Supporting Information

Cooperative Cu/Pd-Catalyzed Borocarbonylation of Ethylene

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Contents

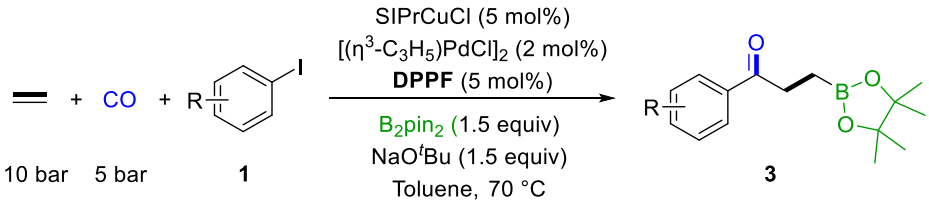
1. General information

Unless otherwise noted, all reactions were carried out under carbon monoxide (CO) or nitrogen atmosphere. All reagents were from commercial sources and used as received without further purification. All solvents were dried by standard techniques and distilled prior to use. Column chromatography was performed on silica gel (200-300 meshes) using petroleum ether (bp. 60~90 °C), dichloromethane and ethyl acetate as eluent. All NMR spectra were recorded at ambient temperature using Bruker Avance III 400 MHz NMR, Bruker Avance III HD 700 MHz NMR spectrometers. ^{1}H NMR chemical shifts are reported relative to TMS and were referenced via residual proton resonances of the corresponding deuterated solvent ($CDCl_3$: 7.26 ppm; d_6-DMSO: 2.50 ppm) whereas $^{13}C\{^{1}H\}$ NMR spectra are reported relative to TMS via the carbon signals of the deuterated solvent ($CDCl_3$: 77.0 ppm; d_6-DMSO: 39.5 ppm. Data for ^{1}H are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet, br = broad), coupling constant (Hz), and integration. All ^{13}C NMR spectra were broad-band 1H decoupled. All reactions were monitored by GC-FID or NMR analysis, GC-yields were calculated using hexadecane as internal standard. All measurements were carried out at room temperature unless otherwise stated. HRMS data was obtained with Micromass HPLC-Q-TOF mass spectrometer (ESI) or Agilent 6540 Accurate-MS spectrometer (Q-TOF).

Because of the high toxicity of carbon monoxide, all the reactions should be performed in an autoclave. The laboratory should be well-equipped with a CO detector and alarm system.

2. General Procedures

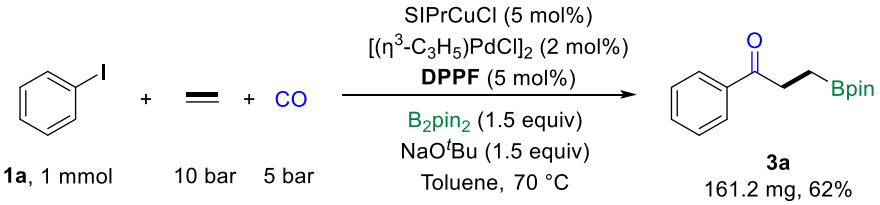
2.1 General Procedure



A vial (4 mL) was charged with DPPF (5.0 mol%), $[(\eta^3\text{-}C_3H_5)PdCl]_2$ (2.0 mol%), SIPrCuCl (5.0 mol%), B_2pin_2 (76.2 mg, 1.5 equiv), NaO^{t}Bu (28.8 mg, 1.5 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 **1** (0.2 mmol, 1.0 equiv) was added, and 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 5 bar of CO and 10 bar of ethylene were adjusted at ambient temperature. Then, the reaction was performed for 24 h at 70 °C. After the reaction was complete, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. The reaction was diluted with EA (ethyl acetate) and filtered through a pad of silica gel (a pipette with about 3 cm silica gel). The filtrate was concentrated under reduced pressure and the residue was directly purified by column chromatography to afford the corresponding products **3**.

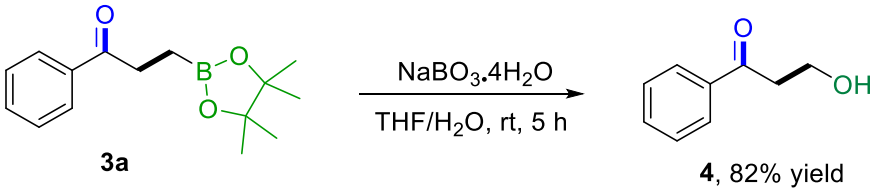
Note: Column chromatography should be performed quickly to prevent product decomposition on silica. (We typically aim to complete the column in 10-15 min for a reaction on this scale.)

2.2 Procedure for 1.0 mmol scale reaction



A vial (4 mL) was charged with DPPF (5.0 mol%), $[(\eta^3\text{-}C_3H_5)PdCl]_2$ (2.0 mol%), SIPrCuCl (5.0 mol%), B_2pin_2 (381 mg, 1.5 equiv), NaO^{t}Bu (144 mg, 1.5 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 **1** (1 mmol, 1.0 equiv) was added, and 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 5 bar of CO and 10 bar of ethylene were adjusted at ambient temperature. Then, the reaction was performed for 24 h at 70 °C. After the reaction was complete, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. The reaction was diluted with EA (ethyl acetate) and filtered through a pad of silica gel. The filtrate was concentrated under reduced pressure and the residue was directly purified by column chromatography to afford the corresponding products **3** as a slight yellow oil in 62% yield (161.2 mg).

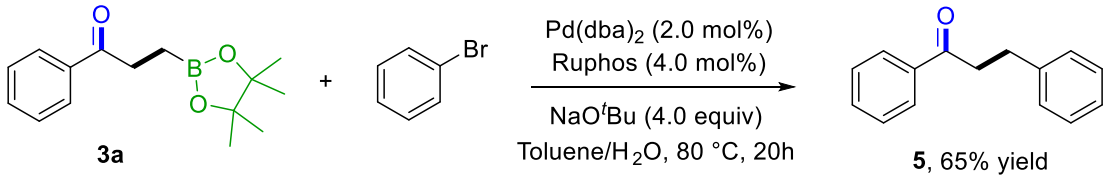
2.3 Procedure for Compound 4



A Schlenk tube with a magnetic stir bar was charged with **3a** (26.1 mg, 0.1 mmol, 1.0 equiv), $NaBO_3.4H_2O$ (101 mg, 0.4 mmol, 4.0 equiv), then THF (0.5 mL) and H_2O (0.5 mL) were added. The reaction allowed to stir at room temperature for 5 h and

extracted with EtOAc (3 x 2 mL). The combined organic phase was dried over Na_2SO_4 and concentrated. The residue was purified by flash chromatography (PE/EA= 5/1) to give **4** as a colorless oil (12.3 mg, 82% yield). **^{1}H NMR** (700 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.0 Hz, 2H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 2H), 4.03 (t, *J* = 5.3 Hz, 2H), 3.23 (t, *J* = 5.4 Hz, 2H), 2.74 (s, 1H). **^{13}C NMR** (176 MHz, $CDCl_3$) δ 200.5, 136.7, 133.6, 128.7, 128.1, 58.1, 40.4. **HRMS** (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_9H_{11}O$ 151.0754; Found: 151.0749.

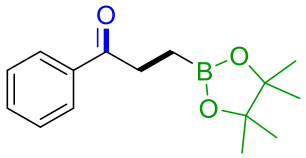
2.4 Procedure for Compound 5



Compound **3a** (39 mg, 0.15 mmol, 1.0 equiv), bromobenzene (28.3 mg, 0.18 mmol, 1.2 equiv), NaO^{t}Bu (57.6 mg, 0.6 mmol, 4.0 equiv), $Pd(dba)_2$ (2.0 mol%) Ruphos (4.0 mol%), toluene (0.5 mL) and H_2O (50 uL) were added to a Schlenk flask. The mixture was degassed with N_2 three times. Then the reaction was heated at 80 °C for 20 h. After the reaction mixture was cooled to room temperature, EA (10 mL) and H_2O (5 mL) were added, and the organic layer was separated. The aqueous layer was extracted with Et_2O (10 mL × 2). The combined organic phases were washed with brine, and dried over anhydrous Na_2SO_4. The residue was purified by flash chromatography (PE/EA= 50/1) to give **5** as a white solid (20.5 mg, 65% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.06 – 7.87 (m, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.34 – 7.12 (m, 5H), 3.39 – 3.20 (m, 2H), 3.15 – 2.96 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.3, 141.3, 136.9, 133.1, 128.6, 128.6, 128.4, 128.1, 126.2, 40.5, 30.2. **HRMS** (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{15}H_{15}O$ 211.1117; Found: 211.1109.

4. Characterization Data

Note: the carbon directly attached to the boron atom was not detected due to quadrupolar relaxation.



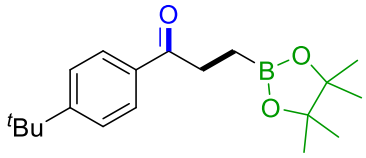
1-Phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3a)

35.8 mg, 69% yield, slight yellow oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.5 Hz, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 2H), 3.15 (t, *J* = 7.0 Hz, 2H), 1.25 (s, 12H), 1.08 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 200.6, 137.0, 132.8, 128.5, 128.0, 83.1, 33.7, 24.8.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{15}H_{22}BO_3$ 261.1665; Found: 261.1664.



1-(4-(*tert*-Butyl)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3b)

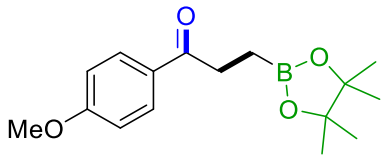
45.5 mg, 72% yield, slight yellow oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.5 Hz, 2H), 7.38 (d, *J* = 8.5 Hz, 2H), 3.06 (t, *J* = 7.0 Hz, 2H), 1.26 (s, 9H), 1.18 (s, 12H), 0.99 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 199.2, 155.3, 133.40, 126.9, 124.4, 82.0, 34.0, 32.6, 30.1, 23.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.21.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{19}H_{30}BO_3$ 317.2291; Found: 317.2286.



1-(4-Methoxyphenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3c)

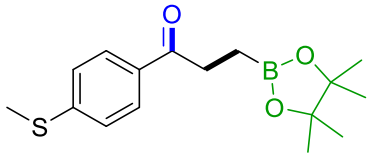
38.9 mg, 67% yield, slight yellow oil. Eluent: pentane/ethyl acetate = 15/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.5 Hz, 2H), 6.92 (d, *J* = 8.5 Hz, 2H), 3.86 (s, 3H), 3.10 (t, *J* = 7.0 Hz, 2H), 1.25 (s, 12H), 1.06 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 199.1, 163.2, 130.2, 113.6, 83.1, 55.4, 33.3, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.30.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{24}BO_4$ 291.1771; Found: 291.1772.



