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Manuscript Title:

Palladium-catalyzed Silyl C(sp^3)-H Bond Activation

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Contents:

1) General Information

Unless otherwise noted, all starting materials were commercially available and were used without further purification. Solvents were purified by M-Braun SPS-800 Solvent Purification System. *n*-BuLi and PhLi were obtained from Acros. All reactions were carried out under a dry and oxygen-free nitrogen atmosphere in slight positive pressure by using Schlenk techniques.

^{1}H and ^{13}C NMR spectra were recorded on a JEOL JNM-AL300 spectrometer (FT, 300 MHz for ^{1}H; 75 MHz for ^{13}C), or a Bruker ARX400 spectrometer (FT, 400 MHz for ^{1}H; 100 MHz for ^{13}C) at room temperature, unless otherwise noted. High-resolution mass spectra (HRMS) were recorded on a Bruker Apex IV FTMS mass spectrometer using ESI (electrospray ionization). GC analyses were recorded on SHIMADZU GC-2010 spectrometer using FID.

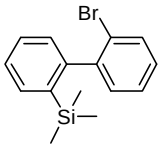
2) Synthesis and Characterization of Starting Materials 1, 3, 5,7 and 10

Preparation of 1a-g:

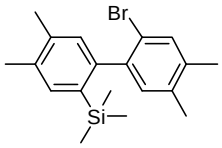
Compounds **1a-g** were prepared according the literature method.[1] To a solution of 4.72 g (20 mmol) of *o*-dibromobenzene in 50 mL of THF was added dropwise, under an atmosphere of nitrogen, 6.25 mL of a 1.6 M solution of *n*-BuLi (10 mmol) in *n*-hexane while the temperature was maintained at -78 °C. After addition, the mixture was warmed to 0 °C and subsequently hydrolyzed with 10 mL of 3 M HCl solution. The organic solvents were removed by rotary evaporation, and the residue was extracted with diethyl ether. The combined filtrates were concentrated under reduced pressure and the crude product was purified by using silicon gel column with petroleum ether as eluent to give the pure product of 2,2'-dibromobiphenyl 2.37 g (76%).

To a solution of 624 mg (2.0 mmol) of 2,2'-dibromobiphenyl in 10 mL of THF at -78 °C was added dropwise, under an atmosphere of argon, 1.3 mL of 1.6 M solution of *n*-BuLi (2.1 mmol) in hexane. After addition, the mixture was stirred for 15 min and 325 mg (3 mmol) of chlorotrialkylsilane was added dropwise. The mixture was warmed to room temperature, a saturated solution of NH_4Cl in water added, and the mixture

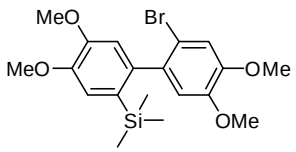
extracted with diethyl ether. The organic fractions were combined, washed (brine), dried (Na_2SO_4), concentrated under reduced pressure and the crude product was purified by using silica gel column with petroleum ether as eluent to give the pure product of (2'-bromobiphenyl-2-yl)trialkylsilane.



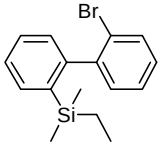
1a:[1] Colorless liquid, isolated yield 85% (518 mg); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.65-7.61 (m, 2H), 7.40-7.37 (m, 2H), 7.33 (d, J = 6.6 Hz, 1H), 7.30-7.13 (m, 3H), -0.01 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.03, 124.32, 126.46, 126.84, 128.28, 128.92, 129.62, 131.56, 132.29, 134.51, 138.35, 144.21, 147.38.



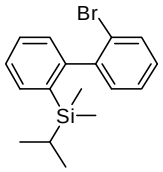
1b: Colorless solid, isolated yield 90% (650 mg); mp: 89.8-91.2 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.39 (s, 1H), 7.37 (s, 1H), 7.00 (s, 1H), 6.94 (s, 1H), 2.33 (s, 3H), 2.29 (s, 3H), 2.28 (s, 3H), 2.22 (s, 3H), 0.00 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.19, 19.09, 19.23, 19.62, 19.64, 120.79, 131.21, 132.82, 133.09, 134.77, 134.79, 135.31, 135.90, 136.79, 137.37, 141.48, 145.27; HRMS (ESI, m/z) calcd for $[C_{19}H_{25}BrSi]H^+$: 361.0982; found 361.0991.



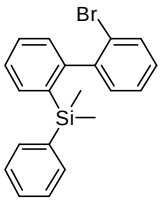
1c: Colorless solid, isolated yield 88% (748 mg); mp: 126.8-127.2 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.06 (s, 1H), 7.04 (s, 1H), 6.76 (s, 1H), 6.66 (s, 1H), 3.91 (s, 3H), 3.90 (s, 3H), 3.83 (s, 3H), 3.82 (s, 3H), 0.01 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.25, 55.70, 55.75, 55.89, 56.14, 113.73, 114.54, 114.67, 114.80, 116.53, 129.66, 136.12, 140.77, 147.28, 147.47, 148.70, 148.74; HRMS (ESI, m/z) calcd for $[C_{19}H_{25}BrO_4Si]H^+$: 425.0778; found 425.0790.



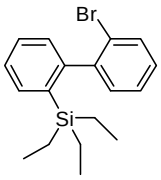
1d: Colorless liquid, isolated yield 87% (555 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.64-7.60 (m, 2H), 7.40-7.37 (m, 2H), 7.32 (t, J = 6.8 Hz, 1H), 7.25-7.20 (m, 2H), 7.14 (d, J = 8.8 Hz, 1H), 0.88-0.83 (m, 3H), 0.54-0.50 (m, 2H), -0.03 (s, 3H), -0.11 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -2.46, 7.54, 8.13, 124.34, 126.44, 126.74, 128.21, 128.88, 129.73, 131.52, 132.33, 134.83, 137.46, 144.38, 147.57.



1e: Colorless liquid, isolated yield 88% (586 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69 (d, *J* = 8.8 Hz, 2H), 7.46-7.43 (m, 2H), 7.37 (t, *J* = 7.4 Hz, 1H), 7.31-7.24 (m, 2H), 7.22 (d, *J* = 9.2 Hz, 1H), 0.96-0.93 (m, 7H), 0.07 (s, 3H), -0.13 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -4.49, -4.46, 13.99, 17.69, 17.73, 124.38, 126.40, 126.62, 128.16, 128.83, 129.77, 131.47, 132.30, 135.09, 136.91, 144.47, 147.60; HRMS (ESI, m/z) calcd for $[C_{17}H_{21}BrSi]Na^+$: 355.0488; found 355.0495.



1f: Colorless liquid, isolated yield 64% (470 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.66 (d, *J* = 8.8 Hz, 1H), 7.59 (d, *J* = 9.2 Hz, 1H), 7.45-7.30 (m, 7H), 7.22-7.14 (m, 3H), 6.99 (d, *J* = 9.2 Hz, 1H), 0.35 (s, 3H), 0.20 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -2.19, -0.99, 124.28, 126.39, 126.80, 127.56, 128.64, 128.72, 128.84, 129.94, 131.67, 132.24, 133.99, 135.61, 136.46, 139.21, 143.85, 147.80; HRMS (ESI, m/z) calcd for $[C_{20}H_{19}BrSi]Na^+$: 389.0332; found 389.0336.

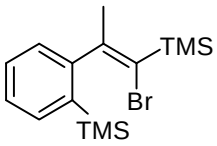


1g: Colorless liquid, isolated yield 82% (569 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.64-7.58 (m, 2H), 7.39-7.36 (m, 2H), 7.30 (t, *J* = 7.2 Hz, 1H), 7.23-7.19 (m, 2H), 7.14 (d, *J* = 8.8 Hz, 1H), 0.82 (t, *J* = 7.6 Hz, 9H), 0.54-0.39 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 3.67, 7.47, 124.21, 126.40, 126.53, 128.06, 128.85, 130.01, 131.40, 132.35, 135,50, 135.56, 144.52, 147.95.

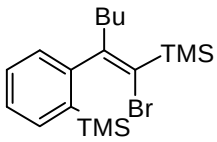
Preparation of 3a and 3b:

To a toluene (100 mL) solution of Cp_2ZrCl_2 (1.75 g, 6 mmol) was added an ether solution of PhLi (6 mL, 2 M, 12 mmol) at 0 °C. After stirring for 2 h, trimethyl(prop-1-ynyl)silane (673 mg, 6 mmol) was added to the mixture at 0 °C. The mixture was warmed to 110 °C and stirred for 9 h. Then, NBS (3.20 g, 18 mmol) and CuCl (653 mg, 6.6 mmol) were added at 0 °C, and the mixture was stirred for 12 h at room temperature. A saturated aqueous solution of $Na_2S_2O_3$ was added, and the mixture was extracted with hexane. The combined extract was washed with brine, dried over Na_2SO_4, filtered, and evaporated. The residue was purified by by silica gel column with petroleum ether as an eluent to afford product (*Z*)-(1-bromo-2-(2-bromophenyl)prop-1-enyl)trimethylsilane (1.41 g, 84%).

To a solution of 750 mg (2.15 mmol) (*Z*)-(1-bromo-2-(2-bromophenyl)prop-1-enyl)trimethylsilane in 10 mL of THF was added dropwise, under an atmosphere of nitrogen, 1.40 mL of 1.6 M solution of *n*-BuLi (2.2 mmol) in *n*-hexane while the temperature was maintained at -78 °C. After addition, the mixture was stirred for 30 min and 282 mg (2.6 mmol) of chlorotrimethylsilane was added dropwise. Then the mixture was warmed to 20 °C, stirred for 1 h and subsequently hydrolyzed with 2 mL of a 3 M HCl solution. The mixture was extracted with with hexane. The combined extract was dried over Na_2SO_4, filtered, and evaporated. The residue was purified by using silicon gel column with hexane as eluent to give the pure product of (*Z*)-(2-(1-bromo-1-(trimethylsilyl)hex-1-en-2-yl)phenyl)trimethylsilane 676 mg (92%).



3a: Colorless oil, isolated yield 92% (676 mg): ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.58-7.55 (m, 1H), 7.38-7.33 (m, 1H), 7.29-7.23 (m, 1H), 6.95-6.92 (m, 1H), 2.14 (s, 3H), 0.35 (s, 9H), 0.28 (s, 9H), ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.59, 0.72, 25.78, 125.36, 126.00, 126.97, 129.09, 135.05, 135.49, 152.37, 153.05; HRMS (ESI, m/z) calcd. for $[C_{15}H_{25}BrSi_2]Na^+$ 353.0570; found 353.0579.



3b: Colorless oil, isolated yield 41% (367 mg); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.60-7.57 (m, 1H), 7.36-7.24 (m, 2H), 6.93-6.90 (m, 1H), 2.74-2.64 (m, 1H), 2.22-2.12 (m, 1H), 1.43-1.19 (m, 4H), 0.85 (t, *J* = 7.05 Hz, 3H), 0.35 (s, 9H), 0.27 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.74, 0.88, 13.95, 22.81, 31.32, 38.75, 125.97, 126.44, 128.25, 128.59, 135.16, 135.79, 150.49, 157.84; HRMS (ESI, m/z) calcd. for $[C_{18}H_{31}BrSi_2]Na^+$ 405.1040; found 405.1050.

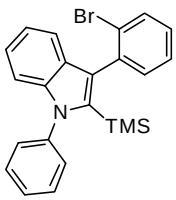
Preparation of 5a-k and **7a-c:**

((2-Bromophenyl)ethynyl)trimethylsilane was prepared according the literature method.[2] Under nitrogen, 1-bromo-2-iodobenzene (5.66 g, 20 mmol), ethynyltrimethylsilane (2.16 g, 22 mmol), $PdCl_2(PPh_3)_2$ (70 mg, 0.5 mol%), CuI (38 mg, 1 mol%) was added in 15 mL THF and 15 mL NEt_3. The reaction mixture was stirred at room temperature until complete consumption of the starting material as monitored by TLC. After the reaction was finished, diethyl ether was poured into the mixture. The mixture was then washed with brine, extracted with diethyl ether, dried with anhydrous

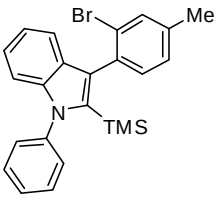
Na_2SO_4, and evaporated under vacuum. The residue was purified by by using silica gel column with petroleum ether as an eluent to afford ((2-bromophenyl)ethynyl) trimethylsilane (5 g, >99%).

(*Z*)-(2-(2-Bromophenyl)-1-iodo-2-(2-iodophenyl)vinyl)trimethylsilane was prepared according literature.[3] To a toluene (100 mL) solution of Cp_2ZrCl_2 (3212 mg, 11 mmol) was added an ether solution of PhLi (11 mL, 2 M, 22 mmol) at 0 °C. After stirring for 2 h, ((2-bromophenyl)ethynyl)trimethylsilane (2530 mg, 10 mmol) was added to the mixture at 0 °C. The mixture was warmed to 100 °C and stirred for 12 h. Then, iodine (10.16 g, 40 mmol) and CuCl (2.08 g, 21 mmol) was added at 0 °C, and the mixture was stirred 12 h at room temperature. A saturated aqueous solution of $Na_2S_2O_3$ was added, and the mixture was extracted with hexane. The combined extract was washed with brine, dried over Na_2SO_4, filtered, and evaporated. The residue was purified by by using SiO_2 column with petroleum ether as an eluent to afford product (*Z*)-(2-(2-bromophenyl)-1-iodo-2-(2-iodophenyl)vinyl)trimethylsilane (5.43 g, 93%).

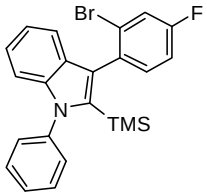
Compounds **5a-k** and **7a-c** were prepared by a modified procedure according the literature.[4] Under nitrogen, $Pd(OAc)_2$ (5 mol%) and Xantphos (10 mol%) was added in 5 mL toluene. After this reaction mixture was stirred at room temperature for 15 min, (*Z*)-(2-(2-bromophenyl)-1-iodo-2-(2-iodophenyl)vinyl)trimethylsilane (1 mmol), amine (1.2 mmol), Cs_2CO_3(2 mmol) were added and this reaction mixture was stirred at 120 °C for 10 h. The reaction mixture was quenched with water and extracted with Et_2O. The extraction was washed with brine and dried over Na_2SO_4. The solvent was then evaporated in vacuo and the residue was purified by using silica gel column with petroleum ether and ethyl acetate as eluent (100:1) to afford the final products.



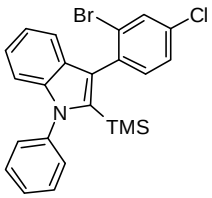
5a: Colorless solid, isolated yield 82% (349 mg); mp: 124.1-124.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.90 (d, *J* = 7.6 Hz, 1H), 7.72-7.63 (m, 6H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.49-7.36 (m, 2H), 7.34-7.27 (m, 3H), 0.01 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.25, 110.24, 119.73, 119.84, 122.75, 126.24, 126.76, 128.07, 128.20, 128.30, 128.35, 128.82, 129.07, 129.15, 132.47, 133.51, 138.03, 140.21, 140.37; HRMS (ESI, m/z) calcd for $[C_{23}H_{22}BrNSi]H^+$: 420.0778; found 420.0772.



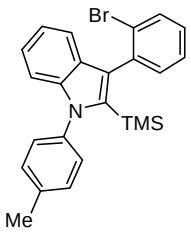
5b: Colorless solid, isolated yield 81% (352 mg); mp: 154.1-155.2 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.53-7.45 (m, 6H), 7.32-7.27 (m, 2H), 7.16-7.07 (m, 4H), 2.40 (s, 3H), -0.19 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.30, 20.89, 110.19, 119.76, 122.68, 125.82, 127.64, 128.11, 128.14, 128.25, 128.94, 129.13, 132.89, 132.92, 133.14, 134.77, 138.04, 138.89, 140.09, 140.39; HRMS (ESI, m/z) calcd for $[C_{24}H_{24}BrNSi]H^+$: 434.0934; found 434.0932.



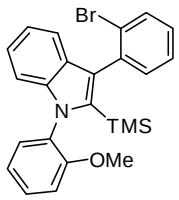
5c: Colorless solid, isolated yield 86% (377 mg); mp: 122.2-123.9 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.71-7.57 (m, 7H), 7.45 (d, J = 7.5 Hz, 1H), 7.36-7.27 (m, 4H), 0.19 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.26, 110.30, 114.01 (d, J = 21.0 Hz), 119.49, 119.79, 119.92, 122.82, 126.26 (d, J = 9.3 Hz), 127.08, 127.99, 128.27, 128.98 (d, J = 8.0 Hz), 129.18, 133.99, 134.07 (d, J = 3.75 Hz), 138.32, 140.11, 140.18, 161.74 (d, J = 248.48 Hz); HRMS (ESI, m/z) calcd for $[C_{23}H_{21}BrFNSi]H^+$: 438.0683; found 438.0682.



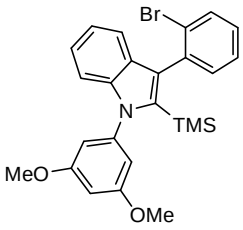
5d: Coloress solid, isolated yield 83% (377 mg); mp: 116.8-118.2 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.73 (s, 1H), 7.53-7.45 (m, 4H), 7.24 (d, J = 8.4 Hz, 2H), 7.20-7.16(m, 3H), 7.15-7.07 (m, 2H), -0.18 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.32, 110.35, 119.49, 119.99, 122.87, 126.55, 126.99, 127.09, 127.83, 128.32, 128.96, 129.02, 129.20, 132.10, 133.72, 134.03, 136.72, 138.28, 140.17; HRMS (ESI, m/z) calcd for $[C_{23}H_{21}BrClNSi]H^+$: 454.0388; found 454.0382.



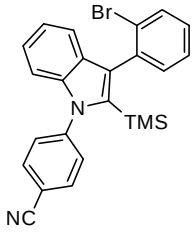
5e: Coloress solid, isolated yield 86% (373 mg); mp: 125.2-126.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.69 (d, J = 7.8 Hz, 1H), 7.43 (d, J = 7.5 Hz, 1H), 7.37-7.20 (m, 7H), 7.15-7.05 (m, 3H), 2.46 (s, 3H), -0.19 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.29, 21.27, 110.29, 119.65, 119.71, 122.61, 126.24, 126.73, 127.97, 128.02, 128.77, 129.71 (2C), 132.42, 133.50, 137.65, 138.05, 138.08, 138.10, 140.23; HRMS (ESI, m/z) calcd for $[C_{24}H_{24}BrNSi]H^+$: 434.0934; found 434.0943.



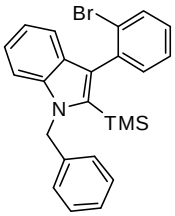
5f: Pale yellow oil, isolated yield 81% (365 mg); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.69 (d, J = 8.1 Hz, 1H), 7.47-7.33 (m, 4H), 7.28-7.21 (m, 2H), 7.14-7.04 (m, 4H), 6.93 (t, J = 3.9 Hz, 1H), 3.71 (s, 3H), -0.21 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -0.13, 55.28, 110.17, 111.58, 119.41, 119.54, 120.38, 122.41, 126.41, 126.61, 126.66, 128.13, 128.60, 128.68, 129.90, 131.53, 132.30, 132.38, 133.65, 138.15, 140.13, 156.54; HRMS (ESI, m/z) calcd for $[C_{24}H_{24}BrNOSi]H^{+}$: 450.0883; found 450.0882.



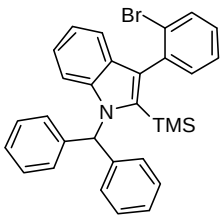
5g: Pale yellow solid, isolated yield 92% (442 mg); mp: 116.2-117.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.69 (d, J = 8.1 Hz, 1H), 7.45-7.33 (m, 2H), 7.29-7.14 (m, 4H), 7.08 (t, J = 7.05 Hz, 1H), 6.71 (s, 1H), 6.58 (s, 2H), 3.82 (s, 6H), -0.12 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.30, 55.52, 100.46, 106.98, 110.38, 119.75, 119.88, 122.79, 126.11, 126.76, 127.94, 128.37, 128.84, 132.46, 133.47, 137.76, 137.94, 139.75, 141.94, 160.94; HRMS (ESI, m/z) calcd for $[C_{25}H_{26}BrNO_2Si]H^{+}$: 480.0989; found 480.0990.



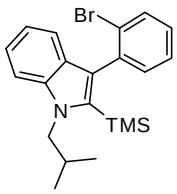
5h: Pale yellow solid, isolated yield 84% (366 mg); mp: 62.5-63.2 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.86 (d, J = 7.5 Hz, 2H), 7.72 (d, J = 7.8 Hz, 1H), 7.63-7.60 (m, 2H), 7.43-7.36 (m, 2H), 7.31-7.29 (m, 2H), 7.26-7.10 (m, 3H), -0.17 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.38, 100.46, 109.68, 111.65, 118.20, 120.19, 120.61, 123.48, 125.88, 126.91, 128.36, 129.15, 129.43, 130.18, 132.61, 133.28, 137.28, 137.63, 139.57, 144.69; HRMS (ESI, m/z) calcd for $[C_{24}H_{21}BrN_2Si]H^{+}$: 445.0730; found 445.0721.



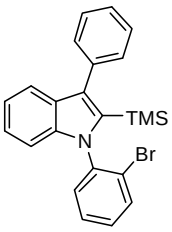
5i: Colorless solid, isolated yield 79% (343 mg); mp: 83.8-83.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.66 (d, J = 7.8 Hz, 1H), 7.40-7.29 (m, 2H), 7.26-7.17 (m, 5H), 7.10 (d, J = 3.6 Hz, 2H), 7.03-7.01 (m, 1H), 6.92 (d, J = 7.5 Hz, 2H), 5.55 (s, 2H), 0.01 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.23, 49.68, 109.86, 119.56, 119.66, 122.62, 125.67, 126.68, 127.08, 128.63, 128.72, 128.78, 128.90. 132.25, 133.37, 137.05, 138.15, 138.25, 138.27, 138.75; HRMS (ESI, m/z) calcd for $[C_{24}H_{24}BrNSi]H^{+}$: 434.0934; found 434.0943.



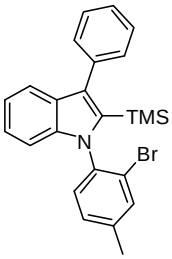
5j: Pale yellow solid, isolated yield 74% (377 mg); mp: 128.6-129.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.68 (d, J = 9.2 Hz, 1H), 7.43 (d, J = 9.2 Hz, 1H), 7.37-7.23 (m, 10H), 7.16-7.11 (m, 4H), 6.93 (d, J = 7.4 Hz, 1H), 6.83 (d, J = 8.2 Hz, 1H), 6.65 (d, J = 8.4 Hz, 1H), 0.03 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.37, 65.20, 113.16, 119.19, 119.55, 122.09, 126.70, 126.84, 127.43, 127.54, 128.06, 128.27, 128.46, 128.77, 129.95, 132,24, 133.27, 138.22, 138.24, 139.41; HRMS (ESI, m/z) calcd for $[C_{24}H_{24}BrNSi]H^{+}$: 510.1247; found 510.1255.



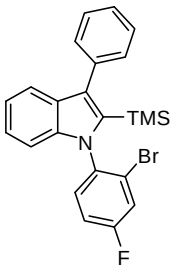
5k: Colorless solid, isolated yield 83% (332 mg); mp: 98.0-98.6 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.66 (d, J = 8.1 Hz, 1H), 7.39-7.28 (m, 3H), 7.22-7.17 (m, 3H), 7.03 (t, J = 7.5 Hz, 1H), 4.08 (t, J = 8.4 Hz, 2H), 2.39-2.30 (m, 1H), 0.94 (d, J = 6.6 Hz, 3H), 0.88 (d, J = 6.6 Hz, 3H), 0.13 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 1.07, 20.05, 20.29, 29.74, 53.39, 110.27, 119.02, 119.64, 122.00, 126.59, 126.61, 126.97, 128.49, 128.66, 132.25, 133.48, 136.75, 138.24, 138.60; HRMS (ESI, m/z) calcd for $[C_{21}H_{26}BrNSi]H$+: 400.1091; found 400.1082.



7a: Yellow oil, isolated yield 72% (301 mg); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.76 (d, J = 7.5 Hz, 1H), 7.52-7.36 (m, 9H), 7.20-7.07 (m, 2H), 6.89 (d, J = 7.8 Hz, 1H), -0.17 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.54, 110.45, 119.51, 120.08, 122.96, 124.61, 126.86, 127.93, 128.12, 128.69, 130.03, 130.13, 131.02, 131.63, 133.53, 136.57, 136.93, 139.54, 139.93; HRMS (ESI, m/z) calcd. for $[C_{23}H_{22}BrNSi]H^{+}$: 420.0777; found 420.0785.



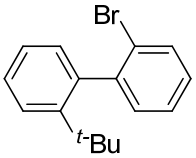
7b: Yellow oil, isolated yield 68% (295 mg); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.58-7.52 (m, 1H), 7.49-7.24 (m, 7H), 7.16-7.09 (m, 3H), 6.89 (d, J = 7.5 Hz, 1H), 2.43 (s, 3H), -0.15 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.59, 20.94, 110.46, 119.44, 119.97, 122.86, 124.15, 126.79, 127.89, 128.65, 128.83, 129.91, 131.03, 131.12, 133.84, 136.67, 136.99, 137.18, 139.65, 140.42; HRMS (ESI, m/z) calcd. for $[C_{24}H_{24}BrNSi]H^{+}$: 434.0934; found 434.0940.



7c:Yellow oil, isolated yield 76% (332 mg); ^{1}H NMR (300 MHz, $CDCl_3$): δ: 7.50-7.38 (m, 8H), 7.21-7.10 (m, 3H), 6.89-6.84 (m, 1H), -0.12 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.59, 110.19, 115.24 (d, J = 24.1 Hz), 119.59, 120.25, 120.67 (d, J = 25.3 Hz), 123.13, 125.28 (d, J = 9.9 Hz), 126.94, 127.95, 128.78, 130.35, 130.98, 132.41 (d, J = 7.5 Hz), 136.28, 136.42, 136.90, 139.67, 161.97 (d, J = 251 Hz); HRMS (ESI, m/z) calcd. for $[C_{23}H_{22}BrFNSi]H^{+}$: 438.0683; found 438.0686.

Preparation of 10:

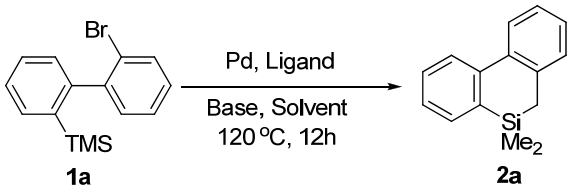
1-(*tert*-Butyl)-2-iodobenzene was prepared according the literature method.[5] Under nitrogen, 1-(*tert*-butyl)-2-iodobenzene (2 mmol), 2-bromobenzeneboronic acid (2.4 mmol), $Pd(PPh_3)_4$ (5 mol%) and K_2CO_3 (6 mmol) were added in 10 mL ethanol and this reaction mixture was stirred at 90 °C for 8 h. The reaction mixture was quenched with water and extracted with Et_2O. The extraction was washed with brine and dried over Na_2SO_4. The solvent was then evaporated in vacuo and the residue was purified by using silica gel column with petroleum ether to afford the product 2-bromo-2'-*tert*-butyldibromobiphenyl.



Colorless liquid, isolated yield 51% (291 mg); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.59-7.66 (m, 2H), 7.24-7.40 (m, 5H), 6.96-6.99 (d, J = 7.5 Hz, 1H), 1.25 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 32.17, 36.53, 124.93, 125.16, 126.08, 127.34, 127.72, 128.40, 131.82, 132.10, 132.29, 139.98, 145.53, 147.21.

3) Reaction Condition Optimization of the Pd-catalyzed Reaction of 1a

STable 1. Reaction Condition Optimization of the Pd-Catalyzed Reaction of **1a**[a]



1a **2a**

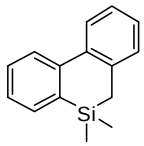
Entry	Pd (5%)	Ligand	Base	Solvent	Yield[b]

1	$Pd(OAc)_2$	P*t*-Bu_3	NaO*t*-Bu	Dioxane	45
2	$[Pd(\pi\text{-allyl})Cl]_2$	P*t*-Bu_3	NaO*t*-Bu	Dioxane	45
3	$Pd_2(dba)_3$	P*t*-Bu_3	NaO*t*-Bu	Dioxane	13
4	$Pd(PPh_3)_4$	P*t*-Bu_3	NaO*t*-Bu	Dioxane	75 (64)
5	$PdBr_2$	P*t*-Bu_3	NaO*t*-Bu	Dioxane	68
6	$Pd(PPh_3)_4$	PCy_3	NaO*t*-Bu	Dioxane	41
7	$Pd(PPh_3)_4$	X-phose	NaO*t*-Bu	Dioxane	56
8	$Pd(PPh_3)_4$	DPPF	NaO*t*-Bu	Dioxane	11
9	$Pd(PPh_3)_4$	Xantphos	NaO*t*-Bu	Dioxane	16
10	$Pd(PPh_3)_4$	Ph_3P	NaO*t*-Bu	Dioxane	28
11	$Pd(PPh_3)_4$	$(2\text{-furan})_3P$	NaO*t*-Bu	Dioxane	51
12	$Pd(PPh_3)_4$	DPPP	NaO*t*-Bu	Dioxane	Trace
13	$Pd(PPh_3)_4$	DPE-phos	NaO*t*-Bu	Dioxane	53
14	$Pd(PPh_3)_4$		NaO*t*-Bu	Dioxane	63
15	$Pd(PPh_3)_4$	P*t*-Bu_3	LiO*t*-Bu	Dioxane	9
16	$Pd(PPh_3)_4$	P*t*-Bu_3	KO*t*-Bu	Dioxane	Trace
17	$Pd(PPh_3)_4$	P*t*-Bu_3	Cs_2CO_3	Dioxane	Trace
18	$Pd(PPh_3)_4$	P*t*-Bu_3	K_2CO_3	Dioxane	Trace
19	$Pd(PPh_3)_4$	P*t*-Bu_3	CsF	Dioxane	Trace
20	$Pd(PPh_3)_4$	P*t*-Bu_3	NEt_3	Dioxane	Trace
21	$Pd(PPh_3)_4$	P*t*-Bu_3	TBAF	Dioxane	Trace
22	$Pd(PPh_3)_4$	P*t*-Bu_3	KOH	Dioxane	Trace
23	$Pd(PPh_3)_4$	P*t*-Bu_3	NaOAc	Dioxane	Trace
24	$Pd(PPh_3)_4$	P*t*-Bu_3	NaO*t*-Bu	Toluene	77 (70)
25	$Pd(PPh_3)_4$	P*t*-Bu_3	NaO*t*-Bu	DMF	Trace
26	$Pd(PPh_3)_4$	P*t*-Bu_3	NaO*t*-Bu	CH_3CN	Trace
27	$Pd(PPh_3)_4$	P*t*-Bu_3	NaO*t*-Bu	NMP	Trace
28	$Pd(PPh_3)_4$	P*t*-Bu_3	NaO*t*-Bu	DME	Trace
29[c]	$Pd(PPh_3)_4$	P*t*-Bu_3	NaO*t*-Bu	Toluene	83 (74)[c]

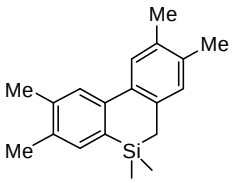
[a] Conditions: **1a** (0.3 mmol), Pd catalyst (5 mol%), ligand (10 mol%), base (0.9 mmol), solvent (2 mL), 120 °C, 12 h. [b] GC yield (*n*-$C_{12}H_{26}$ as internal standard), isolated yield in parenthesis. [c] with TBAB (20 mol%).

4) Typical Procedure for the Preparation and Characterization of 2, 4, 6 and 8

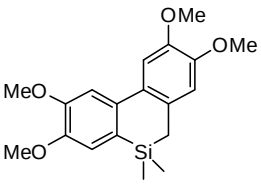
Under the protection of nitrogen, $Pd(PPh_3)_4$ (5 mol%), P*t*-Bu_3 (10 mol%) and NaO*t*-Bu (0.9 mmol), were added in 2 mL of toluene. After this reaction mixture was stirred at room temperature for 15 min, **1**, **3**, **5**, or **7** (0.3 mmol), TBAB (20 mol%) were added and this reaction mixture was stirred at 120 °C for 12 h. The reaction mixture was quenched with water and extracted with Et_2O. The extraction was washed with brine and dried over $MgSO_4$. The solvent was then evaporated in vacuo and the residue was purified by using silicon gel column with petroleum ether and ethyl acetate as eluent to afford the final products.



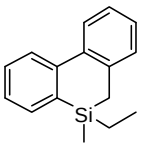
2a: Colorless oil, isolated yield 74% (50 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.68 (d, *J* = 8.0 Hz, 1H), 7.61 (d, *J* = 7.6 Hz, 1H), 7.54 (d, *J* = 8.4 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 1H), 7.30 (t, *J* = 7.2 Hz, 1H), 7.26-7.16 (m, 3H), 2.17 (s, 2H), 0.21 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -4.35 (2C), 21.25, 126.11 (2C), 126.56, 127.45, 127.93, 130.19, 131.21, 132.52, 135.48, 136.17, 137.55, 144.44; HRMS (ESI, m/z) calcd for $[C_{15}H_{16}Si]H^+$: 225.1094; found 225.1093.



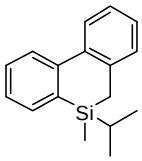
2b: Colorless solid, isolated yield 58% (49 mg); mp: 94.2-94.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.48 (s, 1H), 7.37 (s, 1H), 7.27 (s, 1H), 6.94 (s, 1H), 2.32 (s, 3H), 2.29 (s, 3H), 2.27 (s, 3H), 2.23 (s, 3H), 2.06 (s, 2H), 0.19 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -4.09, 19.26, 19.31, 19.45, 20.12, 20.60, 127.12, 128.80, 132.29, 132.70, 133.07, 133.78, 133.88, 134.52, 134.96, 135.32, 138.48, 142.32; HRMS (ESI, m/z) calcd for $[C_{19}H_{24}Si]H^+$: 281.1720; found 281.1716.



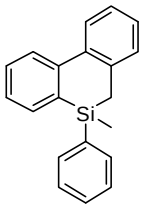
2c: Colorless solid, isolated yield 42% (43 mg); mp: 155.2-155.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.16 (s, 1H), 7.09 (s, 1H), 6.99 (s, 1H), 6.71 (s, 1H), 3.97 (s, 3H), 3.94 (s, 3H), 3.93 (s, 3H), 3.91 (s, 3H), 2.06 (s, 2H), 0.21 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -4.16 , 20.70, 55.78, 55.89, 55.95, 56.19, 109.63, 111.31, 114.43, 114.82, 126.46, 128.35, 129.77, 137.95, 147.05, 147.51, 147.71, 150.51; HRMS (ESI, m/z) calcd for $[C_{19}H_{24}O_4Si]H^+$: 345.1517; found 345.1522.



