.0, 134.1, 134.5, 135.4, 136.5, 145.1, 150.3; **IR** (ATR) 2944, 1366, 1168, 1063 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{25}H_{33}BrNaO_4SSi$ $[M+Na]^+$: 559.0944; Found: 559.0936.

*Synthesis of silyl ether **13b***

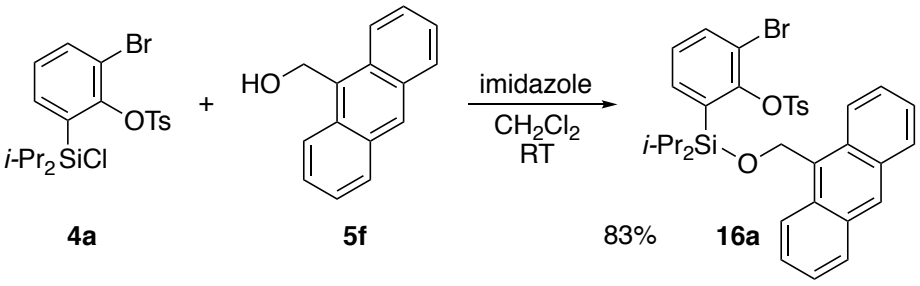


4a **5e** 99% **13b**

To a solution of alcohol **5e** (prepared according to the reported procedure,[7] 48.3 mg, 0.502 mmol) in CH_2Cl_2 (2.5 mL) were added imidazole (67.9 mg, 0.997 mmol) and silyl chloride **4a** (262 mg, 0.551 mmol). After stirring for 11 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 93/7) to afford silyl ether **13b** (266 mg, 99%) as a white solid.

13b: **mp** 88–89 °C; **^{1}H NMR** (600 MHz, acetone-d_6) δ 1.07 (d, 6H, J = 7.8 Hz), 1.20 (d, 6H, J = 7.2 Hz), 1.66 (qq, 2H, J = 7.8, 7.2 Hz), 1.75 (d, 3H, J = 7.2 Hz), 2.50 (s, 3H), 4.40 (d, 2H, J = 4.5 Hz), 5.49 (dq, 1H, J = 10.2, 7.2 Hz), 5.83 (dt, 1H, J = 14.6, 4.5 Hz), 6.07 (brdd, 1H, J = 11.9, 10.2 Hz), 6.74 (brdd, 1H, J = 14.6, 11.9 Hz), 7.33 (dd, 1H, J = 7.8, 7.8 Hz), 7.52 (d, 2H, J = 8.1 Hz), 7.69–7.72 (m, 2H), 7.89 (d, 2H, J = 8.1 Hz); **^{13}C NMR** (150 MHz, acetone-d_6) δ 13.4, 14.6, 18.3, 18.9, 21.7, 65.0, 118.4, 125.5, 126.3, 129.0, 129.4, 129.9, 130.7, 132.8, 134.7, 135.3, 136.6, 137.4, 146.7, 151.2; **IR** (ATR) 2947, 1398, 1357, 1197 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{25}H_{33}BrNaO_4SSi$ $[M+Na]^+$: 559.0944; Found: 559.0931.

*Synthesis of silyl ether **16a***



4a **5f** 83% **16a**

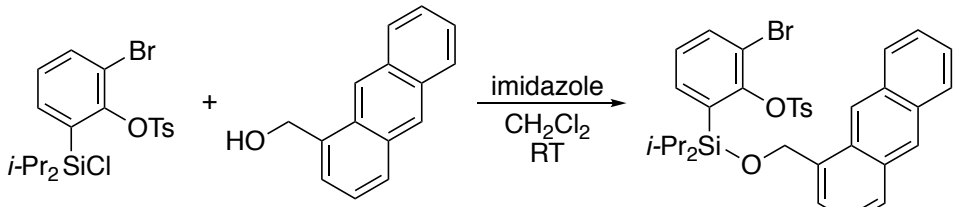
To a solution of alcohol **5f** (purchased from Tokyo Chemical Industry Co., Ltd., 127 mg, 0.610 mmol) in CH_2Cl_2 (2.5 mL) were added imidazole (86.6 mg, 1.27 mmol) and silyl chloride **4a**

[7] A. B. Smith, III, S. M. Pitram, A. M. Boldi, M. J. Gaunt, C. Sfouggatakis and W. H. Moser, *J. Am. Chem. Soc.,* 2003, **125**, 14435–14445.

(238 mg, 0.500 mmol). After stirring for 1.5 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 93/7) to afford silyl ether **16a** (269 mg, 83%) as a pale yellow solid.

16a: **mp** 240 °C (decomp.); **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.08 (d, 6H, *J* = 7.3 Hz), 1.25 (d, 6H, *J* = 7.3 Hz), 1.72 (qq, 2H, *J* = 7.3, 7.3 Hz), 2.46 (s, 3H), 5.77 (s, 2H), 6.93 (dd, 1H, *J* = 7.8, 7.8 Hz), 7.30 (d, 2H, *J* = 8.1 Hz), 7.45–7.52 (m, 5H), 7.54 (dd, 1H, *J* = 7.8, 1.8 Hz), 7.82 (d, 2H, *J* = 8.1 Hz), 8.02 (dd, 2H, *J* = 6.3, 2.4 Hz), 8.36 (dd, 2H, *J* = 6.6, 2.4 Hz), 8.46 (s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 14.0, 18.2, 18.6, 21.8, 58.9, 117.5, 124.9, 125.0, 125.7, 127.8, 127.9, 128.6, 128.9, 129.5, 130.3, 131.5, 131.6, 134.0, 134.4, 135.5, 137.2, 145.2, 150.3; **IR** (ATR) 2943, 1359, 1171, 1073 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{34}H_{35}BrNaO_4SSi$ $[M+Na]^+$: 669.1101; Found: 669.1109.

*Synthesis of silyl ether **16b***



4a **5g** 77% **16b**

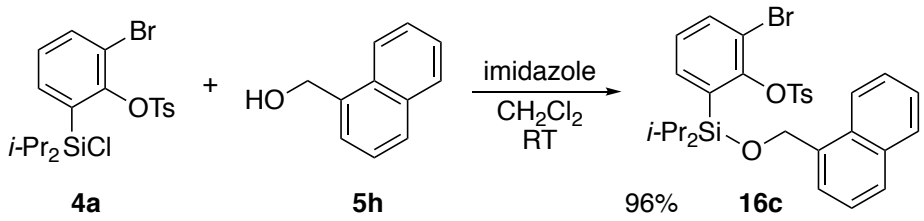
To a solution of alcohol **5g** (prepared according to the reported procedure,[8] 104 mg, 0.499 mmol) in CH_2Cl_2 (2.5 mL) were added imidazole (67.0 mg, 0.984 mmol) and silyl chloride **4a** (262 mg, 0.551 mmol). After stirring for 3 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 93/7) to afford silyl ether **16b** (247 mg, 76%) as a pale yellow solid.

16b: **mp** 232 °C (decomp.); **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.16 (d, 6H, *J* = 7.3 Hz), 1.28 (d, 6H, *J* = 7.3 Hz), 1.75 (qq, 2H, *J* = 7.3, 7.3 Hz), 2.43 (s, 3H), 5.43 (s, 2H), 7.09 (dd, 1H, *J* = 7.8, 7.5 Hz), 7.29 (d, 2H, *J* = 8.1 Hz), 7.43–7.50 (m, 3H), 7.57 (dd, 1H, *J* = 7.8, 1.7 Hz), 7.65 (dd, 1H, *J* = 7.5, 1.7 Hz), 7.69 (d, 1H, *J* = 6.0 Hz), 7.84 (d, 2H, *J* = 8.1 Hz), 7.96 (d, 1H, *J* = 9.0 Hz), 7.97–

[8] F. Xie, K. Sivakumar, Q. Zeng, M. A. Bruckman, B. Hodges and Q. Wang, *Tetrahedron*, 2008, **64**, 2906–2914.

8.02 (m, 2H), 8.46 (s, 1H), 8.50 (s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 14.0, 18.1, 18.7, 21.7, 64.3, 117.8, 121.9, 122.9, 125.0, 125.4, 126.9, 127.8, 127.87, 127.94, 128.5, 128.6, 129.1, 129.5, 131.4, 131.5, 131.8, 133.9, 134.4, 135.6, 136.3, 136.4, 145.2, 150.4 (several signals overlapped); **IR** (ATR) 2942, 1362, 1197, 1093 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{34}H_{35}BrNaO_4SSi$ $[M+Na]^+$: 669.1101; Found: 669.1082.

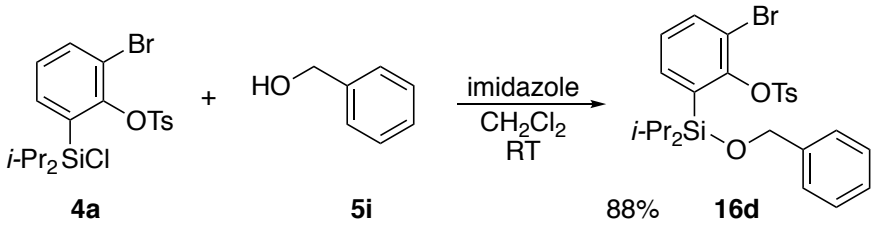
*Synthesis of silyl ether **16c***



To a solution of alcohol **5h** (purchased from Tokyo Chemical Industry Co., Ltd., 79.8 mg, 0.504 mmol) in CH_2Cl_2 (2.5 mL) were added imidazole (69.3 mg, 1.02 mmol) and silyl chloride **4a** (262 mg, 0.551 mmol). After stirring for 3 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 93/7) to afford silyl ether **16c** (290 mg, 96%) as a white solid.

16c: **mp** 135–136 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.13 (d, 6H, *J* = 7.3 Hz), 1.24 (d, 6H, *J* = 7.3 Hz), 1.72 (qq, 2H, *J* = 7.3, 7.3 Hz), 2.44 (s, 3H), 5.31 (s, 2H), 7.08 (dd, 1H, *J* = 7.8, 7.5 Hz), 7.30 (d, 2H, *J* = 8.1 Hz), 7.46–7.52 (m, 3H), 7.56 (dd, 1H, *J* = 7.8, 1.7 Hz), 7.61 (dd, 1H, *J* = 7.5, 1.7 Hz), 7.72 (d, 1H, *J* = 7.2 Hz), 7.79 (d, 1H, *J* = 8.4 Hz), 7.83 (d, 2H, *J* = 8.1 Hz), 7.87–7.90 (m, 1H), 7.90–7.94 (m, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 14.0, 18.1, 18.7, 21.7, 64.0, 117.7, 123.1, 123.5, 125.5, 125.6, 125.9, 127.5, 127.8, 128.6, 129.5, 130.6, 133.5, 133.9, 134.4, 135.6, 136.38, 136.43, 145.2, 150.3 (several signals overlapped); **IR** (ATR) 2941, 1402, 1200, 1034 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{30}H_{33}BrNaO_4SSi$ $[M+Na]^+$: 619.0944; Found: 619.0924.