1-(4-(Methylthio)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3d)

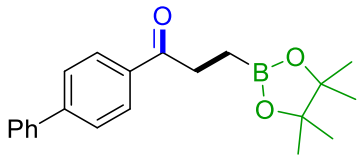
38.6 mg, 63% yield, colorless sticky oil. Eluent: pentane/ethyl acetate = 8/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.5 Hz, 2H), 7.25 (d, *J* = 8.5 Hz, 2H), 3.11 (t, *J* = 7.0 Hz, 2H), 2.51 (s, 3H), 1.25 (s, 12H), 1.06 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 199.6, 145.3, 133.3, 128.4, 124.9, 83.1, 33.4, 25.0, 24.8, 14.9.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.53.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{24}BO_3S$ 307.1542; Found: 307.1549.



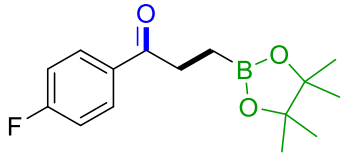
1-([1,1'-Biphenyl]-4-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3e)

51.8 mg, 77% yield, pale yellow solid. Eluent: pentane/ethyl acetate = 15/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.5 Hz, 2H), 7.66 (d, *J* = 7.5 Hz, 2H), 7.62 (d, *J* = 7.5 Hz, 2H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.39 (t, *J* = 7.5 Hz, 1H), 3.18 (t, *J* = 7.0 Hz, 2H), 1.26 (s, 12H), 1.10 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 200.2, 145.4, 140.0, 135.7, 128.9, 128.6, 128.1, 127.3, 127.1, 83.1, 33.8, 25.1, 24.8.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{21}H_{26}BO_3$ 337.1979; Found: 337.1985.



1-(4-Fluorophenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3f)

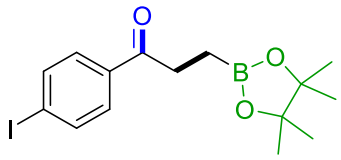
33.9 mg, 61% yield, colorless oil. Eluent: pentane/ethyl acetate = 10/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 8.06 – 7.94 (m, 2H), 7.11 (t, *J* = 8.5 Hz, 2H), 3.12 (t, *J* = 6.9 Hz, 2H), 1.25 (s, 12H), 1.07 (t, *J* = 6.9 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 199.0, 165.6 (d, *J* = 253.9 Hz), 133.4, 130.6 (d, *J* = 9.2 Hz), 115.5 (d, *J* = 21.8 Hz), 83.2, 33.6, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.10.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{15}H_{21}BO_3F$ 279.1571; Found: 279.1570.



1-(4-Iodophenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3g)

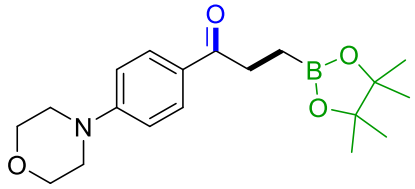
37.0 mg, 48% yield, yellow oil. Eluent: pentane/ethyl acetate = 15/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.5 Hz, 2H), 7.68 (d, *J* = 8.5 Hz, 2H), 3.10 (t, *J* = 7.0 Hz, 2H), 1.28 – 1.23 (m, 12H), 1.07 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 199.9, 137.8, 136.2, 129.5, 100.6, 83.2, 33.6, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 33.72.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{15}H_{221}BO_3I$ 386.0631; Found: 386.0627.



1-(4-Morpholinophenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3h)

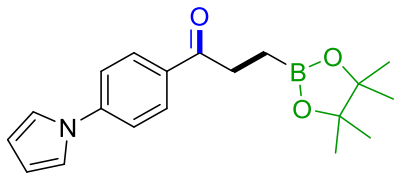
32.5 mg, 47% yield, white solid. Eluent: pentane/ethyl acetate = 3/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.5 Hz, 2H), 3.94 – 3.76 (m, 4H), 3.37 – 3.23 (m, 4H), 3.07 (t, *J* = 7.0 Hz, 2H), 1.25 (s, 12H), 1.04 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 198.9, 154.1, 129.9, 127.9, 113.4, 83.0, 66.6, 47.7, 33.1, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.87.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{19}H_{29}BNO_4$ 346.2193; Found: 346.2198.



1-(4-(1H-Pyrrol-1-yl)phenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3i)

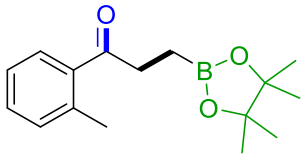
38.4 mg, 59% yield, slight yellow oil. Eluent: pentane/ethyl acetate = 5/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.5 Hz, 2H), 7.44 (d, *J* = 8.5 Hz, 2H), 7.17 (d, *J* = 20.5 Hz, 2H), 6.39 (d, *J* = 15.5 Hz, 2H), 3.15 (t, *J* = 7.0 Hz, 2H), 1.26 (s, 12H), 1.09 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 199.2, 143.8, 133.9, 129.8, 119.4, 119.0, 111.5, 83.2, 33.6, 25.0, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.16.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{19}H_{25}BNO_3$ 326.1931; Found: 326.1930.



3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(o-tolyl)propan-1-one (3j)

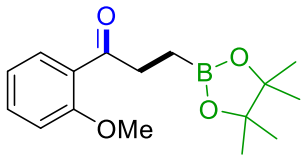
38.9 mg, 71% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.5 Hz, 1H), 7.26 (t, *J* = 7.5 Hz, 1H), 7.16 (dd, *J* = 13.5, 7.5 Hz, 2H), 2.98 (t, *J* = 7.0 Hz, 2H), 2.40 (s, 3H), 1.18 (s, 12H), 0.98 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 204.9, 138.3, 137.7, 131.7, 130.9, 128.2, 125.6, 83.1, 36.7, 25.0, 24.8, 21.1.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.12.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{24}BO_3$ 275.1821; Found: 275.1825.



1-(2-Methoxyphenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3k)

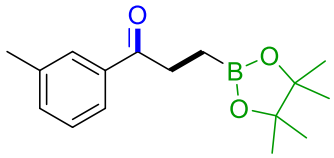
40.6 mg, 70% yield, slight yellow oil. Eluent: pentane/ethyl acetate = 15/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.69 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.48 – 7.37 (m, 1H), 7.09 – 6.87 (m, 2H), 3.88 (s, 3H), 3.13 (t, *J* = 7.0 Hz, 2H), 1.25 (s, 12H), 1.00 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 202.9, 158.6, 133.1, 130.3, 128.3, 120.5, 111.5, 83.0, 55.5, 39.0, 25.0, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 33.99.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{24}BO_4$ 291.1771; Found: 291.1779.



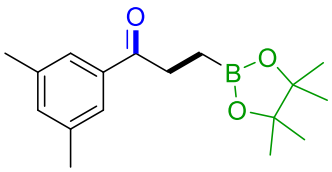
3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(m-tolyl)propan-1-one (3m)

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

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.78 – 7.60 (m, 2H), 7.33 – 7.22 (m, 2H), 3.06 (t, *J* = 7.0 Hz, 2H), 2.33 (s, 3H), 1.18 (s, 12H), 0.99 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 200.8, 138.2, 137.0, 133.5, 128.5, 128.3, 125.2, 83.1, 33.7, 25.0, 24.8, 21.4.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{24}BO_3$ 275.1821; Found: 275.1821.



1-(3,5-Dimethylphenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3n)

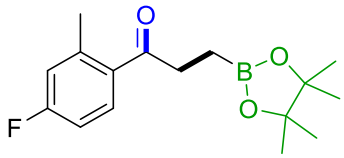
36.9 mg, 64% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.57 (s, 2H), 7.17 (s, 1H), 3.12 (t, *J* = 7.0 Hz, 2H), 2.36 (s, 6H), 1.26 (s, 12H), 1.06 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 201.0, 138.0, 137.1, 134.4, 125.8, 83.1, 33.8, 24.8, 21.2.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.30.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{17}H_{26}BO_3$ 289.1978; Found: 289.1972.



1-(4-Fluoro-2-methylphenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3o)

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

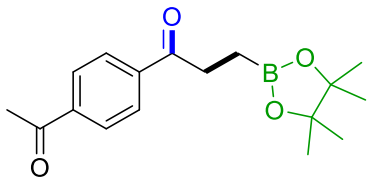
^{1}H NMR (700 MHz, $CDCl_3$) δ 7.31 (d, *J* = 9.0 Hz, 1H), 7.19 – 7.14 (m, 1H), 7.03 (t, *J* = 8.0 Hz, 1H), 3.00 (t, *J* = 7.0 Hz, 2H), 2.41 (s, 3H), 1.24 (s, 12H), 1.09 – 1.01 (m, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 203.8 (d, *J* = 2.5 Hz), 161.3, 159.9, 139.5 (d, *J* = 5.8 Hz), 133.1 (d, *J* = 7.6 Hz), 117.6 (d, *J* = 20.8 Hz), 114.9 (d, *J* = 22.3 Hz), 83.2, 36.7, 24.8, 20.2.

^{19}F NMR (376 MHz, $CDCl_3$) δ -117.15.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.15.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{23}BO_3F$ 293.1727; Found: 293.1725.



1-(4-Acetylphenyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3p)

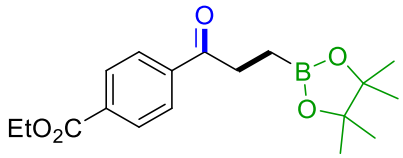
36.2 mg, 60% yield, slight yellow solid. Eluent: pentane/ethyl acetate = 6/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 8.02 (q, *J* = 8.5 Hz, 4H), 3.16 (t, *J* = 7.0 Hz, 2H), 2.63 (s, 3H), 1.24 (s, 12H), 1.09 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 200.1, 197.6, 140.2, 139.9, 128.4, 128.2, 83.2, 34.1, 26.9, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.26.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{17}H_{24}BO_4$ 303.1771; Found: 303.1773.



Ethyl 4-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propanoyl)benzoate (3q)

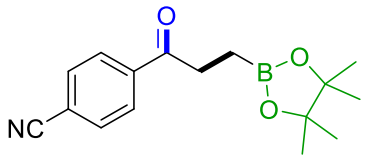
39.9 mg, 60% yield, slight yellow sticky oil. Eluent: pentane/ethyl acetate = 6/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.5 Hz, 2H), 8.00 (d, *J* = 8.5 Hz, 2H), 4.40 (q, *J* = 7.0 Hz, 2H), 3.16 (t, *J* = 7.0 Hz, 2H), 1.40 (t, *J* = 7.0 Hz, 3H), 1.24 (s, 12H), 1.09 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 200.2, 165.9, 140.1, 134.0, 129.7, 127.9, 83.2, 61.4, 34.1, 24.8, 14.3.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.06.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{18}H_{26}BO_5$ 333.1891; Found: 333.1897.



4-(3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)propanoyl)benzonitrile (3r)

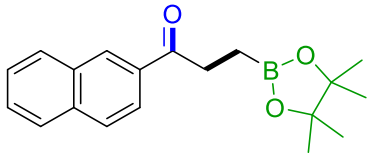
35.4 mg, 62% yield, light yellow oil. Eluent: pentane/ethyl acetate = 8/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.5 Hz, 2H), 7.76 (d, *J* = 7.5 Hz, 1H), 3.15 (t, *J* = 7.0 Hz, 2H), 1.25 (s, 12H), 1.10 (t, *J* = 6.5 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 199.3, 140.0, 132.4, 128.4, 118.1, 116.1, 83.3, 34.1, 25.0, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 33.88.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{21}BNO_3$ 286.1614; Found: 286.1617.



