Supporting Information

Copper-Catalyzed Borofunctionalization of Styrenes with B_2pin_2 and CO

Yang Yuan,[a]† Fu-Peng Wu,[a]† and Xiao-Feng Wu*[a,b]

[a] Leibniz-Institut für Katalyse e.V., Albert-Einstein-Straße 29a, 18059 Rostock, Germany, E-mail: xiao-feng.wu@catalysis.de
[b] Dalian National Laboratory for Clean Energy, Dalian Institute of Chemical Physics, Chinese Academy of Sciences, 116023, Dalian, Liaoning, China, E-mail: xwu2020@dicp.ac.cn

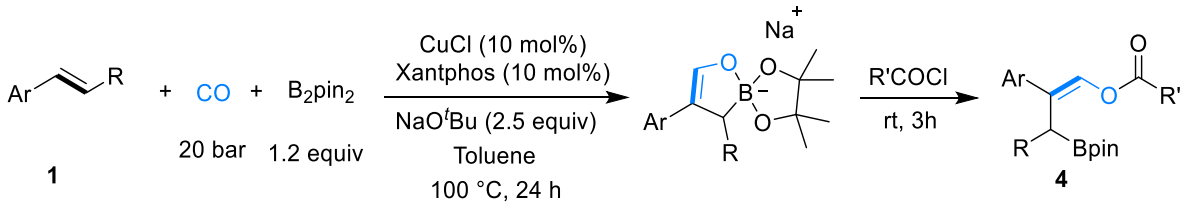
Contents

1. General information

All commercial reagents were purchased from Sigma-Aldrich, Strem, Acros, TCI or Alfa Aesar and used as such unless stated otherwise. Solvents (Anhydrous and under inert atmosphere) were collected from The Solvent purification system by M BRAUN and used under standard schlenk technique. NMR spectra were recorded on Bruker Avance 300 MHz and Bruker ARX 400 MHz spectrometers. Multiplets were assigned as s(singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublet), m (multiplet) and br. s (broad singlet). Coupling constants reported to 0.5 or 1.0 Hz accuracy. GC-yields were calculated using hexadecane as internal standard. All measurements were carried out at room temperature unless otherwise stated. Electron impact (EI) mass spectra were recorded on AMD 402 mass spectrometer (70 eV). The data are given as mass units per charge (m/z). Gas chromatography analysis was performed on an Agilent HP-7890A instrument with an FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d., 0.25 μm film thickness) using argon as carrier gas. The products were isolated from the reaction mixture by column chromatography on silica gel 60, 0.063-0.2 mm, 70-230 mesh (Merck).

2. General Procedures

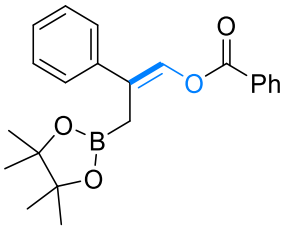
General Procedure for Cu-catalyzed Borocarbonylation of Styrenes with B_2pin_2 and CO.



A vial (4 mL) was charged with CuCl (1.98 mg, 10.0 mol%), Xantphos (11.6 mg, 10.0 mol%), NaO^{t}Bu (48.1 mg, 2.5 equiv), B_2pin_2 (60.1 mg, 1.2 equiv), and a stirring bar. The vial was closed by PTFE/white rubber septum (Wheaton 13 mm Septa) and phenolic cap and connected with atmosphere with a needle. The vial was evacuated under vacuum and recharged with argon for three times. Then, toluene (1.0 mL) was injected under argon by using a syringe. After that, styrenes **1** (0.2 mmol, 1.0 equiv), was added, the vial (or several vials) was placed in an alloy plate, which was transferred into a 300 mL autoclave of the 4560 series from Parr Instruments. After flushing the autoclave three times with CO, a pressure of 20 bar of CO was adjusted at ambient temperature. Then, the reaction was performed for 24 hours at 100 °C. After 24 hours, the autoclave was cooled down with ice water to room temperature and the pressure was

released carefully. Then, Acid chlorides (0.4 mmol, 2.0 equiv) was added to the vials by using a micro syringe, the reaction was stirred for 3 more hours at room temperature. After that, the solution was then filtered through celite and concentrated in vacuo. The residue was purified by column chromatography to afford the corresponding products **4**.

3. Characterization Data



***(E)*-2-Phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4a)**

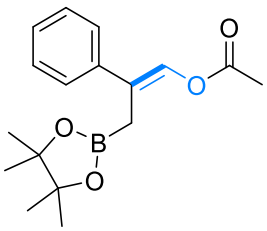
53.9 mg, 74% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 – 8.07 (m, 2H), 7.68 (t, *J* = 1.0 Hz, 1H), 7.58 – 7.49 (m, 1H), 7.46 – 7.34 (m, 4H), 7.31 – 7.24 (m, 2H), 7.22 – 7.16 (m, 1H), 2.23 (s, 2H), 1.08 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.5, 139.7, 133.5, 132.1, 130.2, 129.6, 128.6, 128.5, 127.3, 126.4, 123.9, 83.6, 77.5, 77.1, 76.8, 24.7.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.85.

HRMS (ESI-TOF) m/z: Calcd. for $C_{22}H_{25}BO_4$ $[M+Na]^+$: 387.1747, Found: 387.1752.



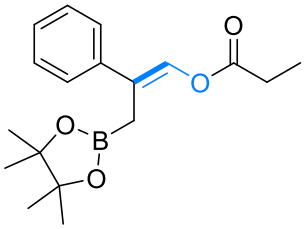
***(E)*-2-Phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl acetate (4b)**

42.3 mg, 70% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (t, *J* = 1.0 Hz, 1H), 7.43 – 7.38 (m, 2H), 7.36 – 7.24 (m, 3H), 2.22 (s, 3H), 2.20 (s, 2H), 1.18 (s, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 168.0, 139.4, 131.9, 128.3, 127.1, 126.2, 123.0, 83.4, 24.6, 20.9.

HRMS (ESI-TOF) m/z: Calcd. for $C_{17}H_{23}BO_4$ $[M+Na]^+$: 325.1589, Found: 325.1591.



(*E*)-2-Phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl propionate (4c)

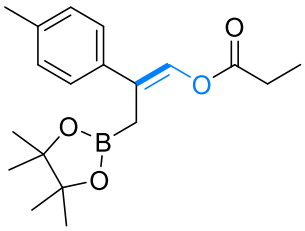
35.4 mg, 56% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (t, *J* = 1.0 Hz, 1H), 7.32 – 7.28 (m, 2H), 7.26 – 7.20 (m, 2H), 7.19 – 7.14 (m, 1H), 2.41 (q, *J* = 7.5 Hz, 2H), 2.10 (s, 2H), 1.15 (t, *J* = 7.5 Hz, 3H), 1.08 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 139.5, 131.9, 128.3, 127.0, 126.2, 122.9, 83.4, 27.6, 24.6, 9.0.

^{11}B NMR (128 MHz, $CDCl_3$) δ 33.02.

HRMS (ESI-TOF) m/z: Calcd. for $C_{18}H_{25}BO_4$ $[M+Na]^+$: 339.1746, Found: 339.1749.



(*E*)-3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-2-(p-tolyl)prop-1-en-1-yl propionate (4d)

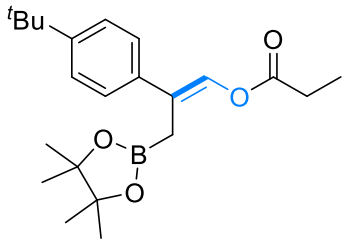
45.6 mg, 69% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.32 – 7.27 (m, 2H), 7.17 – 7.10 (m, 2H), 2.50 (q, *J* = 7.5 Hz, 2H), 2.35 (s, 3H), 2.18 (s, 2H), 1.24 (t, *J* = 7.5 Hz, 3H), 1.19 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 136.7, 136.5, 131.5, 129.0, 126.0, 122.7, 83.4, 27.6, 24.6, 21.1, 9.0.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.75.

HRMS (ESI-TOF) m/z: Calcd. for $C_{19}H_{27}BO_4$ $[M+Na]^+$: 353.1903, Found: 353.1903.



(*E*)-2-(4-(*tert*-Butyl)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl propionate (4e)

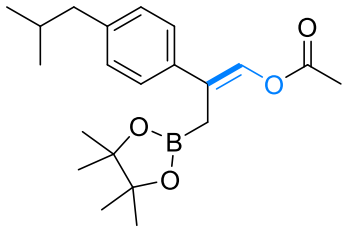
52.9 mg, 71% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (t, *J* = 1.0 Hz, 1H), 7.35 (s, 4H), 2.51 (q, *J* = 7.5 Hz, 2H), 2.18 (s, 2H), 1.33 (s, 9H), 1.24 (t, *J* = 7.5 Hz, 3H), 1.19 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 150.0, 136.6, 131.6, 125.9, 125.2, 122.7, 83.4, 34.5, 31.3, 27.6, 24.6, 9.0.

^{11}B NMR (128 MHz, $CDCl_3$) δ 33.06.

HRMS (ESI-TOF) m/z: Calcd. for $C_{22}H_{33}BO_4$ $[M+Na]^+$: 395.2373, Found: 395.2375.



(*E*)-2-(4-Isobutylphenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl acetate (4f)

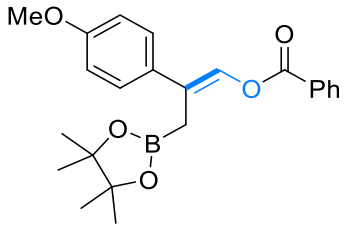
48.7 mg, 68% yield, E/Z = 5/1, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (t, *J* = 1.0 Hz, 1H), 7.39 – 7.24 (m, 2H), 7.16 – 7.02 (m, 2H), 2.47 (dd, *J* = 7.0, 3.0 Hz, 2H), 2.21 (s, 3H), 2.19 (s, 2H), 1.86 (dt, *J* = 13.5, 7.0 Hz, 1H), 1.18 (s, 12H), 0.91 (d, *J* = 6.0 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.0, 157.9, 140.6, 136.7, 131.5, 129.1, 129.1, 126.0, 125.9, 122.9, 83.5, 83.4, 45.1, 30.2, 25.0, 24.6, 22.3, 20.9.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.67.

HRMS (ESI-TOF) m/z: Calcd. for $C_{22}H_{25}BO_4$ $[M+Na]^+$: 381.2216, Found: 381.2218.



(*E*)-2-(4-Methoxyphenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4g)

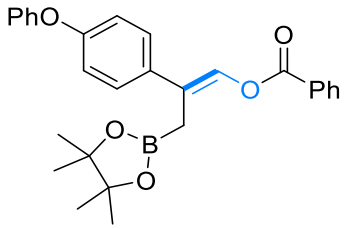
57.6 mg, 73% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1-10/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 – 7.99 (m, 2H), 7.58 (t, *J* = 1.0 Hz, 1H), 7.51 – 7.44 (m, 1H), 7.40 – 7.32 (m, 2H), 7.31 – 7.24 (m, 2H), 6.82 – 6.71 (m, 2H), 3.70 (s, 3H), 2.17 (s, 2H), 1.04 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4, 159.0, 133.3, 132.0, 131.0, 130.0, 129.6, 128.5, 127.4, 123.3, 113.8, 83.5, 55.3, 24.6.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.56.

HRMS (ESI-TOF) m/z: Calcd. for $C_{23}H_{27}BO_5$ $[M+Na]^+$: 417.1852, Found: 417.1854.



(*E*)-2-(4-Phenoxyphenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4h)

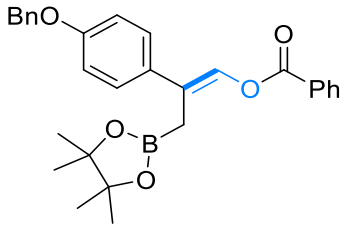
62.1 mg, 68% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 – 8.20 (m, 2H), 7.75 – 7.73 (m, 1H), 7.68 – 7.60 (m, 1H), 7.56 – 7.49 (m, 2H), 7.47 – 7.43 (m, 2H), 7.40 – 7.32 (m, 2H), 7.18 – 7.09 (m, 1H), 7.07 – 6.99 (m, 4H), 2.32 (s, 2H), 1.19 (s, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 163.4, 157.3, 156.5, 134.7, 133.4, 131.6, 130.1, 129.7, 129.5, 128.5, 127.7, 123.2, 118.9, 118.8, 83.5, 24.6.

^{11}B NMR (96 MHz, $CDCl_3$) δ 32.67.

HRMS (ESI-TOF) m/z: Calcd. for $C_{28}H_{29}BO_5$ $[M+Na]^+$: 479.2010, Found: 479.2016.



(*E*)-2-(4-(Benzyloxy)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4i)

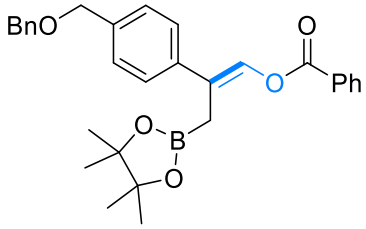
59.3 mg, 63% yield, white solid. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (dd, *J* = 8.5, 1.5 Hz, 2H), 7.74 (t, *J* = 1.0 Hz, 1H), 7.67 – 7.59 (m, 1H), 7.55 – 7.34 (m, 9H), 6.99 (d, *J* = 9.0 Hz, 2H), 5.11 (s, 2H), 2.32 (s, 2H), 1.19 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4, 158.2, 137.0, 133.4, 132.3, 131.1, 130.1, 129.6, 128.6, 128.5, 128.0, 127.5, 127.4, 123.3, 83.5, 70.0, 24.7.

^{11}B NMR (128 MHz, $CDCl_3$) δ 33.13.

HRMS (ESI-TOF) m/z: Calcd. for $C_{29}H_{31}BO_5$ $[M+Na]^+$: 493.2167, Found: 493.2158.



(*E*)-2-(4-((Benzyloxy)methyl)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4j)

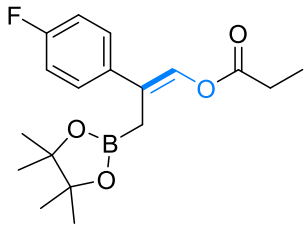
51.3 mg, 53% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 – 8.18 (m, 2H), 7.78 (s, 1H), 7.66 – 7.57 (m, 1H), 7.54 – 7.43 (m, 4H), 7.40 – 7.32 (m, 6H), 7.35 – 7.28 (m, 1H), 4.57 (s, 2H), 4.56 (s, 2H), 2.31 (s, 2H), 1.17 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4, 138.9, 138.3, 137.2, 133.4, 132.0, 130.1, 129.5, 128.5, 128.4, 127.9, 127.8, 127.7, 126.3, 123.5, 83.5, 72.0, 71.8, 24.7.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.64.

HRMS (ESI-TOF) m/z: Calcd. for $C_{30}H_{33}BO_5$ $[M+Na]^+$: 507.2324, Found: 507.2325.



(*E*)-2-(4-Fluorophenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl propionate (4k)

41.4 mg, 62% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

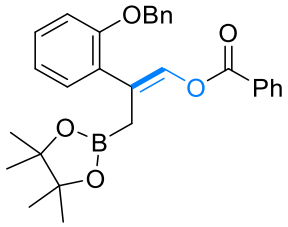
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.36 – 7.31 (m, 2H), 7.04 – 6.94 (m, 2H), 2.48 (q, *J* = 7.5 Hz, 2H), 2.14 (s, 2H), 1.22 (t, *J* = 7.5 Hz, 3H), 1.16 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.41, 162.11 (d, *J* = 246.0 Hz), 135.55 (d, *J* = 3.5 Hz), 131.78, 127.79 (d, *J* = 8.0 Hz), 122.05, 115.24, 115.02, 83.48, 27.56, 24.59, 8.97.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.69.

^{19}F NMR (282 MHz, $CDCl_3$) δ -115.69 – -115.80 (m).

HRMS (ESI-TOF) m/z: Calcd. for $C_{18}H_{24}BFO_4$ $[M+Na]^+$: 357.1652, Found: 357.1655.



(*E*)-2-(2-(Benzyloxy)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4l)

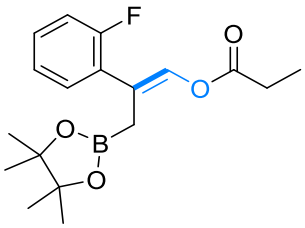
57.4 mg, 61% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 – 8.17 (m, 2H), 7.65 – 7.56 (m, 2H), 7.53 – 7.44 (m, 4H), 7.41 – 7.28 (m, 4H), 7.25 – 7.18 (m, 1H), 6.99 – 6.87 (m, 2H), 5.15 (s, 2H), 2.37 (s, 2H), 1.12 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4, 156.6, 137.3, 133.3, 133.1, 130.6, 130.1, 129.7, 129.4, 128.5, 128.4, 127.7, 127.2, 122.5, 120.8, 112.6, 83.2, 70.3, 24.7.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.55.

HRMS (ESI-TOF) m/z: Calcd. for $C_{29}H_{31}BO_5$ $[M+Na]^+$: 493.2167, Found: 493.2165.



(*E*)-2-(2-Fluorophenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl propionate (4m)

38.8 mg, 58% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

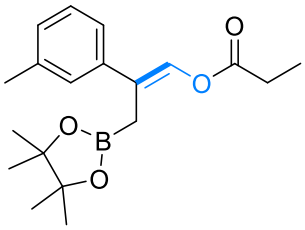
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.40 (m, 1H), 7.34 – 7.28 (m, 1H), 7.27 – 7.18 (m, 1H), 7.15 – 6.99 (m, 2H), 2.51 (q, *J* = 7.5 Hz, 1H), 2.18 (s, 1H), 1.24 (t, *J* = 7.5 Hz, 2H), 1.18 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.20, 160.43 (d, *J* = 247.5 Hz), 133.90 (d, *J* = 4.0 Hz), 130.00 (d, *J* = 4.0 Hz), 128.60 (d, *J* = 8.5 Hz), 127.41 (d, *J* = 13.5 Hz), 123.89 (d, *J* = 3.5 Hz), 118.48, 115.82, 115.59, 83.36, 27.54, 24.61, 8.95.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.53.

^{19}F NMR (282 MHz, $CDCl_3$) δ -113.90 (dt, J = 12.2, 6.5 Hz).

HRMS (ESI-TOF) m/z: Calcd. for $C_{18}H_{24}BFO_4$ $[M+Na]^+$: 357.1652, Found: 357.1651.



(*E*)-3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-2-(m-tolyl)prop-1-en-1-yl propionate (4n)

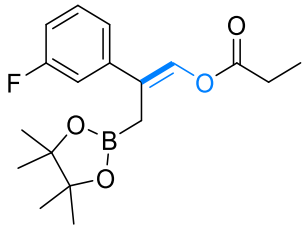
43.6 mg, 66% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.21 – 7.17 (m, 3H), 7.05 (ddd, *J* = 4.5, 3.5, 1.0 Hz, 1H), 2.49 (q, *J* = 7.5 Hz, 2H), 2.34 (s, 3H), 2.16 (s, 2H), 1.22 (t, *J* = 7.5 Hz, 3H), 1.17 (s, 11H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 139.4, 137.8, 131.8, 128.2, 127.8, 126.9, 123.3, 122.9, 83.4, 27.6, 24.6, 21.5, 9.0.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.75.

HRMS (ESI-TOF) m/z: Calcd. for $C_{19}H_{27}BO_4$ $[M+Na]^+$: 353.1903, Found: 353.1905.



(*E*)-2-(3-Fluorophenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl propionate (4o)

41.4 mg, 62% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

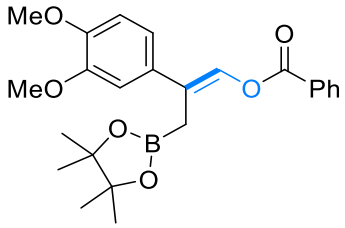
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (t, *J* = 1.0 Hz, 1H), 7.33 – 7.24 (m, 1H), 7.18 (ddd, *J* = 8.0, 1.5, 1.0 Hz, 1H), 7.11 (ddd, *J* = 10.5, 2.5, 2.0 Hz, 1H), 7.00 – 6.90 (m, 1H), 2.51 (q, *J* = 7.5 Hz, 2H), 2.17 (s, 2H), 1.25 (t, *J* = 7.5 Hz, 3H), 1.19 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.31, 162.88 (d, *J* = 245.0 Hz), 141.89 (d, *J* = 8.0 Hz), 132.56, 129.71 (d, *J* = 8.5 Hz), 121.93, 121.75 (d, *J* = 3.0 Hz), 113.88, 113.67, 113.24, 113.02, 83.54, 27.55, 24.59, 8.95.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.7.

^{19}F NMR (282 MHz, $CDCl_3$) δ -113.54 – -113.68 (m).

HRMS (ESI-TOF) m/z: Calcd. for $C_{18}H_{24}BFO_4$ $[M+Na]^+$: 357.1652, Found: 357.1655.



(*E*)-2-(3,4-Dimethoxyphenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4p)

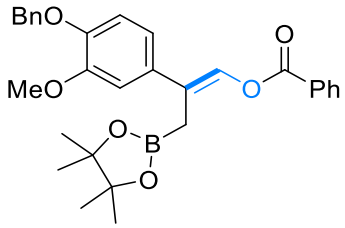
52.6 mg, 62% yield, colorless oil. Eluent: pentane/ethyl acetate = 15/1-5/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (dd, *J* = 8.5, 1.5 Hz, 2H), 7.70 (s, 1H), 7.63 – 7.55 (m, 1H), 7.51 – 7.45 (m, 2H), 7.04 – 6.97 (m, 2H), 6.84 (d, *J* = 8.0 Hz, 1H), 3.91 (s, 3H), 3.88 (s, 3H), 2.29 (s, 2H), 1.16 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4, 148.8, 148.4, 133.4, 132.4, 131.2, 130.0, 129.5, 128.5, 123.5, 118.7, 111.1, 109.6, 83.5, 55.9, 55.9, 24.7.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.59.

HRMS (ESI-TOF) m/z: Calcd. for $C_{24}H_{29}BO_6$ $[M+Na]^+$: 447.1959, Found: 447.1959.



(*E*)-2-(4-(Benzyloxy)-3-methoxyphenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4q)

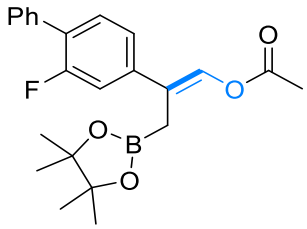
52.0 mg, 52% yield, colorless oil. Eluent: pentane/ethyl acetate = 15/1-5/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 – 8.15 (m, 2H), 7.70 (s, 1H), 7.63 – 7.57 (m, 1H), 7.52 – 7.41 (m, 4H), 7.40 – 7.29 (m, 3H), 7.04 (d, *J* = 2.0 Hz, 1H), 6.93 (dd, *J* = 8.5, 2.0 Hz, 1H), 6.85 (d, *J* = 8.5 Hz, 1H), 5.17 (s, 2H), 3.93 (s, 3H), 2.28 (s, 2H), 1.15 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4, 149.5, 147.5, 137.2, 133.4, 133.0, 131.3, 130.0, 129.5, 128.5, 128.5, 127.8, 127.3, 123.5, 118.6, 114.0, 110.1, 83.5, 71.1, 56.0, 24.7.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.77.

HRMS (ESI-TOF) m/z: Calcd. for $C_{30}H_{33}BO_6$ $[M+Na]^+$: 523.2272, Found: 523.2281.



(*E*)-2-(2-Fluoro-[1,1'-biphenyl]-4-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl acetate (4r)

49.1 mg, 62% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

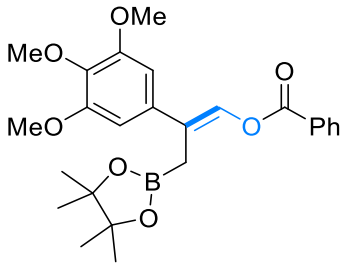
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (t, *J* = 1.0 Hz, 1H), 7.49 – 7.45 (m, 2H), 7.39 – 7.32 (m, 2H), 7.31 – 7.26 (m, 2H), 7.20 – 7.09 (m, 2H), 2.14 (s, 3H), 2.11 (s, 2H), 1.12 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.8, 159.7 (d, *J* = 247.5 Hz), 140.7 (d, *J* = 8.0 Hz), 135.6, 132.6, 130.5 (d, *J* = 4.0 Hz), 128.9 (d, *J* = 3.0 Hz), 127.6, 127.5 (d, *J* = 13.5 Hz), 122.0 (d, *J* = 3.0 Hz), 121.7, 113.9, 113.6, 83.6, 24.6, 20.9.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.65.

^{19}F NMR (282 MHz, $CDCl_3$) δ -118.24 – -118.51 (m).

HRMS (ESI-TOF) m/z: Calcd. for $C_{23}H_{26}BFO_4$ $[M+Na]^+$: 419.1809, Found: 419.1810.



(*E*)-3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-2-(3,4,5-trimethoxyphenyl)prop-1-en-1-yl benzoate (4s)

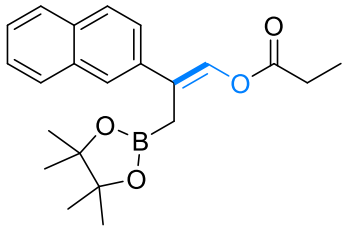
63.6 mg, 70% yield, colorless oil. Eluent: pentane/ethyl acetate = 15/1-5/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 – 7.88 (m, 2H), 7.51 (s, 1H), 7.44 – 7.36 (m, 1H), 7.32 – 7.21 (m, 2H), 6.49 (s, 2H), 3.69 (s, 6H), 3.64 (s, 3H), 2.08 (s, 2H), 0.96 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4, 153.1, 137.4, 135.5, 133.5, 131.7, 130.1, 129.4, 128.5, 123.9, 103.6, 83.5, 60.9, 56.1, 24.7.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.56.

HRMS (ESI-TOF) m/z: Calcd. for $C_{25}H_{31}BO_7$ $[M+Na]^+$: 477.2064, Found: 477.2069.



(*E*)-2-(Naphthalen-2-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl propionate (4t)

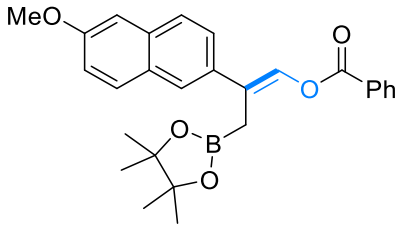
41.7 mg, 57% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.75 (m, 4H), 7.70 (s, 1H), 7.57 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.49 – 7.40 (m, 2H), 2.52 (q, *J* = 7.5 Hz, 2H), 2.29 (s, 2H), 1.25 (t, *J* = 7.6 Hz, 3H), 1.16 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 136.7, 133.5, 132.6, 132.5, 128.0, 127.9, 127.5, 126.1, 125.6, 124.8, 124.3, 122.6, 83.5, 27.6, 24.6, 9.0.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.81.

HRMS (ESI-TOF) m/z: Calcd. for $C_{22}H_{27}BO_4$ $[M+Na]^+$: 389.1904, Found: 389.1906.



(*E*)-2-(6-Methoxynaphthalen-2-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4u)

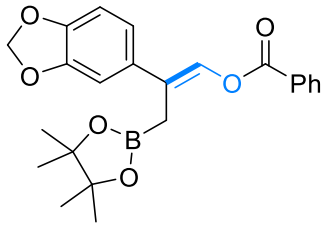
60.4 mg, 68% yield, white solid. Eluent: pentane/ethyl acetate = 30/1-15/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 – 8.22 (m, 2H), 7.94 (s, 1H), 7.83 (d, *J* = 1.5 Hz, 1H), 7.75 (t, *J* = 9.0 Hz, 2H), 7.68 – 7.61 (m, 2H), 7.58 – 7.50 (m, 2H), 7.21 – 7.12 (m, 2H), 3.95 (s, 3H), 2.44 (s, 2H), 1.18 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4, 157.6, 134.5, 133.8, 133.4, 132.0, 130.1, 129.5, 129.5, 129.0, 128.5, 126.8, 125.0, 124.7, 123.6, 118.9, 105.7, 83.5, 55.3, 24.7.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.69.

HRMS (ESI-TOF) m/z: Calcd. for $C_{27}H_{29}BO_5$ $[M+Na]^+$: 467.2010, Found: 467.2018.



(*E*)-2-(Benzo[*d*][1,3]dioxol-5-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4v)

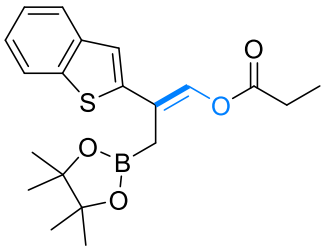
47.4 mg, 58% yield, white solid. Eluent: pentane/ethyl acetate = 30/1-15/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (dd, *J* = 8.5, 1.5 Hz, 2H), 7.66 (t, *J* = 1.0 Hz, 1H), 7.62 – 7.56 (m, 1H), 7.51 – 7.44 (m, 2H), 6.99 – 6.88 (m, 2H), 6.83 – 6.75 (m, 1H), 5.95 (s, 2H), 2.25 (s, 2H), 1.17 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4, 147.7, 146.9, 133.8, 133.4, 131.4, 130.1, 129.5, 128.5, 123.6, 119.8, 108.2, 106.9, 101.0, 83.5, 24.7.

^{11}B NMR (128 MHz, $CDCl_3$) δ 32.27.

HRMS (ESI-TOF) m/z: Calcd. for $C_{23}H_{25}BO_6$ $[M+Na]^+$: 431.1646, Found: 431.1644.



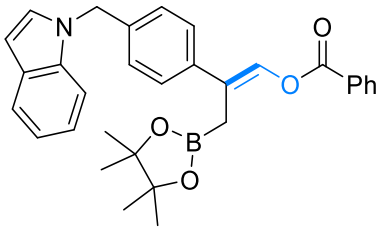
(*E*)-2-(Benzo[b]thiophen-2-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl propionate (4w)

40.9 mg, 55% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.92 (m, 1H), 7.87 – 7.81 (m, 1H), 7.47 (t, *J* = 1.0 Hz, 1H), 7.40 – 7.29 (m, 3H), 2.52 (q, *J* = 7.5 Hz, 2H), 2.22 (s, 2H), 1.24 (t, *J* = 7.5 Hz, 3H), 1.14 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 140.3, 137.9, 135.9, 133.4, 124.2, 124.2, 123.4, 123.3, 122.7, 117.8, 83.5, 27.6, 24.6, 24.6, 9.0.

HRMS (ESI-TOF) m/z: Calcd. for $C_{20}H_{25}BO_4S$ $[M+Na]^+$: 395.1467, Found: 395.1469.



(*E*)-2-(4-((*1H*-Indol-1-yl)methyl)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4x)

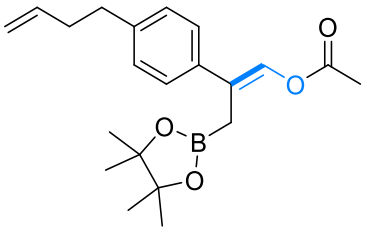
66.1 mg, 67% yield, yellow oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.29 – 8.19 (m, 2H), 7.77 (t, *J* = 1.0 Hz, 1H), 7.75 – 7.59 (m, 2H), 7.58 – 7.47 (m, 2H), 7.47 – 7.39 (m, 2H), 7.38 – 7.29 (m, 1H), 7.22 – 7.11 (m, 5H), 6.61 (dd, *J* = 3.0, 1.0 Hz, 1H), 5.36 (s, 2H), 2.31 (s, 2H), 1.18 (s, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 163.4, 139.1, 136.4, 136.3, 133.5, 132.1, 130.1, 129.5, 128.8, 128.5, 128.2, 127.0, 126.7, 123.4, 121.7, 121.0, 119.6, 109.8, 101.7, 83.5, 49.9, 24.7.

^{11}B NMR (96 MHz, $CDCl_3$) δ 32.35.

HRMS (ESI-TOF) m/z: Calcd. for $C_{31}H_{32}BNO_4$ $[M+Na]^+$: 516.2322, Found: 516.2324.



(*E*)-2-(4-(But-3-en-1-yl)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl acetate (4y)

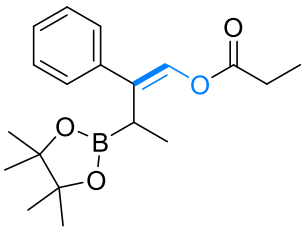
47.0 mg, 66% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 (t, *J* = 1.0 Hz, 1H), 7.32 (d, *J* = 8.5 Hz, 2H), 7.14 (dd, *J* = 8.0, 0.5 Hz, 2H), 5.98 – 5.77 (m, 1H), 5.12 – 4.95 (m, 2H), 2.81 – 2.67 (m, 2H), 2.44 – 2.31 (m, 2H), 2.21 (s, 3H), 2.18 (s, 2H), 1.18 (s, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 168.0, 140.8, 138.0, 136.9, 131.6, 128.4, 126.1, 122.9, 114.9, 83.4, 35.5, 35.0, 24.6, 20.9.

^{11}B NMR (96 MHz, $CDCl_3$) δ 32.67.

HRMS (ESI-TOF) m/z: Calcd. for $C_{21}H_{29}BO_4$ $[M+Na]^+$: 379.2060, Found: 379.2061.



(*E*)-2-Phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)but-1-en-1-yl propionate (4z)

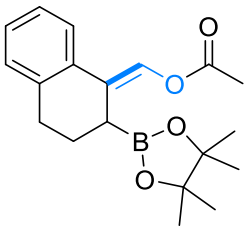
36.3 mg, 55% yield (from trans-β-methylstyrene); 38.3 mg, 58% yield (from cis- β-methylstyrene), colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 – 7.11 (m, 6H), 2.72 – 2.44 (m, 3H), 1.29 – 1.24 (m, 9H), 1.24 – 1.21 (m, 9H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.3, 139.4, 132.3, 129.5, 128.2, 127.2, 126.9, 83.3, 27.6, 24.9, 24.6, 14.5, 9.0.

^{11}B NMR (96 MHz, $CDCl_3$) δ 33.64.

HRMS (ESI-TOF) m/z: Calcd. for $C_{19}H_{27}BO_4$ $[M+Na]^+$: 353.1903, Found: 353.1907.



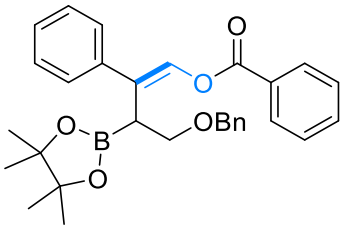
(*E*)-(2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-3,4-dihydronaphthalen-1(*2H*)-ylidene)methyl acetate (4aa)

38.1 mg, 58% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, *J* = 2.0 Hz, 1H), 7.55 – 7.49 (m, 1H), 7.17 – 7.07 (m, 3H), 2.88 – 2.72 (m, 1H), 2.69 – 2.55 (m, 2H), 2.19 (s, 3H), 1.97 – 1.80 (m, 2H), 1.20 (s, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 138.3, 132.7, 130.2, 128.5, 126.7, 126.2, 123.7, 122.7, 83.3, 30.1, 24.8, 24.5, 23.7, 20.9.

HRMS (ESI-TOF) m/z: Calcd. for $C_{19}H_{25}BO_4$ $[M+Na]^+$: 351.1746, Found: 351.1748.



(*E*)-4-(Benzyloxy)-2-phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)but-1-en-1-yl benzoate (4ab)

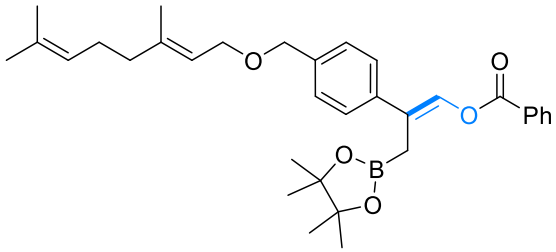
45.5 mg, 47% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1-15/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 – 8.20 (m, 2H), 7.66 (s, 1H), 7.65 – 7.59 (m, 1H), 7.59 – 7.45 (m, 4H), 7.41 – 7.20 (m, 8H), 4.58 – 4.36 (m, 2H), 4.18 – 3.88 (m, 2H), 3.05 (dd, *J* = 9.5, 6.5 Hz, 1H), 1.21 (s, 6H), 1.19 (s, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 163.2, 139.7, 138.8, 133.4, 133.1, 130.6, 130.3, 129.3, 128.9, 128.5, 128.2, 128.1, 127.7, 127.4, 127.2, 127.1, 126.5, 83.6, 72.8, 69.6, 25.1, 24.3.

^{11}B NMR (96 MHz, $CDCl_3$) δ 32.02.

HRMS (ESI-TOF) m/z: Calcd. for $C_{30}H_{33}BO_5$ $[M+Na]^+$: 507.2324, Found: 507.2326.



(*E*)-2-(4-((((*E*)-3,7-Dimethylocta-2,6-dien-1-yl)oxy)methyl)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4ac)

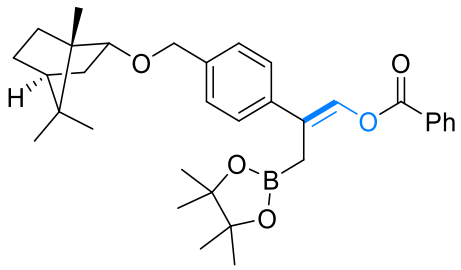
67.9 mg, 64% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.24 – 8.16 (m, 2H), 7.75 (s, 1H), 7.65 – 7.57 (m, 1H), 7.54 – 7.39 (m, 4H), 7.31 (d, *J* = 8.5 Hz, 2H), 5.41 (td, *J* = 7.0, 1.5 Hz, 1H), 5.13 – 5.03 (m, 1H), 4.50 (s, 2H), 4.00 (dd, *J* = 7.0, 1.0 Hz, 2H), 2.29 (s, 2H), 2.07 (d, *J* = 3.0 Hz, 4H), 1.76 (q, *J* = 1.0 Hz, 3H), 1.68 (s, 3H), 1.62 – 1.57 (m, 3H), 1.16 (s, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 163.4, 140.6, 138.7, 137.6, 133.4, 131.9, 130.1, 129.5, 128.5, 127.9, 126.2, 123.9, 123.5, 121.9, 83.5, 71.8, 66.3, 32.3, 26.7, 25.7, 25.0, 24.6, 23.5, 17.7.

^{11}B NMR (96 MHz, $CDCl_3$) δ 30.15.

HRMS (ESI-TOF) m/z: Calcd. for $C_{33}H_{43}BO_5$ $[M+Na]^+$: 553.3106, Found: 553.3110.



(*E*)-3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-2-(4-((((*1S*,*2S*,*4S*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)oxy)methyl)phenyl)prop-1-en-1-yl benzoate (4ad)

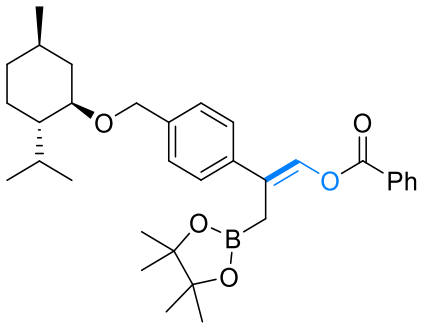
62.6 mg, 59% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 – 8.14 (m, 3H), 7.76 (s, 1H), 7.70 – 7.56 (m, 2H), 7.53 – 7.40 (m, 3H), 7.34 – 7.29 (m, 1H), 4.66 – 4.38 (m, 2H), 3.76 – 3.65 (m, 1H), 2.30 (s, 2H), δ 2.19 – 2.04 (m, 2H), 1.78 – 1.62 (m, 2H), 1.39 – 1.23 (m, 3H), 1.16 (s, 12H), 0.91 (s, 3H), 0.84 (d, *J* = 8.0 Hz, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 163.4, 138.5, 134.5, 133.4, 131.8, 130.6, 130.1, 129.5, 128.9, 128.5, 127.3, 126.1, 123.6, 84.2, 83.5, 71.3, 49.3, 47.9, 45.1, 36.1, 28.3, 26.8, 24.7, 19.8, 18.9, 14.0.

^{11}B NMR (96 MHz, $CDCl_3$) δ 32.42.

HRMS (ESI-TOF) m/z: Calcd. for $C_{33}H_{43}BO_5$ $[M+Na]^+$: 553.3106, Found: 553.3101.



(*E*)-2-(4-((((*1R,2S,5R*)-2-Isopropyl-5-methylcyclohexyl)oxy)methyl)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl benzoate (4ae)

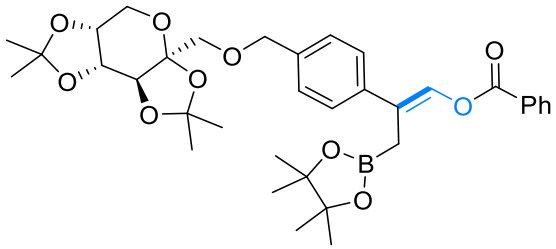
54.3 mg, 51% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.26 – 8.15 (m, 2H), 7.76 (s, 1H), 7.65 – 7.57 (m, 1H), 7.52 – 7.40 (m, 4H), 7.36 – 7.30 (m, 2H), 4.53 (dd, *J* = 75.0, 11.5 Hz, 2H), 3.17 (td, *J* = 10.5, 4.0 Hz, 1H), 2.38 – 2.11 (m, 4H), 1.77 – 1.57 (m, 2H), 1.45 – 1.24 (m, 3H), 1.16 (s, 12H), 0.93 (dd, *J* = 10.5, 7.0 Hz, 9H), 0.73 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 163.4, 138.6, 138.1, 133.4, 131.9, 130.1, 129.5, 128.9, 128.5, 127.9, 126.1, 123.6, 83.5, 78.7, 70.1, 48.4, 40.4, 34.6, 31.6, 25.6, 24.7, 23.3, 22.4, 21.1, 16.1.

^{11}B NMR (96 MHz, $CDCl_3$) δ 32.31.

HRMS (ESI-TOF) m/z: Calcd. for $C_{33}H_{45}BO_5$ [M+Na]$^+$: 555.3263, Found: 555.3269.



(*E*)-3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-2-(4-((((*3aS,5aR,8aR,8bS*)-2,2,7,7-tetramethyltetrahydro-3a*H*-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-3a-yl)methoxy)methyl)phenyl)prop-1-en-1-yl benzoate (4af)

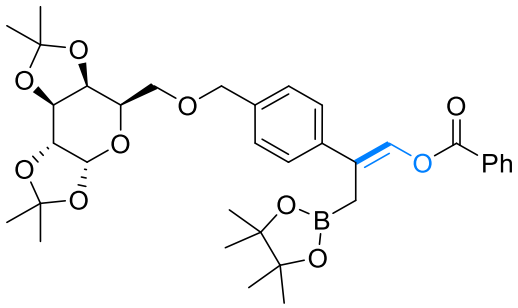
86.6 mg, 68% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1-5/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.19 (dd, *J* = 8.5, 1.5 Hz, 2H), 7.74 (s, 1H), 7.64 – 7.55 (m, 1H), 7.52 – 7.40 (m, 4H), 7.31 (d, *J* = 8.5 Hz, 2H), 4.71 – 4.54 (m, 3H), 4.44 (d, *J* = 2.5 Hz, 1H), 4.25 – 4.18 (m, 1H), 3.92 (dd, *J* = 13.0, 2.0 Hz, 1H), 3.73 (dd, *J* = 13.0, 1.0 Hz, 1H), 3.67 – 3.55 (m, 2H), 2.29 (s, 2H), 1.58 – 1.51 (m, 3H), 1.42 (s, 6H), 1.33 (s, 3H), 1.14 (s, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 163.4, 138.8, 137.0, 133.4, 131.9, 130.1, 129.5, 128.5, 127.7, 126.1, 123.5, 108.9, 108.6, 102.8, 83.5, 73.5, 71.6, 71.1, 70.2, 70.1, 61.0, 26.6, 25.9 , 25.5, 25.0, 24.6, 24.1.

^{11}B NMR (96 MHz, $CDCl_3$) δ 33.17.

HRMS (ESI-TOF) m/z: Calcd. for $C_{35}H_{45}BO_{10}$ [M+Na]$^+$: 659.3009, Found: 659.3009.



(*E*)-3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-2-(4-((((*3aR,5R,5aS,8aS,8bR*)-2,2,7,7-tetramethyltetrahydro-5*H*-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)methoxy)methyl)phenyl)prop-1-en-1-yl benzoate (4ag)

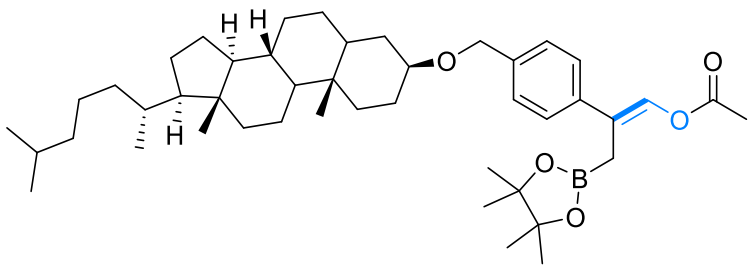
91.7 mg, 72% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1-5/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.31 – 8.13 (m, 2H), 7.74 (s, 1H), 7.65 – 7.54 (m, 1H), 7.52 – 7.40 (m, 4H), 7.32 (d, *J* = 8.5 Hz, 2H), 5.55 (d, *J* = 5.0 Hz, 1H), 4.70 – 4.50 (m, 3H), 4.38 – 4.24 (m, 2H), 4.01 (td, *J* = 6.0, 1.5 Hz, 1H), 3.77 – 3.57 (m, 2H), 2.29 (s, 2H), 1.54 (s, 3H), 1.44 (s, 3H), 1.34 (d, *J* = 2.5 Hz, 6H), 1.15 (s, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 163.4, 138.8, 137.3, 133.4, 131.9, 130.1, 129.5, 128.5, 127.8, 126.2, 123.5, 109.2, 108.6, 96.4, 83.5, 73.0, 71.2, 70.7, 70.6, 68.8, 66.9, 26.1, 26.0, 25.0, 24.6, 24.5.

^{11}B NMR (96 MHz, $CDCl_3$) δ 32.34.

HRMS (ESI-TOF) m/z: Calcd. for $C_{35}H_{45}BO_{10}$ [M+Na]$^+$: 659.3009, Found: 659.3019.



(*E*)-2-(4-((((*3S,8R,10S,13R,14S,17R*)-10,13-Dimethyl-17-((*R*)-6-methylheptan-2-yl)hexadecahydro-1*H*-cyclopenta[a]phenanthren-3-yl)oxy)methyl)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl acetate (4ah)

82.9 mg, 59% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1.

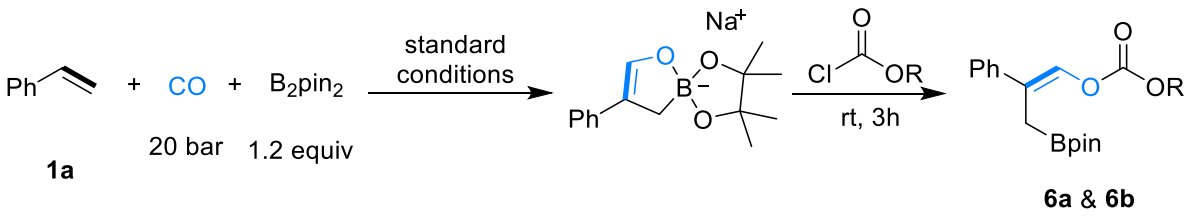
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 (s, 1H), 7.39 – 7.29 (m, 2H), 7.29 – 7.24 (m, 2H), 4.52 (s, 2H), 3.36 – 3.21 (m, 1H), 2.48 (q, *J* = 7.5 Hz, 2H), 2.15 (s, 2H), 1.98 – 1.68 (m, 6H), 1.63 – 1.41 (m, 8H), 1.26 (s, 17H), 1.16 (s, 12H), 0.92 – 0.83 (m, 12H), 0.80 (s, 3H), 0.64 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.4, 138.5, 138.1, 131.8, 127.6, 126.1, 122.7, 83.5, 83.4, 77.8, 69.4, 56.5, 56.3, 54.5, 44.9, 42.6, 40.1, 39.5, 37.0, 36.2, 35.8, 35.5, 34.9, 32.1, 28.9, 28.3, 28.0, 27.6, 25.0, 24.6, 24.2, 23.8, 22.8, 22.6, 21.2, 18.7, 12.3, 12.1, 9.0.

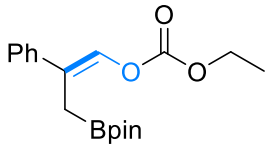
^{11}B NMR (96 MHz, $CDCl_3$) δ 30.83.

HRMS (ESI-TOF) m/z: Calcd. for $C_{46}H_{73}BO_5$ $[M+Na]^+$: 739.5456, Found: 739.5459.

4. Procedure for the synthesis of 6a & 6b.



A vial (4 mL) was charged with CuCl (1.98 mg, 10.0 mol%), Xantphos (11.6 mg, 10.0 mol%), NaO^tBu (48.1 mg, 2.5 equiv), B_2pin_2 (60.1 mg, 1.2 equiv), and a stirring bar. The vial was closed by PTFE/white rubber septum (Wheaton 13 mm Septa) and phenolic cap and connected with atmosphere with a needle. The vial was evacuated under vacuum and recharged with argon for three times. Then, toluene (1.0 mL) was injected under argon by using a syringe. After that, styrene **1a** (0.2 mmol, 1.0 equiv) was added, the vial (or several vials) was placed in an alloy plate, which was transferred into a 300 mL autoclave of the 4560 series from Parr Instruments. After flushing the autoclave three times with CO, a pressure of 20 bar of CO was adjusted at ambient temperature. Then, the reaction was performed for 24 hours at 100 °C. After 24 hours, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. Then, chloroformates (0.4 mmol, 2.0 equiv) was added to the vials by using a micro syringe, the reaction was stirred for 3 more hours at room temperature. After that, the solution was then filtered through celite and concentrated in vacuo. The residue was purified by column chromatography to afford the corresponding products **6a-6b**.



***(E)*-Ethyl (2-phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl) carbonate (6a)**

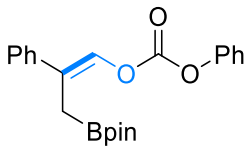
47.2 mg, 71% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 – 7.08 (m, 6H), 4.21 (q, *J* = 7.0 Hz, 2H), 2.17 – 2.03 (m, 2H), 1.28 (t, *J* = 7.0 Hz, 3H), 1.08 (s, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 152.9, 139.2, 133.3, 128.3, 127.1, 126.2, 123.0, 83.4, 64.5, 24.6, 14.3.

^{11}B NMR (96 MHz, $CDCl_3$) δ 32.72.

HRMS (ESI-TOF) m/z: Calcd. for $C_{28}H_{25}BO_5$ $[M+Na]^+$: 355.1696, Found: 355.1694.



***(E)*-Phenyl (2-phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl) carbonate (6b)**

45.6 mg, 60% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

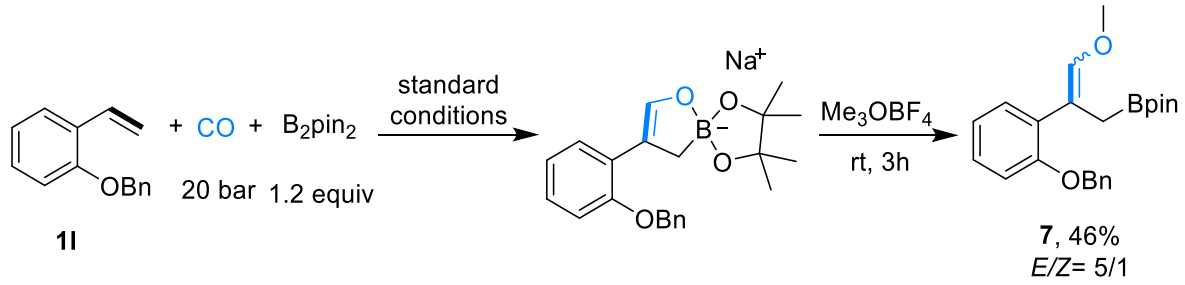
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 – 7.40 (m, 4H), 7.38 – 7.23 (m, 7H), 2.26 (s, 2H), 1.22 (s, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 151.4, 151.0, 138.9, 133.2, 129.6, 128.4, 127.4, 126.3, 126.2, 124.2, 121.0, 83.6, 24.6.

^{11}B NMR (96 MHz, $CDCl_3$) δ 32.59.

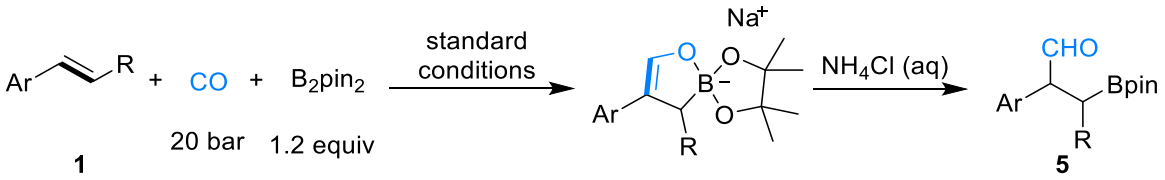
HRMS (ESI-TOF) m/z: Calcd. for $C_{22}H_{25}BO_5$ $[M+Na]^+$: 403.2473, Found: 403.2476.