*Synthesis of silyl ether **16d***

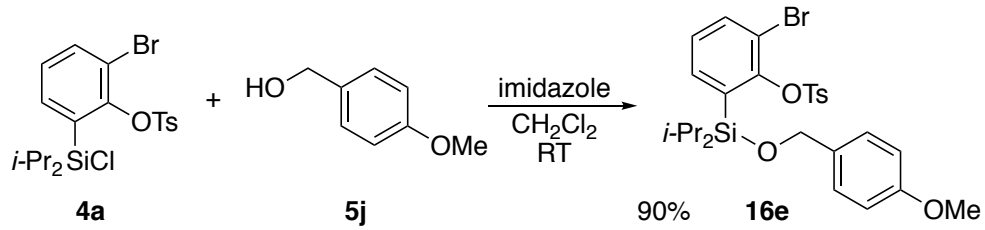


4a **5i** 88% **16d**

To a solution of alcohol **5i** (purchased from Tokyo Chemical Industry Co., Ltd., 77 μL, 0.75 mmol) in CH_2Cl_2 (2.5 mL) were added imidazole (67.1 mg, 0.986 mmol) and silyl chloride **4a** (239 mg, 0.502 mmol). After stirring for 2 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 93/7) to afford silyl ether **16d** (243 mg, 88%) as a white solid.

16d: **mp** 82–83 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.09 (d, 6H, *J* = 7.2 Hz), 1.21 (d, 6H, *J* = 7.8 Hz), 1.66 (qq, 2H, *J* = 7.8, 7.2 Hz), 2.46 (s, 3H), 4.86 (s, 2H), 7.11 (dd, 1H, *J* = 7.8, 7.5 Hz), 7.27 (t, 1H, *J* = 7.5 Hz), 7.32 (d, 2H, *J* = 8.4 Hz), 7.36 (dd, 2H, *J* = 7.5, 7.5 Hz), 7.39 (d, 2H, *J* = 7.5 Hz), 7.56 (dd, 1H, *J* = 7.8, 1.5 Hz), 7.59 (dd, 1H, *J* = 7.5, 1.5 Hz), 7.83 (d, 2H, *J* = 8.4 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 13.9, 18.0, 18.6, 21.8, 65.5, 117.7, 125.8, 126.9, 127.7, 128.2, 128.6, 129.5, 134.0, 134.5, 135.5, 136.4, 141.2, 145.2, 150.3; **IR** (ATR) 2944, 1397, 1196, 1054 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{26}H_{31}BrNaO_4SSi$ $[M+Na]^+$: 569.0788; Found: 569.0768.

*Synthesis of silyl ether **16e***



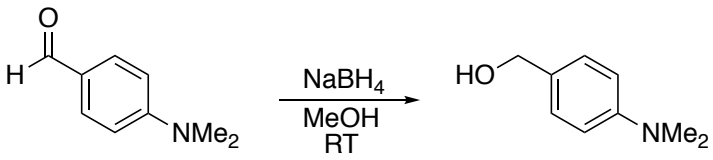
4a **5j** 90% **16e**

To a solution of alcohol **5j** (purchased from Tokyo Chemical Industry Co., Ltd., 105 mg, 0.760 mmol) in CH_2Cl_2 (2.5 mL) were added imidazole (68.4 mg, 0.994 mmol) and silyl chloride **4a** (238 mg, 0.500 mmol). After stirring for 3 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 9/1) to afford silyl ether **16e** (260 mg, 90%) as a white solid.

16e: **mp** 68–69 °C; **^{1}H NMR** (600 MHz, acetone-d_6) δ 1.08 (d, 6H, J = 7.8 Hz), 1.20 (d, 6H, J = 7.8 Hz), 1.68 (qq, 2H, J = 7.8, 7.8 Hz), 2.49 (s, 3H), 3.80 (s, 3H), 4.84 (s, 2H), 6.91–6.95 (m, 2H), 7.30 (dd, 1H, J = 7.8, 7.8 Hz), 7.36 (d, 2H, J = 8.4 Hz), 7.50 (d, 2H, J = 8.4 Hz), 7.69–7.72 (m, 2H), 7.87 (d, 2H, J = 8.4 Hz); **^{13}C NMR** (150 MHz, acetone-d_6) δ 14.5, 18.3, 18.9, 21.6, 55.5, 66.1, 114.5, 118.3, 128.3, 129.0, 129.4, 130.7, 134.0, 134.7, 135.3, 136.6, 137.5, 146.7, 151.2, 159.9; **IR** (ATR) 2941, 1513, 1397, 1247 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{27}H_{33}BrNaO_5SSi$ $[M+Na]^+$: 599.0894; Found: 599.0877.

*Synthesis of benzyl alcohol **5k***

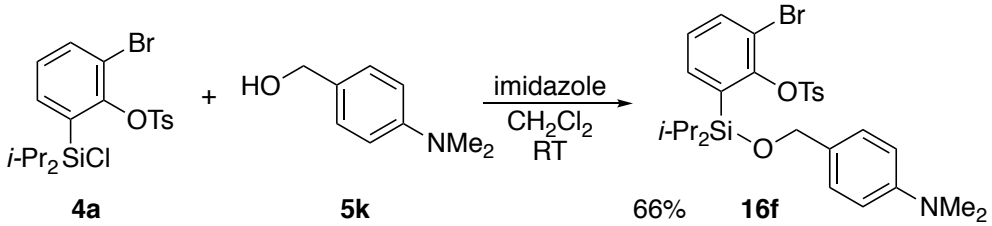


S4 91% **5k**

To a solution of aldehyde **S4** (purchased from Tokyo Chemical Industry Co., Ltd., 450 mg, 3.02 mmol) in MeOH (6.0 mL) was added sodium borohydride (137 mg, 3.62 mmol). After stirring for 20 min at room temperature, the reaction was quenched by adding saturated aqueous NH_4Cl, and the mixture was extracted with EtOAc (x6). 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 = 3/2) to afford alcohol **5k** (417 mg, 91%) as colorless oil.

5k: Spectral data matched that reported in the literature.[9]

*Synthesis of silyl ether **16f***



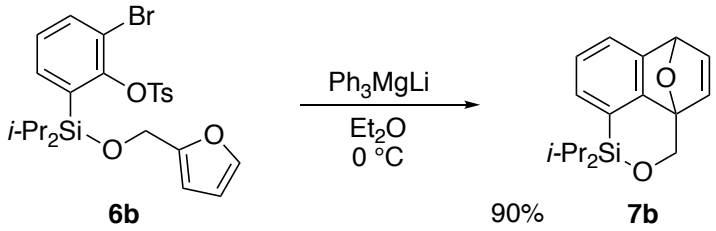
4a **5k** 66% **16f**

[9] A. Orita, H. Taniguchi and J. Otera, *Chem. Asian J.*, 2006, **1**, 430–437.

To a solution of alcohol **5k** (114 mg, 0.754 mmol) in CH_2Cl_2 (2.5 mL) were added imidazole (69.1 mg, 1.01 mmol) and silyl chloride **4a** (238 mg, 0.500 mmol). After stirring for 1 h at room temperature, the reaction was quenched by adding saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2 (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 = 85/15) to afford silyl ether **16f** as a white solid, contaminated with impurity. This material was further purified by reprecipitation (hexane/CH_2Cl_2) to afford **16f** (148 mg, 50%) as a white solid. The mother liquor concentrated in vacuo, and the residue was purified by gel-permeation chromatography [YMC-GPC T4000® (2.0 cm φ×60 cm)+T2000® (2.0 cm φ×60 cm), EtOAc, flow rate 7.0 mL/min] to afford **16f** (47.4 mg, 16%) as a white solid.

16f: **mp** 117 °C (decomp.); **^{1}H NMR** (600 MHz, acetone-d_6) δ 1.07 (d, 6H, J = 7.2 Hz), 1.20 (d, 6H, J = 7.2 Hz), 1.67 (qq, 2H, J = 7.2, 7.2 Hz), 2.49 (s, 3H), 2.93 (s, 6H), 4.78 (s, 2H), 6.73–6.78 (m, 2H), 7.26 (d, 2H, J = 8.4 Hz), 7.29 (dd, 1H, J = 7.8, 7.2 Hz), 7.50 (d, 2H, J = 8.1 Hz), 7.70 (dd, 1H, J = 7.8, 1.5 Hz), 7.72 (dd, 1H, J = 7.2, 1.5 Hz), 7.88 (d, 2H, J = 8.1 Hz); **^{13}C NMR** (150 MHz, acetone-d_6) δ 14.6, 18.4, 19.0, 21.6, 40.7, 66.5, 113.2, 118.3, 128.2, 128.9, 129.4, 129.7, 130.7, 134.8, 135.3, 136.5, 137.6, 146.7, 151.0, 151.2; **IR** (ATR) 2931, 1525, 1369, 1168 cm^{-1}; **HRMS** (ESI): Calcd. for $C_{28}H_{37}BrNO_4SSi$ $[M+H]^+$: 590.1390; Found: 590.1376.

*Synthesis of cycloadduct **7b***



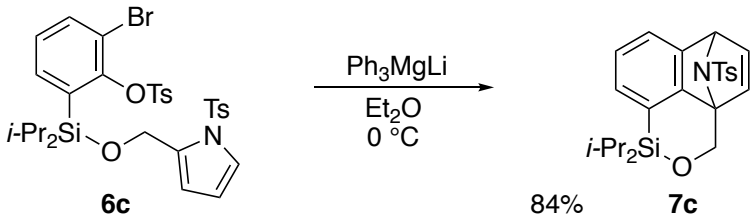
6b 90% **7b**

According to the typical procedure, cycloadduct **7b** was prepared from the reaction of bromoaryl tosylate **6b** (44.0 mg, 0.0819 mmol) in Et_2O (1.6 mL) and Ph_3MgLi [prepared from PhLi (0.70 M in cyclohexane and Et_2O, 0.29 mL, 0.20 mmol) and PhMgBr (0.83 M in THF, 0.13 mL, 0.11 mmol) in Et_2O (0.40 mL)] at 0 °C for 10 min. Purification by PTLC (hexane/Et_2O = 93/7) afforded **7b** (21.0 mg, 90%) as colorless oil.

7b: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.94 (d, 3H, J = 7.8 Hz), 0.97 (d, 3H, J = 7.2 Hz), 1.09 (d, 3H, J = 7.2 Hz), 1.12 (d, 3H, J = 7.8 Hz), 1.14 (qq, 1H, J = 7.8, 7.2 Hz), 1.26 (qq, 1H, J = 7.8, 7.2 Hz), 4.40 (d, 1H, J = 10.2 Hz), 4.61 (d, 1H, J = 10.2 Hz), 5.71 (d, 1H, J = 1.8 Hz), 6.96 (dd,

1H, *J* = 7.4, 7.4 Hz), 7.03 (brd, 1H, *J* = 5.4 Hz), 7.06 (d, 1H, *J* = 7.4 Hz), 7.09 (d, 1H, *J* = 5.4 Hz), 7.27 (d, 1H, *J* = 7.4 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 12.1, 13.6, 17.01, 17.03, 17.3, 17.7, 64.0, 82.1, 87.4, 121.2, 124.4, 124.8, 128.4, 143.4, 143.8, 147.3, 157.3; **IR** (neat) 2942, 1463, 1082, 991 cm^{-1}; **HRMS** (APCI) Calcd. for $C_{17}H_{23}O_2Si$ $[M+H]^+$: 287.1462; Found: 287.1462.