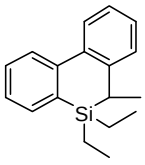
2d: Colorless oil, isolated yield 69% (49 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.68 (d, *J* = 7.6 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 1H), 7.51 (d, *J* = 7.2 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 1H), 7.28 (t, *J* = 7.2 Hz, 1H), 7.24-7.13 (m, 3H), 2.20-2.12 (m, 2H), 0.89 (t, *J* = 7.8 Hz, 3H), 0.70-0.64 (m, 2H), 0.21 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -6.46, 4.37, 7.33, 19.50, 126.09, 126.14, 126.47, 127.43, 127.94, 130.17, 131.16, 132.81, 134.66, 136.18, 137.68, 144.70; HRMS (ESI, m/z) calcd for $[C_{16}H_{18}Si]H^+$: 239.1251; found 239.1247.



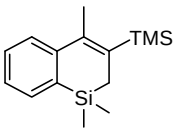
2e: Colorless oil, isolated yield 79% (60 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69 (d, *J* = 8.0 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 7.52 (d, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 1H), 7.28 (t, *J* = 7.4 Hz, 1H), 7.23-7.14 (m, 3H), 2.22-2.14 (m, 2H), 0.90-0.87 (m, 7H), 0.23 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -8.21, 11.37, 17.39, 17.54, 18.64, 126.09, 126.16, 126.42, 127.46, 127.96, 130.14, 131.08, 133.22, 134.21, 136.22, 137.87, 144.90; HRMS (ESI, m/z) calcd for $[C_{17}H_{20}Si]H^+$: 253.1407; found 253.1407.



2f: Colorless oil, isolated yield 53% (46 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.74 (d, *J* = 7.6 Hz, 1H), 7.63 (d, *J* = 8.0 Hz, 1H), 7.50-7.43 (m, 4H), 7.35-7.14 (m, 7H), 2.54-2.32 (m, 2H), 0.51 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -5.90, 20.55, 126.17, 126.34, 126.62, 127.61, 127.82, 127.98, 129.55, 130.51, 131.45, 133.63, 133.77, 134.44, 135.22, 135.49, 137.67, 144.90; HRMS (ESI, m/z) calcd for $[C_{20}H_{18}Si]H^+$: 287.1251; found 287.1251.

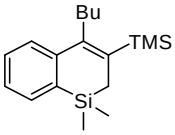


2g: Colorless oil, isolated yield 41% (33 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67 (d, *J* = 7.6 Hz, 1H), 7.56 (d, *J* = 9.6 Hz, 1H), 7.50-7.43 (m, 2H), 7.29-7.21 (m, 4H), 2.34-2.28 (m, 1H), 1.21 (d, *J* = 7.2 Hz, 3H), 0.97 (t, *J* = 7.8 Hz, 3H), 0.85 (d, *J* = 7.8 Hz, 3H), 0.81-0.65 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 0.91, 2.21, 7.35, 7.42, 14.65, 21.82, 126.05, 126.08, 126.46, 127.74, 128.30, 128.67, 130.13, 132.80, 133.62, 137.46, 141.95, 144.73; HRMS (ESI, m/z) calcd for $[C_{18}H_{22}Si]H^+$: 267.1564; found 267.1558.

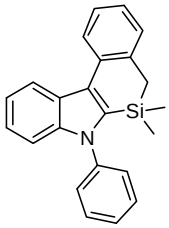


4a: Colorless oil, isolated yield 46% (36 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (d, *J* = 7.2 Hz, 1H), 7.37-7.35 (m, 2H), 7.20-7.17 (m, 1H), 2.23 (s, 3H), 1.52 (s, 2H), 0.23 (s, 9H), 0.21 (s, 6H); ^{13}C NMR (75 MHz,

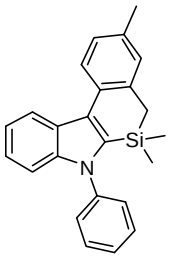
$CDCl_3$) δ: -4.46, 0.66, 17.07, 23.01, 124.84, 126.02, 129.53, 131.73, 133.07, 135.61, 141.17, 145.91; HRMS (ESI, m/z) calcd for $[C_{15}H_{24}Si_2]H^+$: 259.1333; found 259.1330.



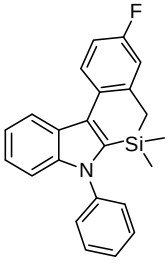
4b: Colorless oil, isolated yield 76% (69 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44 (d, *J* = 6.8 Hz, 1H), 7.37-7.35 (m, 2H), 7.20-7.16 (m, 1H), 2.66 (t, *J* = 7.4 Hz, 2H), 1.48 (s, 2H), 1.32-1.26 (m, 4H), 0.86 (t, *J* = 6.8 Hz, 3H), 0.23 (s, 9H), 0.21 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -4.61, 0.88, 14.08, 16.99, 22.91, 32.49, 35.26, 124.84, 125.90, 129.47, 131.91, 134.81, 136.76, 144.15, 146.87; HRMS (ESI, m/z) calcd for $[C_{18}H_{30}Si_2]H^+$: 303.1959; found 303.1967.



6a: Pale yellow solid, isolated yield 91% (93 mg); mp: 110.5-111.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.18-8.15 (m, 1H), 7.99 (d, *J* = 7.6 Hz, 1H), 7.50-7.46 (m, 2H), 7.42-7.38 (m, 3H), 7.34-7.32 (m, 1H), 7.29-7.20 (m, 4H), 7.08 (t, *J* = 7.4 Hz, 1H), 2.20 (s, 2H), -0.04 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -3.55, 22.64, 110.63, 120.42, 120.54, 122.79, 125.01, 125.53, 125.70, 126.15, 126.29, 127.21, 127.72, 129.34, 131.07, 134.49, 134.60, 138.05, 140.26, 140.34; HRMS (ESI, m/z) calcd for $[C_{23}H_{21}NSi]H^+$: 340.1516; found 340.1520.

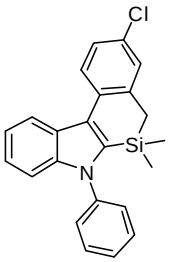


6b: Colorless solid, isolated yield 86% (91 mg); mp: 133.8-134.9 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.16-8.13 (m, 1H), 7.88 (d, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.4 Hz, 2H), 7.42-7.40 (m, 3H), 7.33-7.31 (m, 1H), 7.21-7.19 (m, 2H), 7.09 (d, *J* = 7.6 Hz, 2H), 2.33 (s, 3H), 2.16 (s, 2H), -0.04 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -3.48, 21.06, 22.56, 110.58, 120.41, 120.45, 122.71, 125.58, 126.17, 126.33, 127.20, 127.64, 128.36, 129.32, 131.71, 132.00, 134.38, 134.45, 137.57, 140.29, 140.32; HRMS (ESI, m/z) calcd for $[C_{24}H_{24}NSi]H^+$: 354.1673; found 354.1676.

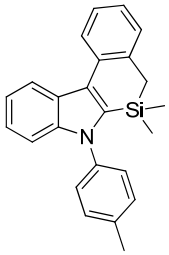


6c: Colorless solid, isolated yield 80% (86 mg); mp: 155.2-156.0 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.09 (d, *J* = 9.2 Hz, 1H), 7.92 (d, *J* = 8.4 Hz, 1H), 7.51 (t, *J* = 7.2 Hz, 2H), 7.45-7.41 (m, 3H), 7.35-7.31 (m, 1H), 7.23-7.21 (m, 2H), 6.98 (d, *J* = 8.8 Hz, 2H), 2.18 (s, 2H), -0.03 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -3.63, 22.98, 110.72, 112.14 (d, *J* = 20.4 Hz), 117.75 (d, *J* = 20.4 Hz), 120.10, 120.63, 122.93, 124.80, 125.96, 127.18, 127.32 (d, *J* = 32.1 Hz), 127.8, 129.41, 130.67 (d, *J* = 3.1 Hz), 137.21 (d, *J* = 7.4

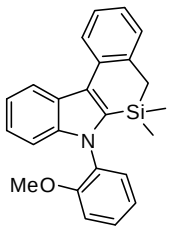
Hz), 137.44, 140.19, 140.26, 160.24 (d, J = 242.3 Hz); HRMS (ESI, m/z) calcd for $[C_{23}H_{21}FNSi]H^+$: 358.1422; found 358.1429.



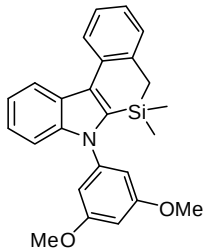
6d: Pale yellow solid, isolated yield 61% (68 mg); mp: 156.7-158.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.10-8.08 (m, 1H), 7.89 (d, J = 9.2 Hz, 1H), 7.53 (t, J = 7.4 Hz, 2H), 7.48-7.42 (m, 3H), 7.35-7.33 (m, 1H), 7.26-7.22 (m, 4H), 2.18 (s, 2H), -0.02 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -3.60, 22.70, 110.79, 120.15, 120.77, 123.02, 124.59, 125.68, 125.95, 127.27 (2C), 127.93, 129.45, 130.00, 130.77, 133.19, 136.67, 138.11, 140.15, 140.40; HRMS (ESI, m/z) calcd for $[C_{23}H_{21}ClNSi]H^+$: 374.1126; found 374.1121.



6e: Colorless solid, isolated yield 88% (93 mg); mp: 165.8-167.0 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.18 (d, J = 8.8 Hz, 1H), 8.01 (d, J = 8.4 Hz, 1H), 7.34-7.27 (m, 7H), 7.23 (t, J = 5.4 Hz, 2H), 7.11 (t, J = 7.4 Hz, 1H), 2.46 (s, 3H), 2.23 (s, 2H), 0.03 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -3.47, 21.19, 22.67, 110.71, 120.39, 120.43, 122.68, 124.93, 125.24, 125.68, 126.06, 126.26, 127.04, 129.92, 131.08, 134.50, 134.71, 137.63 (2C), 138.19, 140.49; HRMS (ESI, m/z) calcd for $[C_{24}H_{23}NSi]H^+$: 354.1673; found 354.1675.

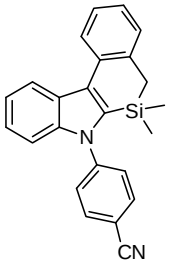


6f: Pale yellow solid, isolated yield 85% (94 mg); mp: 118.2-119.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.16 (d, J = 6.8 Hz, 1H), 8.02 (d, J = 7.6 Hz, 1H), 7.40 (t, J = 8.0 Hz, 1H), 7.30-7.22 (m, 3H), 7.20-7.17 (m, 2H), 7.08-7.01 (m, 4H), 3.66 (s, 3H), 2.25-2.16 (m, 2H), -0.04 (s, 3H), -0.13 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -4.56, -3.56, 22.66, 55.42, 110.98, 111.89, 120.22, 120.33, 120.60, 122.45, 124.74, 124.91, 125.61, 125.79, 126.15, 128.54, 129.64, 129.94, 131.09, 134.54, 134.88, 138.77, 140.83, 155.80; HRMS (ESI, m/z) calcd for $[C_{24}H_{23}NOSi]H^+$: 370.1622; found 370.1623.

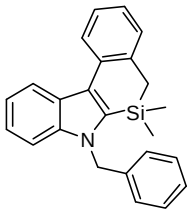


6g: Yellow oil, isolated yield 87% (104 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.15 (d, J = 8.8 Hz, 1H), 7.97 (d, J = 7.6 Hz, 1H), 7.45-7.42 (m, 1H), 7.29-7.20 (m, 4H), 7.08 (d, J = 7.4 Hz, 1H), 6.61 (s, 2H), 6.54 (s, 1H), 3.79 (s, 6H), 2.21 (s, 2H), 0.06 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -3.49, 22.75, 55.53, 99.91, 105.44 (2C), 110.86, 120.43, 120.59, 122.85, 125.07, 125.58, 125.70, 126.21, 126.32, 131.07, 134.56,

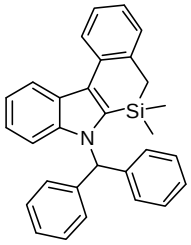
137.80, 140.08, 141.92, 161.21; HRMS (ESI, m/z) calcd for $[C_{25}H_{25}NO_2Si]H^+$: 400.1727; found 400.1728.



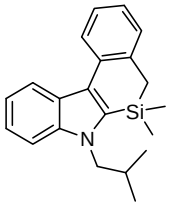
6h: Colorless solid, isolated yield 71% (78 mg); mp: 238.9-239.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.17-8.14 (m, 1H), 7.94 (d, *J* = 7.6 Hz, 1H), 7.83 (d, *J* = 8.8 Hz, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.39-7.35 (m, 1H), 7.32-7.25 (m, 4H), 7.14 (t, *J* = 7.4 Hz, 1H), 2.21 (s, 2H), 0.02 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -3.36, 22.42, 110.13, 110.92, 118.17, 120.71, 121.32, 123.51, 125.59, 125.83, 126.48, 126.75, 127.38, 127.44, 131.08, 133.43, 133.91, 134.41, 137.14, 139.65, 144.33; HRMS (ESI, m/z) calcd for $[C_{24}H_{20}N_2Si]H^+$: 365.1469; found 365.1469.



6i: Pale yellow solid, isolated yield 64% (68 mg); mp: 109.4-110.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.19-8.16 (m, 1H), 8.02 (d, *J* = 7.6 Hz, 1H), 7.27-7.16 (m, 8H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.92 (d, *J* = 6.4 Hz, 1H), 5.41 (s, 2H), 2.23 (s, 2H), 0.11 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -3.25, 22.45, 49.74, 109.96, 120.13, 120.77, 122.59, 124.66, 124.77, 125.71, 125.87, 126.09, 126.13, 127.33, 128.67, 131.08, 134.03, 134.73, 137.65, 138.01, 140.10; HRMS (ESI, m/z) calcd for $[C_{24}H_{24}NSi]H^+$: 354.1673; found 354.1671.

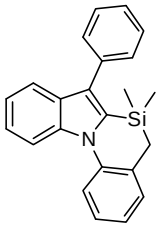


6j: Pale yellow solid, isolated yield 94% (121 mg); mp: 129.7-130.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.14 (d, *J* = 8.0 Hz, 1H), 8.00 (d, *J* = 7.2 Hz, 1H), 7.31-7.23 (m, 8H), 7.17 (t, *J* = 3.8 Hz, 4H), 7.10-7.04 (m, 2H), 7.00 (s, 1H), 6.90 (t, *J* = 7.6 Hz, 1H), 6.72 (d, *J* = 8.8 Hz, 1H), 2.27 (s, 2H), 0.17 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -2.81, 22.58, 66.57, 113.26, 119.83, 120.72, 122.08, 124.82, 124.88, 125.65, 126.32, 127.41, 127.73, 128.14, 128.56, 130.99, 134.01, 134.65, 139.17, 139.21, 139.51; HRMS (ESI, m/z) calcd for $[C_{30}H_{28}NSi]H^+$: 430.1986; found 430.1988.

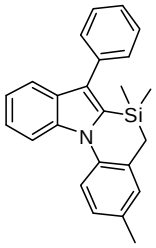


6k: Pale yellow oil, isolated yield 51% (49 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.12 (d, *J* = 8.0 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 1H), 7.37 (d, *J* = 8.4 Hz, 1H), 7.25-7.21 (m, 3H), 7.16 (t, *J* = 7.6 Hz, 1H), 7.06 (t, *J* = 7.4 Hz, 1H), 4.01 (d, *J* = 7.6 Hz, 2H), 2.28-2.17 (m, 3H), 0.93 (d, *J* = 6.4 Hz, 6H), 0.36 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -2.75, 20.41, 22.64, 30.30, 53.97, 110.23, 119.76, 120.56, 122.15, 124.14, 124.67, 125.67, 125.98, 126.25,

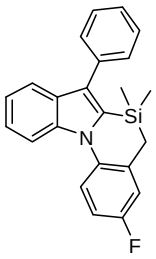
130.88, 134.12, 134.85, 137.14, 139.90; HRMS (ESI, m/z) calcd for $[C_{21}H_{26}NSi]H^+$: 320.1829; found 320.1831.



8a: Yellow solid, isolated yield 57% (58 mg); mp: 129.1-130.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.88 (d, *J* = 8.4 Hz, 1H), 7.78 (d, *J* = 7.6 Hz, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.54 (d, *J* = 6.8 Hz, 2H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.35 (t, *J* = 7.4 Hz, 1H), 7.30-7.26 (m, 3H), 7.17 (t, *J* = 7.0 Hz, 1H), 7.12 (t, *J* = 7.4 Hz, 1H), 2.13 (s, 2H), 0.04 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -3.37, 20.36, 111.76, 119.84, 120.60, 121.52, 123.16, 124.83, 125.98, 126.81, 128.34, 128.53, 128.67, 129.76, 130.44, 131.56, 134.95, 136.01, 137.67, 137.83; HRMS (ESI, m/z) calcd for $[C_{23}H_{22}NSi]H^+$: 340.1516; found 340.1519.



8b: Pale yellow oil, isolated yield 84% (89 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.86 (d, *J* = 8.4 Hz, 1H), 7.78 (d, *J* = 7.6 Hz, 1H), 7.63 (d, *J* = 8.0 Hz, 1H), 7.54 (d, *J* = 7.6 Hz, 2H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.36 (d, *J* = 7.6 Hz, 1H), 7.28 (t, *J* = 7.0 Hz, 1H), 7.16 (t, *J* = 7.4 Hz, 1H), 7.11 (s, 1H), 7.08 (d, *J* = 8.0 Hz, 1H), 2.36 (s, 3H), 2.09 (s, 2H), 0.04 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -3.33, 20.26, 20.89, 111.73, 119.79, 120.42, 121.35, 123.05, 126.52, 126.75, 128.25, 128.32, 128.51, 129.78, 130.17, 132.21, 134.35, 135.01, 135.39, 136.11, 137.65; HRMS (ESI, m/z) calcd for $[C_{24}H_{24}NSi]H^+$: 354.1673; found 354.1675.



8c: Yellow solid, isolated yield 68% (73 mg); mp: 135.6-136.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.82-7.77 (m, 2H), 7.70-7.67 (m, 1H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.37 (d, *J* = 7.6 Hz, 1H), 7.29 (t, *J* = 7.6 Hz, 1H), 7.22-7.16 (m, 1H), 7.03-6.95 (m, 2H), 2.12 (s, 2H), 0.05 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: -3.45, 20.76, 111.35, 112.44 (d, *J* = 21.6 Hz), 117.87 (d, *J* = 21.6 Hz), 119.94, 120.66, 122.67 (d, *J* = 8.7 Hz), 123.32, 126.90, 128.38, 128.49, 128.67, 129.71, 133.12 (d, *J* = 8.0 Hz), 133.92 (d, *J* = 2.5 Hz), 134.60, 135.85, 137.66, 159.46 (d, *J* = 242.3 Hz); HRMS (ESI, m/z) calcd for $[C_{24}H_{24}FNSi]H^+$: 358.1422; found 358.1427.

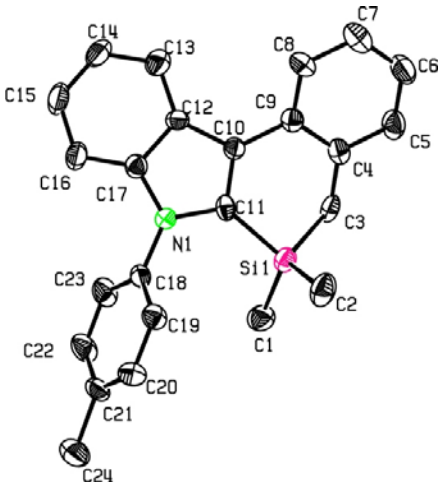
5) X-ray Crystallographic Studies of 6e and 8a

The single crystals of **6e** suitable for X-ray analysis were grown in mixed solvent of hexane, diethyl ether and ethyl acetate. Data collections for **6e** were performed at 20 °C on a Rigaku RAXIS RAPID IP diffractometer, using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The determination of crystal class and unit cell parameters was carried out by the Rapid-AUTO (Rigaku 2000) program package for **6e**. The raw frame data were processed using Crystal Structure (Rigaku/MSC 2000) for **6e** to yield the reflection data file. The structure of **6e** was solved by use of SHELXTL program. Refinement was performed on F2 anisotropically for all the non-hydrogen atoms by the full-matrix least-squares method. The hydrogen atoms were placed at the calculated positions and were included in the structure calculation without further refinement of the parameters. Crystal data, data collection and processing parameters for compounds **6e** are summarized in **STable 2**. Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-823538 (**6e**). Copies of these data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

STable 2. Crystal data and structure refinement for **6e**.[6]

Identification code	a	
Empirical formula	C24 H23 N Si	
Formula weight	353.52	
Temperature	298(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.3619(19) Å	α= 87.11(3) °
	b = 10.083(2) Å	β= 82.02(3) °
	c = 10.655(2) Å	γ = 88.24(3) °
Volume	994.5(3) Å^3	
Z	2	
Density (calculated)	1.181 Mg/m^3	
Absorption coefficient	0.125 mm^{-1}	
F(000)	376	
Crystal size	0.30 x 0.30 x 0.20 mm^3	
Theta range for data collection	2.72 to 27.48°.	
Index ranges	-12<=h<=12, -13<=k<=13, -13<=l<=13	
Reflections collected	6104	
Independent reflections	4195 [R(int) = 0.1614]	
Completeness to theta = 27.48°	92.0 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9755 and 0.9636	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4195 / 0 / 239	
Goodness-of-fit on F^2	0.996	

Final R indices [I>2sigma(I)]	R1 = 0.0664, wR2 = 0.1178
R indices (all data)	R1 = 0.1855, wR2 = 0.1398
Extinction coefficient	0.043(3)
Largest diff. peak and hole	0.303 and -0.336 e.$^{-3}$

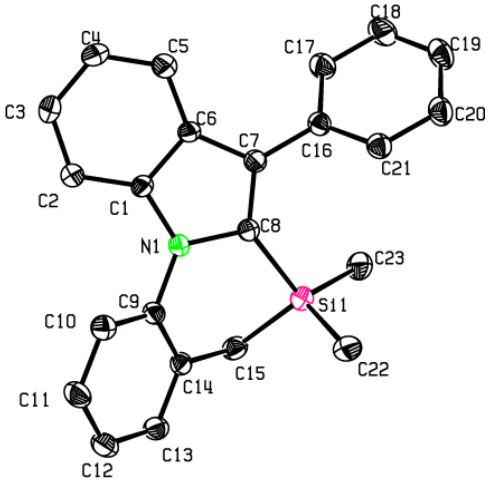


SFigure 1. ORTEP drawing of **6e** with 30% probability thermal ellipsoids. Hydrogen atoms have been omitted for clarity.

The single crystals of **8a** suitable for X-ray analysis were grown in mixed solvent of hexane, diethyl ether and ethyl acetate. Data collections for **8a** were performed at 20 °C on a Rigaku RAXIS RAPID IP diffractometer, using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The determination of crystal class and unit cell parameters was carried out by the Rapid-AUTO (Rigaku 2000) program package for **8a**. The raw frame data were processed using Crystal Structure (Rigaku/MSC 2000) for **8a** to yield the reflection data file. The structures of **8a** were solved by use of SHELXTL program. Refinement was performed on F2 anisotropically for all the non-hydrogen atoms by the full-matrix least-squares method. The hydrogen atoms were placed at the calculated positions and were included in the structure calculation without further refinement of the parameters. Crystal data, data collection and processing parameters for compounds **8a** are summarized in **STable 3**. Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-824123 (**8a**). Copies of these data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

STable 3. Crystal data and structure refinement for **8a**.[6]

Identification code	ly	
Empirical formula	C23 H21 N Si	
Formula weight	339.50	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 13.268(3) Å	α= 90°
	b = 10.072(2) Å	β= 91.60(3) °
	c = 13.822(3) Å	γ = 90°
Volume	1846.4(6) Å^3	
Z	4	
Density (calculated)	1.221 Mg/m^3	
Absorption coefficient	0.132 mm^{-1}	
F(000)	720	
Crystal size	0.30 x 0.30 x 0.20 mm^3	
Theta range for data collection	2.10 to 27.48°.	
Index ranges	-17<=h<=17, -12<=k<=13, -17<=l<=17	
Reflections collected	7929	
Independent reflections	4229 [R(int) = 0.0480]	
Completeness to theta = 27.48°	99.9 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9742 and 0.9616	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4229 / 0 / 229	
Goodness-of-fit on F^2	1.004	
Final R indices [I>2sigma(I)]	R1 = 0.0484, wR2 = 0.0865	
R indices (all data)	R1 = 0.0965, wR2 = 0.0899	
Extinction coefficient	0.0276(10)	
Largest diff. peak and hole	0.274 and -0.202 e. Å $^{-3}$	

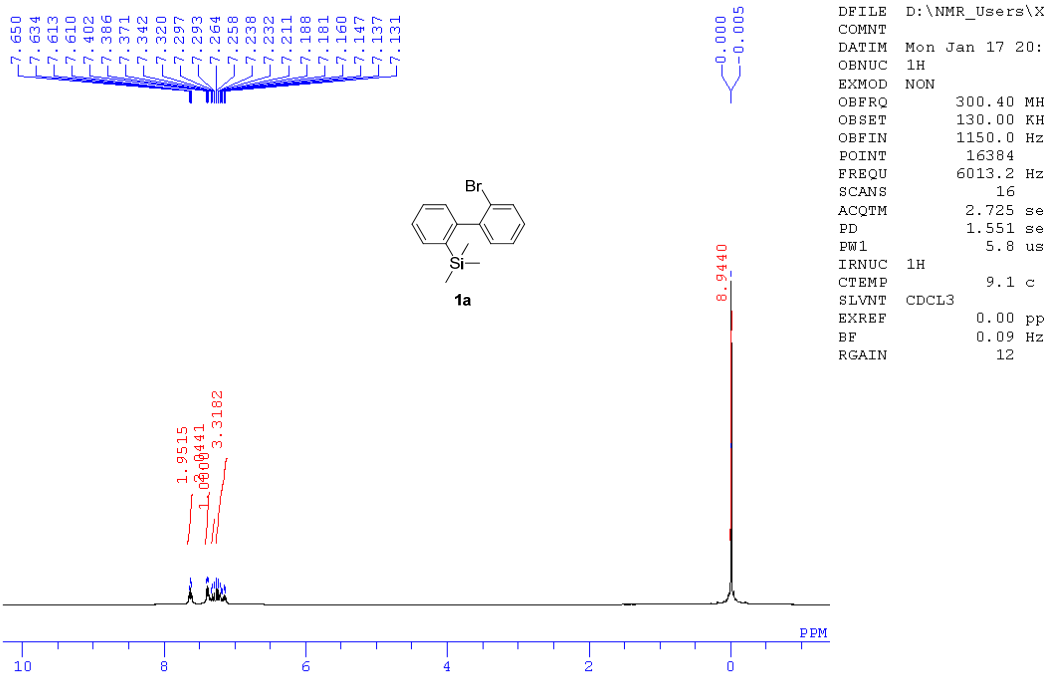


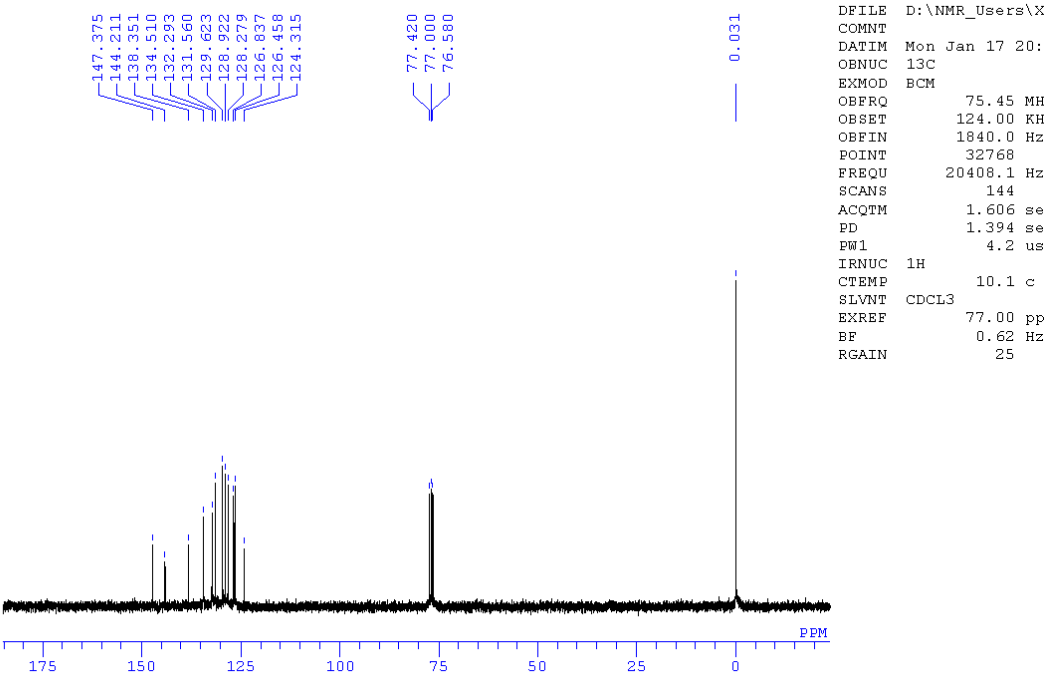
SFigure 2. ORTEP drawing of **8a** with 30% probability thermal ellipsoids. Hydrogen atoms have been omitted for clarity.

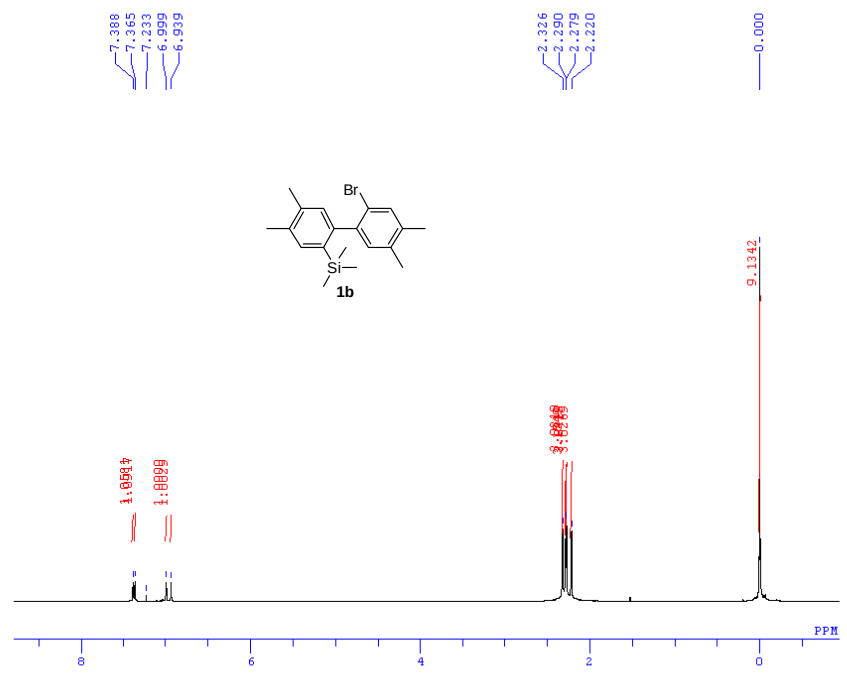
References:

1. G. P. M. van Klink, H. J. R. de Boer, G. Schat, O. S. Akkerman, F. Bickelhaupt and A. L. Spek, *Organometallics,* 2002, **21**, 2119.
2. K. Sonogashira, Y. Tohda and N. Hagihara, *Tetrahedron Lett.,* 1975, **44**, 4467.
3. H.-J. Zhang, Z. Song, C. Wang, C. Bruneau and Z. Xi, *Tetrahedron Lett*., 2008, **49**, 624.
4. a) M. C. Willis, G. N. Brace and I. P. Holmes, *Angew. Chem., Int. Ed.,* 2005, **44**, 403. b) M. C. Willis, G. N. Brace, T. J. K. Findlay and I. P. Holmes, *Ad*v. *Synth. Catal.,* 2006, **348**, 851. c) A. J. Fletcher, M. N. Bax and M. C. Willis, *Chem. Commun.,* 2007, 4764.
5. Z. Gan, K. Kawamura, K. Eda and M. Hayashi, *J. Organomet. Chem.,* 2010, **695**, 2022.
6. G. M. Sheldrick, SHELXTL 5.10 for Windows NT: *Structure Determination Software Programs*; Bruker Analytical X-ray Systems, Inc.: Madison, WI, **1997**.