5. Procedure for the synthesis of 7.

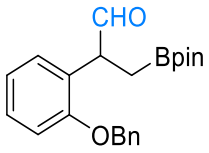


A vial (4 mL) was charged with CuCl (1.98 mg, 10.0 mol%), Xantphos (11.6 mg, 10.0 mol%), NaO^{t}Bu (48.1 mg, 2.5 equiv), B_2pin_2 (60.1 mg, 1.2 equiv), and a stirring bar. The vial was closed by PTFE/white rubber septum (Wheaton 13 mm Septa) and phenolic cap and connected with atmosphere with a needle. The vial was evacuated under vacuum and recharged with argon for three times. Then, toluene (1.0 mL) was injected under argon by using a syringe. After that, styrene **1l** (0.2 mmol, 1.0 equiv) was added, the vial was placed in an alloy plate, which was transferred into a 300 mL autoclave of the 4560 series from Parr Instruments. After flushing the autoclave three times with CO, a pressure of 20 bar of CO was adjusted at ambient temperature. Then, the reaction was performed for 24 hours at 100 °C. After 24 hours, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. Then, trimethyloxonium tetrafluoroborate (0.3 mmol, 1.5 equiv) was added to the vials by using a micro syringe, the reaction was stirred for 3 more hours at room temperature. After that, the solution was then filtered through celite and concentrated in vacuo. The residue was purified by column chromatography (eluent: pentane/ethyl acetate = 30/1-20/1) to afford the corresponding products **7** as a colorless oil (34.9 mg, 46% yield, E/Z= 5/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.50 – 7.42 (m, 2H), 7.41 – 7.32 (m, 4H), 7.22 – 7.16 (m, 1H), 6.92 – 6.83 (m, 2H), 6.30 (s, 1H), 5.09 (s, 2H), 3.62 (s, 3H), 2.15 – 2.00 (m, 2H), 1.15 (s, 12H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 156.5, 145.9, 130.8, 130.1, 128.4, 127.6, 127.3, 127.2, 120.8, 113.7, 112.7, 82.9, 70.4, 59.4, 24.7. **^{11}B NMR** (96 MHz, $CDCl_3$) δ 33.36. **HRMS** (ESI-TOF) m/z: Calcd. for $C_{22}H_{25}BO_5$ $[M+Na]^+$: 403.2060, Found: 403.2056.

6. Procedure for the synthesis of 5.



A vial (4 mL) was charged with CuCl (1.98 mg, 10.0 mol%), Xantphos (11.6 mg, 10.0 mol%), NaO^{t}Bu (48.1 mg, 2.5 equiv), B_2pin_2 (60.1 mg, 1.2 equiv), and a stirring bar. The vial was closed by PTFE/white rubber septum (Wheaton 13 mm Septa) and phenolic cap and connected with atmosphere with a needle. The vial was evacuated under vacuum and recharged with argon for three times. Then, toluene (1.0 mL) was injected under argon by using a syringe. After that, styrene **1** (0.2 mmol, 1.0 equiv) was added, the vial (or several vials) was placed in an alloy plate, which was transferred into a 300 mL autoclave of the 4560 series from Parr Instruments. After flushing the autoclave three times with CO, a pressure of 20 bar of CO was adjusted at ambient temperature. Then, the reaction was performed for 24 hours at 100 °C. After 24 hours, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. The reaction was quenched with 0.5 mL saturated aqueous $NHCl_4$ solution [saturated deuterium water (D_2O) solution of $NHCl_4$] and stirred for 5 minutes at room temperature. After that, the solution was extracted with EA (5 mL×3). The organic layer concentrated in vacuo and the residue was purified by column chromatography (eluent: pentane/ethyl acetate = 30/1-20/1) to afford the corresponding products **5b, 5c,** and **5d.**



2-(2-(Benzyloxy)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propanal (5b)

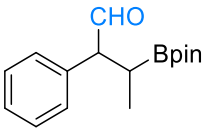
46.9 mg, 64% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.62 (s, 1H), 7.32 – 7.05 (m, 7H), 6.91 – 6.83 (m, 2H), 5.04 (s, 2H), 4.03 (dd, *J* = 8.5, 7.0 Hz, 1H), 1.42 (dd, *J* = 16.0, 8.5 Hz, 1H), 1.13 (s, 6H), 1.11 (s, 6H), 1.09 – 1.02 (m, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 201.8, 156.3, 136.9, 130.0, 128.6, 127.8, 127.0, 121.2, 112.2, 83.2, 70.1, 49.7, 24.7, 24.7.

^{11}B NMR (96 MHz, $CDCl_3$) δ 33.04.

HRMS (ESI-TOF) m/z: Calcd. for $C_{22}H_{27}BO_4$ $[M+Na]^+$: 389.1904, Found: 389.1898.



2-Phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butanal (5c)

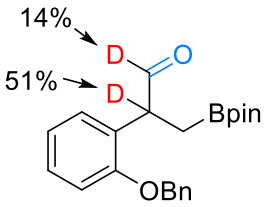
26.9 mg, 49% yield, 1/1 *dr*, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.83 – 9.46 (m, 1H), 7.42 – 7.22 (m, 4H), 7.20 – 7.14 (m,12H), 3.62 (d, *J* = 10.5 Hz, 1H), 1.88 – 1.46 (m, 1H), 1.29 (d, *J* = 19.5 Hz, 6H), 1.06 (s, 6H), 1.05 – 0.80 (m, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 201.3, 201.2, 136.7, 136.1, 129.4, 129.4, 129.0, 128.7, 127.5, 127.4, 83.3, 83.2, 62.9, 61.4, 24.7, 24.5, 24.4, 13.2, 12.7.

^{11}B NMR (128 MHz, $CDCl_3$) δ 33.21.

HRMS (ESI-TOF) m/z: Calcd. for $C_{16}H_{23}BO_3$ $[M+Na]^+$: 297.1640, Found: 297.1647.

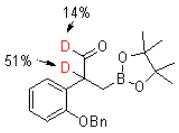


(5d)

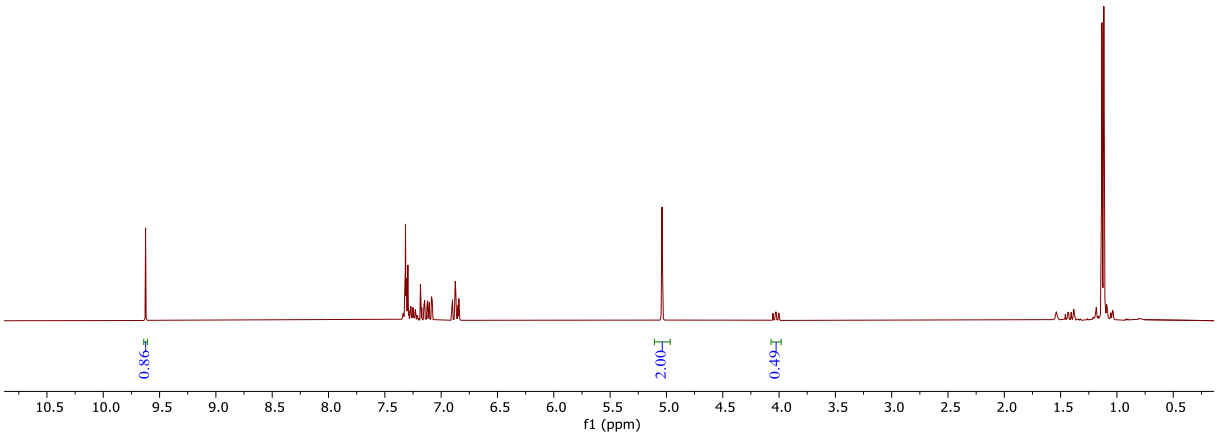
47.7 mg, 61% yield, colorless oil. Eluent: pentane/ethyl acetate = 30/1-20/1.

^{13}C NMR (75 MHz, $CDCl_3$) δ 201.9, 201.9, 156.2, 136.9, 130.0, 129.9, 128.6, 127.8, 127.0, 121.2, 112.2, 83.2, 70.1, 49.7, 24.7, 24.7.

Yang Yuan YY-w-168-D — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 22 — 300.13 MHz

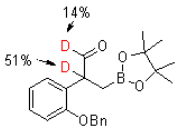


5d

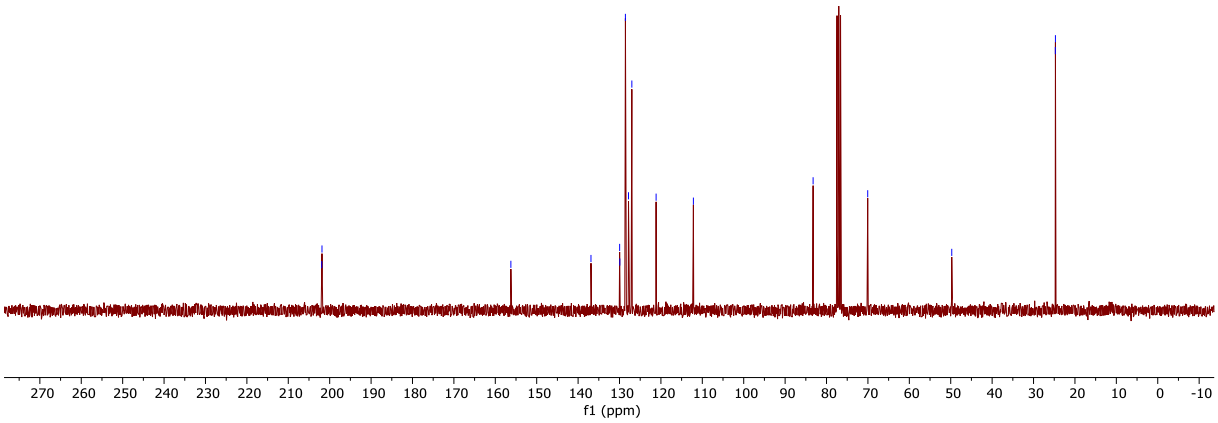


Yang Yuan YY-w-168-D — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 22 — 75.48 MHz

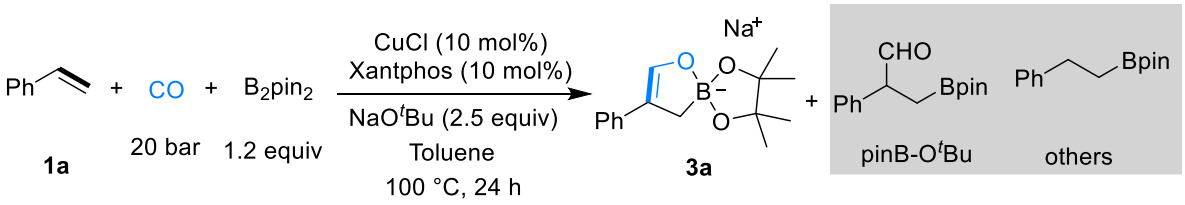
201.92
201.86
156.24
136.89
129.97
129.93
128.57
127.83
127.02
121.16
112.15
83.24
70.05
49.75
24.73
24.71



5d



7. Analysis of the ^{11}B NMR spectroscopies after reaction[1]



A vial (4 mL) was charged with CuCl (1.98 mg, 10.0 mol%), Xantphos (11.6 mg, 10.0 mol%), NaO^{t}Bu (48.1 mg, 2.5 equiv), B_2pin_2 (60.1 mg, 1.2 equiv), and a stirring bar. The vial was closed by PTFE/white rubber septum (Wheaton 13 mm Septa) and phenolic cap and connected with atmosphere with a needle. The vial was evacuated under vacuum and recharged with argon for three times. Then, toluene (1.0 mL) was injected under argon by using a syringe. After that, styrene **1a** (0.2 mmol, 1.0 equiv) was added, the vial was placed in an alloy plate, which was transferred into a 300 mL autoclave of the 4560 series from Parr Instruments. After flushing the autoclave three times with CO, a pressure of 20 bar of CO was adjusted at ambient temperature. Then, the reaction was performed for 24 hours at 100 °C. After 24 hours, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. After that, remove the solvent (toluene) and add DMSO-*d*6 as deuterium solvent. Then, the mixture was transferred to an NMR tube and the raw ^{11}B NMR spectra was measured immediately (**Figure 1a**). For the ^{11}B NMR spectra of **3a**, wash the residue with pentane (5 mL) two times after removed the solvent (toluene) and then, measured the ^{11}B NMR spectra in DMSO-*d*6 (**Figure 1b**).

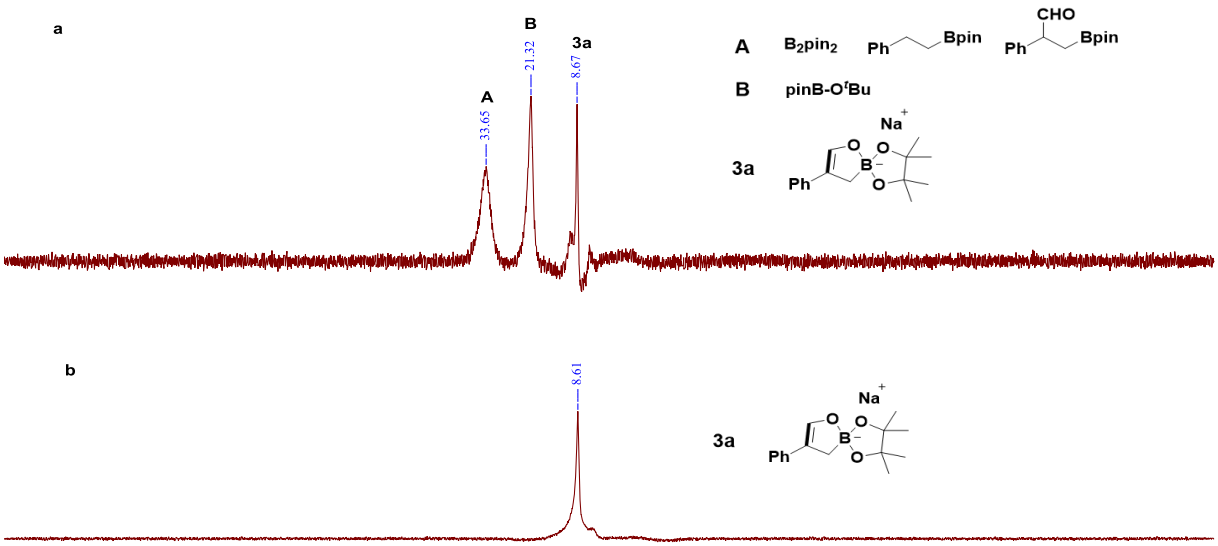
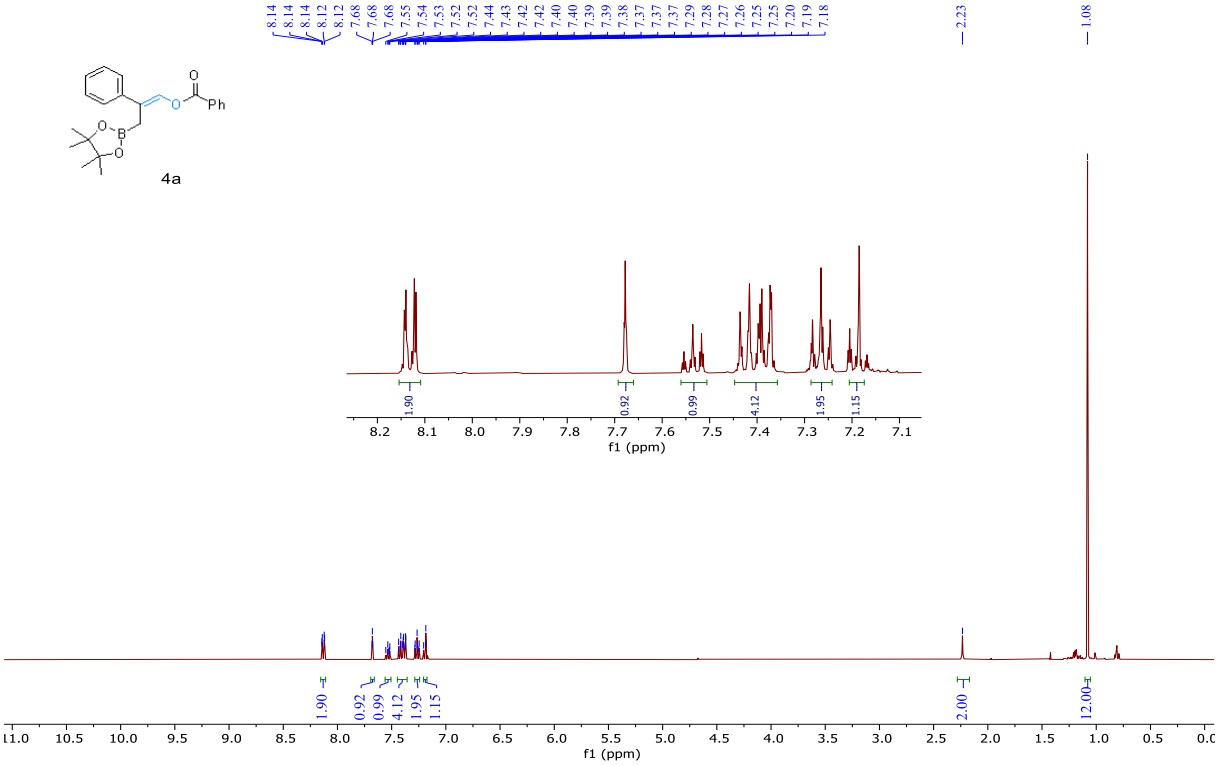


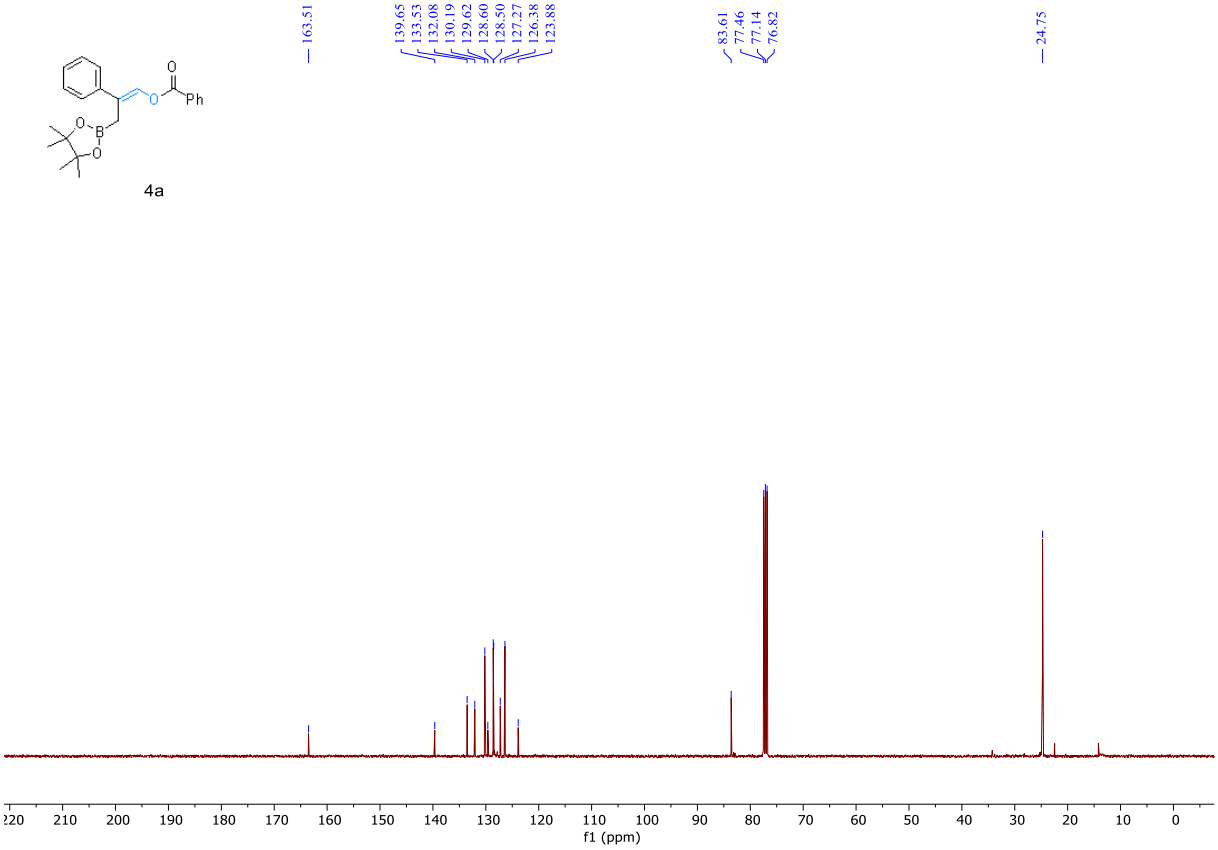
Figure 1. ^{11}B NMR spectroscopies (96.3 HZ, DMSO-*d*6)

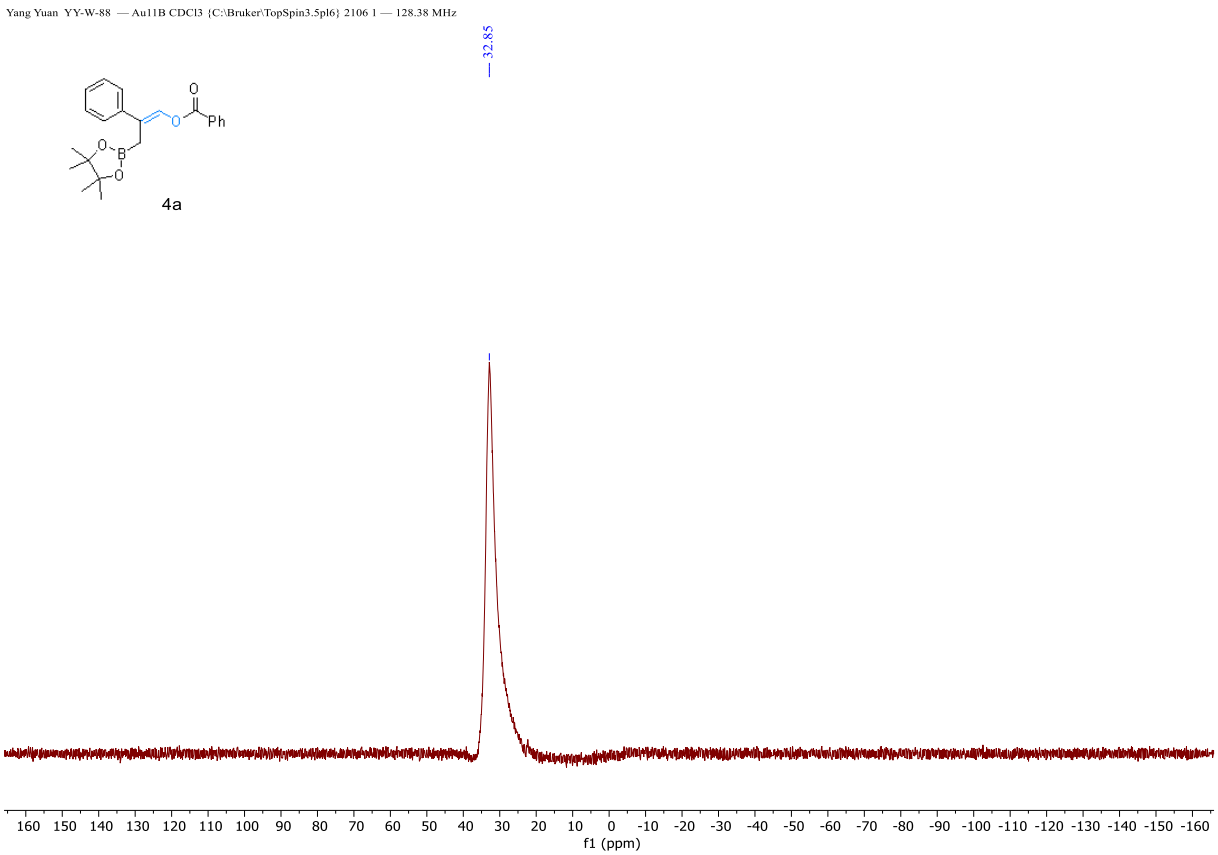
8. References.

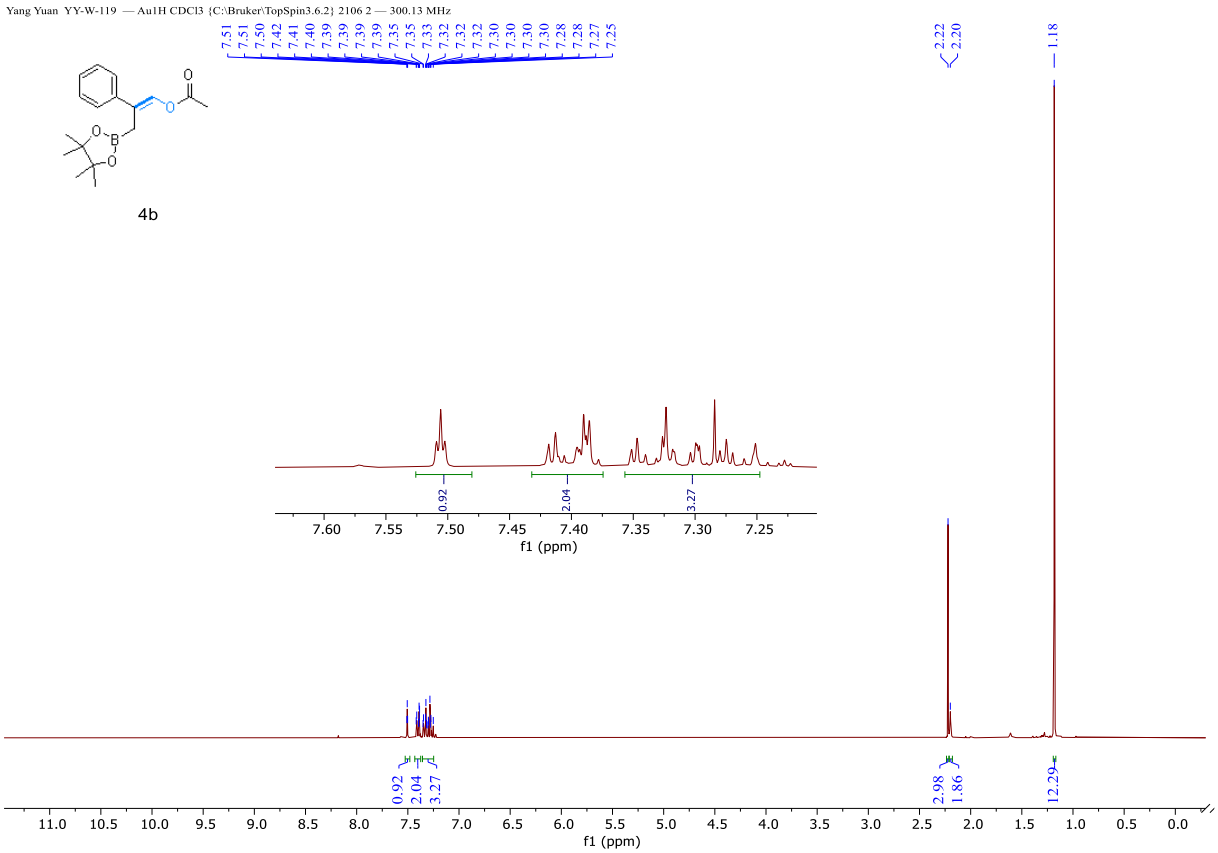
[1] a) M. V. Joannou, B. S. Moyer, S. J. Meek, *J. Am. Chem. Soc.* **2015**, *137*, 6176-6179; b) B. Lee, P. J. Chirik, *J. Am. Chem. Soc.* **2020**, *142*, 2429-2437; c) C. Kim, B. Roh, H. G. Lee, *Chem. Sci.* **2021**, *12*, 3668-3673; d) L. Zhang, J. Cheng, B. Carry, Z. Hou, *J. Am. Chem. Soc.* **2012**, *134*, 14314-14317; e) B. Carry, L. Zhang, M. Nishiura, Z. Hou, *Angew. Chem. Int. Ed.* **2016**, *55*, 6257-6260; f) Z. Li, L. Zhang, M. Nishiura, G. Luo, Y. Luo, Z. Hou, *J. Am. Chem. Soc.* **2020**, *142*, 1966-1974.

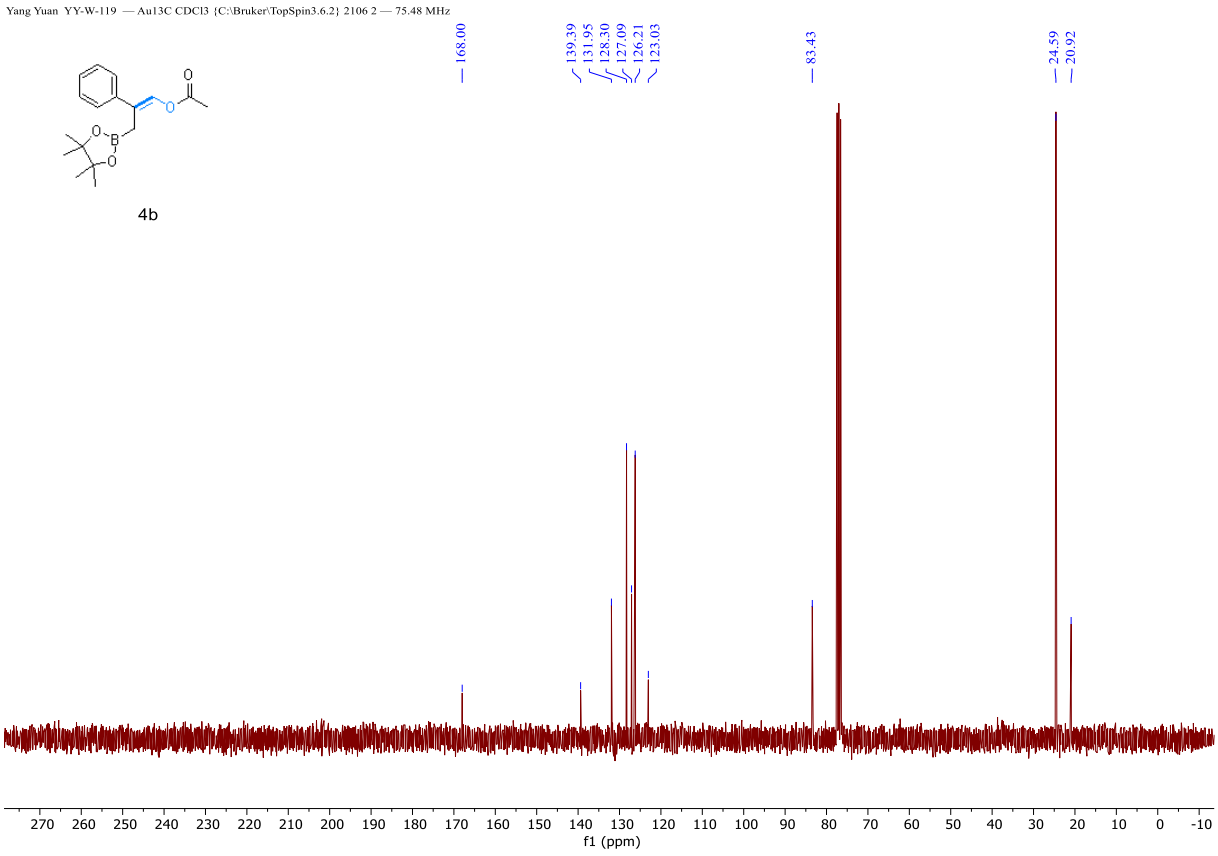
9. NMR Spectra

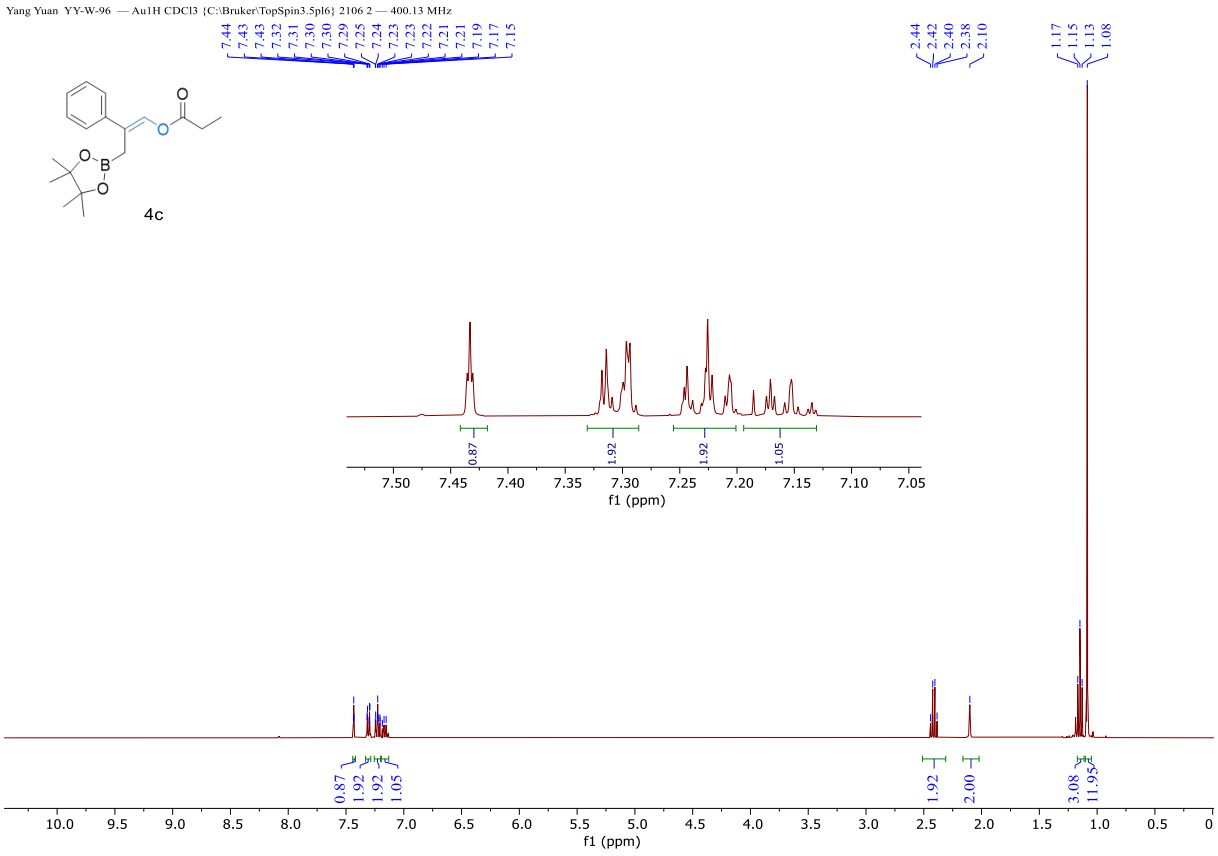


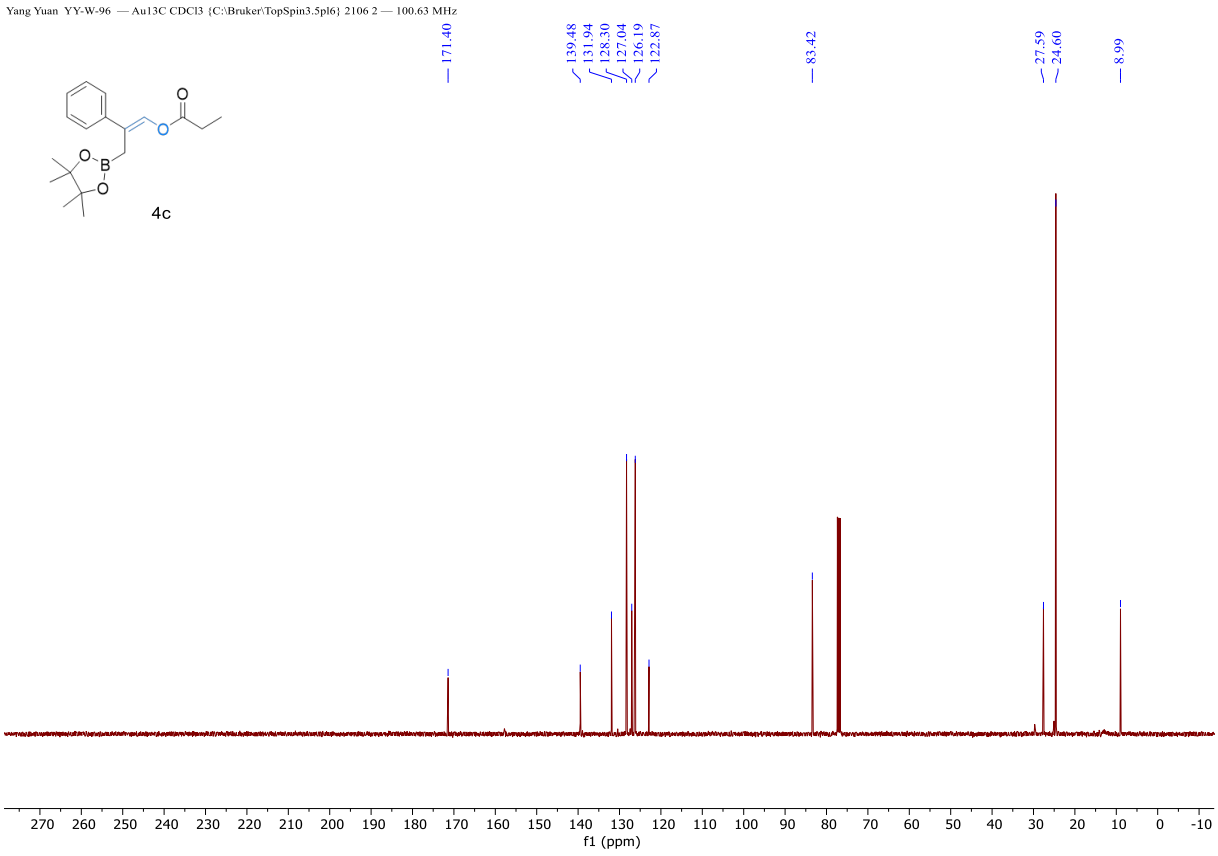


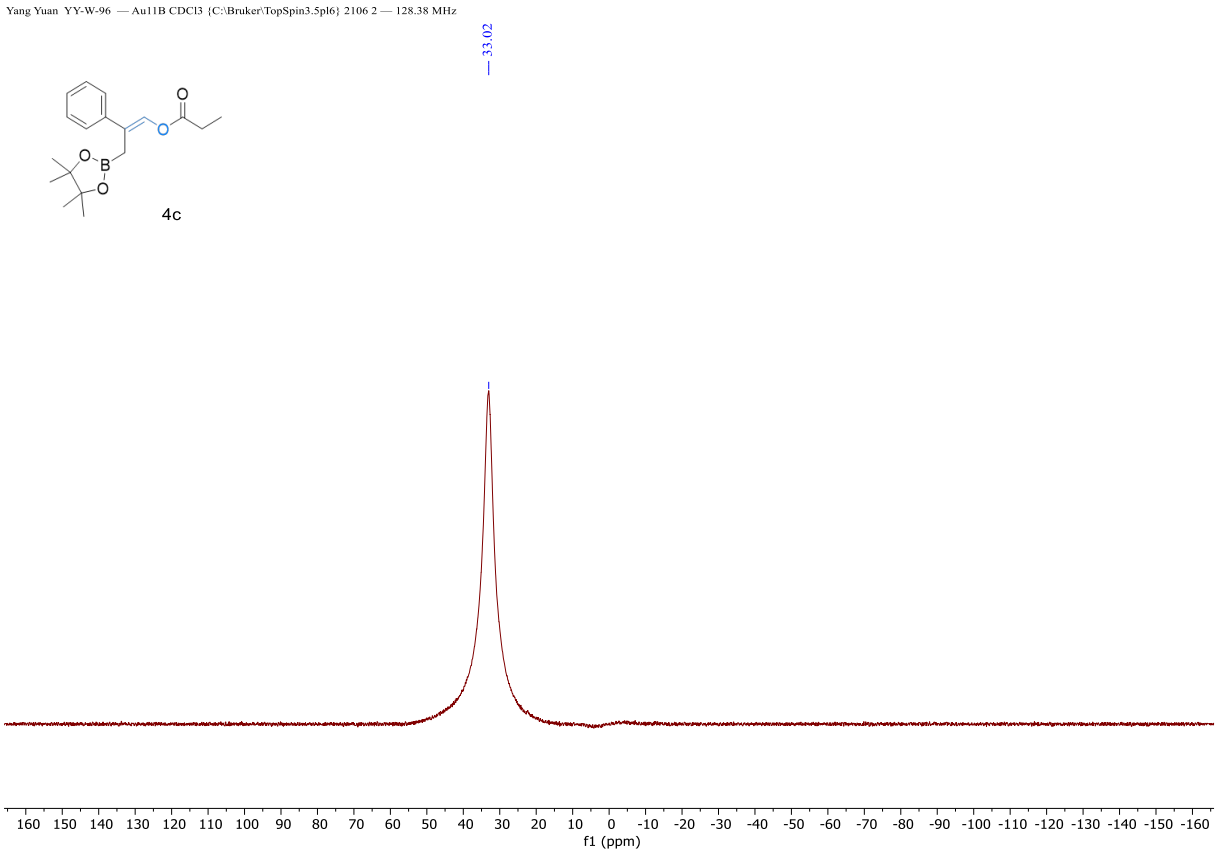

Yang Yuan YY-W-88 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 1 — 128.38 MHz
32.85
O
O
Ph
O
B
O
4a
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)


Yang Yuan YY-W-119 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 2 — 300.13 MHz
4b
f1 (ppm)

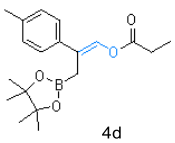

Yang Yuan YY-W-119 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 2 — 75.48 MHz
4b
f1 (ppm)


Yang Yuan YY-W-96 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 2 — 400.13 MHz
7.44
7.43
7.43
7.32
7.31
7.30
7.30
7.29
7.25
7.24
7.23
7.23
7.22
7.21
7.21
7.19
7.17
7.15
2.44
2.42
2.40
2.38
2.10
1.17
1.15
1.13
1.08
4c
0.87
1.92
1.92
1.05
f1 (ppm)
0.87
1.92
1.92
1.05
1.92
2.00
3.08
11.95
f1 (ppm)


Yang Yuan YY-W-96 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 2 — 100.63 MHz
171.40
139.48
131.94
128.30
127.04
126.19
122.87
83.42
27.59
24.60
8.99
4c
f1 (ppm)

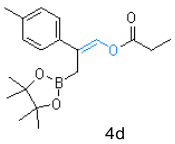

Yang Yuan YY-W-96 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 2 — 128.38 MHz
33.02
O
O
B
O
O
4c
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

Yang Yuan YY-W-102-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 8 — 400.13 MHz

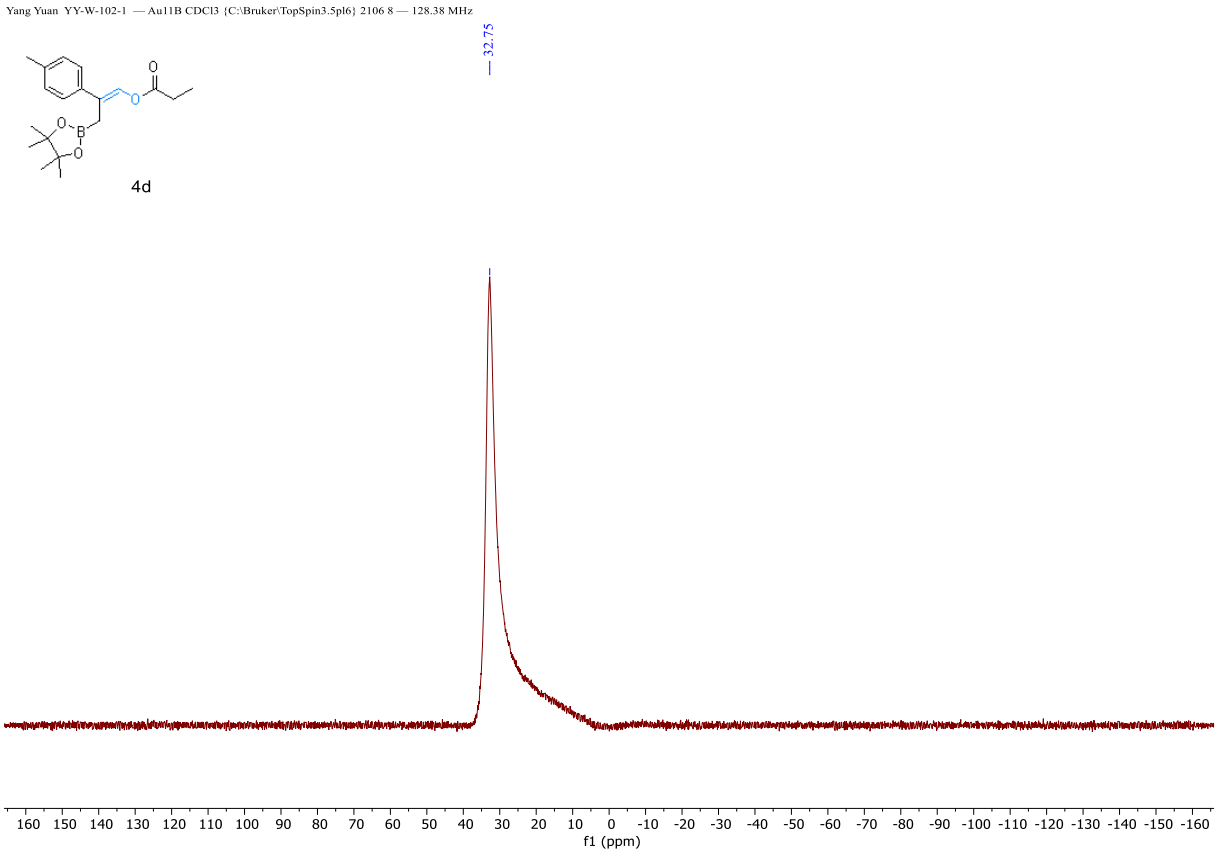


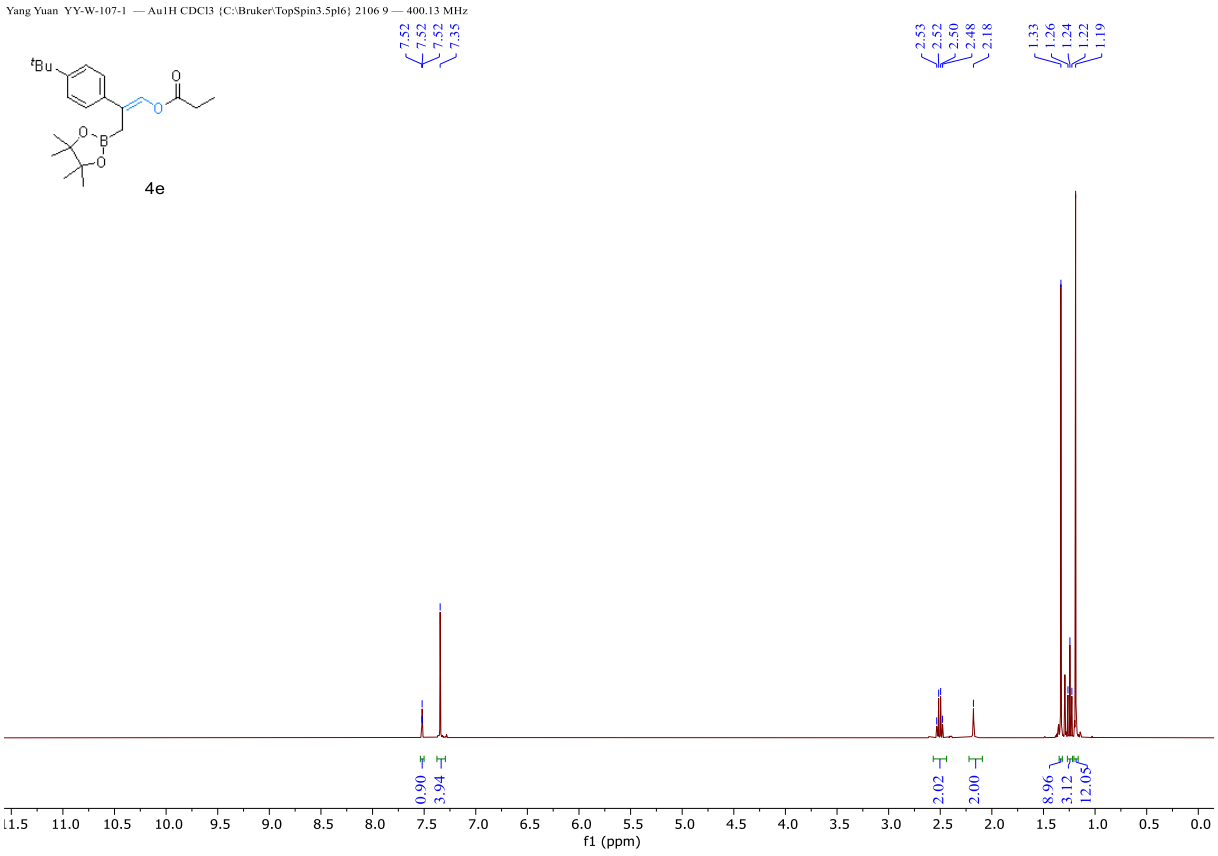
4d

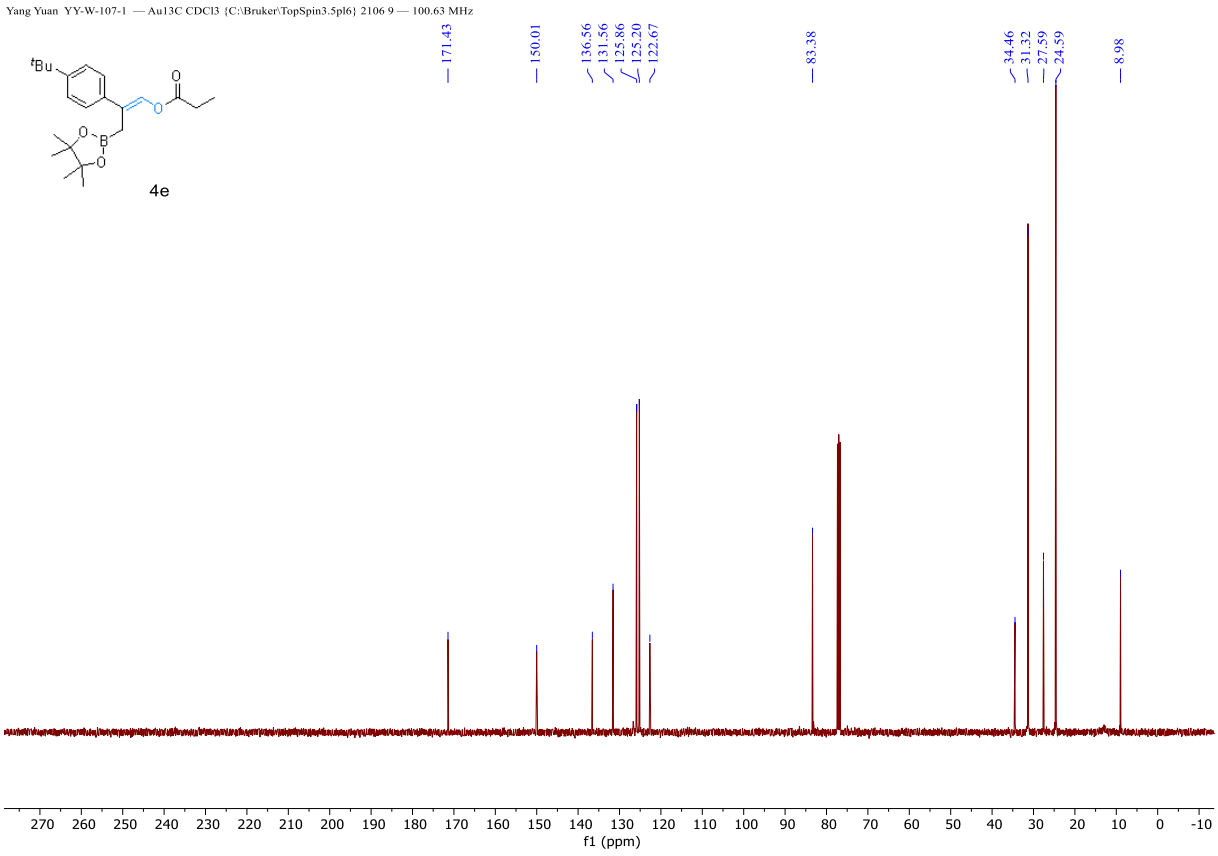
Yang Yuan YY-W-102-1 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 8 — 100.63 MHz