*Synthesis of cycloadduct **7c***

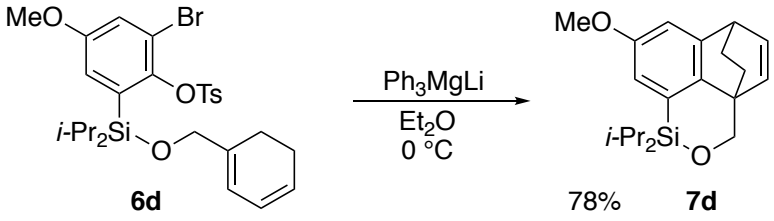


6c 84% **7c**

According to the typical procedure, cycloadduct **7c** was prepared from the reaction of bromoaryl tosylate **6c** (85.9 mg, 0.124 mmol) in Et_2O (2.5 mL) and Ph_3MgLi [prepared from PhLi (0.74 M in cyclohexane and Et_2O, 0.40 mL, 0.30 mmol) and PhMgBr (0.63 M in THF, 0.26 mL, 0.16 mmol) in Et_2O (0.60 mL)] at 0 °C for 20 min. Purification by PTLC (hexane/Et_2O = 3/2) afforded **7c** (46.1 mg, 84%) as a white solid.

7c: **mp** 137 °C (decomp.); **^{1}H NMR** (600 MHz, acetone-d_6) δ 0.86 (d, 3H, *J* = 7.2 Hz), 0.87 (d, 3H, *J* = 7.2 Hz), 1.09 (qq, 1H, *J* = 7.2, 7.2 Hz), 1.14 (d, 3H, *J* = 7.8 Hz), 1.15 (d, 3H, *J* = 7.2 Hz), 1.31 (qq, 1H, *J* = 7.8, 7.2 Hz), 2.40 (s, 3H), 4.74 (d, 1H, *J* = 11.4 Hz), 4.81 (d, 1H, *J* = 11.4 Hz), 5.61 (d, 1H, *J* = 1.8 Hz), 6.56 (d, 1H, *J* = 5.7 Hz), 6.61 (brd, 1H, *J* = 5.7 Hz), 6.95 (dd, 1H, *J* = 7.8, 7.2 Hz), 7.07 (d, 1H, *J* = 7.8 Hz), 7.31 (d, 1H, *J* = 7.2 Hz), 7.36 (d, 2H, *J* = 7.9 Hz), 7.60 (d, 2H, *J* = 7.9 Hz); **^{13}C NMR** (150 MHz, acetone-d_6) δ 11.7, 13.3, 16.5, 16.6, 16.8, 17.3, 20.6, 62.7, 68.4, 74.5, 121.8, 124.4, 125.0, 128.2, 128.5, 129.8, 136.6, 140.8, 143.1, 143.7, 146.1, 157.5; **IR** (ATR) 2945, 1337, 1159, 1026 cm^{-1}; **HRMS** (ESI) Calcd. for $C_{24}H_{29}NNaO_3SSi$ $[M+Na]^+$: 462.1530; Found: 462.1512.

*Synthesis of cycloadduct **7d***

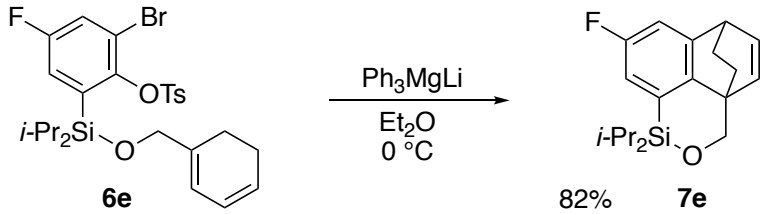


6d 78% **7d**

According to the typical procedure, cycloadduct **7d** was prepared from the reaction of bromoaryl tosylate **6d** (58.1 mg, 0.100 mmol) in Et_2O (2.0 mL) and Ph_3MgLi [prepared from PhLi (0.74 M in cyclohexane and Et_2O, 0.33 mL, 0.24 mmol) and PhMgBr (0.95 M in THF, 0.14 mL, 0.13 mmol) in Et_2O (0.50 mL)] at 0 °C for 10 min. Purification by PTLC (hexane/EtOAc = 97/3 x2) afforded **7d** (25.7 mg, 78%) as colorless oil.

7d: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.97 (d, 3H, *J* = 7.2 Hz), 1.01 (d, 3H, *J* = 7.8 Hz), 1.11 (d, 3H, *J* = 7.2 Hz), 1.14 (d, 3H, *J* = 7.2 Hz), 1.16 (qq, 1H, *J* = 7.8, 7.2 Hz), 1.29 (qq, 1H, *J* = 7.2, 7.2 Hz), 1.34 (ddd, 1H, *J* = 10.2, 10.2, 4.4 Hz), 1.44–1.50 (m, 1H), 1.61–1.72 (m, 2H), 3.79 (s, 3H), 3.84–3.87 (m, 1H), 4.28 (d, 1H, *J* = 11.4 Hz), 4.57 (d, 1H, *J* = 11.4 Hz), 6.06 (d, 1H, *J* = 7.8 Hz), 6.55 (dd, 1H, *J* = 7.8, 7.2 Hz), 6.70 (d, 1H, *J* = 2.4 Hz), 6.81 (d, 1H, *J* = 2.4 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 12.7, 13.3, 17.3, 17.4, 17.5, 17.9, 27.5, 30.3, 41.3, 44.6, 55.3, 68.4, 110.3, 113.7, 127.3, 135.4, 136.2, 143.2, 145.7, 156.5; **IR** (neat) 2943, 1596, 1464, 1254, 1071 cm^{-1}; **HRMS** (ESI) Calcd. for $C_{20}H_{29}O_2Si$ $[M+H]^+$: 329.1931; Found: 329.1924.

*Synthesis of cycloadduct **7e***

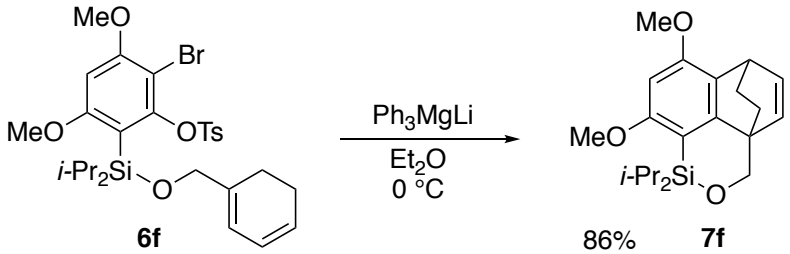


According to the typical procedure, cycloadduct **7e** was prepared from the reaction of bromoaryl tosylate **6e** (57.1 mg, 0.101 mmol) in Et_2O (2.0 mL) and Ph_3MgLi [prepared from PhLi (0.74 M in cyclohexane and Et_2O, 0.33 mL, 0.24 mmol) and PhMgBr (0.95 M in THF, 0.14 mL, 0.13 mmol) in Et_2O (0.50 mL)] at 0 °C for 10 min. Purification by PTLC (hexane/EtOAc = 95/5) afforded **7e** (26.2 mg, 82%) as colorless oil.

7e: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.95 (d, 3H, *J* = 7.8 Hz), 1.01 (d, 3H, *J* = 7.8 Hz), 1.10 (d, 3H, *J* = 7.8 Hz), 1.13 (d, 3H, *J* = 7.8 Hz), 1.17 (qq, 1H, *J* = 7.8, 7.8 Hz), 1.29 (qq, 1H, *J* = 7.8, 7.8 Hz), 1.36 (ddd, 1H, *J* = 11.0, 11.0, 4.4 Hz), 1.44–1.50 (m, 1H), 1.63–1.71 (m, 2H), 3.87–3.90 (m, 1H), 4.29 (d, 1H, *J* = 12.0 Hz), 4.57 (d, 1H, *J* = 12.0 Hz), 6.06 (d, 1H, *J* = 7.8 Hz), 6.55 (dd, 1H, *J* = 7.8, 6.0 Hz), 6.86 (dd, 1H, *J* = 2.6 Hz, J_{HF} = 9.0 Hz), 6.91 (dd, 1H, *J* = 2.6 Hz, J_{HF} = 8.7 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 12.6, 13.2, 17.2, 17.3, 17.4, 17.9, 27.3, 30.1, 41.0, 44.9, 68.2, 111.3 (d, J_{CF} = 22.5 Hz), 114.8 (d, J_{CF} = 19.5 Hz), 128.4 (d, J_{CF} = 4.5 Hz), 135.4, 135.9, 146.3 (d, J_{CF} = 1.5 Hz), 146.5 (d, J_{CF} = 6.0 Hz), 160.2 (d, J_{CF} = 245 Hz); **IR** (neat) 2944,

1453, 1251, 1057 cm^{-1}; **HRMS** (ESI) Calcd. for $C_{19}H_{26}FOSi$ $[M+H]^+$: 317.1732; Found: 317.1733.

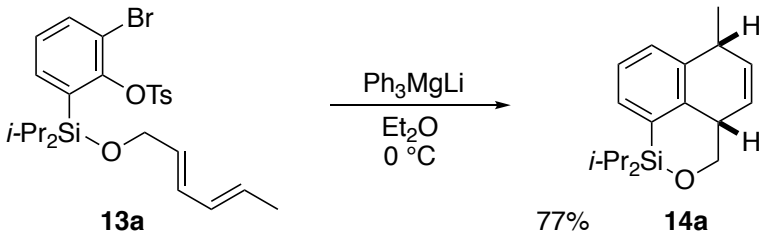
*Synthesis of cycloadduct **7f***



According to the typical procedure, cycloadduct **7f** was prepared from the reaction of bromoaryl tosylate **6f** (64.3 mg, 0.105 mmol) in Et_2O (2.1 mL) and Ph_3MgLi [prepared from PhLi (0.74 M in cyclohexane and Et_2O, 0.34 mL, 0.25 mmol) and PhMgBr (0.95 M in THF, 0.15 mL, 0.14 mmol) in Et_2O (0.50 mL)] at 0 °C for 10 min. Purification by PTLC (hexane/EtOAc = 95/5) afforded **7f** (32.4 mg, 86%) as a white solid.

7f: **mp** 71–77 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.91 (d, 3H, *J* = 7.2 Hz), 1.02 (d, 3H, *J* = 7.8 Hz), 1.05 (d, 3H, *J* = 7.8 Hz), 1.11 (d, 3H, *J* = 7.2 Hz), 1.21 (qq, 1H, *J* = 7.8, 7.2 Hz), 1.27 (qq, 1H, *J* = 7.8, 7.2 Hz), 1.32 (ddd, 1H, *J* = 11.1, 10.4, 4.2 Hz), 1.43 (dddd, 1H, *J* = 11.4, 9.6, 4.2, 2.8 Hz), 1.55–1.61 (m, 1H), 1.65 (ddd, 1H, *J* = 11.1, 9.6, 3.9 Hz), 3.75 (s, 3H), 3.85 (s, 3H), 4.23 (d, 1H, *J* = 11.4 Hz), 4.28–4.31 (m, 1H), 4.49 (d, 1H, *J* = 11.4 Hz), 6.04 (d, 1H, *J* = 7.5 Hz), 6.19 (s, 1H), 6.56 (dd, 1H, *J* = 7.5, 6.6 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 12.8, 13.7, 17.4, 17.6, 18.1, 18.6, 27.2, 29.9, 33.1, 45.1, 54.9, 55.4, 68.3, 90.5, 107.3, 124.2, 135.7, 136.4, 154.0, 155.2, 162.4; **IR** (ATR) 2940, 1571, 1462, 1321, 1208 cm^{-1}; **HRMS** (ESI) Calcd. for $C_{21}H_{31}O_3Si$ $[M+H]^+$: 359.2037; Found: 359.2045.