6) Scanned ^{1}H NMR and ^{13}C NMR Spectra of All New Compounds





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RGAIN           15
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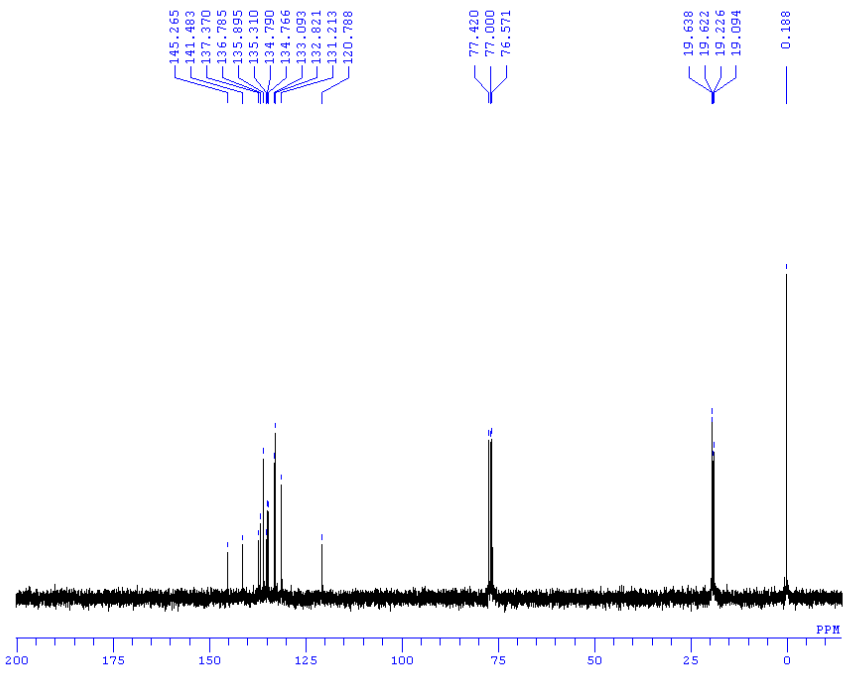


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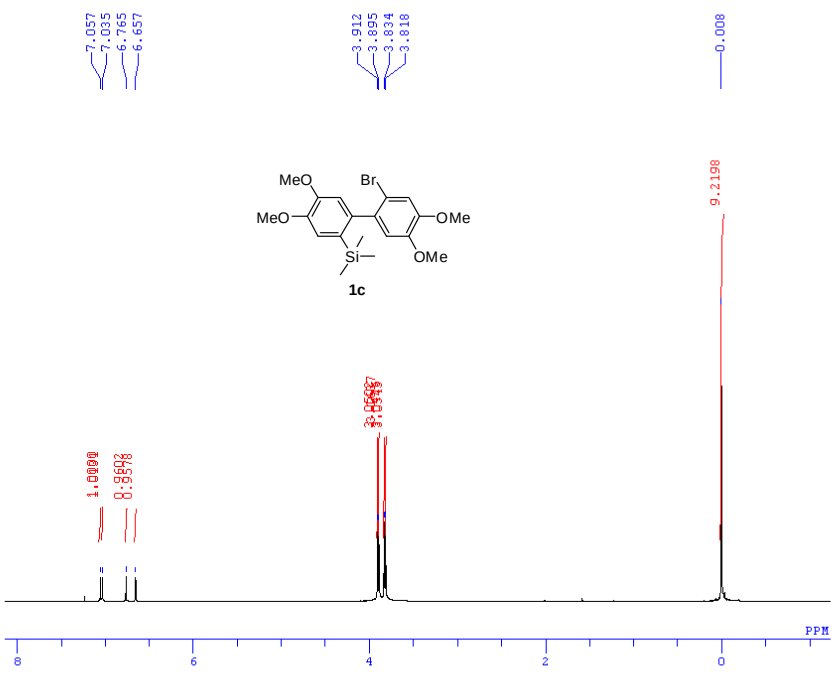


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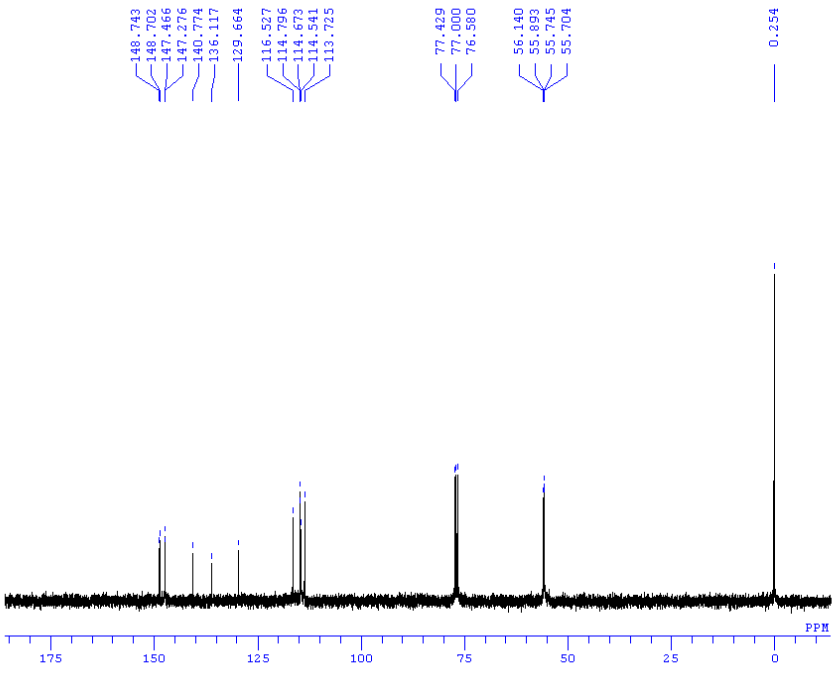


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RGAIN           26
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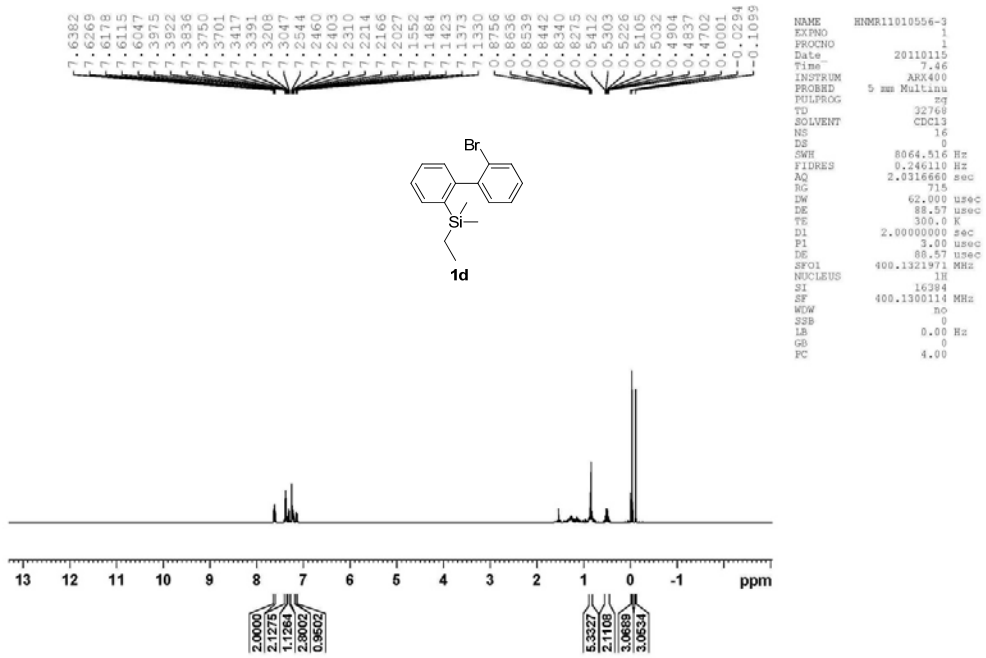
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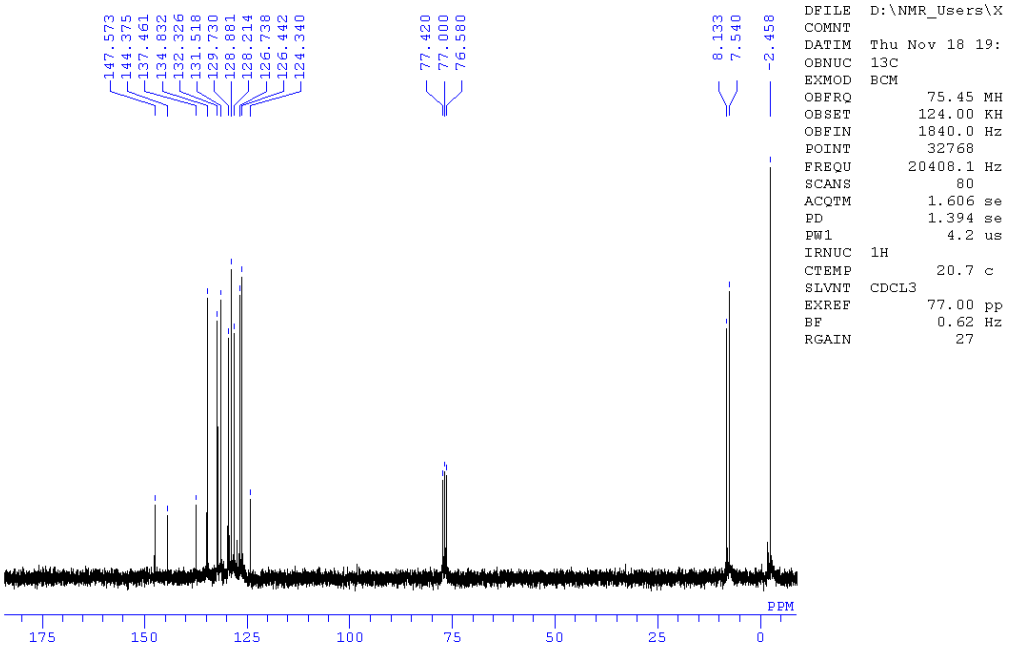
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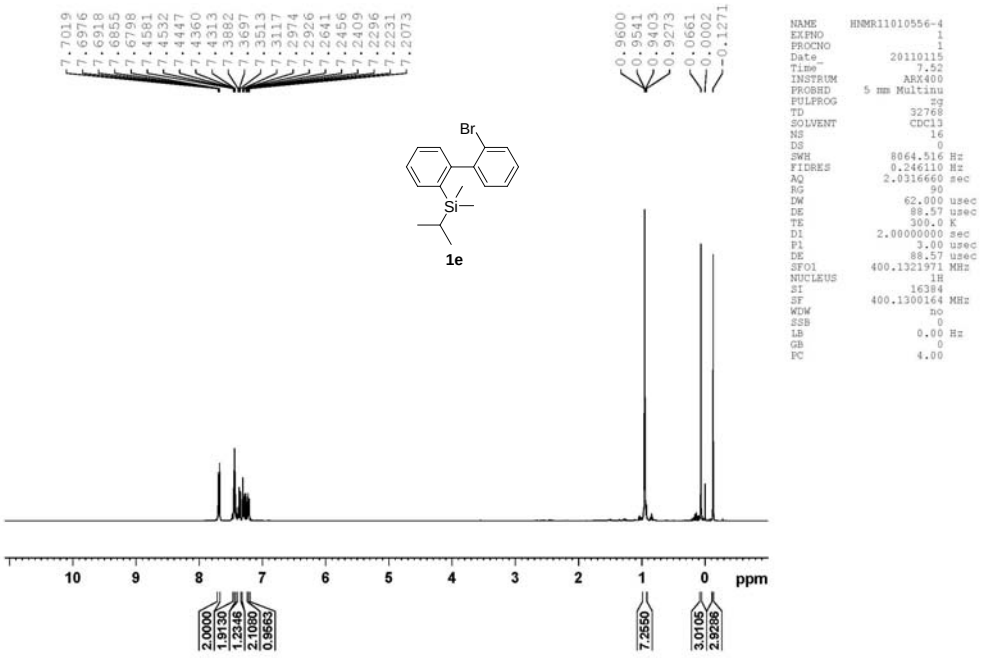

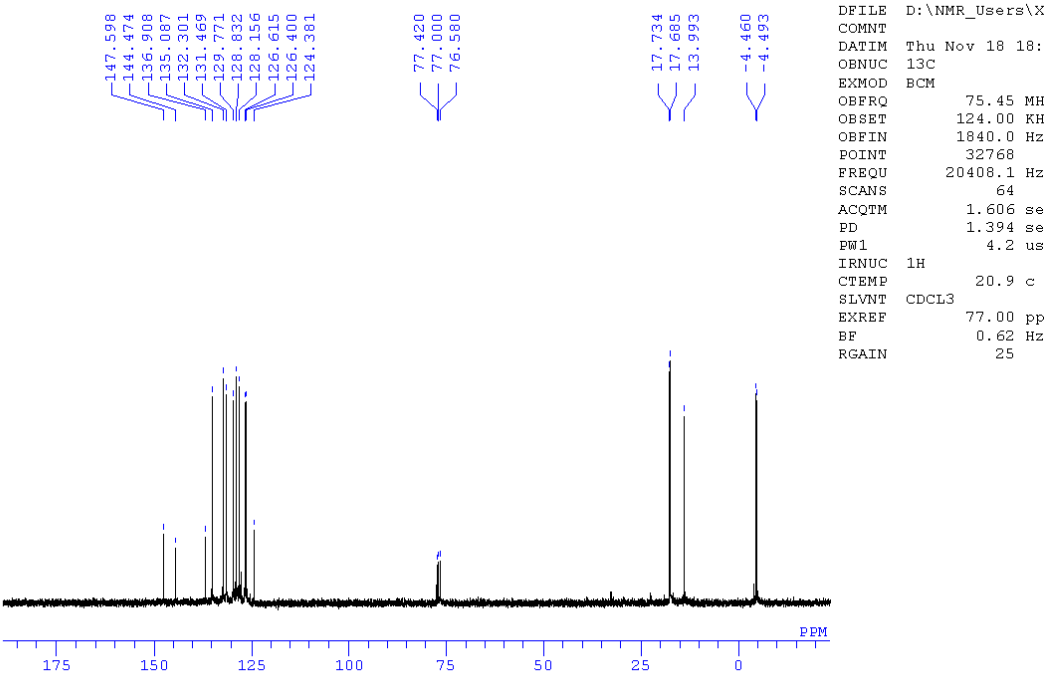

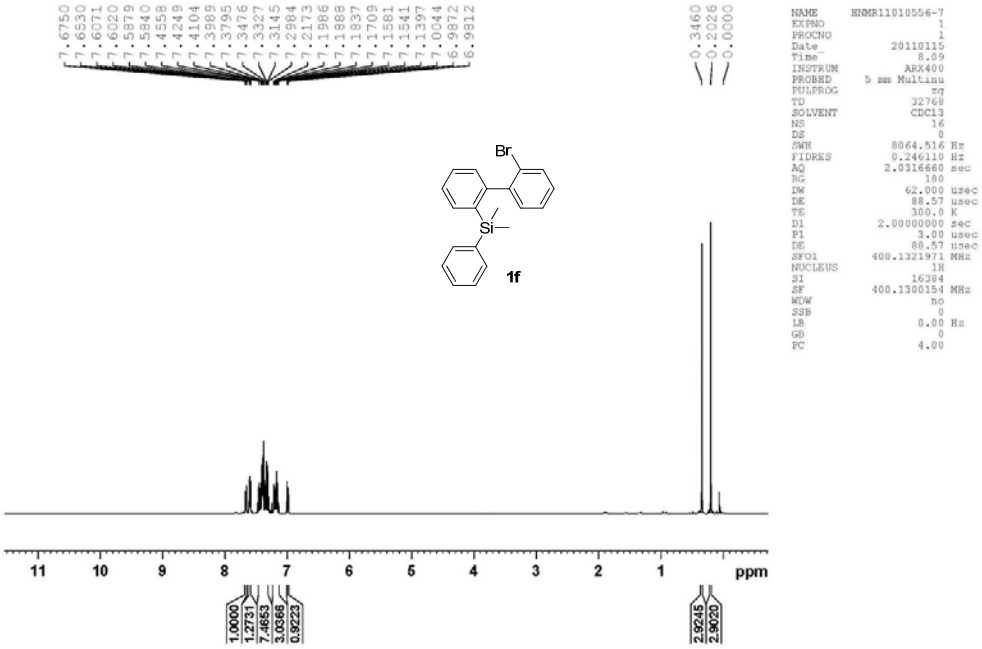

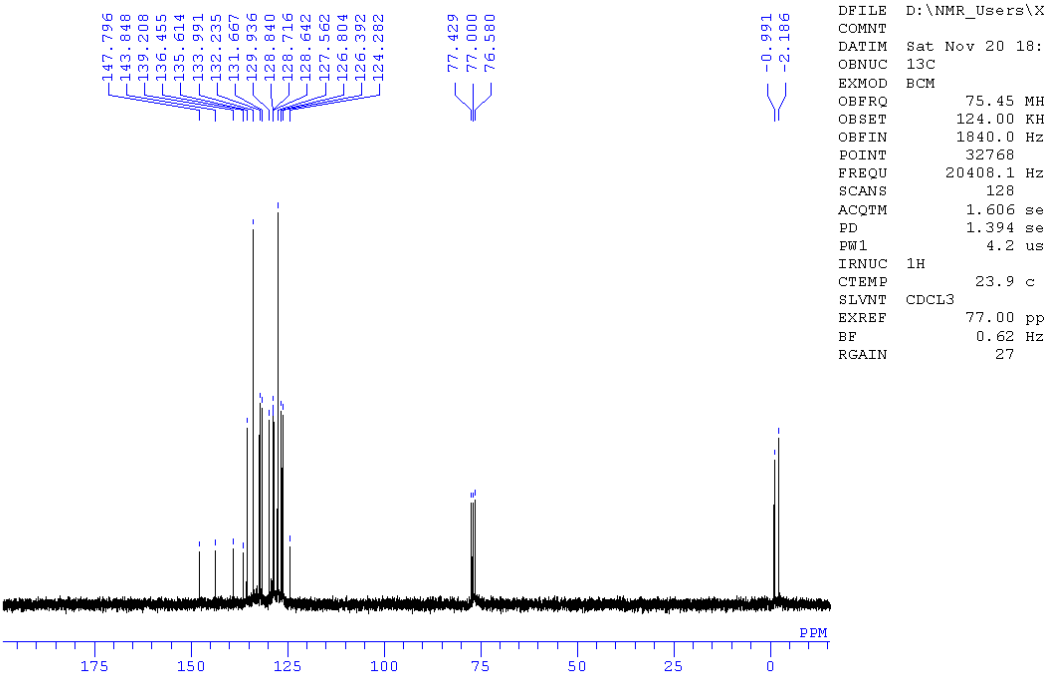

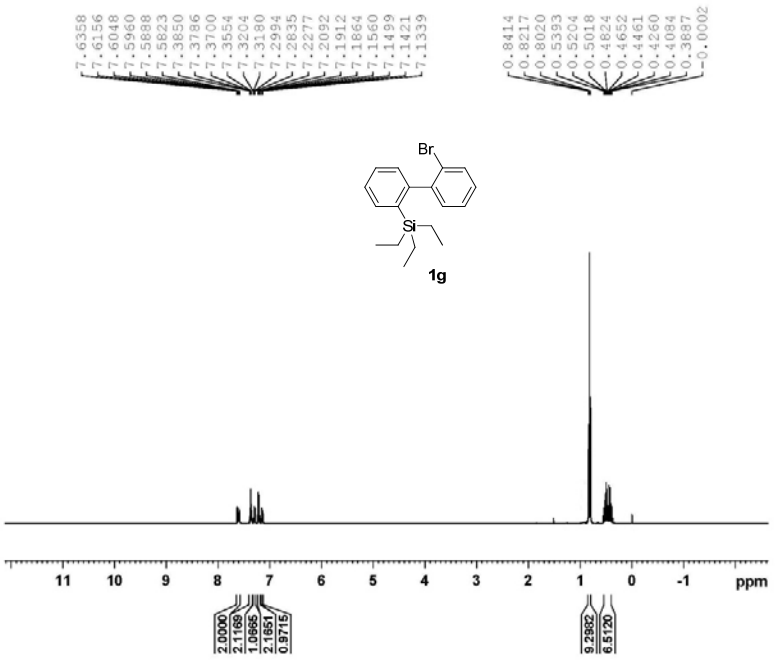
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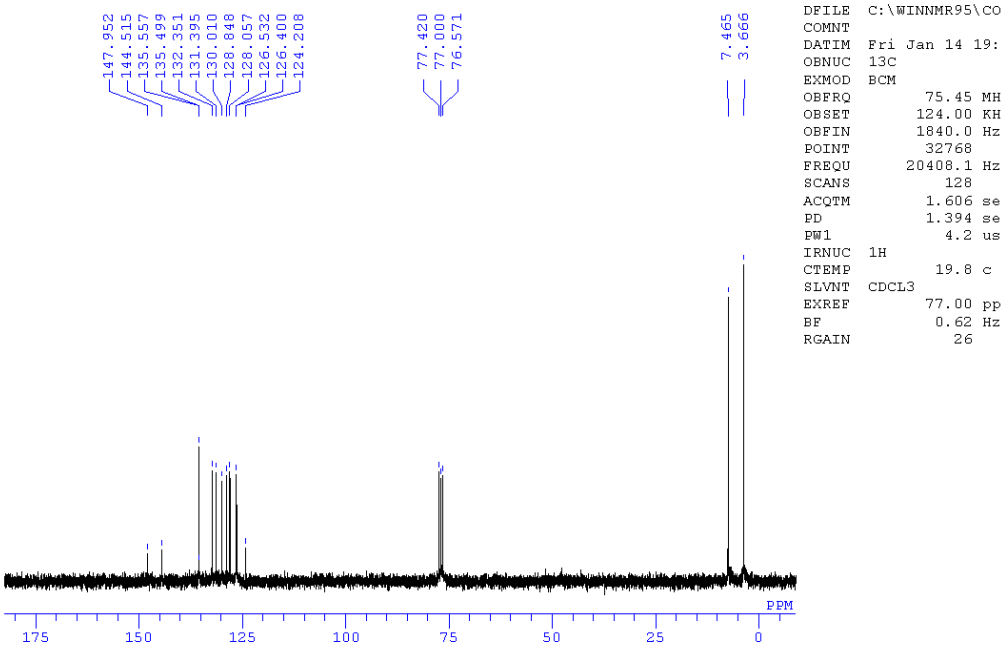
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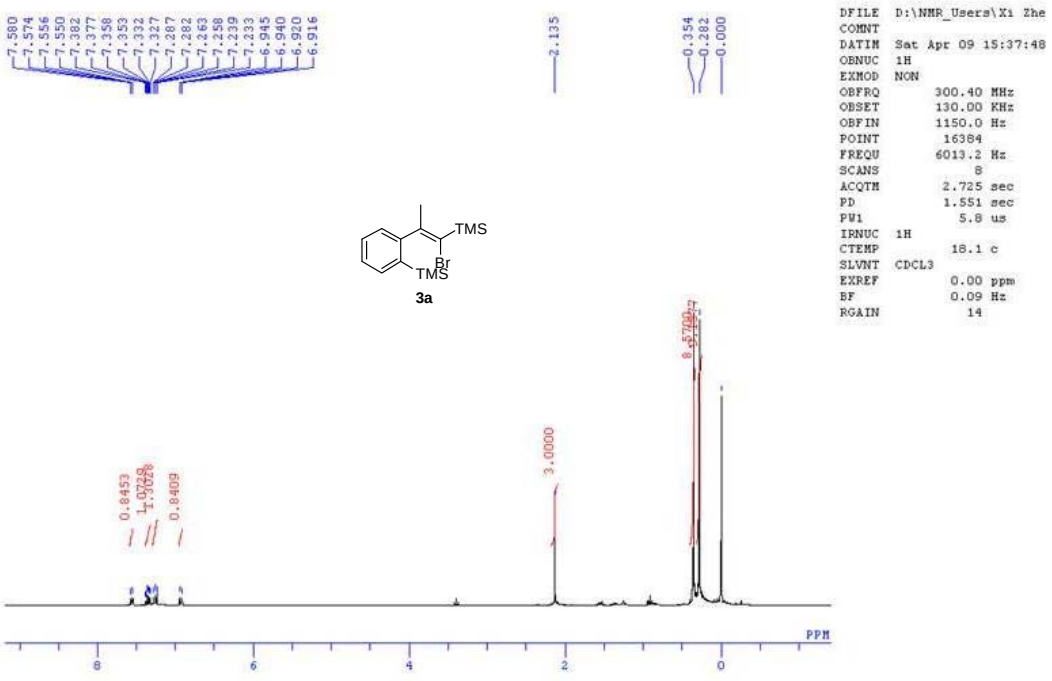
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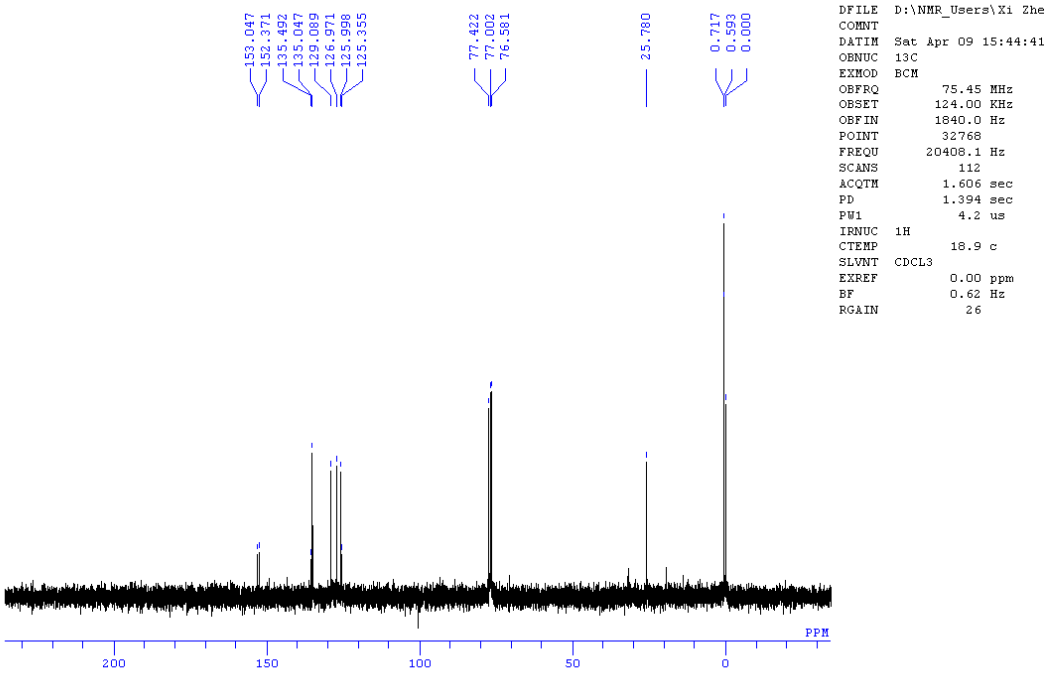
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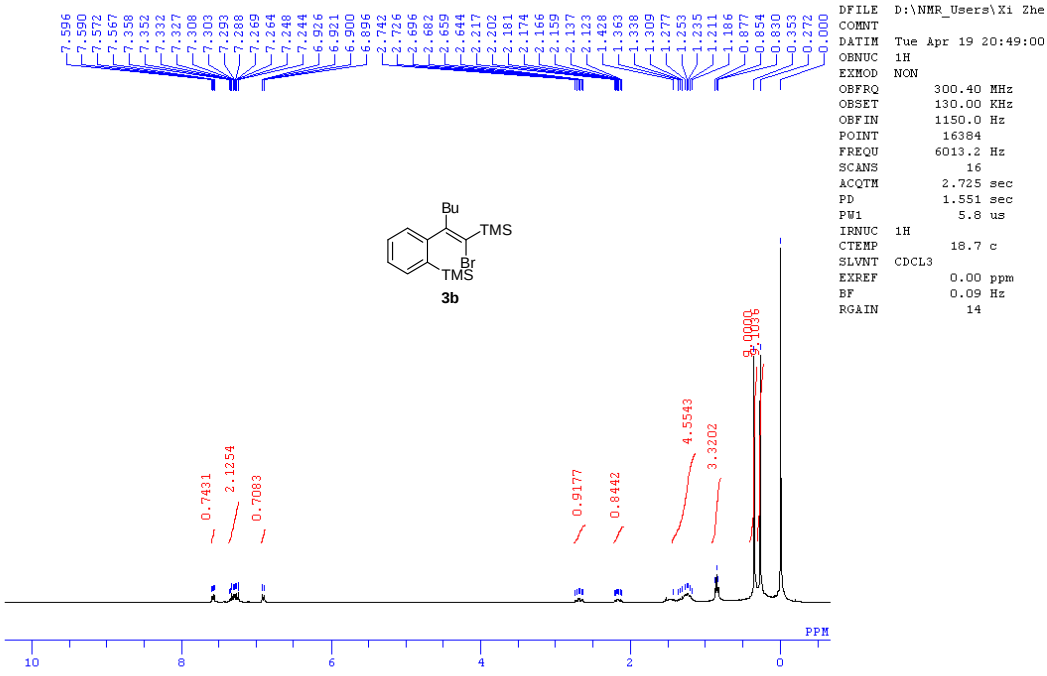
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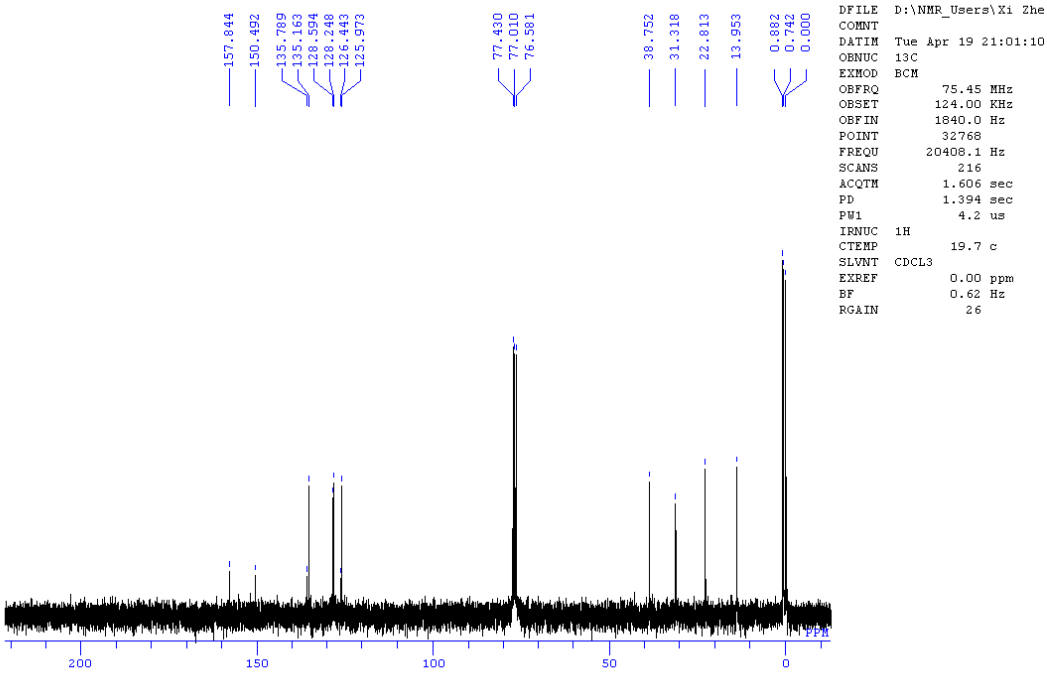
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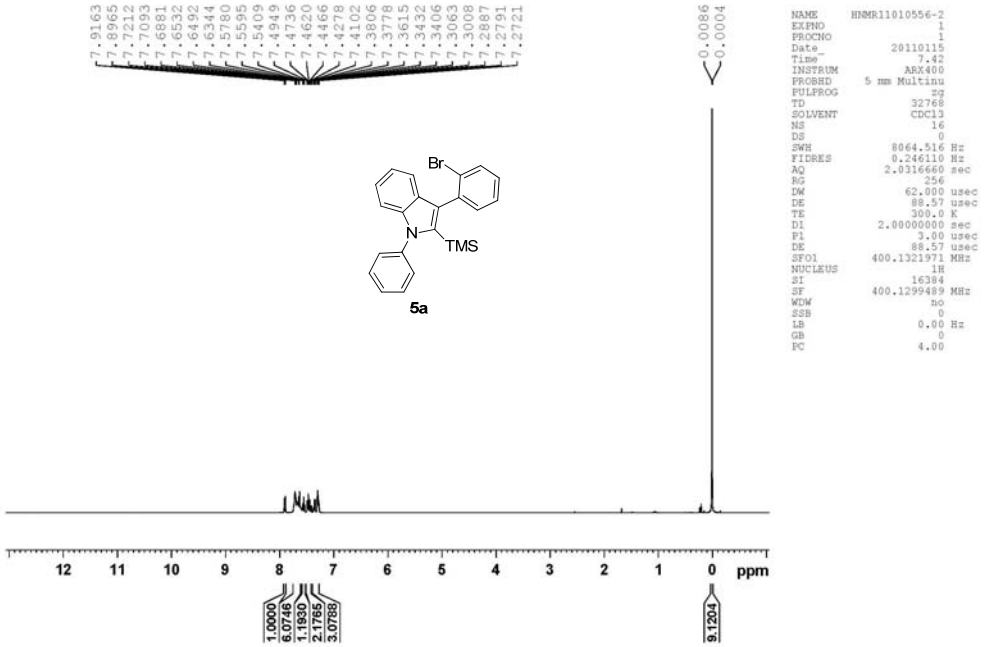

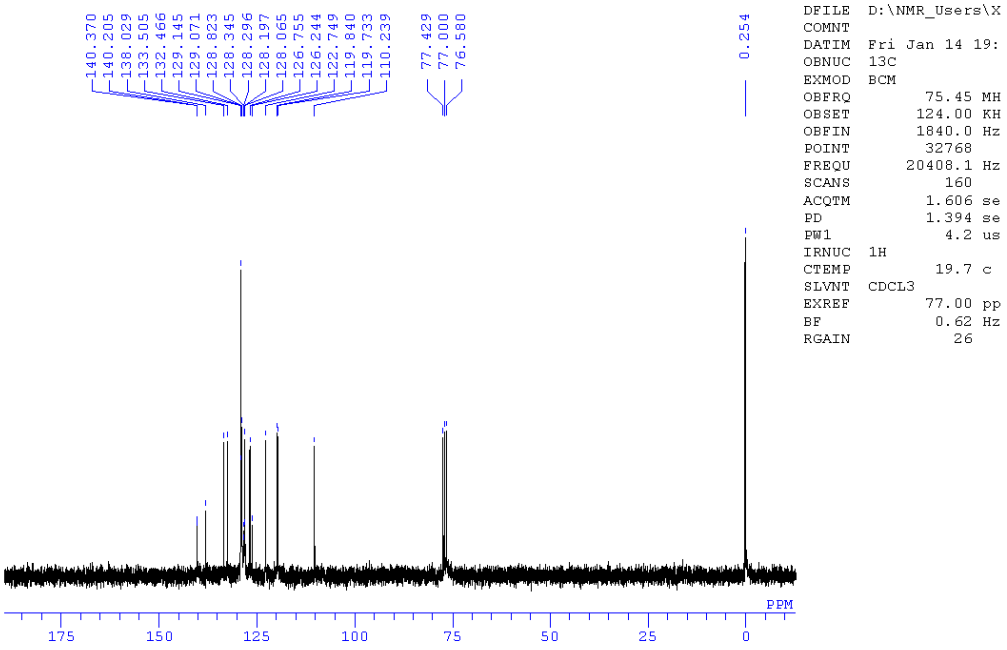

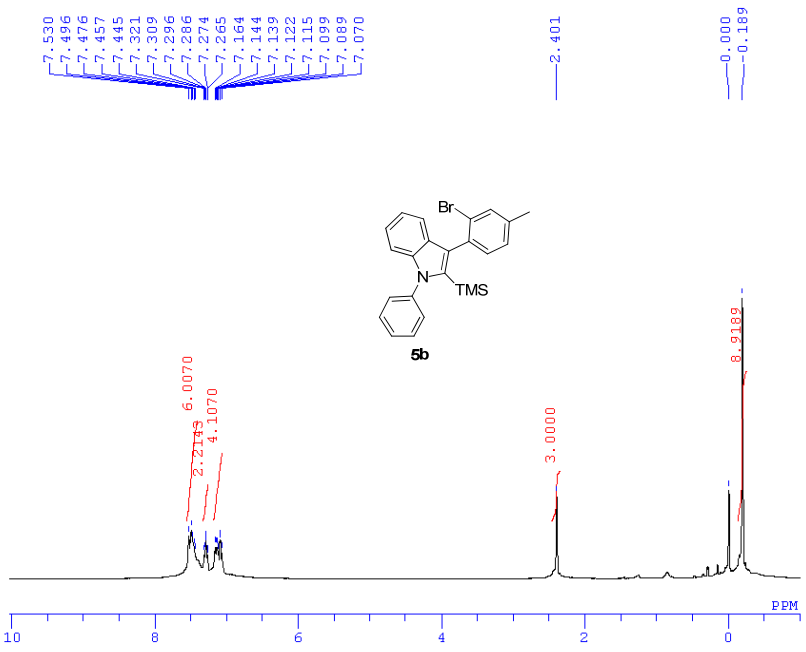


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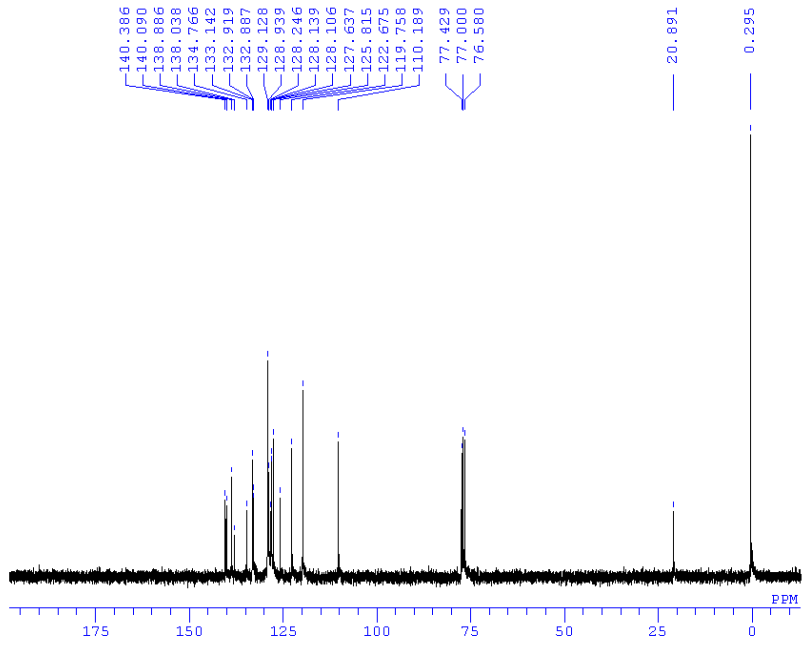