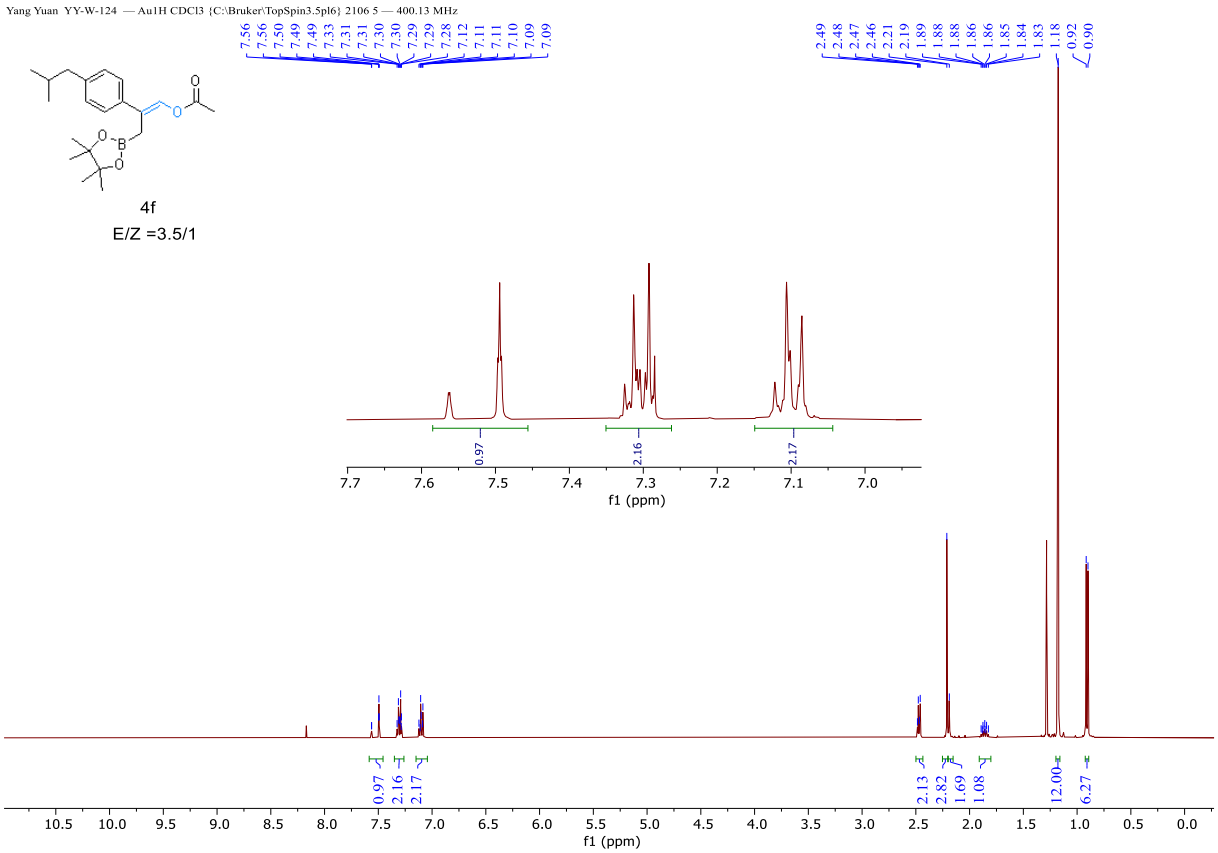


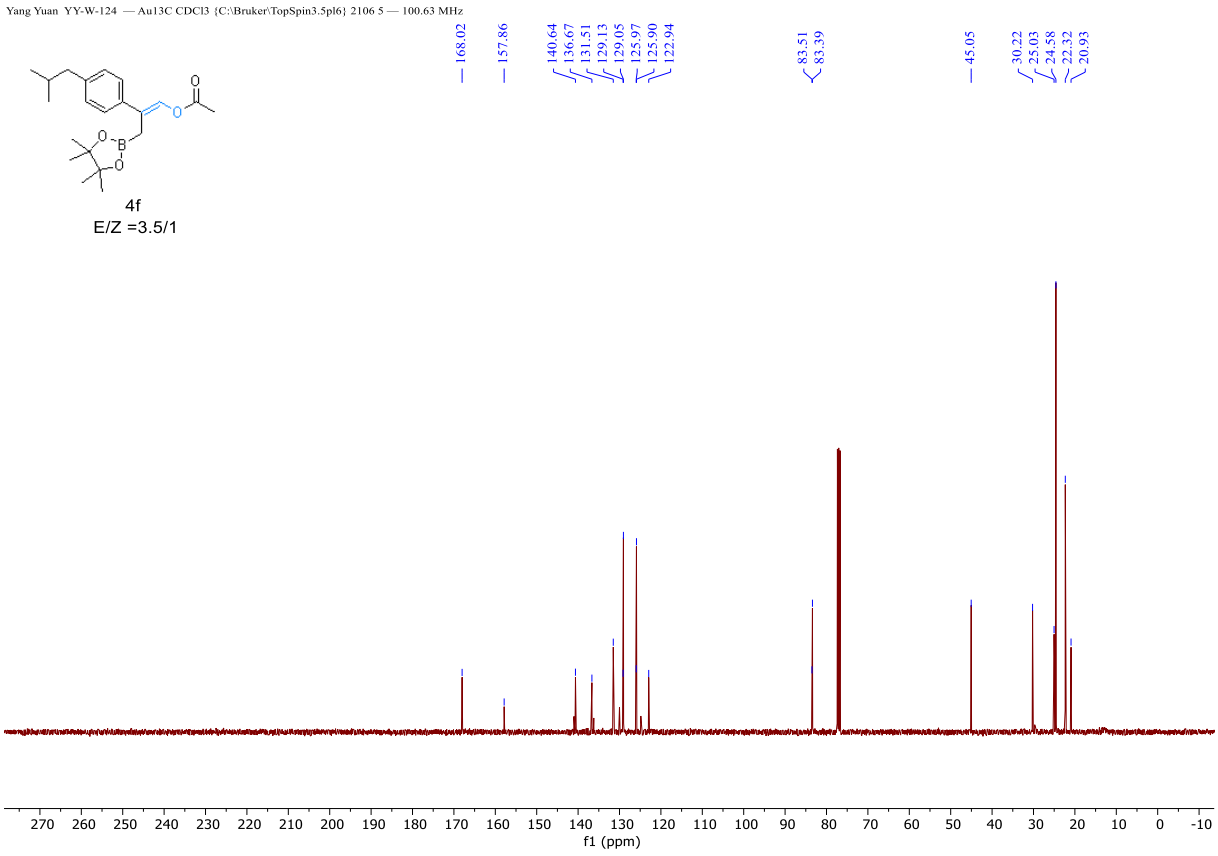
4d


Yang Yuan YY-W-102-1 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 8 — 128.38 MHz
32.75
4d
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

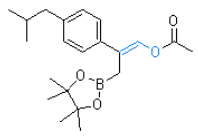

Yang Yuan YY-W-107-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 9 — 400.13 MHz
7.52
7.52
7.52
7.35
2.53
2.52
2.50
2.48
2.18
1.33
1.26
1.24
1.22
1.19
4e
0.90
3.94
2.02
2.00
8.96
3.12
12.05
f1 (ppm)


Yang Yuan YY-W-107-1 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 9 — 100.63 MHz
171.43
150.01
136.56
131.56
125.86
125.20
122.67
83.38
34.46
31.32
27.59
24.59
8.98
4e
f1 (ppm)

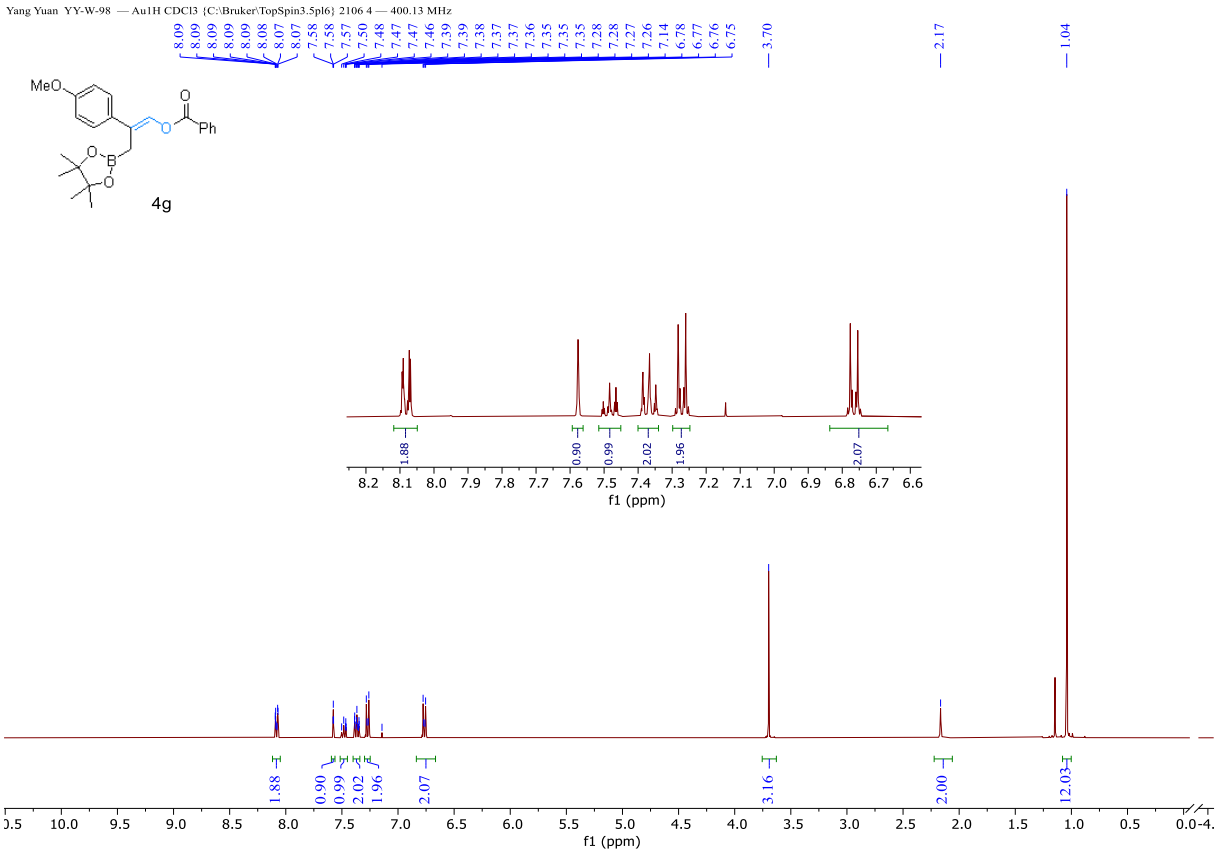

Yang Yuan YY-W-124 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 5 — 400.13 MHz
4f
E/Z =3.5/1
f1 (ppm)

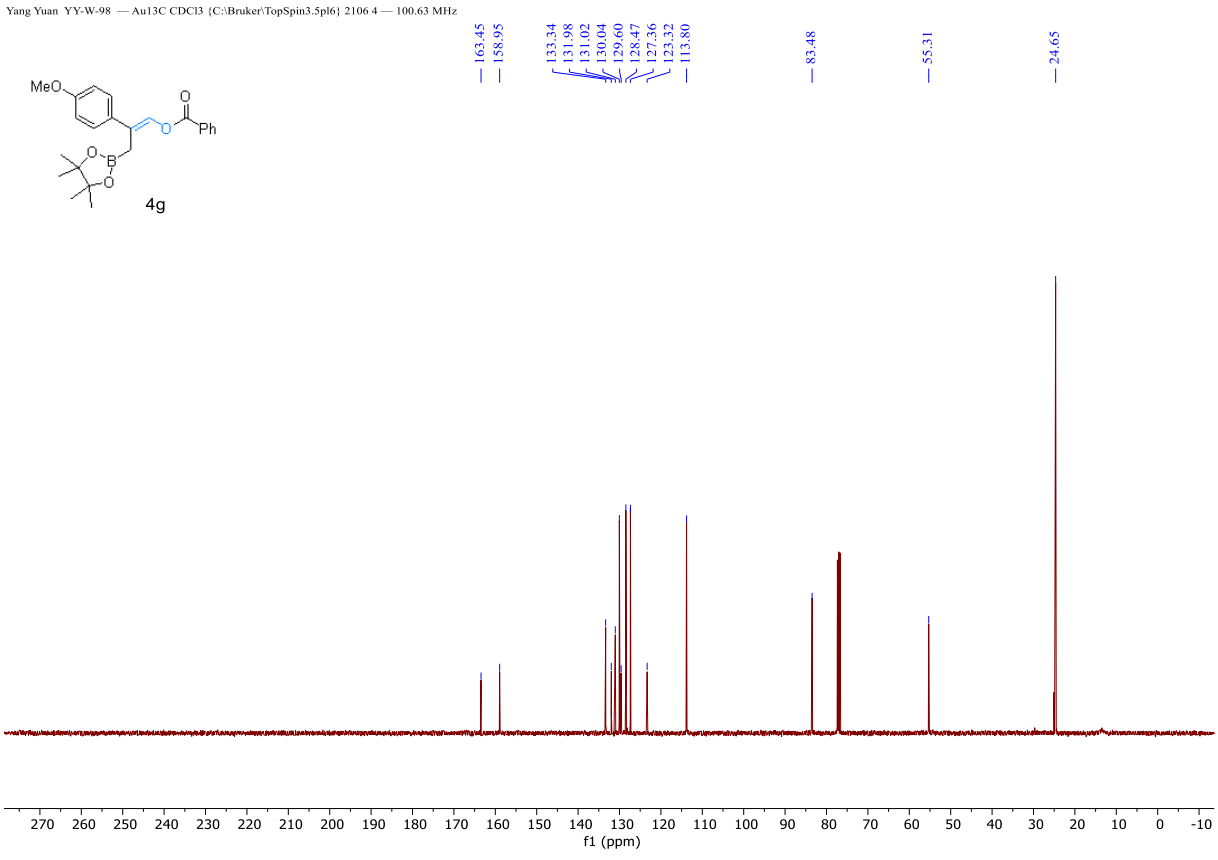

Yang Yuan YY-W-124 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 5 — 100.63 MHz
4f
E/Z =3.5/1
f1 (ppm)

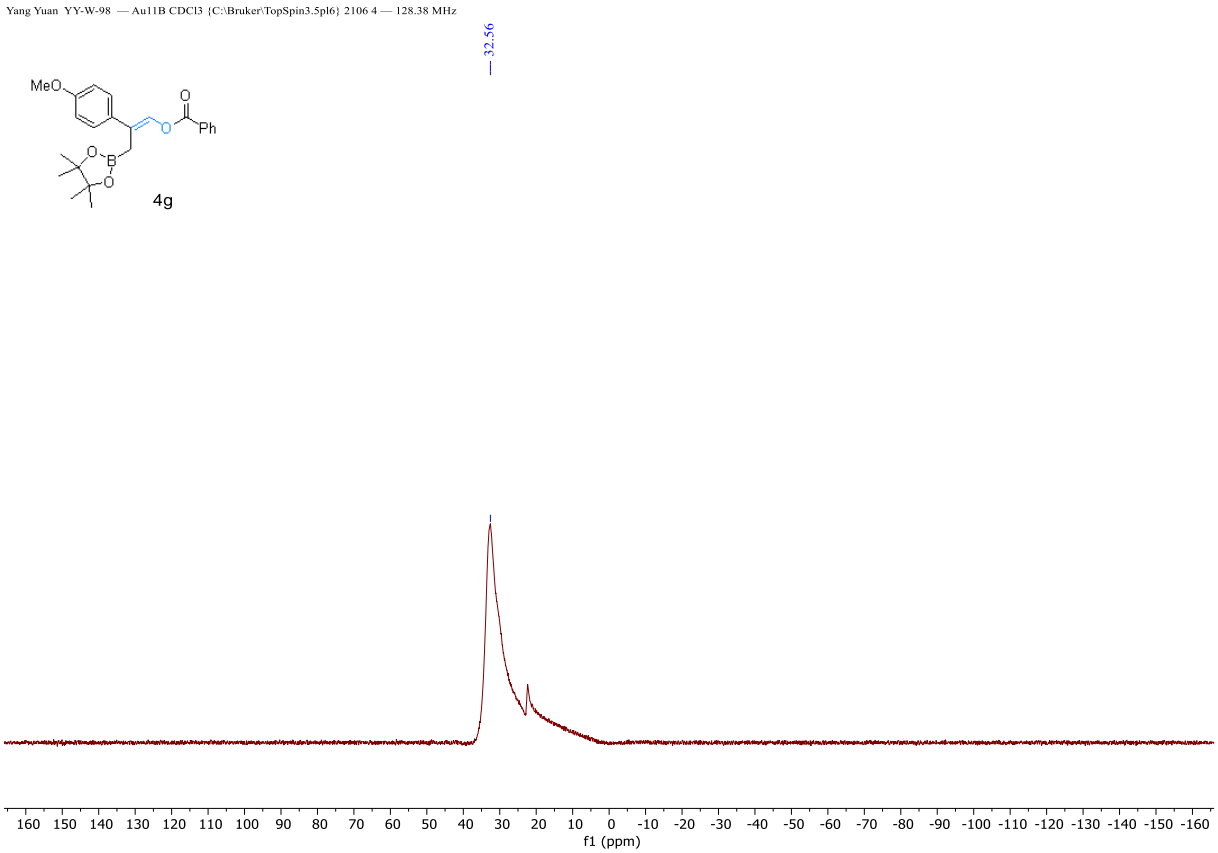
Yang Yuan YY-W-124 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 5 — 128.38 MHz

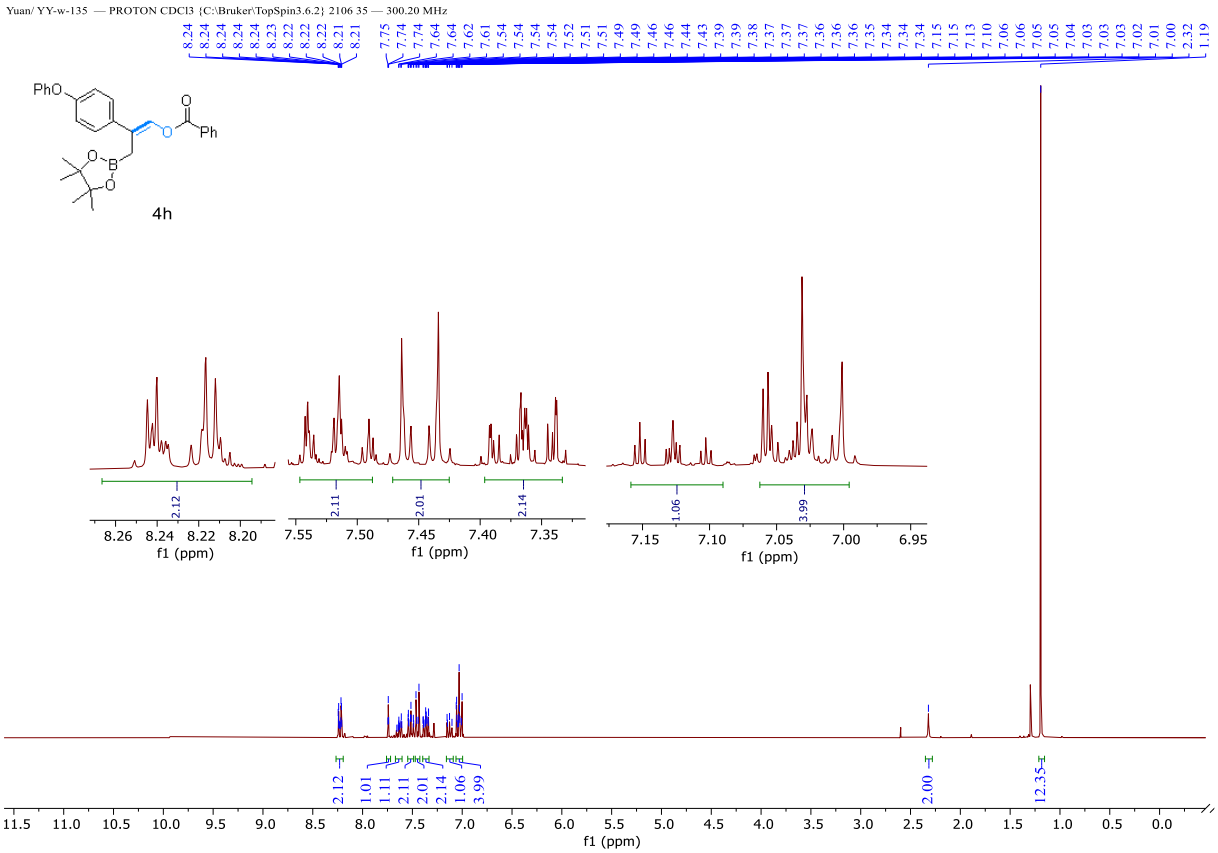


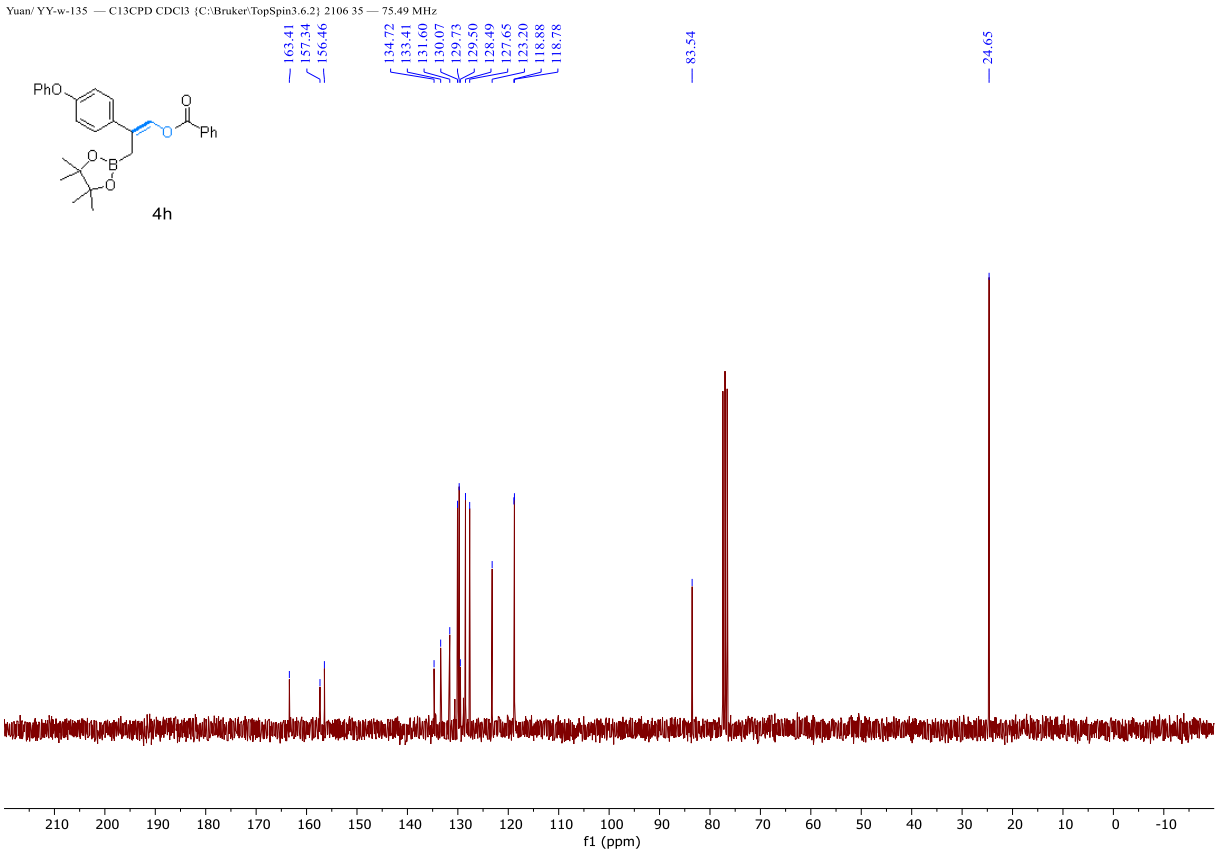
4f
E/Z =3.5/1

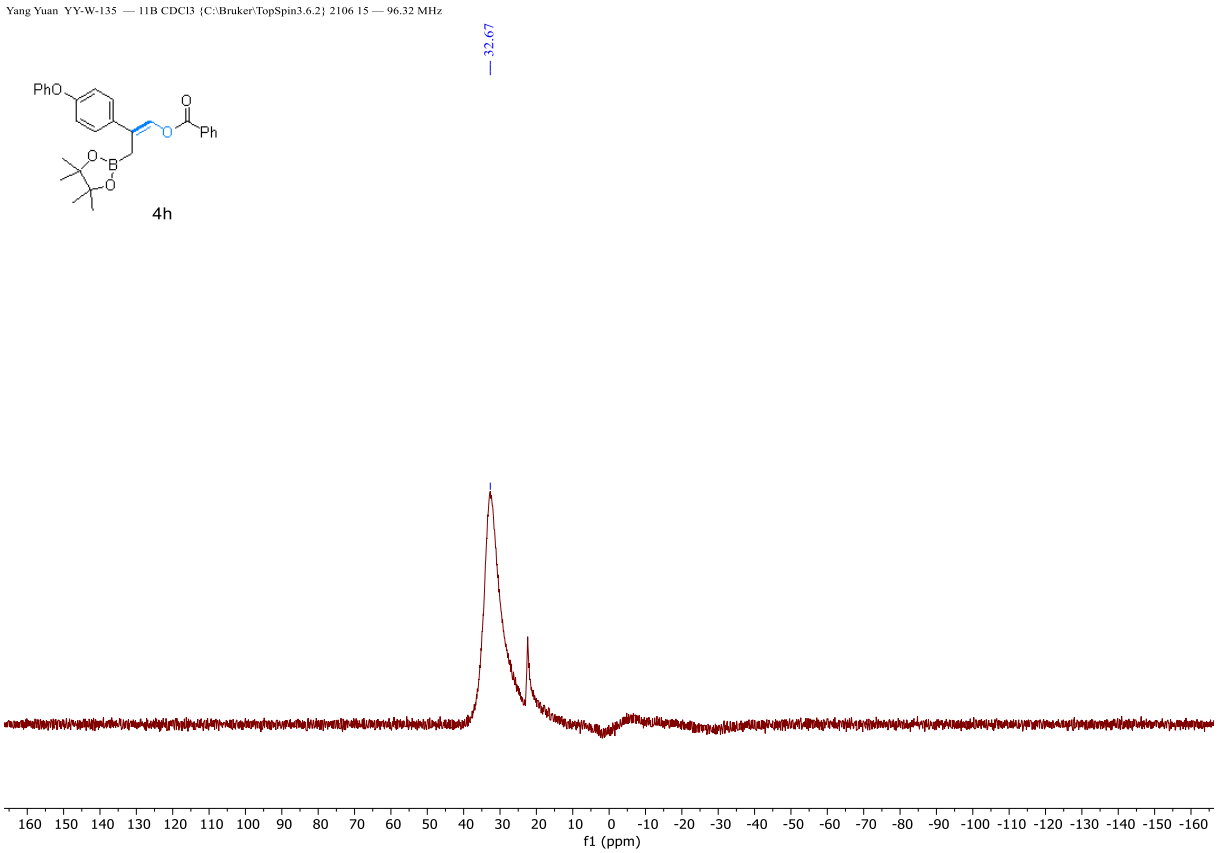

Yang Yuan YY-W-98 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 4 — 400.13 MHz
MeO
O
Ph
O
B
O
4g
f1 (ppm)

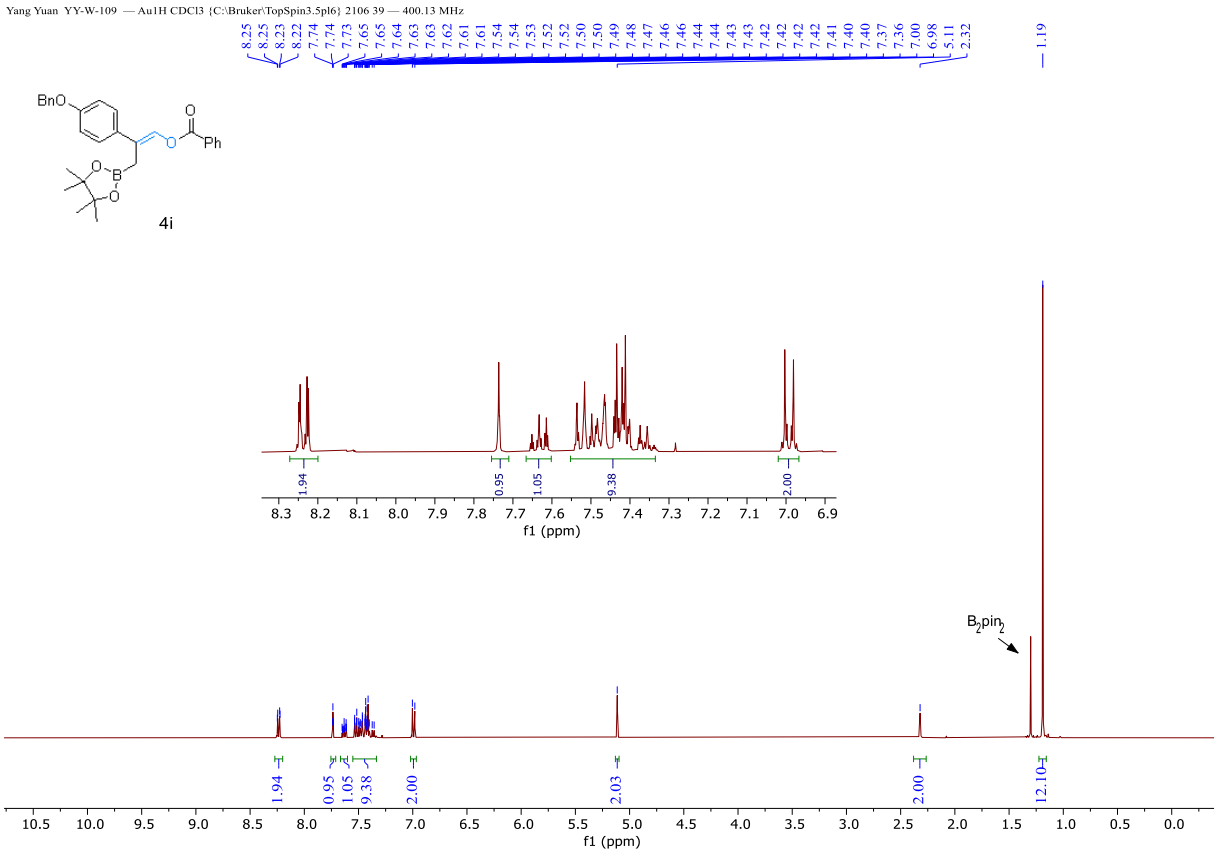

Yang Yuan YY-W-98 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 4 — 100.63 MHz
MeO
O
Ph
O
B
O
4g
f1 (ppm)

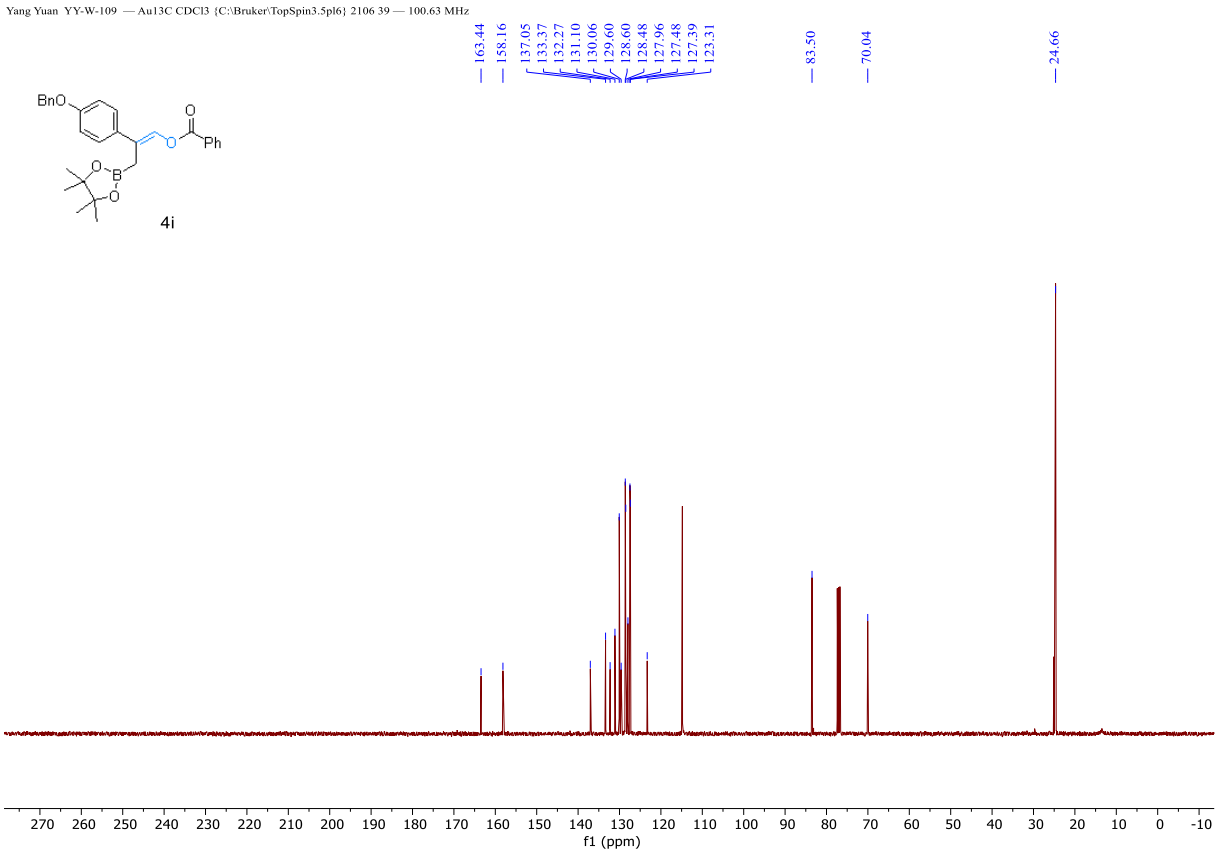

Yang Yuan YY-W-98 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 4 — 128.38 MHz
32.56
MeO
O
O
Ph
O
B
O
4g
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

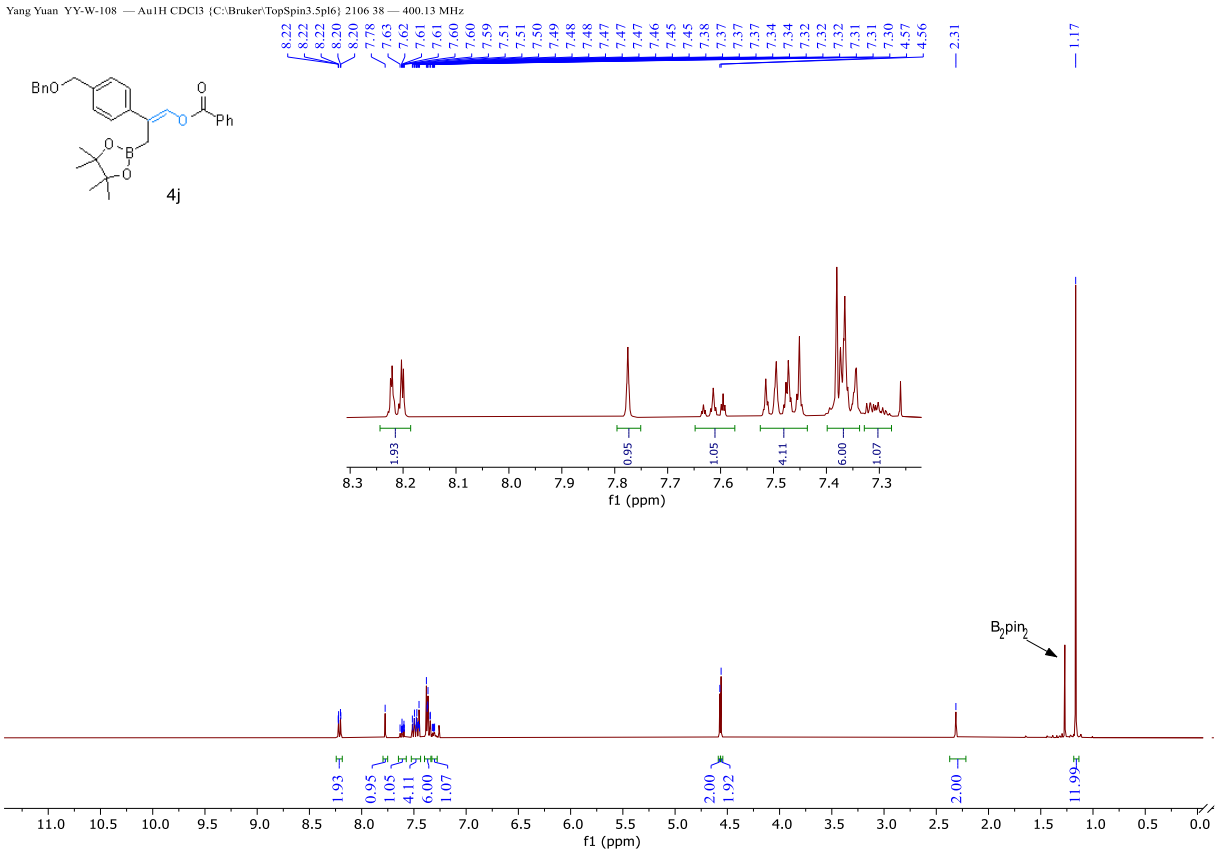

Yuan/ YY-w-135 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 35 — 300.20 MHz
PhO
O
Ph
O
B
O
4h
f1 (ppm)

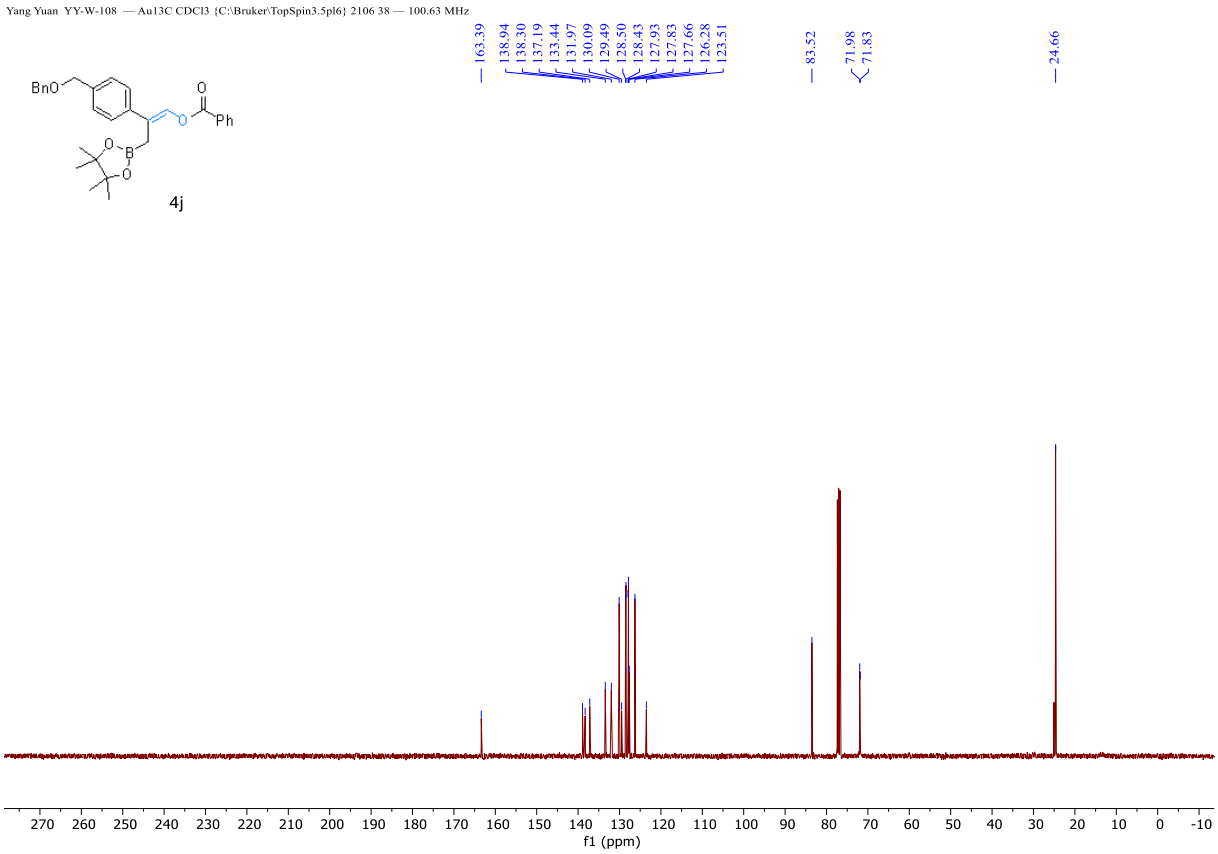

Yuan/ YY-w-135 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 35 — 75.49 MHz
PhO
O
Ph
O
B
O
4h
f1 (ppm)

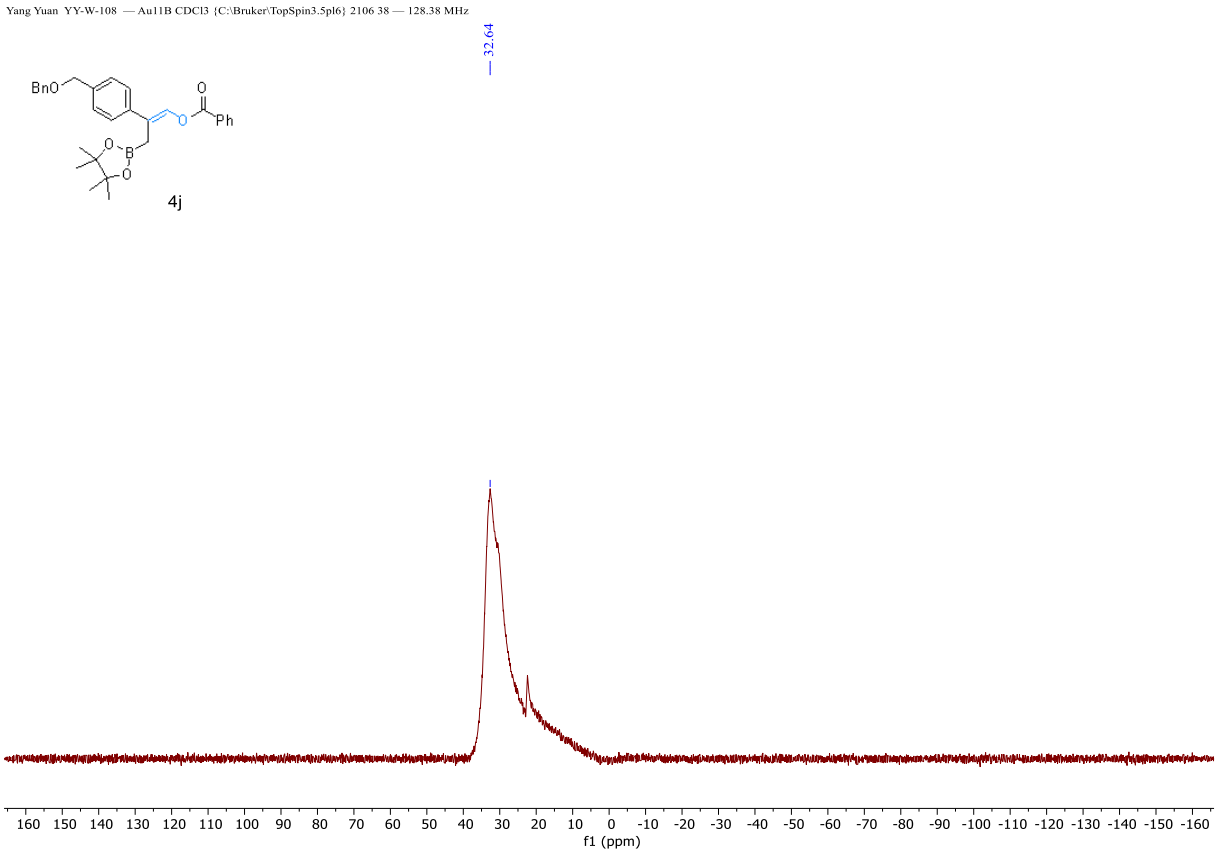

Yang Yuan YY-W-135 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 15 — 96.32 MHz
32.67
PhO
O
Ph
O
O
B
O
4h
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

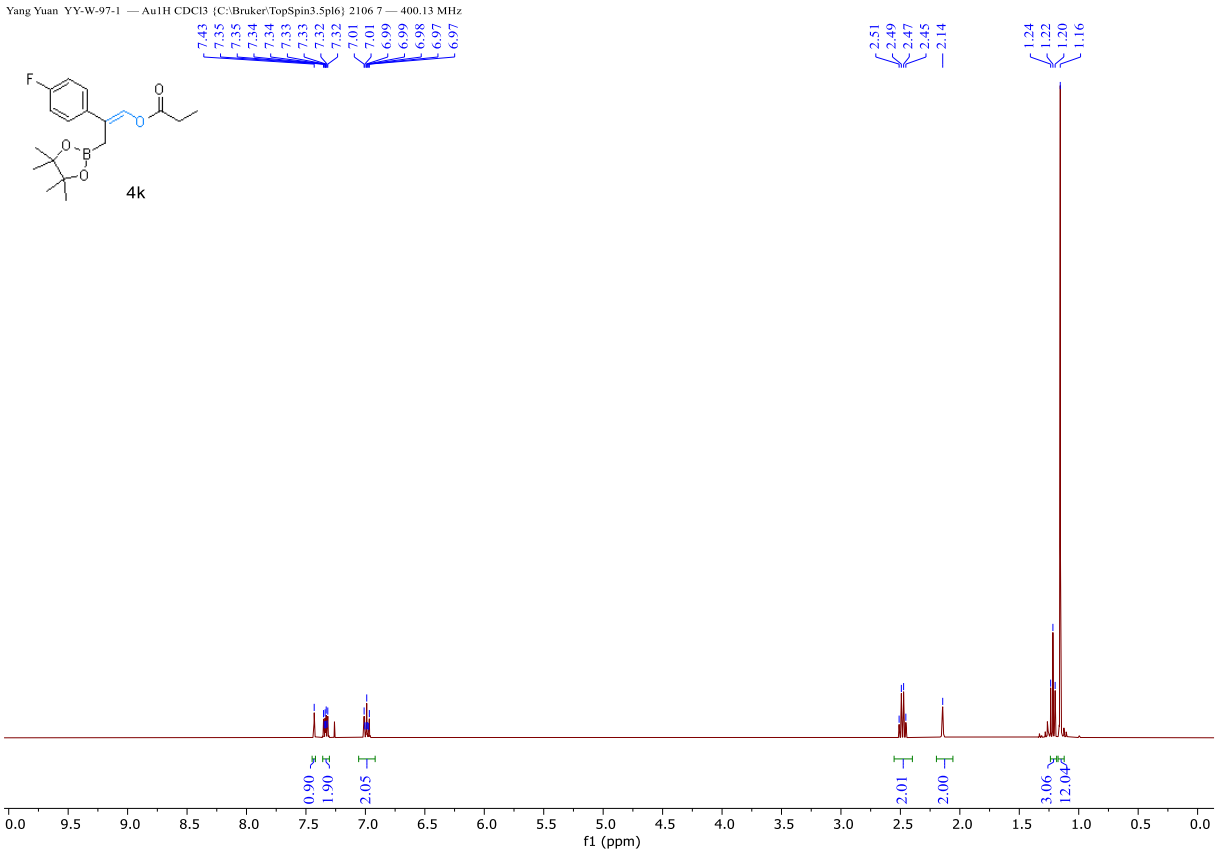

Yang Yuan YY-W-109 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 39 — 400.13 MHz
8.25 8.25 8.23 8.22 7.74 7.74 7.73 7.65 7.65 7.64 7.63 7.63 7.62 7.61 7.61 7.54 7.54 7.53 7.52 7.52 7.50 7.50 7.49 7.48 7.47 7.46 7.46 7.44 7.44 7.43 7.43 7.42 7.42 7.42 7.42 7.41 7.40 7.40 7.37 7.36 7.00 6.98 5.11 2.32 1.19
BnO
O
Ph
O
B
O
4i
1.94 0.95 1.05 9.38 2.00
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9
f1 (ppm)
B2pin2
1.94 0.95 1.05 9.38 2.00 2.03 2.00 12.10
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

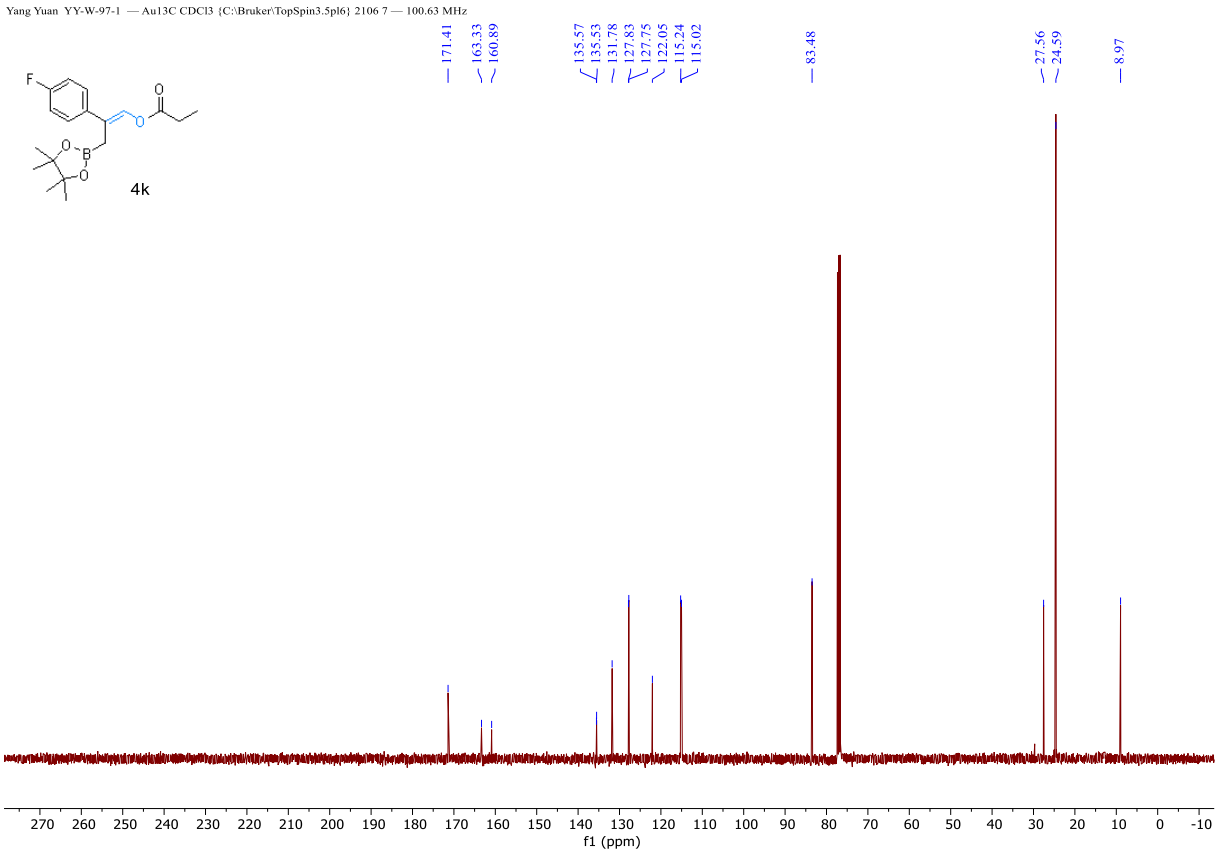

Yang Yuan YY-W-109 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 39 — 100.63 MHz
163.44 158.16 137.05 133.37 132.27 131.10 130.06 129.60 128.48 127.96 127.48 127.39 123.31 83.50 70.04 24.66
BnO
O
Ph
O
B
O
4i
270 260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

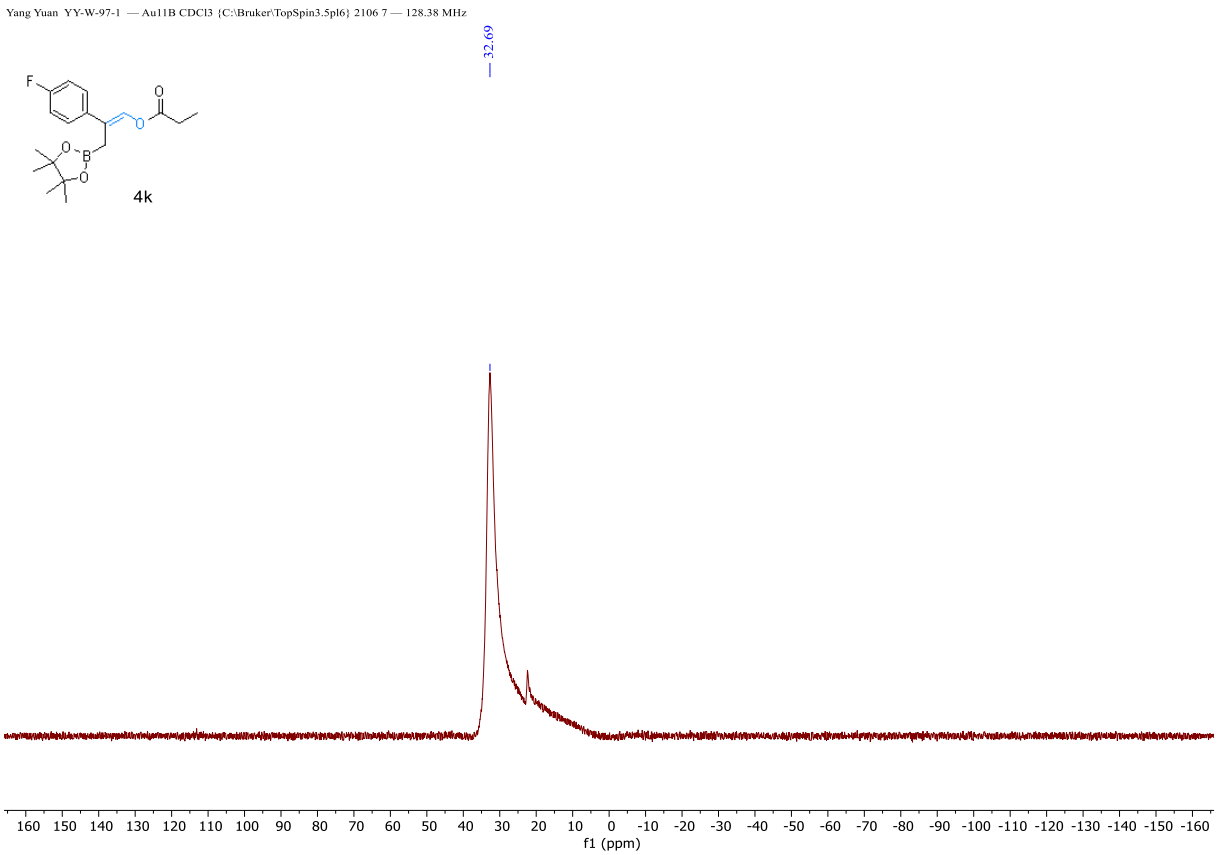

Yang Yuan YY-W-108 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 38 — 400.13 MHz
BnO
O
Ph
O
B
O
4j
f1 (ppm)
B2pin2
f1 (ppm)