*Synthesis of cycloadduct **14a***

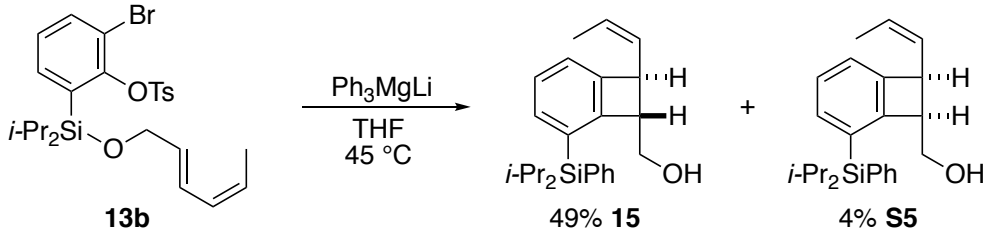


According to the typical procedure, cycloadduct **14a** was prepared from the reaction of bromoaryl tosylate **13a** (53.8 mg, 0.100 mmol) in Et_2O (2.0 mL) and Ph_3MgLi [prepared from PhLi (0.74 M in cyclohexane and Et_2O, 0.33 mL, 0.24 mmol) and PhMgBr (0.94 M in THF,

0.14 mL, 0.13 mmol) in Et_2O (0.50 mL)] at 0 °C for 10 min. Purification by gel-permeation chromatography [YMC-GPC T4000® (2.0 cm φ×60 cm)+T2000® (2.0 cm φ×60 cm), EtOAc, flow rate 7.0 mL/min] afforded **14a** (22.2 mg, 77%) as colorless oil.

14a: **^{1}H NMR** (600 MHz, acetone-d_6) δ 0.96 (d, 3H, J = 7.2 Hz), 0.99 (d, 3H, J = 7.2 Hz), 1.01 (d, 3H, J = 7.8 Hz), 1.09 (d, 3H, J = 7.2 Hz), 1.13 (qq, 1H, J = 7.8, 7.2 Hz), 1.25 (qq, 1H, J = 7.2, 7.2 Hz), 1.37 (d, 3H, J = 7.2 Hz), 3.40–3.48 (m, 2H), 3.65–3.72 (m, 1H), 4.16 (dd, 1H, J = 10.2, 3.6 Hz), 5.61 (brd, 1H, J = 9.6 Hz), 5.87 (brd, 1H, J = 9.6 Hz), 7.27 (dd, 1H, J = 7.7, 7.1 Hz), 7.33 (dd, 1H, J = 7.1, 1.2 Hz), 7.40 (dd, 1H, J = 7.7, 1.2 Hz); **^{13}C NMR** (150 MHz, acetone-d_6) δ 13.6, 13.7, 17.0, 17.1, 17.3, 17.8, 23.3, 33.4, 40.8, 70.0, 123.9, 126.5, 129.0, 131.4, 132.0, 133.7, 138.4, 143.7; **IR** (neat) 2942, 1462, 1104, 1049 cm^{-1}; **HRMS** (ESI) Calcd. for $C_{18}H_{27}OSi$ $[M+H]^+$: 287.1826; Found: 287.1819.

*Synthesis of cycloadducts **15** and **S5***



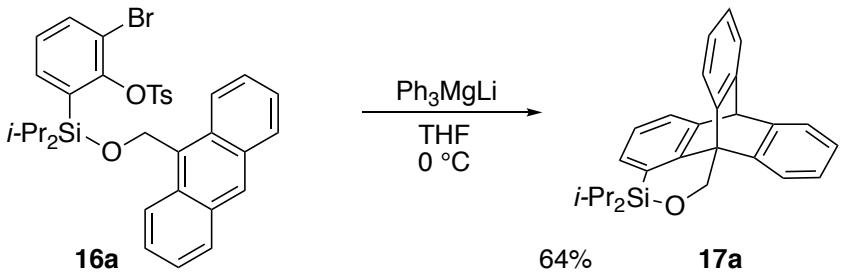
According to the typical procedure, cycloadducts **15** and **S5** were prepared from the reaction of bromoaryl tosylate **13b** (53.7 mg, 0.0999 mmol) in THF (2.0 mL) and Ph_3MgLi [prepared from PhLi (0.65 M in cyclohexane and Et_2O, 0.37 mL, 0.24 mmol) and PhMgBr (0.95 M in THF, 0.14 mL, 0.13 mmol) in THF (0.50 mL)] at 45 °C for 10 min. Purification by PTLC (hexane/acetone = 93/7 x2) afforded **S5** (1.5 mg, 4%) as colorless oil. The fraction containing **15** and impurity was further purified by PTLC (hexane/EtOAc = 9/1) to afford **15** (17.8 mg, 49%) as colorless oil.

15: **^{1}H NMR** (600 MHz, acetone-d_6) δ 0.95 (d, 3H, J = 7.2 Hz), 0.98 (d, 3H, J = 7.2 Hz), 0.99 (d, 3H, J = 7.8 Hz), 1.03 (d, 3H, J = 7.8 Hz), 1.58–1.68 (m, 2H), 1.77 (dd, 3H, J = 6.6, 1.4 Hz), 3.24 (ddd, 1H, J = 8.7, 4.5, 2.0 Hz), 3.41 (ddd, 1H, J = 10.8, 8.7, 5.7 Hz), 3.50–3.55 (m, 1H), 3.67 (ddd, 1H, J = 10.8, 5.6, 4.5 Hz), 4.21 (brd, 1H, J = 9.0 Hz), 5.51 (dqd, 1H, J = 10.7, 6.6, 1.0 Hz), 5.58 (ddq, 1H, J = 10.7, 9.0, 1.4 Hz), 7.10 (d, 1H, J = 7.5 Hz), 7.24 (dd, 1H, J = 7.8, 7.5 Hz), 7.35 (d, 1H, J = 7.8 Hz), 7.39–7.46 (m, 3H), 7.54–7.58 (m, 2H); **^{13}C NMR** (150 MHz, acetone-d_6) δ 10.7, 11.1, 13.5, 18.1, 18.2, 18.4, 45.1, 57.3, 64.8, 123.8, 125.2, 127.7, 128.4, 129.6, 130.2, 132.9, 134.0, 136.4, 136.8, 148.7, 153.3 (several signals overlapped); **IR** (neat)

3407, 2943, 1462, 1392, 1108 cm^{-1}; **HRMS** (ESI) Calcd. for $C_{24}H_{32}NaOSi$ $[M+Na]^+$: 387.2115; Found: 387.2113.

S5: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.94 (d, 3H, *J* = 7.8 Hz), 0.96–1.01 (m, 6H), 1.03 (d, 3H, *J* = 7.2 Hz), 1.44 (dd, 1H, *J* = 9.6, 4.2 Hz), 1.52–1.61 (m, 2H), 1.80 (d, 3H, *J* = 5.4 Hz), 3.41 (ddd, 1H, *J* = 11.4, 9.6, 4.7 Hz), 3.52–3.60 (m, 1H), 3.79 (ddd, 1H, *J* = 9.9, 5.4, 4.7 Hz), 4.45 (dd, 1H, *J* = 9.0, 5.4 Hz), 5.71–5.79 (m, 2H), 7.12 (d, 1H, *J* = 7.2 Hz), 7.25 (dd, 1H, *J* = 7.8, 7.2 Hz), 7.33–7.43 (m, 4H), 7.53 (dd, 2H, *J* = 8.1, 1.5 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 10.0, 10.4, 13.5, 17.7, 17.81, 17.82, 17.9, 41.5, 52.9, 63.0, 123.0, 127.0, 127.6, 128.0, 128.8, 129.4, 129.5, 133.1, 135.87, 135.93, 147.1, 151.2; **IR** (neat) 3452, 2942, 1462, 1392, 1108 cm^{-1}; **HRMS** (ESI) Calcd. for $C_{24}H_{32}NaOSi$ $[M+Na]^+$: 387.2115; Found: 387.2111.

*Synthesis of cycloadduct **17a***

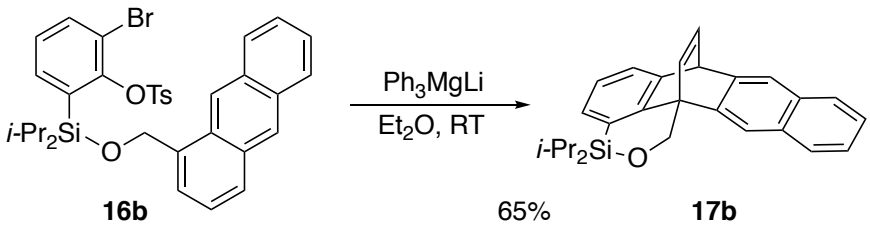


16a 64% **17a**

According to the typical procedure, cycloadduct **17a** was prepared from the reaction of bromoaryl tosylate **16a** (65.1 mg, 0.101 mmol) in THF (2.0 mL) and Ph_3MgLi [prepared from PhLi (0.74 M in cyclohexane and Et_2O, 0.33 mL, 0.24 mmol) and PhMgBr (0.63 M in THF, 0.21 mL, 0.13 mmol) in THF (0.50 mL)] at 0 °C for 10 min. Purification by PTLC (hexane/acetone = 95/5) afforded **17a** (25.4 mg, 64%) as colorless oil.

17a: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.97 (d, 6H, *J* = 7.2 Hz), 1.01 (d, 6H, *J* = 7.8 Hz), 1.25 (qq, 2H, *J* = 7.8, 7.2 Hz), 5.36 (s, 1H), 5.37 (s, 2H), 6.95–7.03 (m, 5H), 7.10 (d, 1H, *J* = 7.2 Hz), 7.35–7.42 (m, 5H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 13.0, 17.3, 17.7, 52.1, 54.2, 62.5, 121.5, 123.5, 124.2, 124.6, 125.0, 125.1, 128.1, 129.7, 145.0, 145.4, 146.6, 152.3; **IR** (neat) 2942, 1458, 1087 cm^{-1}; **HRMS** (APCI) Calcd. for $C_{27}H_{29}OSi$ $[M+H]^+$: 397.1982; Found: 397.1989.

*Synthesis of cycloadduct **17b***

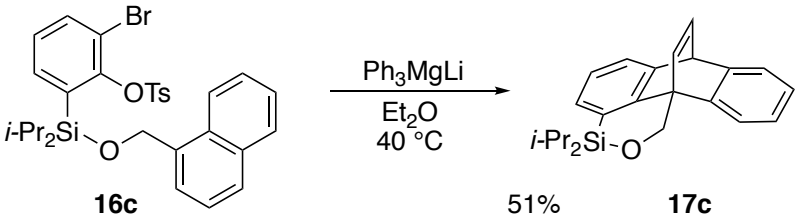


16b 65% **17b**

According to the typical procedure, cycloadduct **17b** was prepared from the reaction of bromoaryl tosylate **16b** (65.0 mg, 0.100 mmol) in Et_2O (2.0 mL) and Ph_3MgLi [prepared from PhLi (0.74 M in cyclohexane and Et_2O, 0.32 mL, 0.24 mmol) and PhMgBr (0.95 M in THF, 0.14 mL, 0.13 mmol) in Et_2O (0.50 mL)] at room temperature for 10 min. Purification by PTLC (hexane/CH_2Cl_2/toluene = 3/1/1 x2) afforded **17b** (25.9 mg, 65%) as colorless oil.