1-(Naphthalen-2-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3s)

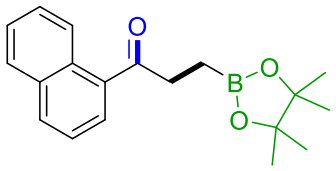
40.9 mg, 66% yield, slight yellow oil. Eluent: pentane/ethyl acetate = 10/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 8.49 (s, 1H), 8.04 (dd, *J* = 8.5, 1.5 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 1H), 7.87 (t, *J* = 8.5 Hz, 2H), 7.62 – 7.51 (m, 2H), 3.29 (t, *J* = 7.0 Hz, 2H), 1.27 (s, 12H), 1.14 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 200.6, 135.5, 134.3, 132.6, 129.5, 129.5, 128.3, 128.2, 127.8, 126.7, 124.0, 83.2, 33.7, 25.0, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.89.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{19}H_{24}BO_3$ 311.1822; Found: 311.1826.



1-(Naphthalen-1-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3t)

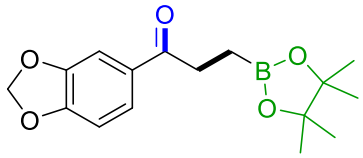
38.4 mg, 62% yield, slight yellow oil. Eluent: pentane/ethyl acetate = 10/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 8.56 (d, *J* = 8.5 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 1H), 7.85 (dd, *J* = 7.5, 3.5 Hz, 2H), 7.61 – 7.43 (m, 3H), 3.22 (t, *J* = 7.0 Hz, 2H), 1.28 (s, 12H), 1.15 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 205.3, 136.5, 133.9, 132.0, 130.1, 128.3, 127.6, 126.9, 126.3, 125.9, 124.4, 83.2, 37.4, 24.9.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.23.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{19}H_{24}BO_3$ 311.1822; Found: 311.1824.



1-(Benzo[d][1,3]dioxol-5-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3u)

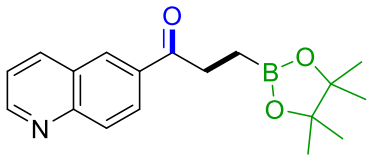
35.3 mg, 58% yield, light yellow oil. Eluent: pentane/ethyl acetate = 8/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.0 Hz, 1H), 7.44 (s, 1H), 6.83 (d, *J* = 8.0 Hz, 1H), 6.02 (s, 2H), 3.07 (t, *J* = 7.0 Hz, 2H), 1.25 (s, 12H), 1.05 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 198.6, 151.4, 148.0, 131.8, 124.1, 107.9, 107.8, 101.7, 83.1, 33.4, 25.0, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.17.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{22}BO_5$ 305.1563; Found: 305.1568.



1-(Quinolin-6-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (3v)

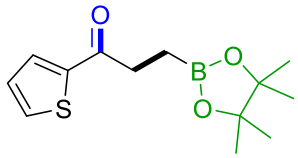
43.5 mg, 70% yield, brown solid. Eluent: pentane/ethyl acetate = 2/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 9.09 – 8.93 (m, 1H), 8.47 (s, 1H), 8.28 (d, *J* = 8.5 Hz, 2H), 8.15 (d, *J* = 9.0 Hz, 1H), 7.47 (dd, *J* = 8.0, 4.0 Hz, 1H), 3.30 (t, *J* = 7.0 Hz, 2H), 1.27 (s, 12H), 1.16 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 199.9, 152.4, 150.0, 137.5, 134.7, 129.7, 129.2, 127.8, 127.5, 121.8, 83.2, 33.9, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.28.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{18}H_{23}BO_3N$ 312.1774; Found: 312.1777.



3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(thiophen-2-yl)propan-1-one (3w)

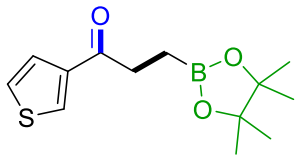
39.4 mg, 74% yield, yellow oil. Eluent: pentane/ethyl acetate = 10/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.73 (d, *J* = 3.5 Hz, 1H), 7.59 (d, *J* = 5.0 Hz, 1H), 7.16 – 7.06 (m, 1H), 3.09 (t, *J* = 7.0 Hz, 2H), 1.25 (s, 12H), 1.09 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 193.6, 144.1, 132.9, 131.5, 127.9, 83.2, 34.2, 25.0, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.24.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{13}H_{20}BO_3S$ 267.1229; Found: 267.1224.



3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(thiophen-3-yl)propan-1-one (3x)

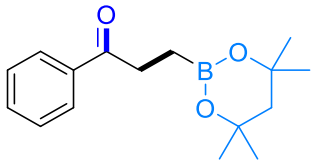
40.5 mg, 76% yield, yellow oil. Eluent: pentane/ethyl acetate = 10/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 8.05 (d, *J* = 2.5 Hz, 1H), 7.55 (d, *J* = 5.0 Hz, 1H), 7.29 (dd, *J* = 5.0, 3.0 Hz, 1H), 3.06 (t, *J* = 7.0 Hz, 2H), 1.25 (s, 12H), 1.06 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 195.0, 142.1, 131.5, 127.0, 126.0, 83.1, 34.8, 25.0, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$) δ 34.17.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{13}H_{20}BO_3S$ 267.1229; Found: 267.1227.



1-Phenyl-3-(4,4,6,6-tetramethyl-1,3,2-dioxaborinan-2-yl)propan-1-one (3y)

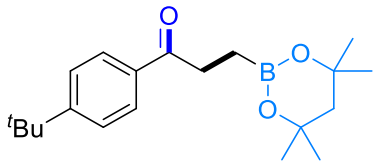
31.8 mg, 58% yield, colorless oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 8.10 – 7.83 (m, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 2H), 3.07 (t, *J* = 7.0 Hz, 1H), 1.80 (s, 2H), 1.29 (s, 12H), 1.00 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 201.7, 137.5, 132.5, 128.4, 128.0, 70.3, 48.8, 33.8, 31.67.

^{11}B NMR (128 MHz, $CDCl_3$) δ 30.26.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{24}BO_3$ 275.1822; Found: 275.1828.



1-(4-(Tert-butyl)phenyl)-3-(4,4,6,6-tetramethyl-1,3,2-dioxaborinan-2-yl)propan-1-one (3z)

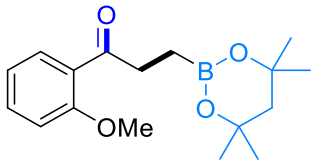
45.5 mg, 69% yield, yellow oil. Eluent: pentane/ethyl acetate = 20/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.0 Hz, 2H), 7.45 (d, *J* = 8.0 Hz, 2H), 3.05 (t, *J* = 7.0 Hz, 2H), 1.80 (s, 2H), 1.30 (s, 12H), 0.98 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 201.3, 156.0, 134.9, 127.9, 125.3, 70.3, 48.8, 35.0, 33.8, 31.7, 31.1.

^{11}B NMR (128 MHz, $CDCl_3$) δ 30.12.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{20}H_{32}BO_3$ 331.2448; Found: 311.2453.



1-(2-Methoxyphenyl)-3-(4,4,6,6-tetramethyl-1,3,2-dioxaborinan-2-yl)propan-1-one (3aa)

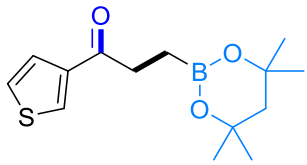
41.4 mg, 68% yield, white solid. Eluent: pentane/ethyl acetate = 15/1.

^{1}H NMR (700 MHz, $CDCl_3$) δ 7.63 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.47 – 7.34 (m, 1H), 7.02 – 6.87 (m, 2H), 3.88 (s, 3H), 3.06 (t, *J* = 7.0 Hz, 2H), 1.80 (s, 2H), 1.33 (s, 12H), 0.93 (t, *J* = 6.9 Hz, 2H).

^{13}C NMR (176 MHz, $CDCl_3$) δ 204.3, 158.2, 132.6, 123.0, 120.4, 111.5, 70.2, 69.4, 55.5, 49.5, 39.1, 31.9, 31.7.

^{11}B NMR (128 MHz, $CDCl_3$) δ 30.29.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{17}H_{26}BO_4$ 305.1927; Found: 305.1921.



3-(4,4,6,6-Tetramethyl-1,3,2-dioxaborinan-2-yl)-1-(thiophen-3-yl)propan-1-one (3ab)

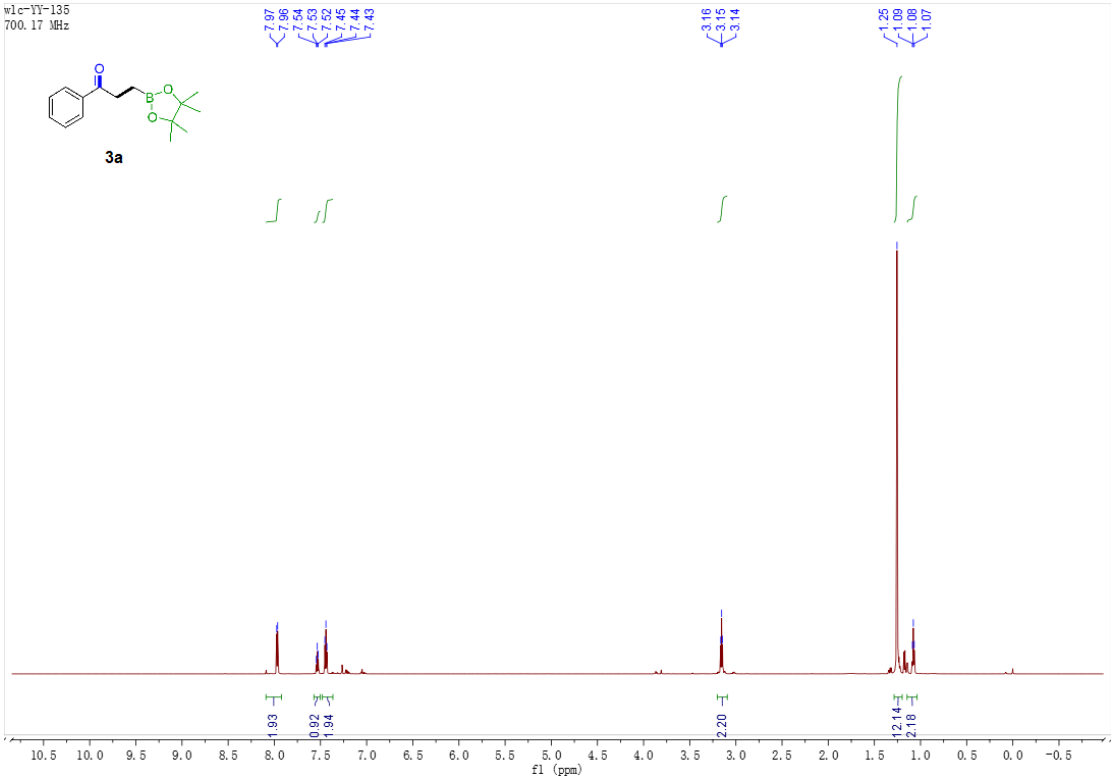
30.8 mg, 55% yield, pale yellow oil. Eluent: pentane/ethyl acetate = 20/1.