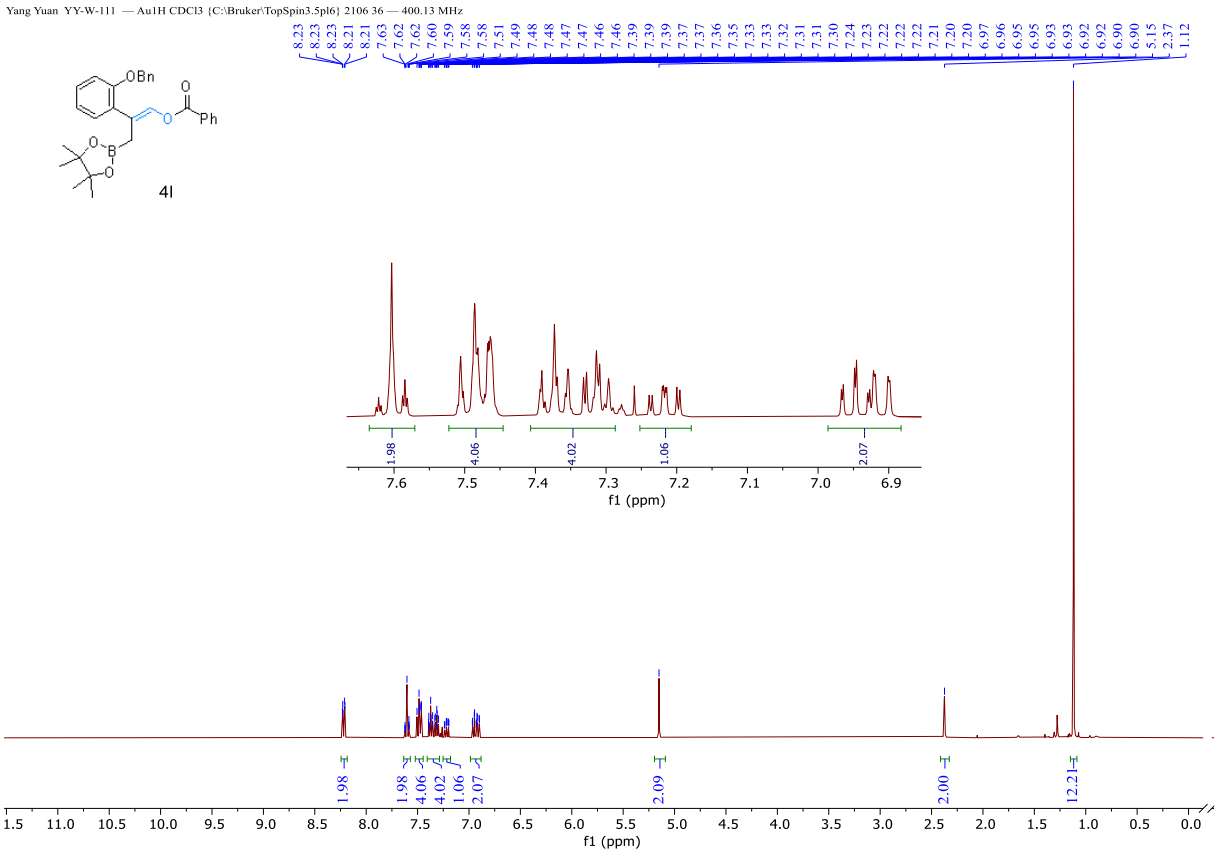

Yang Yuan YY-W-108 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 38 — 100.63 MHz
BnO
O
Ph
O
B
O
4j
f1 (ppm)

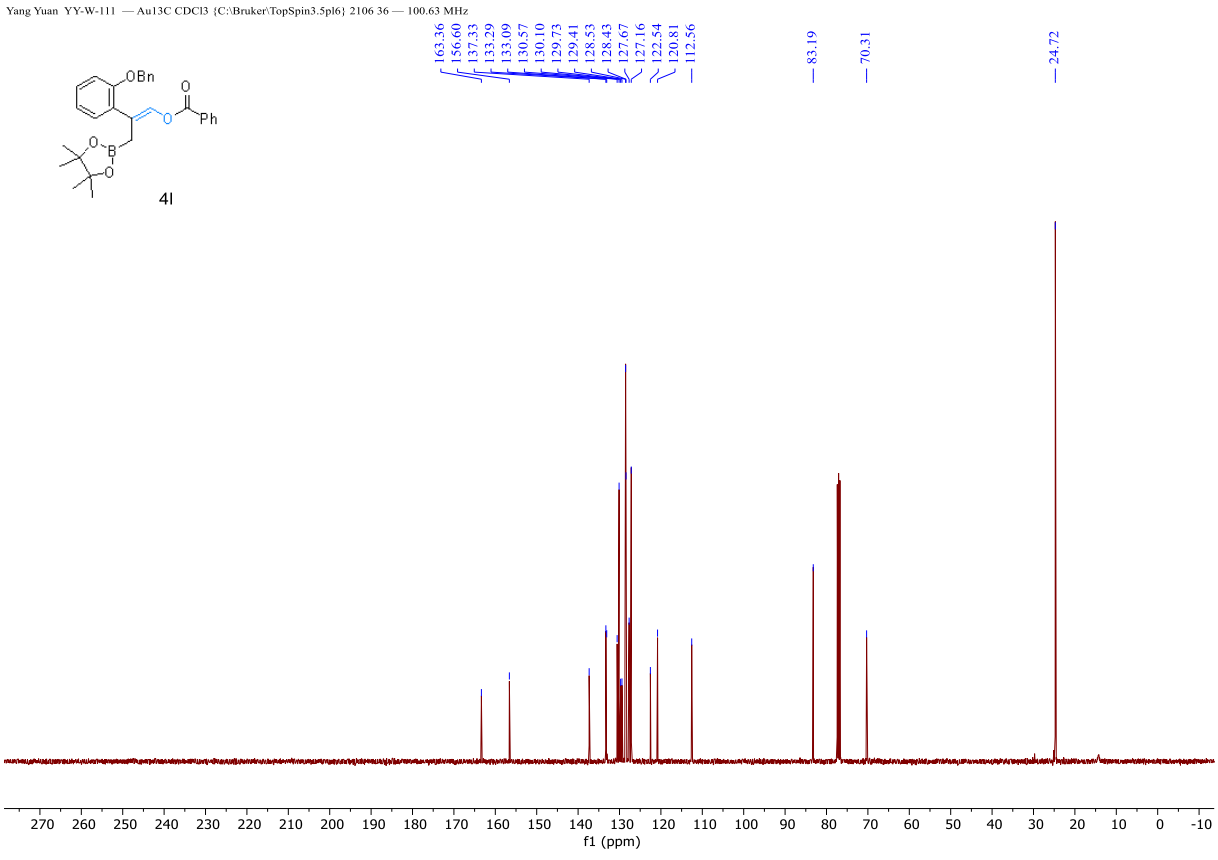

Yang Yuan YY-W-108 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 38 — 128.38 MHz
32.64
BnO
O
Ph
O
B
O
4j
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

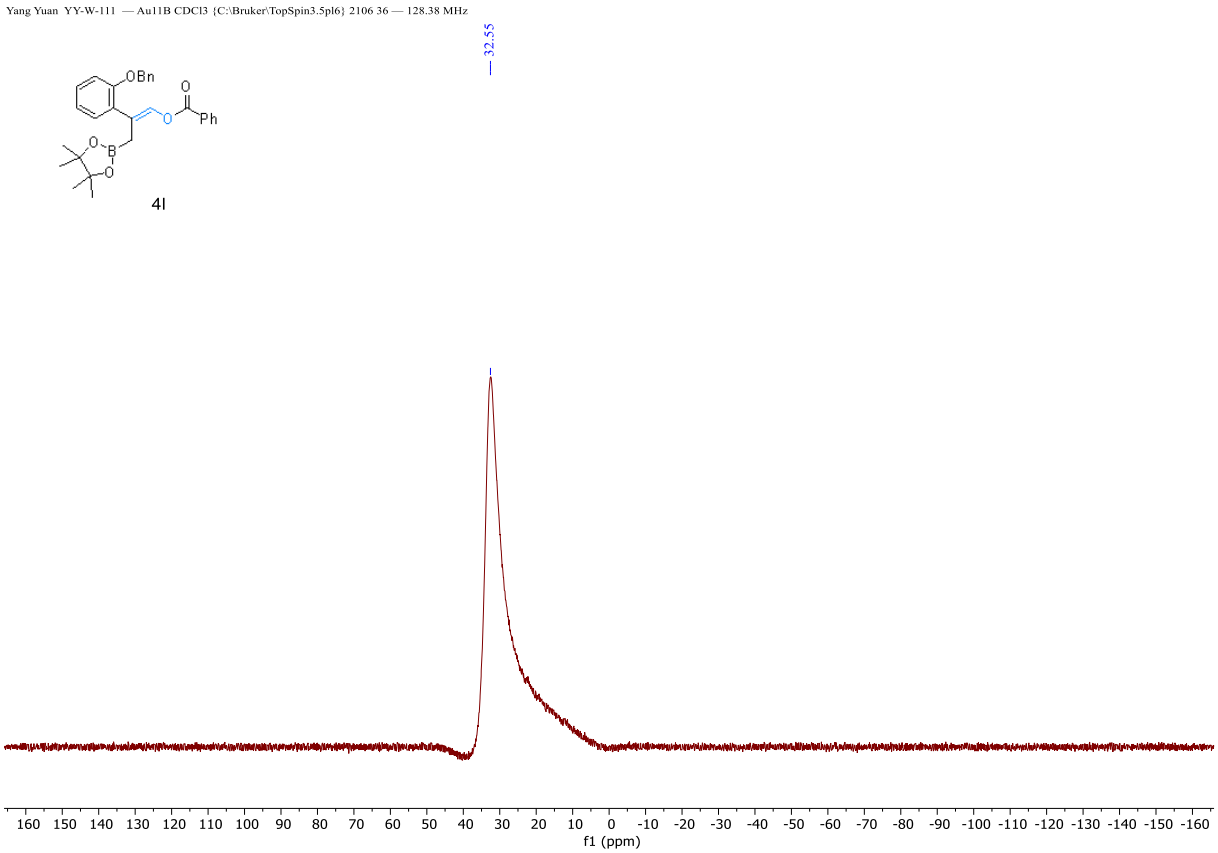

Yang Yuan YY-W-97-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 7 — 400.13 MHz
7.43
7.35
7.35
7.34
7.34
7.33
7.33
7.32
7.32
7.01
7.01
6.99
6.99
6.98
6.97
6.97
2.51
2.49
2.47
2.45
2.14
1.24
1.22
1.20
1.16
F
O
O
B
O
4k
0.90
1.90
2.05
2.01
2.00
3.06
12.04
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Yang Yuan YY-W-97-1 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 7 — 100.63 MHz
171.41
163.33
160.89
135.57
135.53
131.78
127.83
127.75
122.05
115.24
115.02
83.48
27.56
24.59
8.97
F
O
O
B
O
4k
270
260
250
240
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

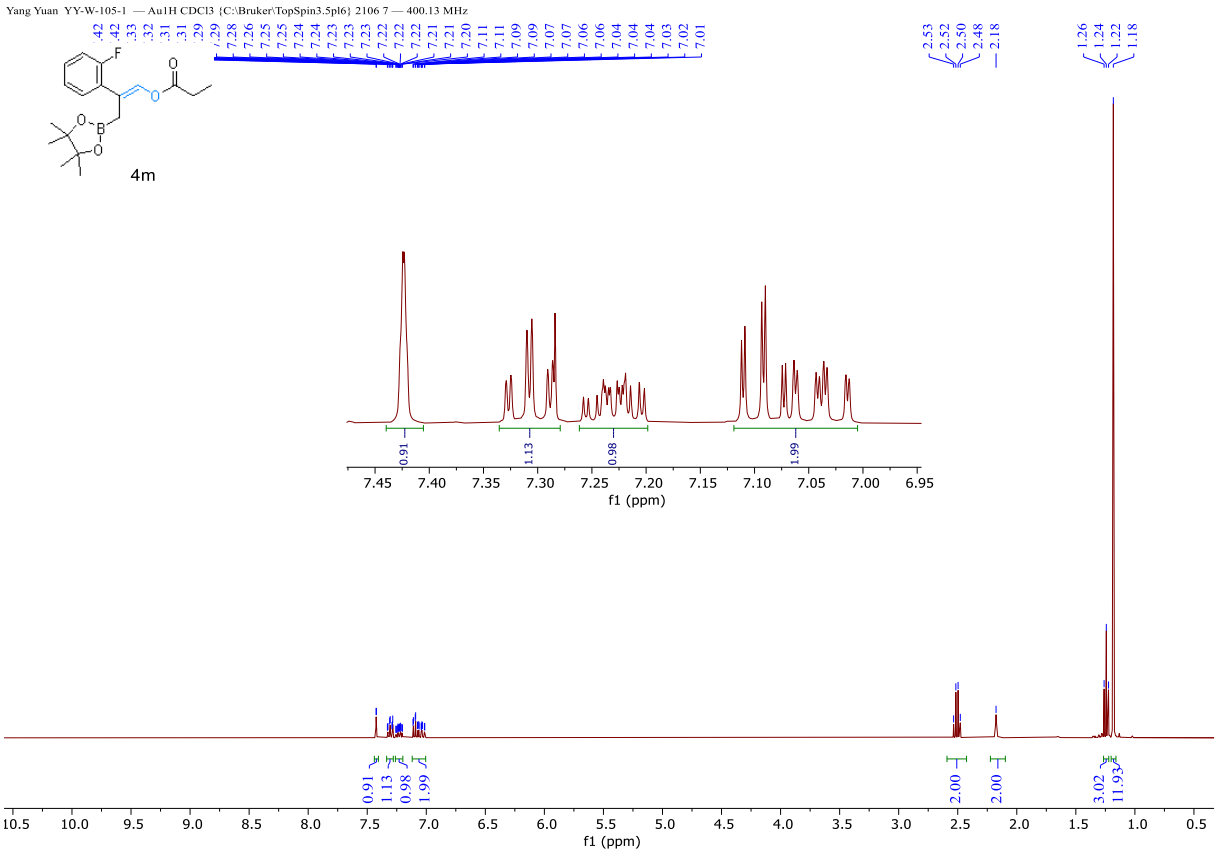

Yang Yuan YY-W-97-1 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 7 — 128.38 MHz
32.69
F
O
O
O
B
O
4k
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

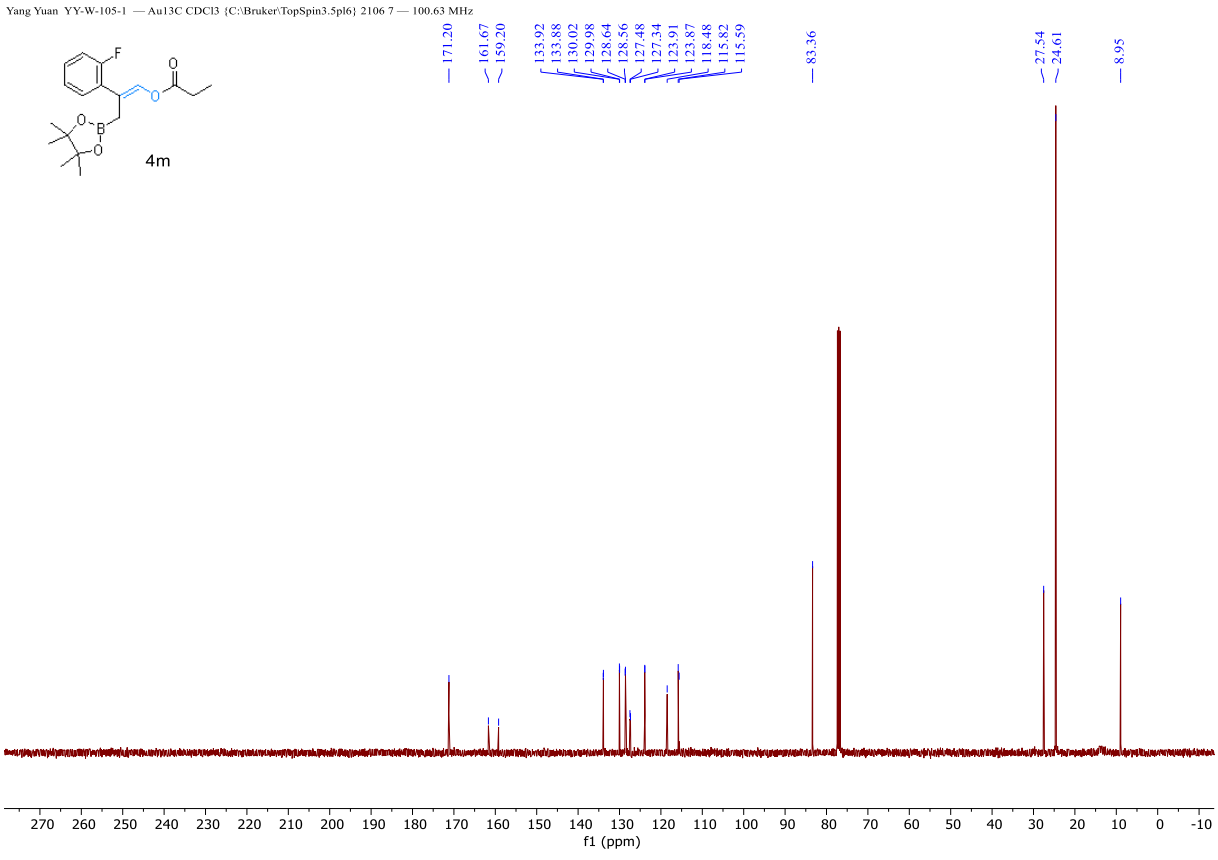

Yang Yuan YY-W-111 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 36 — 400.13 MHz
OBn
O
Ph
O
B
O
4l
f1 (ppm)

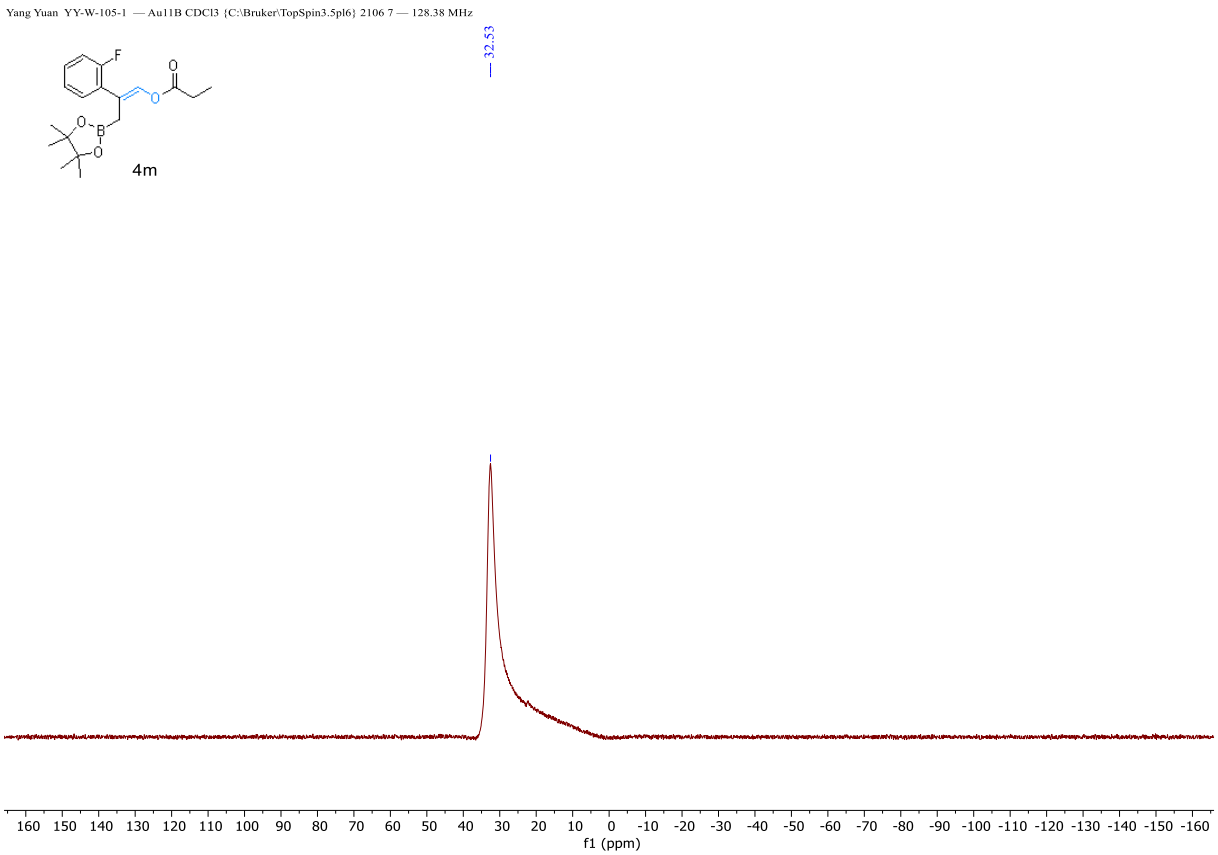

Yang Yuan YY-W-111 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 36 — 100.63 MHz
OBn
O
Ph
O
B
O
4l
f1 (ppm)

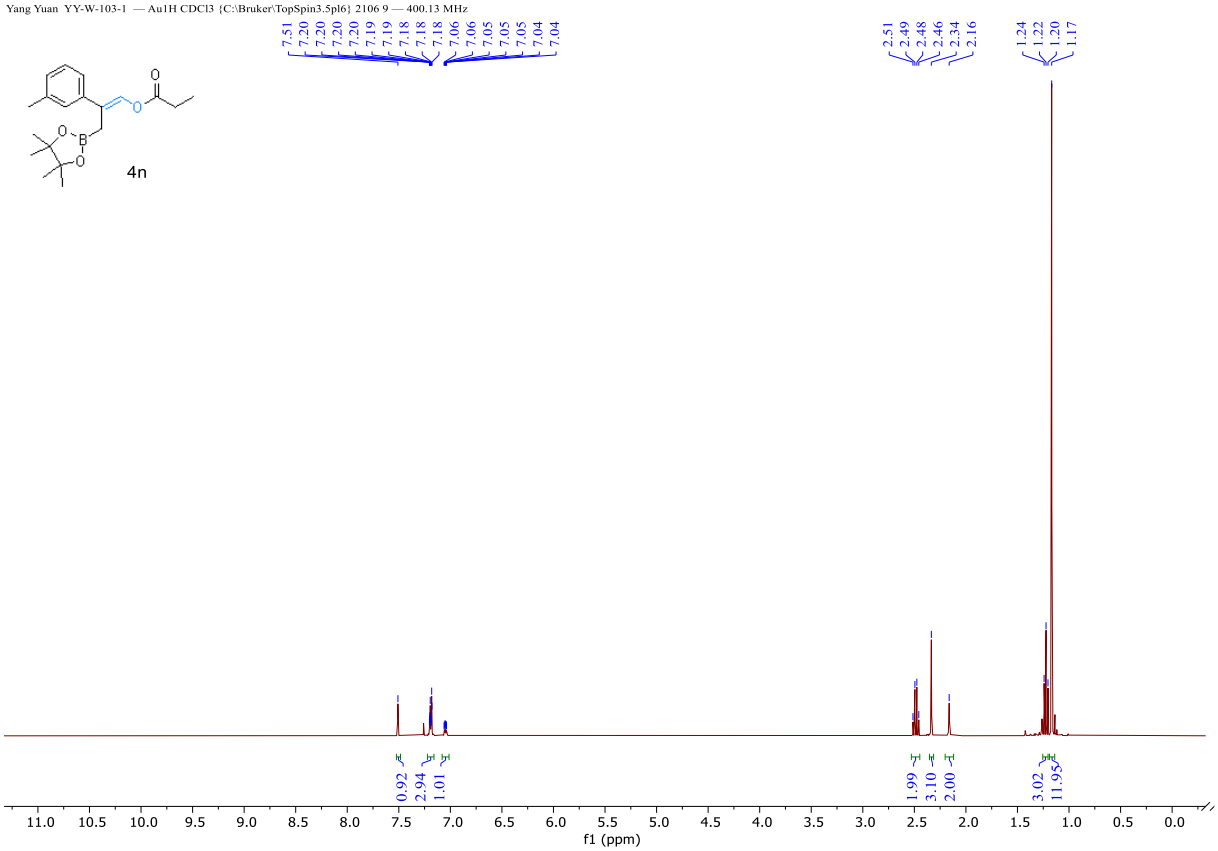

Yang Yuan YY-W-111 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 36 — 128.38 MHz
32.55
OBn
O
O
Ph
O
B
O
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

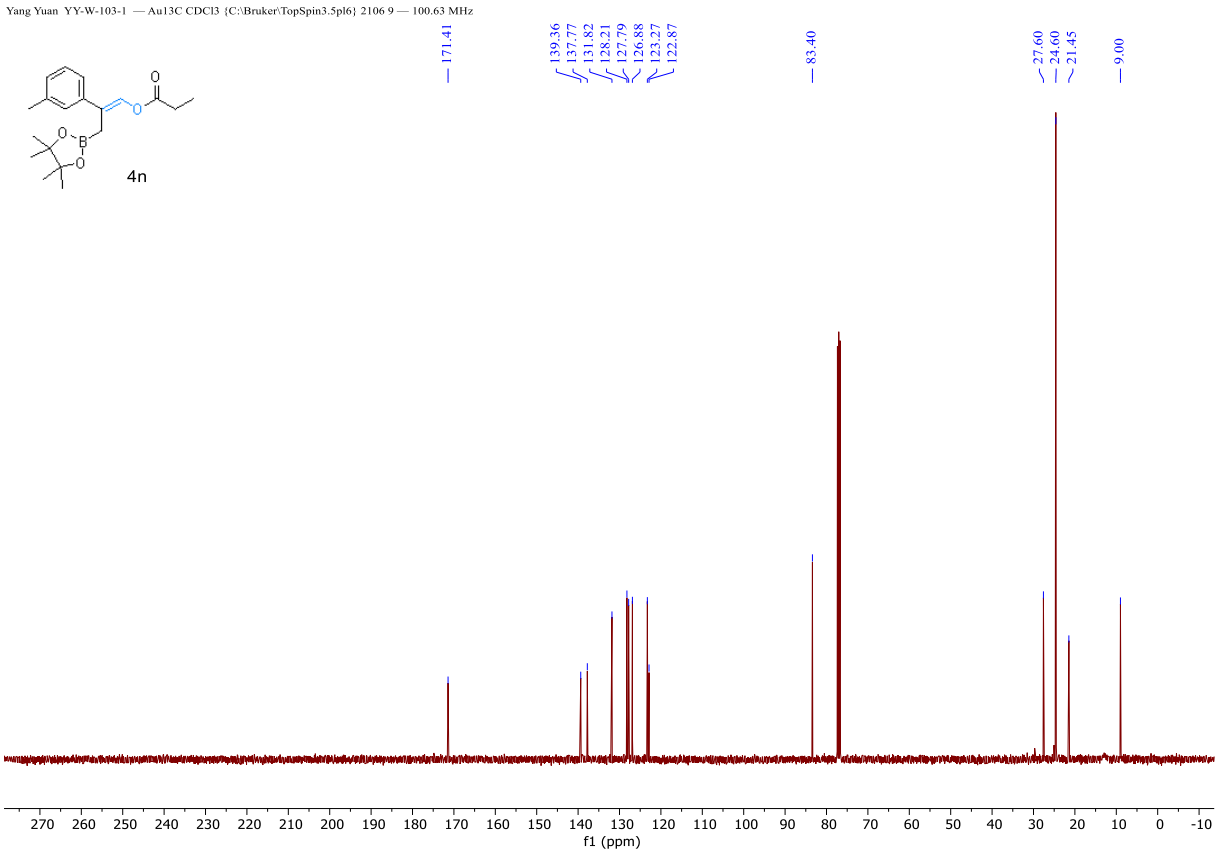
4l

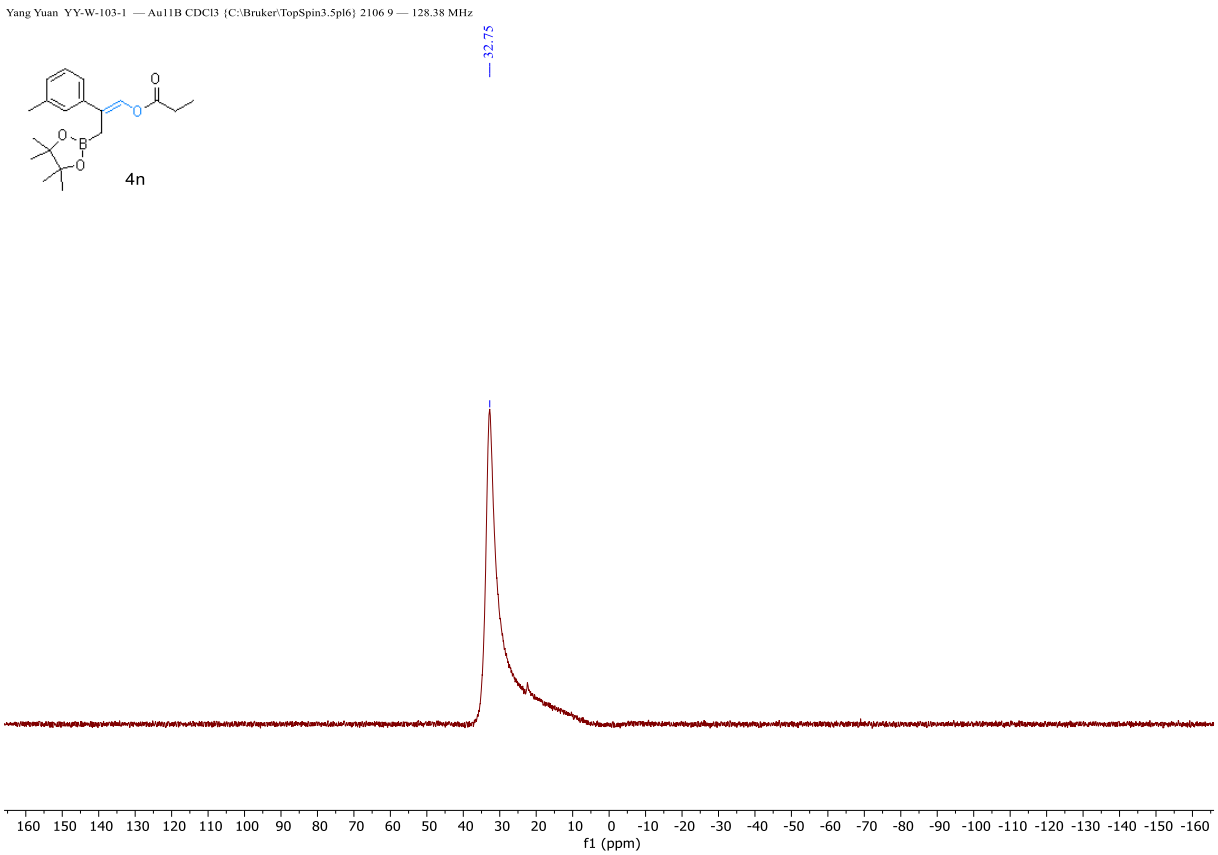

Yang Yuan YY-W-105-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 7 — 400.13 MHz
4m
f1 (ppm)

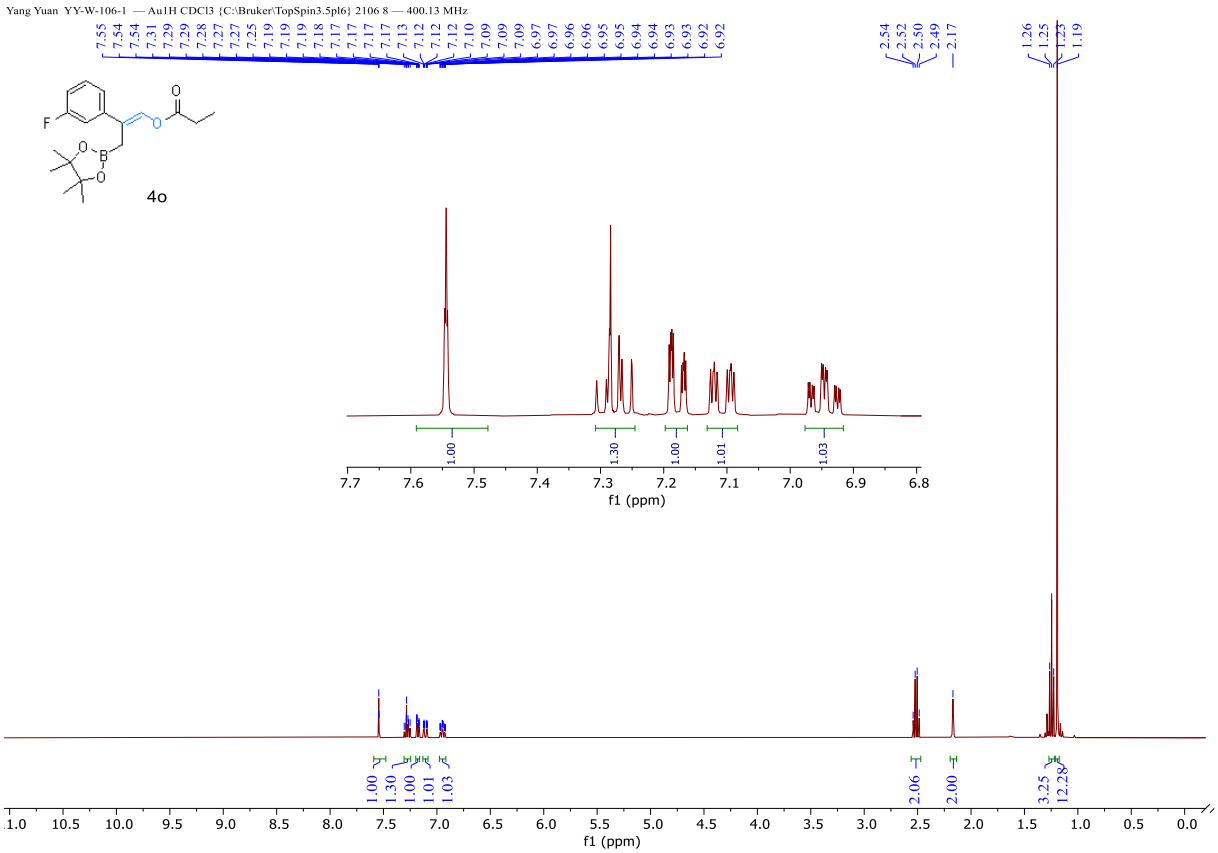

Yang Yuan YY-W-105-1 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 7 — 100.63 MHz
4m
f1 (ppm)

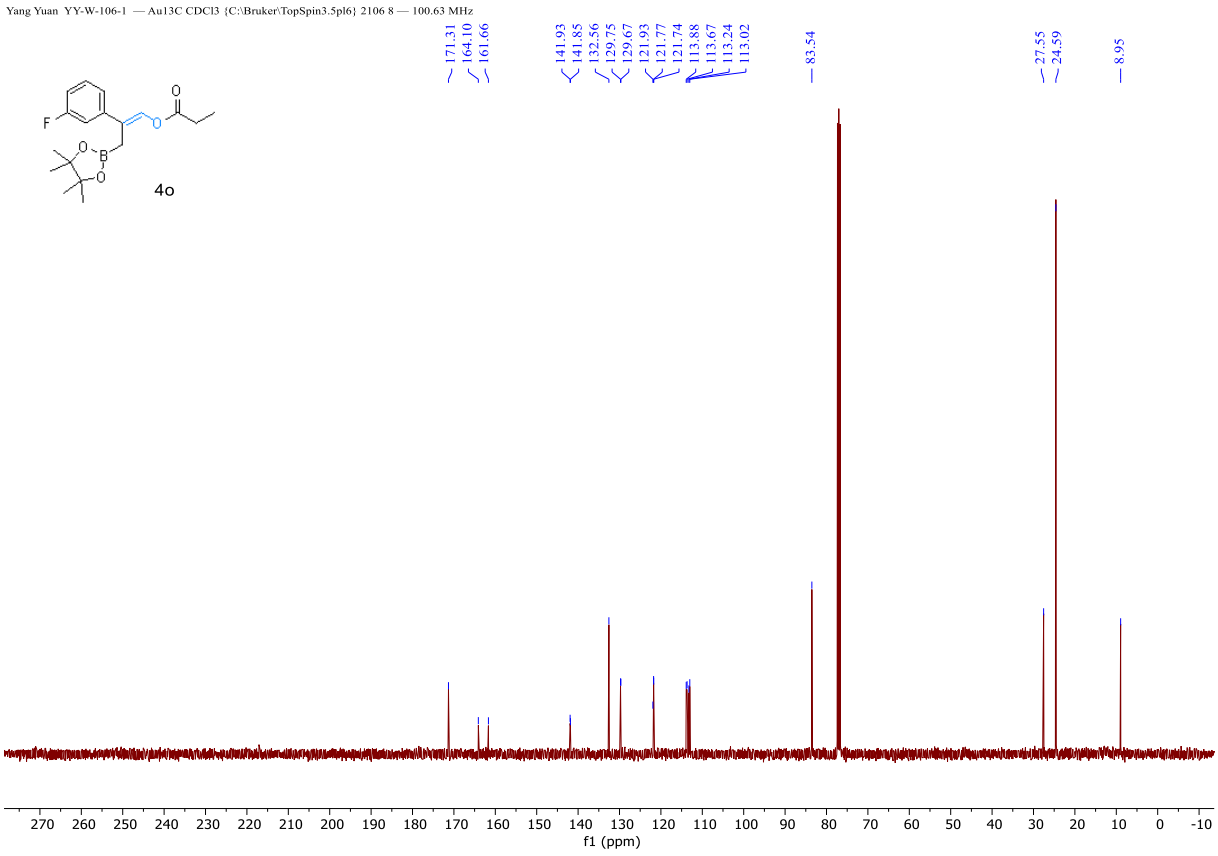

Yang Yuan YY-W-105-1 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 7 — 128.38 MHz
32.53
F
O
O
B
O
4m
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

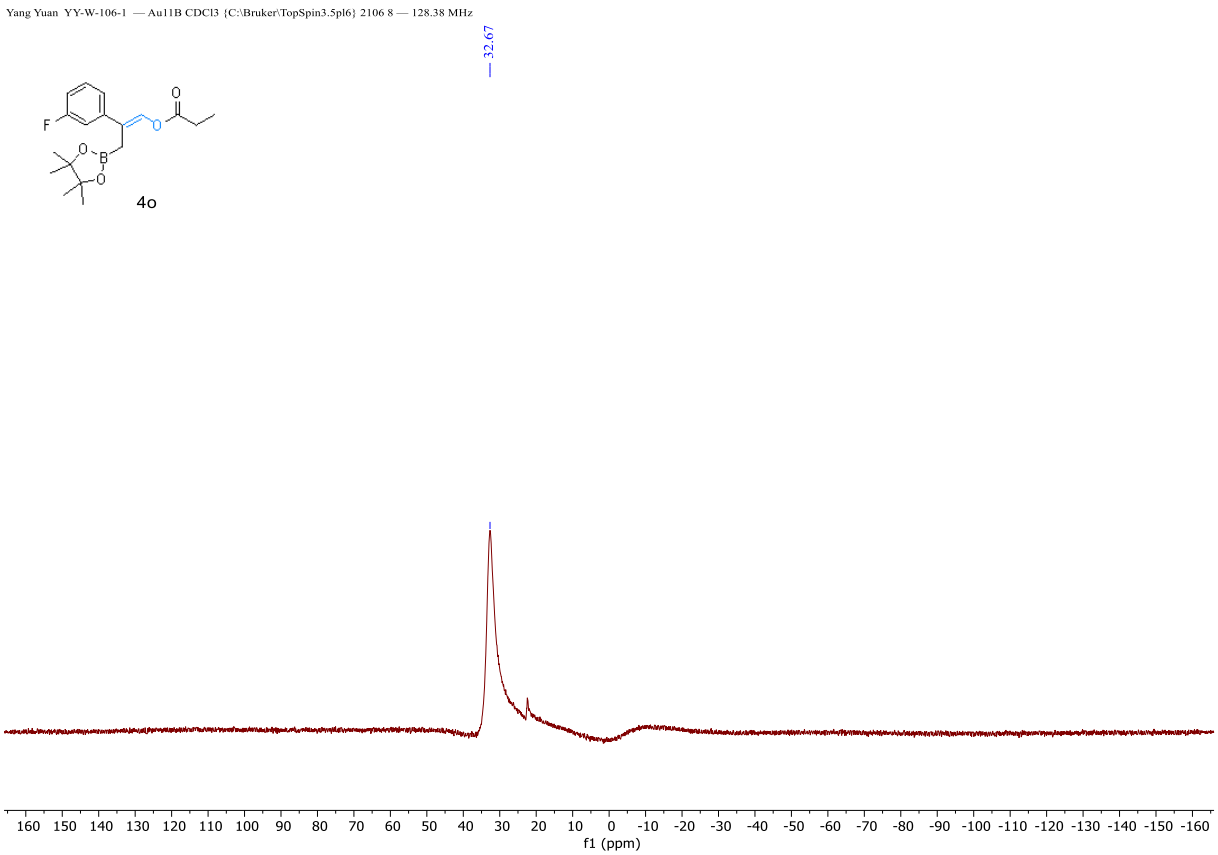

Yang Yuan YY-W-103-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 9 — 400.13 MHz
4n
f1 (ppm)

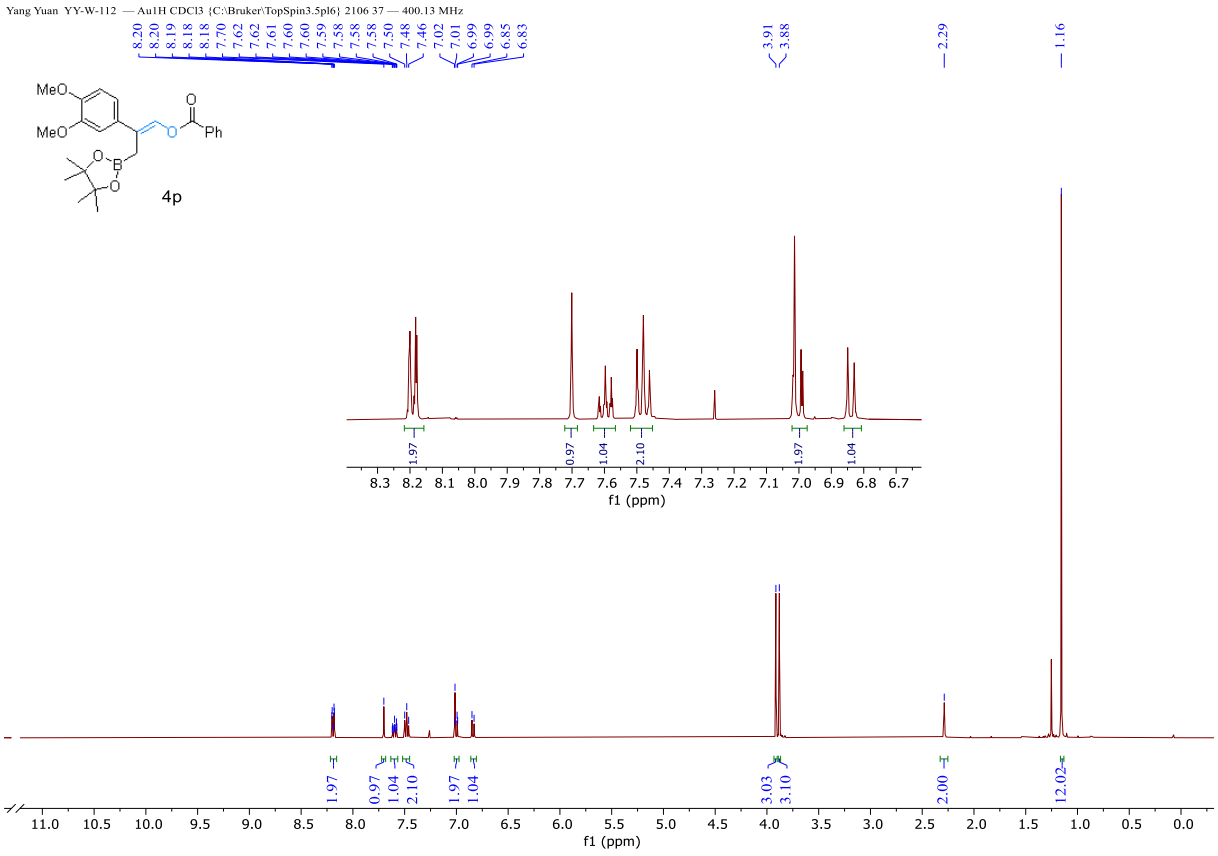

Yang Yuan YY-W-103-1 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 9 — 100.63 MHz
4n
f1 (ppm)

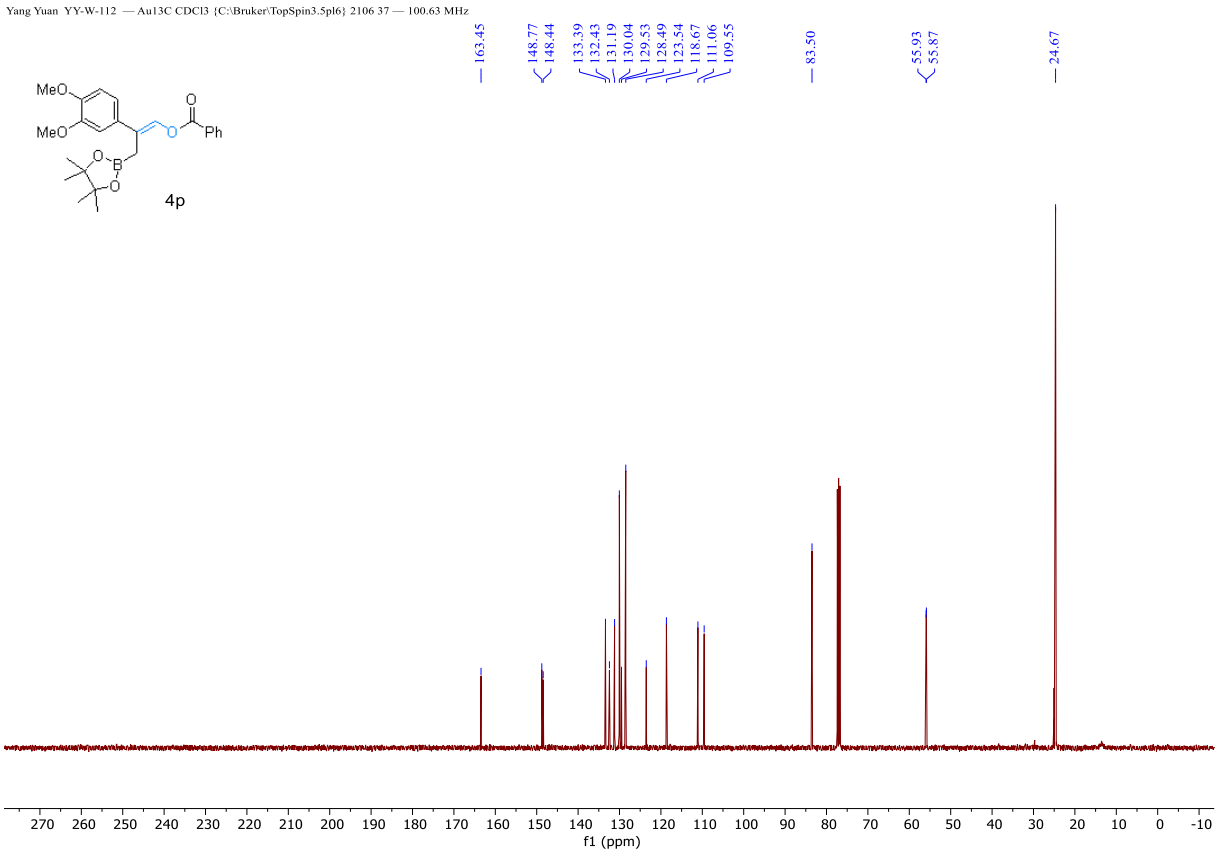

Yang Yuan YY-W-103-1 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 9 — 128.38 MHz
32.75
4n
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

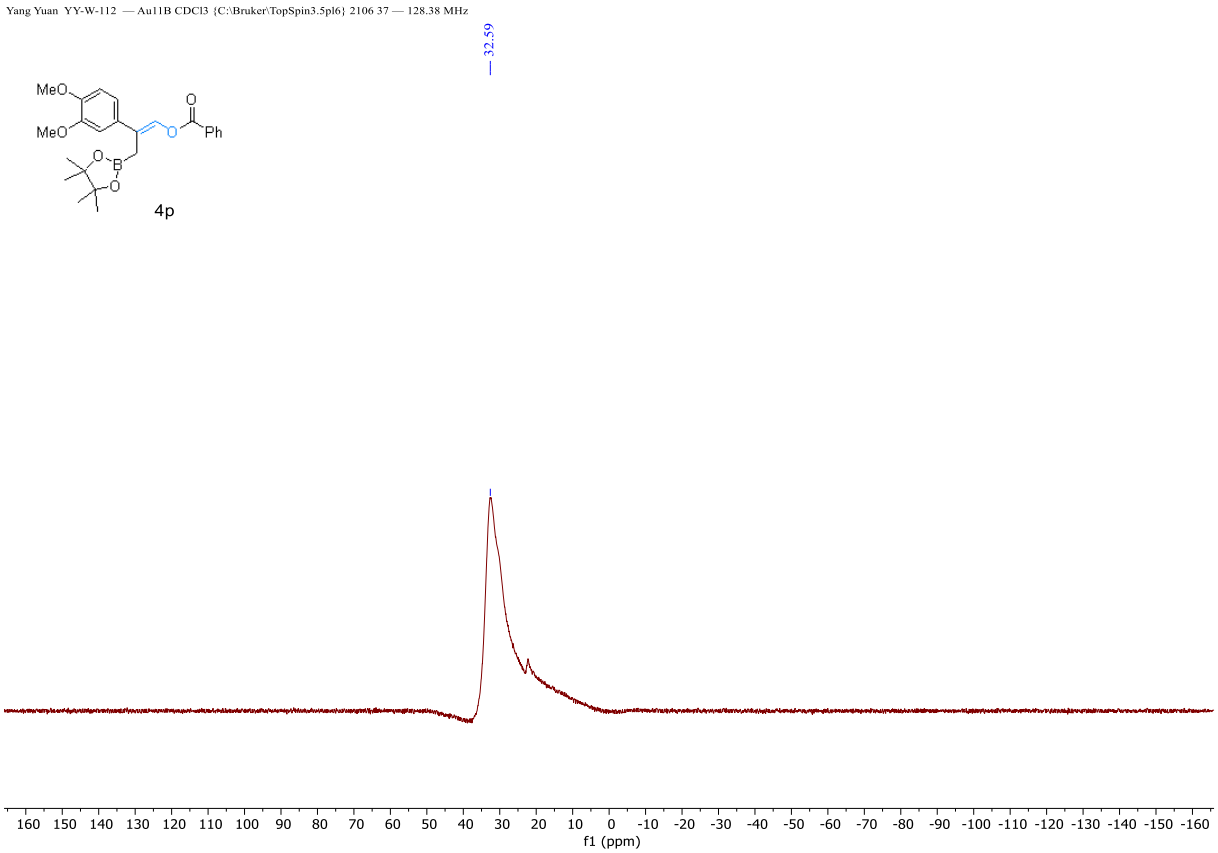

Yang Yuan YY-W-106-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 8 — 400.13 MHz
4o
f1 (ppm)

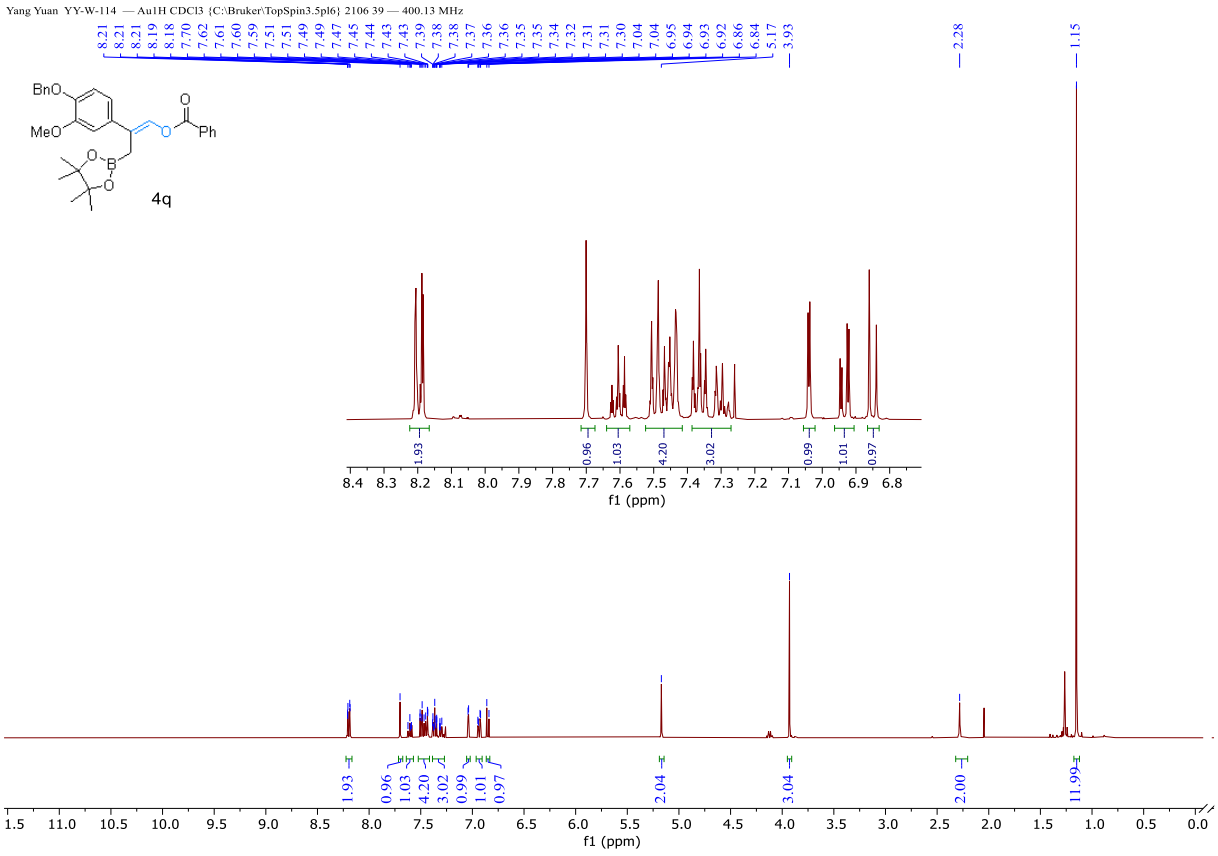

Yang Yuan YY-W-106-1 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 8 — 100.63 MHz
4o
f1 (ppm)