17b: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.75 (d, 3H, *J* = 7.2 Hz), 0.78 (d, 3H, *J* = 7.2 Hz), 1.09 (qq, 1H, *J* = 7.2, 7.2 Hz), 1.21 (d, 3H, *J* = 7.2 Hz), 1.24 (d, 3H, *J* = 7.2 Hz), 1.40 (qq, 1H, *J* = 7.2, 7.2 Hz), 4.89 (d, 1H, *J* = 12.3 Hz), 5.15 (dd, 1H, *J* = 6.0, 0.9 Hz), 5.28 (d, 1H, *J* = 12.3 Hz), 6.41 (dd, 1H, *J* = 7.2, 0.9 Hz), 6.98 (dd, 1H, *J* = 7.5, 7.5 Hz), 7.08–7.12 (m, 2H), 7.34 (d, 1H, *J* = 7.5 Hz), 7.34–7.39 (m, 2H), 7.62 (s, 1H), 7.67 (dd, 1H, *J* = 6.0, 3.6 Hz), 7.73 (dd, 1H, *J* = 6.0, 3.6 Hz), 7.89 (s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 12.6, 13.5, 17.1, 17.2, 17.4, 18.1, 50.8, 52.9, 64.4, 119.3, 120.8, 123.9, 124.2, 125.5, 125.6, 127.2, 127.5, 127.8, 129.3, 131.0, 131.4, 138.9, 140.0, 143.4, 143.9, 145.2, 152.2; **IR** (neat) 2941, 1462, 1093 cm^{-1}; **HRMS** (ESI) Calcd. for $C_{27}H_{29}OSi$ $[M+H]^+$: 397.1982; Found: 397.1989.

*Synthesis of cycloadduct **17c***

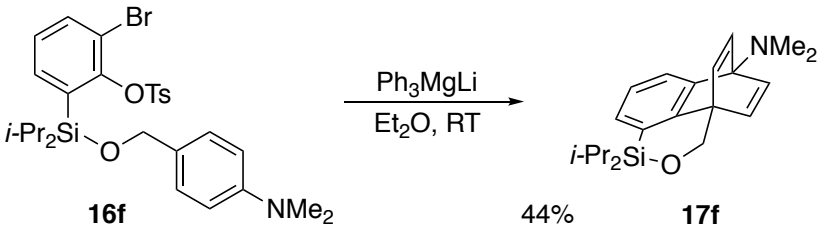


16c 51% **17c**

According to the typical procedure, cycloadduct **17c** was prepared from the reaction of bromoaryl tosylate **16c** (60.0 mg, 0.100 mmol) in Et_2O (2.0 mL) and Ph_3MgLi [prepared from PhLi (0.74 M in cyclohexane and Et_2O, 0.33 mL, 0.24 mmol) and PhMgBr (0.95 M in THF, 0.14 mL, 0.13 mmol) in Et_2O (0.50 mL)] at 40 °C for 10 min. Purification by PTLC (hexane/CH_2Cl_2 = 2/1) afforded **17c** (17.8 mg, 51%) as colorless oil.

17c: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 0.77 (d, 3H, *J* = 7.2 Hz), 0.82 (d, 3H, *J* = 7.2 Hz), 1.09 (qq, 1H, *J* = 7.2, 7.2 Hz), 1.17 (d, 3H, *J* = 7.2 Hz), 1.20 (d, 3H, *J* = 7.2 Hz), 1.37 (qq, 1H, *J* = 7.2, 7.2 Hz), 4.83 (d, 1H, *J* = 12.3 Hz), 5.06 (dd, 1H, *J* = 6.0, 1.5 Hz), 5.18 (d, 1H, *J* = 12.3 Hz), 6.43 (dd, 1H, *J* = 7.2, 1.5 Hz), 6.90–6.95 (m, 2H), 6.98 (ddd, 1H, *J* = 7.5, 7.2, 1.2 Hz), 7.05 (dd, 1H, *J* = 7.8, 1.2 Hz), 7.10 (dd, 1H, *J* = 7.2, 6.0 Hz), 7.25 (brd, 1H, *J* = 7.2 Hz), 7.29 (dd, 1H, *J* = 7.2, 1.2 Hz), 7.52 (d, 1H, *J* = 7.5 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 12.6, 13.5, 17.1, 17.2, 17.4, 18.0, 51.4, 53.6, 64.2, 121.0, 122.9, 123.4, 123.98, 124.01, 124.3, 127.2, 128.8, 139.7, 140.7, 146.2, 146.6, 147.3, 153.3; **IR** (neat) 2941, 1462, 1090 cm^{-1}; **HRMS** (ESI) Calcd. for $C_{23}H_{27}OSi$ $[M+H]^+$: 347.1826; Found: 347.1833.

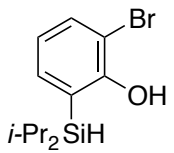
*Synthesis of cycloadduct **17f***



16f 44% **17f**

According to the typical procedure, cycloadduct **17f** was prepared from the reaction of bromoaryl tosylate **16f** (59.0 mg, 0.100 mmol) in Et_2O (2.0 mL) and Ph_3MgLi [prepared from PhLi (0.65 M in cyclohexane and Et_2O, 0.37 mL, 0.24 mmol) and PhMgBr (0.96 M in THF, 0.14 mL, 0.13 mmol) in Et_2O (0.50 mL)] at room temperature for 10 min. Purification by PTLC (hexane/EtOAc = 4/1) afforded **17f** (14.9 mg, 44%) as colorless oil.

17f: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 1.01 (d, 6H, *J* = 7.8 Hz), 1.04 (d, 6H, *J* = 7.2 Hz), 1.22 (qq, 2H, *J* = 7.8, 7.2 Hz), 2.76 (s, 6H), 4.71 (s, 2H), 6.68 (d, 2H, *J* = 6.9 Hz), 6.93 (dd, 1H, *J* = 7.2, 7.2 Hz), 6.98 (dd, 1H, *J* = 7.2, 1.2 Hz), 7.02 (d, 2H, *J* = 6.9 Hz), 7.41 (dd, 1H, *J* = 7.2, 1.2 Hz); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 12.9, 17.3, 17.7, 42.1, 54.5, 66.3, 77.3, 122.1, 122.5, 125.6, 127.4, 139.8, 141.3, 147.9, 155.6; **IR** (neat) 2943, 1462, 1392, 1086 cm^{-1}; **HRMS** (ESI) Calcd. for $C_{21}H_{30}NOSi$ $[M+H]^+$: 340.2091; Found: 340.2093.



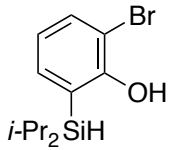
2a

Current Data Parameters
NAME AN2−1683−1
EXPNO 10
PROCNO 1

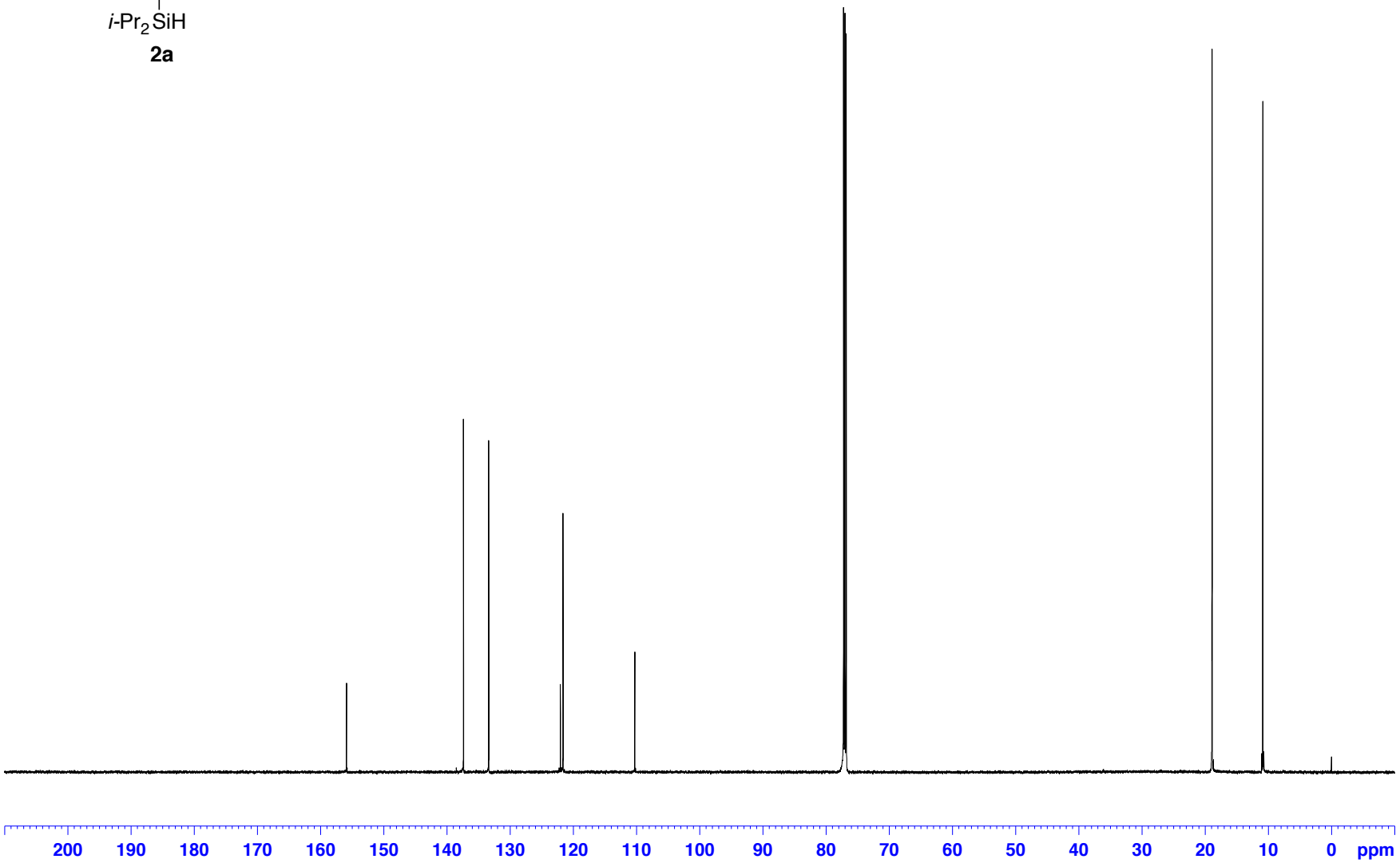
F2 − Acquisition Parameters
Date_ 20180209
Time 16.29
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300151 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



2a



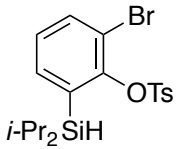
Current Data Parameters
NAME AN2−1448−1
EXPNO 11
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170629
Time 14.05
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028096 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



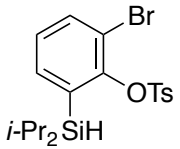
3a

Current Data Parameters
NAME AN2–1687–1–1
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180215
Time 4.58
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 17.5
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3a

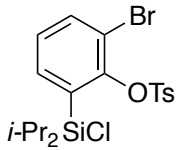
Current Data Parameters
NAME AN2-1687-1-1
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180215
Time 5.50
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 - Processing parameters
SI 32768
SF 150.9028095 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



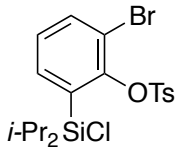
4a

Current Data Parameters
NAME AN2–1752–cr
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180616
Time 13.34
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4a

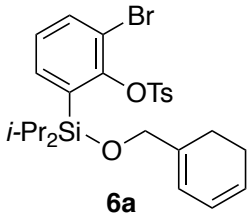
Current Data Parameters
NAME AN2−1491−1
EXPNO 11
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170801
Time 1.48
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028081 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