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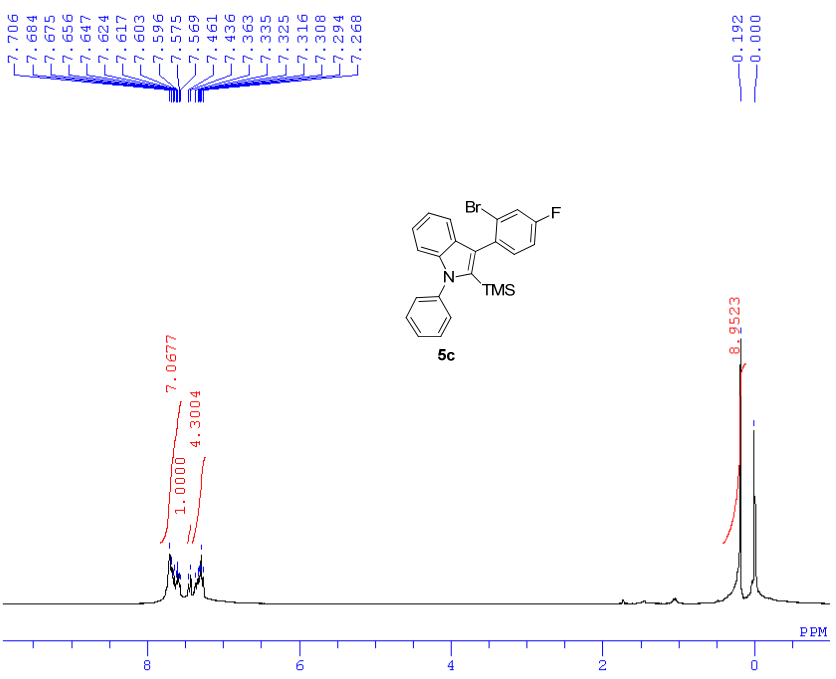


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RGAIN            14
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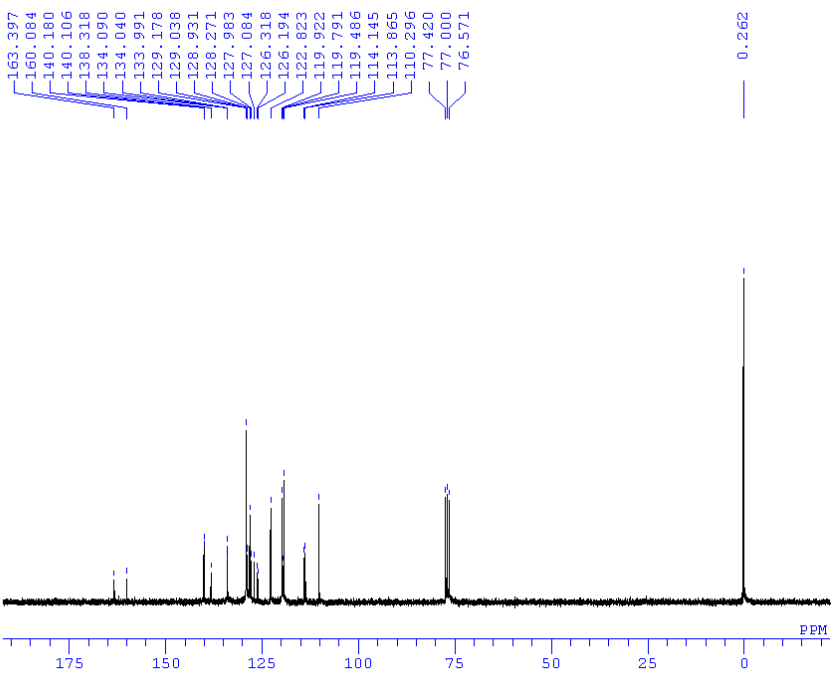
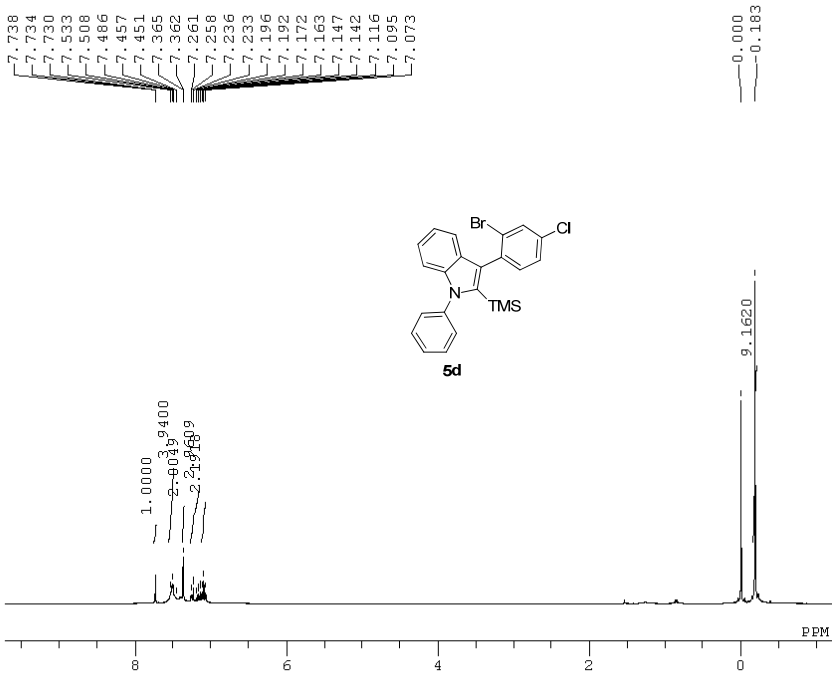
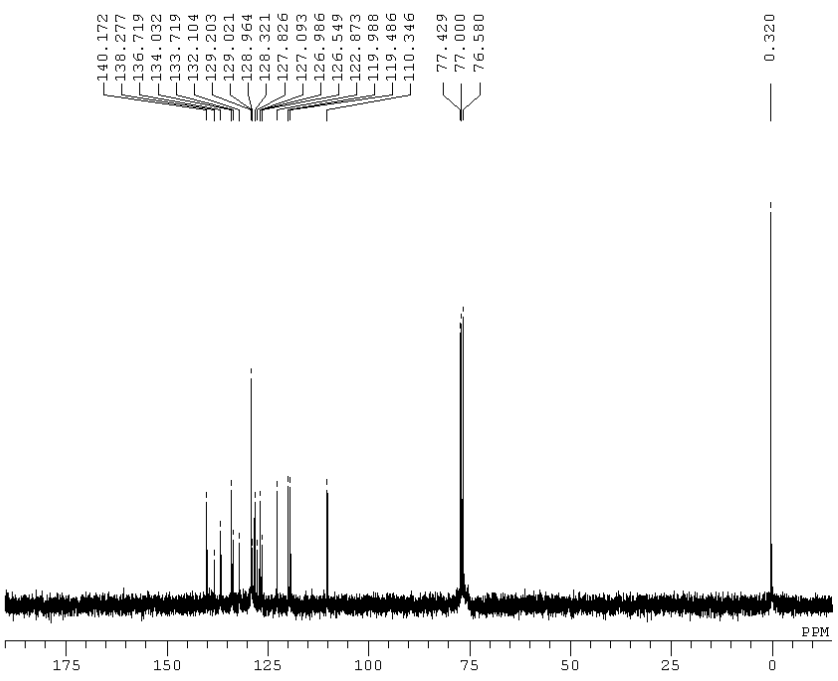


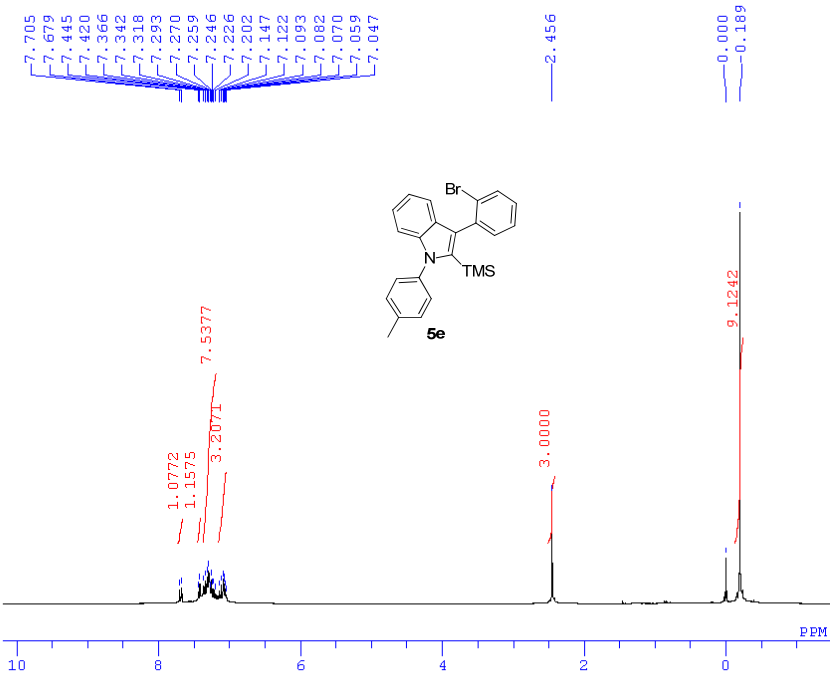

```
DFILE  D:\NMR_Users\X
COMNT
DATIM  Fri Jan 21 20:
OBNUC  13C
EXMOD  BCM
OBFRQ         75.45 MH
OBSET        124.00 KH
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           344
ACQTM         1.606 se
PD            1.394 se
PW1             4.2 us
IRNUC  1H
CTEMP          11.3 c
SLVNT  CDCL3
EXREF         77.00 pp
BF             0.62 Hz
RGAIN            27
```



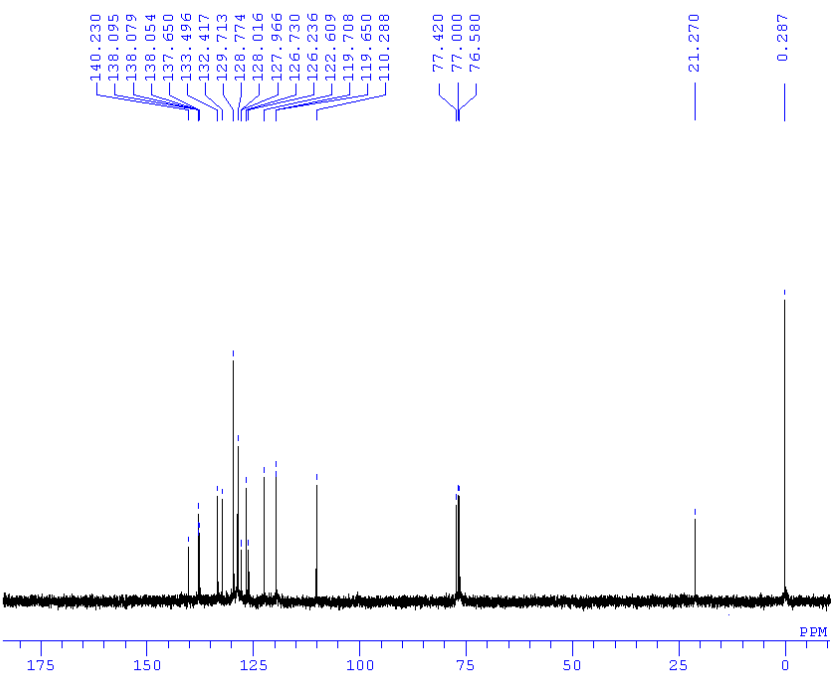

```
DFILE  D:\NMR_Users\X
COMNT
DATIM  Fri Dec 17 19:
OBNUC  1H
EXMOD  NON
OBFRQ        300.40 MH
OBSET        130.00 KH
OBFIN        1150.0 Hz
POINT         16384
FREQU        6013.2 Hz
SCANS             8
ACQTM         2.725 se
PD            1.551 se
PW1             5.8 us
IRNUC  1H
CTEMP          13.8 c
SLVNT  CDCL3
EXREF          0.00 pp
BF             0.09 Hz
RGAIN            15
```



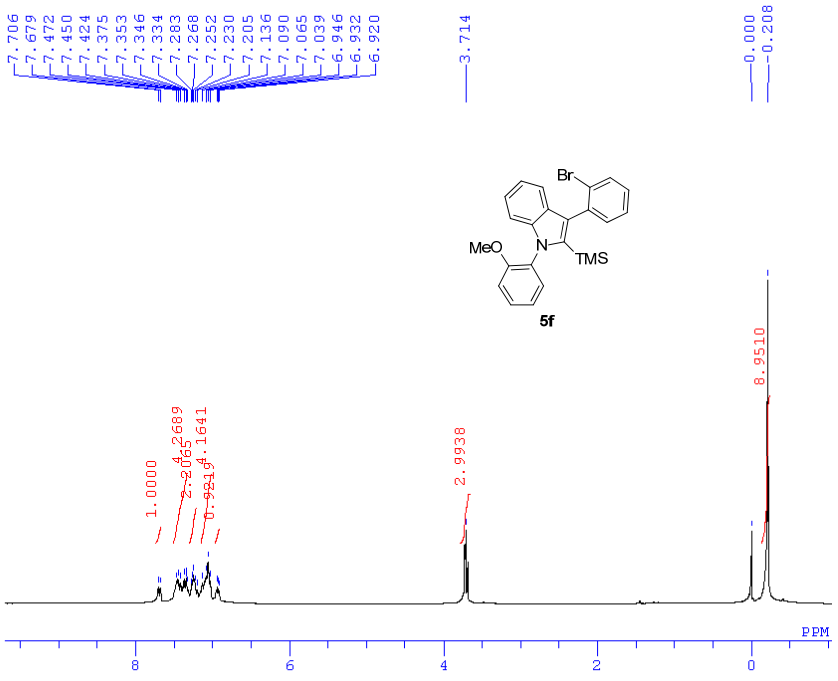

```
DFILE  C:\WINNMR95\CO
COMNT
DATIM  Fri Dec 17 19:
OBNUC  13C
EXMOD  BCM
OBFRQ         75.45 MH
OBSET        124.00 KH
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           288
ACQTM         1.606 se
PD            1.394 se
PW1             4.2 us
IRNUC  1H
CTEMP          14.6 c
SLVNT  CDCL3
EXREF         77.00 pp
BF             0.62 Hz
RGAIN            27
```



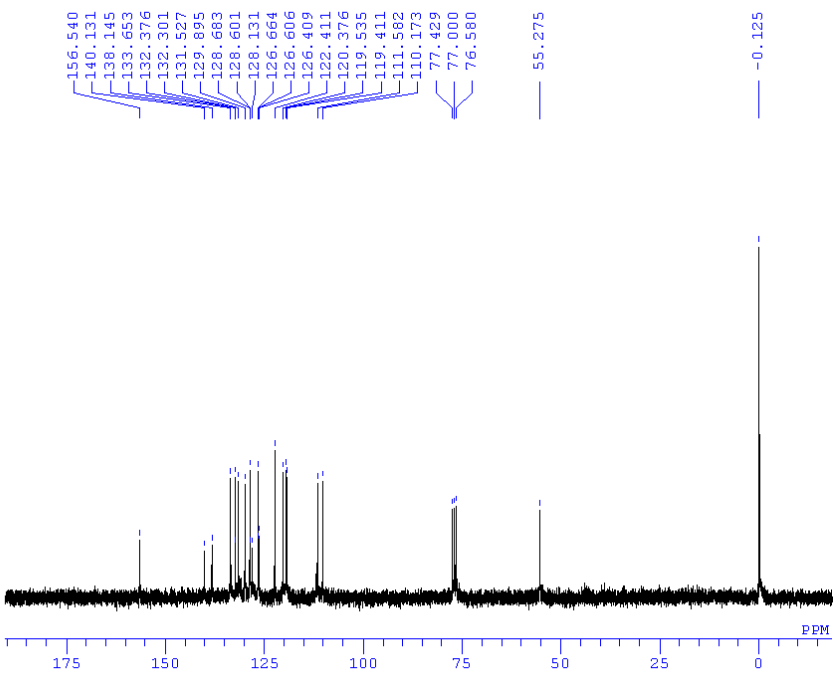

```
DFILE  D:\NMR_Users\X
COMNT
DATIM  Tue Dec 07 22:
OBNUC  1H
EXMOD  NON
OBFRQ        300.40 MH
OBSET        130.00 KH
OBFIN        1150.0 Hz
POINT         16384
FREQU        6013.2 Hz
SCANS             8
ACQTM         2.725 se
PD            1.551 se
PW1             5.8 us
IRNUC  1H
CTEMP          14.5 c
SLVNT  CDCL3
EXREF          0.00 pp
BF             0.09 Hz
RGAIN            13
```



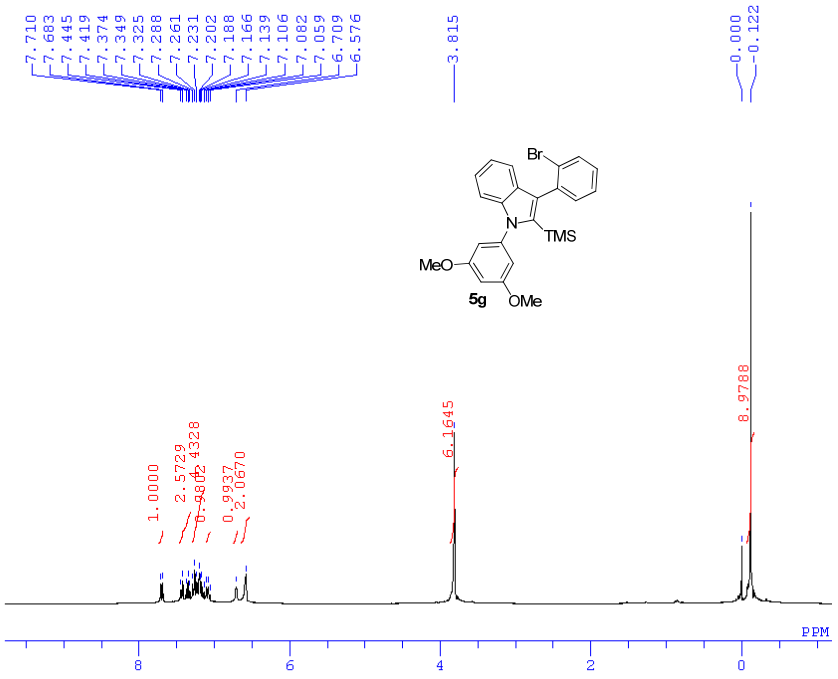

```
DFILE  D:\NMR_Users\X
COMNT
DATIM  Tue Dec 07 21:
OBNUC  13C
EXMOD  BCM
OBFRQ         75.45 MH
OBSET        124.00 KH
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           104
ACQTM         1.606 se
PD            1.394 se
PW1             4.2 us
IRNUC  1H
CTEMP          15.1 c
SLVNT  CDCL3
EXREF         77.00 pp
BF             0.62 Hz
RGAIN            26
```

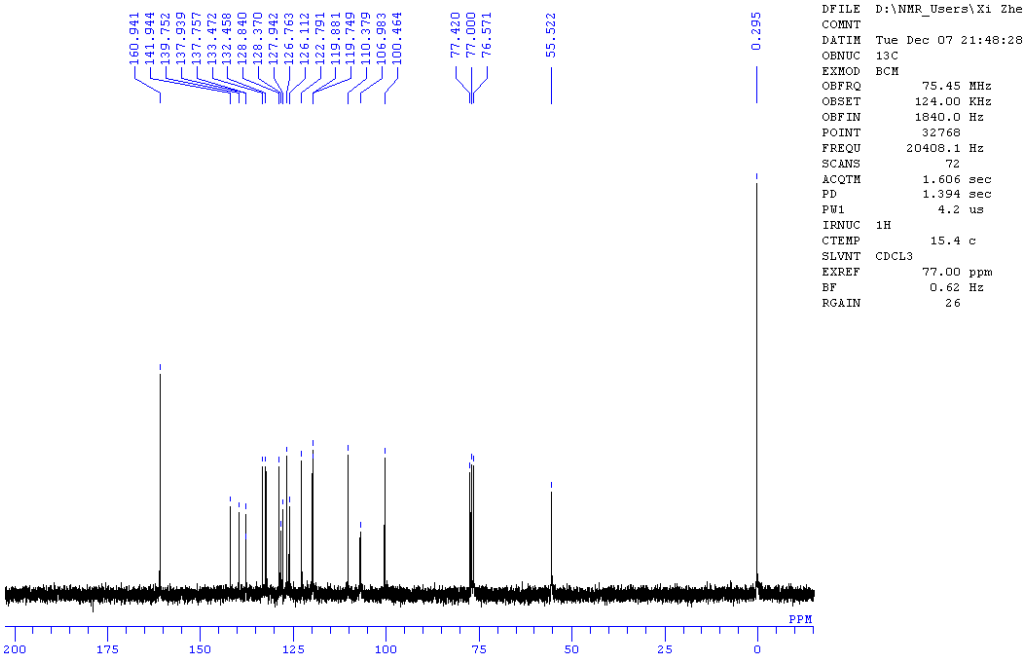


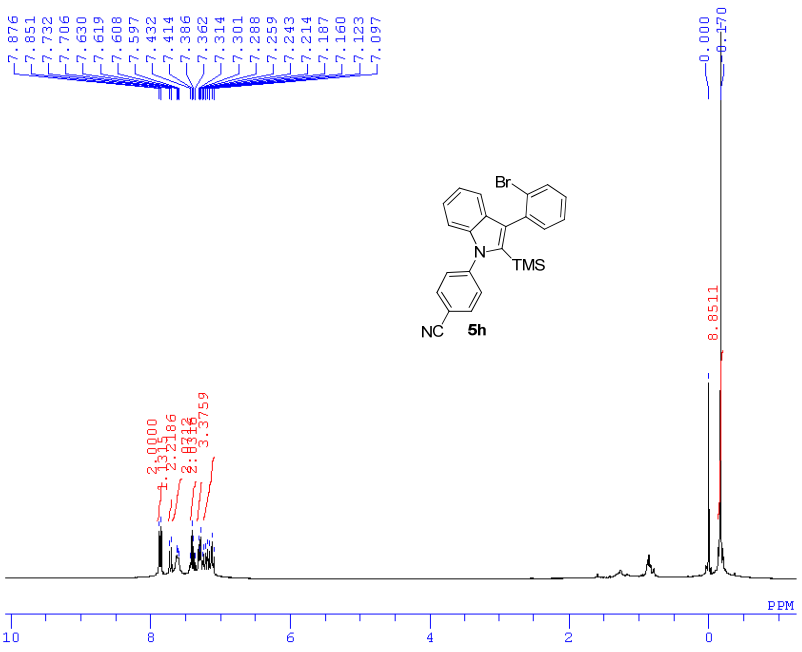

```
DFILE  D:\NMR_Users\X
COMNT
DATIM  Fri Jan 21 20:
OBNUC  1H
EXMOD  NON
OBFRQ       300.40 MH
OBSET       130.00 KH
OBFIN       1150.0 Hz
POINT        16384
FREQU       6013.2 Hz
SCANS           16
ACQTM        2.725 se
PD           1.551 se
PW1           5.8 us
IRNUC  1H
CTEMP         10.6 c
SLVNT  CDCL3
EXREF         0.00 pp
BF            0.09 Hz
RGAIN           13
```



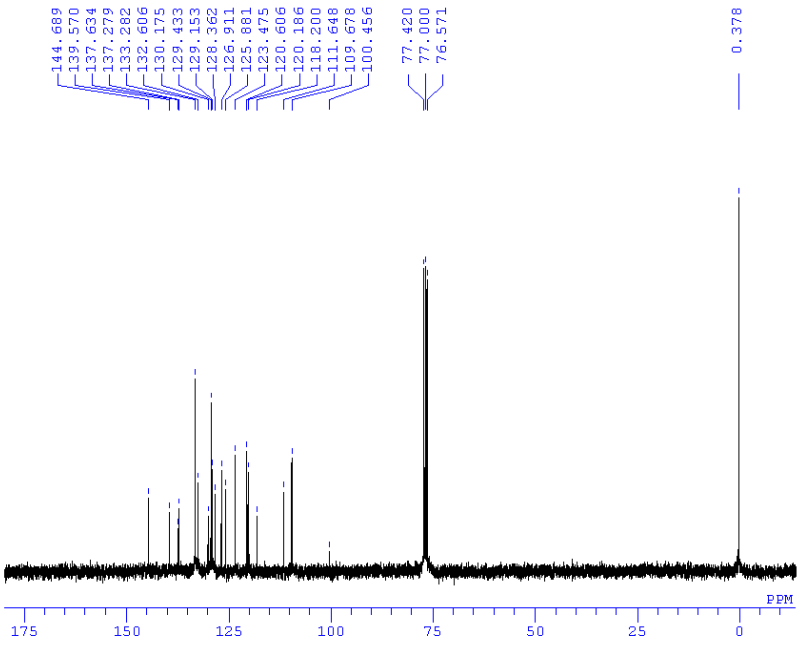

```
DFILE  D:\NMR_Users\X
COMNT
DATIM  Sun Nov 28 16:
OBNUC  13C
EXMOD  BCM
OBFRQ        75.45 MH
OBSET       124.00 KH
OBFIN       1840.0 Hz
POINT        32768
FREQU      20408.1 Hz
SCANS           96
ACQTM        1.606 se
PD           1.394 se
PW1            4.2 us
IRNUC  1H
CTEMP         15.9 c
SLVNT  CDCL3
EXREF        77.00 pp
BF            0.63 Hz
RGAIN           26
```


7.710
7.683
7.445
7.419
7.374
7.349
7.325
7.288
7.261
7.231
7.202
7.188
7.166
7.139
7.106
7.082
7.059
6.709
6.576
3.815
0.000
-0.122
Br
N
TMS
MeO
OMe
5g
DFILE D:\NMR_Users\X
COMNT
DATIM Tue Dec 07 22:
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MH
OBSET 130.00 KH
OBFIN 1150.0 Hz
POINT 16384
FREQU 6013.2 Hz
SCANS 8
ACQTM 2.725 se
PD 1.551 se
PW1 5.8 us
IRNUC 1H
CTEMP 14.5 c
SLVNT CDCL3
EXREF 0.00 pp
BF 0.09 Hz
RGAIN 12
1.0000
2.5729
0.9805
4.4328
0.9937
2.0670
6.1645
8.9788
PPM
8
6
4
2
0

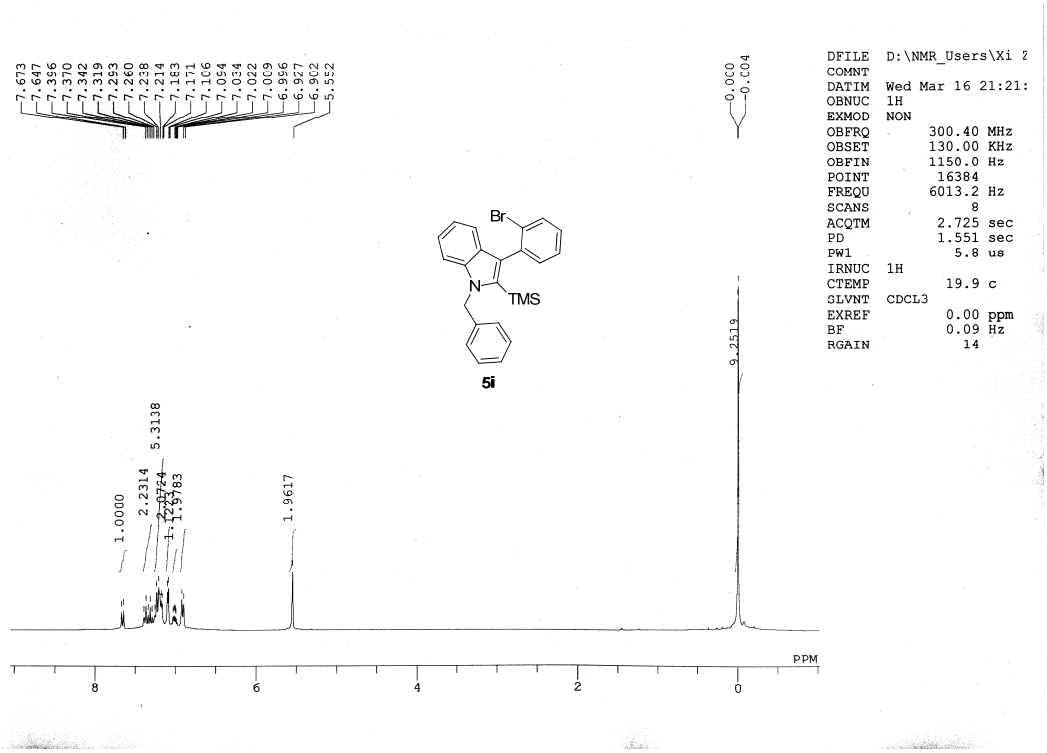

160.941
141.944
139.752
137.939
137.757
133.472
132.458
128.840
128.370
127.942
126.763
126.112
122.791
119.881
119.749
110.379
106.983
100.464
77.420
77.000
76.571
55.522
0.295
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Tue Dec 07 21:48:28
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 72
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 15.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26
PPM
200
175
150
125
100
75
50
25
0

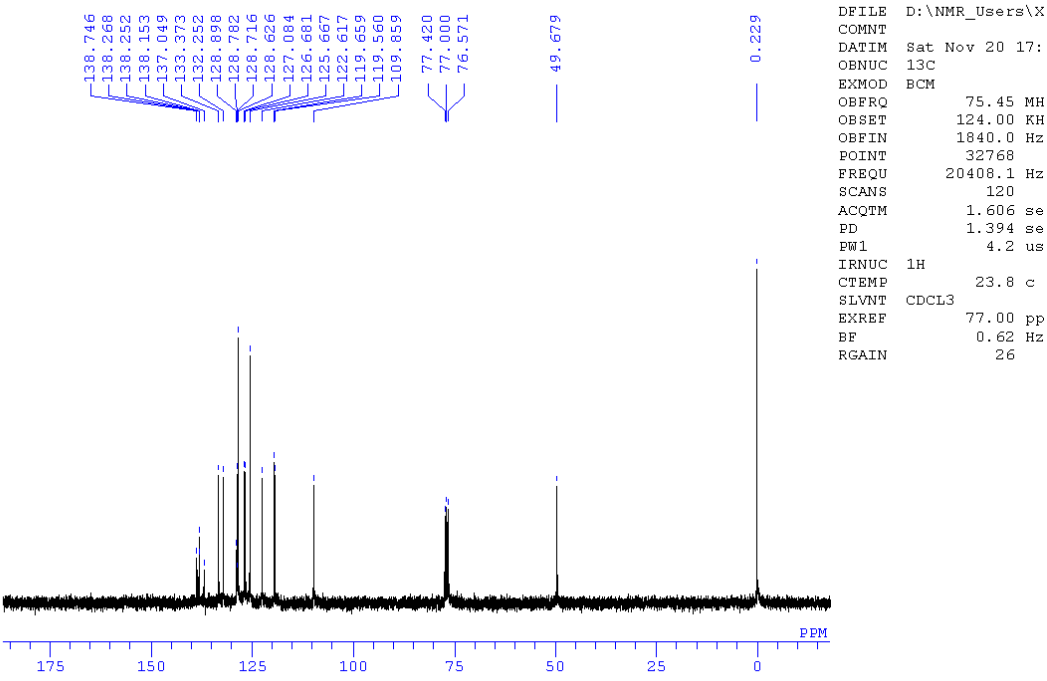


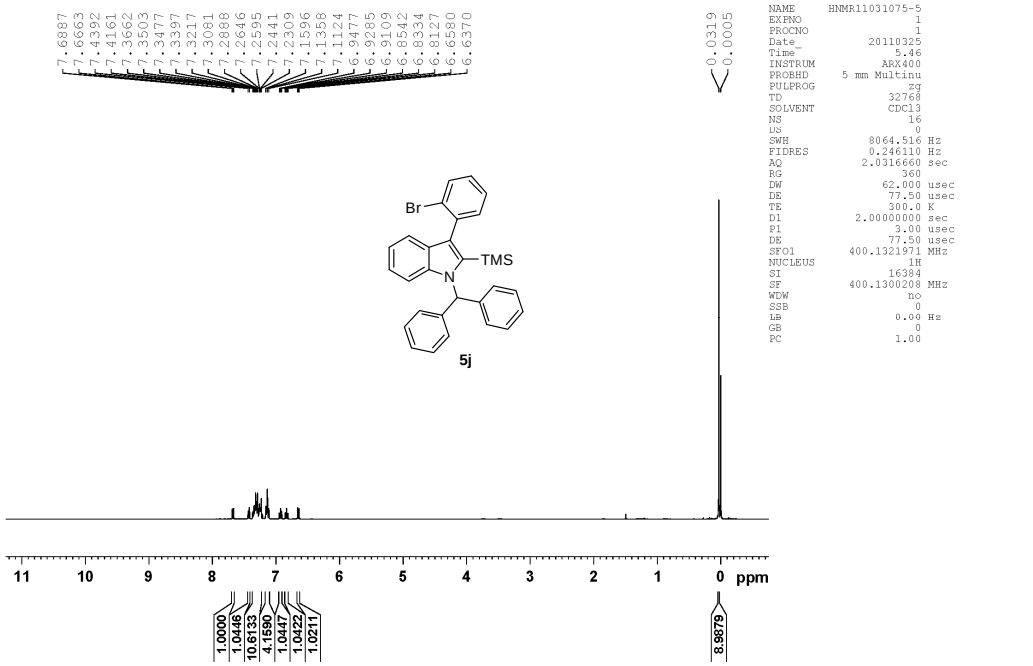

```
DFILE  D:\NMR_Users\X
COMNT
DATIM  Sun Jan 09 19:
OBNUC  1H
EXMOD  NON
OBFRQ       300.40 MH
OBSET       130.00 KH
OBFIN       1150.0 Hz
POINT        16384
FREQU       6013.2 Hz
SCANS           16
ACQTM        2.725 se
PD           1.551 se
PW1            5.8 us
IRNUC  1H
CTEMP         11.3 c
SLVNT  CDCL3
EXREF          0.00 pp
BF             0.09 Hz
RGAIN            14
```