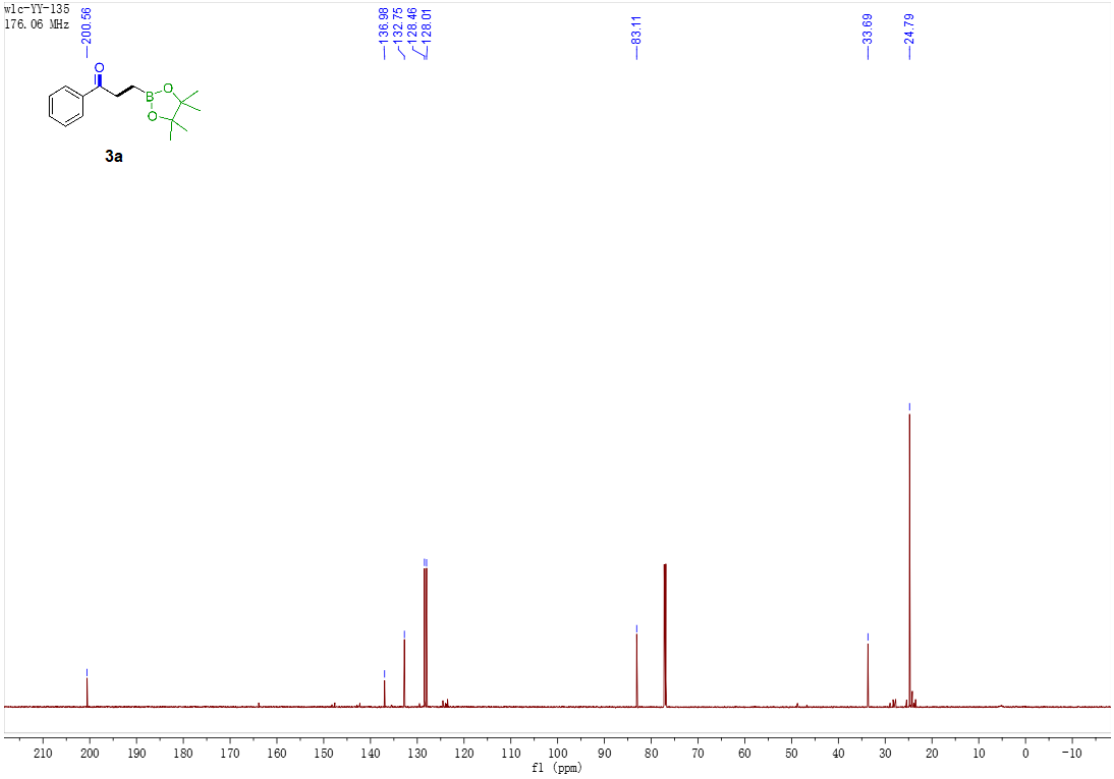
^{1}H NMR (700 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.55 (d, *J* = 5.0 Hz, 1H), 7.30 – 7.27 (m, 1H), 2.97 (t, *J* = 7.0 Hz, 2H), 1.56 (s, 2H), 1.30 (s, 12H), 0.99 (t, *J* = 7.0 Hz, 2H).

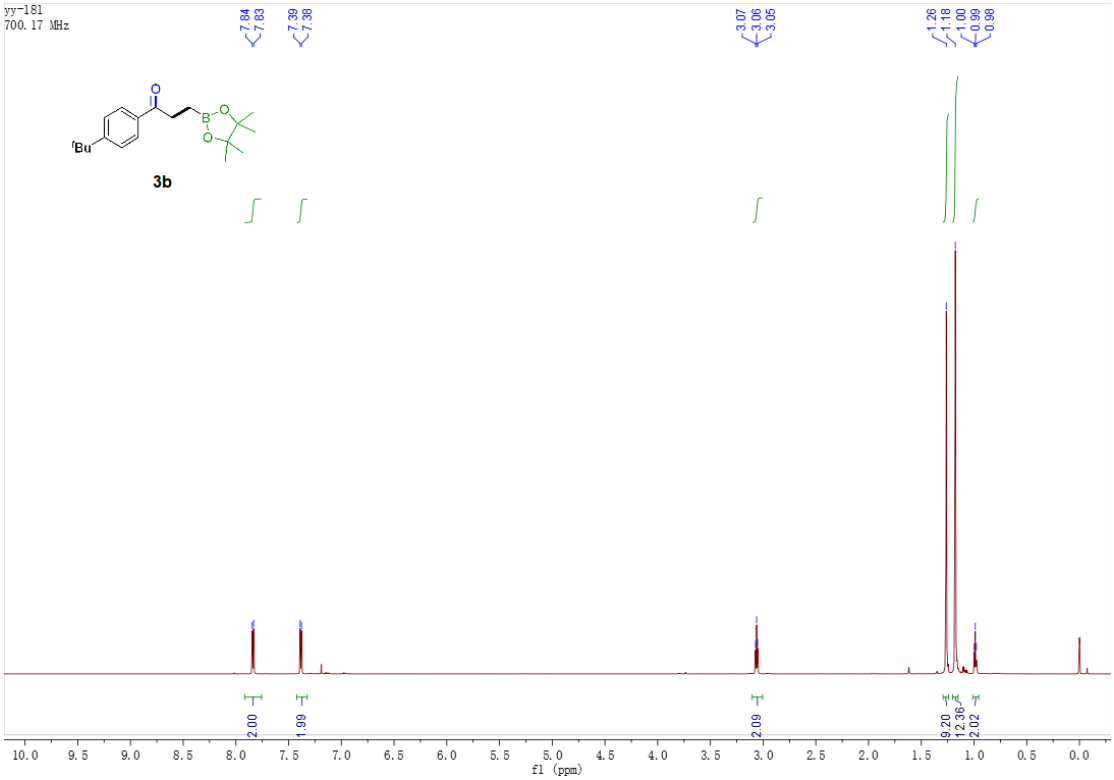
^{13}C NMR (176 MHz, $CDCl_3$) δ 196.14, 142.61, 131.22, 127.08, 125.86, 69.38, 49.51, 31.93, 31.69.

HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{14}H_{22}BO_3S$ 281.1385; Found: 281.1380.