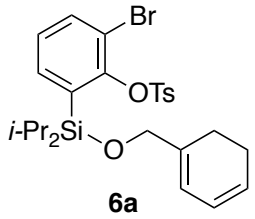
6a

Current Data Parameters
NAME AN2−1455−data
EXPNO 31
PROCNO 1

F2 − Acquisition Parameters
Date_ 20180517
Time 0.02
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 15.79
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300152 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



6a

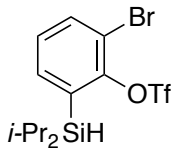
Current Data Parameters
NAME AN2–1455–data
EXPNO 32
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180517
Time 0.54
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028094 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



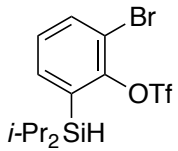
3e

Current Data Parameters
NAME AN2−1331−data
EXPNO 10
PROCNO 1

F2 − Acquisition Parameters
Date_ 20180427
Time 14.52
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3e

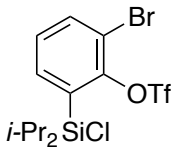
Current Data Parameters
NAME AN2−1331−data
EXPNO 12
PROCNO 1

F2 − Acquisition Parameters
Date_ 20180428
Time 3.33
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65356
SOLVENT CDCl3
NS 4096
DS 4
SWH 36057.691 Hz
FIDRES 0.551712 Hz
AQ 0.9062698 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028071 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



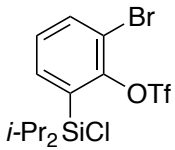
4e

Current Data Parameters
NAME AN2–1712–cr
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180427
Time 23.00
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300148 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4e

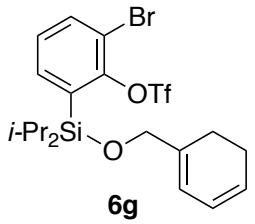
Current Data Parameters
NAME AN2–1712–cr
EXPNO 12
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180428
Time 0.05
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028081 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



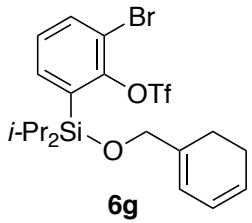
6g

Current Data Parameters
NAME AN2–1402–1
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20170531
Time 20.50
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300144 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



6g

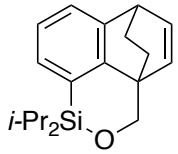
Current Data Parameters
NAME AN2−1402−1
EXPNO 12
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170601
Time 7.43
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65356
SOLVENT CDCl3
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.551712 Hz
AQ 0.9062698 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



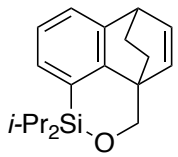
7a

Current Data Parameters
NAME AN2–1470–2
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20170714
Time 20.18
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300156 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7a

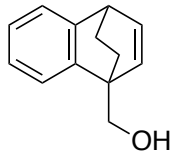
Current Data Parameters
NAME AN2−1406−3
EXPNO 11
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170602
Time 12.21
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028095 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



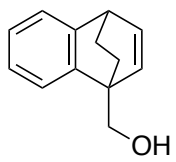
9

Current Data Parameters
NAME AN2−1619−data
EXPNO 11
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171215
Time 4.03
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300178 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



9

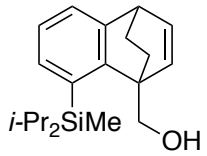
Current Data Parameters
NAME AN2–1619–data
EXPNO 12
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171215
Time 5.45
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65356
SOLVENT CDCl3
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.551712 Hz
AQ 0.9062698 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028099 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



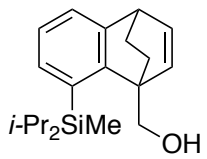
10

Current Data Parameters
NAME AN2−1534−1
EXPNO 13
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170914
Time 11.48
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 17.5
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300193 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



10

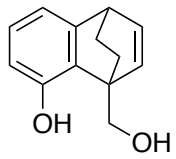
Current Data Parameters
NAME AN2−1534−1
EXPNO 11
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170914
Time 1.02
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

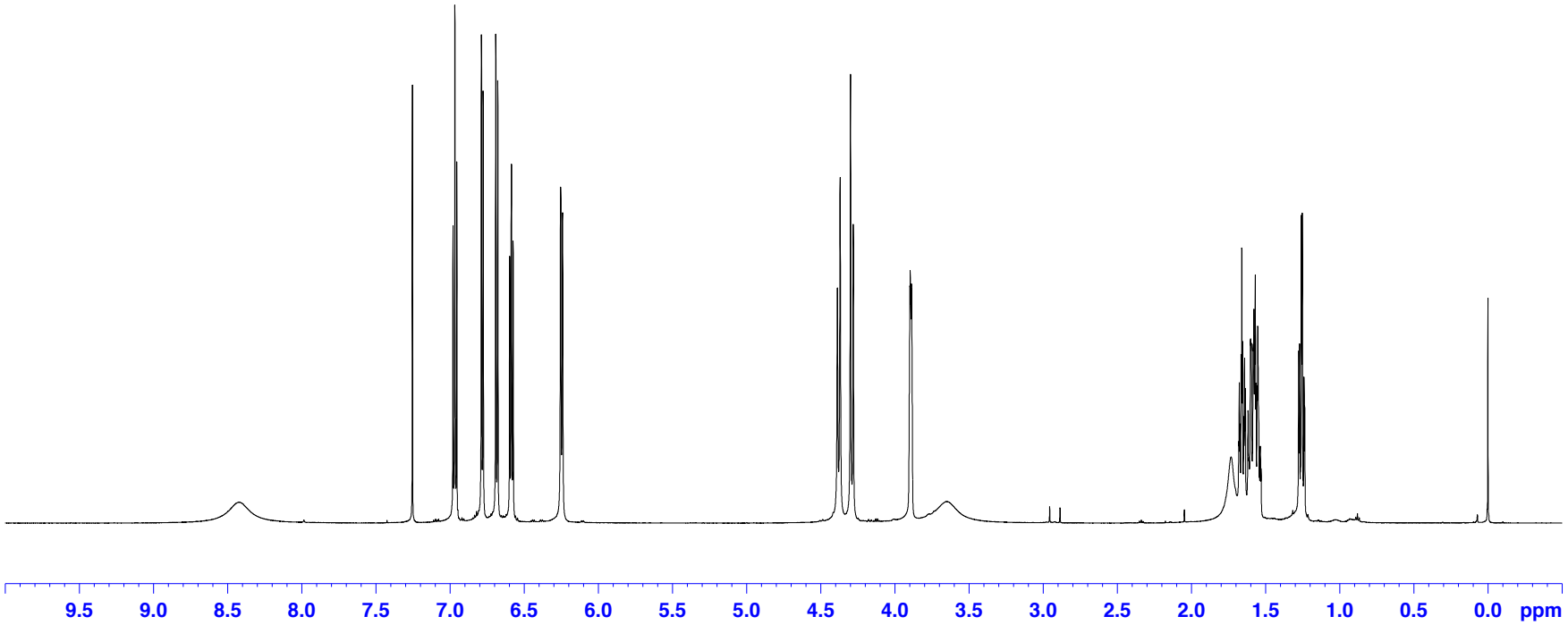
======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028105 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



11

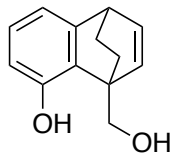


Current Data Parameters
NAME AN2–1532–2
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20170912
Time 14.38
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300188 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



11

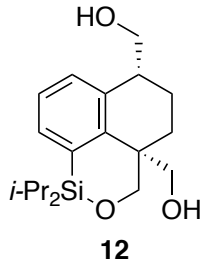
Current Data Parameters
NAME AN2−1532−2
EXPNO 11
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170913
Time 1.02
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028106 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



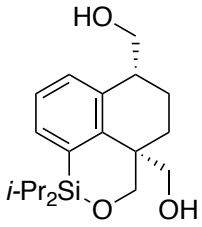
12

Current Data Parameters
NAME AN2–1677–data
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180202
Time 0.02
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 17.5
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



12

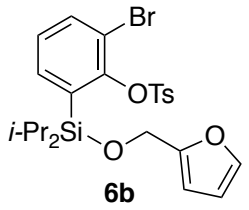
Current Data Parameters
NAME AN2–1677–data
EXPNO 12
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180202
Time 0.54
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028091 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



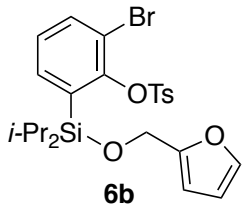
6b

Current Data Parameters
NAME AN2–1492–1
EXPNO 20
PROCNO 1

F2 – Acquisition Parameters
Date_ 20170802
Time 23.33
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300161 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



6b

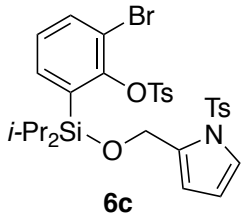
Current Data Parameters
NAME AN2−1492−1
EXPNO 21
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170803
Time 1.45
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



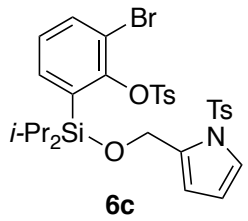
6c

Current Data Parameters
NAME AN2–NTs–precursor–data
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171215
Time 22.59
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 18.96
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



6c

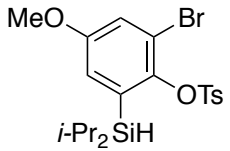
Current Data Parameters
NAME AN2–NTs–precursor–data
EXPNO 12
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171216
Time 1.44
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9026744 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



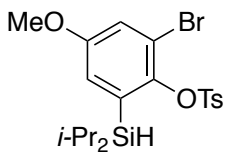
3b

Current Data Parameters
NAME AN2–1578–3–1
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171026
Time 16.16
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300147 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3b

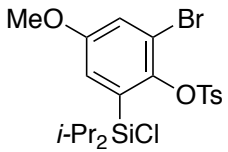
Current Data Parameters
NAME AN2–1578–3–1
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171027
Time 0.53
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028081 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



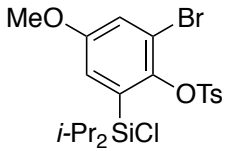
4b

Current Data Parameters
NAME AN2–1586–cr
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171031
Time 22.39
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300156 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4b

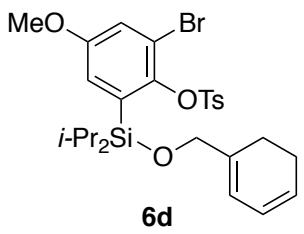
Current Data Parameters
NAME AN2–1586–cr
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171031
Time 23.08
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028095 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



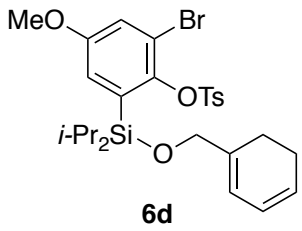
6d

Current Data Parameters
NAME AN2−1587−data
EXPNO 30
PROCNO 1

F2 − Acquisition Parameters
Date_ 20180530
Time 22.40
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 17.5
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



6d

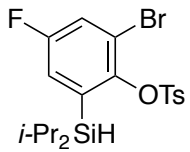
Current Data Parameters
NAME AN2−1587−data
EXPNO 11
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171104
Time 16.59
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028094 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



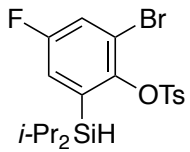
3c

Current Data Parameters
NAME AN2–1562–GPC–2
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171016
Time 21.58
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 18.96
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300147 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3c