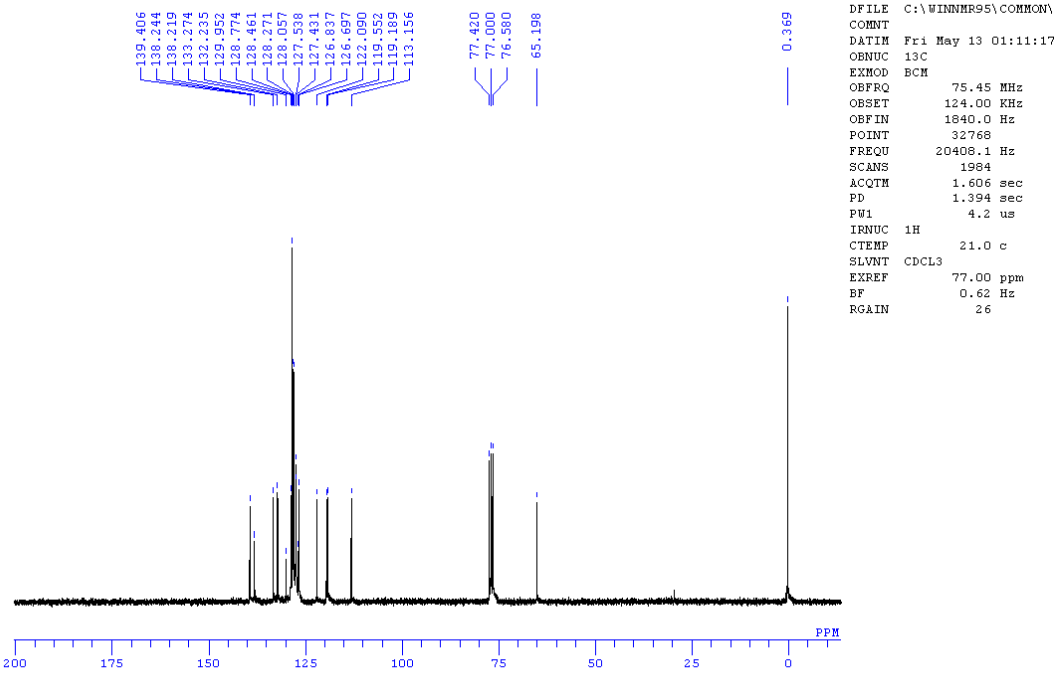


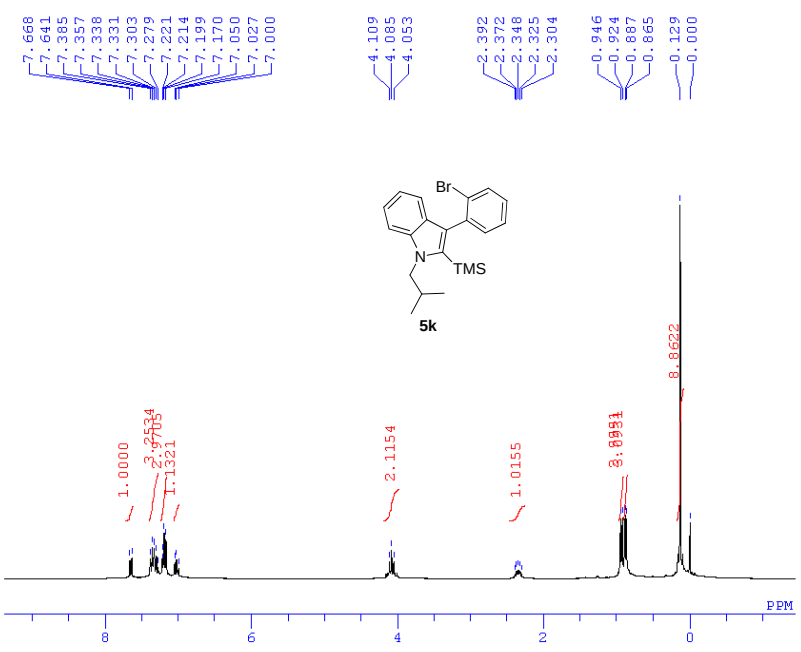

```
DFILE  D:\NMR_Users\X
COMNT
DATIM  Sun Jan 09 19:
OBNUC  13C
EXMOD  BCM
OBFRQ        75.45 MH
OBSET       124.00 KH
OBFIN       1840.0 Hz
POINT        32768
FREQU      20408.1 Hz
SCANS           504
ACQTM        1.606 se
PD           1.394 se
PW1            4.2 us
IRNUC  1H
CTEMP         11.7 c
SLVNT  CDCL3
EXREF         77.00 pp
BF             0.62 Hz
RGAIN            25
```


7.673
7.647
7.396
7.370
7.342
7.319
7.293
7.260
7.238
7.214
7.183
7.171
7.106
7.054
7.034
7.022
7.009
6.996
6.927
6.902
5.552
0.000
-0.004
Br
N
TMS
5i
9.2519
5.3138
2.2314
2.0774
1.9783
1.9617
1.0000
PPM
8
6
4
2
0
DFILE D:\NMR_Users\Xi 2
COMNT
DATIM Wed Mar 16 21:21:
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 16384
FREQU 6013.2 Hz
SCANS 8
ACQTM 2.725 sec
PD 1.551 sec
PW1 5.8 us
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.09 Hz
RGAIN 14

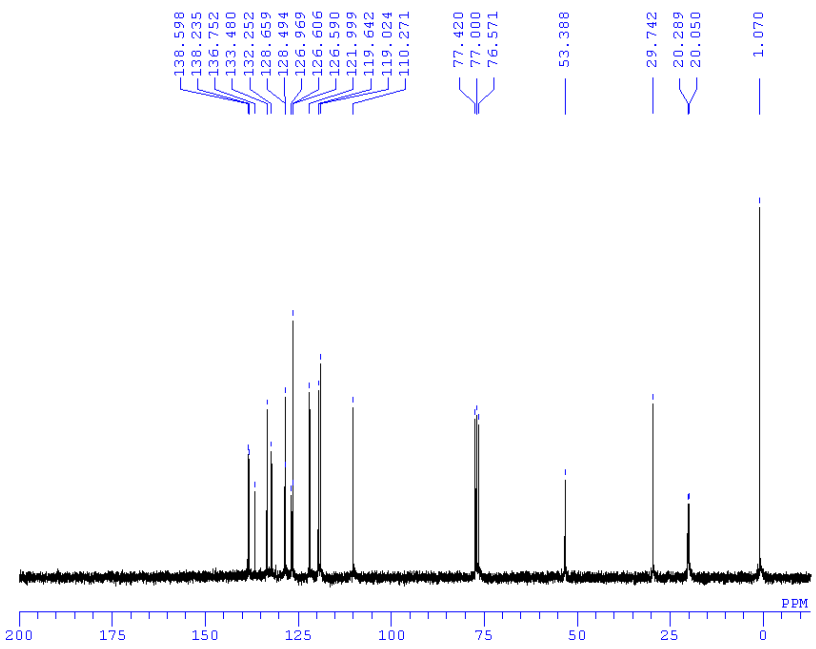

138.746
138.268
138.252
138.153
137.049
133.373
132.252
128.898
128.782
128.716
128.626
127.084
126.681
125.667
122.617
119.659
119.560
109.859
77.420
77.000
76.571
49.679
0.229
PPM
175
150
125
100
75
50
25
0
DFILE D:\NMR_Users\X
COMNT
DATIM Sat Nov 20 17:
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MH
OBSET 124.00 KH
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 120
ACQTM 1.606 se
PD 1.394 se
PW1 4.2 us
IRNUC 1H
CTEMP 23.8 c
SLVNT CDCL3
EXREF 77.00 pp
BF 0.62 Hz
RGAIN 26



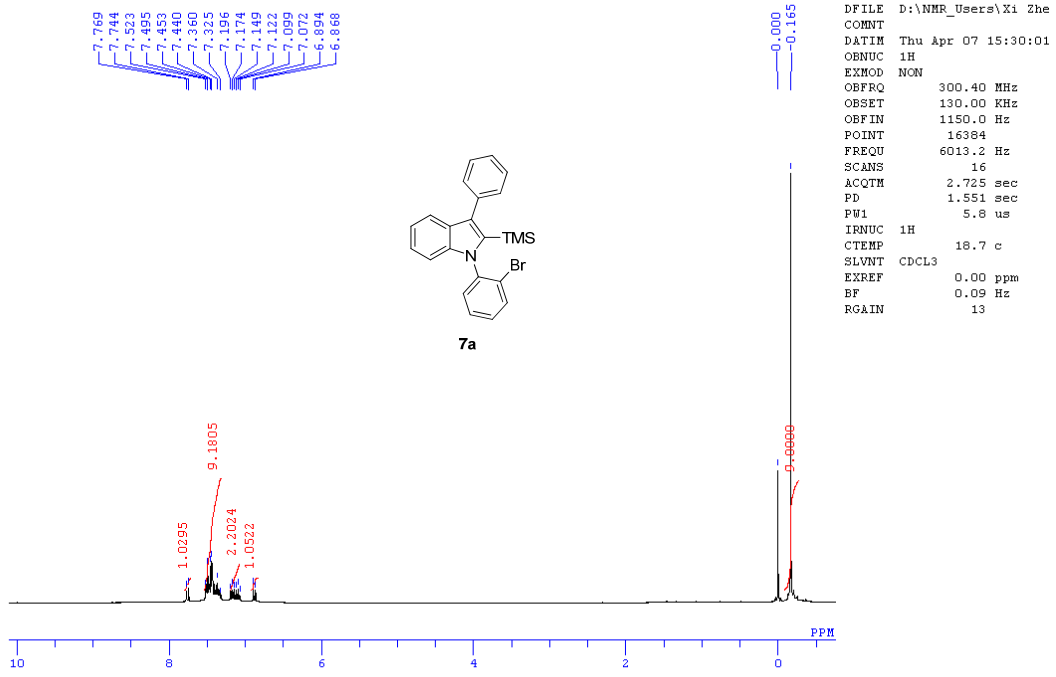


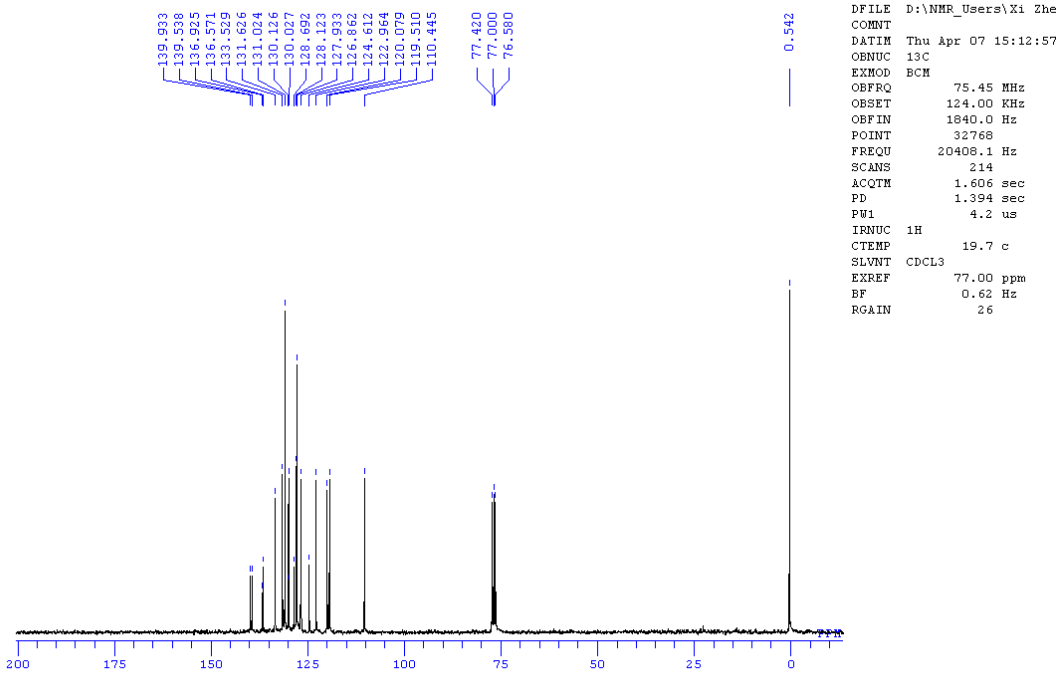


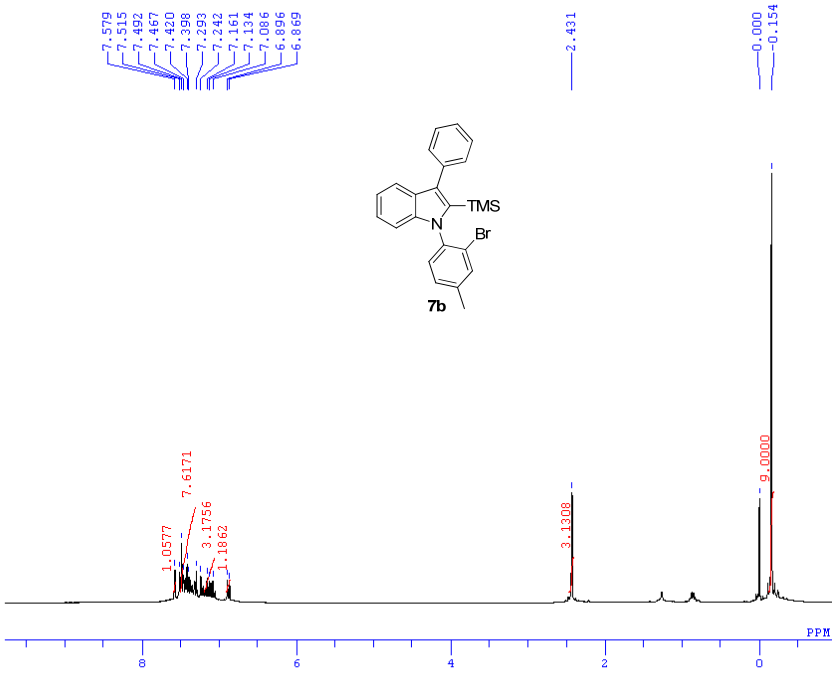

```
DFILE  D:\NMR_Users\X
COMNT
DATIM  Sun Jan 09 19:
OBNUC  1H
EXMOD  NON
OBFRQ       300.40 MH
OBSET       130.00 KH
OBFIN       1150.0 Hz
POINT        16384
FREQU       6013.2 Hz
SCANS            8
ACQTM        2.725 se
PD           1.551 se
PW1            5.8 us
IRNUC  1H
CTEMP         11.3 c
SLVNT  CDCL3
EXREF         0.00 pp
BF            0.09 Hz
RGAIN           13
```

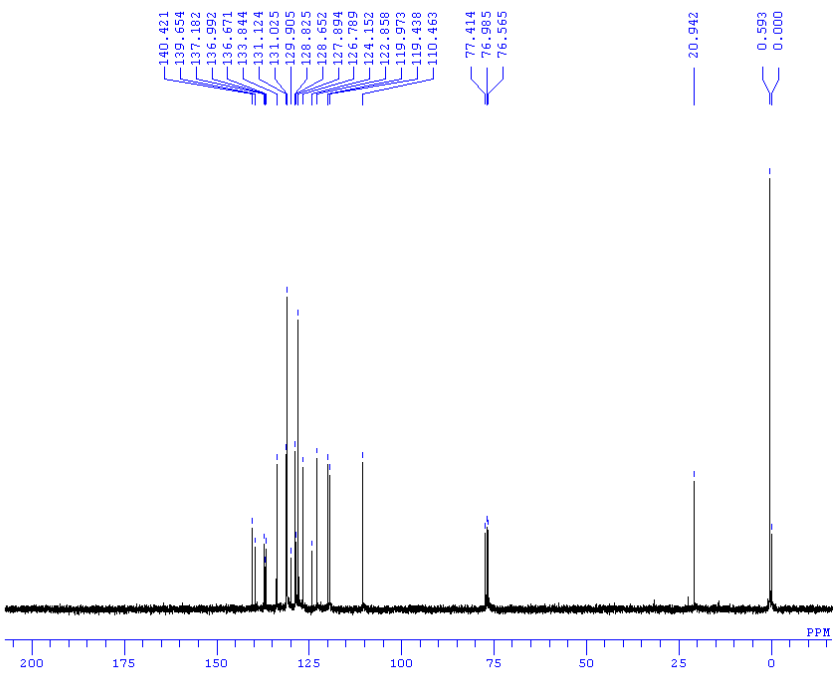


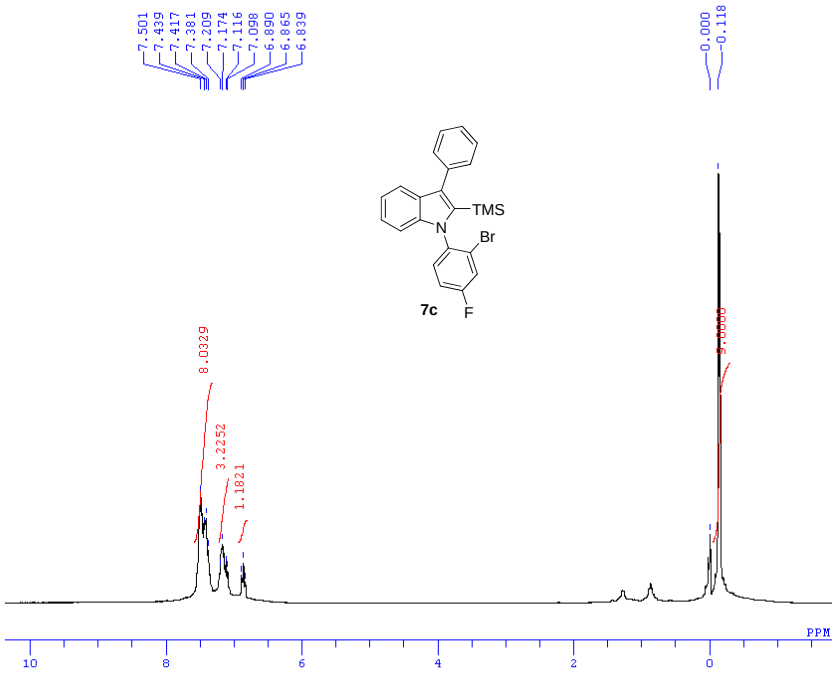

```
DFILE  C:\WINNMR95\CO
COMNT
DATIM  Sun Jan 09 19:
OBNUC  13C
EXMOD  BCM
OBFRQ        75.45 MH
OBSET       124.00 KH
OBFIN       1840.0 Hz
POINT        32768
FREQU      20408.1 Hz
SCANS          248
ACQTM        1.606 se
PD           1.394 se
PW1            4.2 us
IRNUC  1H
CTEMP         12.0 c
SLVNT  CDCL3
EXREF        77.00 pp
BF            0.62 Hz
RGAIN           26
```


7.769
7.744
7.523
7.495
7.453
7.440
7.360
7.325
7.196
7.174
7.149
7.122
7.099
7.072
6.894
6.868
0.000
0.165
TMS
Br
7a
9.1805
1.0295
2.2024
1.0522
1.0000
PPM
10
8
6
4
2
0
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Thu Apr 07 15:30:01
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 16384
FREQU 6013.2 Hz
SCANS 16
ACQTM 2.725 sec
PD 1.551 sec
PW1 5.8 us
IRNUC 1H
CTEMP 18.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.09 Hz
RGAIN 13

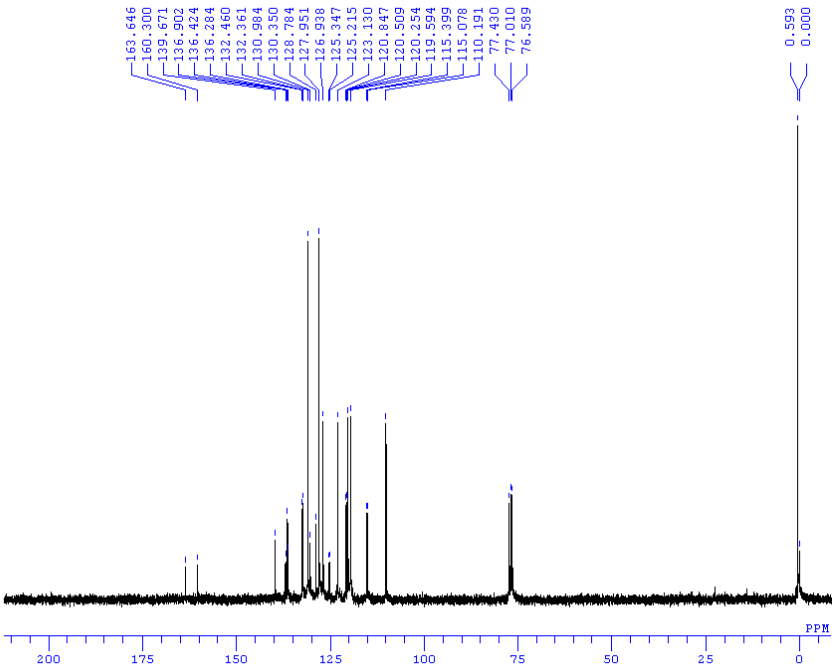

139.933
139.538
136.925
136.571
133.529
131.626
131.024
130.126
130.027
128.692
128.123
127.933
126.862
124.612
122.964
120.079
119.510
110.445
77.420
77.000
76.580
0.542
PPM
200
175
150
125
100
75
50
25
0
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Thu Apr 07 15:12:57
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 214
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26


7.579
7.515
7.492
7.467
7.420
7.398
7.293
7.242
7.161
7.134
7.086
6.896
6.869
2.431
0.000
0.154
TMS
N
Br
7b
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Fri Apr 15 16:12:11
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 16384
FREQU 6013.2 Hz
SCANS 16
ACQTM 2.725 sec
PD 1.551 sec
PW1 5.8 us
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.09 Hz
RGAIN 13
7.6171
3.1756
1.1862
1.0577
3.1308
9.0000
PPM
8
6
4
2
0

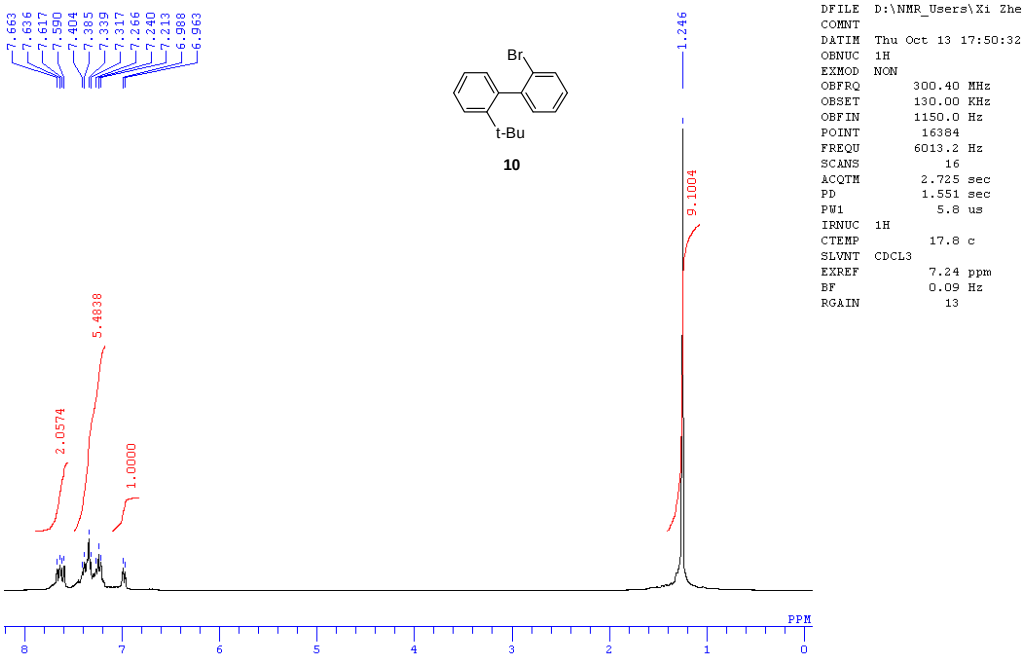

140.421
139.654
137.182
136.992
136.671
133.844
131.124
131.025
129.905
128.825
128.652
127.994
126.789
124.152
122.858
119.973
119.438
110.463
77.414
76.985
76.565
20.942
0.593
0.000
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Fri Apr 15 16:08:23
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 220
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.62 Hz
RGAIN 28
PPM
200
175
150
125
100
75
50
25
0

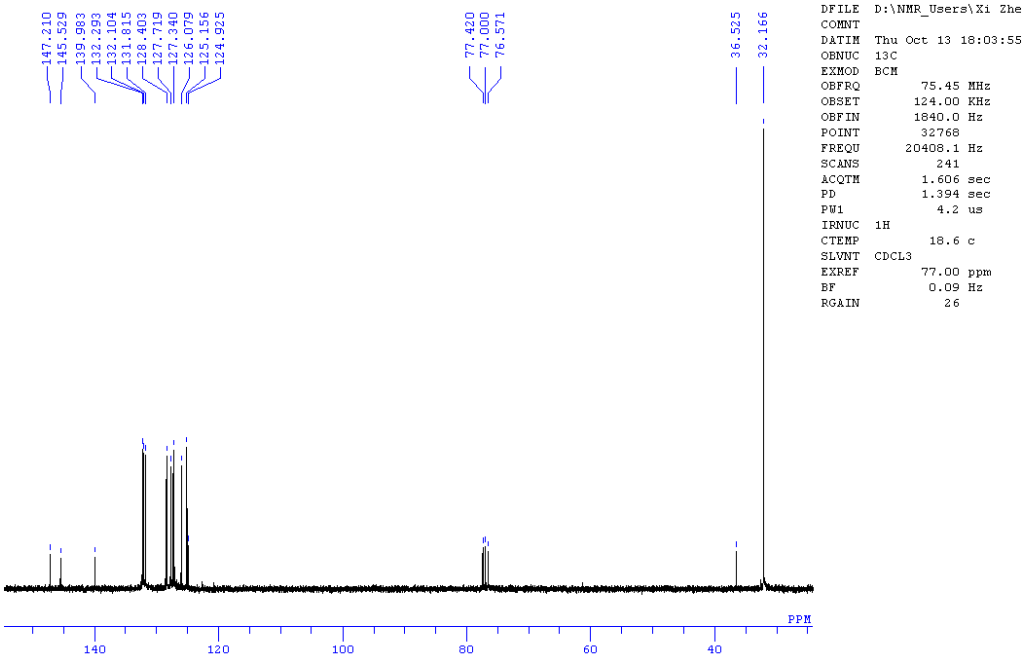


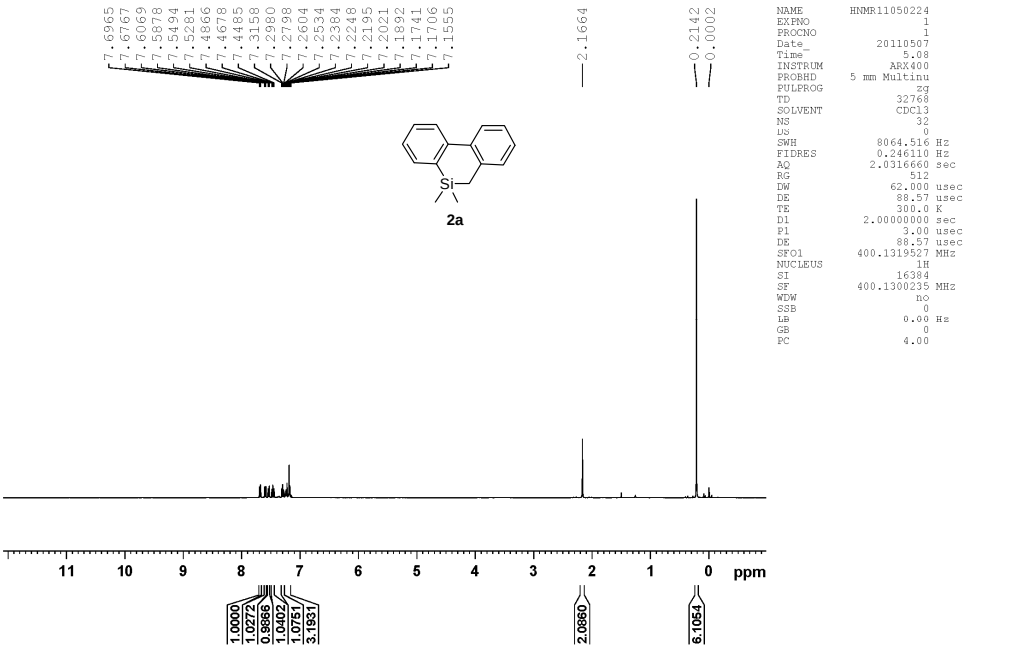

```
DFILE  C:\WINNMR95\COMMON\
COMNT
DATIM  Sat Apr 16 20:45:21
OBNUC  1H
EXMOD  NON
OBFRQ       300.40 MHz
OBSET       130.00 KHz
OBFIN       1150.0 Hz
POINT        16384
FREQU       6013.2 Hz
SCANS           16
ACQTM        2.725 sec
PD           1.551 sec
PW1            5.8 us
IRNUC  1H
CTEMP         19.7 c
SLVNT  CDCL3
EXREF         0.00 ppm
BF            0.09 Hz
RGAIN           12
```

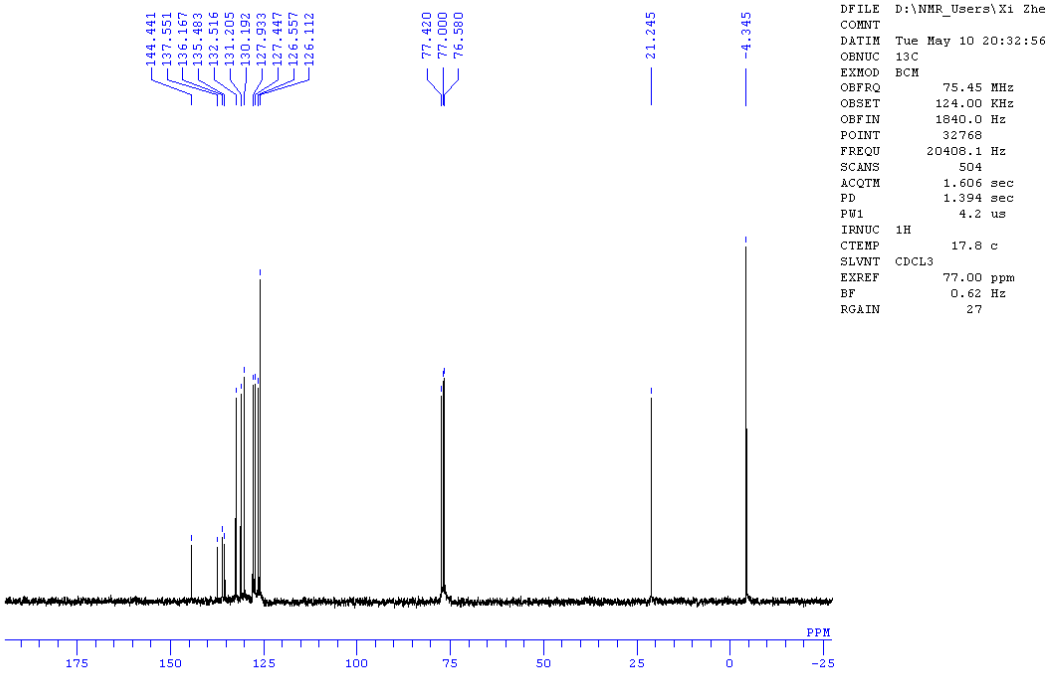


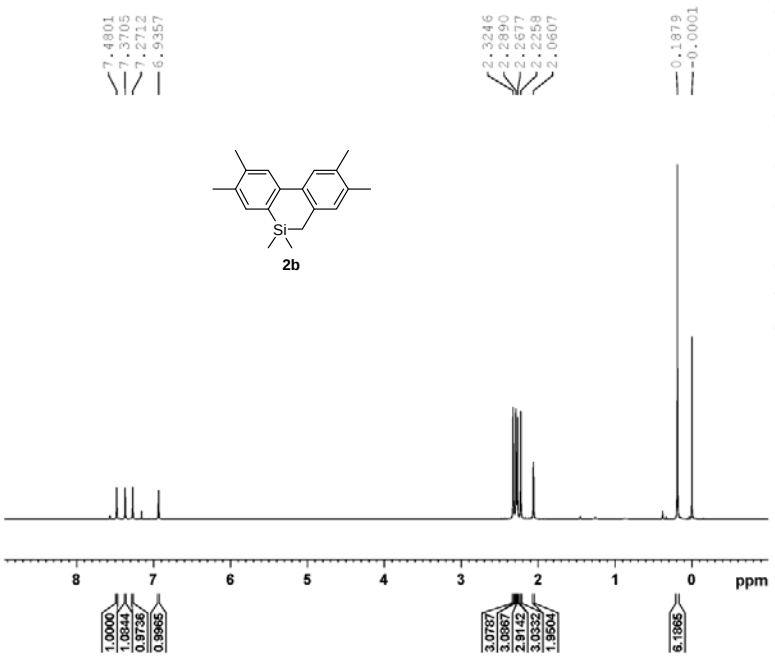

```
DFILE  C:\WINNMR95\COMMON\
COMNT
DATIM  Sat Apr 16 20:32:06
OBNUC  13C
EXMOD  BCM
OBFRQ        75.45 MHz
OBSET       124.00 KHz
OBFIN       1840.0 Hz
POINT        32768
FREQU      20408.1 Hz
SCANS          473
ACQTM        1.606 sec
PD           1.394 sec
PW1            4.2 us
IRNUC  1H
CTEMP         20.3 c
SLVNT  CDCL3
EXREF         0.00 ppm
BF            0.62 Hz
RGAIN           26
```