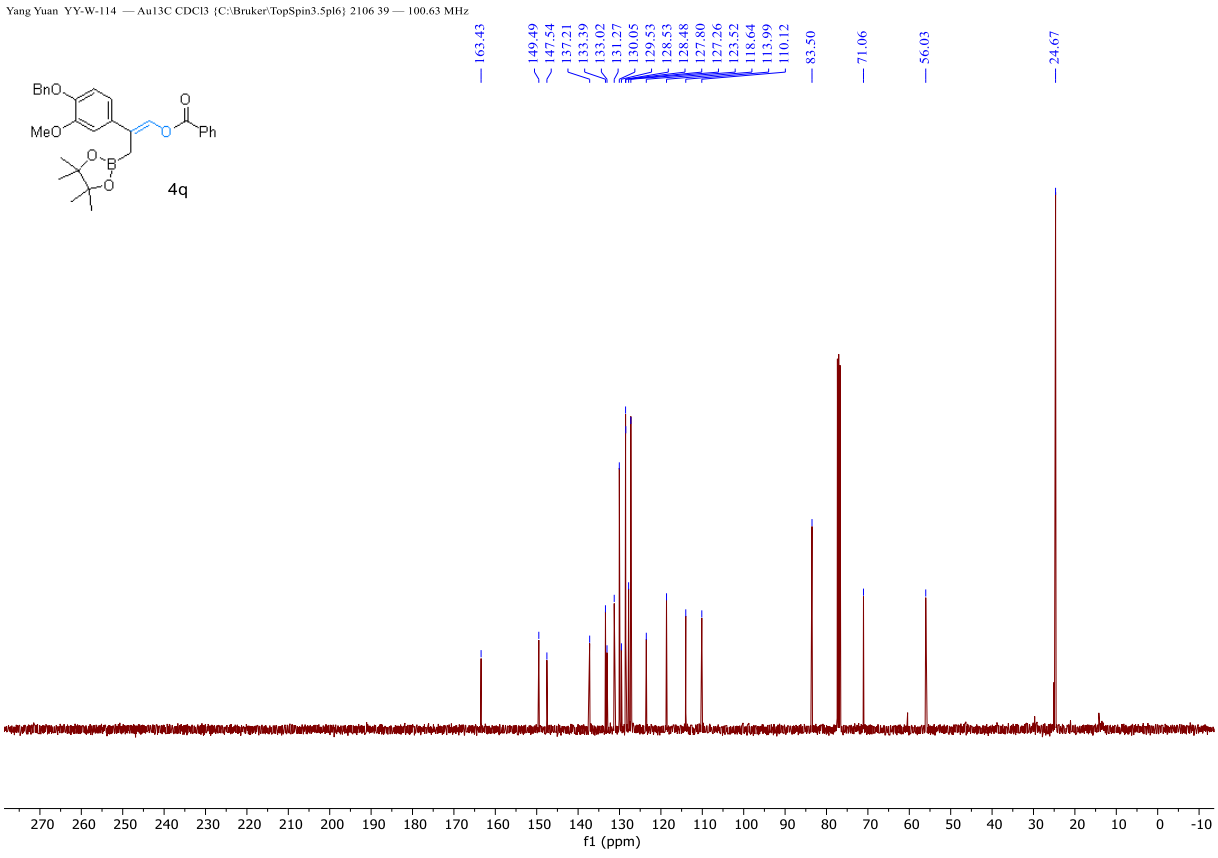

Yang Yuan YY-W-106-1 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 8 — 128.38 MHz
32.67
F
O
O
B
O
4o
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

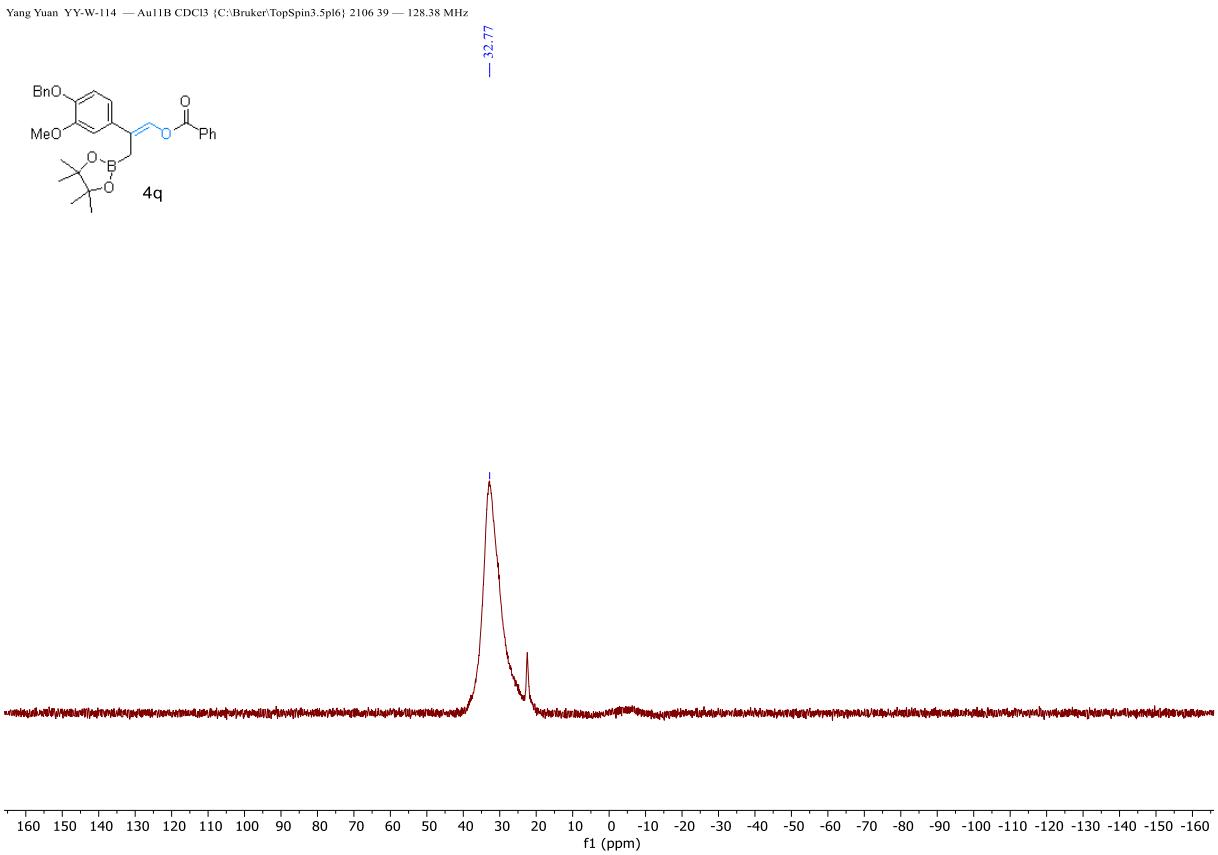

Yang Yuan YY-W-112 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 37 — 400.13 MHz
8.20 8.20 8.19 8.18 8.18 7.70 7.62 7.62 7.61 7.60 7.60 7.59 7.58 7.58 7.58 7.50 7.48 7.46 7.02 7.01 6.99 6.99 6.85 6.83
3.91 3.88
2.29
1.16
MeO
MeO
O
O
Ph
O
B
O
4p
1.97 0.97 1.04 2.10 1.97 1.04
f1 (ppm)
1.97 0.97 1.04 2.10 3.03 3.10 2.00 12.02
f1 (ppm)

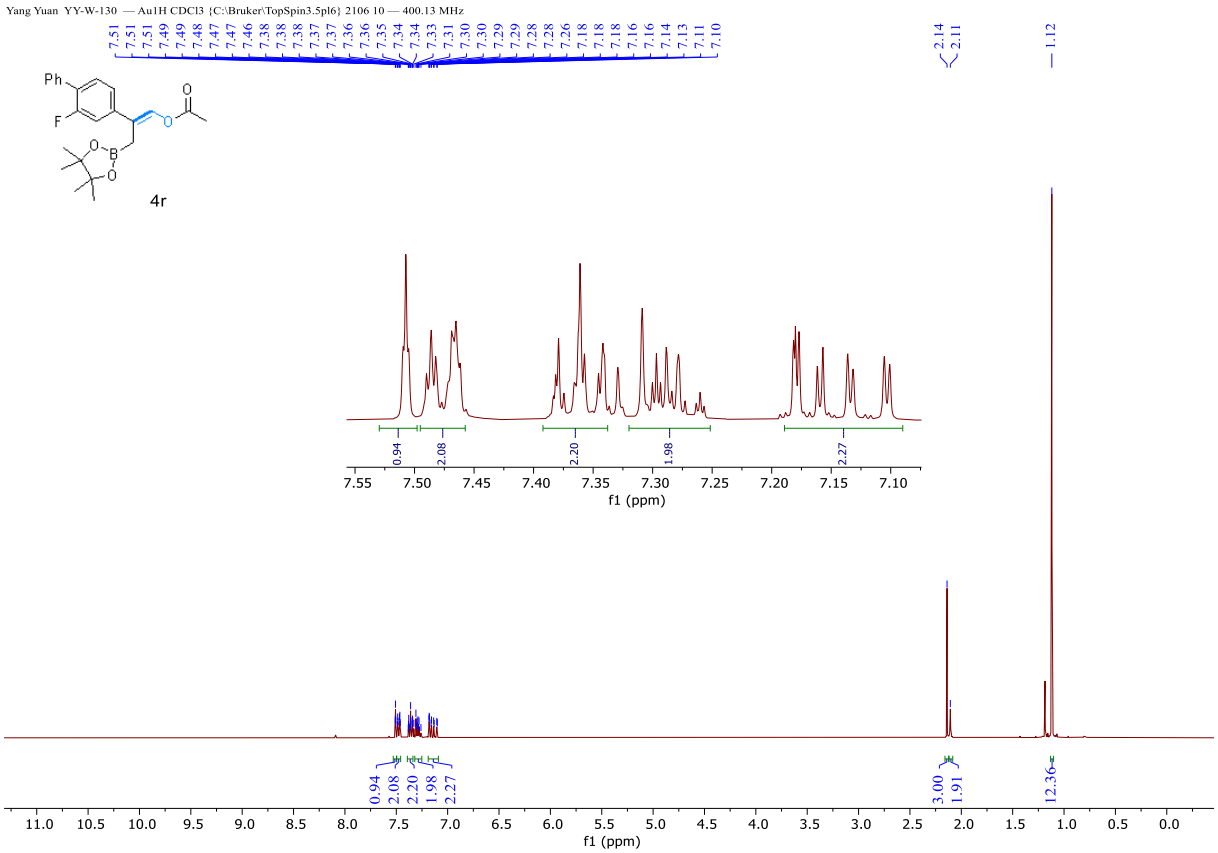

Yang Yuan YY-W-112 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 37 — 100.63 MHz
163.45
148.77 148.44
133.39 132.43 131.19 130.04 129.53 128.49 123.54 118.67 111.06 109.55
83.50
55.93 55.87
24.67
MeO
MeO
O
O
Ph
O
B
O
4p
f1 (ppm)

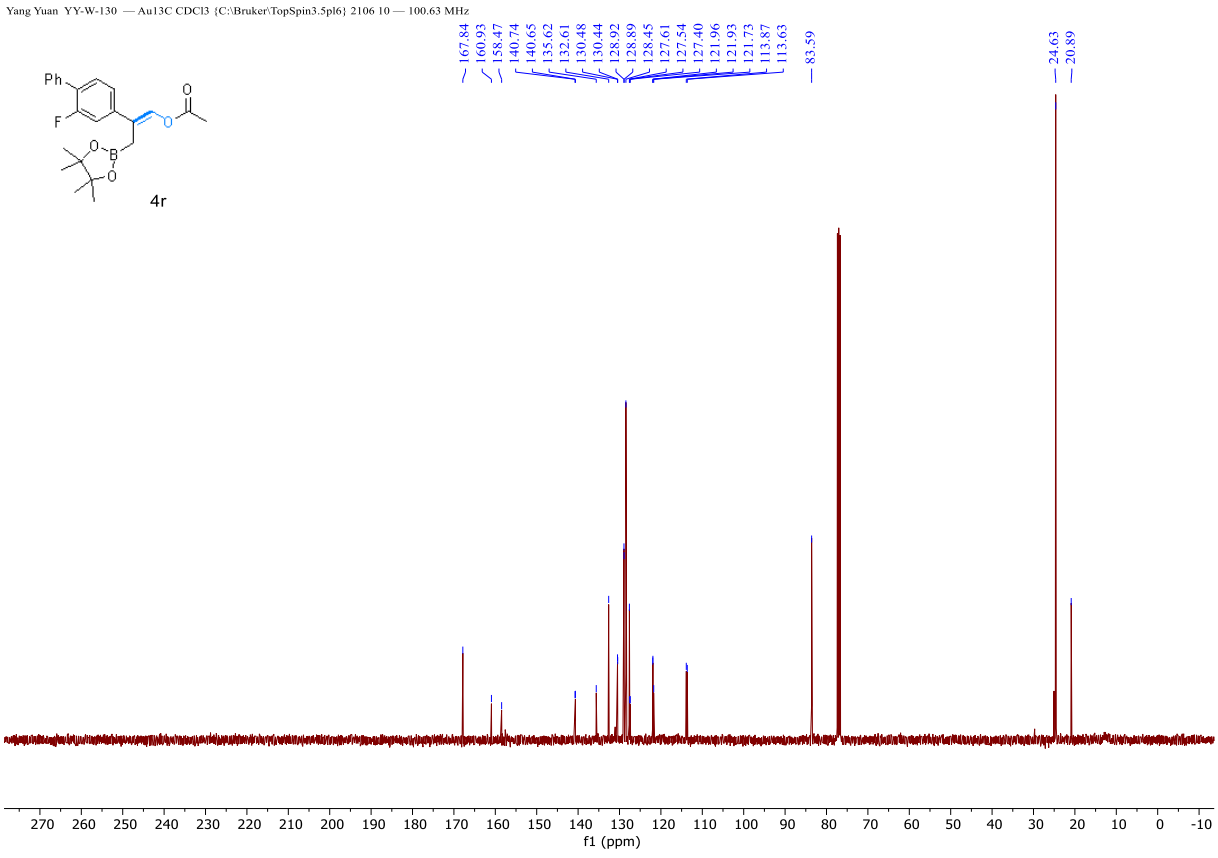

Yang Yuan YY-W-112 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 37 — 128.38 MHz
32.59
MeO
MeO
O
O
Ph
O
B
O
4p
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

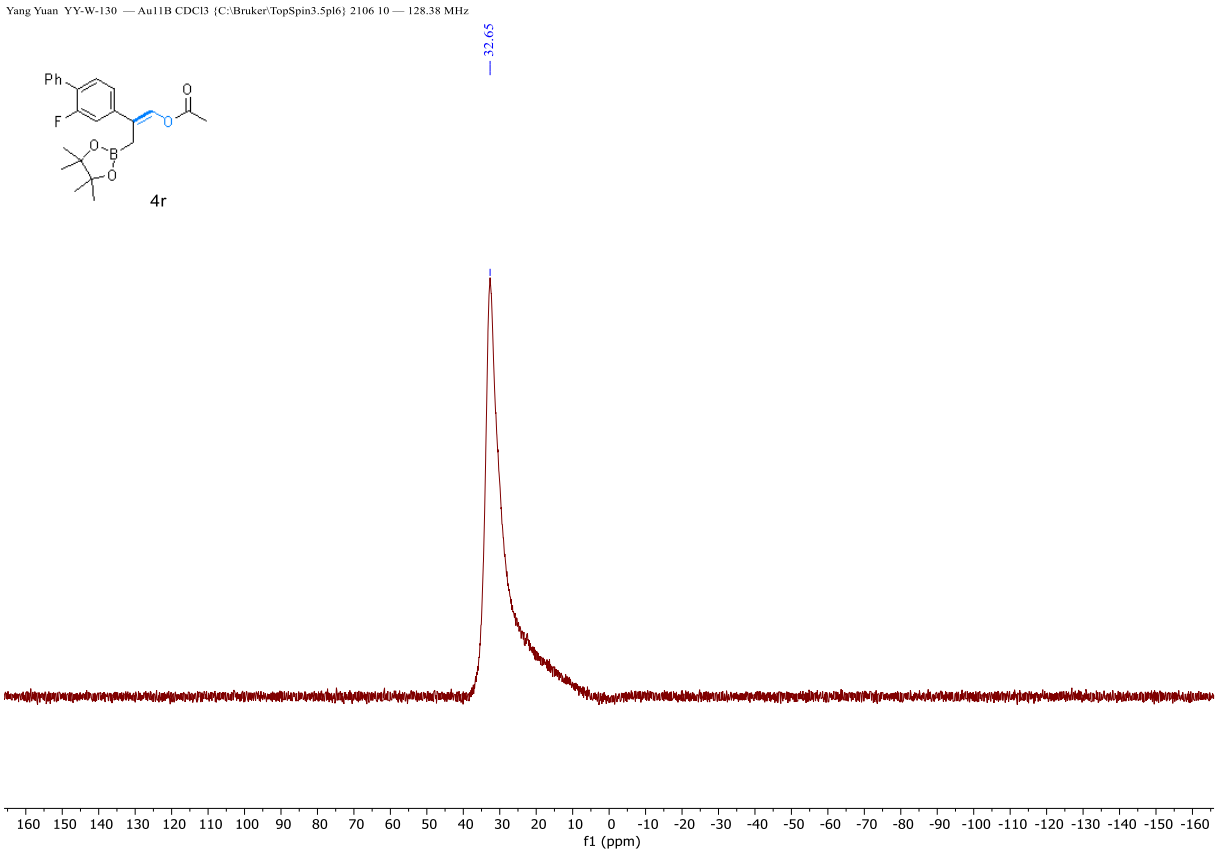

Yang Yuan YY-W-114 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 39 — 400.13 MHz
BnO
MeO
O
Ph
O
B
O
4q
f1 (ppm)

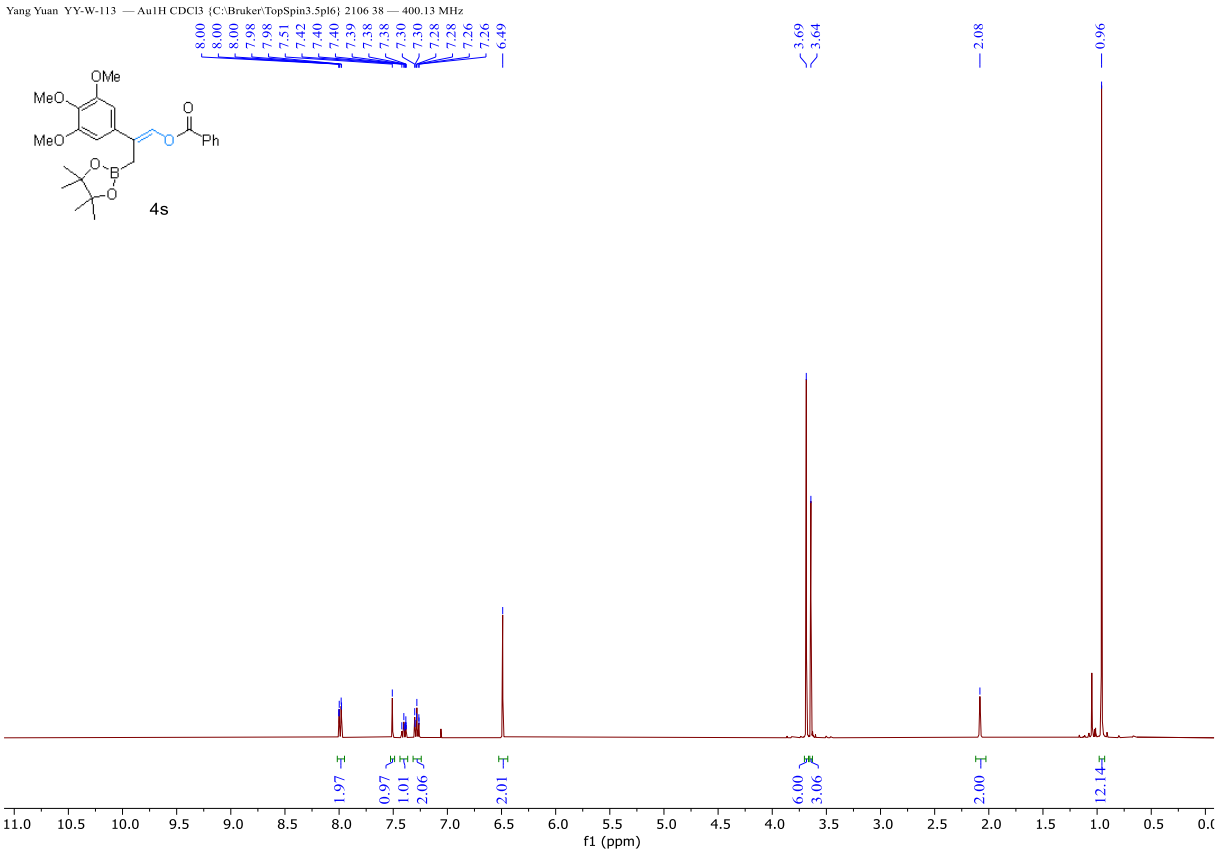

Yang Yuan YY-W-114 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 39 — 100.63 MHz
BnO
MeO
O
Ph
O
B
O
4q
f1 (ppm)

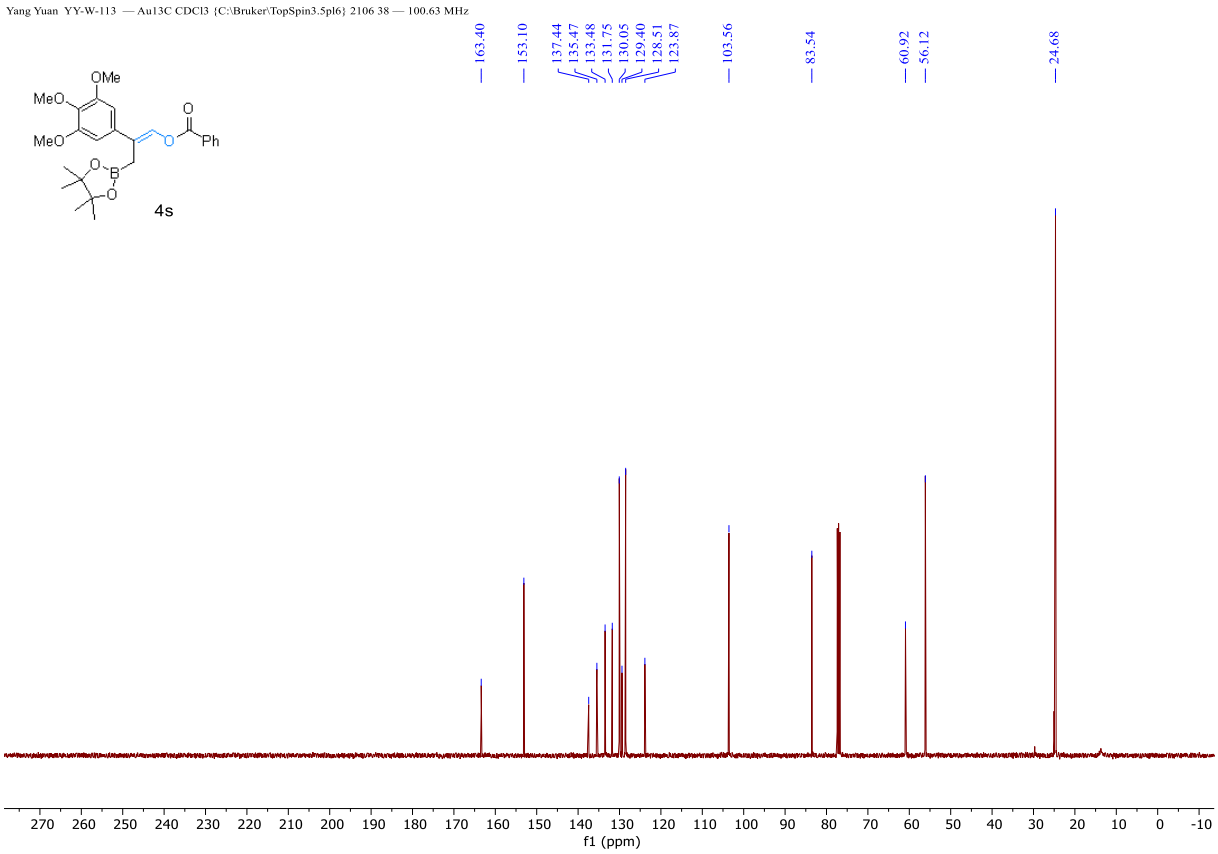

Yang Yuan YY-W-114 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 39 — 128.38 MHz
32.77
BnO
MeO
O
O
Ph
O
B
O
4q
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

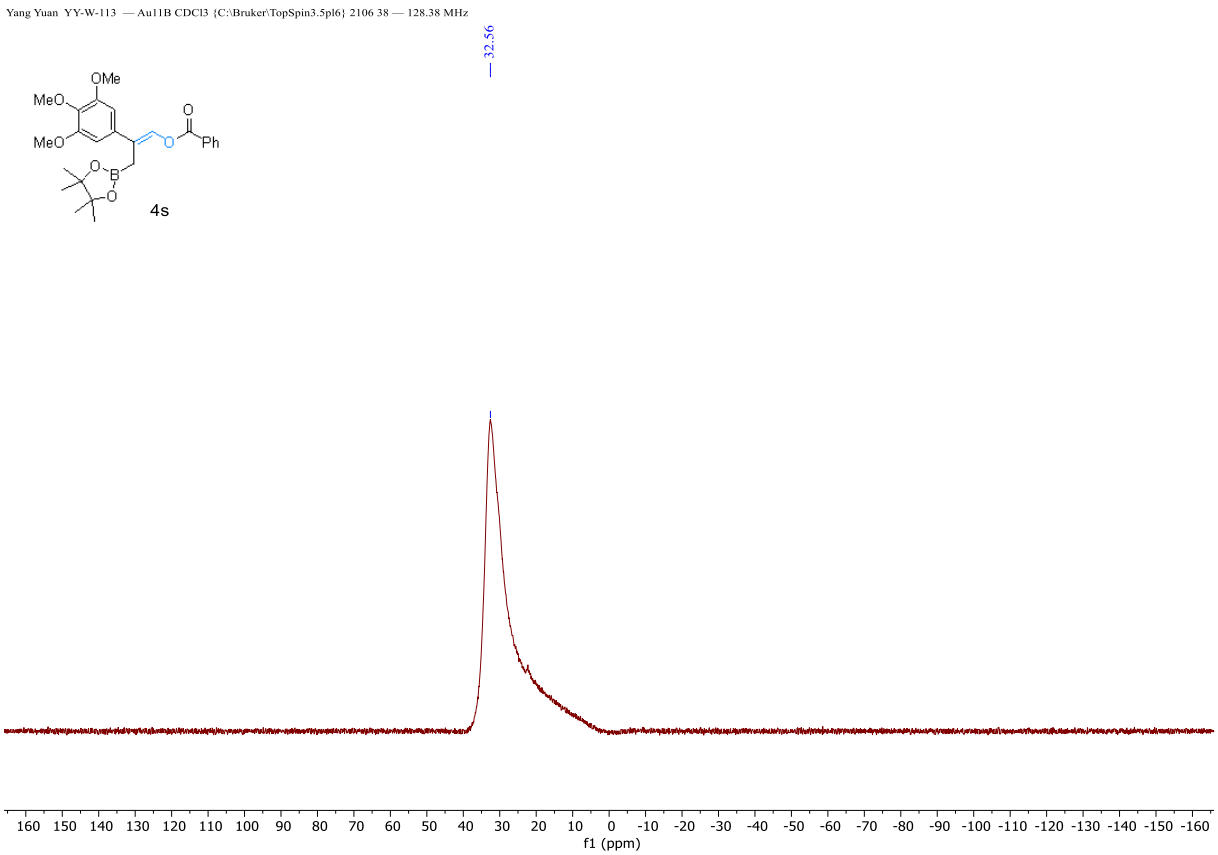

Yang Yuan YY-W-130 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 10 — 400.13 MHz
4r
f1 (ppm)

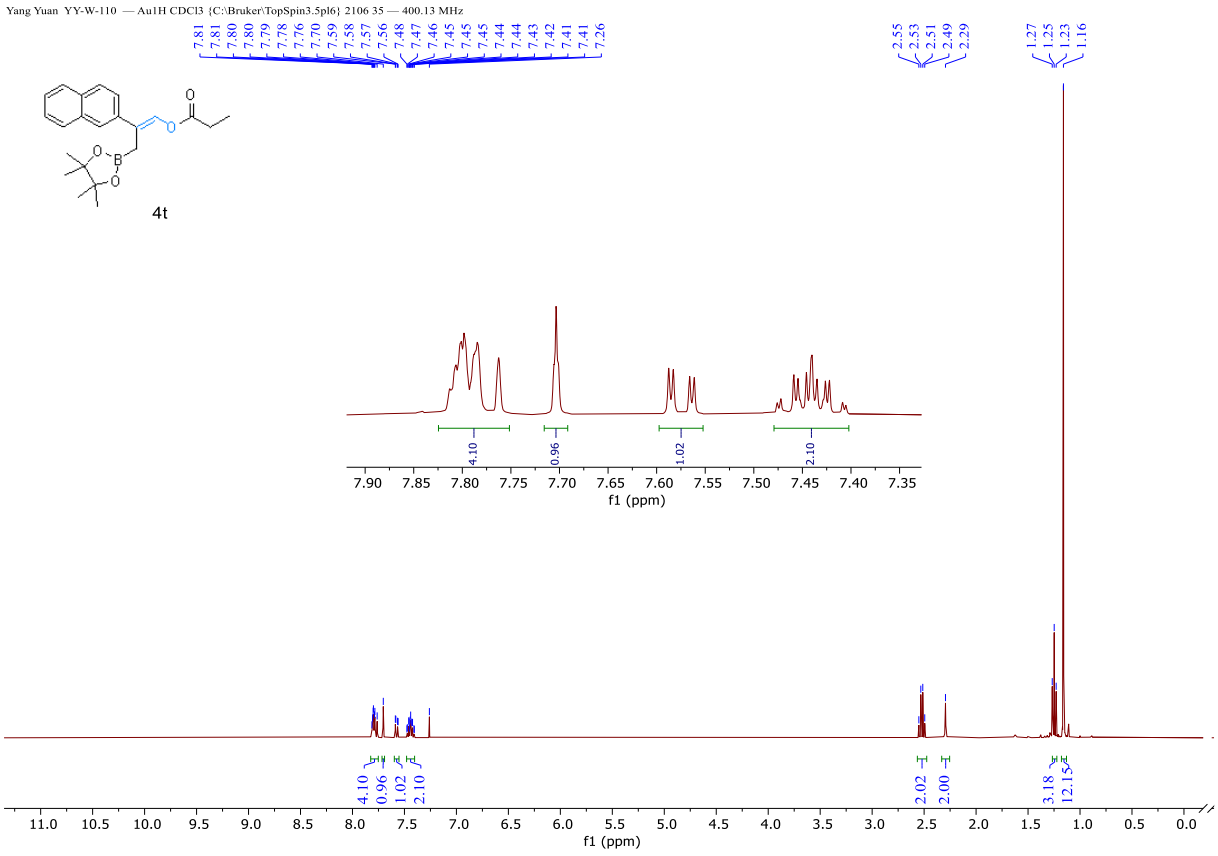

Yang Yuan YY-W-130 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 10 — 100.63 MHz
4r
f1 (ppm)

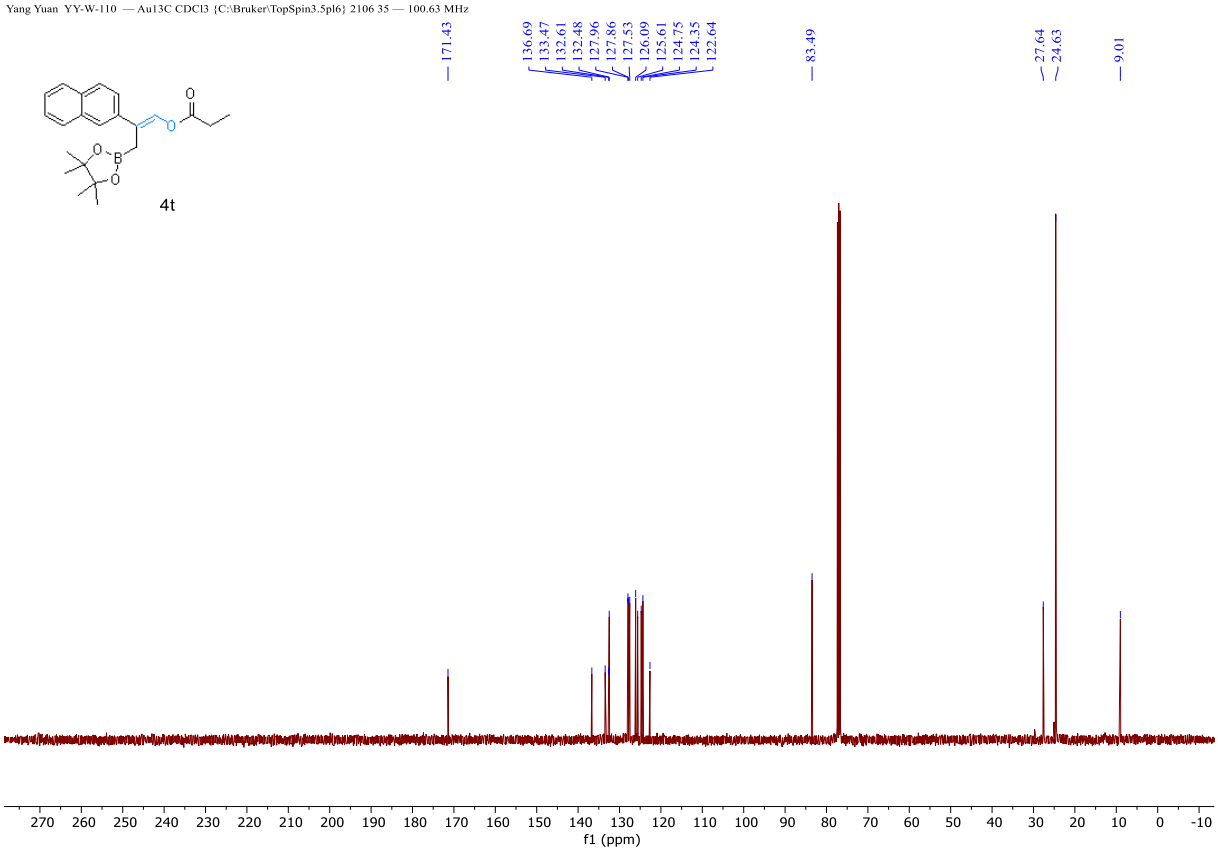

Yang Yuan YY-W-130 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 10 — 128.38 MHz
32.65
Ph
F
O
O
B
O
4r
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

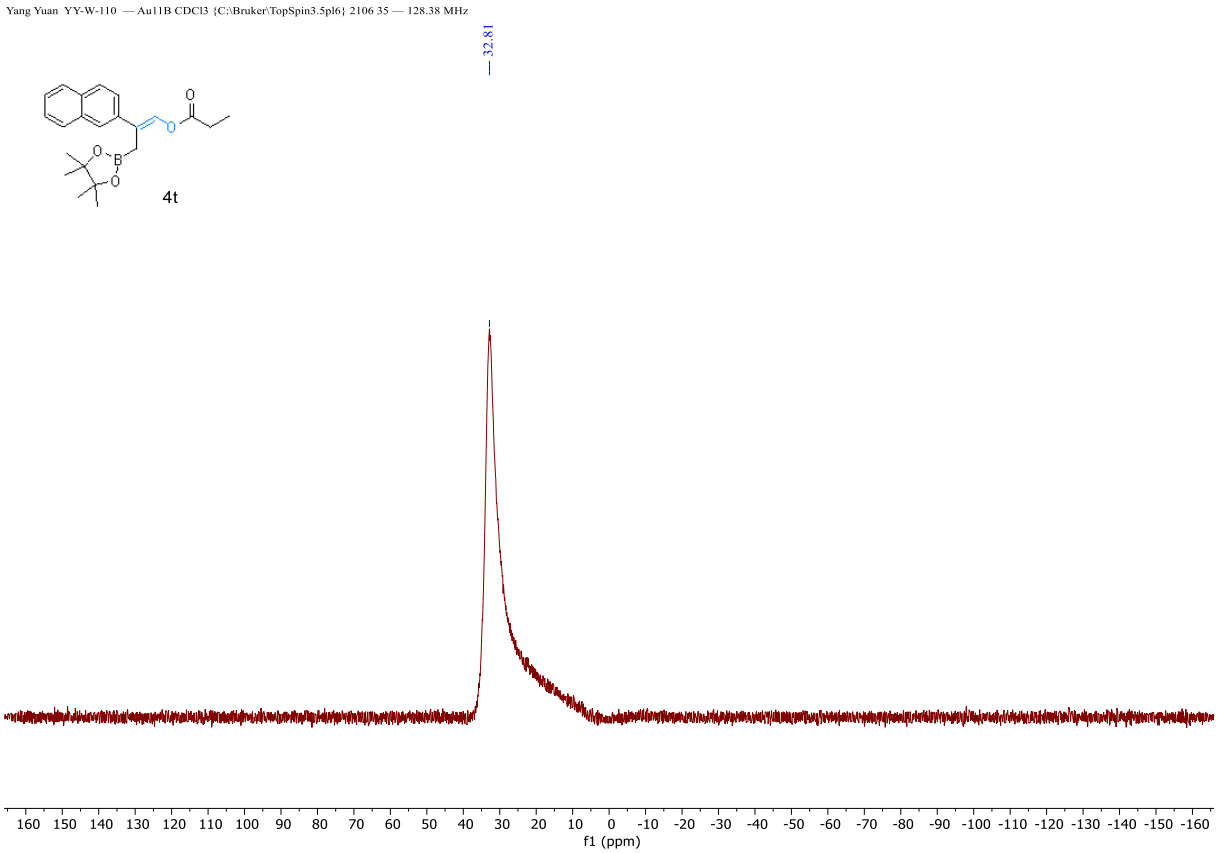

Yang Yuan YY-W-113 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 38 — 400.13 MHz
8.00
8.00
8.00
7.98
7.98
7.98
7.51
7.42
7.40
7.40
7.39
7.38
7.38
7.30
7.30
7.28
7.28
7.26
7.26
6.49
3.69
3.64
2.08
0.96
OMe
MeO
MeO
O
O
Ph
B
O
O
4s
1.97
0.97
1.01
2.06
2.01
6.00
3.06
2.00
12.14
f1 (ppm)

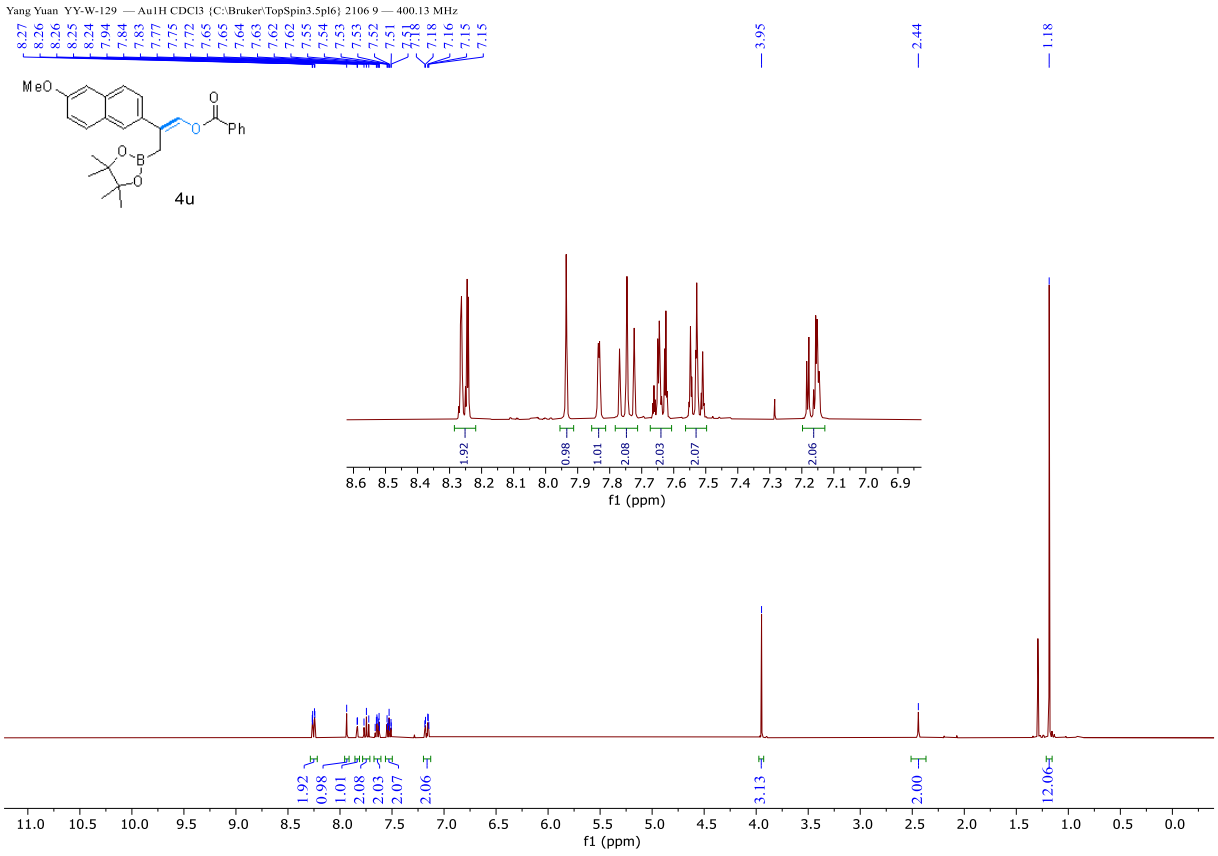

Yang Yuan YY-W-113 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 38 — 100.63 MHz
163.40
153.10
137.44
135.47
133.48
131.75
130.05
129.40
128.51
123.87
103.56
83.54
60.92
56.12
24.68
OMe
MeO
MeO
O
O
Ph
B
O
O
4s
f1 (ppm)

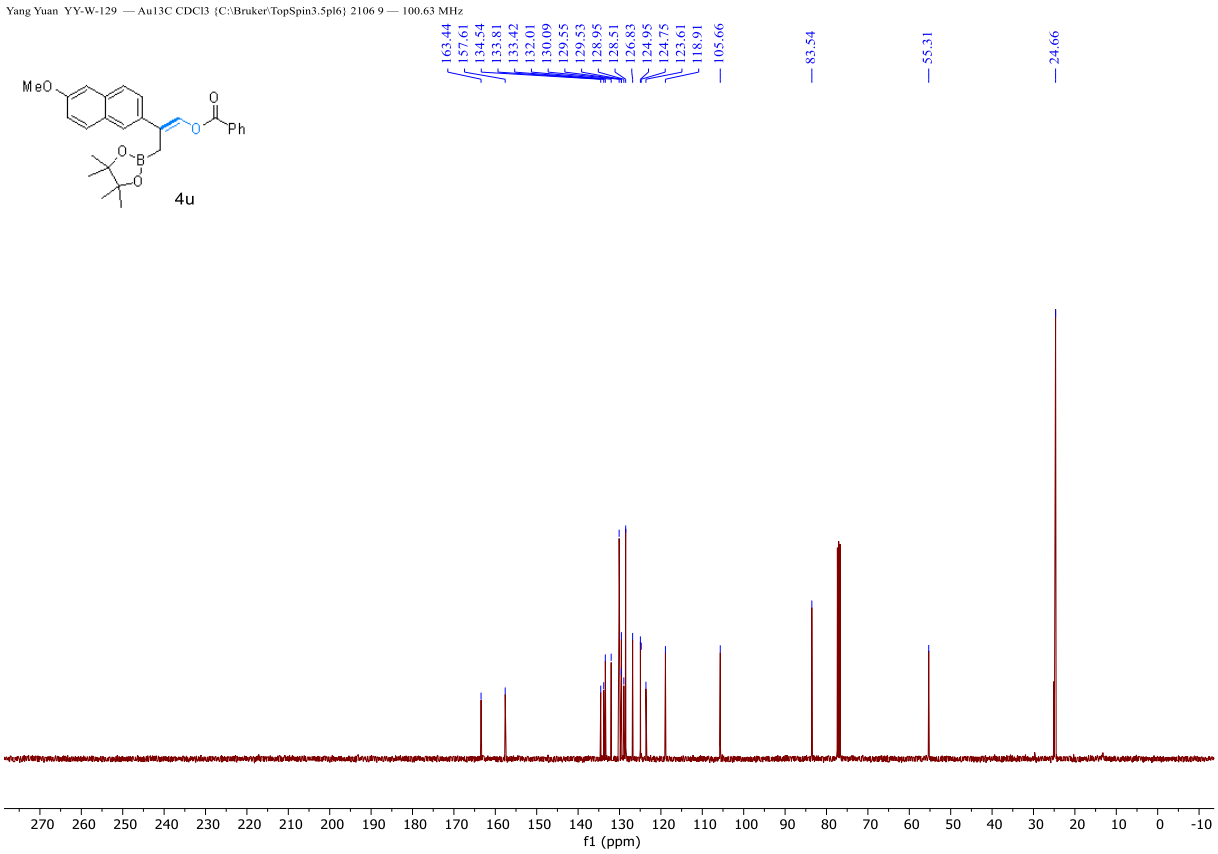

Yang Yuan YY-W-113 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 38 — 128.38 MHz
32.56
OMe
MeO
MeO
O
O
Ph
O
B
O
4s
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

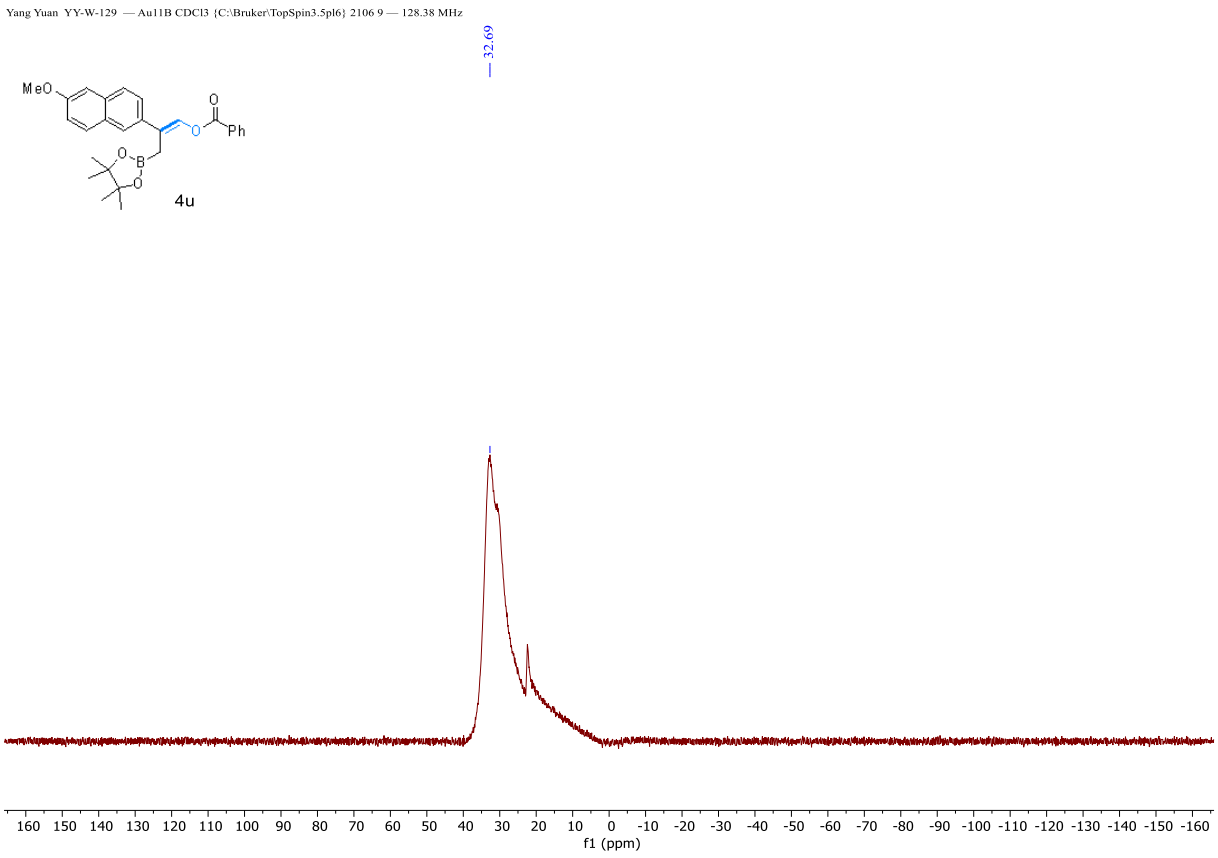

Yang Yuan YY-W-110 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 35 — 400.13 MHz
7.81 7.81 7.80 7.80 7.79 7.78 7.76 7.70 7.59 7.58 7.57 7.56 7.48 7.47 7.46 7.45 7.45 7.45 7.44 7.43 7.42 7.41 7.41 7.26
2.55 2.53 2.51 2.49 2.29
1.27 1.25 1.23 1.16
4t
4.10 0.96 1.02 2.10
2.02 2.00 3.18 12.15
f1 (ppm)


Yang Yuan YY-W-110 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 35 — 100.63 MHz
171.43
136.69 133.47 132.61 132.48 127.96 127.86 127.53 126.09 125.61 124.75 124.35 122.64
83.49
27.64 24.63
9.01
4t
f1 (ppm)

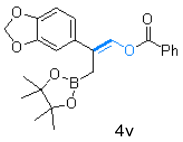

Yang Yuan YY-W-110 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 35 — 128.38 MHz
32.81
4t
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)


Yang Yuan YY-W-129 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 9 — 400.13 MHz
8.27
8.26
8.26
8.25
8.24
7.94
7.84
7.83
7.77
7.75
7.72
7.65
7.65
7.64
7.63
7.62
7.62
7.55
7.54
7.53
7.53
7.52
7.51
7.51
7.18
7.16
7.15
7.15
3.95
2.44
1.18
MeO
O
Ph
O
B
O
4u
1.92
0.98
1.01
2.08
2.03
2.07
2.06
8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9
f1 (ppm)
1.92
0.98
1.01
2.08
2.03
2.07
2.06
3.13
2.00
12.06
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Yang Yuan YY-W-129 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 9 — 100.63 MHz
163.44
157.61
134.54
133.81
133.42
132.01
130.09
129.55
129.53
128.95
128.51
126.83
124.95
124.75
123.61
118.91
105.66
83.54
55.31
24.66
MeO
O
Ph
O
B
O
4u
270 260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

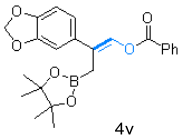

Yang Yuan YY-W-129 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 9 — 128.38 MHz
32.69
MeO
O
O
Ph
O
B
O
4u
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

Yang Yuan YY-W-126 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 7 — 400.13 MHz