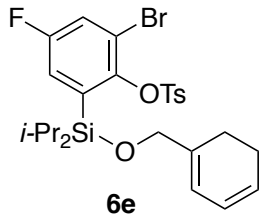
Current Data Parameters
NAME AN2–1562–GPC–2
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171018
Time 1.45
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65356
SOLVENT CDCl3
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.551712 Hz
AQ 0.9062698 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028082 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



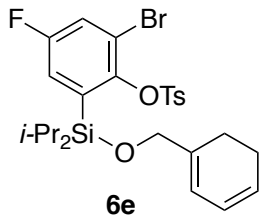
6e

Current Data Parameters
NAME AN2−1604−data
EXPNO 11
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171123
Time 3.05
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300150 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



6e

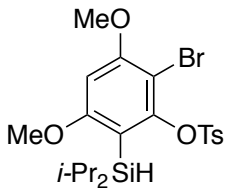
Current Data Parameters
NAME AN2−1604−data
EXPNO 12
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171123
Time 3.56
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028084 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



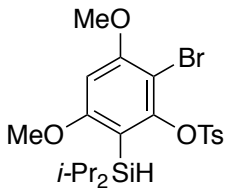
3d

Current Data Parameters
NAME AN2−1572−1
EXPNO 11
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171020
Time 18.07
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300143 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3d

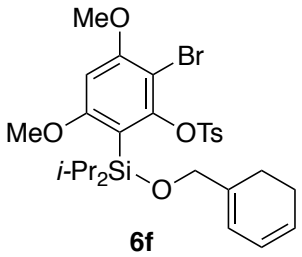
Current Data Parameters
NAME AN2–1572–1
EXPNO 12
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171020
Time 19.49
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65356
SOLVENT CDCl3
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.551712 Hz
AQ 0.9062698 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028081 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



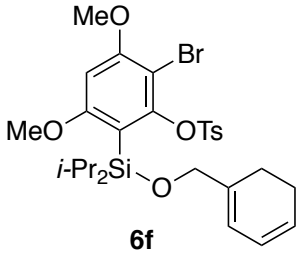
6f

Current Data Parameters
NAME AN2–1602–1–1
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171120
Time 16.17
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 18.96
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300151 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



6f

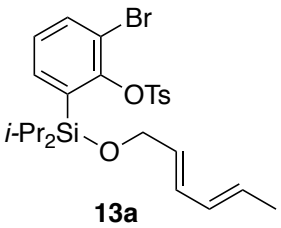
Current Data Parameters
NAME AN2-1602-1-1
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171120
Time 18.29
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 - Processing parameters
SI 32768
SF 150.9028084 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



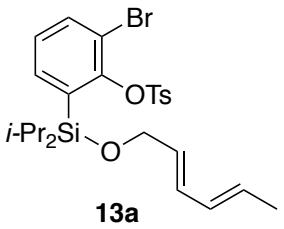
13a

Current Data Parameters
NAME AN2–1504–data
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20170823
Time 20.26
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 293.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



13a

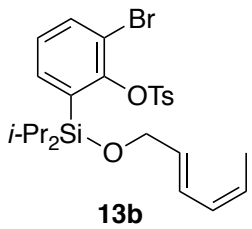
Current Data Parameters
NAME AN2–1504–data
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20170824
Time 1.46
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 293.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



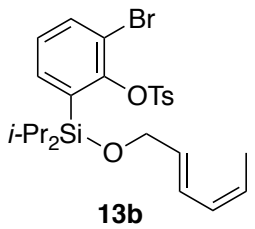
13b

Current Data Parameters
NAME AN2−1579−1
EXPNO 30
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171028
Time 17.18
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 17.5
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



13b

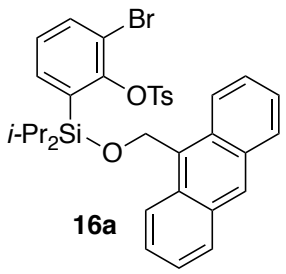
Current Data Parameters
NAME AN2–1579–1
EXPNO 32
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171029
Time 7.12
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9026731 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



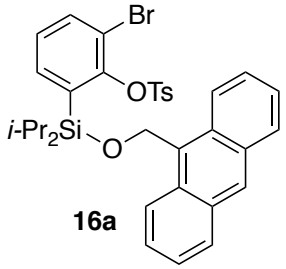
16a

Current Data Parameters
NAME AN2–1524–1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20170831
Time 21.59
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300164 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



16a

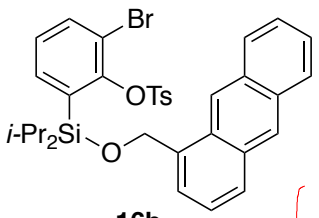
Current Data Parameters
NAME AN2−1524−1
EXPNO 20
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170901
Time 1.45
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65356
SOLVENT CDCl3
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.551712 Hz
AQ 0.9062698 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028081 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



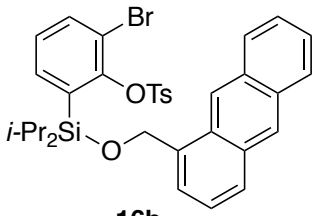
16b

Current Data Parameters
NAME AN2–1606–data
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171125
Time 6.03
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300181 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



16b

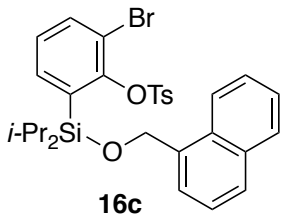
Current Data Parameters
NAME AN2–1606–data
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171125
Time 6.54
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028099 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



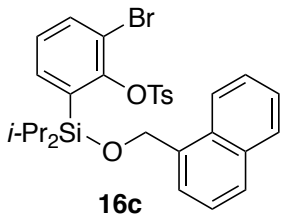
16c

Current Data Parameters
NAME AN2−1605−data
EXPNO 10
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171125
Time 5.03
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



16c

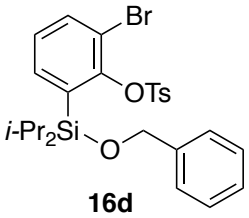
Current Data Parameters
NAME AN2–1605–data
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171125
Time 5.54
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028097 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



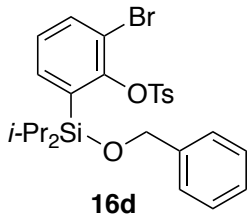
16d

Current Data Parameters
NAME AN2−1613−data
EXPNO 10
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171130
Time 22.24
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



16d

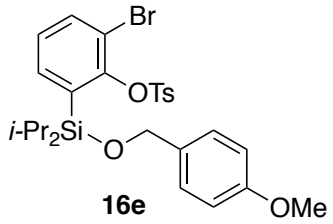
Current Data Parameters
NAME AN2–1613–data
EXPNO 12
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171201
Time 1.29
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028096 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



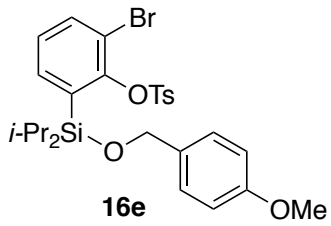
16e

Current Data Parameters
NAME AN2–1630–trit–1
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180925
Time 22.24
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



16e

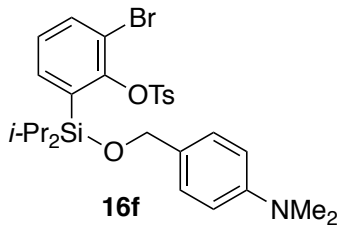
Current Data Parameters
NAME AN2–1630–trit–1
EXPNO 12
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180926
Time 0.56
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9026751 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



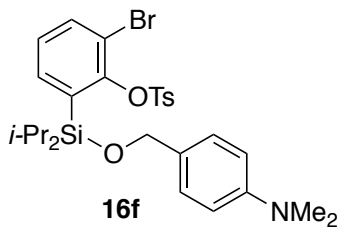
16f

Current Data Parameters
NAME AN2–1821–GPC–1
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180921
Time 15.10
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



16f

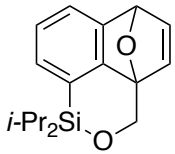
Current Data Parameters
NAME AN2–1821–GPC–1
EXPNO 22
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180923
Time 0.58
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9026749 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



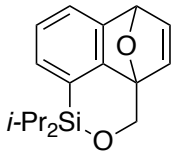
7b

Current Data Parameters
NAME AN2−1497−1
EXPNO 10
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170804
Time 12.42
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 17.5
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300173 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7b

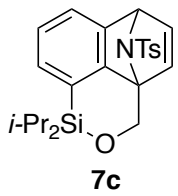
Current Data Parameters
NAME AN2−1497−1
EXPNO 11
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170805
Time 3.00
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028092 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



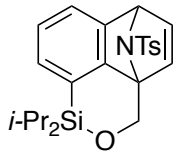
7c

Current Data Parameters
NAME AN2−1530−data
EXPNO 10
PROCNO 1

F2 − Acquisition Parameters
Date_ 20170912
Time 0.59
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 15.79
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300107 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7c

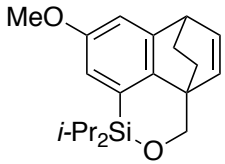
Current Data Parameters
NAME AN2–1530–data
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20170912
Time 1.50
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



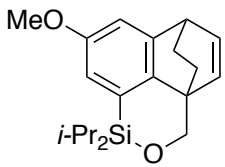
7d

Current Data Parameters
NAME AN2−1596−1
EXPNO 10
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171110
Time 11.15
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 18.96
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300165 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7d

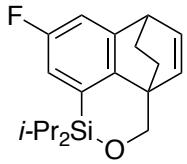
Current Data Parameters
NAME AN2−1596−1
EXPNO 12
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171111
Time 0.54
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028092 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



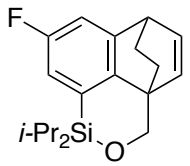
7e

Current Data Parameters
NAME AN2–1608–1
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171122
Time 13.15
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300155 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7e

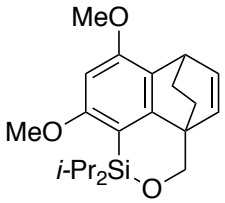
Current Data Parameters
NAME AN2−1608−1
EXPNO 12
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171123
Time 5.59
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028088 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



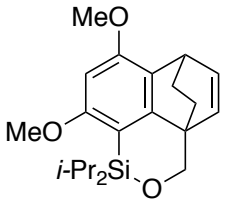
7f

Current Data Parameters
NAME AN2–1607–1
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171123
Time 4.07
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 299.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300159 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7f

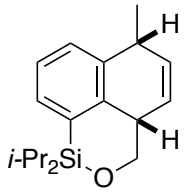
Current Data Parameters
NAME AN2−1607−1
EXPNO 12
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171123
Time 4.58
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028092 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