5. NMR Spectra



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7.39
7.38
3.07
3.06
3.05
1.26
1.18
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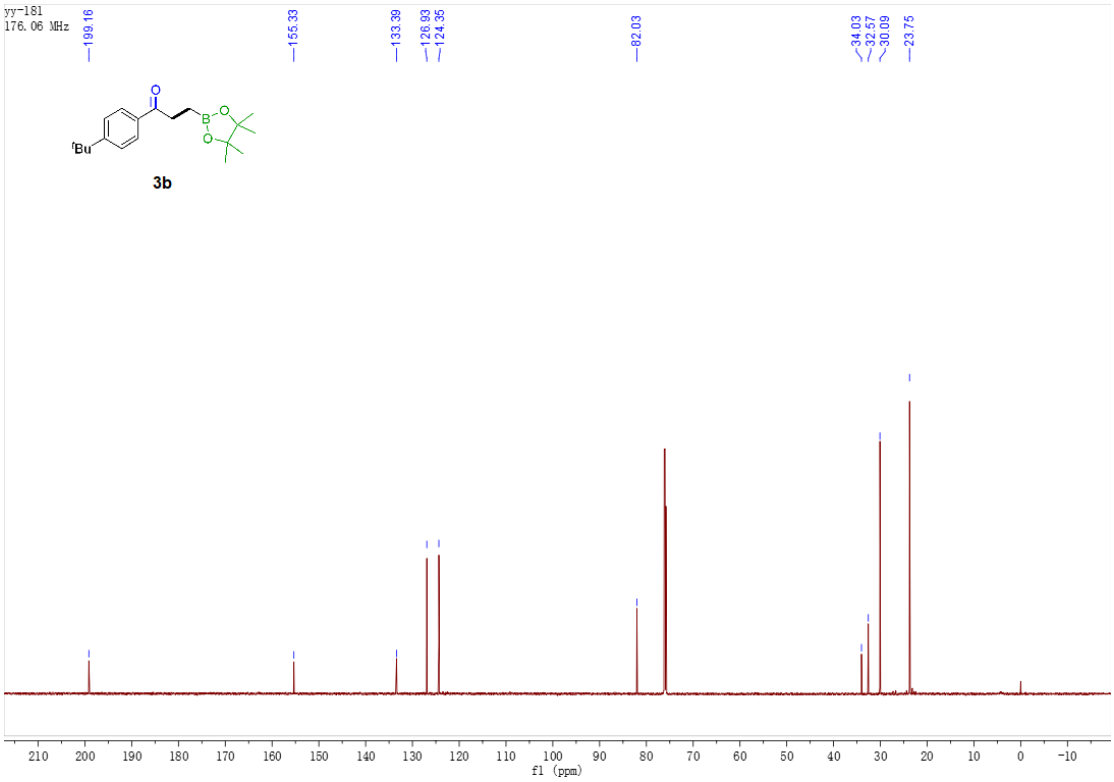
0.98
tBu
3b
2.00
1.99
2.09
9.20
12.36
2.02
f1 (ppm)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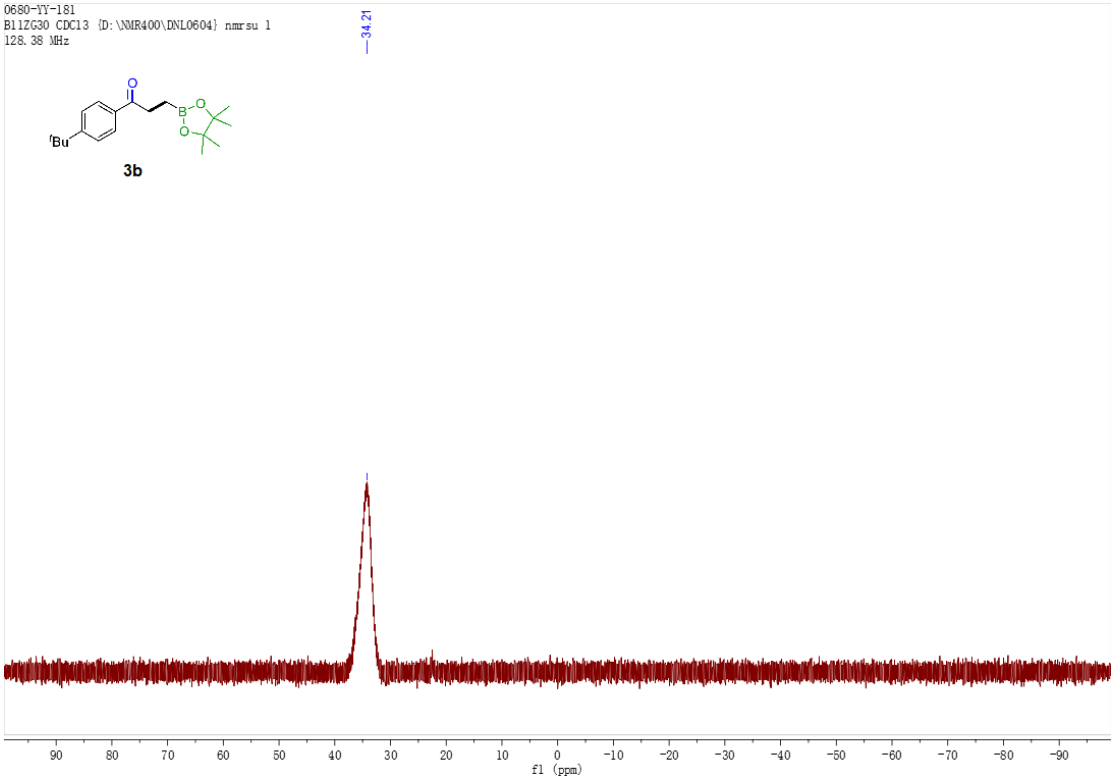
3b
f1 (ppm)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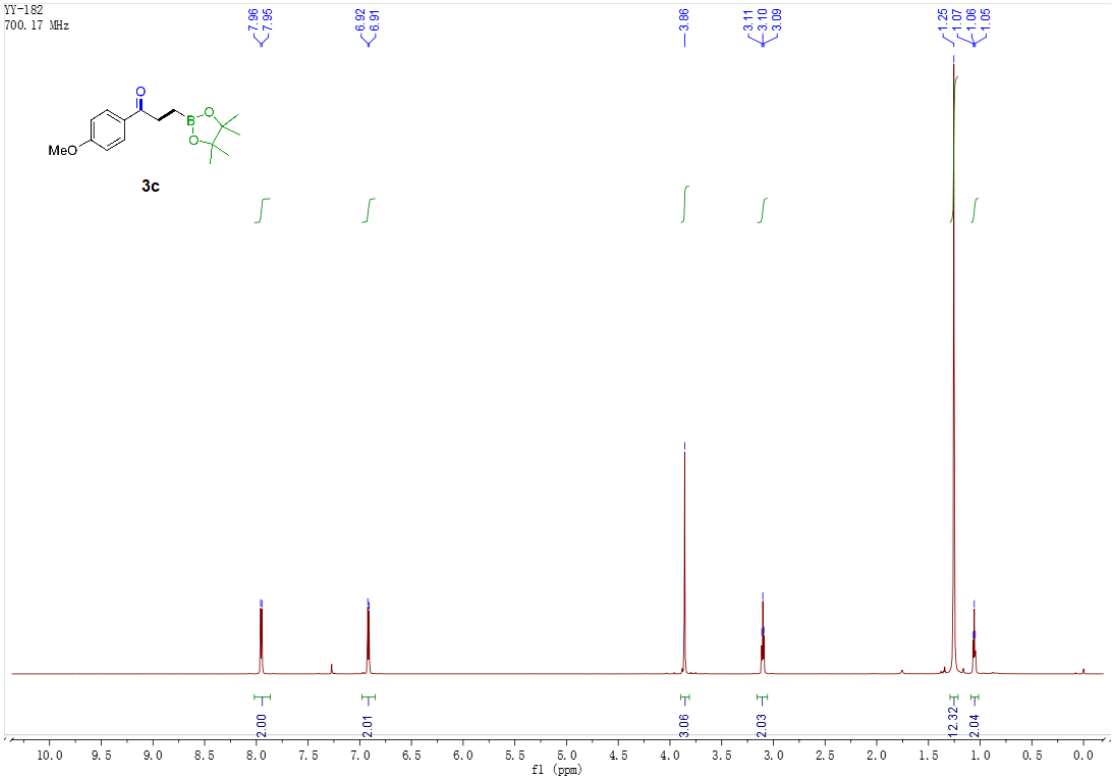
f1 (ppm)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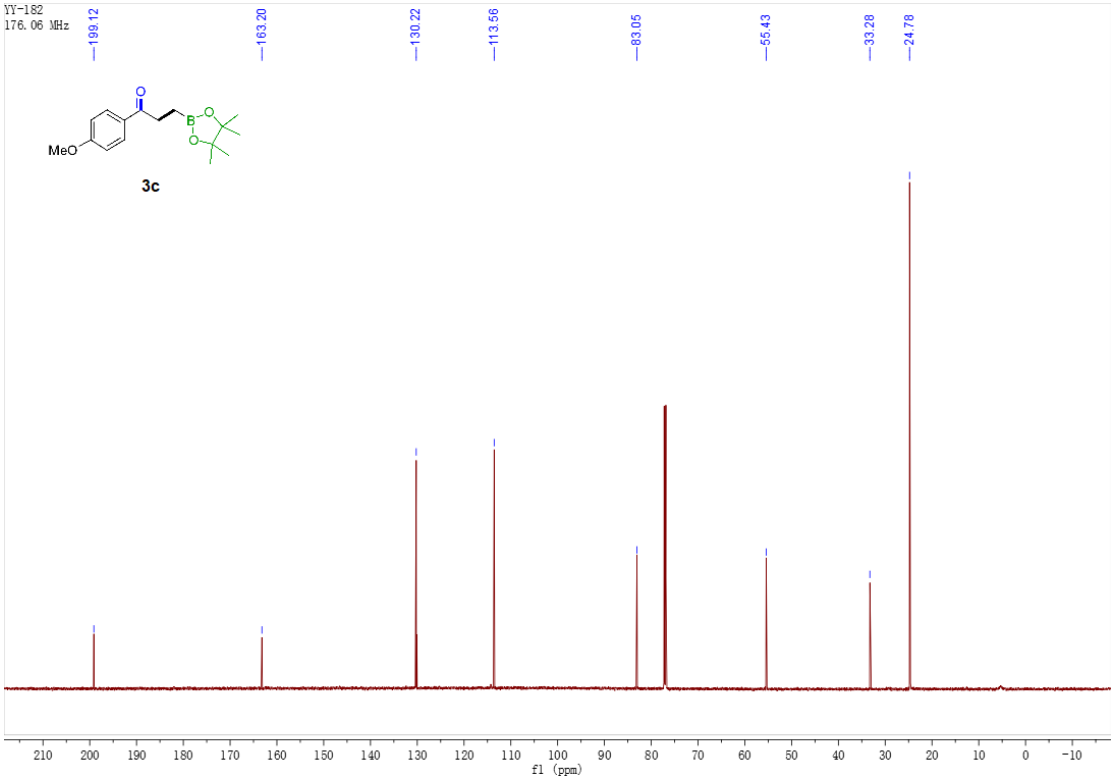
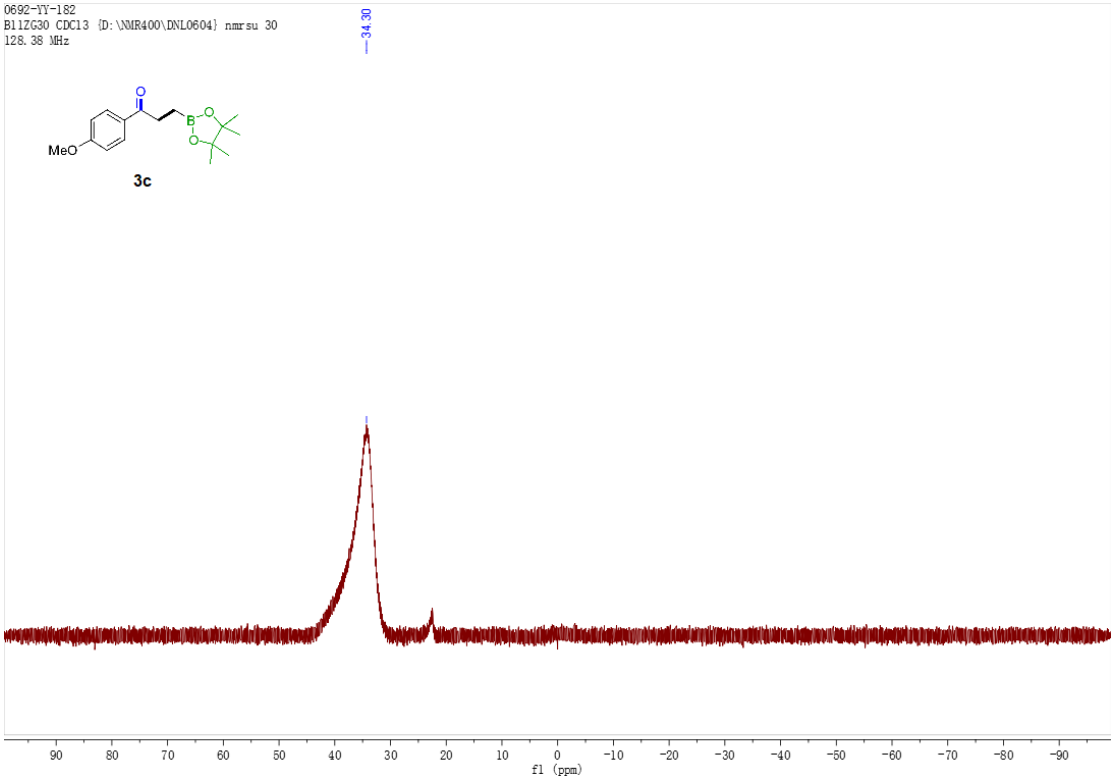
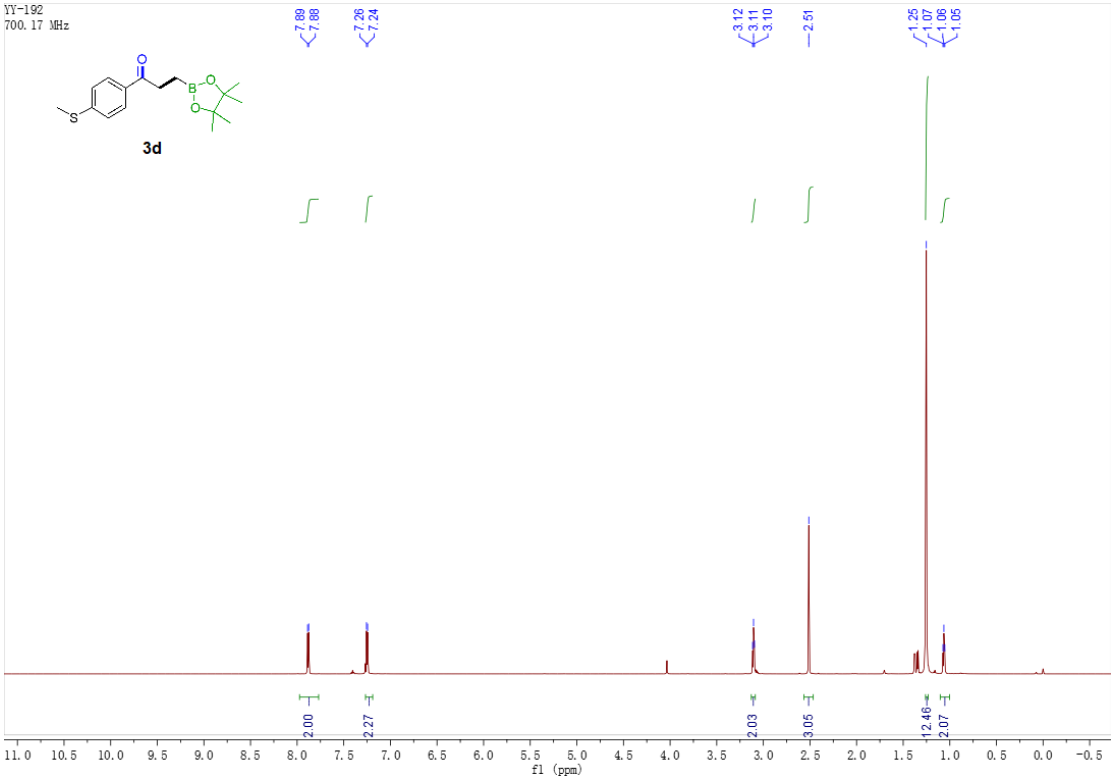
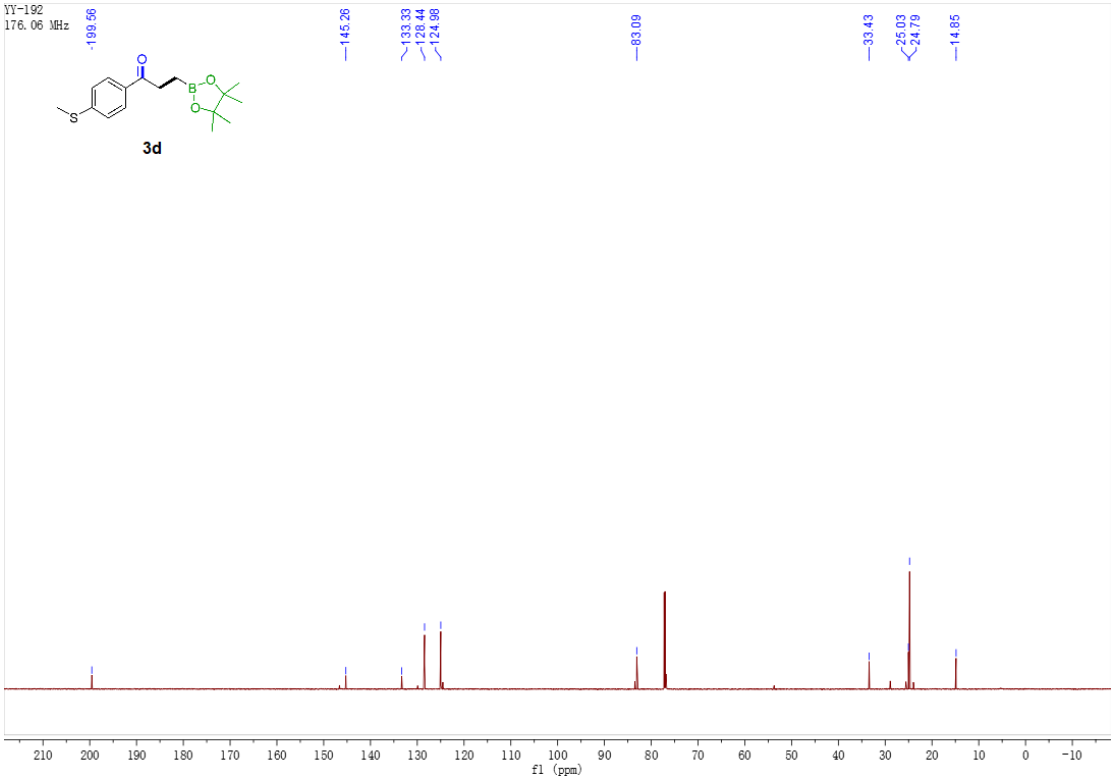
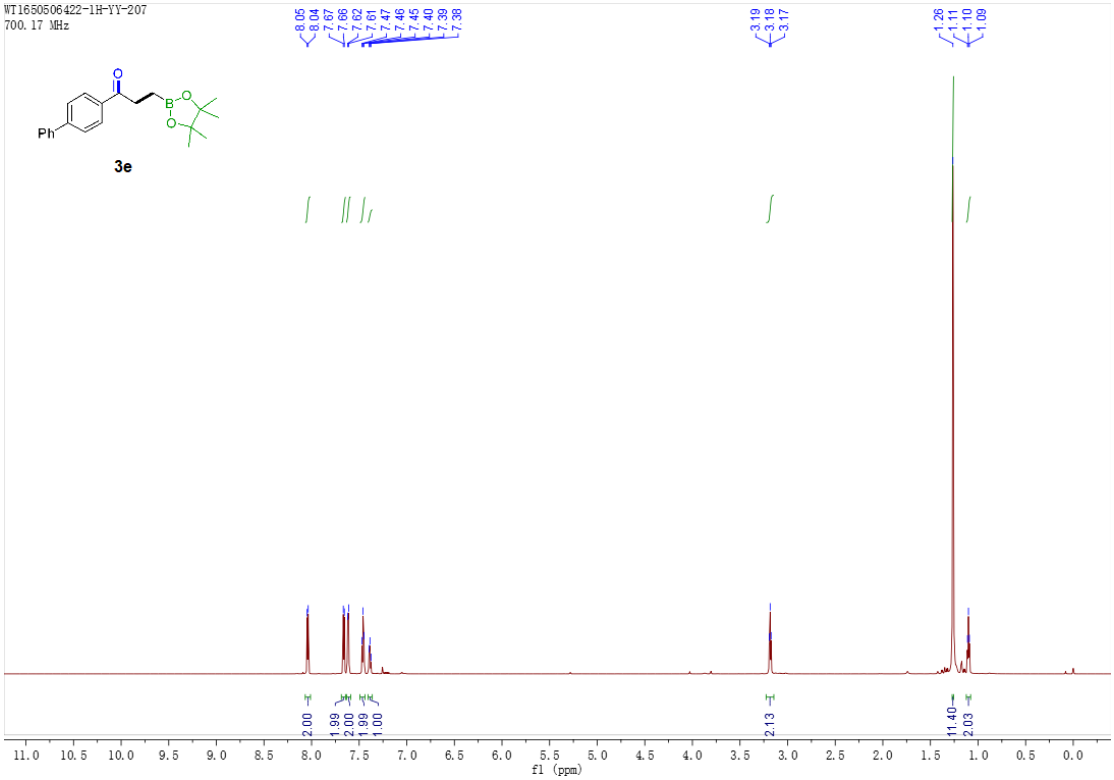
3c
2.00
2.01
3.06
2.03
12.32
2.04
f1 (ppm)