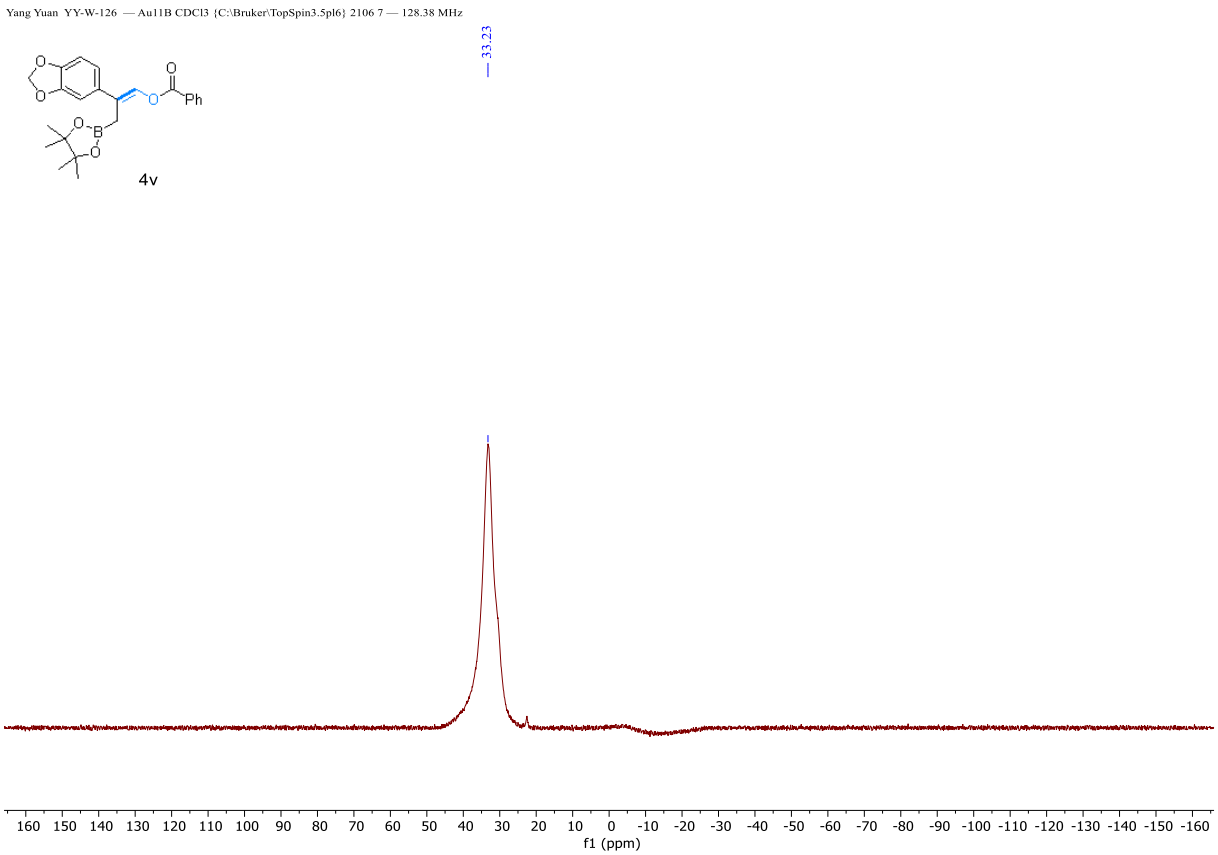


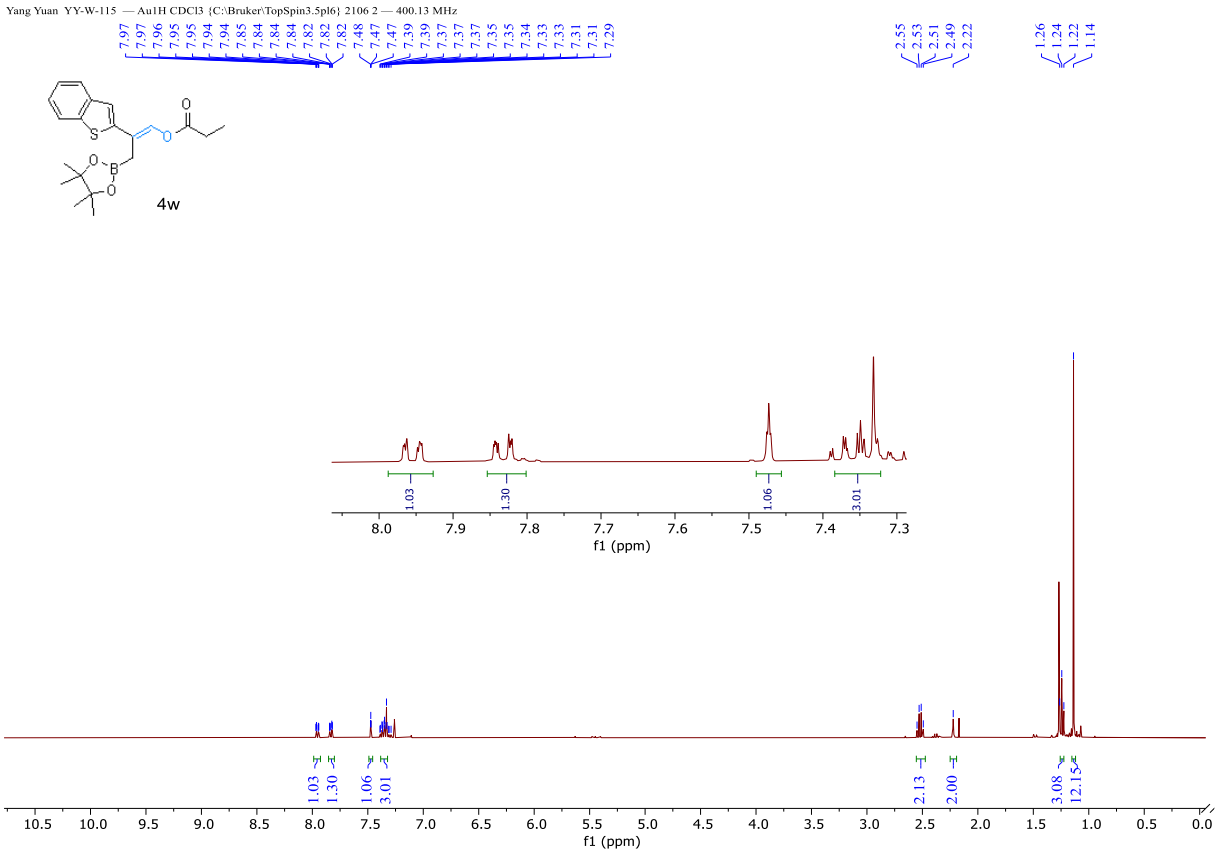
4v

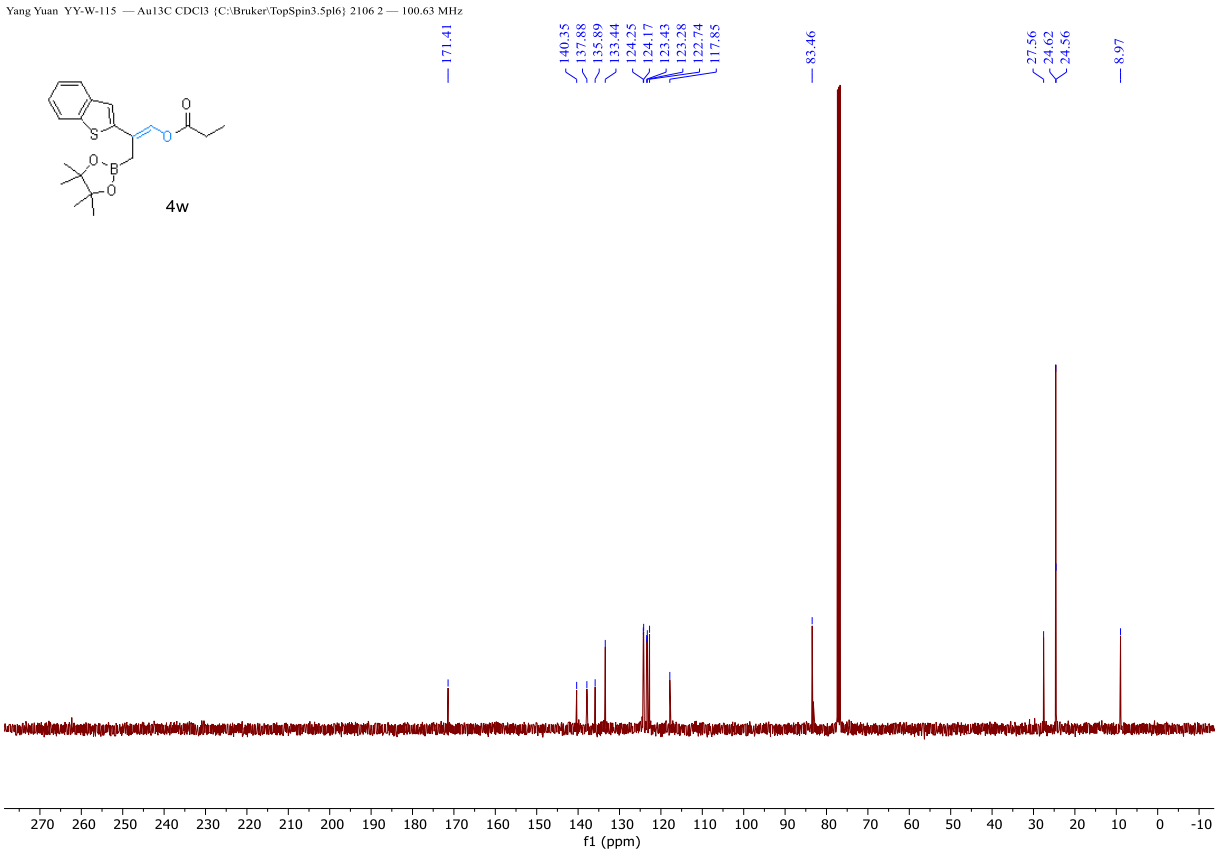
Yang Yuan YY-W-126 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 7 — 100.63 MHz

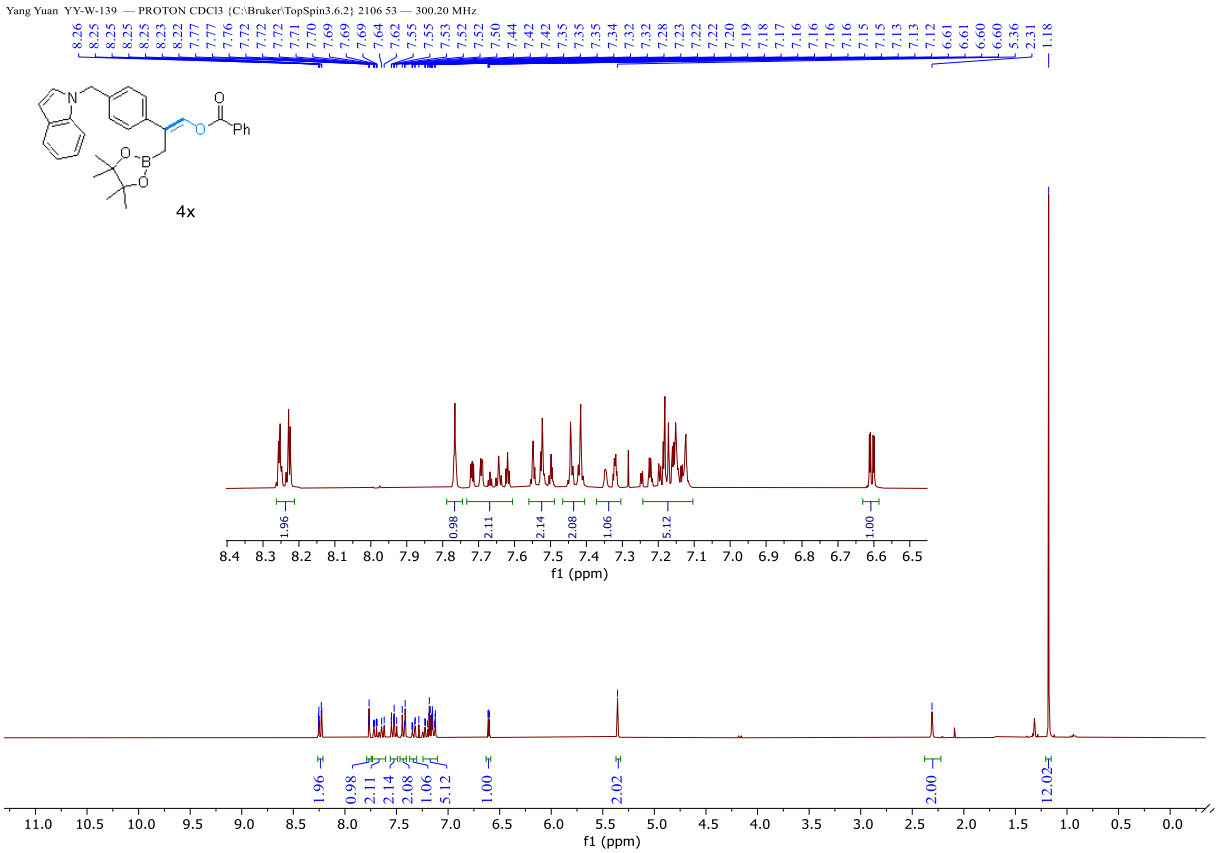


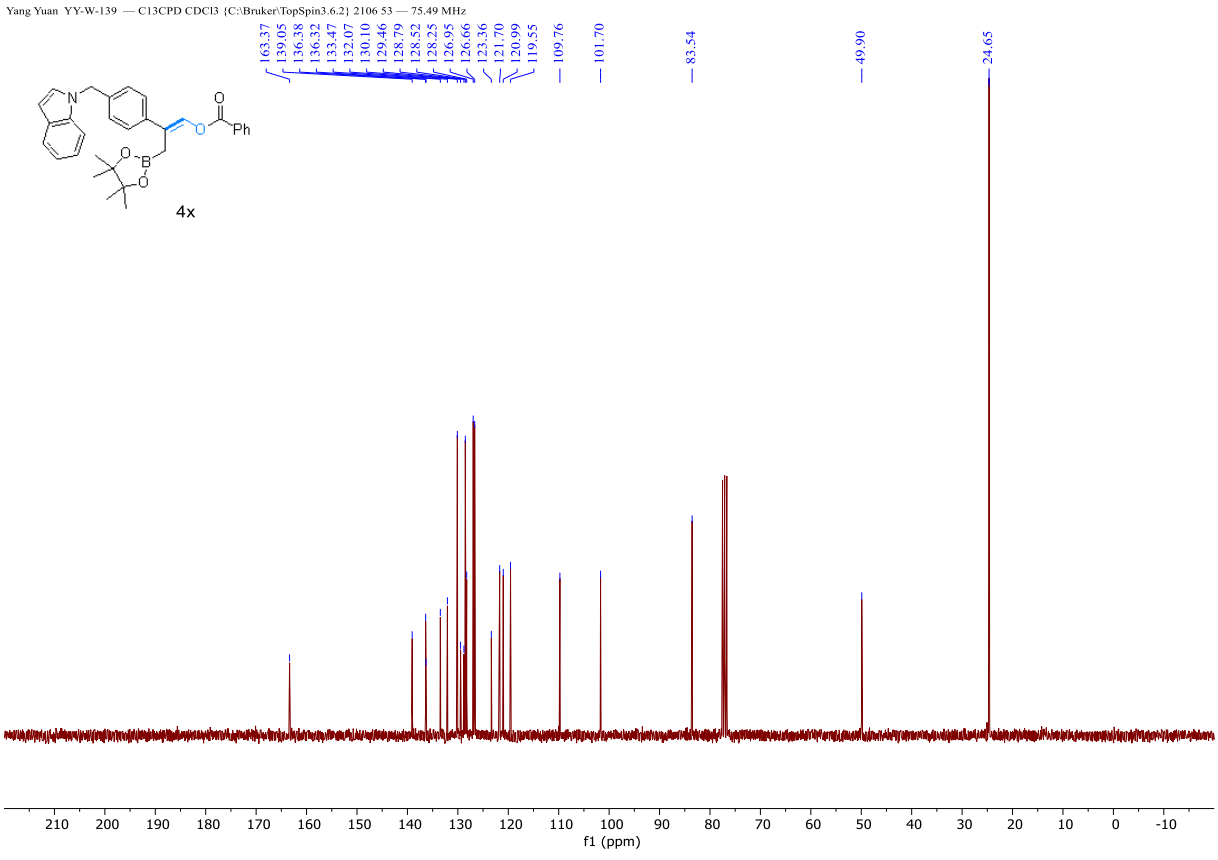
4v


Yang Yuan YY-W-126 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 7 — 128.38 MHz
33.23
O
O
O
O
B
O
Ph
4v
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

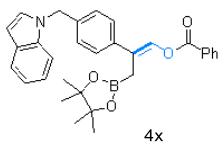

Yang Yuan YY-W-115 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 2 — 400.13 MHz
7.97 7.97 7.96 7.95 7.95 7.94 7.94 7.85 7.84 7.84 7.84 7.82 7.82 7.82 7.48 7.47 7.47 7.39 7.39 7.37 7.37 7.37 7.35 7.35 7.34 7.33 7.31 7.31 7.29
2.55 2.53 2.51 2.49 2.22
1.26 1.24 1.22 1.14
4w
1.03 1.30 1.06 3.01
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3
f1 (ppm)
1.03 1.30 1.06 3.01 2.13 2.00 3.08 12.15
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Yang Yuan YY-W-115 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 2 — 100.63 MHz
171.41
140.35 137.88 135.89 133.44 124.25 124.17 123.43 123.28 122.74 117.85
83.46
27.56 24.62 24.56
8.97
4w
270 260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

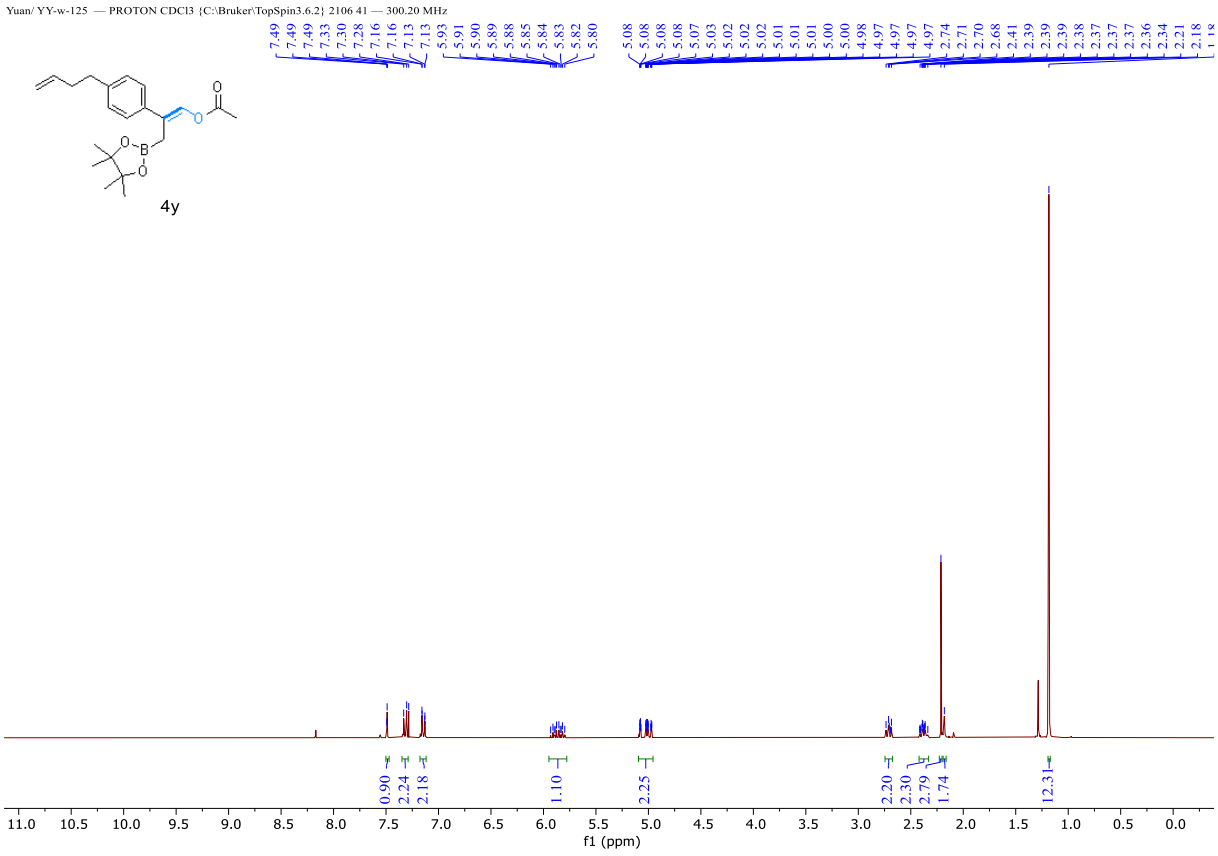

Yang Yuan YY-W-139 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 53 — 300.20 MHz
4x
f1 (ppm)

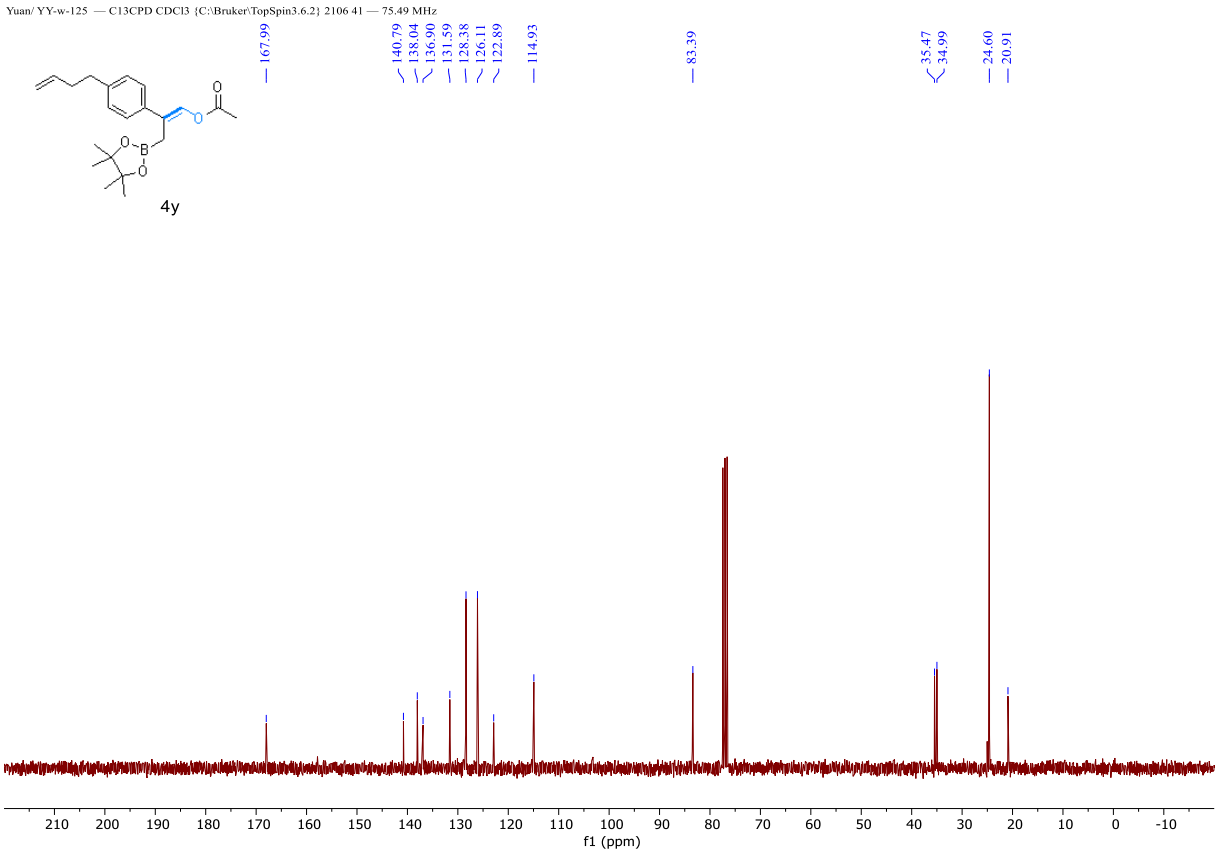

Yang Yuan YY-W-139 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 53 — 75.49 MHz
4x
f1 (ppm)

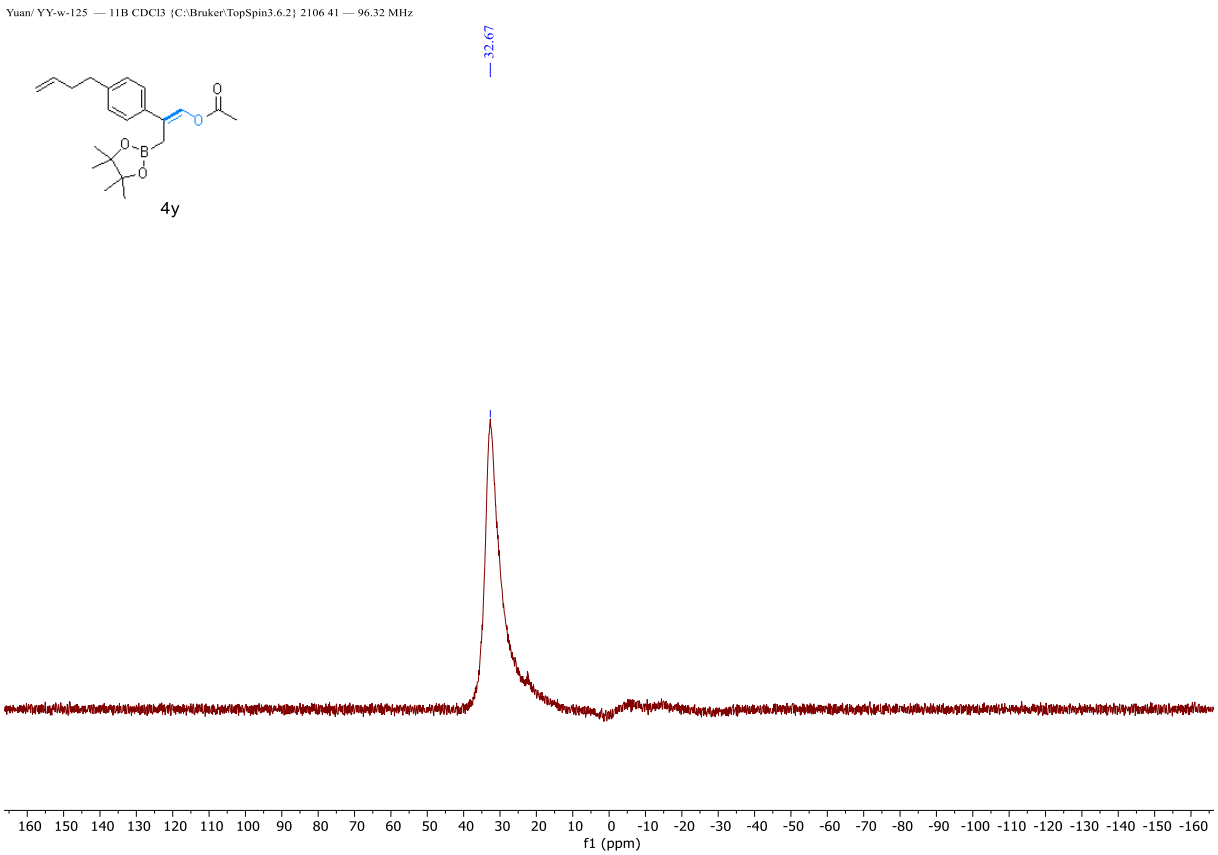
Yang Yuan YY-W-139 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 53 — 96.32 MHz

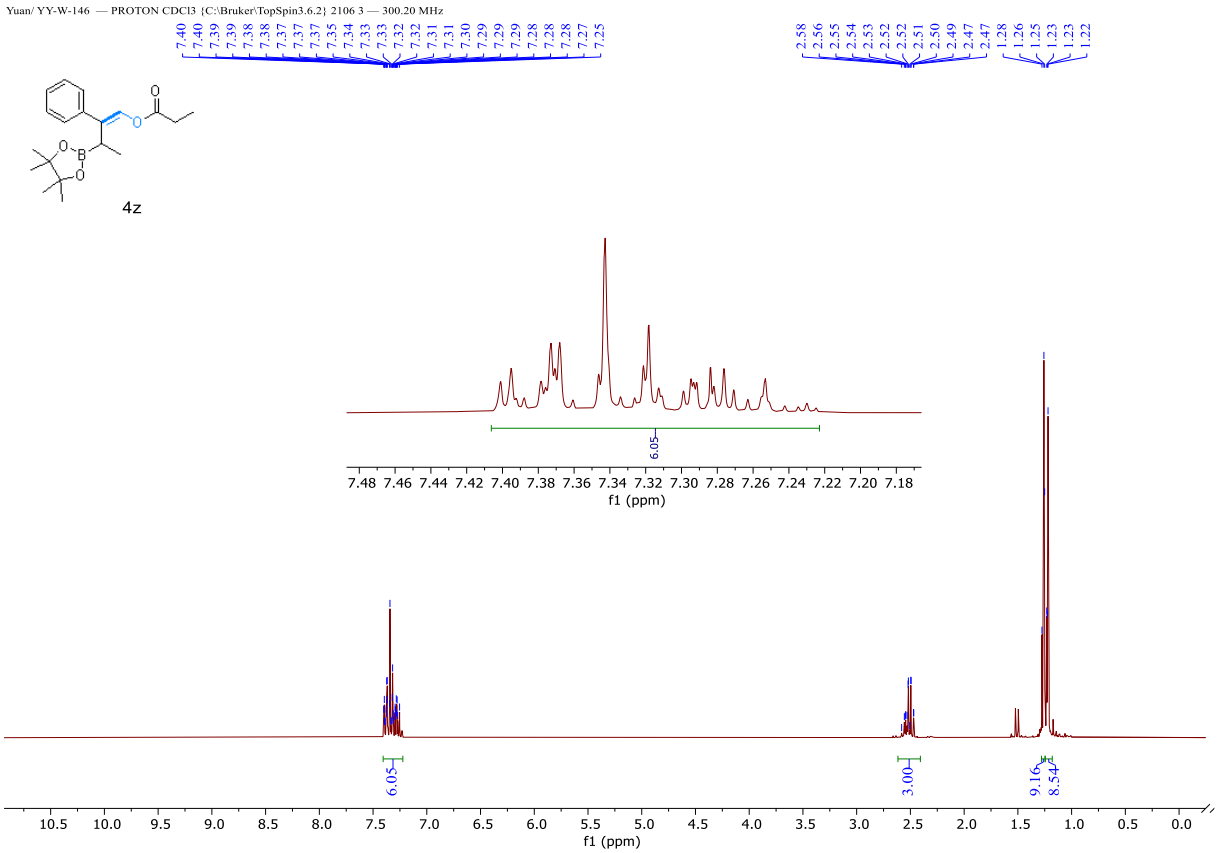


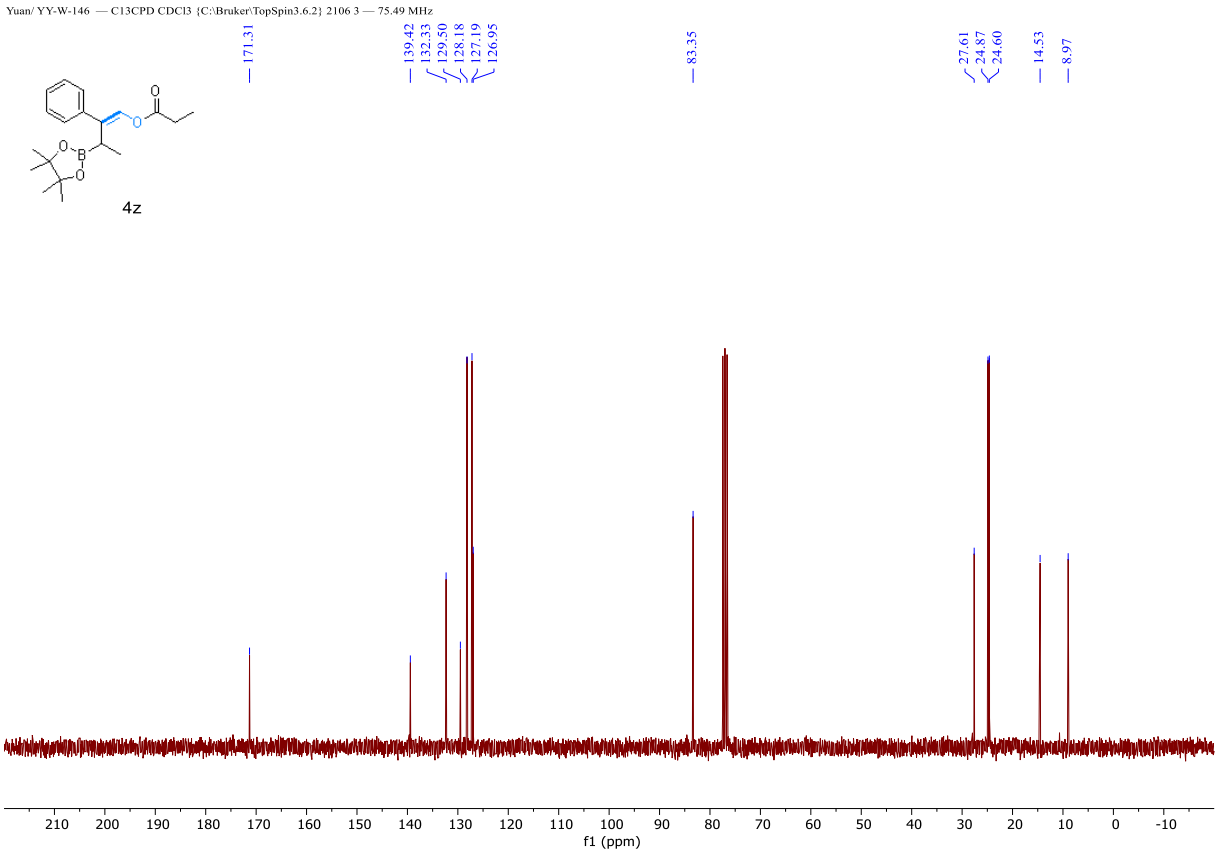
4x


Yuan/ YY-w-125 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 41 — 300.20 MHz
4y
0.90
2.24
2.18
1.10
2.25
2.20
2.30
2.79
1.74
12.31
f1 (ppm)

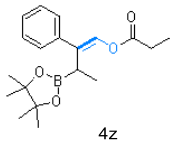

Yuan/ YY-w-125 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 41 — 75.49 MHz
4y
167.99
140.79
138.04
136.90
131.59
128.38
126.11
122.89
114.93
83.39
35.47
34.99
24.60
20.91
f1 (ppm)


Yuan/ YY-w-125 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 41 — 96.32 MHz
32.67
O
O
B
O
O
4y
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

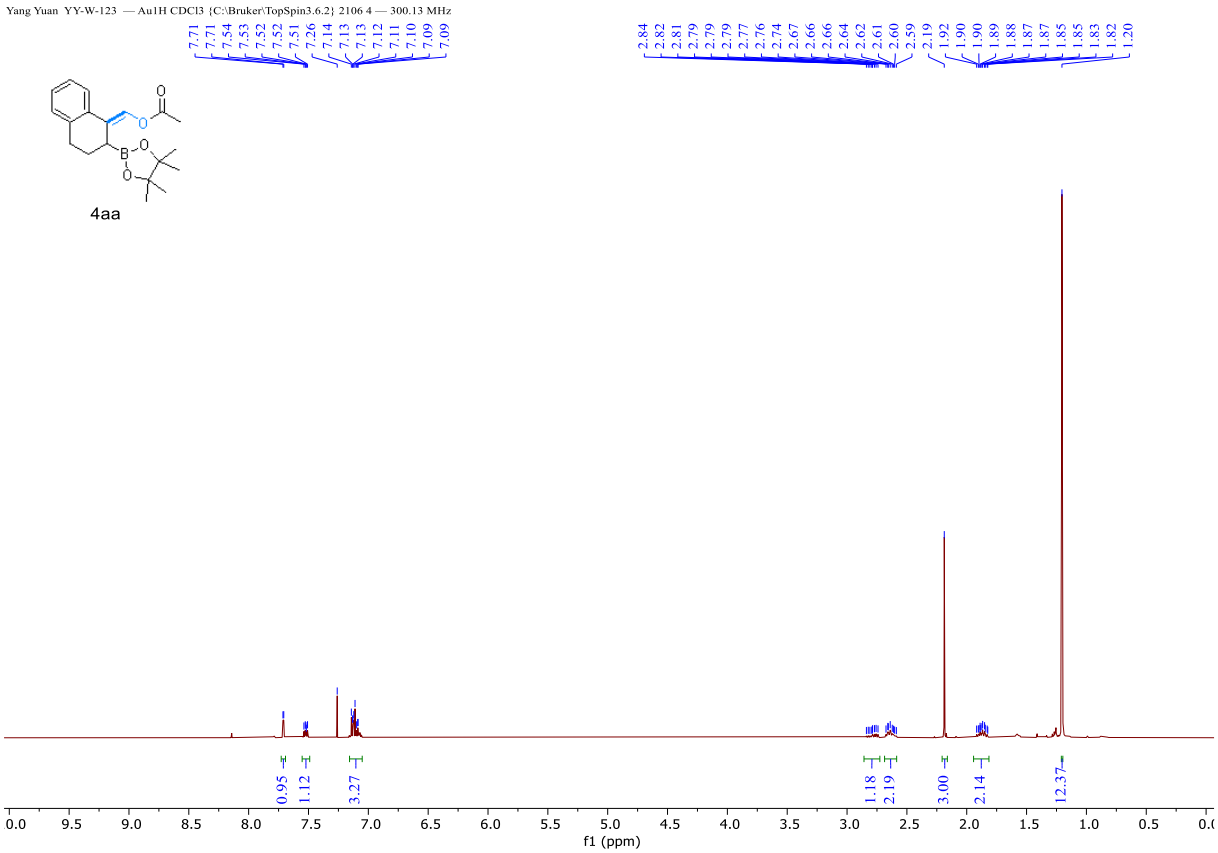

Yuan/ YY-W-146 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 3 — 300.20 MHz
7.40 7.40 7.39 7.39 7.38 7.38 7.37 7.37 7.37 7.35 7.34 7.33 7.33 7.32 7.32 7.31 7.31 7.30 7.29 7.29 7.28 7.28 7.28 7.27 7.25
2.58 2.56 2.55 2.54 2.53 2.52 2.52 2.51 2.50 2.49 2.47 2.47
1.28 1.26 1.25 1.23 1.23 1.22
4z
6.05
7.48 7.46 7.44 7.42 7.40 7.38 7.36 7.34 7.32 7.30 7.28 7.26 7.24 7.22 7.20 7.18
f1 (ppm)
6.05
3.00
9.16
8.54
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

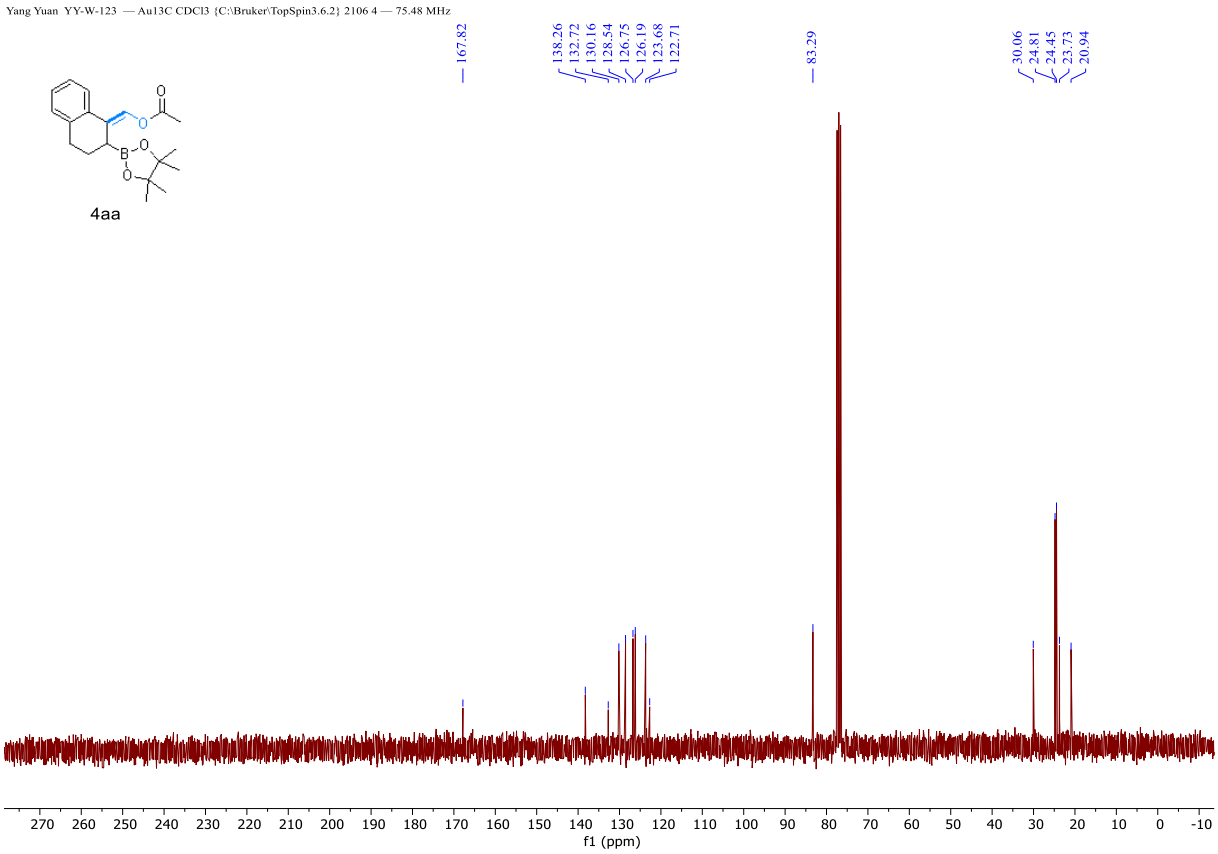

Yuan/ YY-W-146 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 3 — 75.49 MHz
171.31
139.42
132.33
129.50
128.18
127.19
126.95
83.35
27.61
24.87
24.60
14.53
8.97
4z
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

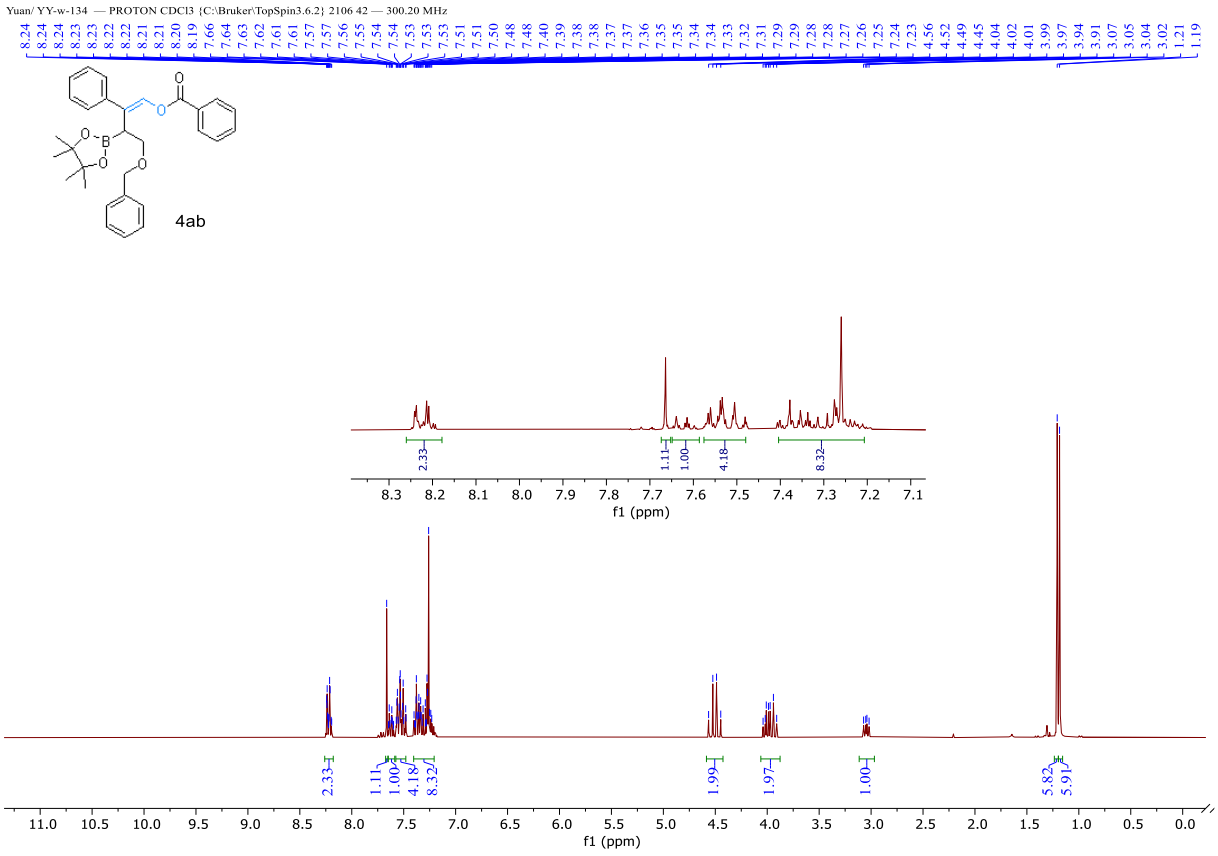
Yuan/ YY-W-146 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 3 — 96.32 MHz

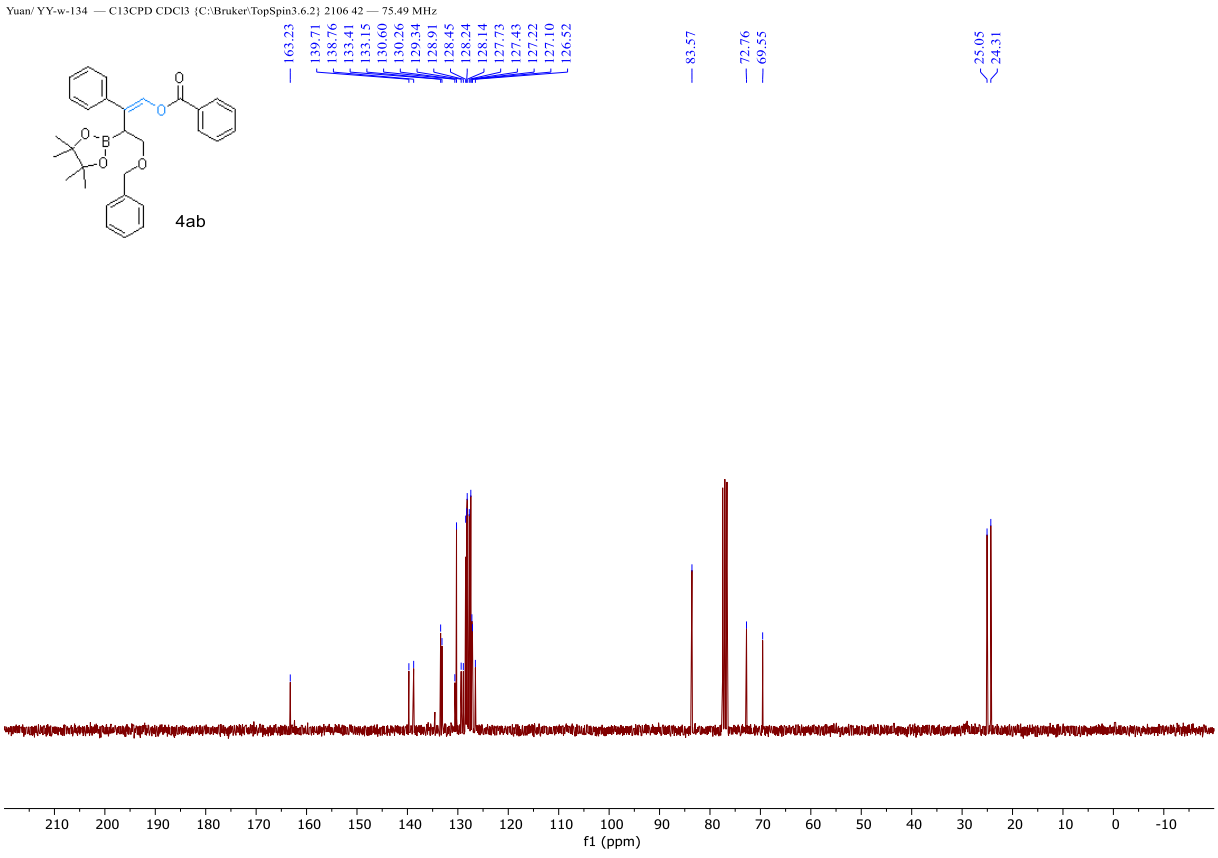


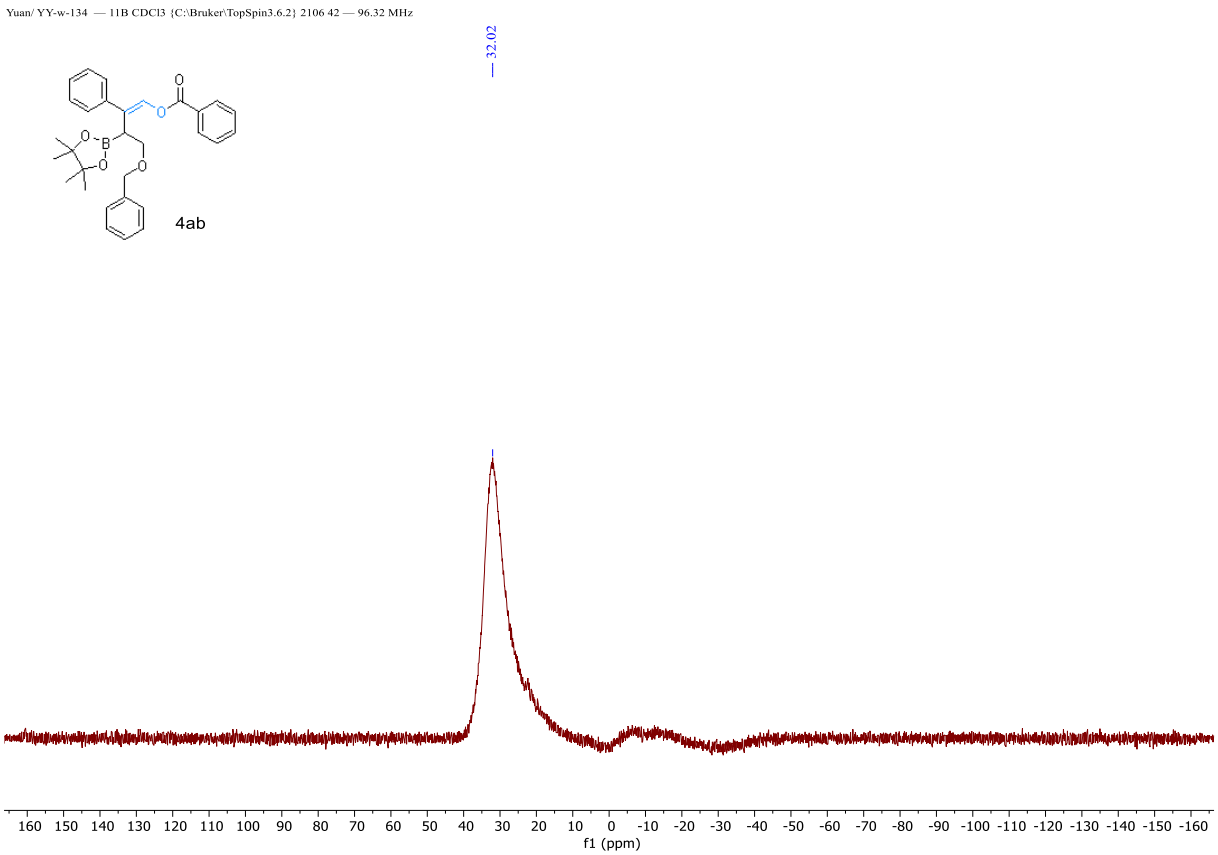
4z

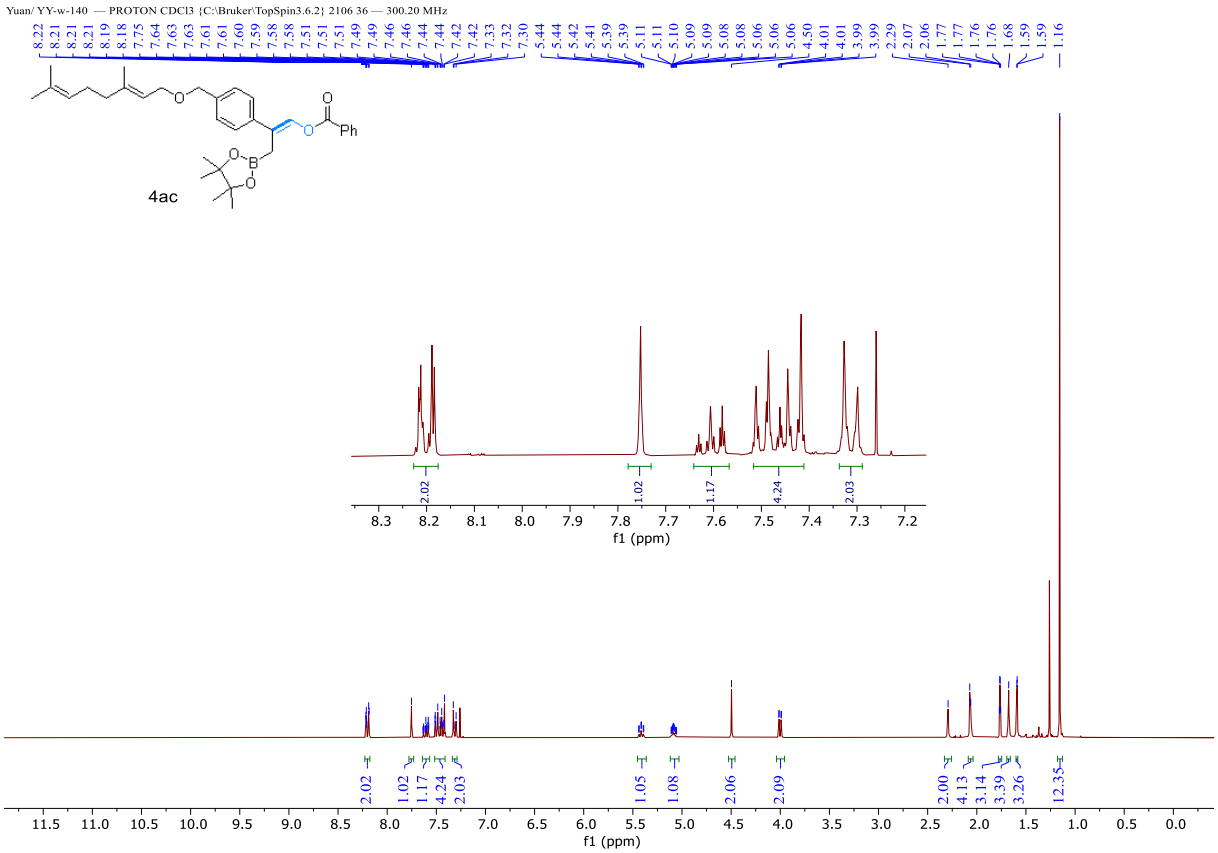

Yang Yuan YY-W-123 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 4 — 300.13 MHz
7.71 7.71 7.54 7.53 7.52 7.52 7.51 7.26 7.14 7.13 7.13 7.12 7.11 7.10 7.09 7.09
2.84 2.82 2.81 2.79 2.79 2.79 2.77 2.76 2.74 2.67 2.66 2.66 2.64 2.62 2.61 2.60 2.59 2.19 1.92 1.90 1.90 1.89 1.88 1.87 1.87 1.85 1.85 1.83 1.82 1.20
4aa
0.95 1.12 3.27 1.18 2.19 3.00 2.14 12.37
f1 (ppm)