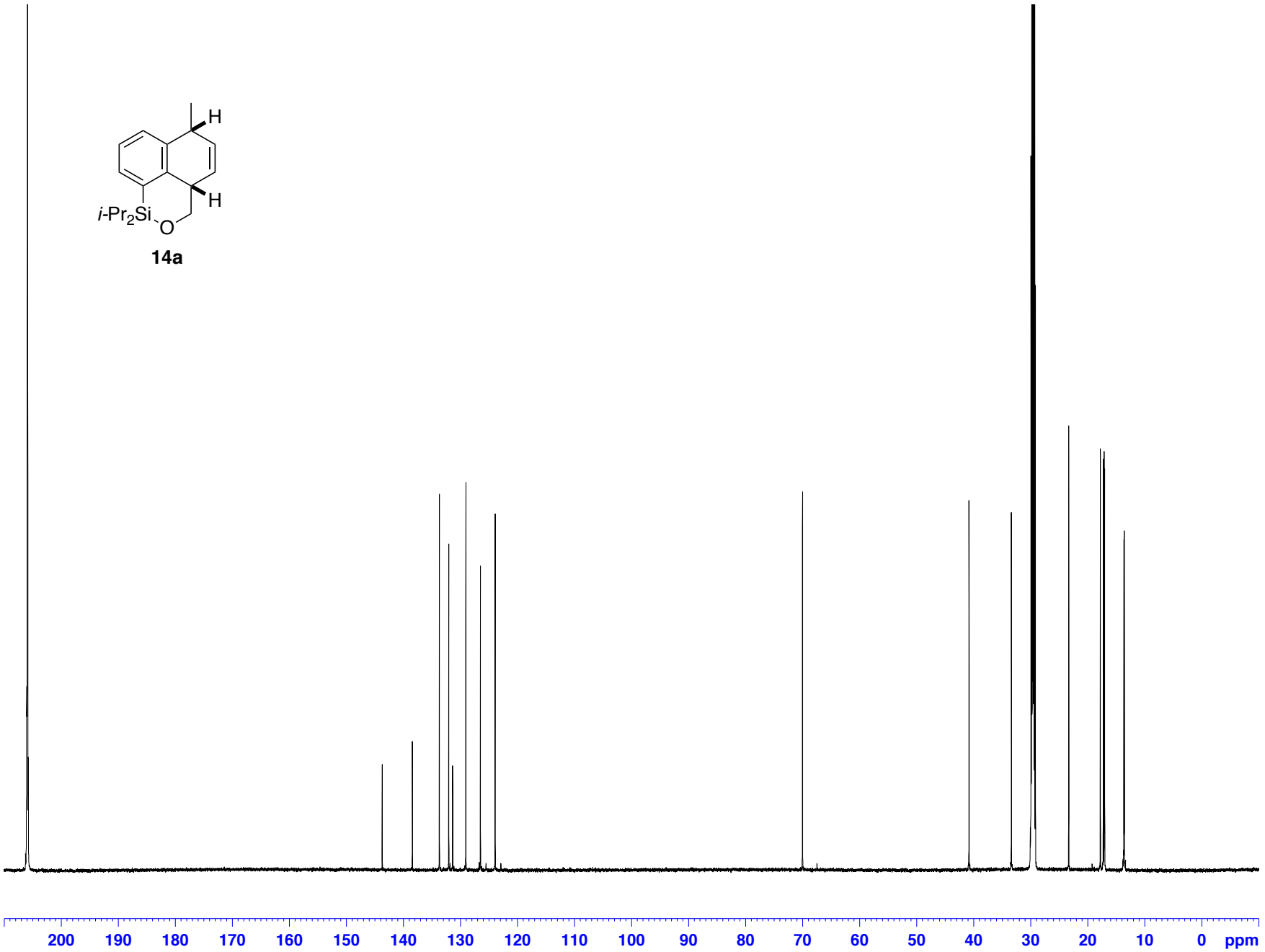
14a

Current Data Parameters
NAME AN2–1717–1
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180516
Time 12.42
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 17.5
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



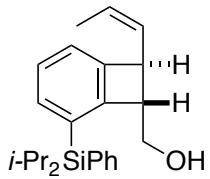
Current Data Parameters
NAME AN2–1507,09–GPC–3
EXPNO 22
PROCNO 1

F2 – Acquisition Parameters
Date_ 20170827
Time 6.14
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9027160 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



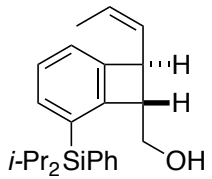
15

Current Data Parameters
NAME AN2–1817–2–1
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180908
Time 17.20
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



15

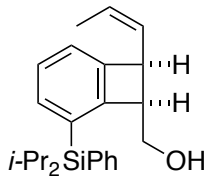
Current Data Parameters
NAME AN2–1817–2–1
EXPNO 22
PROCNO 1

F2 – Acquisition Parameters
Date_ 20180911
Time 0.51
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9026718 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



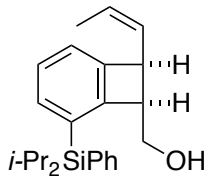
S5

Current Data Parameters
NAME AN2–1857–2–1
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters
Date_ 20181017
Time 14.24
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



S5

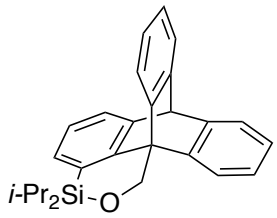
Current Data Parameters
NAME AN2–1857–2–1
EXPNO 22
PROCNO 1

F2 – Acquisition Parameters
Date_ 20181018
Time 7.26
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65356
SOLVENT CDCl3
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.551712 Hz
AQ 0.9062698 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028101 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



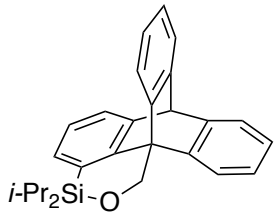
17a

Current Data Parameters
NAME AN2–1531–1–1
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20170912
Time 2.03
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 17.5
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300276 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



17a

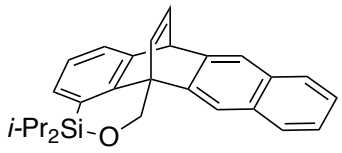
Current Data Parameters
NAME AN2-1531-1-1
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170912
Time 2.54
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

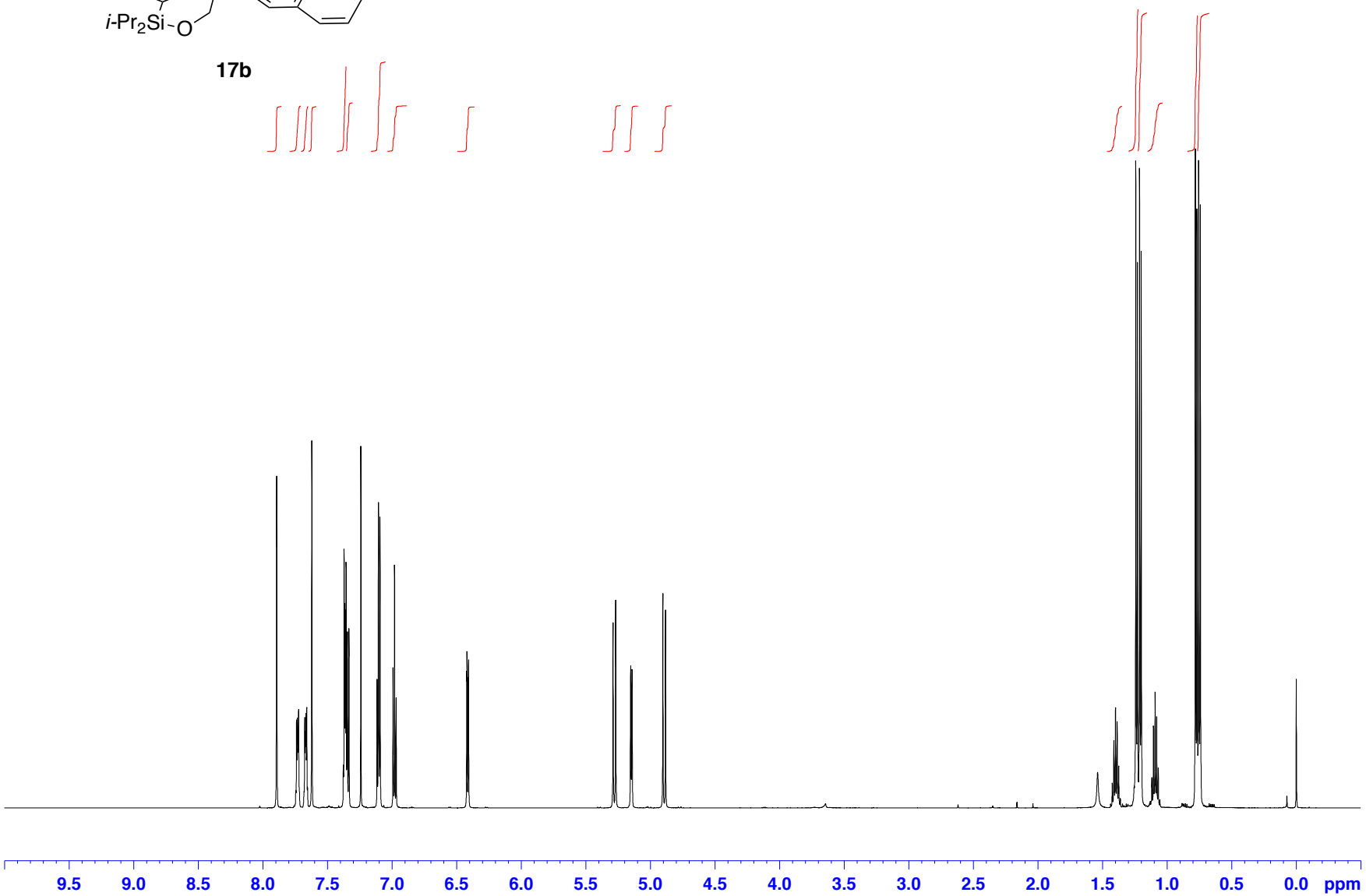
======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 - Processing parameters
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



17b

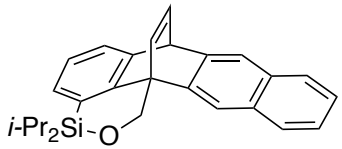


Current Data Parameters
NAME AN2−1610−data
EXPNO 20
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171130
Time 22.20
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 17.5
DW 41.600 usec
DE 10.00 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300255 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



17b

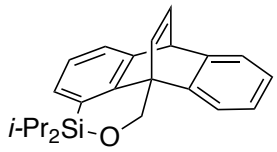
Current Data Parameters
NAME AN2–1610–data
EXPNO 22
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171201
Time 0.28
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 – Processing parameters
SI 32768
SF 150.9028126 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



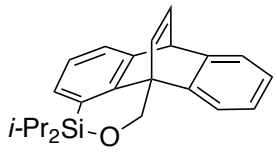
17c

Current Data Parameters
NAME AN2–1609–3
EXPNO 11
PROCNO 1

F2 – Acquisition Parameters
Date_ 20171125
Time 8.33
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 17.5
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 – Processing parameters
SI 65536
SF 600.1300211 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



17c

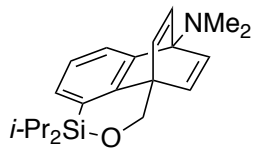
Current Data Parameters
NAME AN2−1609−3
EXPNO 12
PROCNO 1

F2 − Acquisition Parameters
Date_ 20171125
Time 8.59
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028106 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



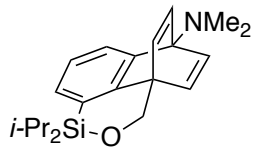
17f

Current Data Parameters
NAME AN2−1822−3
EXPNO 11
PROCNO 1

F2 − Acquisition Parameters
Date_ 20180919
Time 8.08
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 usec
PLW1 23.00000000 W

F2 − Processing parameters
SI 65536
SF 600.1300151 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



17f

Current Data Parameters
NAME AN2−1822−3
EXPNO 12
PROCNO 1

F2 − Acquisition Parameters
Date_ 20180919
Time 8.34
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 14.00000000 W
PLW12 0.64286000 W
PLW13 0.32335001 W

F2 − Processing parameters
SI 32768
SF 150.9028084 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40