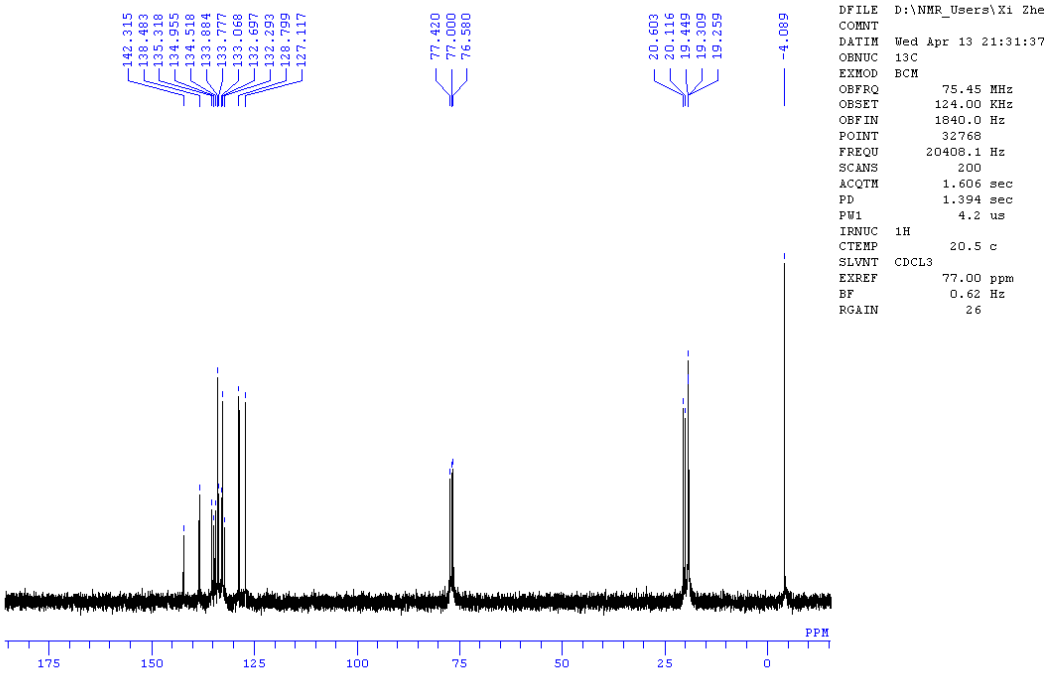


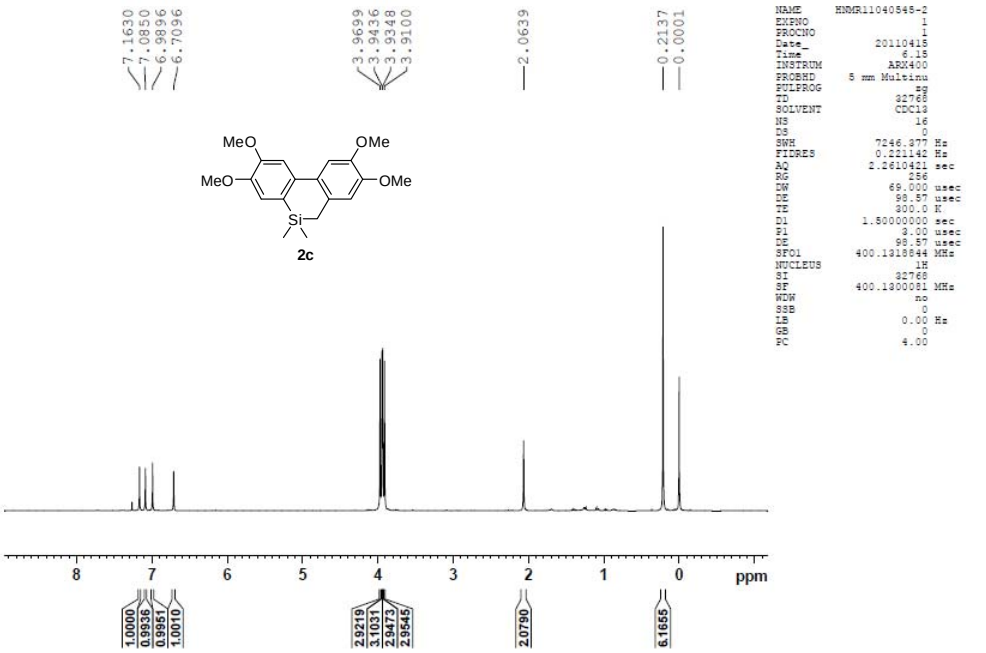


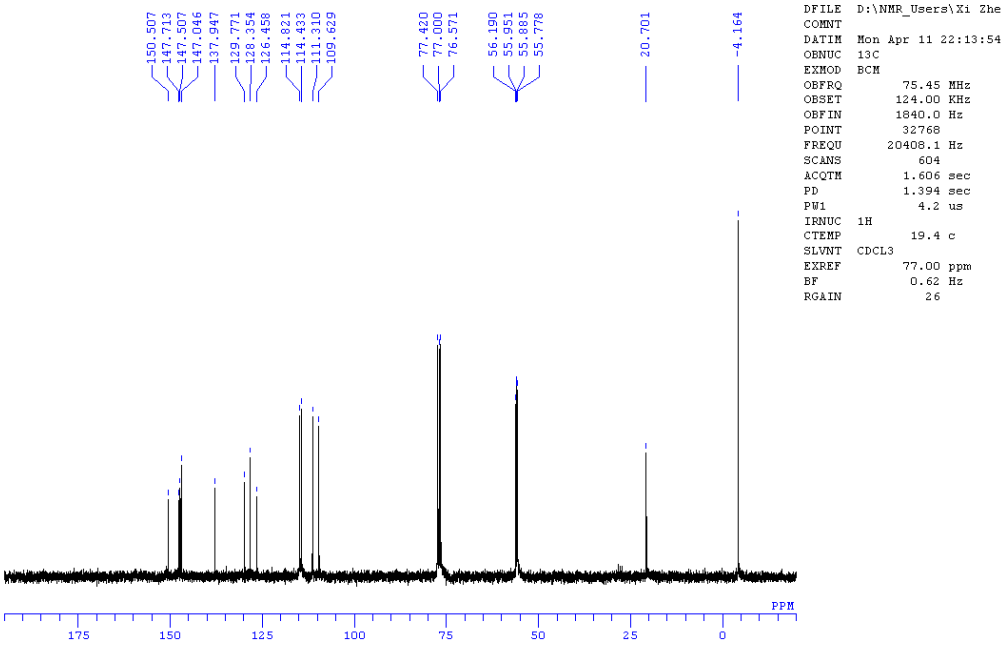

7.6965
7.6767
7.6069
7.5878
7.5494
7.5281
7.4866
7.4678
7.4485
7.3158
7.2980
7.2798
7.2604
7.2534
7.2384
7.2248
7.2195
7.2021
7.1892
7.1741
7.1706
7.1555
2.1664
0.2142
0.0002
Si
2a
NAME HNMR11050224
EXPNO 1
PROCNO 1
Date_ 20110507
Time 5.08
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8064.516 Hz
FIDRES 0.246110 Hz
AQ 2.0316660 sec
RG 512
DW 62.000 usec
DE 88.57 usec
TE 300.0 K
D1 2.00000000 sec
P1 3.00 usec
DE 88.57 usec
SFO1 400.1319527 MHz
NUCLEUS 1H
SI 16384
SF 400.1300235 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.0000
1.0272
0.9986
1.0402
1.0751
3.1931
2.0860
6.1054

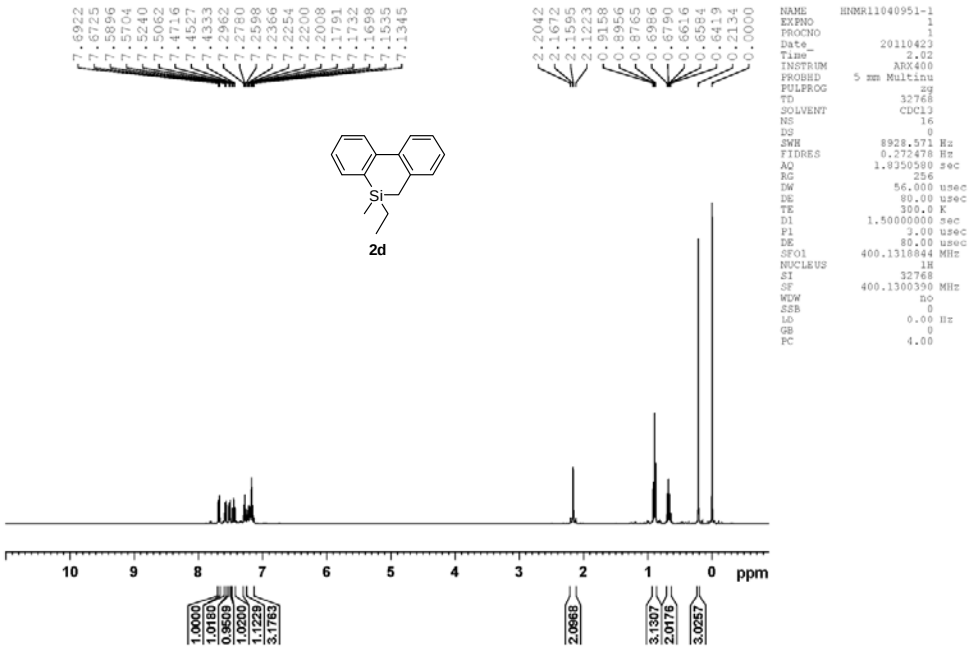

144.441
137.551
136.167
135.483
132.516
131.205
130.192
127.933
127.447
126.557
126.112
77.420
77.000
76.580
21.245
-4.345
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Tue May 10 20:32:56
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 504
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 17.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 27
PPM
175 150 125 100 75 50 25 0 -25

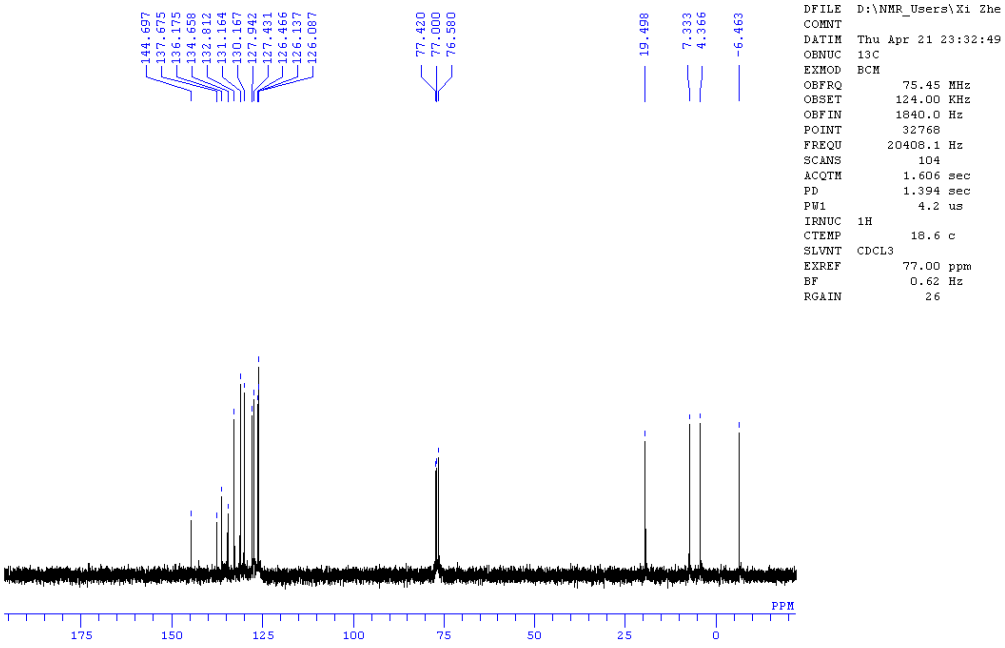

7.4801
7.3705
7.2712
6.9357
2.3246
2.2890
2.2677
2.2258
2.0607
0.1879
-0.0001
NAME HNMR11040545-1
EXPNO 1
PROCNO 1
Date_ 20110415
Time_ 6.10
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.26104421 sec
RG 180
DW 69.000 usec
DE 98.57 usec
TE 300.0 K
D1 1.50000000 sec
P1 2.00 usec
DE 98.57 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300504 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
Si
2b
8
7
6
5
4
3
2
1
0
ppm
1.0000
1.0844
0.9736
0.9965
3.0787
3.0867
2.9142
3.0332
1.9504
6.1865

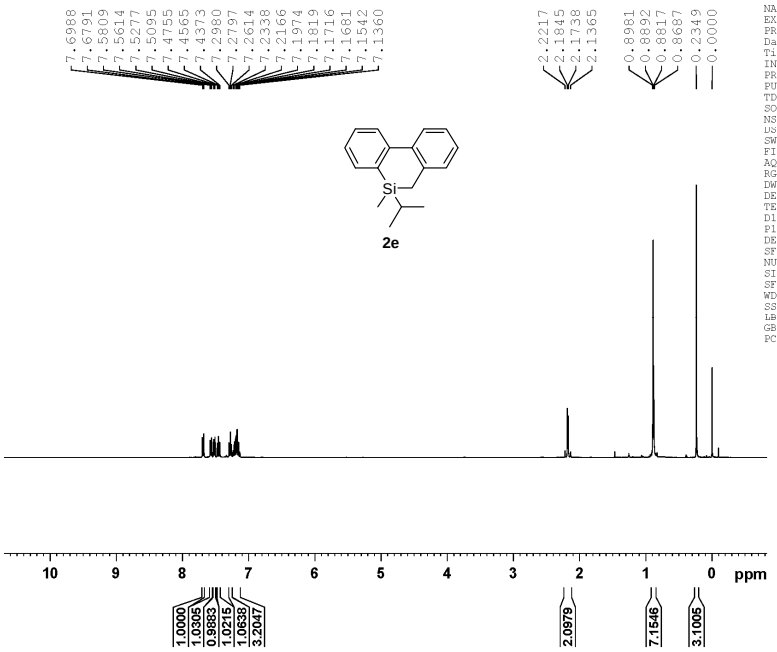

142.315
138.483
135.318
134.955
134.518
133.884
133.777
133.068
132.697
132.293
128.799
127.117
77.420
77.000
76.580
20.603
20.116
19.449
19.309
19.259
-4.089
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Wed Apr 13 21:31:37
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 200
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26
PPM
175
150
125
100
75
50
25
0


7.1630
7.0850
6.9896
6.7096
3.9699
3.9436
3.9348
3.9100
2.0639
0.2137
0.0001
MeO
OMe
MeO
OMe
Si
2c
NAME HNMR11040848-2
EXPNO 1
PROCNO 1
Date_ 20110415
Time 6.15
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 256
DW 69.000 usec
DE 98.57 usec
TE 300.0 K
D1 1.50000000 sec
P1 8.00 usec
DE 98.57 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300081 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
8
7
6
5
4
3
2
1
0
ppm
1.0000
0.9936
0.9951
1.0010
2.9219
3.1031
2.9473
2.9545
2.0790
6.1655

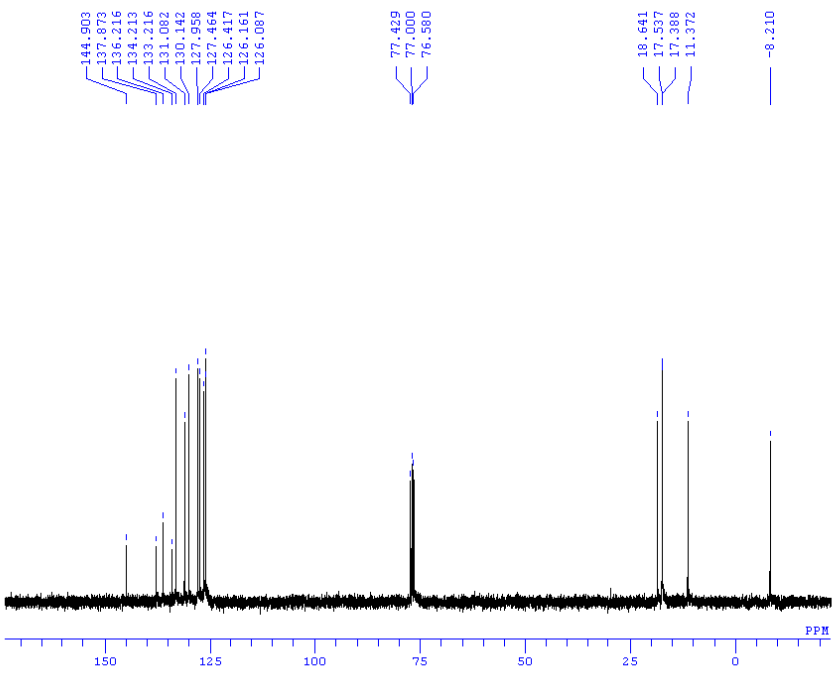

150.507
147.713
147.507
147.046
137.947
129.771
128.354
126.458
114.821
114.433
111.310
109.629
77.420
77.000
76.571
56.190
55.951
55.885
55.778
20.701
-4.164
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Mon Apr 11 22:13:54
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 604
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26
PPM
175
150
125
100
75
50
25
0


7.6922
7.6725
7.5896
7.5704
7.5240
7.5062
7.4716
7.4527
7.4333
7.2962
7.2780
7.2598
7.2366
7.2254
7.2200
7.2008
7.1791
7.1732
7.1698
7.1535
7.1345
2.2042
2.1672
2.1595
2.1223
2.0158
0.8956
0.8765
0.6986
0.6790
0.6616
0.6554
0.6419
0.2134
0.0000
NAME HNMR11040951-1
EXPNO 1
PROCNO 1
Date_ 20110423
Time_ 2.02
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8928.571 Hz
FIDRES 0.272478 Hz
AQ 1.9350580 sec
RG 256
DW 56.000 usec
DE 80.00 usec
TE 300.0 K
D1 1.50000000 sec
P1 3.00 usec
DE 80.00 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300390 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
Si
2d
10
9
8
7
6
5
4
3
2
1
0
ppm
1.0000
1.0180
0.9609
1.0200
1.1228
3.1763
2.0968
3.1307
2.0178
3.0257

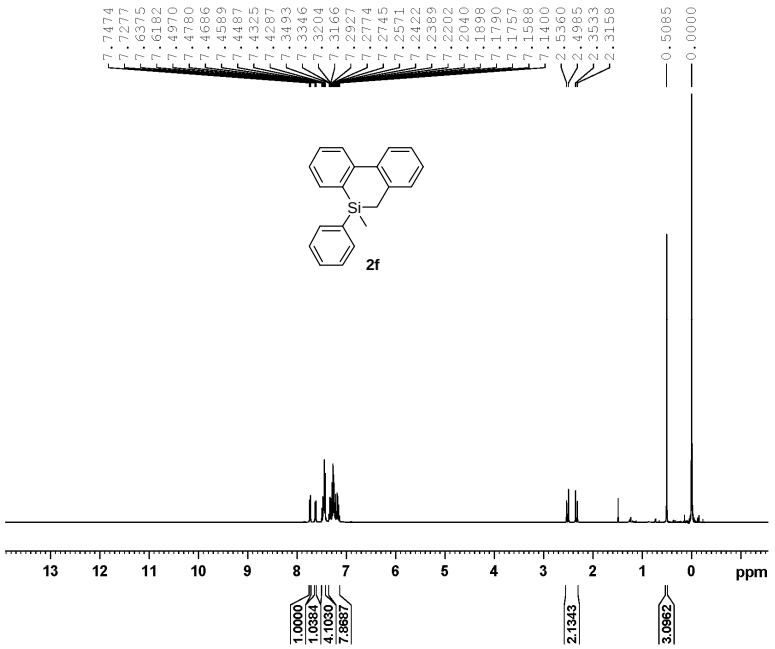

144.697
137.675
136.175
134.658
132.812
131.164
130.167
127.942
127.431
126.466
126.137
126.087
77.420
77.000
76.580
19.498
7.333
4.366
-6.463
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Thu Apr 21 23:32:49
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 104
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26
PPM
175
150
125
100
75
50
25
0



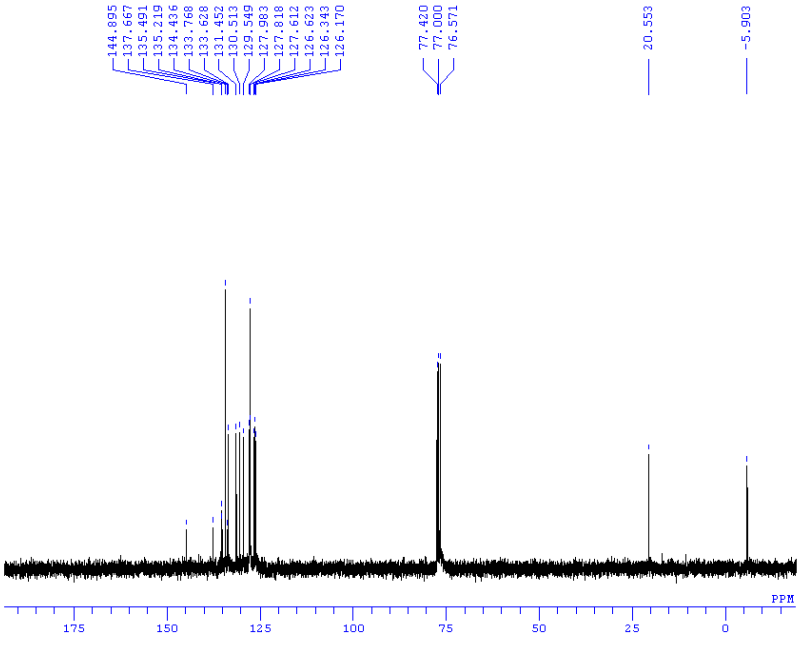

```
NAME        HNMR11031075-2
EXPNO                    1
PROCNO                   1
Date_             20110325
Time                 2.57
INSTRUM           ARX400
PROBHD     5 mm Multinu
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              8064.516 Hz
FIDRES           0.246110 Hz
AQ               2.0316660 sec
RG                    180
DW                62.000 usec
DE                77.50 usec
TE                300.0 K
D1               2.00000000 sec
P1                  3.00 usec
DE                77.50 usec
SFO1          400.1321971 MHz
NUCLEUS               1H
SI                 16384
SF            400.1300356 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



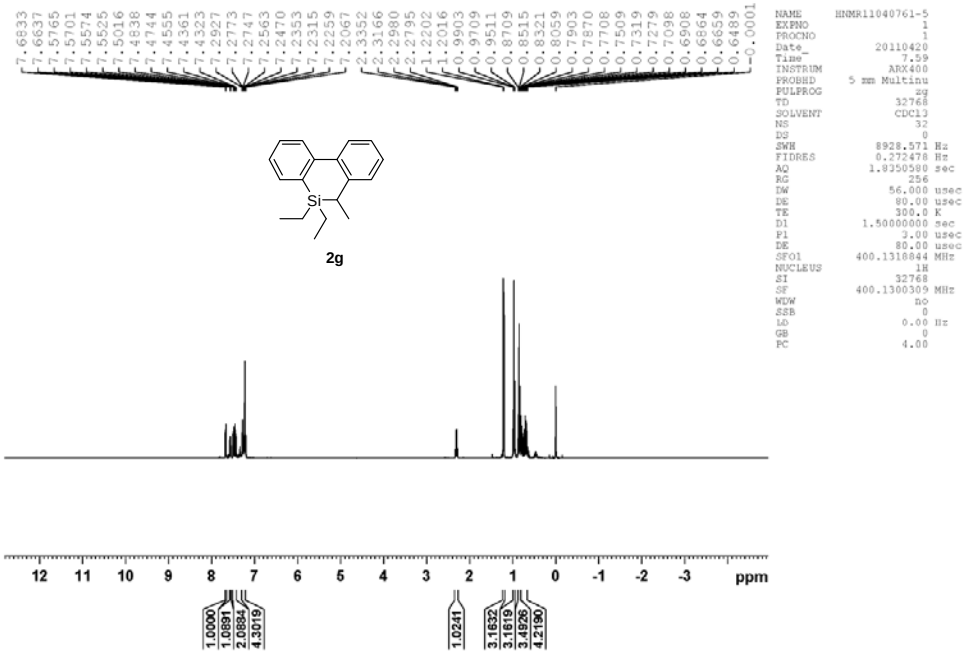

```
DFILE   D:\NMR_Users\Xi Zhe
COMNT
DATIM   Wed Mar 23 20:56:52
OBNUC   13C
EXMOD   BCM
OBFRQ         75.45 MHz
OBSET        124.00 KHz
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           161
ACQTM          1.606 sec
PD             1.394 sec
PW1              4.2 us
IRNUC   1H
CTEMP          24.0 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.62 Hz
RGAIN            26
```

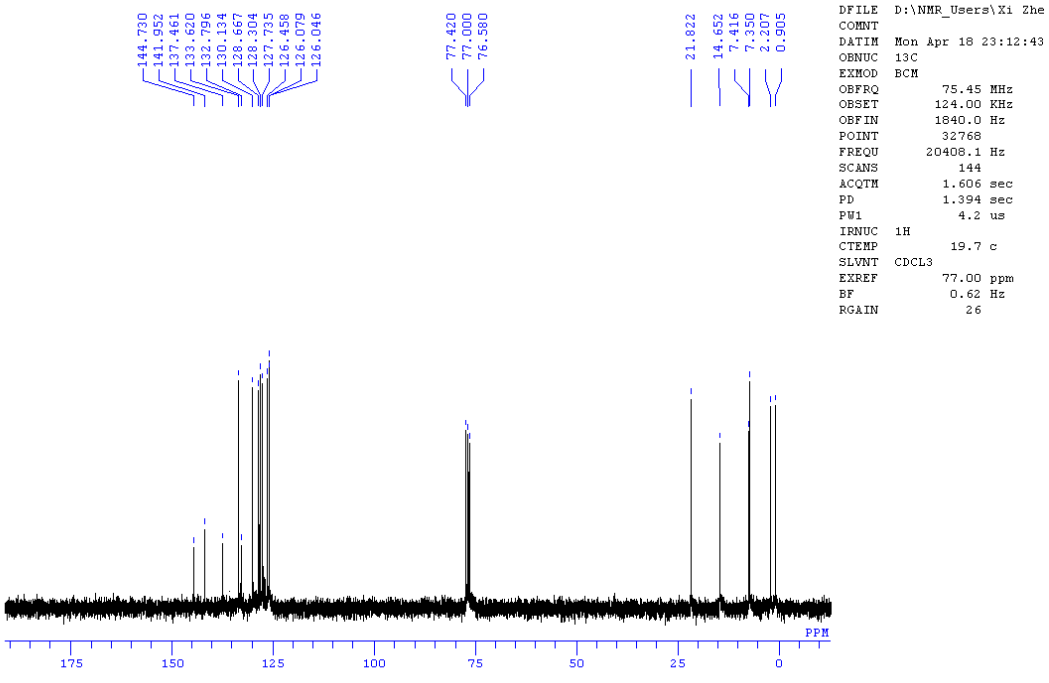


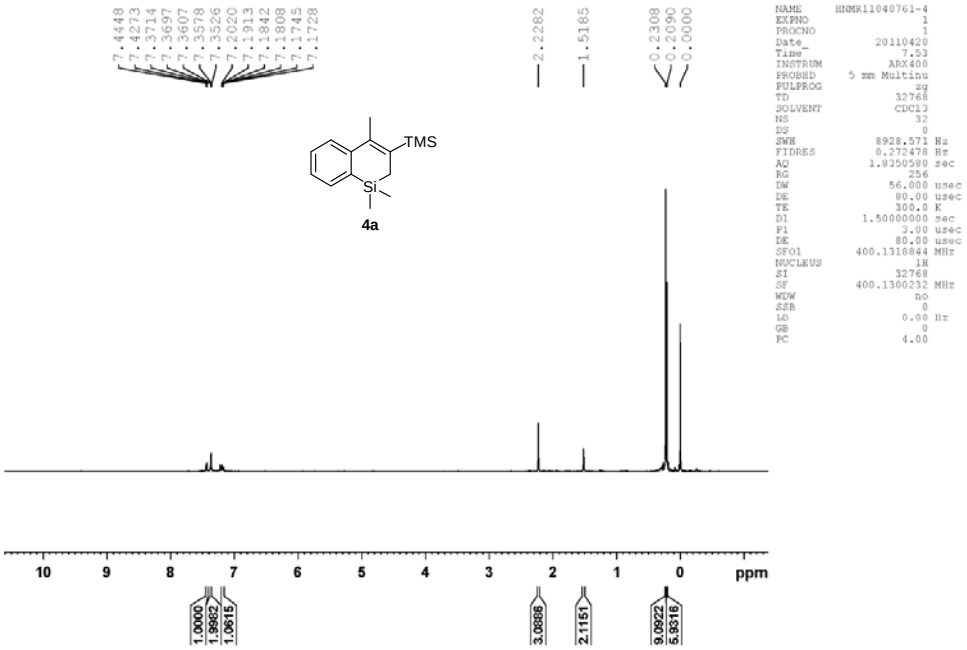

```
NAME        HNMR11040951-2
EXPNO                    1
PROCNO                   1
Date_             20110423
Time                 2.07
INSTRUM           ARX400
PROBHD      5 mm Multinu
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              8928.571 Hz
FIDRES           0.272478 Hz
AQ               1.8350580 sec
RG                    256
DW                56.000 usec
DE                80.00 usec
TE                300.0 K
D1             1.50000000 sec
P1                 3.00 usec
DE                80.00 usec
SFO1         400.1318844 MHz
NUCLEUS              1H
SI                  32768
SF           400.1300251 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 4.00
```

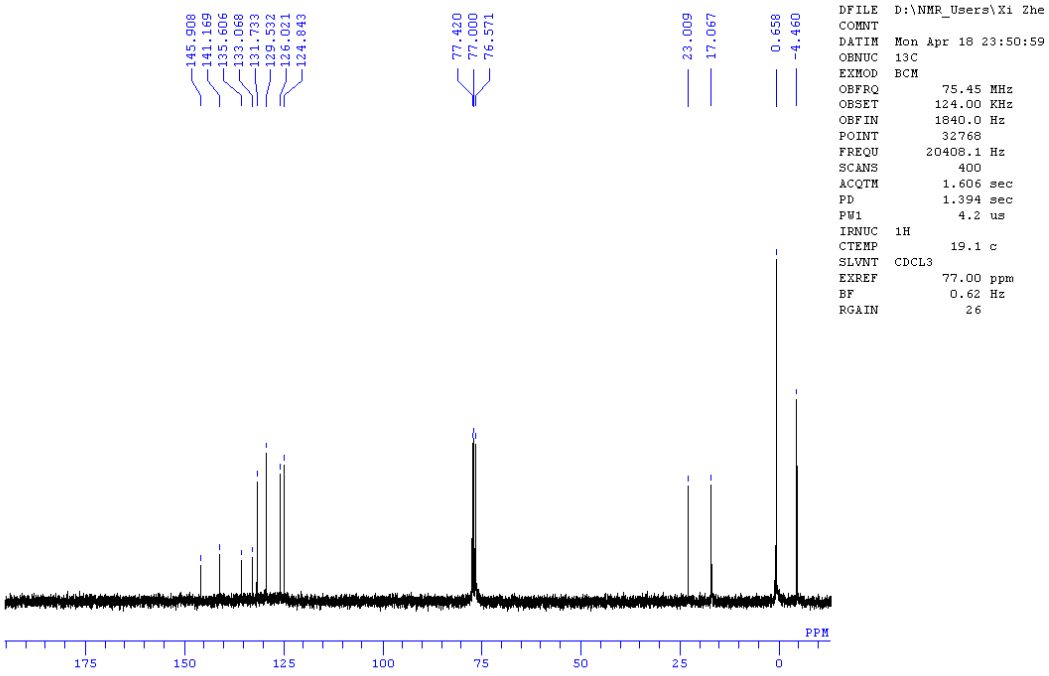


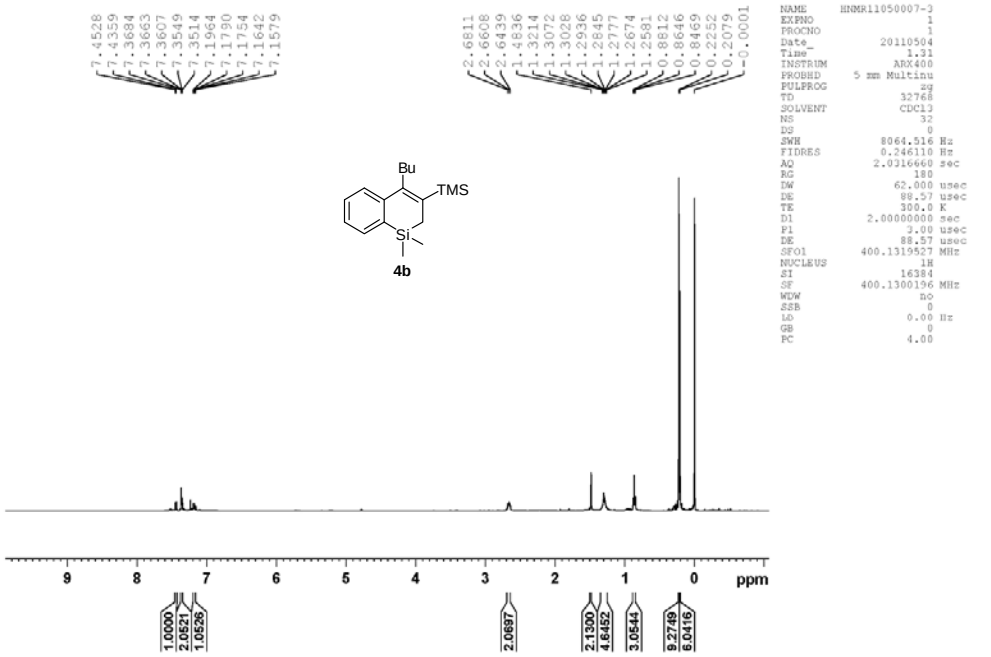