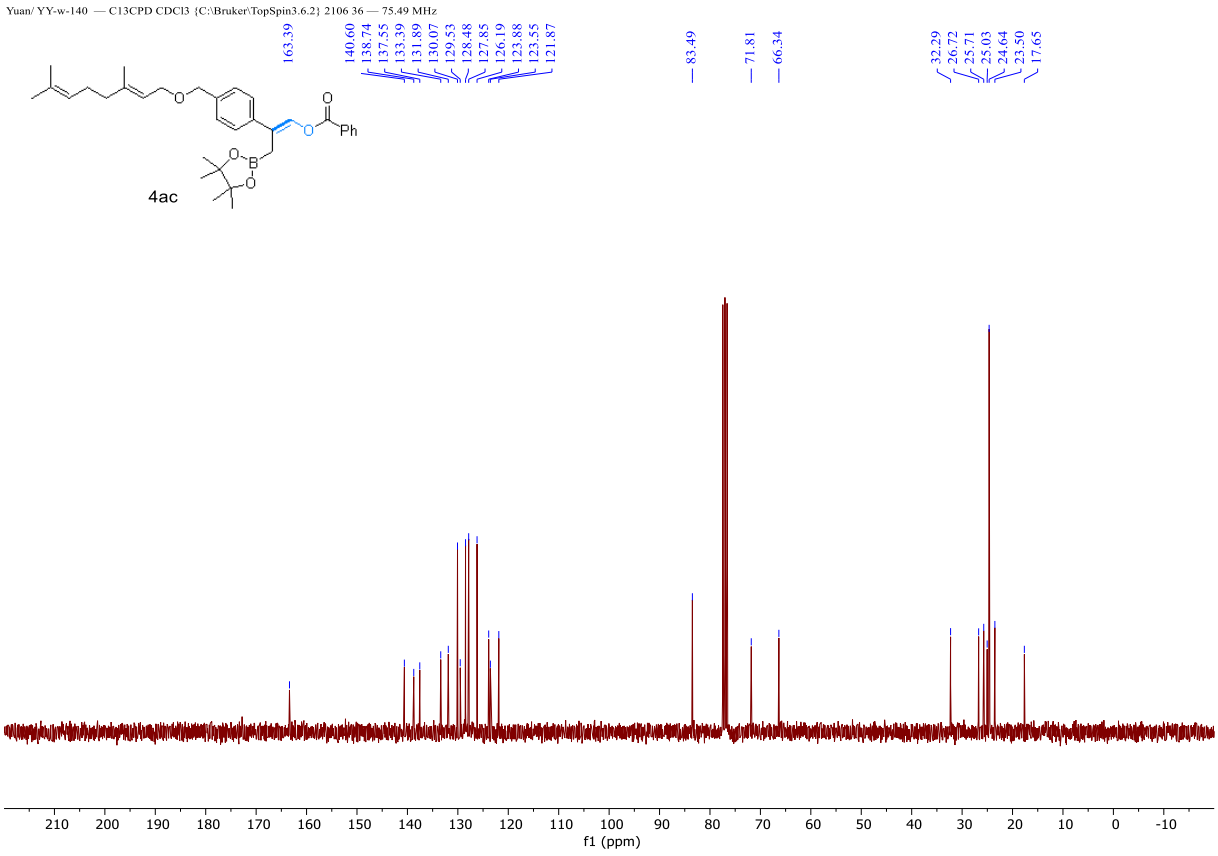

Yang Yuan YY-W-123 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 4 — 75.48 MHz
167.82 138.26 132.72 130.16 128.54 126.75 126.19 123.68 122.71 83.29 30.06 24.81 24.45 23.73 20.94
4aa
f1 (ppm)

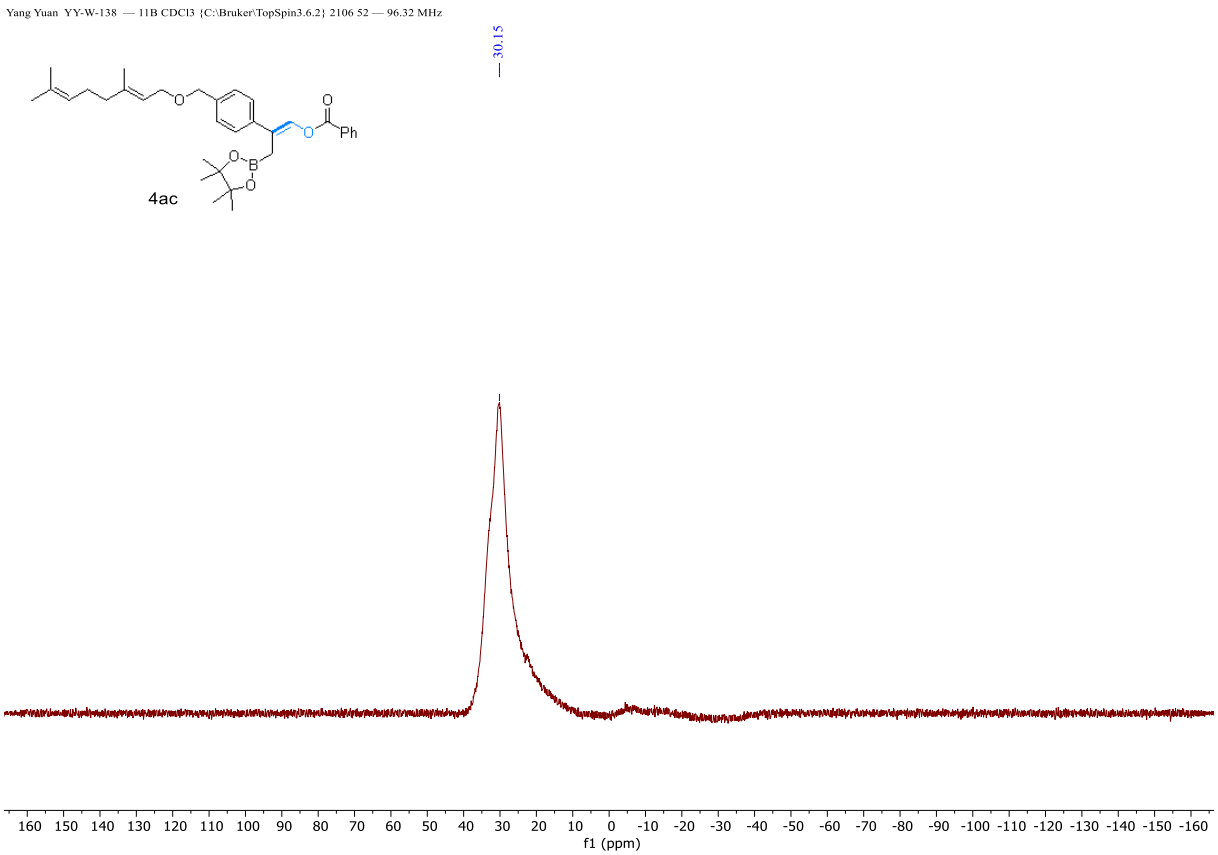

Yuan/ YY-w-134 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 42 — 300.20 MHz
4ab
f1 (ppm)

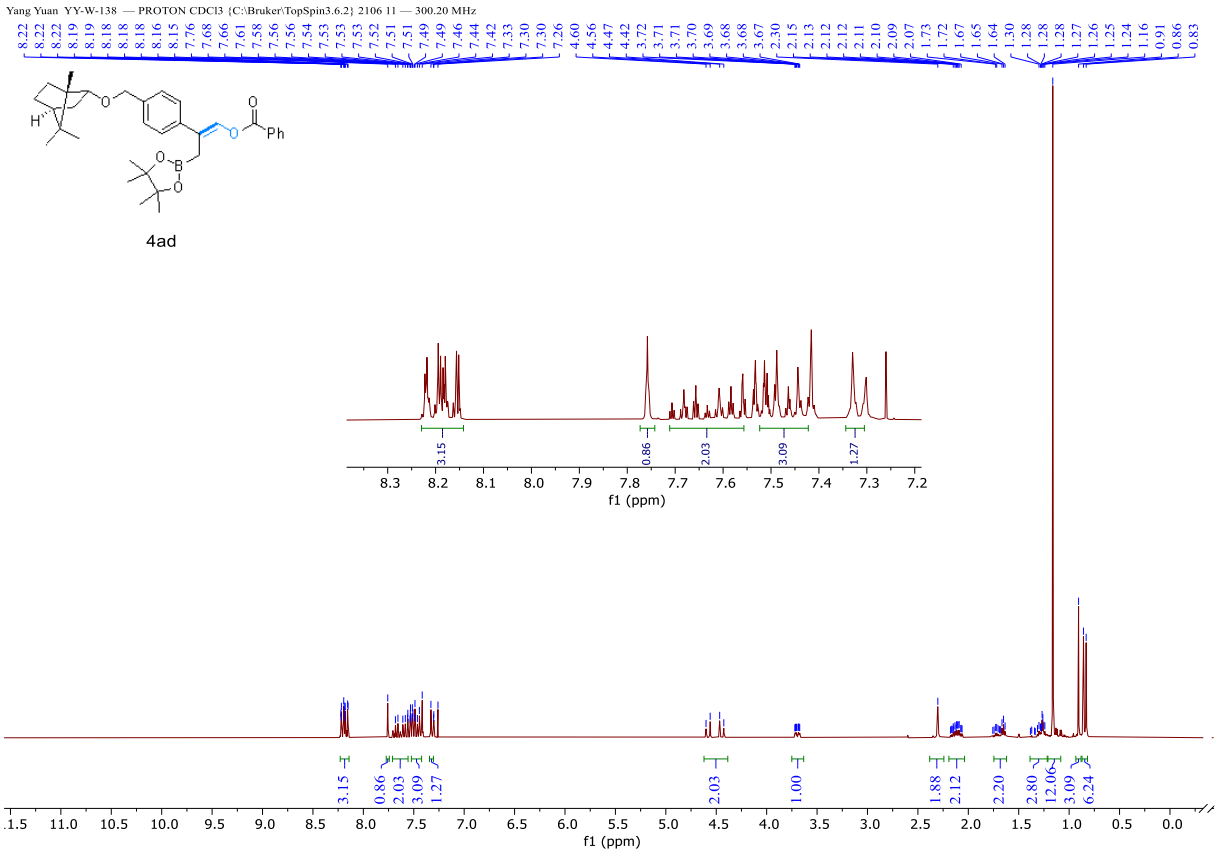

Yuan/ YY-w-134 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 42 — 75.49 MHz
4ab
f1 (ppm)

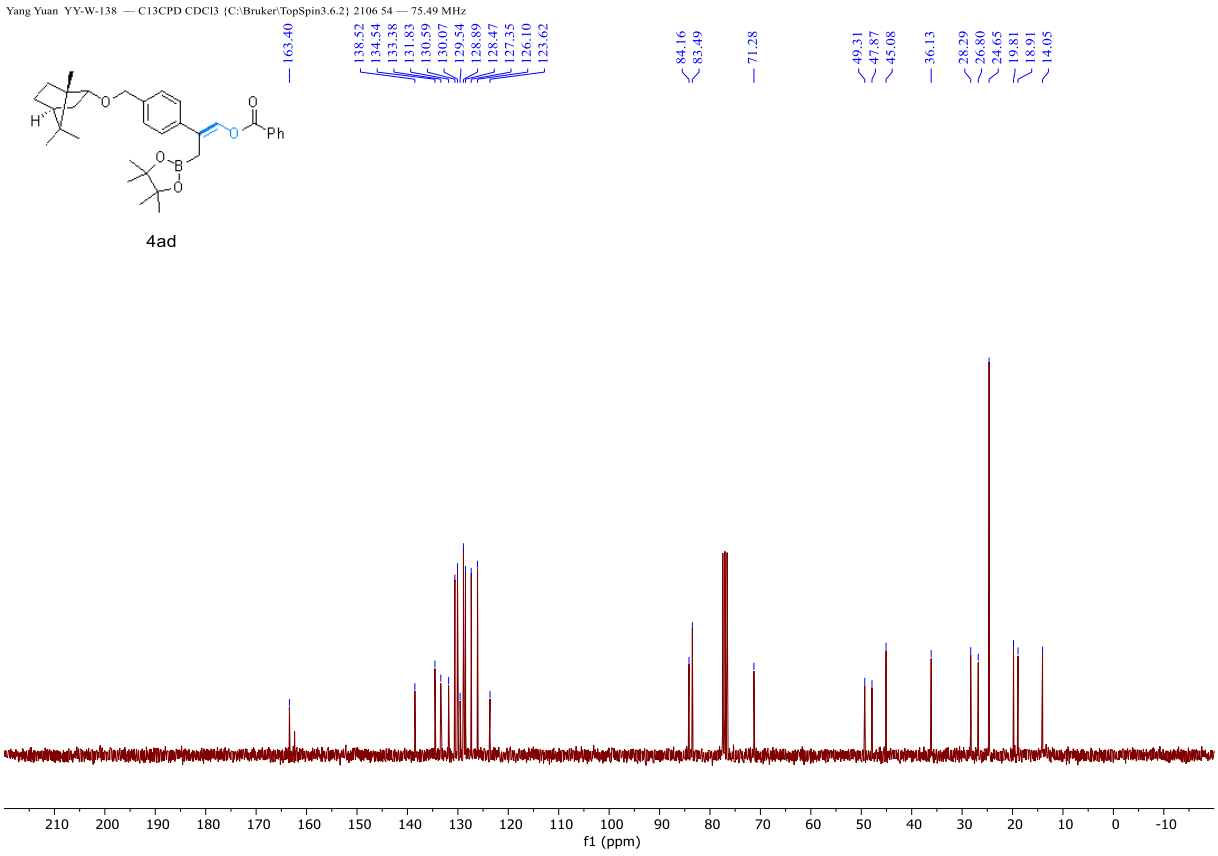

Yuan/ YY-w-134 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 42 — 96.32 MHz
32.02
4ab
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)


Yuan/ YY-w-140 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 36 — 300.20 MHz
4ac
f1 (ppm)

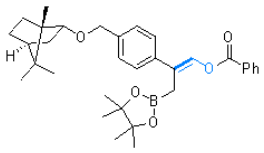

Yuan/ YY-w-140 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 36 — 75.49 MHz
4ac
f1 (ppm)


Yang Yuan YY-W-138 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 52 — 96.32 MHz
30.15
O
Ph
O
O
B
O
4ac
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

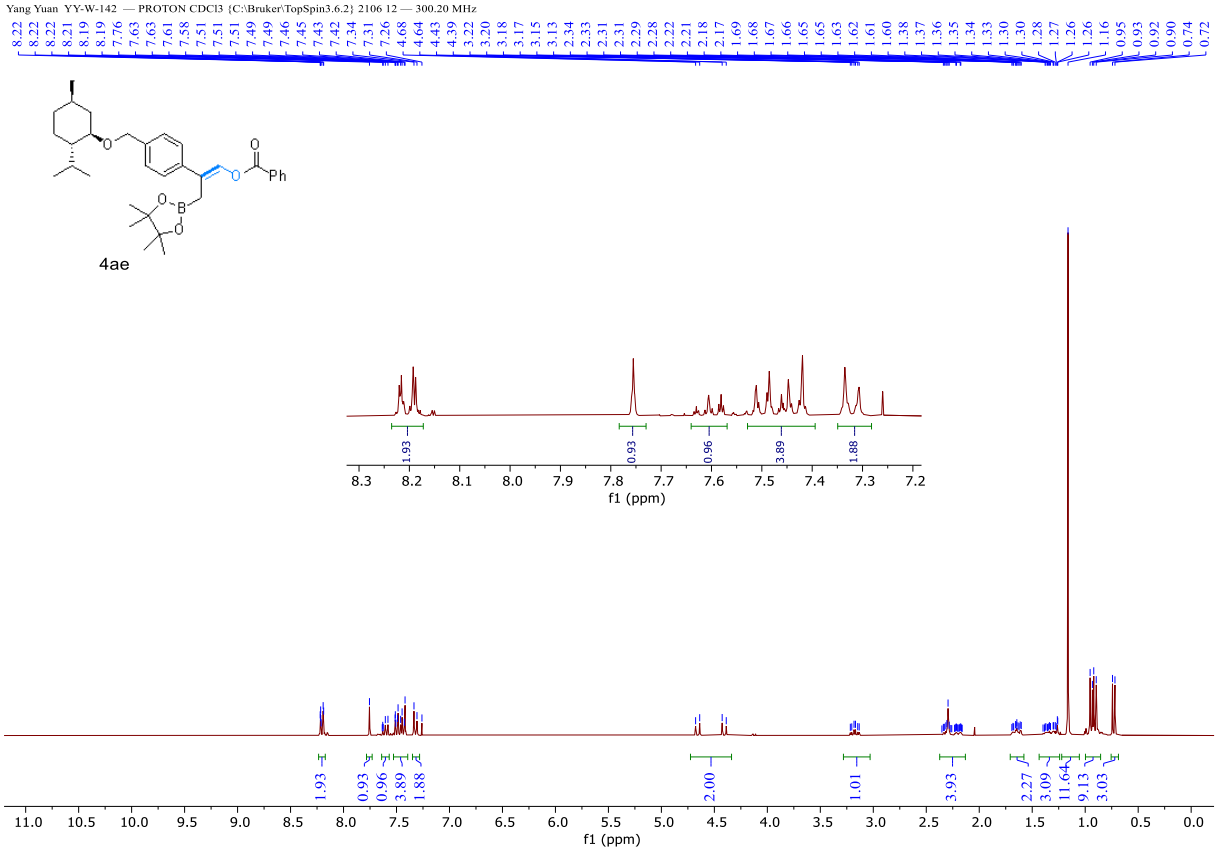

Yang Yuan YY-W-138 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 11 — 300.20 MHz
4ad
f1 (ppm)

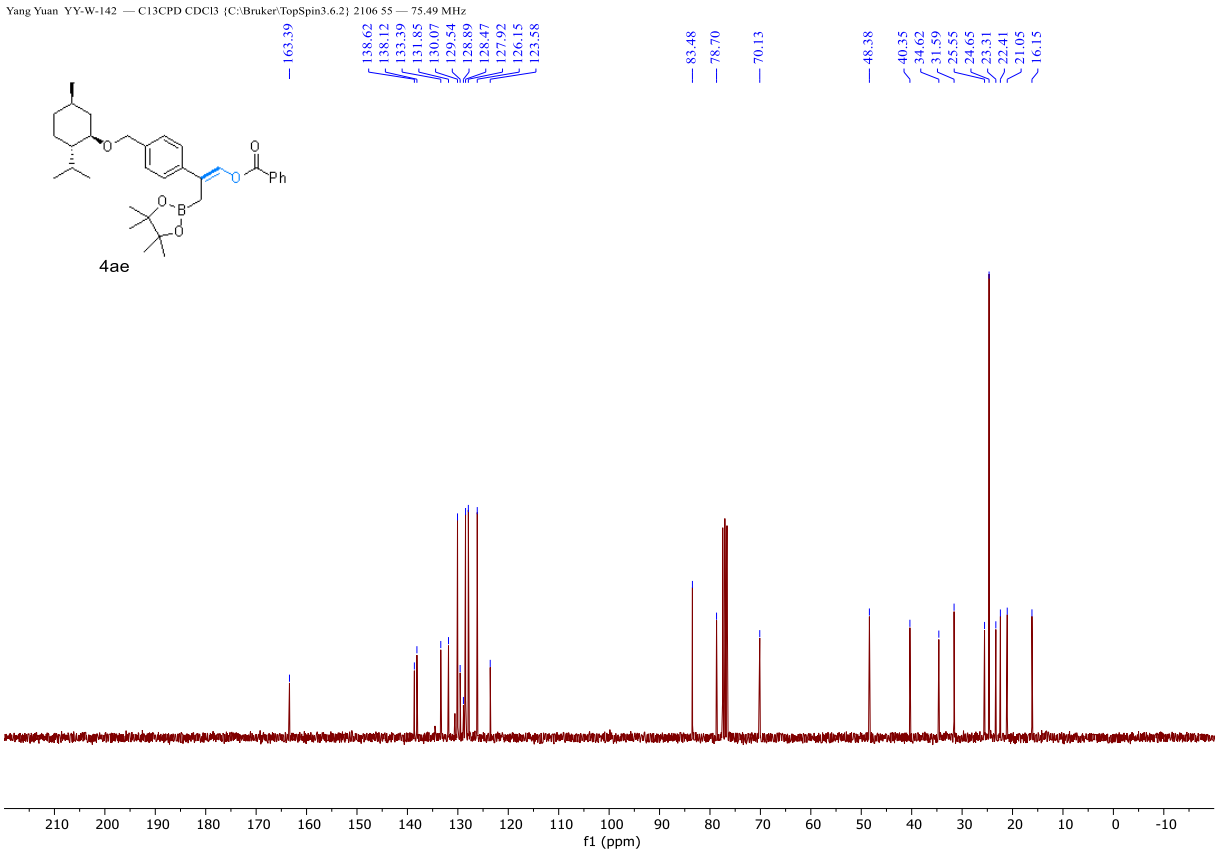

Yang Yuan YY-W-138 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 54 — 75.49 MHz
4ad
f1 (ppm)

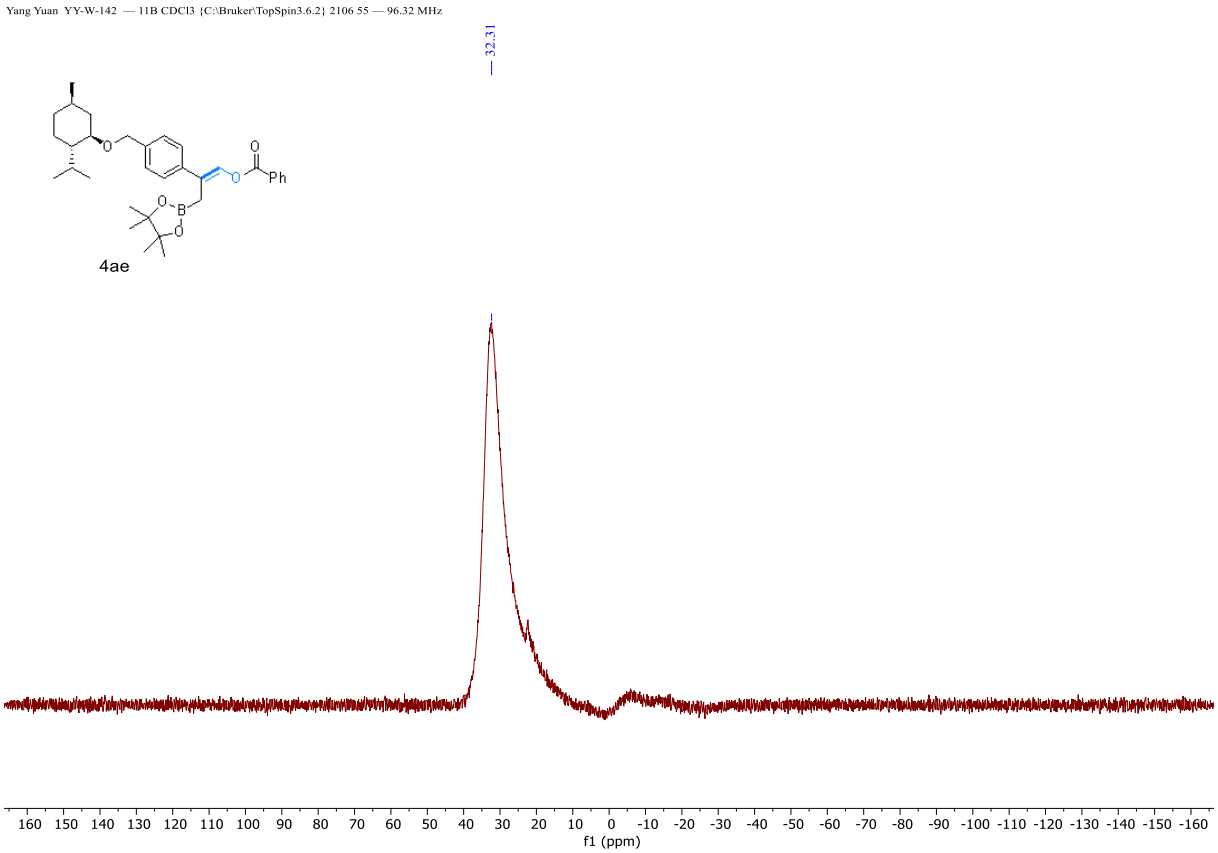
Yang Yuan YY-W-138 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 54 — 96.32 MHz

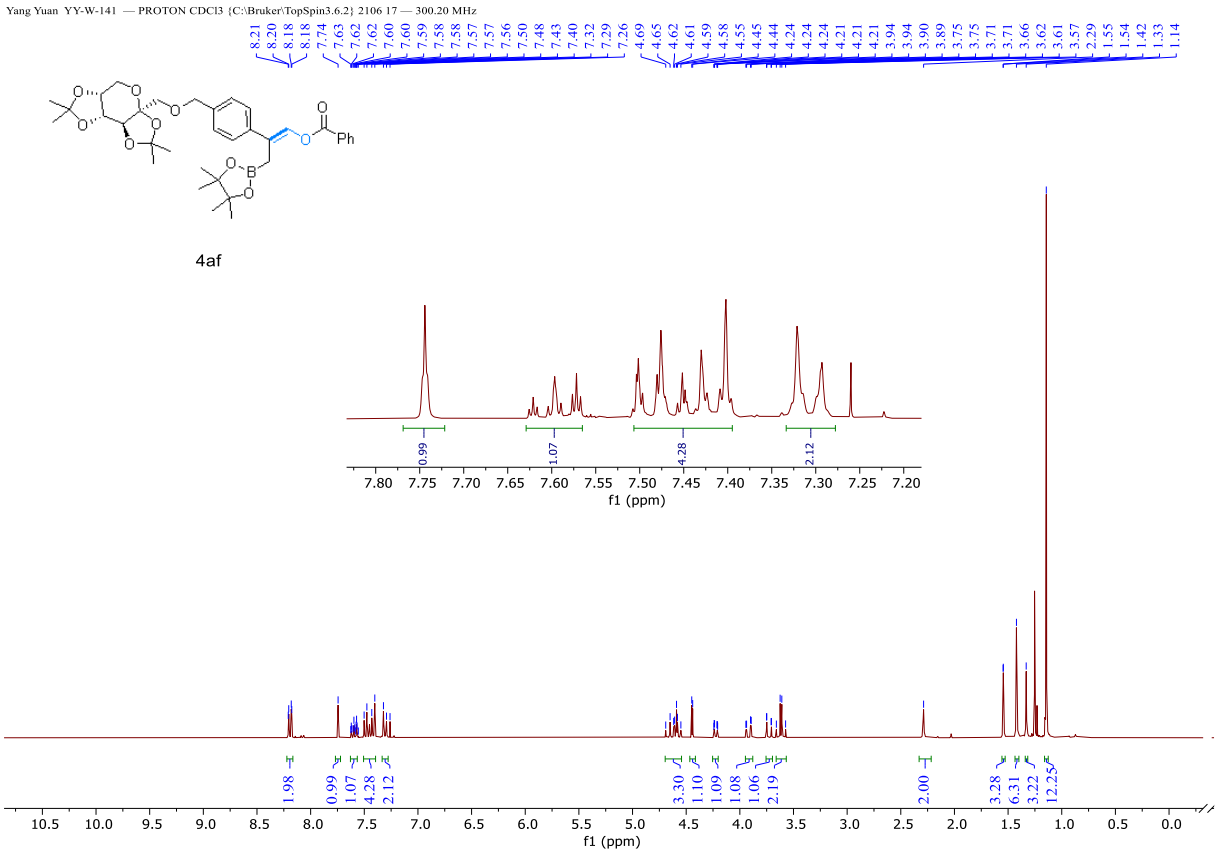


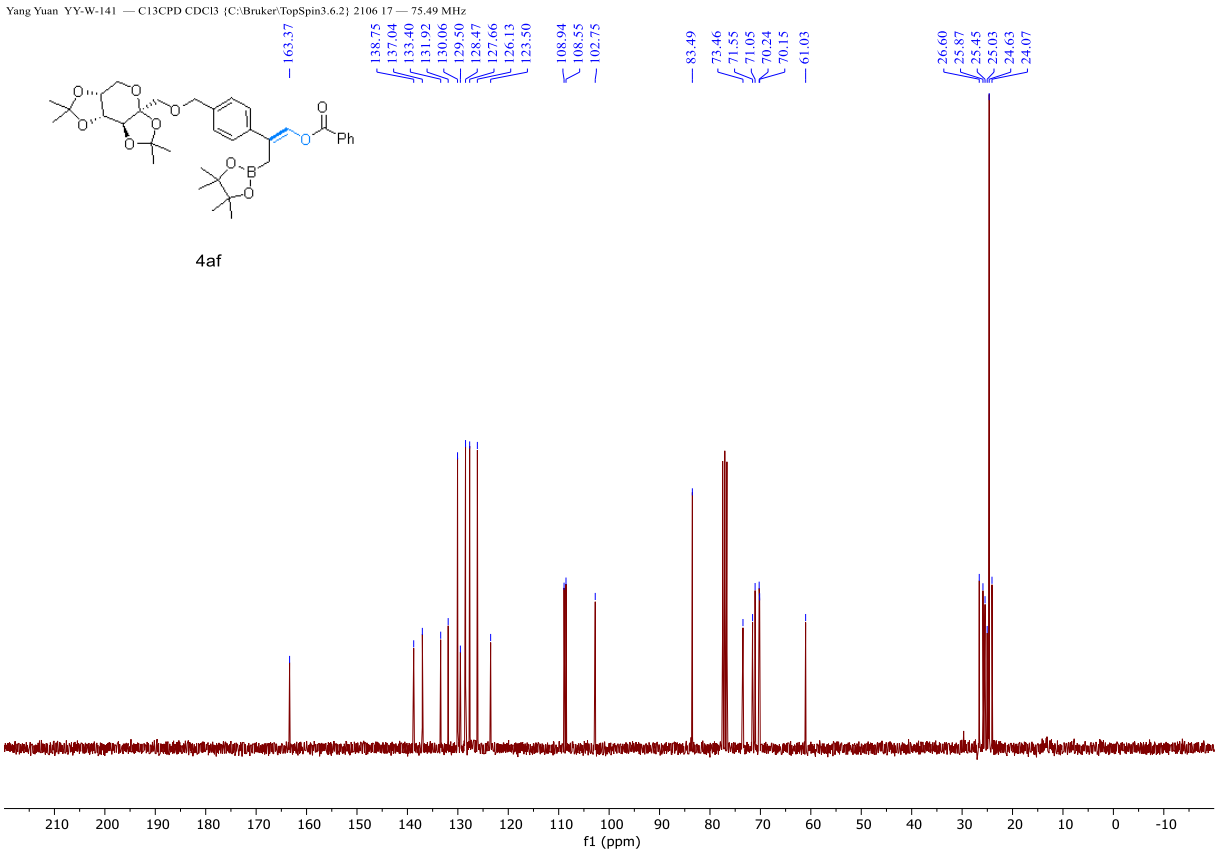
4ad

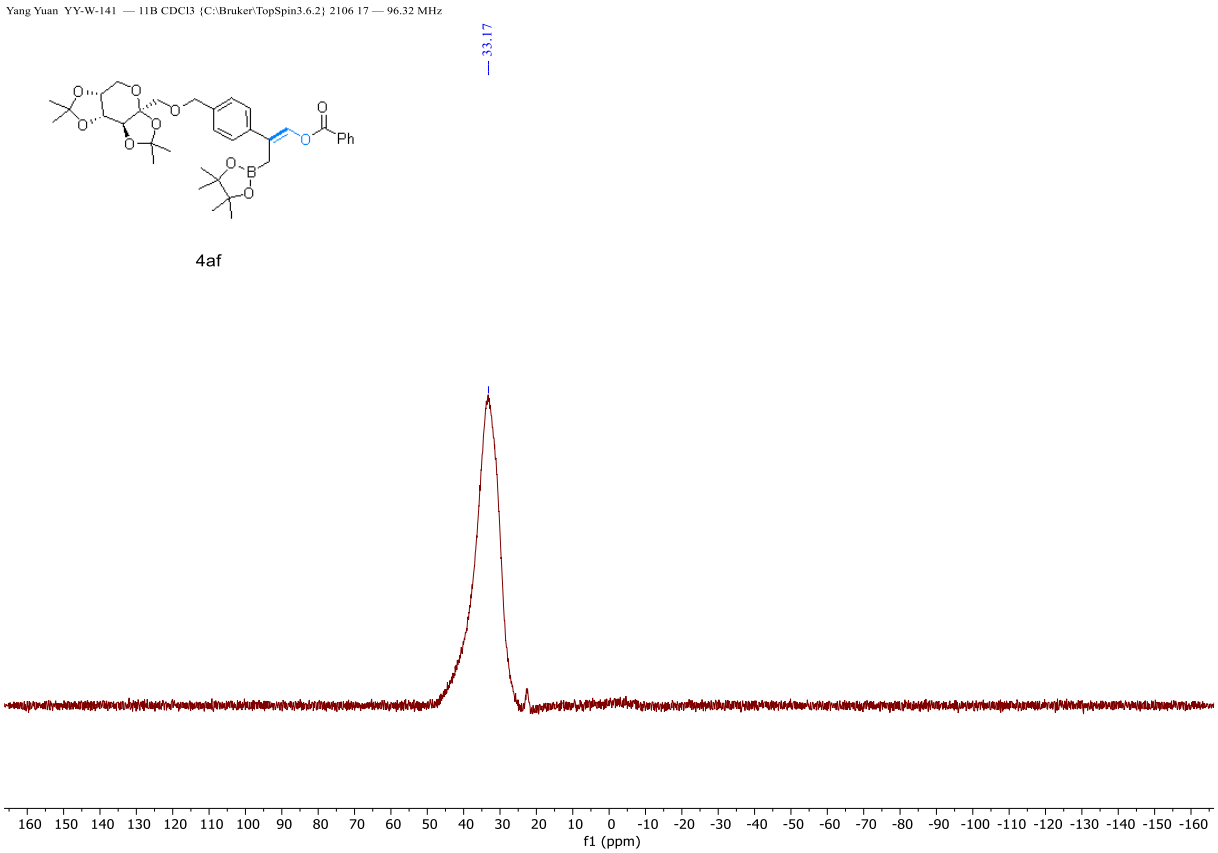

Yang Yuan YY-W-142 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 12 — 300.20 MHz
4ae
f1 (ppm)

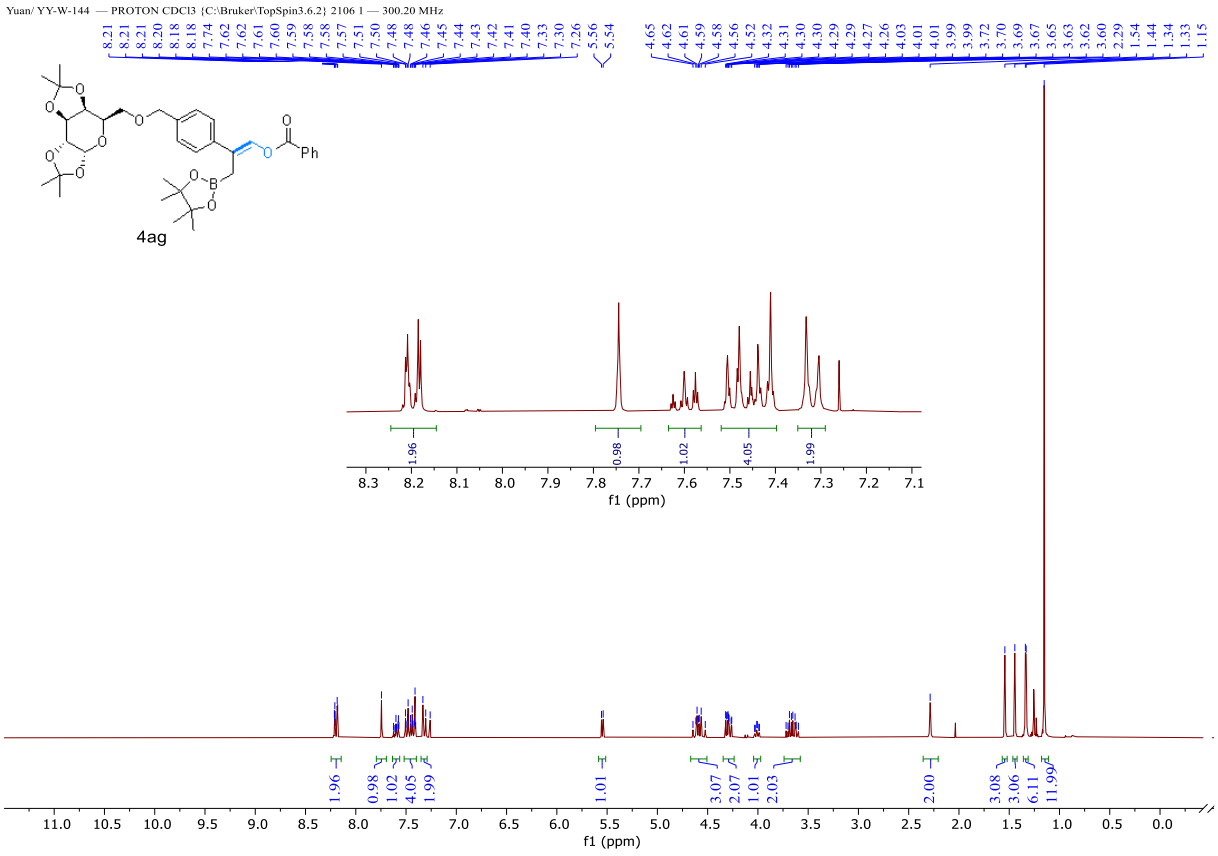

Yang Yuan YY-W-142 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 55 — 75.49 MHz
4ae
f1 (ppm)

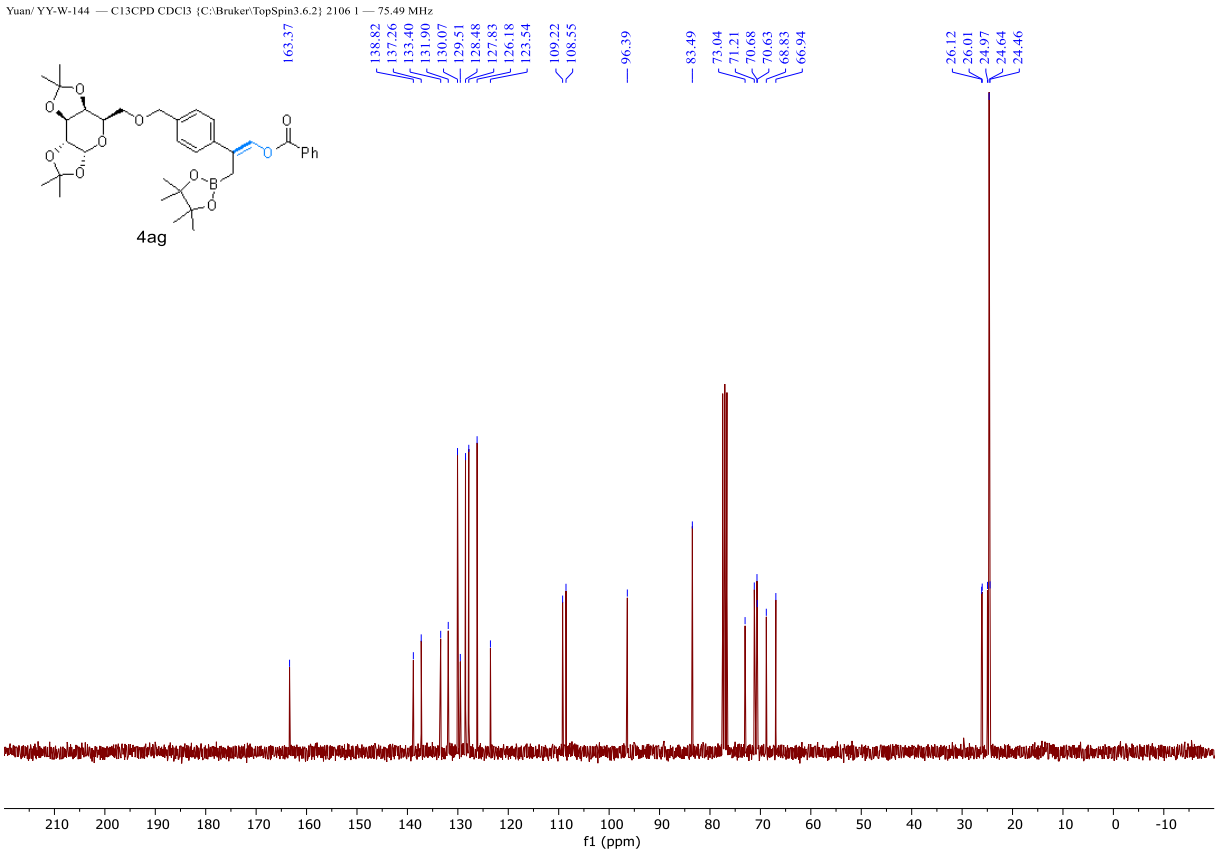

Yang Yuan YY-W-142 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 55 — 96.32 MHz
32.31
O
O
Ph
O
B
O
4ae
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

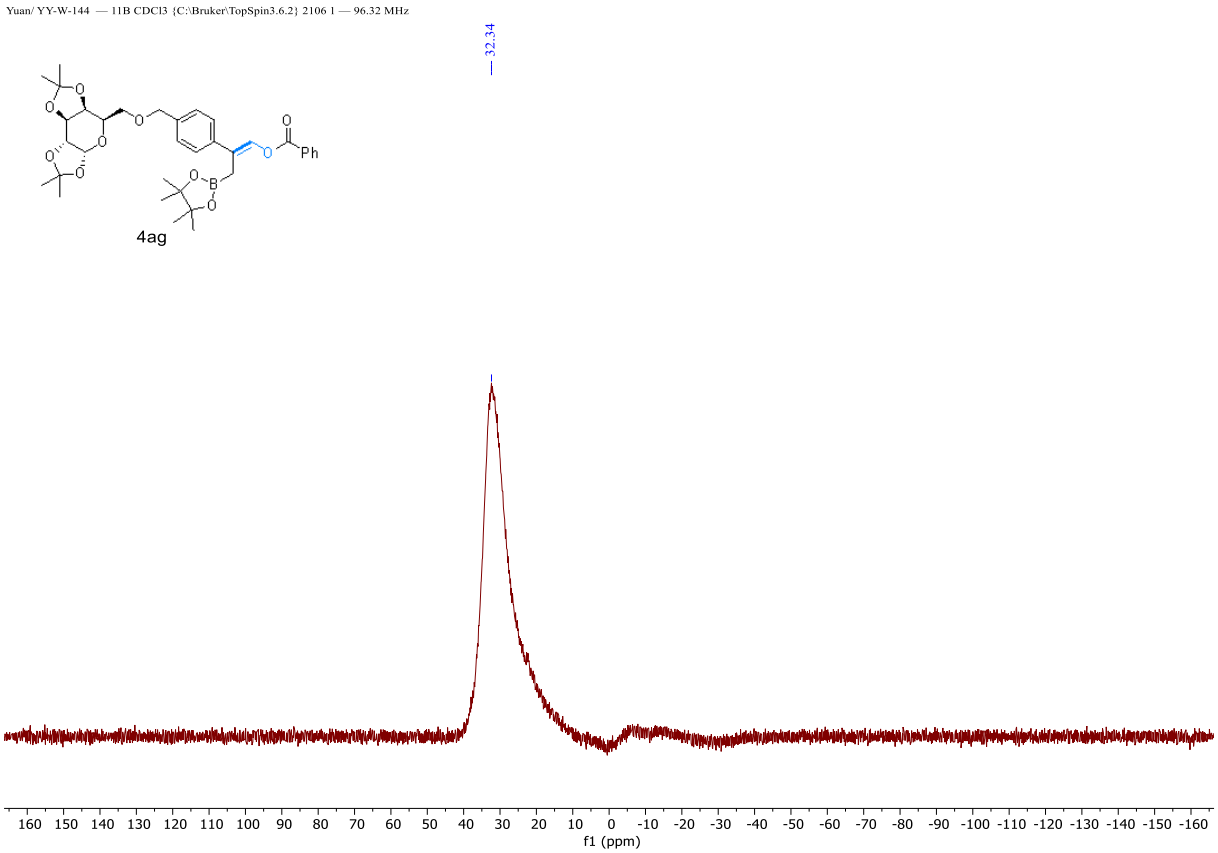

Yang Yuan YY-W-141 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 17 — 300.20 MHz
8.21 8.20 8.18 8.18 7.74 7.63 7.62 7.62 7.60 7.60 7.59 7.58 7.58 7.57 7.57 7.56 7.50 7.48 7.43 7.40 7.32 7.29 7.26 4.69 4.65 4.62 4.61 4.59 4.58 4.55 4.45 4.44 4.24 4.24 4.24 4.21 4.21 4.21 3.94 3.94 3.90 3.89 3.75 3.75 3.71 3.71 3.66 3.62 3.61 3.57 2.29 1.55 1.54 1.42 1.33 1.14
Ph
B
4af
0.99 1.07 4.28 2.12
7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20
f1 (ppm)
1.98 0.99 1.07 4.28 2.12 3.30 1.10 1.09 1.08 1.06 2.19 2.00 3.28 6.31 3.22 12.25
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

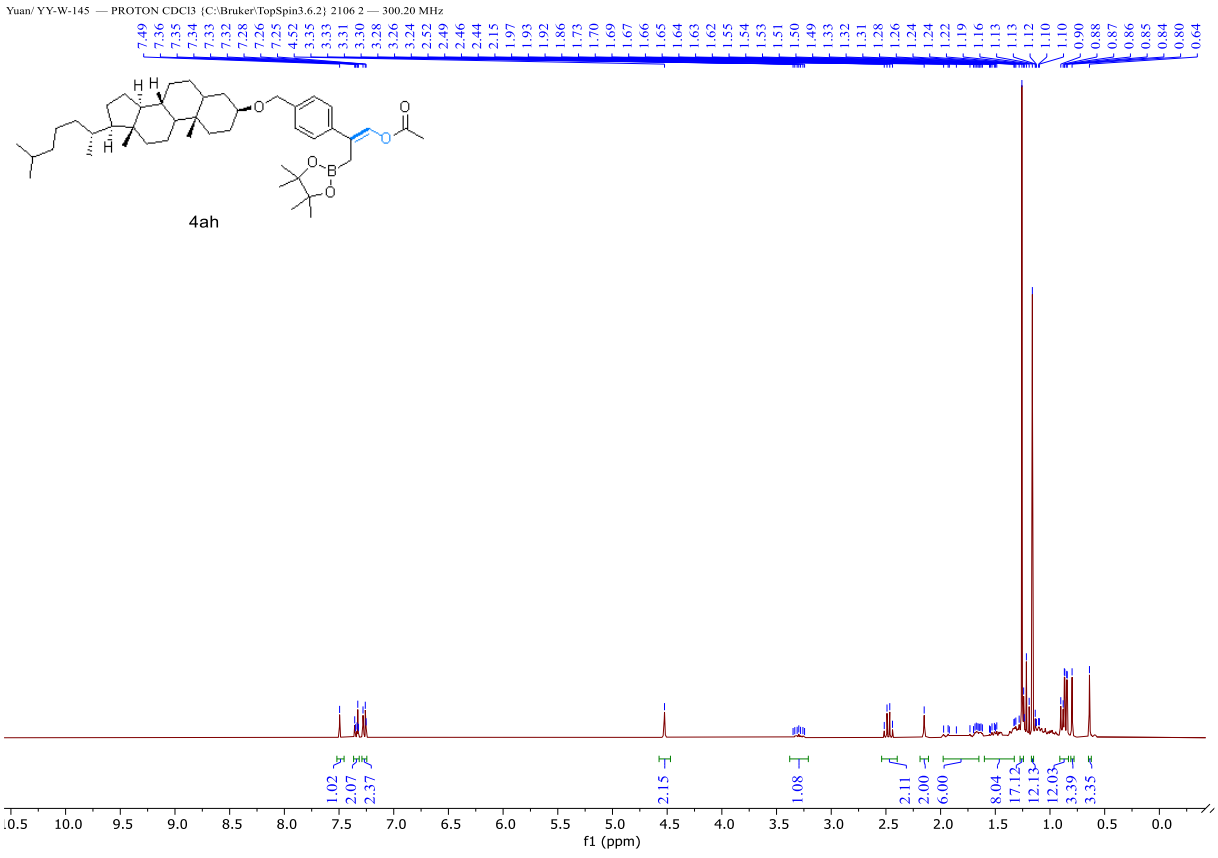

Yang Yuan YY-W-141 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 17 — 75.49 MHz
163.37 138.75 137.04 133.40 131.92 130.06 129.50 128.47 127.66 126.13 123.50 108.94 108.55 102.75 83.49 73.46 71.55 71.05 70.24 70.15 61.03 26.60 25.87 25.45 25.03 24.63 24.07
Ph
B
4af
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

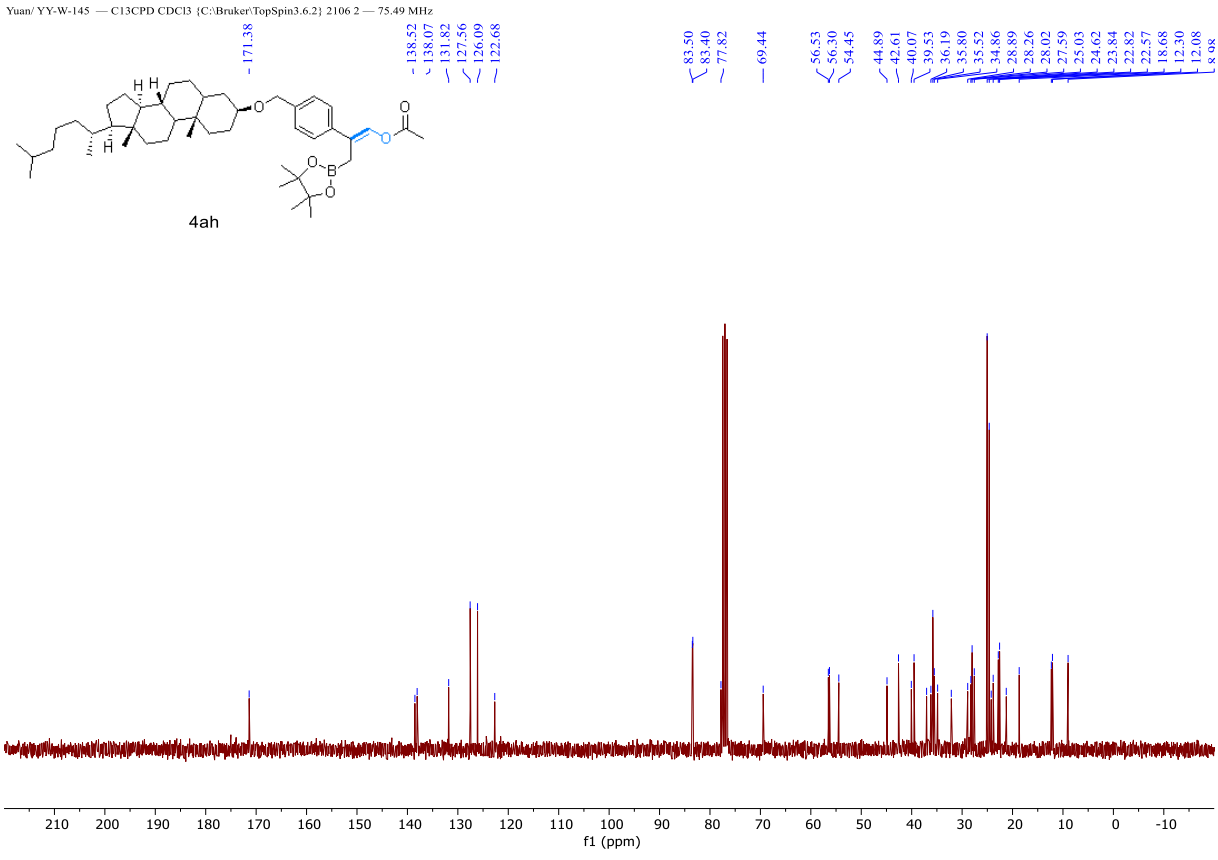

Yang Yuan YY-W-141 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 17 — 96.32 MHz
33.17
Ph
4af
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

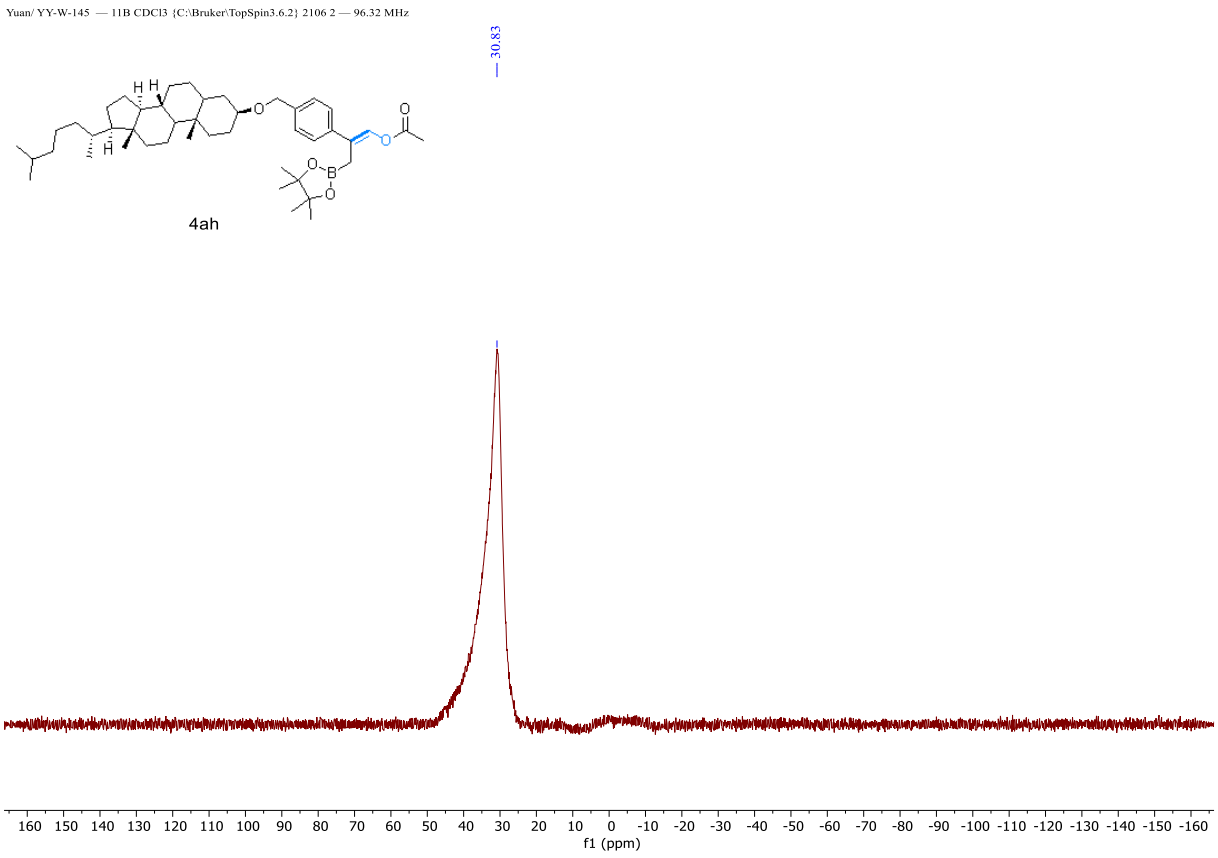

Yuan/ YY-W-144 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 1 — 300.20 MHz
4ag
f1 (ppm)