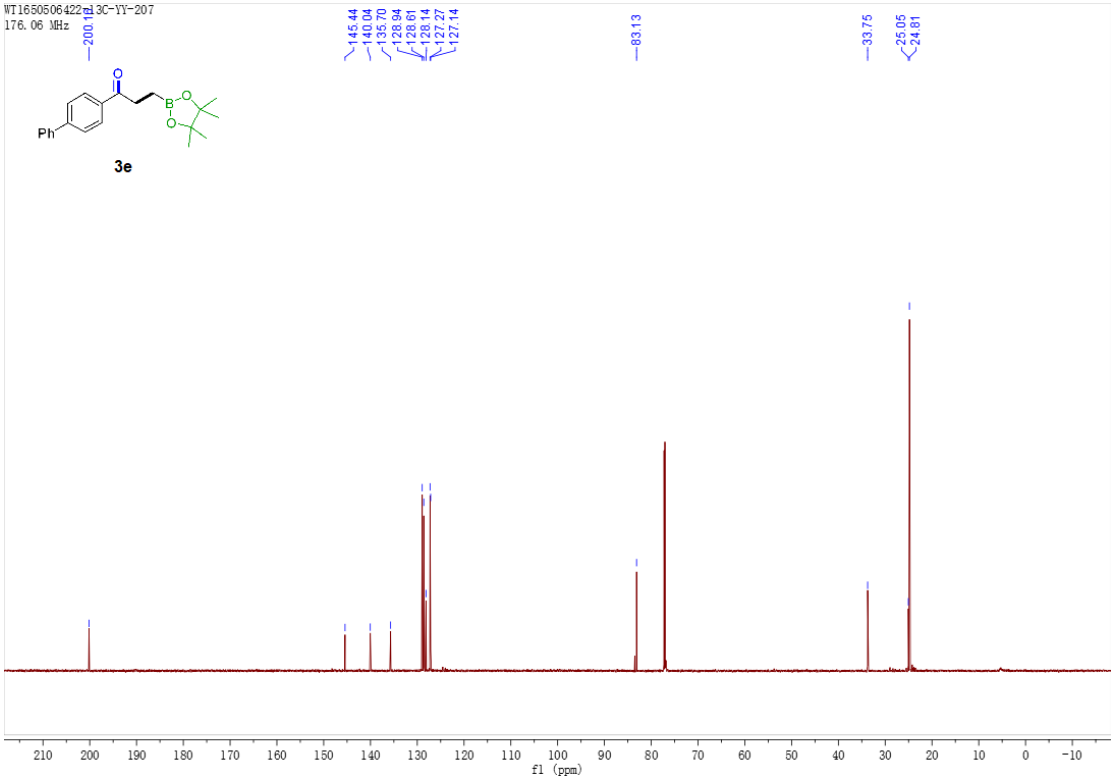

YY-182
176.06 MHz
199.12
163.20
130.22
113.56
83.05
55.43
33.28
24.78
MeO
O
B
O
O
3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


0692-YY-182
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 30
128.38 MHz
34.30
MeO
O
B
O
O
3c
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)


YY-192
700.17 MHz
7.89
7.88
7.26
7.24
3.12
3.11
3.10
2.51
1.25
1.07
1.06
1.05
3d
2.00
2.27
2.03
3.05
12.46
2.07
f1 (ppm)


YY-192
176.06 MHz
199.56
145.26
133.33
128.44
124.98
83.09
33.43
25.03
24.79
14.85
3d
f1 (ppm)


WT1650506422-1H-YY-207
700.17 MHz
8.05
8.04
7.67
7.66
7.62
7.61
7.47
7.46
7.45
7.40
7.39
7.38
3.19
3.18
3.17
1.26
1.11
1.10
1.09
Ph
O
B
O
O
3e
2.00
1.99
2.00
1.99
1.00
2.13
11.40
2.03
f1 (ppm)