```
DFILE   D:\NMR_Users\Xi Zhe
COMNT
DATIM   Thu Apr 21 23:54:41
OBNUC   13C
EXMOD   BCM
OBFRQ        75.45 MHz
OBSET       124.00 KHz
OBFIN      1840.0 Hz
POINT       32768
FREQU      20408.1 Hz
SCANS          336
ACQTM        1.606 sec
PD           1.394 sec
PW1            4.2 us
IRNUC   1H
CTEMP         18.6 c
SLVNT   CDCL3
EXREF        77.00 ppm
BF            0.62 Hz
RGAIN           26
```

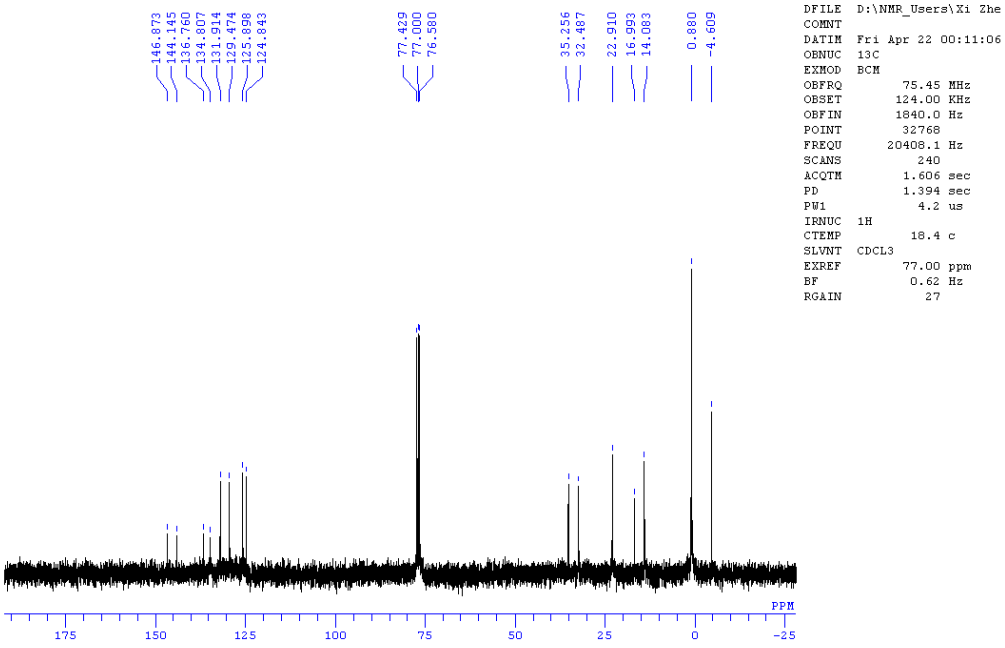

7.6833
7.6637
7.5765
7.5701
7.5574
7.5525
7.5016
7.4838
7.4744
7.4555
7.4361
7.4323
7.2927
7.2773
7.2747
7.2563
7.2470
7.2353
7.2315
7.2259
7.2067
2.3552
2.3166
2.2980
2.2795
1.2202
1.2016
0.9903
0.9709
0.9511
0.8709
0.8515
0.8321
0.8059
0.7903
0.7870
0.7708
0.7509
0.7319
0.7279
0.7098
0.6908
0.6664
0.6659
0.6489
-0.0001
Si
2g
NAME HNMR11040761-5
EXPNO 1
PROCNO 1
Date_ 20110420
Time_ 7.59
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8928.571 Hz
FIDRES 0.272478 Hz
AQ 1.9350580 sec
RG 256
DW 56.000 usec
DE 80.00 usec
TE 300.0 K
D1 1.50000000 sec
P1 2.00 usec
DE 80.00 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300309 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 ppm
1.0000
1.0891
2.0884
4.3019
1.0241
3.1632
3.1619
3.4926
4.2190


144.730
141.952
137.461
133.620
132.796
130.134
128.667
128.304
127.735
126.458
126.079
126.046
77.420
77.000
76.580
21.822
14.652
7.416
7.350
2.207
0.905
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Mon Apr 18 23:12:43
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 144
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26
PPM
175 150 125 100 75 50 25 0


7.4448
7.4273
7.3714
7.3697
7.3607
7.3578
7.3526
7.2020
7.1913
7.1842
7.1808
7.1745
7.1728
2.2282
1.5165
0.2308
0.2090
0.0000
TMS
Si
4a
NAME HNMR11040761-4
EXPNO 1
PROCNO 1
Date_ 20110420
Time_ 7.53
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8928.571 Hz
FIDRES 0.272478 Hz
AQ 1.9350580 sec
RG 256
DW 56.000 usec
DE 80.00 usec
TE 300.0 K
D1 1.50000000 sec
P1 2.00 usec
DE 80.00 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300232 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.0000
1.9962
1.0615
3.0698
2.1151
9.0922
5.9316


145.908
141.169
135.606
133.088
131.733
129.532
126.021
124.843
77.420
77.000
76.571
23.009
17.067
0.658
-4.460
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Mon Apr 18 23:50:59
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 400
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 19.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26
PPM
175
150
125
100
75
50
25
0


7.4528
7.4359
7.3684
7.3663
7.3607
7.3549
7.3514
7.1964
7.1790
7.1754
7.1642
7.1579
2.6811
2.6608
2.6439
1.4836
1.3214
1.3072
1.3028
1.2936
1.2845
1.2777
1.2674
1.2581
0.8812
0.8646
0.8469
0.2252
0.2079
-0.0001
NAME HNMR11050007-3
EXPNO 1
PROCNO 1
Date_ 20110504
Time_ 1.31
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8064.516 Hz
FIDRES 0.246110 Hz
AQ 2.0316660 sec
RG 180
DW 62.000 usec
DE 88.57 usec
TE 300.0 K
D1 2.00000000 sec
P1 2.00 usec
DE 88.57 usec
SFO1 400.1319527 MHz
NUCLEUS 1H
SI 16384
SF 400.1300196 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
Bu
TMS
Si
4b
9
8
7
6
5
4
3
2
1
0
ppm
1.0000
2.0621
1.0536
2.0697
2.1390
4.8452
3.0544
9.2749
8.0416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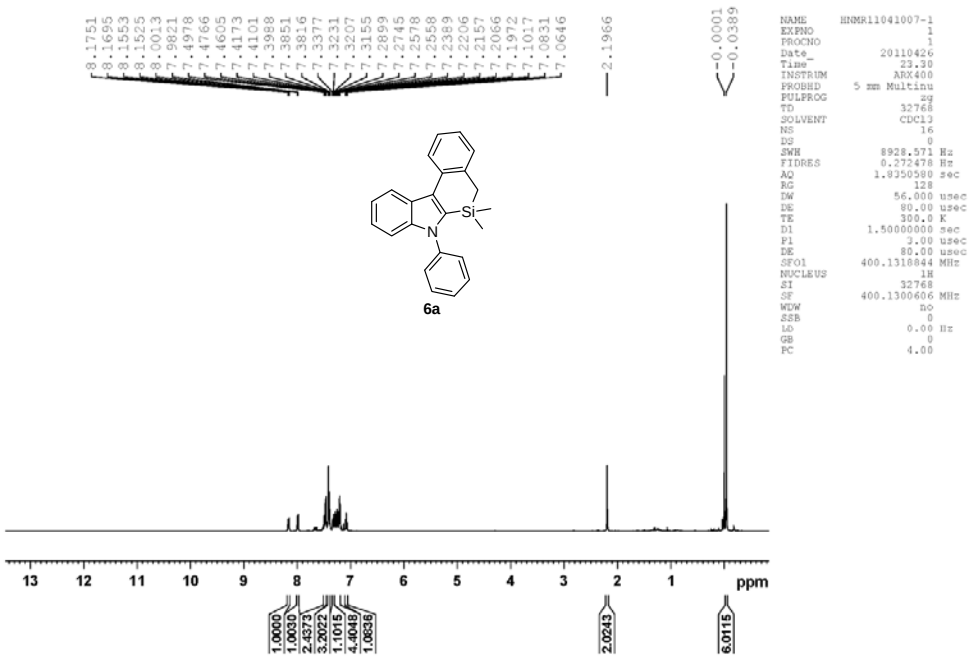
144.145
136.760
134.807
131.914
129.474
125.898
124.843
77.429
77.000
76.580
35.256
32.487
22.910
16.993
14.083
0.880
-4.609
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Fri Apr 22 00:11:06
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 240
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 18.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 27
PPM
175
150
125
100
75
50
25
0
-25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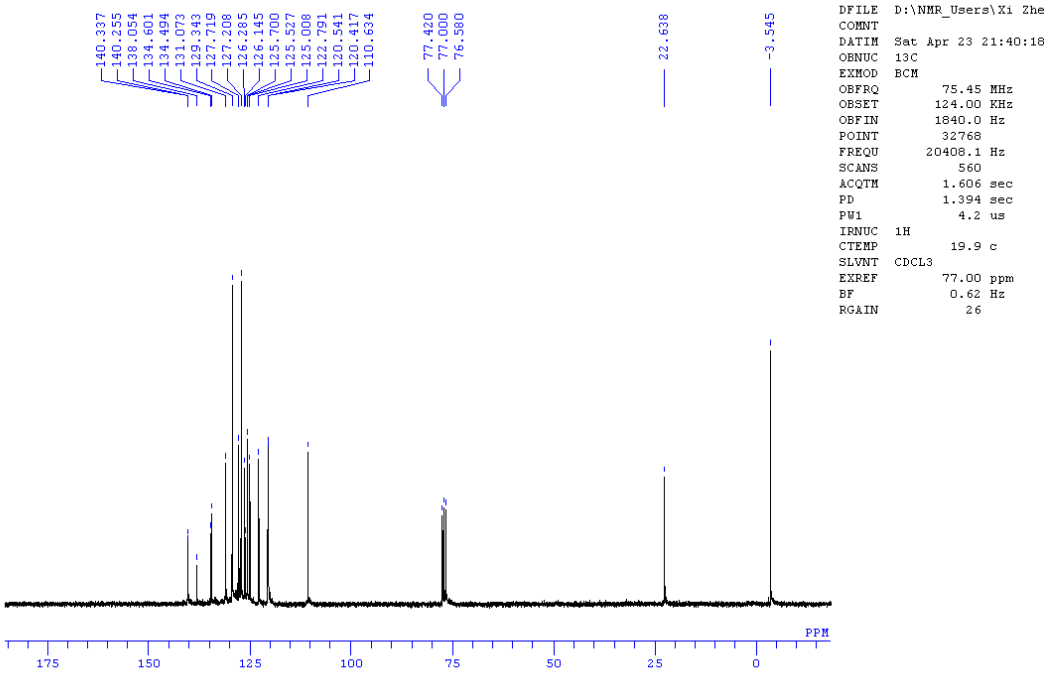
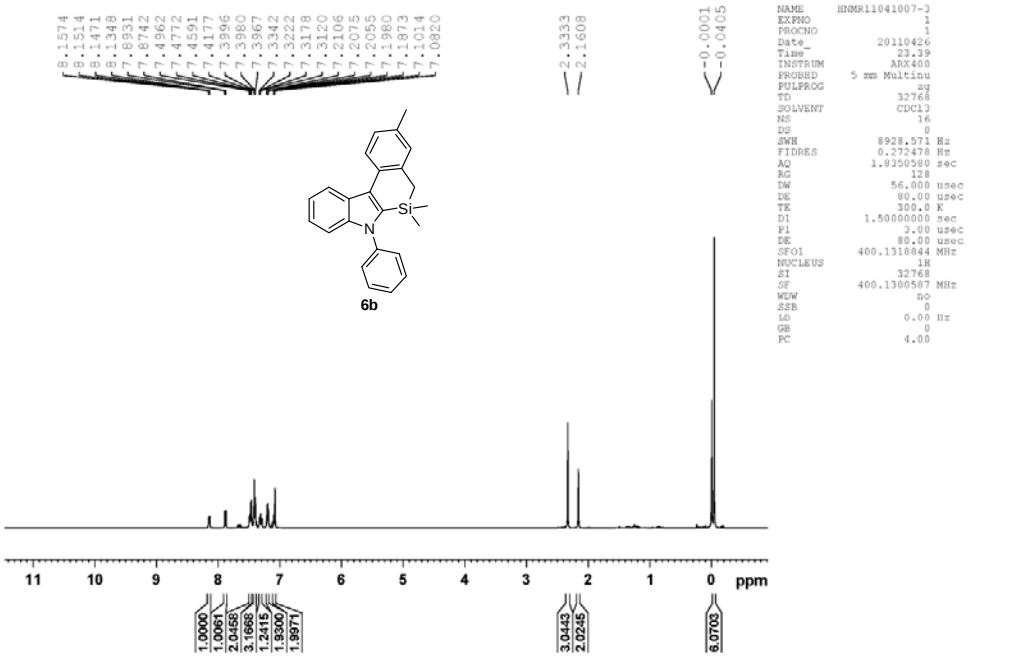
8.1695
8.1553
8.1525
8.0013
7.9821
7.4978
7.4766
7.4605
7.4173
7.4101
7.3988
7.3851
7.3816
7.3377
7.3231
7.3207
7.3155
7.2899
7.2745
7.2578
7.2558
7.2389
7.2206
7.2157
7.2066
7.1972
7.1017
7.0831
7.0646
2.1966
0.0001
-0.0389
Si
N
6a
NAME HNMR11041007-1
EXPNO 1
PROCNO 1
Date_ 20110426
Time_ 23.30
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8928.571 Hz
FIDRES 0.272478 Hz
AQ 1.9350580 sec
RG 128
DW 56.000 usec
DE 80.00 usec
TE 300.0 K
D1 1.50000000 sec
P1 2.00 usec
DE 80.00 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300606 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.0000
1.0030
2.4373
3.2022
1.1015
4.4048
1.0036
2.0243
6.0115


140.337
140.255
138.054
134.601
134.494
131.073
129.343
127.719
127.208
126.285
126.145
125.700
125.527
125.008
122.791
120.541
120.417
110.634
77.420
77.000
76.580
22.638
-3.545
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Sat Apr 23 21:40:18
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 560
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26
PPM
175 150 125 100 75 50 25 0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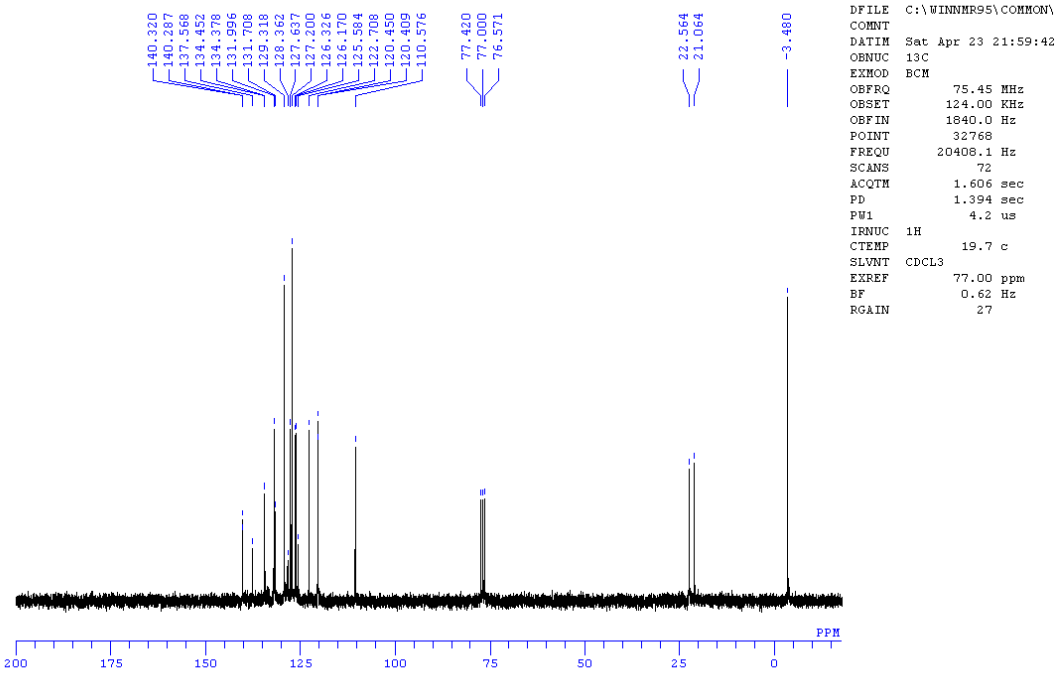
8.1514
8.1471
8.1348
7.8931
7.8742
7.4962
7.4772
7.4591
7.4177
7.3996
7.3980
7.3967
7.3342
7.3222
7.3178
7.3120
7.2106
7.2075
7.2055
7.1980
7.1873
7.1014
7.0820
2.3333
2.1608
-0.0001
-0.0405
Si
N
6b
NAME HNMR11041007-3
EXPNO 1
PROCNO 1
Date_ 20110426
Time_ 23.39
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8928.571 Hz
FIDRES 0.272478 Hz
AQ 1.9350580 sec
RG 128
DW 56.000 usec
DE 80.00 usec
TE 300.0 K
D1 1.50000000 sec
P1 2.00 usec
DE 80.00 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300587 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.0000
1.0061
2.0458
3.1668
1.2415
1.9300
1.9971
3.0443
2.0245
6.0703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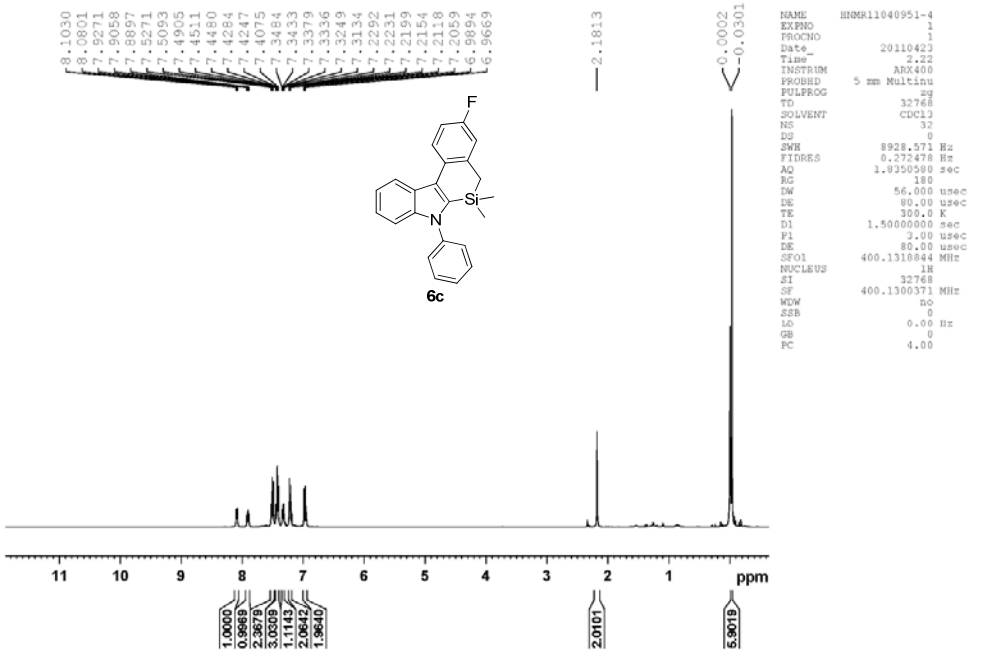
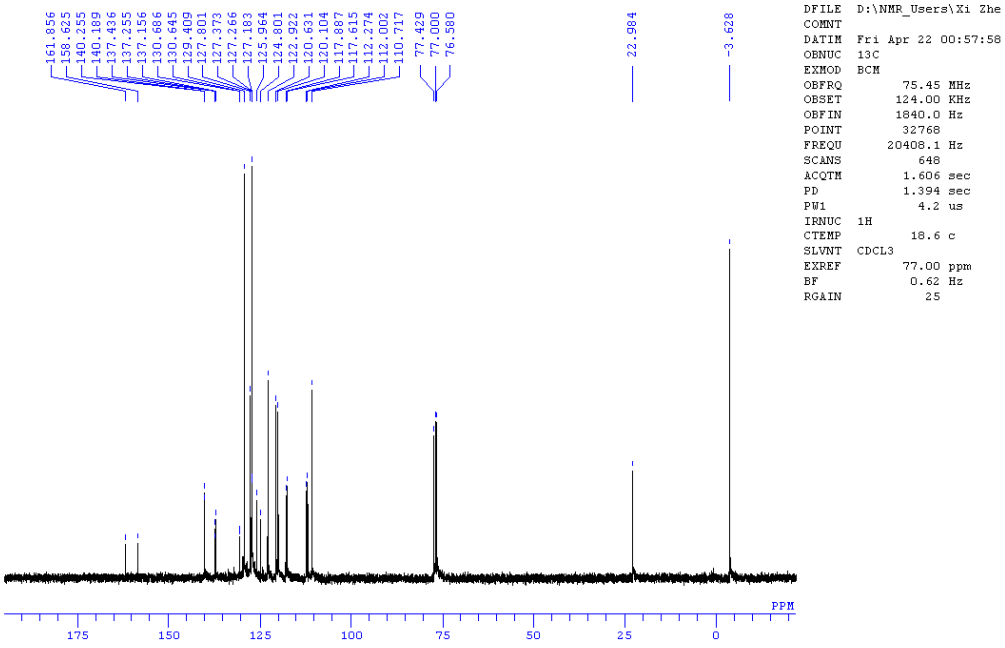
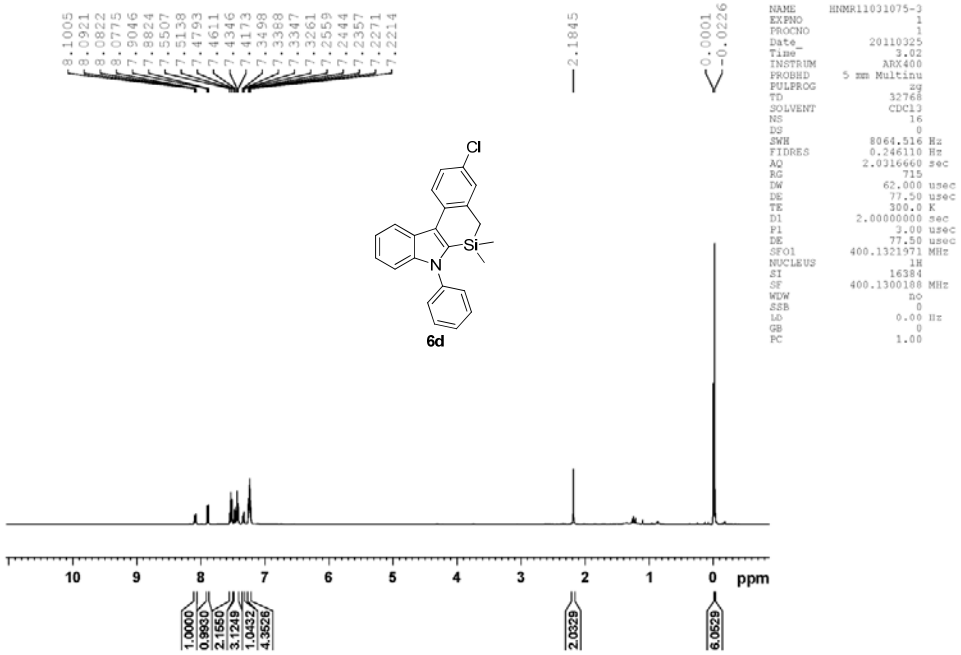
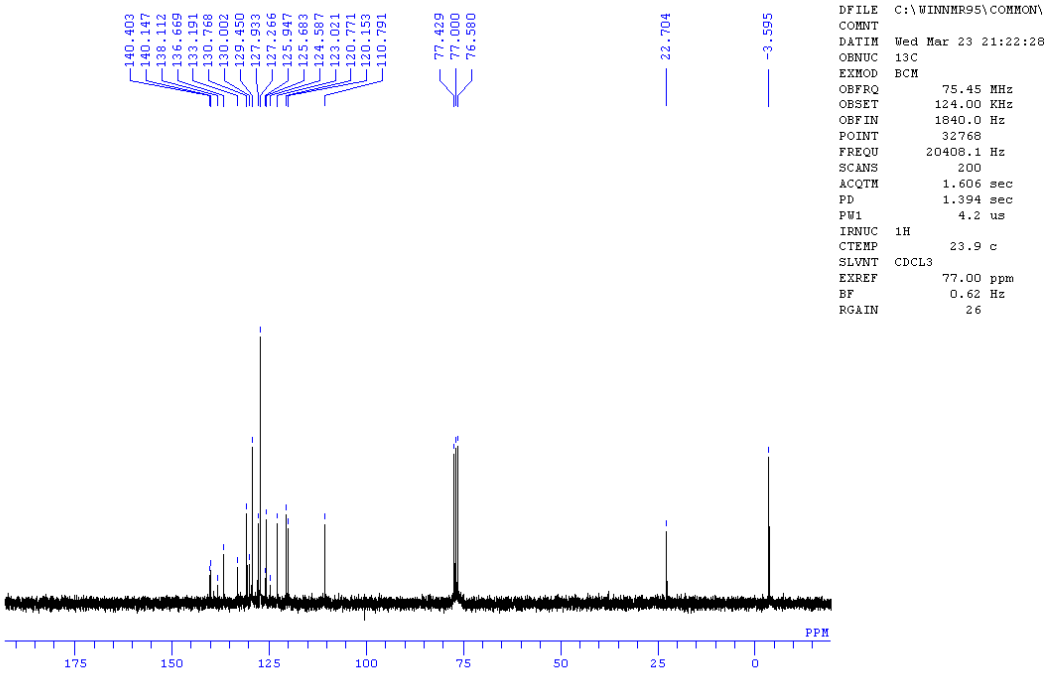
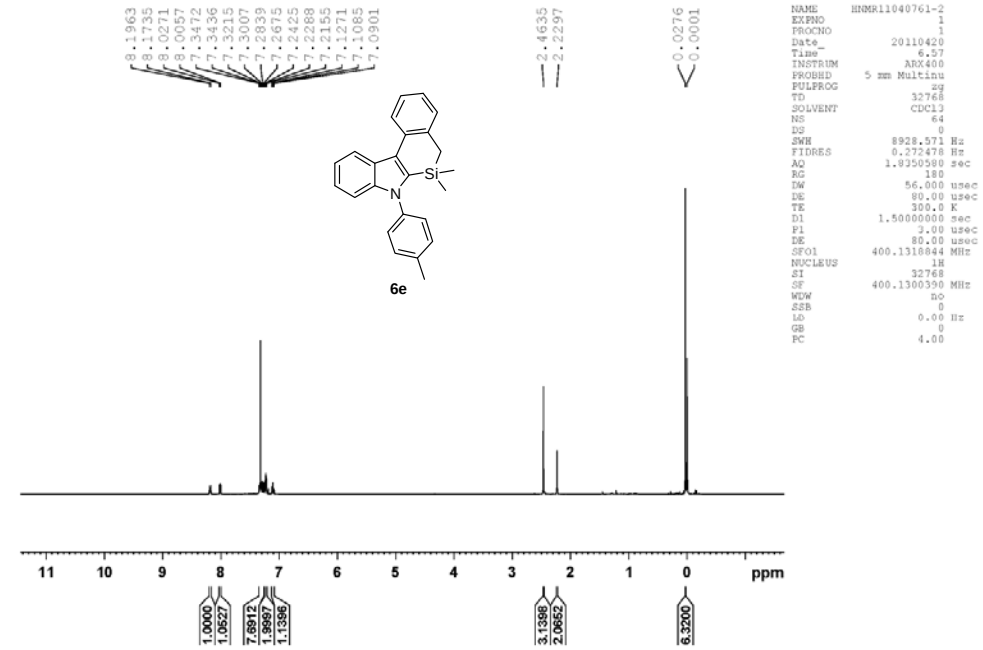
140.287
137.568
134.452
134.378
131.996
131.708
129.318
128.362
127.637
127.200
126.326
126.170
125.584
122.708
120.450
120.409
110.576
77.420
77.000
76.571
22.564
21.064
-3.480
DFILE C:\WINNMR95\COMMON\
COMNT
DATIM Sat Apr 23 21:59:42
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 72
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 27
PPM
200 175 150 125 100 75 50 25 0

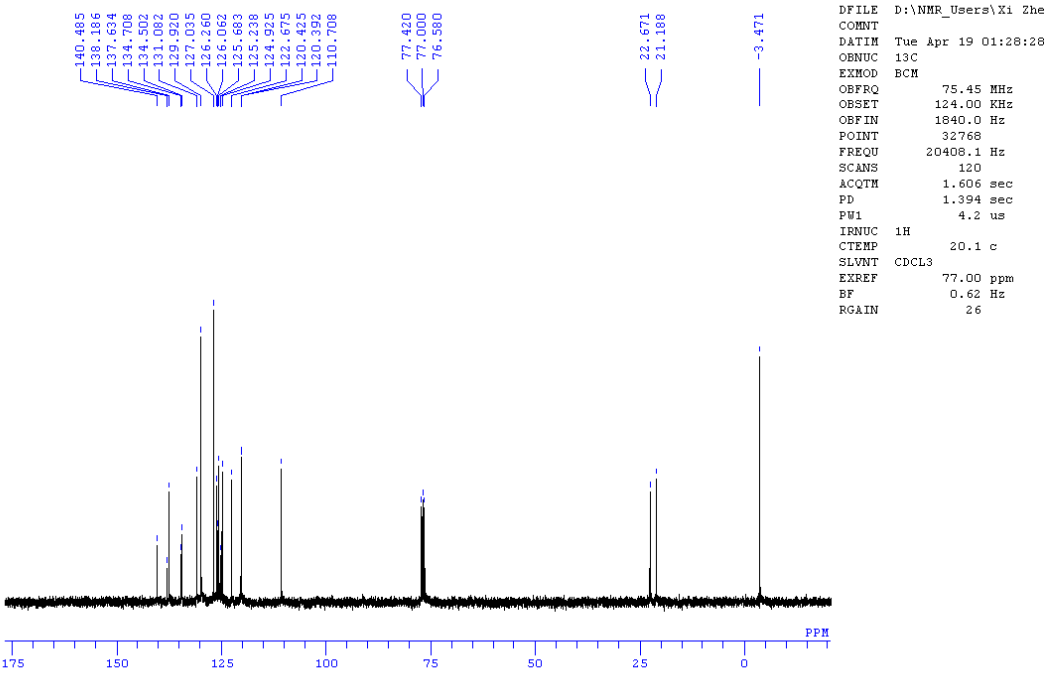


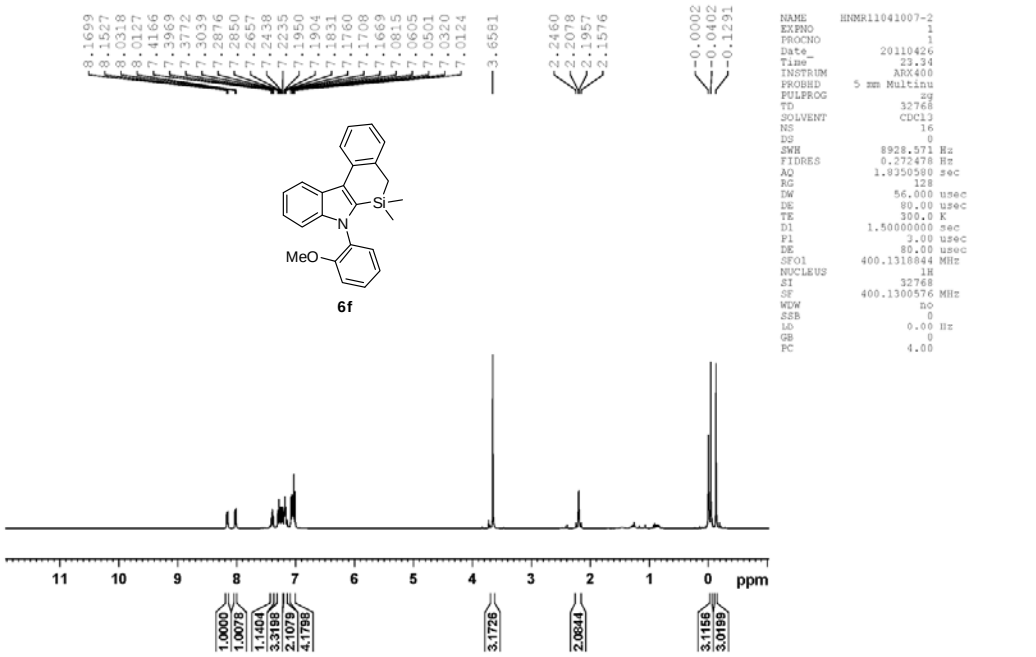



8.1005
8.0921
8.0822
8.0775
7.9046
7.8824
7.5507
7.5138
7.4793
7.4611
7.4346
7.4173
7.3498
7.3388
7.3347
7.3261
7.2559
7.2444
7.2357
7.2271
7.2214
2.1845
0.0001
-0.0226
Cl
Si
N
6d
NAME HNMR11031075-3
EXPNO 1
PROCNO 1
Date_ 20110325
Time_ 3.02
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8064.516 Hz
FIDRES 0.246110 Hz
AQ 2.0316660 sec
RG 715
DW 62.000 usec
DE 77.50 usec
TE 300.0 K
D1 2.00000000 sec
P1 2.00 usec
DE 77.50 usec
SFO1 400.1321971 MHz
NUCLEUS 1H
SI 16384
SF 400.1300180 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
1.0000
0.9930
2.1660
3.1249
1.0432
4.3526
2.0329
6.0529


140.403
140.147
138.112
136.669
133.191
130.768
130.002
129.450
127.933
127.266
125.947
125.683
124.587
123.021
120.771
120.153
110.791
77.429
77.000
76.580
22.704
-3.595
DFILE C:\WINNMR95\COMMON\
COMNT
DATIM Wed Mar 23 21:22:28
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 200
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26
PPM
175 150 125 100 75 50 25 0


8.1963
8.1735
8.0271
8.0057
7.3472
7.3436
7.3215
7.3007
7.2839
7.2675
7.2425
7.2288
7.2155
7.1271
7.1085
7.0901
2.4635
2.2297
0.0276
0.0001
Si
N
6e
NAME HNMR11040761-2
EXPNO 1
PROCNO 1
Date_ 20110420
Time_ 6.57
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 8928.571 Hz
FIDRES 0.272478 Hz
AQ 1.9350580 sec
RG 180
DW 56.000 usec
DE 80.00 usec
TE 300.0 K
D1 1.50000000 sec
P1 2.00 usec
DE 80.00 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300390 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.0000
1.0527