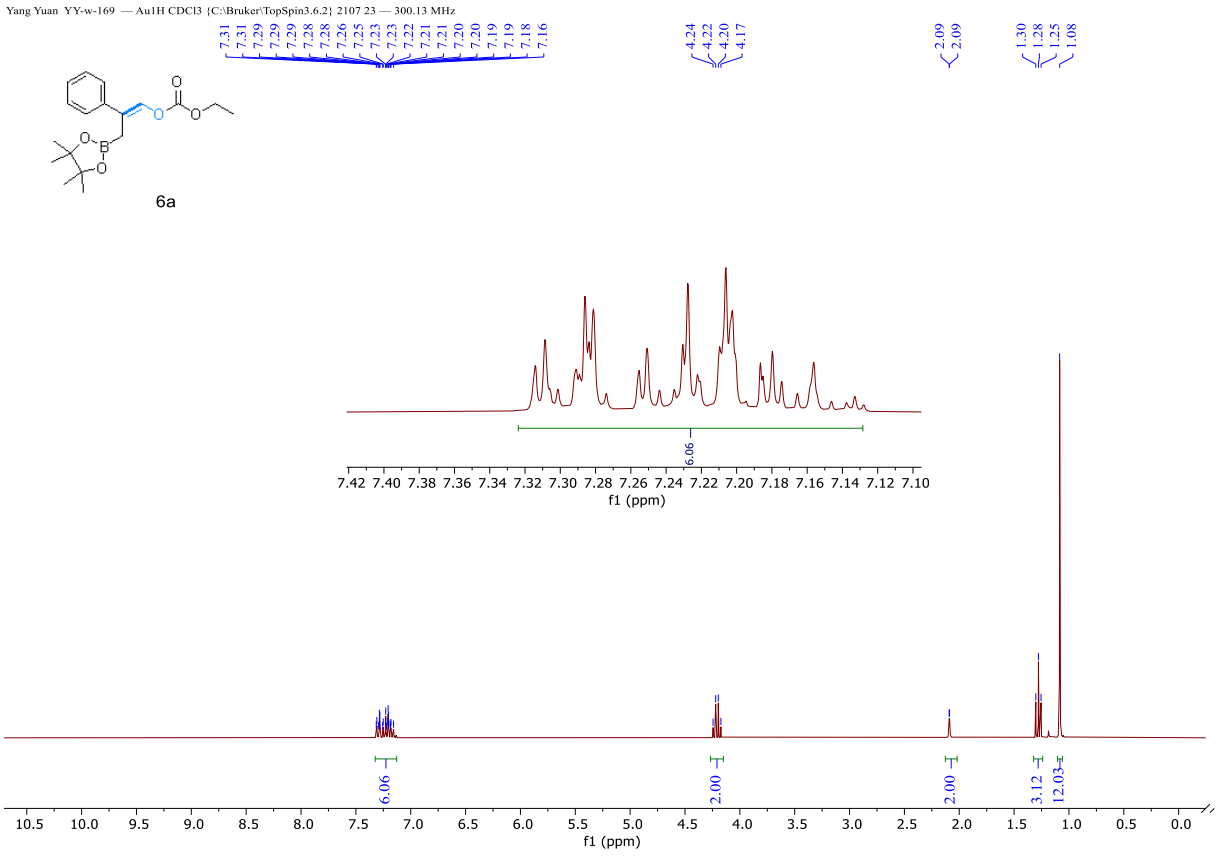

Yuan/ YY-W-144 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 1 — 75.49 MHz
4ag
f1 (ppm)

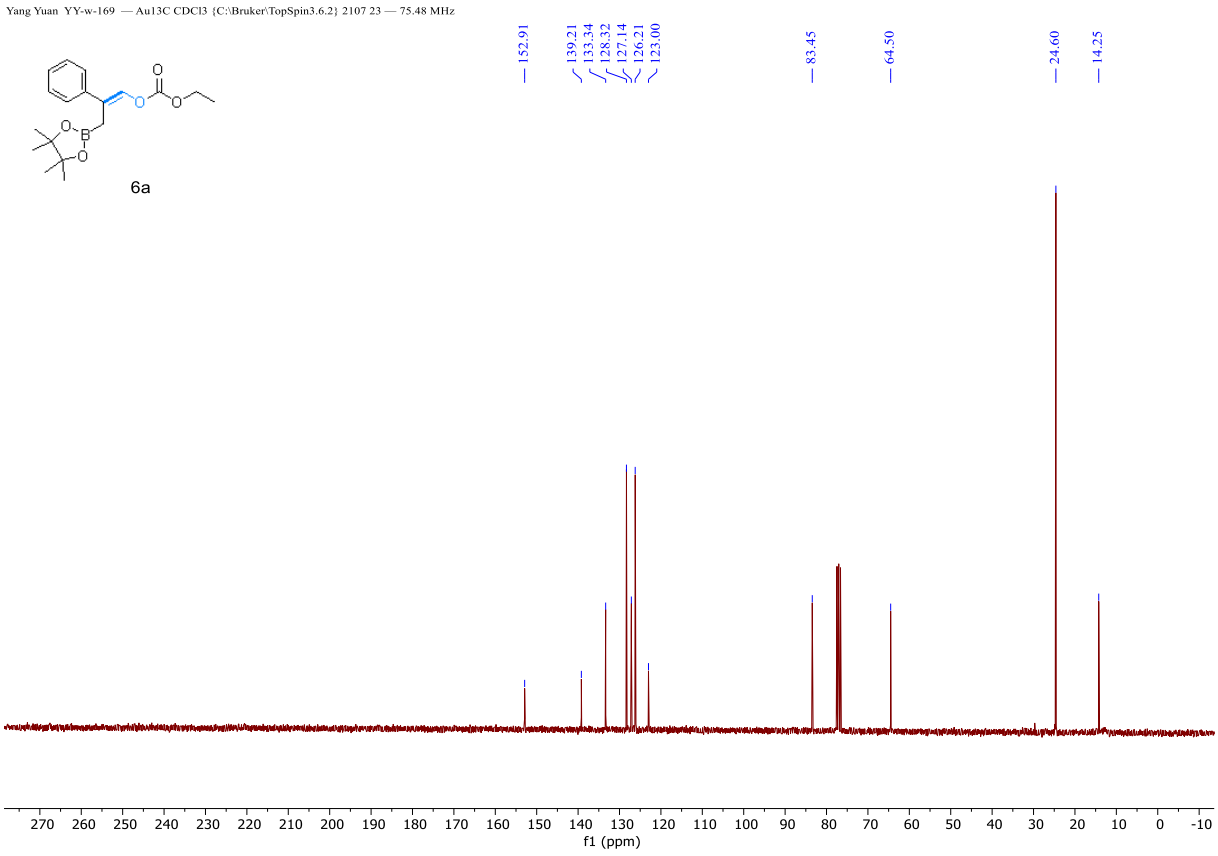

Yuan/ YY-W-144 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 1 — 96.32 MHz
32.34
4ag
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

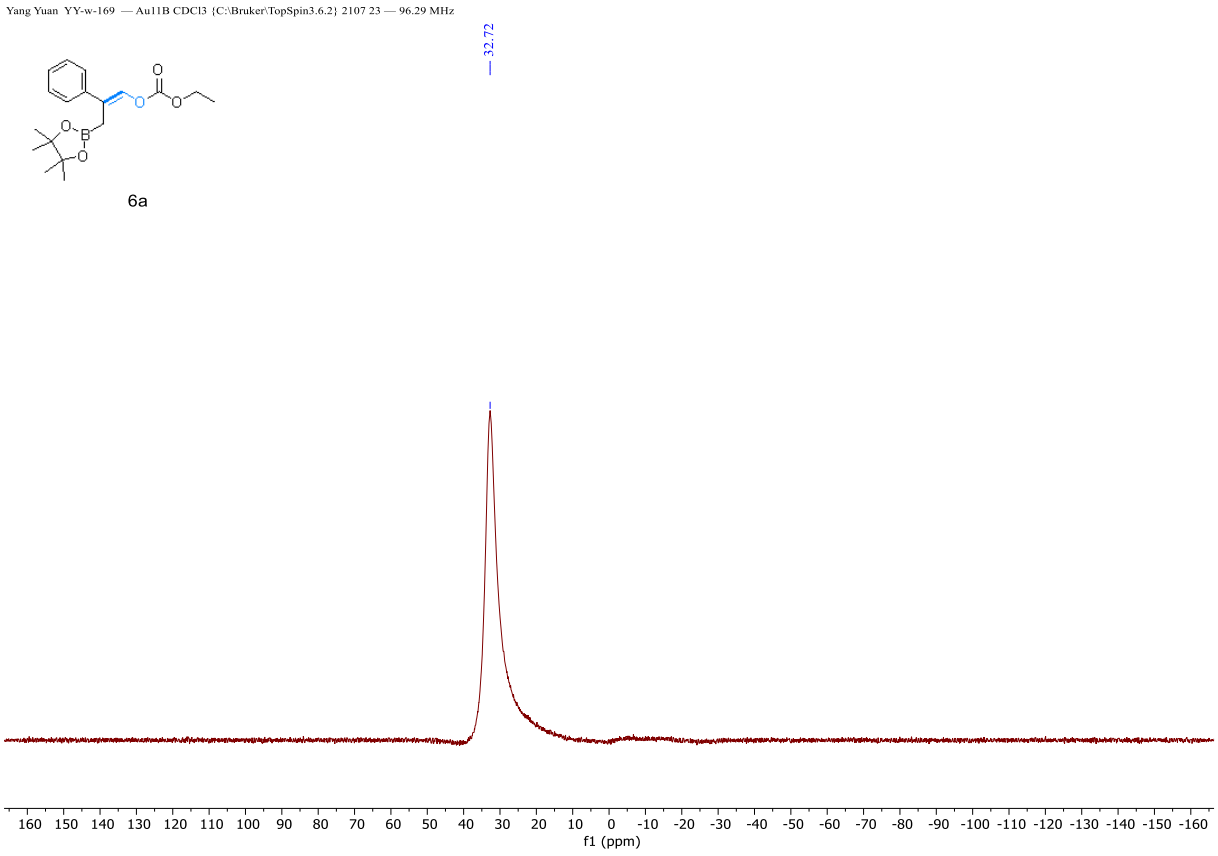

Yuan/ YY-W-145 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 2 — 300.20 MHz
4ah
f1 (ppm)


Yuan/ YY-W-145 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 2 — 75.49 MHz
4ah
f1 (ppm)

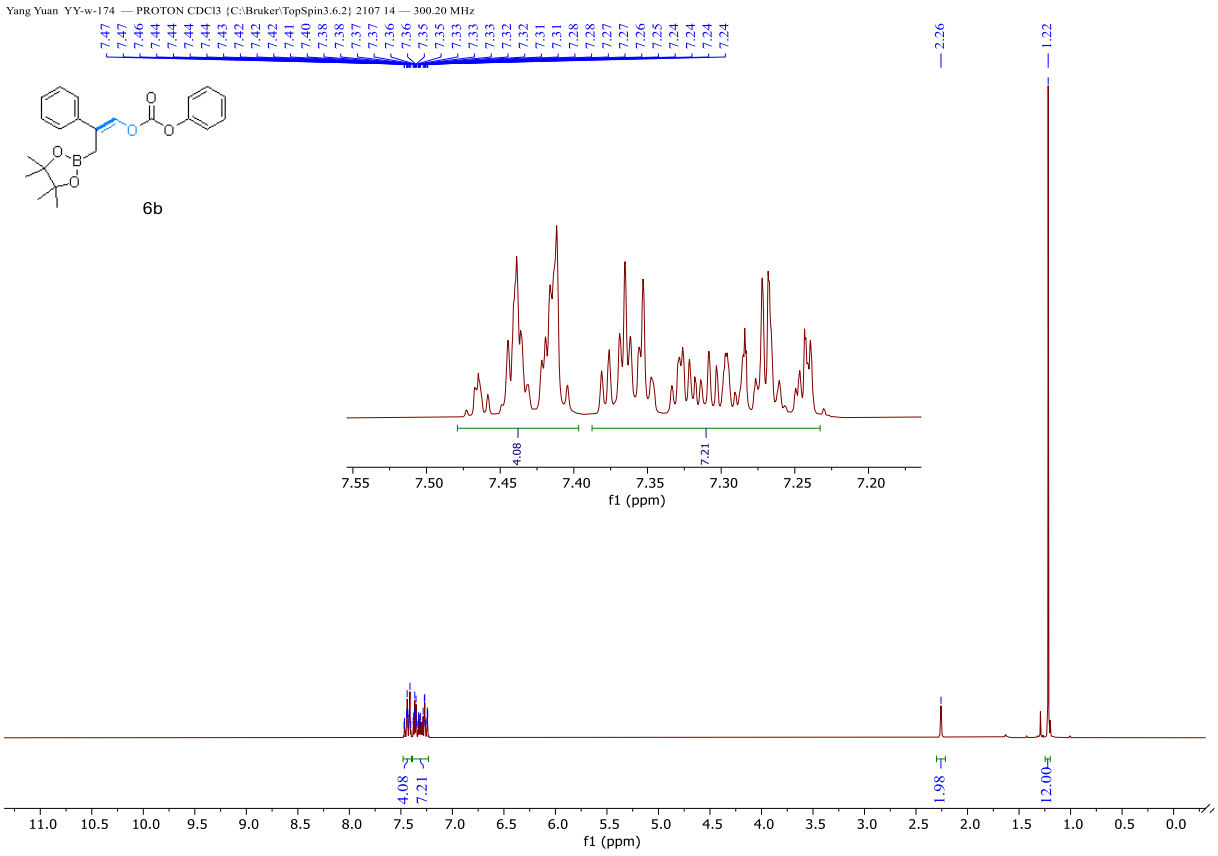

Yuan/ YY-W-145 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 2 — 96.32 MHz
30.83
4ah
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

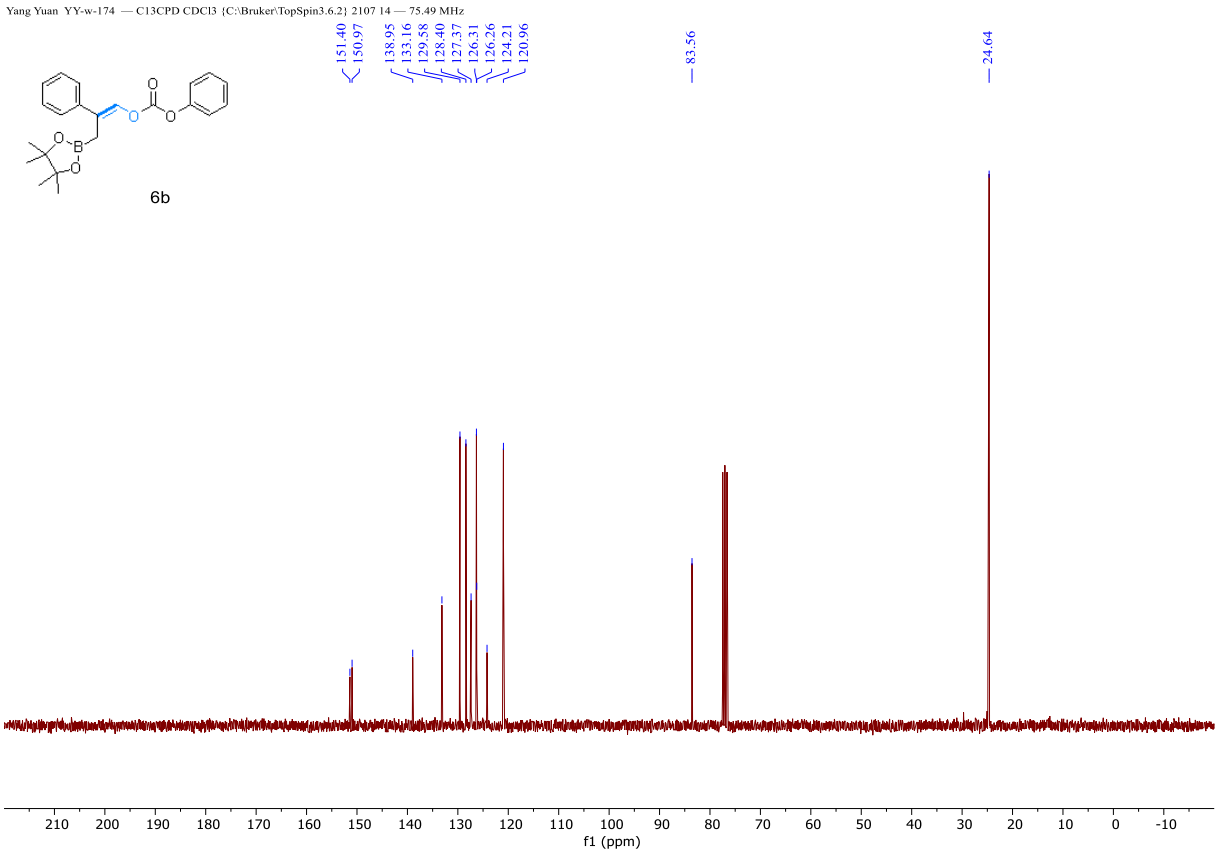

Yang Yuan YY-w-169 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 23 — 300.13 MHz
7.31 7.31 7.29 7.29 7.29 7.28 7.28 7.26 7.25 7.23 7.23 7.22 7.21 7.21 7.20 7.20 7.19 7.19 7.18 7.16
4.24 4.22 4.20 4.17
2.09 2.09
1.30 1.28 1.25 1.08
6a
6.06
7.42 7.40 7.38 7.36 7.34 7.32 7.30 7.28 7.26 7.24 7.22 7.20 7.18 7.16 7.14 7.12 7.10
f1 (ppm)
6.06 2.00 2.00 3.12 12.03
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

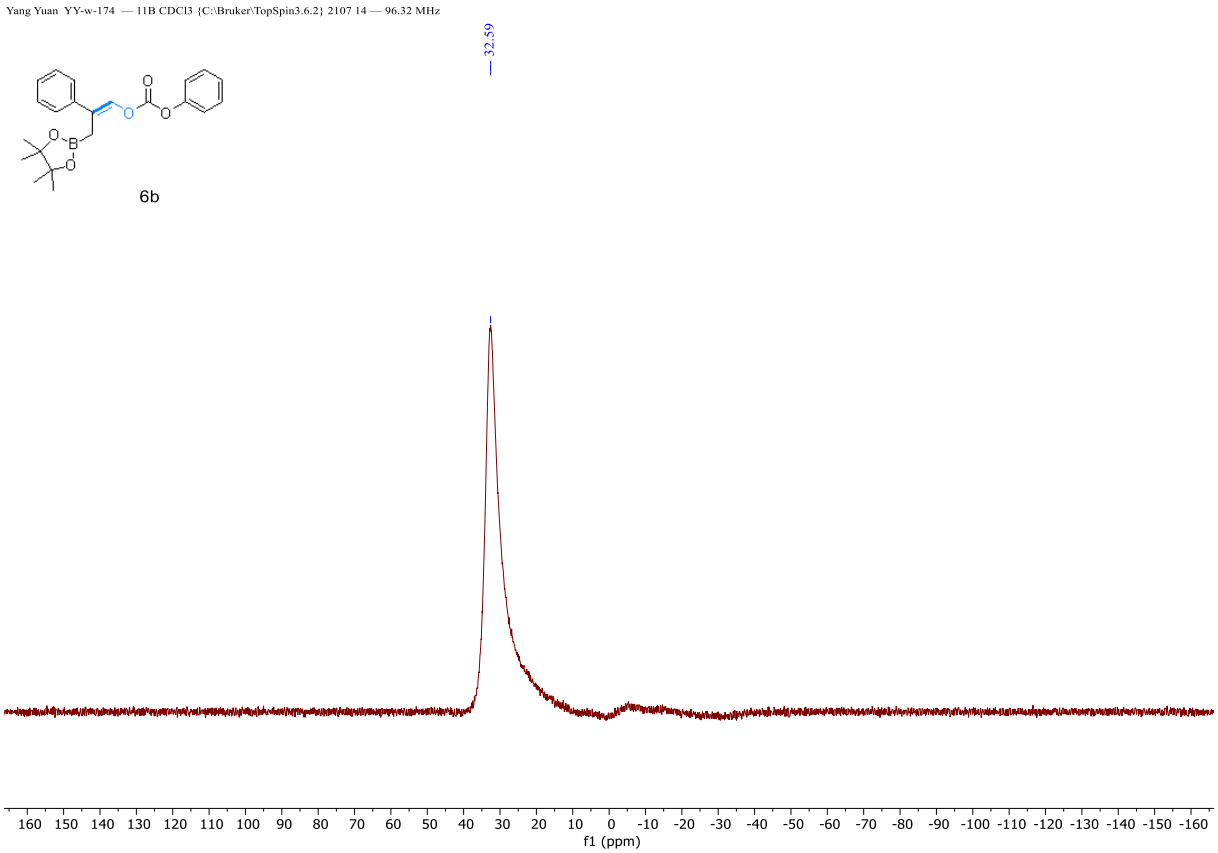

Yang Yuan YY-w-169 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 23 — 75.48 MHz
152.91 139.21 133.34 128.32 127.14 126.21 123.00
83.45
64.50
24.60
14.25
6a
270 260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

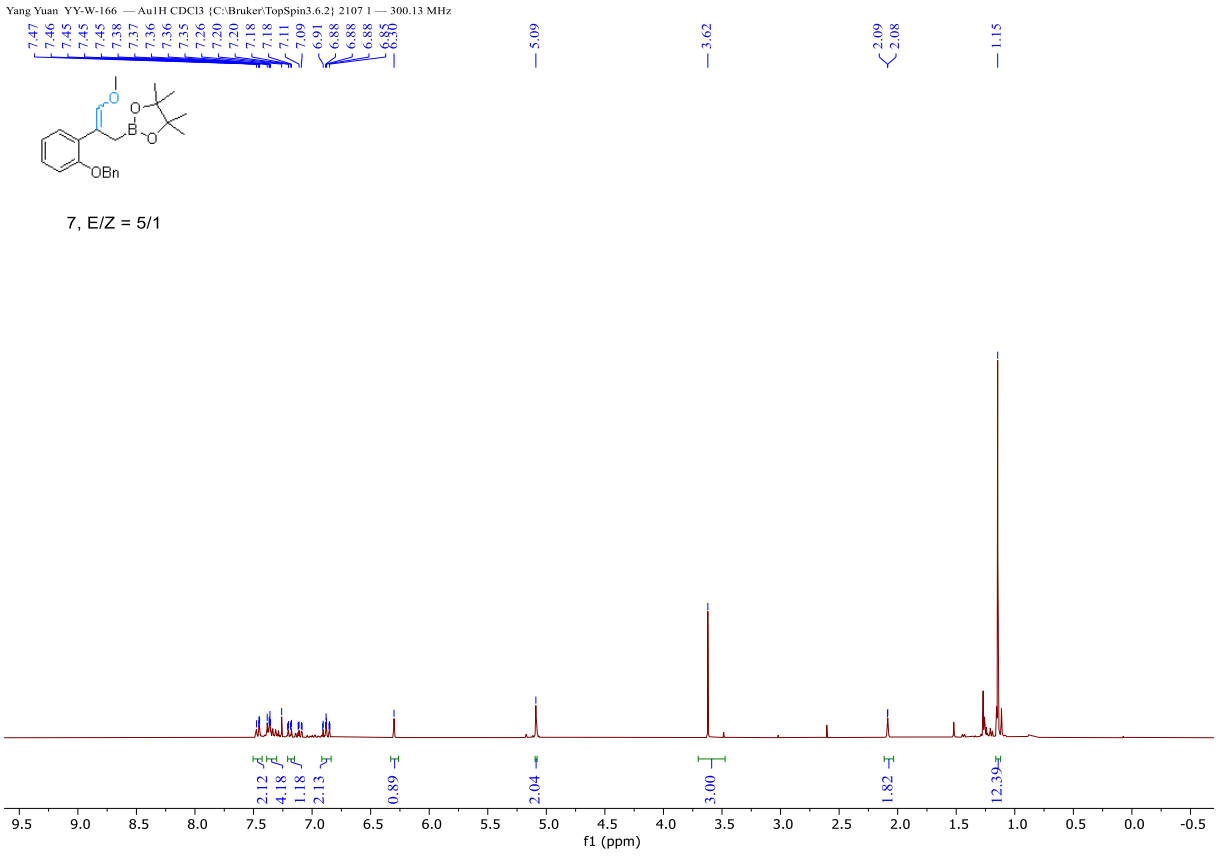

Yang Yuan YY-w-169 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 23 — 96.29 MHz
32.72
O
O
O
O
B
O
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

6a

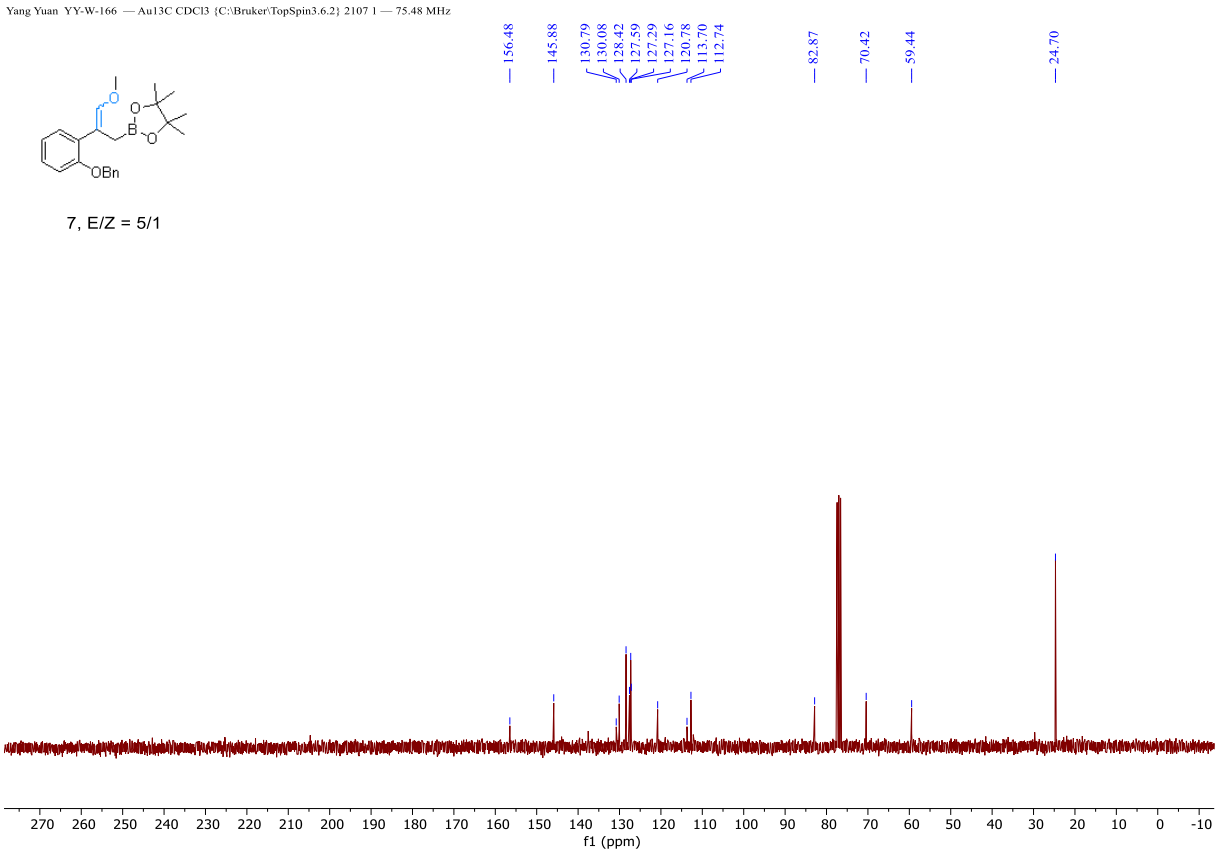

Yang Yuan YY-w-174 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 14 — 300.20 MHz
6b
f1 (ppm)


Yang Yuan YY-w-174 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 14 — 75.49 MHz
6b
f1 (ppm)

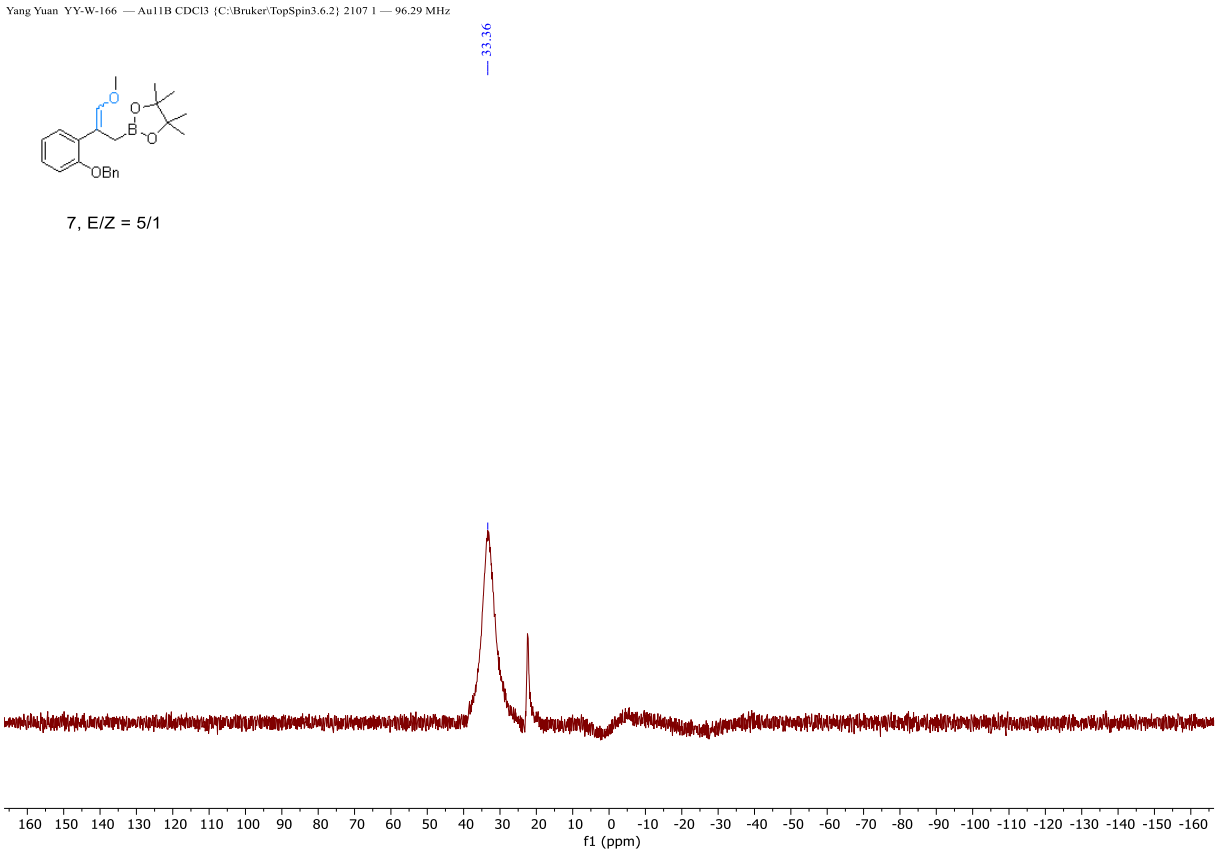

Yang Yuan YY-w-174 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 14 — 96.32 MHz
32.59
6b
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)


Yang Yuan YY-W-166 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 1 — 300.13 MHz
7.47
7.46
7.45
7.45
7.45
7.38
7.37
7.36
7.36
7.35
7.26
7.20
7.20
7.18
7.18
7.11
7.09
6.91
6.88
6.88
6.88
6.85
6.30
5.09
3.62
2.09
2.08
1.15
OBn
2.12
4.18
1.18
2.13
0.89
2.04
3.00
1.82
12.39
f1 (ppm)

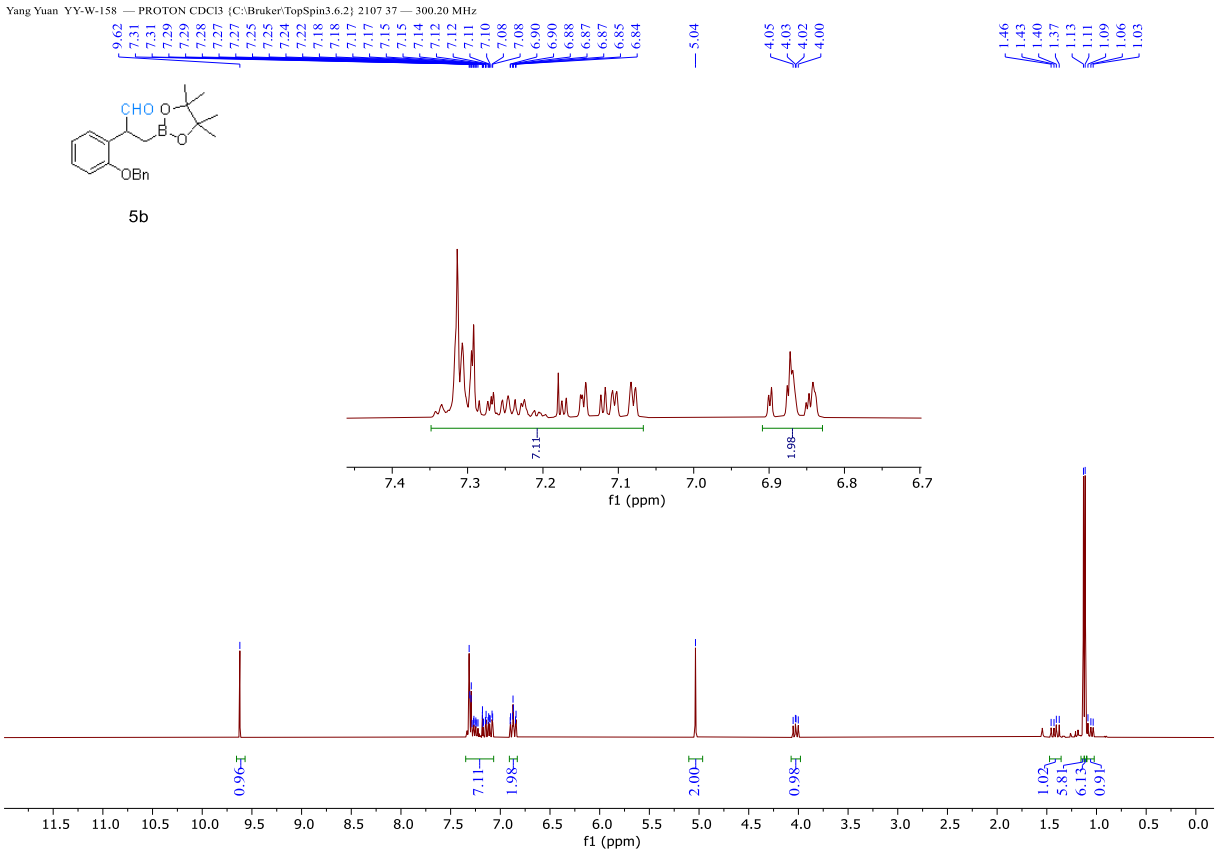
7, E/Z = 5/1

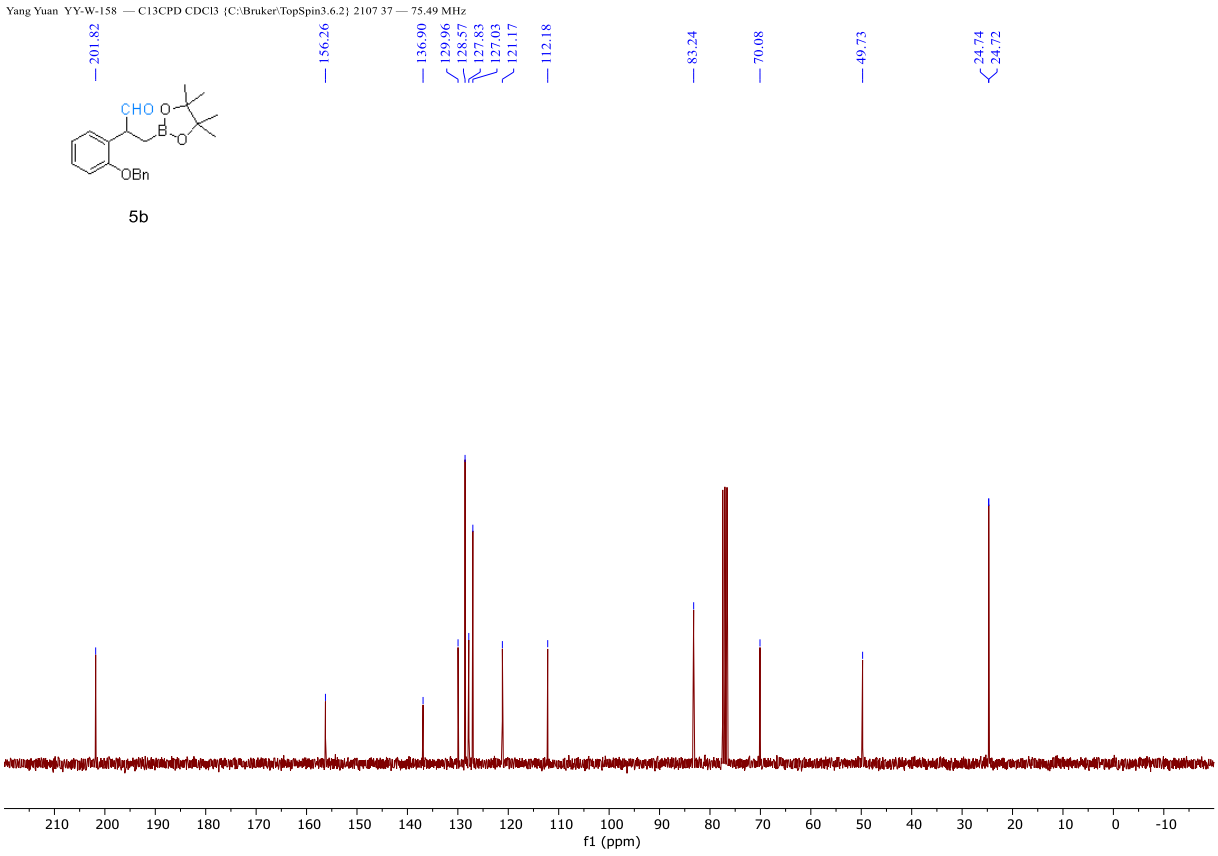

Yang Yuan YY-W-166 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 1 — 75.48 MHz
156.48
145.88
130.79
130.08
128.42
127.59
127.29
127.16
120.78
113.70
112.74
82.87
70.42
59.44
24.70
OBn
f1 (ppm)

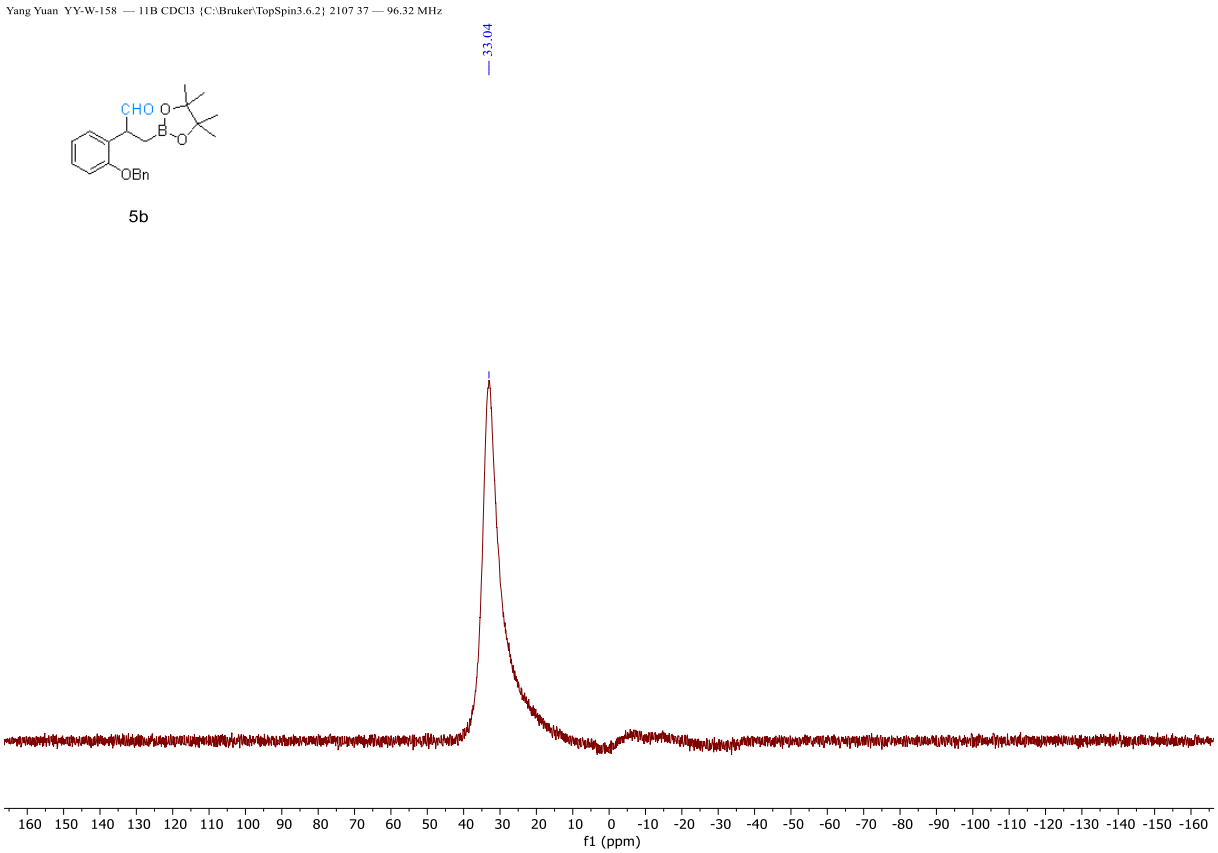
7, E/Z = 5/1


Yang Yuan YY-W-166 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 1 — 96.29 MHz
33.36
O
O
B
O
OBn
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

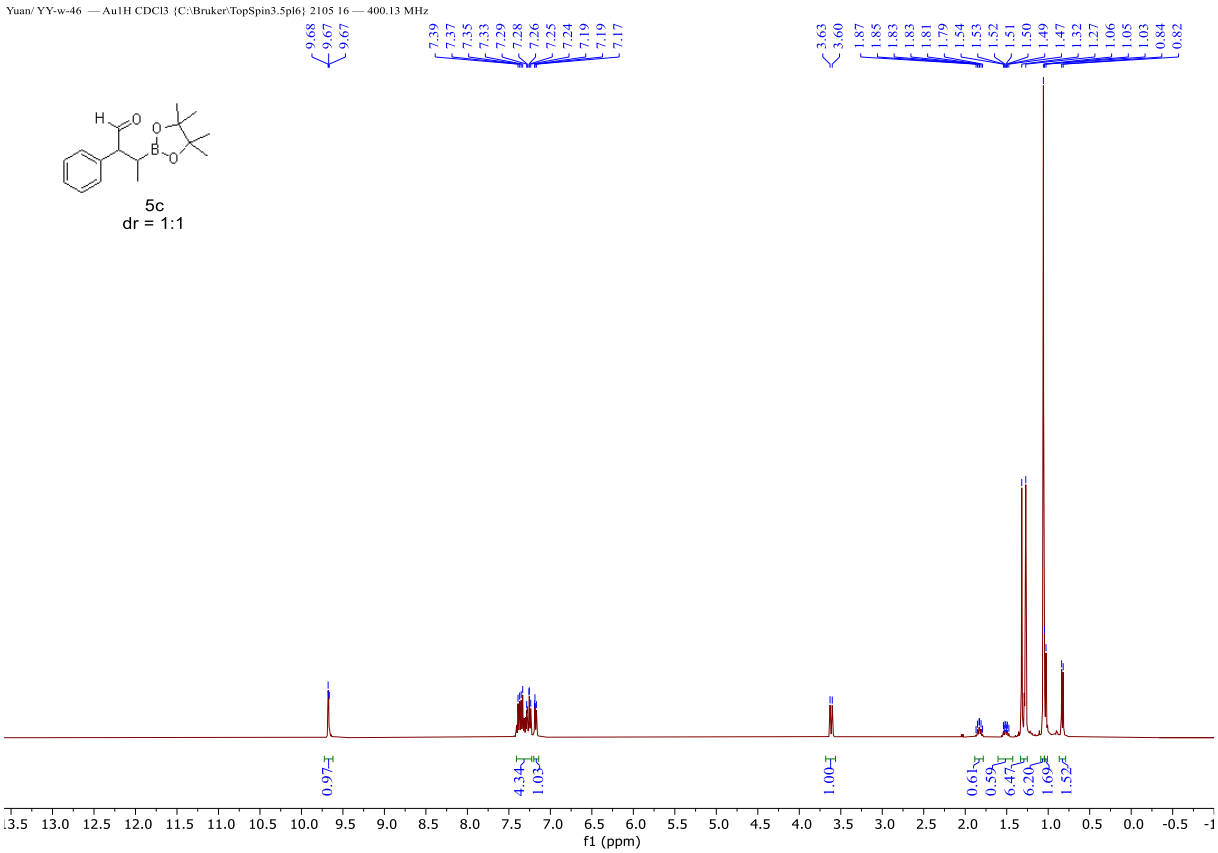
7, E/Z = 5/1

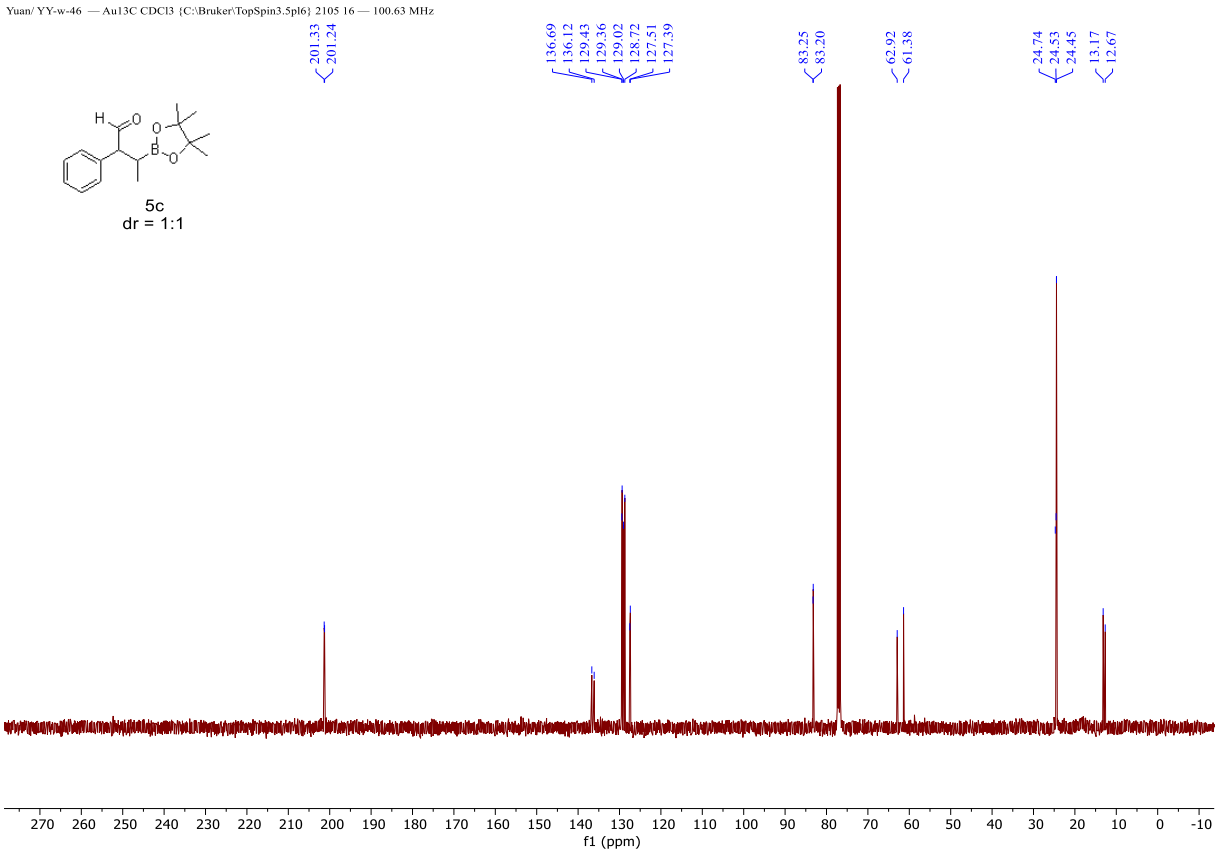

Yang Yuan YY-W-158 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 37 — 300.20 MHz
9.62 7.31 7.31 7.29 7.29 7.28 7.27 7.27 7.25 7.25 7.24 7.22 7.18 7.18 7.17 7.17 7.15 7.15 7.14 7.12 7.12 7.11 7.10 7.08 7.08 6.90 6.90 6.88 6.87 6.87 6.85 6.84
5.04
4.05 4.03 4.02 4.00
1.46 1.43 1.40 1.37 1.13 1.11 1.09 1.06 1.03
CHO
O
B
O
OBn
5b
7.11
1.98
7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7
f1 (ppm)
0.96 7.11 1.98 2.00 0.98 1.02 5.81 6.13 0.91
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

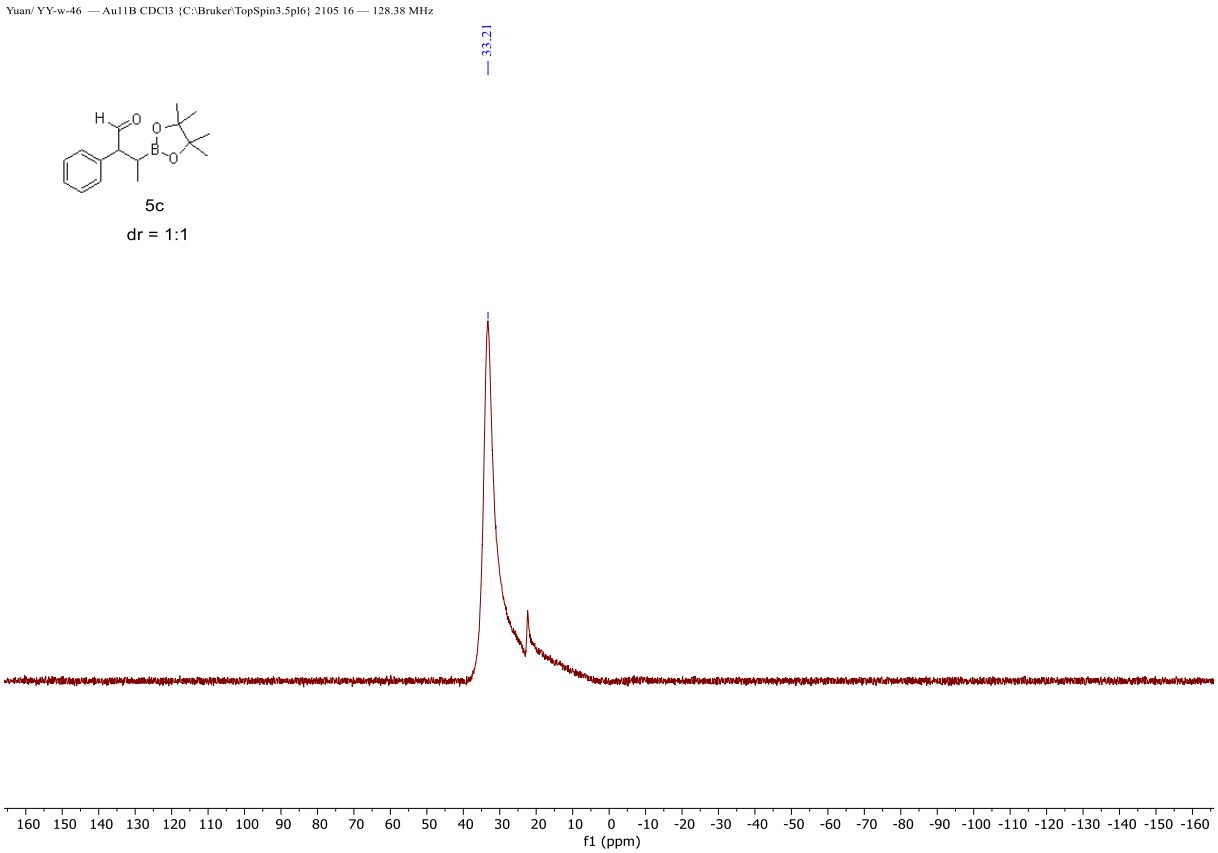

Yang Yuan YY-W-158 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 37 — 75.49 MHz
201.82
156.26
136.90
129.96 128.57 127.83 127.03 121.17
112.18
83.24
70.08
49.73
24.74 24.72
CHO
O
B
O
OBn
5b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Yang Yuan YY-W-158 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 37 — 96.32 MHz
33.04
CHO
O
B
O
OBn
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

5b


Yuan/ YY-w-46 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2105 16 — 400.13 MHz
9.68
9.67
9.67
7.39
7.37
7.35
7.33
7.29
7.28
7.26
7.25
7.24
7.19
7.19
7.17
3.63
3.60
1.87
1.85
1.83
1.83
1.81
1.79
1.54
1.53
1.52
1.51
1.50
1.49
1.47
1.32
1.27
1.06
1.05
1.03
0.84
0.82
5c
dr = 1:1
0.97
4.34
1.03
1.00
0.61
0.59
6.47
6.20
1.69
1.52
f1 (ppm)


Yuan/ YY-w-46 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2105 16 — 100.63 MHz
201.33
201.24
136.69
136.12
129.43
129.36
129.02
128.72
127.51
127.39
83.25
83.20
62.92
61.38
24.74
24.53
24.45
13.17
12.67
5c
dr = 1:1
f1 (ppm)


Yuan/ YY-w-46 — Au11B CDCl3 {C:\Bruker\TopSpin3.5pl6} 2105 16 — 128.38 MHz
33.21
5c
dr = 1:1
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)