WT1650506422-13C-YY-207
176.06 MHz
200.14
145.44
140.04
135.70
128.94
128.61
128.14
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83.13
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25.05
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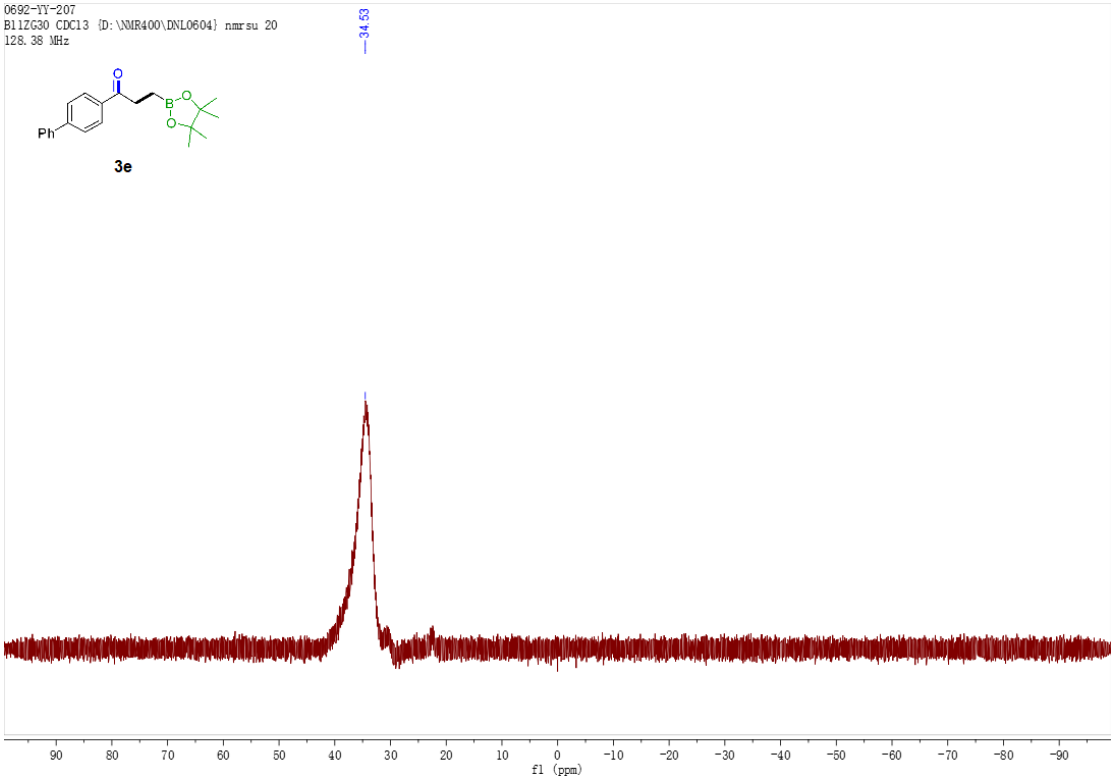
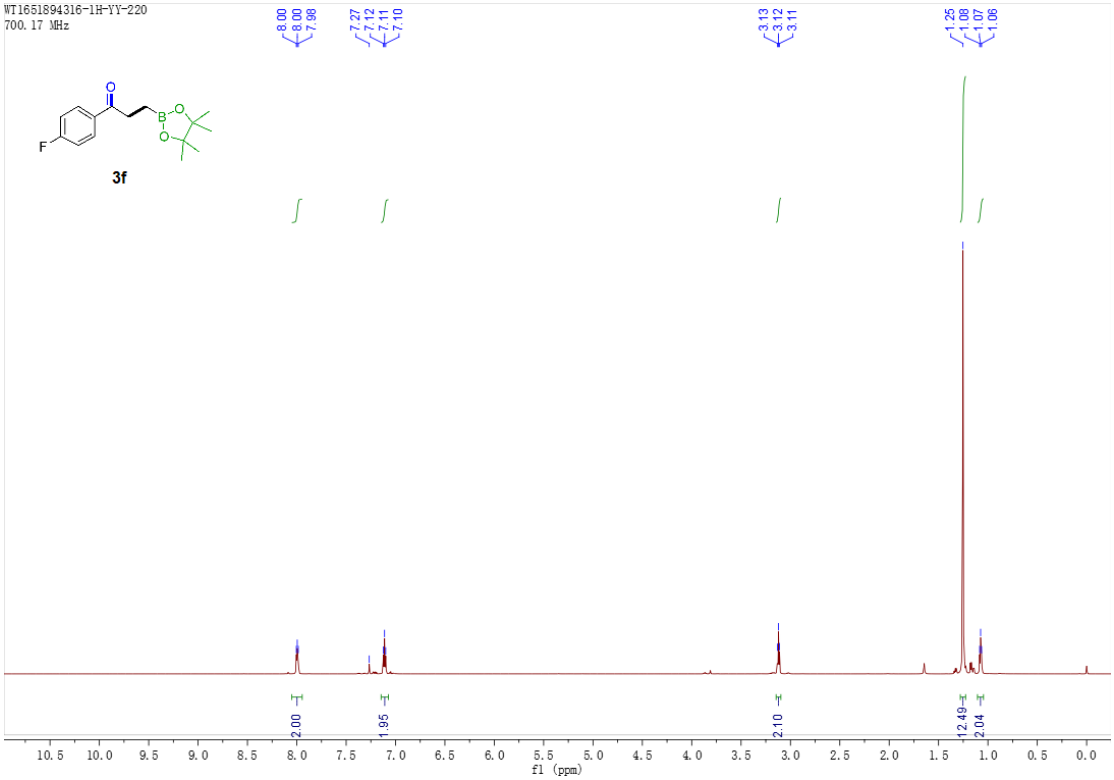
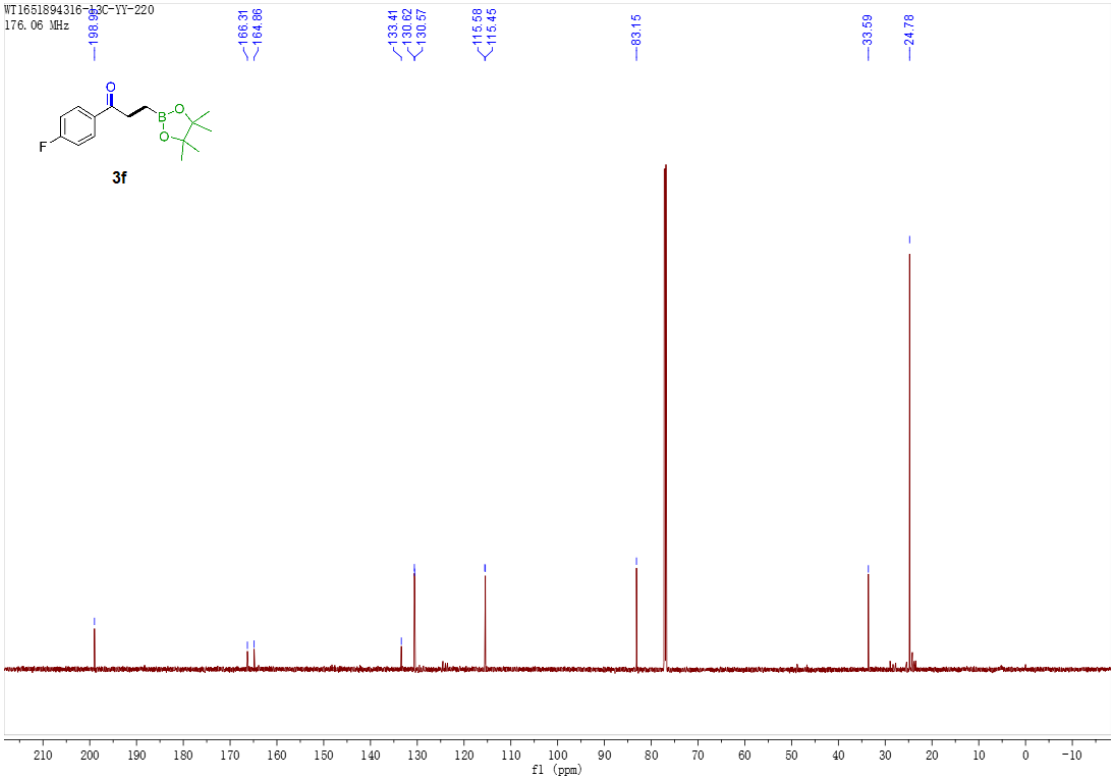
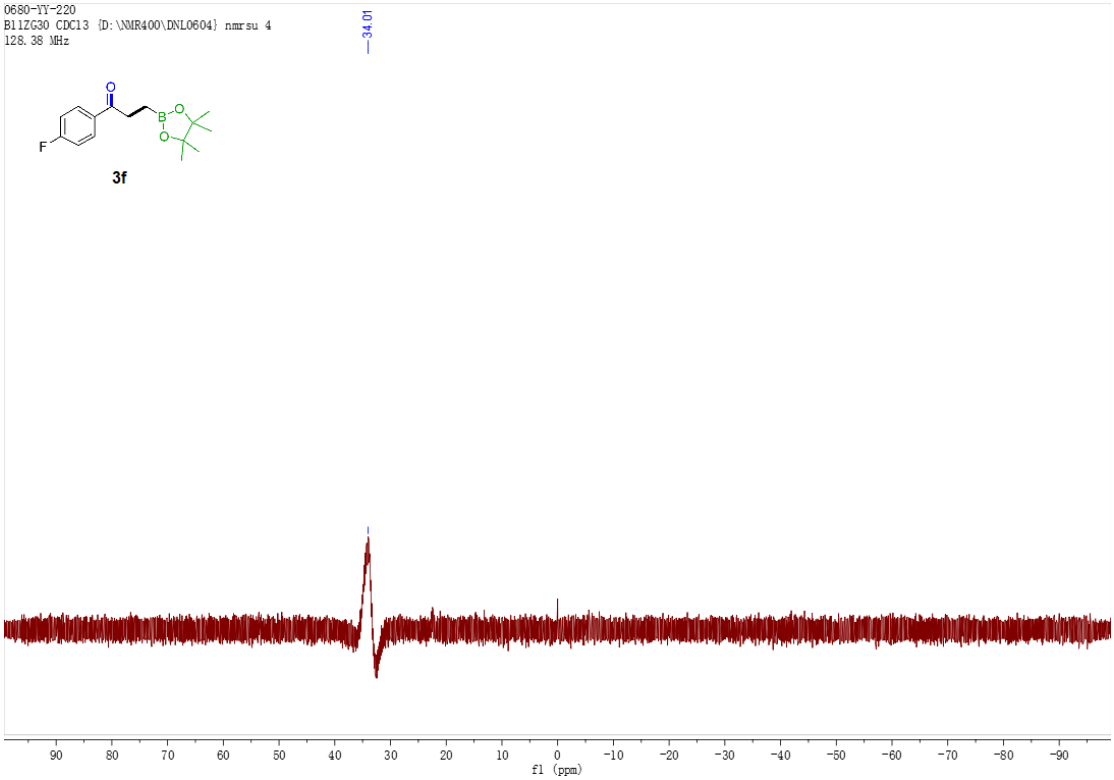
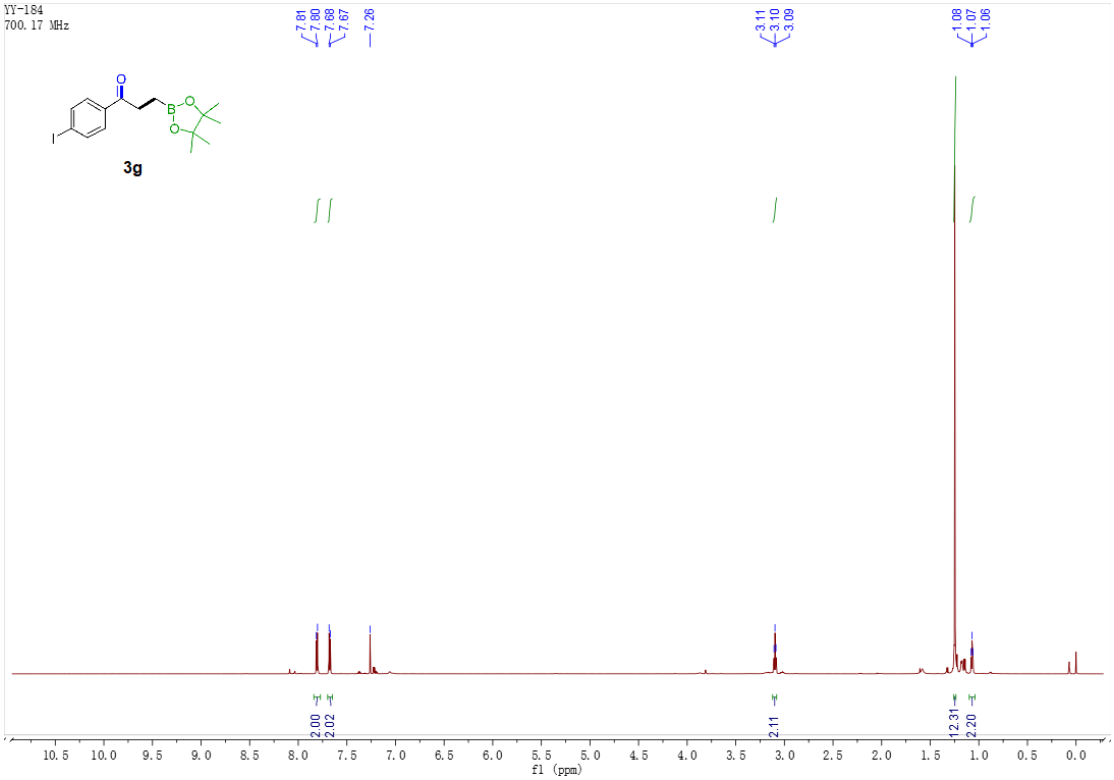
Ph
O
B
O
O
3e
f1 (ppm)