7.6912
1.9997
1.1396
3.1398
2.0652
6.3200


140.485
138.186
137.634
134.708
134.502
131.082
129.920
127.035
126.260
126.062
125.683
125.238
124.925
122.675
120.425
120.392
110.708
77.420
77.000
76.580
22.671
21.188
-3.471
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Tue Apr 19 01:28:28
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 120
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 20.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26
PPM
175 150 125 100 75 50 25 0


8.1699
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8.0318
8.0127
7.4166
7.3969
7.3772
7.3039
7.2876
7.2850
7.2657
7.2438
7.2235
7.1950
7.1904
7.1831
7.1760
7.1708
7.1669
7.0815
7.0605
7.0501
7.0320
7.0124
3.6581
2.2460
2.2078
2.1957
2.1576
-0.0002
-0.0402
-0.1291
Si
N
MeO
6f
NAME HNMR11041007-2
EXPNO 1
PROCNO 1
Date_ 20110426
Time_ 23.34
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8928.571 Hz
FIDRES 0.272478 Hz
AQ 1.9350580 sec
RG 128
DW 56.000 usec
DE 80.00 usec
TE 300.0 K
D1 1.50000000 sec
P1 3.00 usec
DE 80.00 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300576 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
11 10 9 8 7 6 5 4 3 2 1 0 ppm
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1.0078
1.1404
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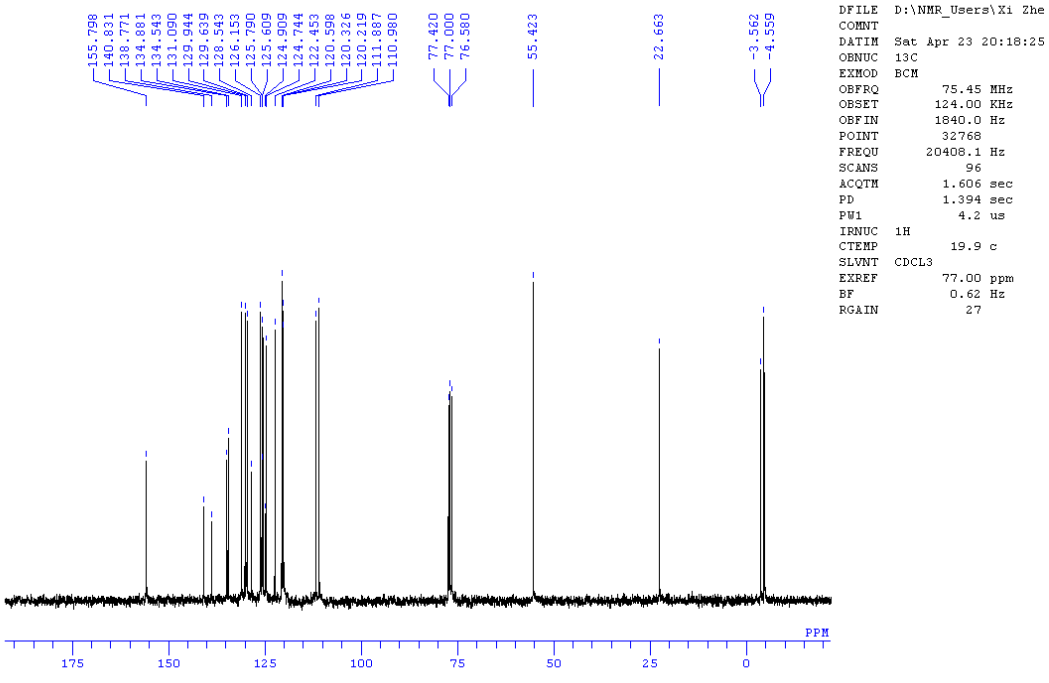
2.1079
4.1798
3.1726
2.0644
3.1156
3.0199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138.771
134.881
134.543
131.090
129.944
129.639
128.543
126.153
125.790
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124.909
124.744
122.453
120.598
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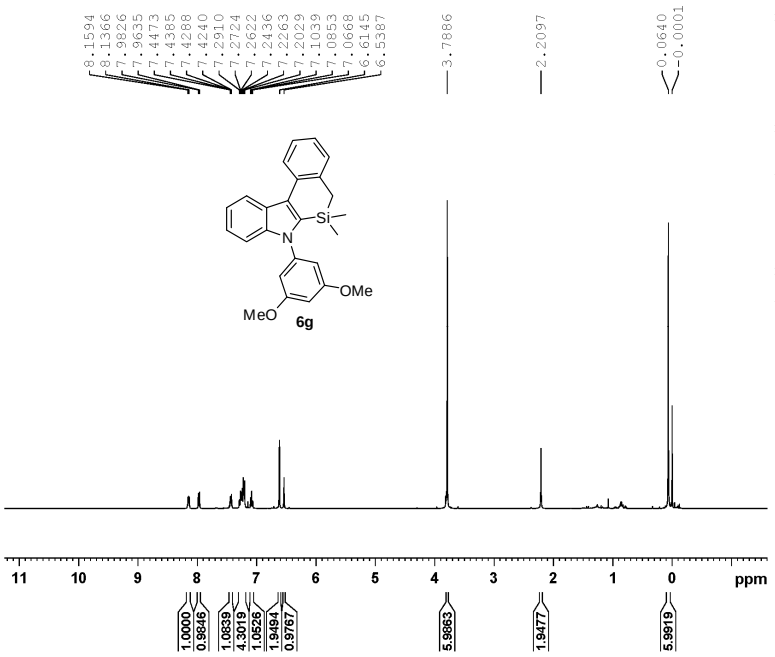
111.887
110.980
77.420
77.000
76.580
55.423
22.663
-3.562
-4.559
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Sat Apr 23 20:18:25
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 96
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 27
PPM
175 150 125 100 75 50 25 0

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EXPNO                    1
PROCNO                   1
Date_             20110415
Time                 6.22
INSTRUM           ARX400
PROBHD     5 mm Multinu
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              7246.377 Hz
FIDRES           0.221142 Hz
AQ               2.2610421 sec
RG                    256
DW                 69.000 usec
DE                 98.57 usec
TE                 300.0 K
D1              1.50000000 sec
P1                  3.00 usec
DE                 98.57 usec
SFO1           400.1318844 MHz
NUCLEUS               1H
SI                 32768
SF             400.1300528 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  4.00
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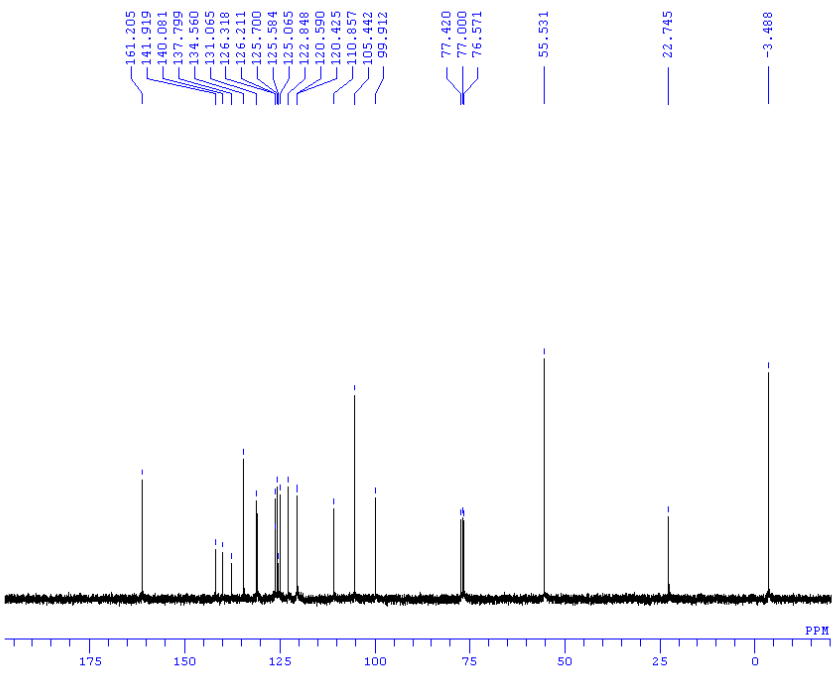


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DFILE   D:\NMR_Users\Xi Zhe
COMNT
DATIM   Mon Mar 28 20:32:20
OBNUC   13C
EXMOD   BCM
OBFRQ        75.45 MHz
OBSET       124.00 KHz
OBFIN       1840.0 Hz
POINT        32768
FREQU      20408.1 Hz
SCANS          120
ACQTM        1.606 sec
PD           1.394 sec
PW1            4.2 us
IRNUC   1H
CTEMP         22.7 c
SLVNT   CDCL3
EXREF        77.00 ppm
BF            0.62 Hz
RGAIN           26
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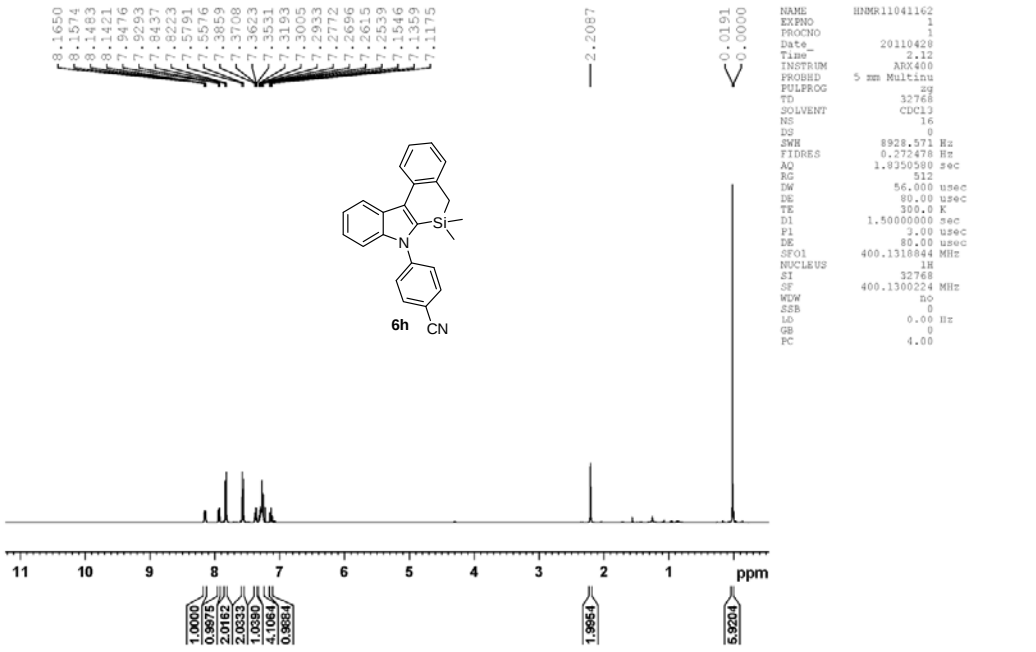

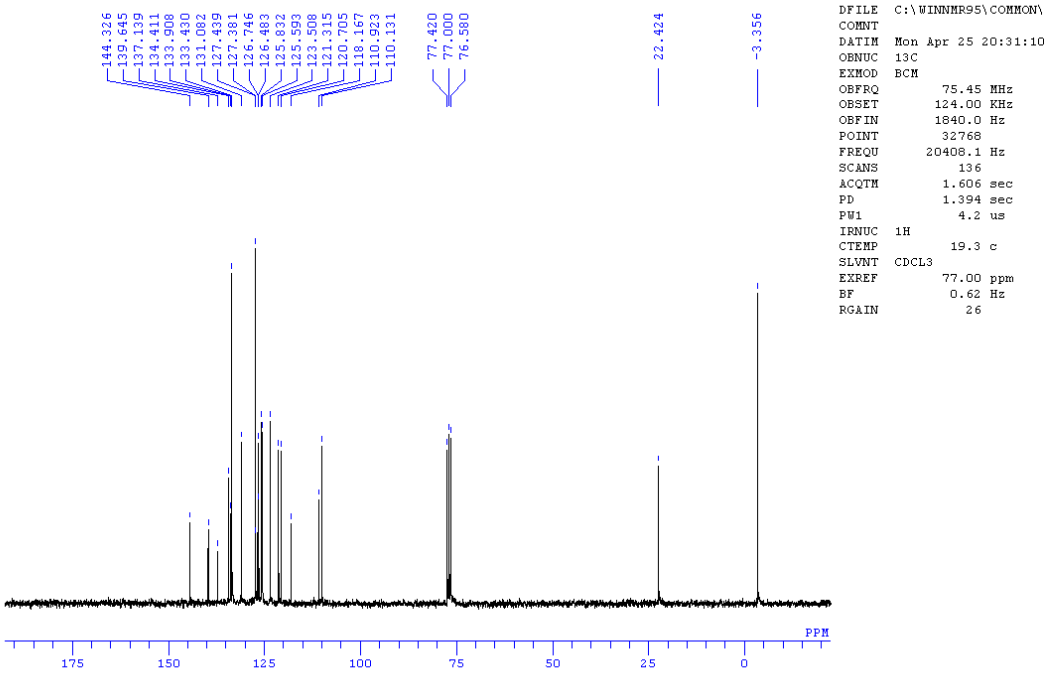

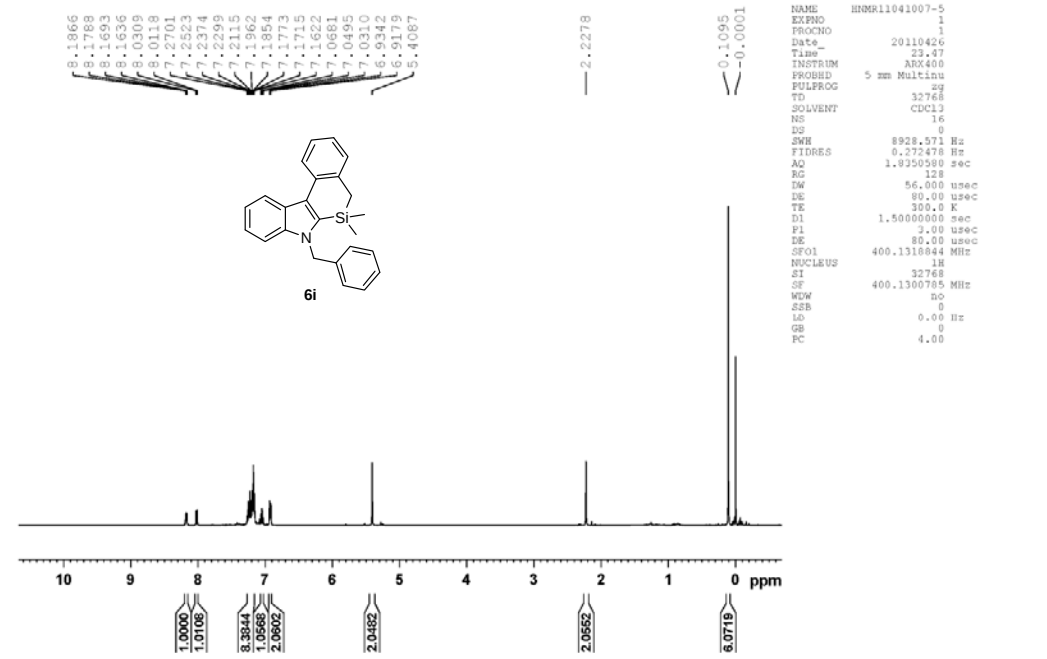

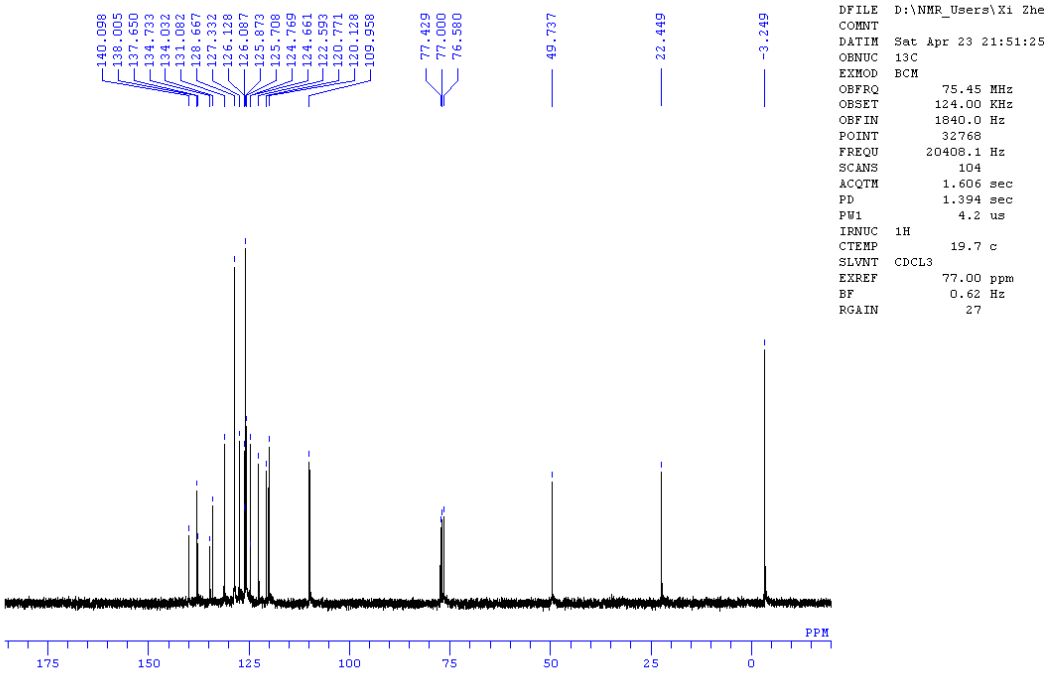

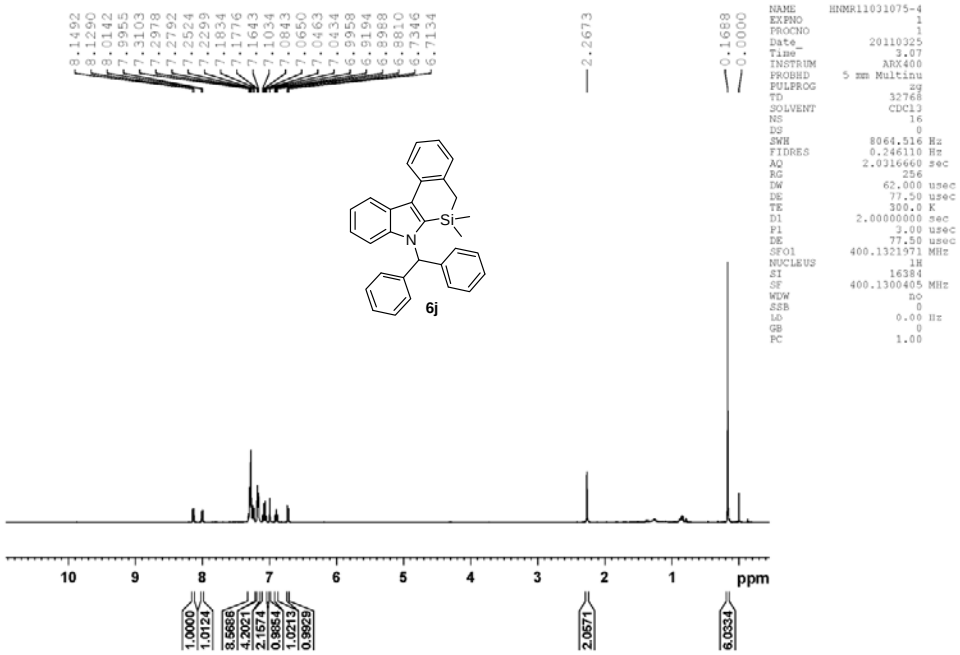
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7.2792
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7.2299
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7.1776
7.1643
7.1034
7.0843
7.0650
7.0463
7.0434
6.9958
6.9194
6.8988
6.8810
6.7346
6.7134
2.2673
0.1688
0.0000
Si
N
6j
NAME HNMR11031075-4
EXPNO 1
PROCNO 1
Date_ 20110325
Time_ 3.07
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8064.516 Hz
FIDRES 0.246110 Hz
AQ 2.0316660 sec
RG 256
DW 62.000 usec
DE 77.50 usec
TE 300.0 K
D1 2.00000000 sec
P1 2.00 usec
DE 77.50 usec
SFO1 400.1321971 MHz
NUCLEUS 1H
SI 16384
SF 400.1300405 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
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4.2021
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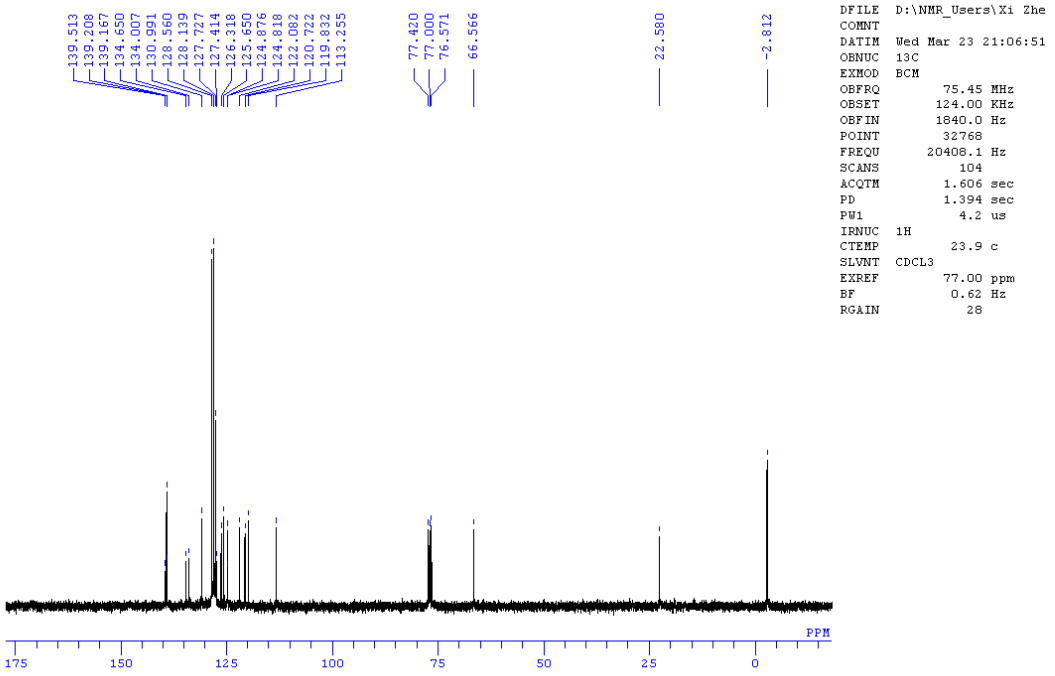
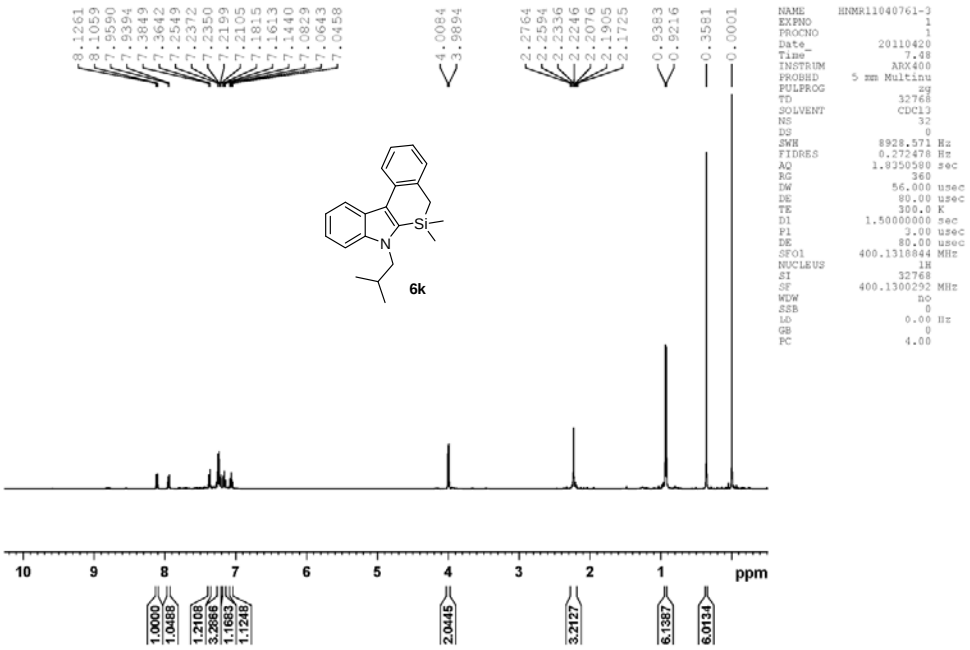
1.0213
0.9929
2.0571
6.0334


139.513
139.208
139.167
134.650
134.007
130.991
128.560
128.139
127.727
127.414
126.318
125.650
124.876
124.818
122.082
120.722
119.832
113.255
77.420
77.000
76.571
66.566
22.580
-2.812
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Wed Mar 23 21:06:51
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 104
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 28
PPM
175
150
125
100
75
50
25
0



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EXPNO 1
PROCNO 1
Date_ 20110420
Time_ 7.48
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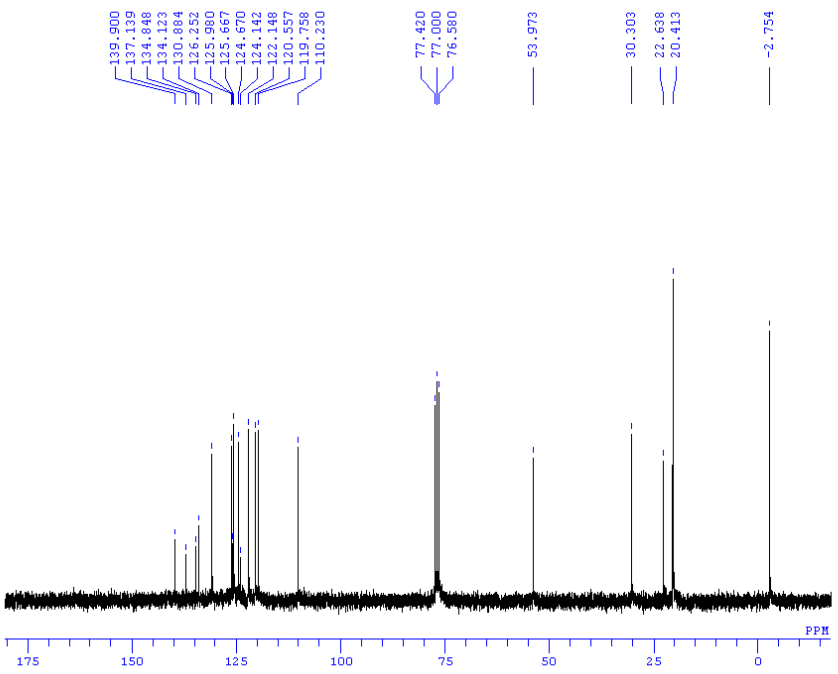
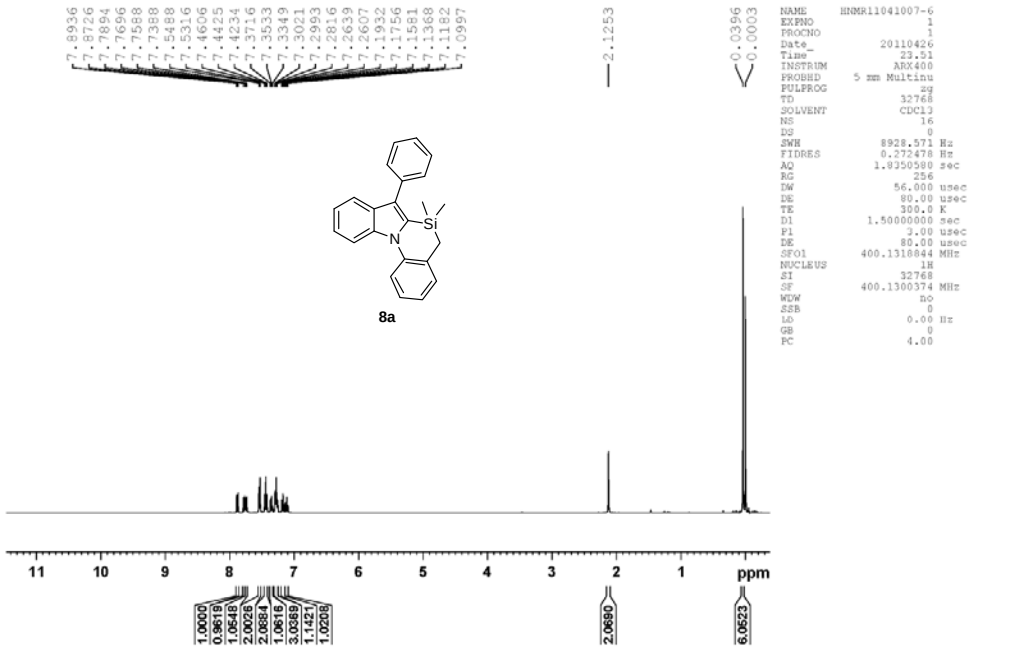
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8928.571 Hz
FIDRES 0.272478 Hz
AQ 1.9350580 sec
RG 360
DW 56.000 usec
DE 80.00 usec
TE 300.0 K
D1 1.50000000 sec
P1 2.00 usec
DE 80.00 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300292 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00



DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Tue Apr 19 00:16:22
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 376
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 27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7.8726
7.7894
7.7696
7.7588
7.7388
7.5488
7.5316
7.4606
7.4425
7.4234
7.3716
7.3533
7.3349
7.3021
7.2993
7.2816
7.2639
7.2607
7.1922
7.1756
7.1581
7.1368
7.1182
7.0997
2.1253
0.0396
0.0003
Si
N
8a
NAME HNMR11041007-6
EXPNO 1
PROCNO 1
Date_ 20110426
Time_ 23.51
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8928.571 Hz
FIDRES 0.272478 Hz
AQ 1.9350580 sec
RG 256
DW 56.000 usec
DE 80.00 usec
TE 300.0 K
D1 1.50000000 sec
P1 2.00 usec
DE 80.00 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300374 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
11 10 9 8 7 6 5 4 3 2 1 ppm
1.0000
0.9619
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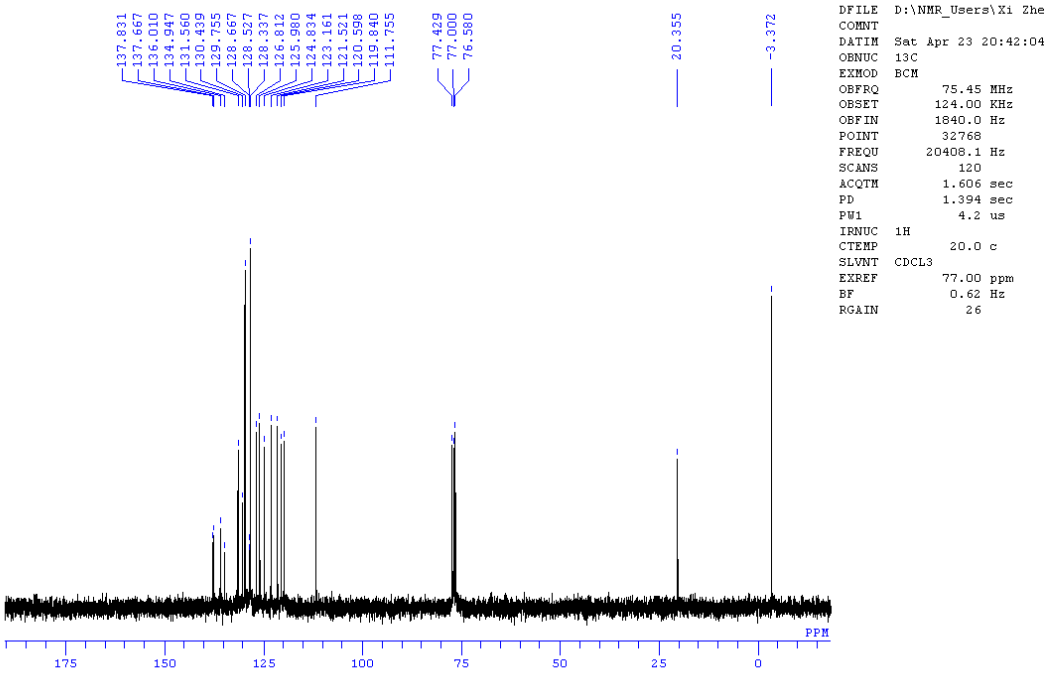
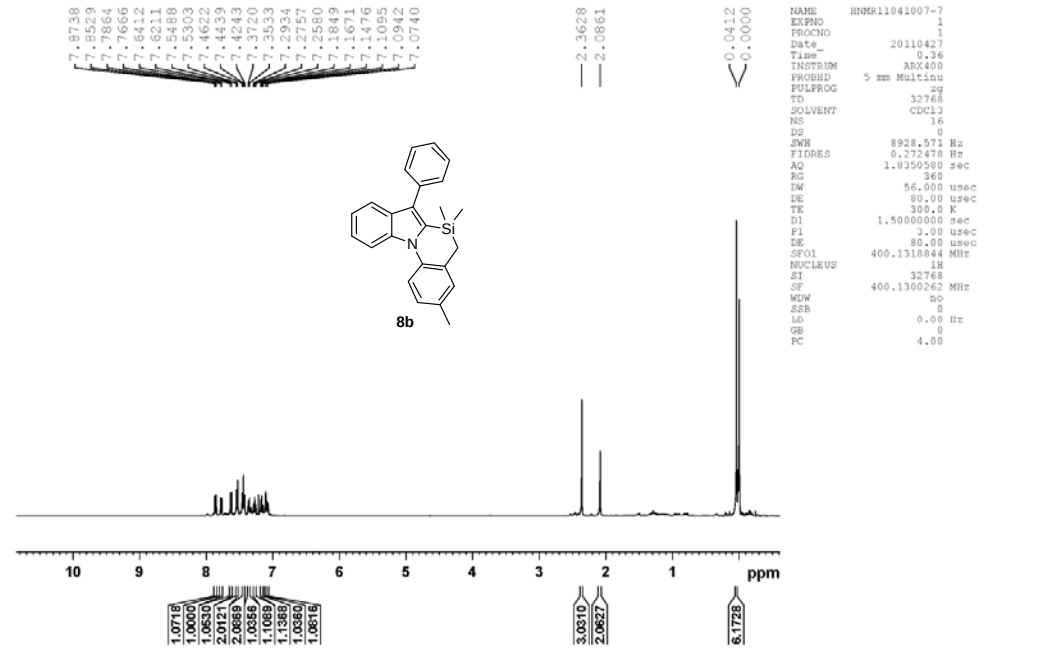
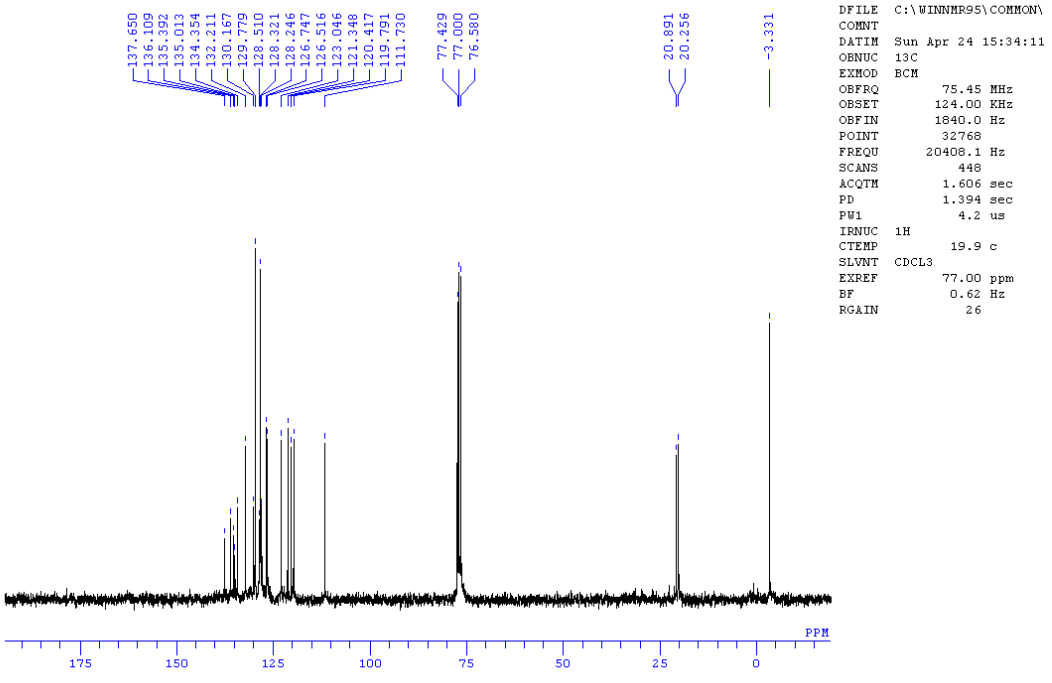
2.0028
2.0084
1.0616
3.0085
1.1427
1.0208
2.0690
6.0523


137.831
137.667
136.010
134.947
131.560
130.439
129.755
128.667
128.527
128.337
126.812
125.980
124.834
123.161
121.521
120.598
119.840
111.755
77.429
77.000
76.580
20.355
-3.372
DFILE D:\NMR_Users\Xi Zhe
COMNT
DATIM Sat Apr 23 20:42:04
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 120
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26
PPM
175 150 125 100 75 50 25 0


7.8738
7.8529
7.7864
7.7666
7.6412
7.6211
7.5488
7.5303
7.4622
7.4439
7.4243
7.3720
7.3533
7.2934
7.2757
7.2580
7.1849
7.1671
7.1476
7.1095
7.0942
7.0740
2.3628
2.0861
0.0412
0.0000
NAME HNMR11041007-7
EXPNO 1
PROCNO 1
Date_ 20110427
Time_ 0.36
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8928.571 Hz
FIDRES 0.272478 Hz
AQ 1.9350580 sec
RG 360
DW 56.000 usec
DE 80.00 usec
TE 300.0 K
D1 1.50000000 sec
P1 2.00 usec
DE 80.00 usec
SFO1 400.1318844 MHz
NUCLEUS 1H
SI 32768
SF 400.1300262 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
Si
N
8b
10
9
8
7
6
5
4
3
2
1
ppm
1.0716
1.0000
1.0630
2.0121
2.0669
1.0358
1.1069
1.1388
1.0360
1.0616
3.0310
2.0627
6.1728


137.650
136.109
135.392
135.013
134.354
132.211
130.167
129.779
128.510
128.321
128.246
126.747
126.516
123.046
121.348
120.417
119.791
111.730
77.429
77.000
76.580
20.891
20.256
-3.331
DFILE C:\WINNMR95\COMMON\
COMNT
DATIM Sun Apr 24 15:34:11
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 448
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26
PPM
175
150
125
100
75
50
25
0