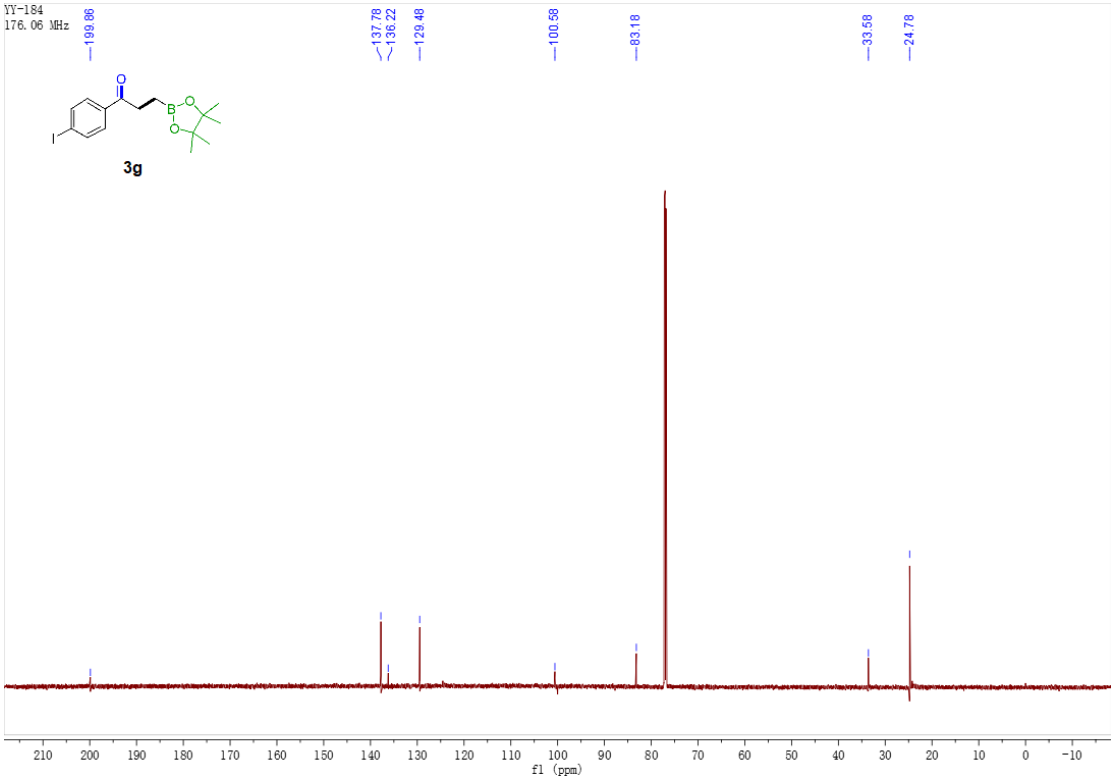

0692-YY-207
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 20
128.38 MHz
34.53
Ph
3e
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

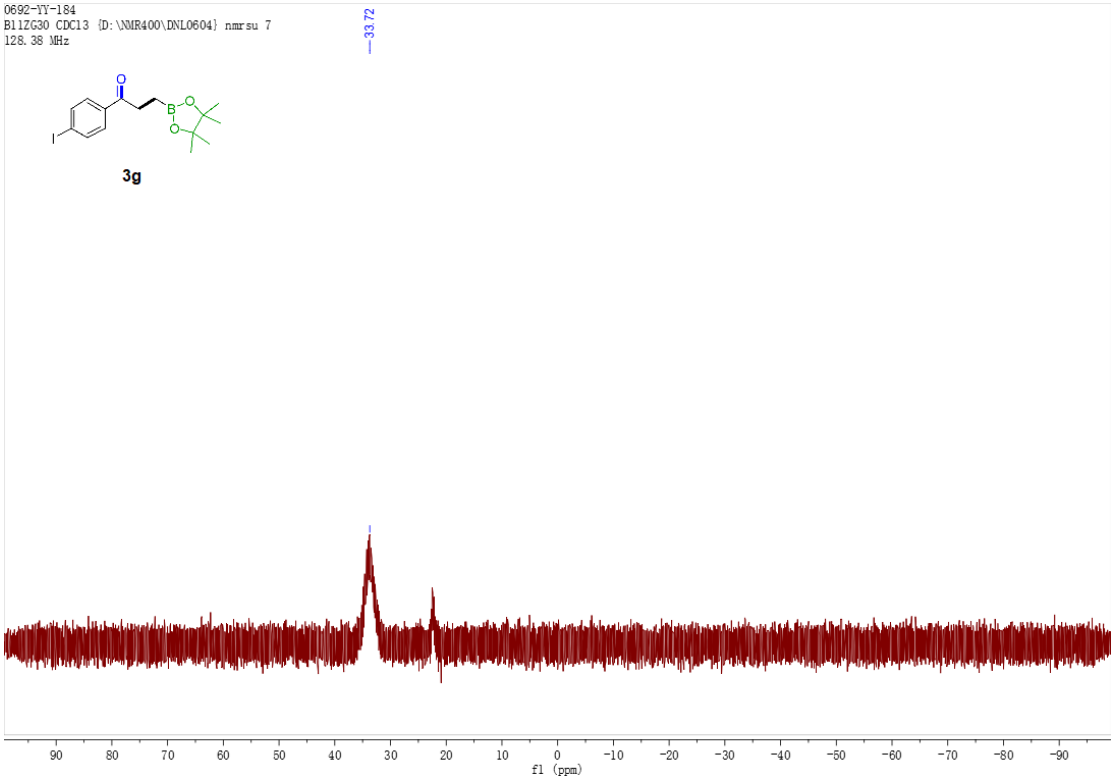

WT1651894316-1H-YY-220
700.17 MHz
8.00
8.00
7.98
7.27
7.12
7.11
7.10
3.13
3.12
3.11
1.25
1.08
1.07
1.06
F
3f
2.00
1.95
2.10
12.49
2.04
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

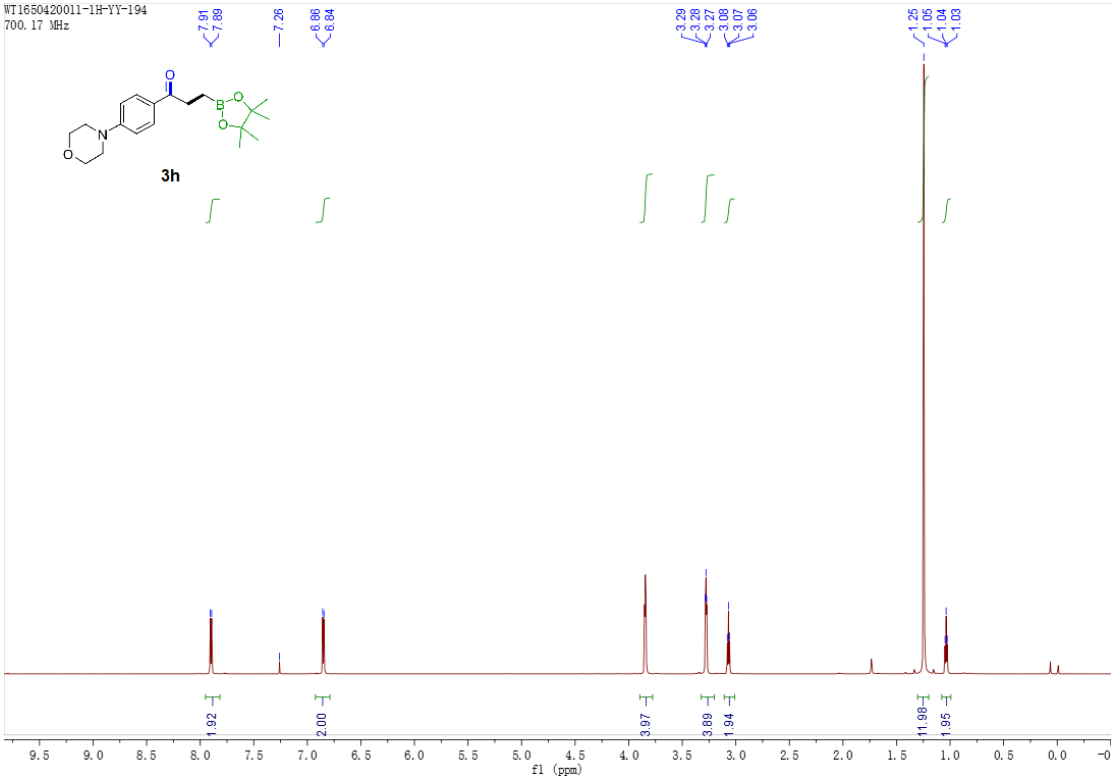

WT1651894316-13C-YY-220
176.06 MHz
198.99
166.31
164.86
133.41
130.62
130.57
115.58
115.45
83.15
33.59
24.78
F
O
B
O
O
3f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

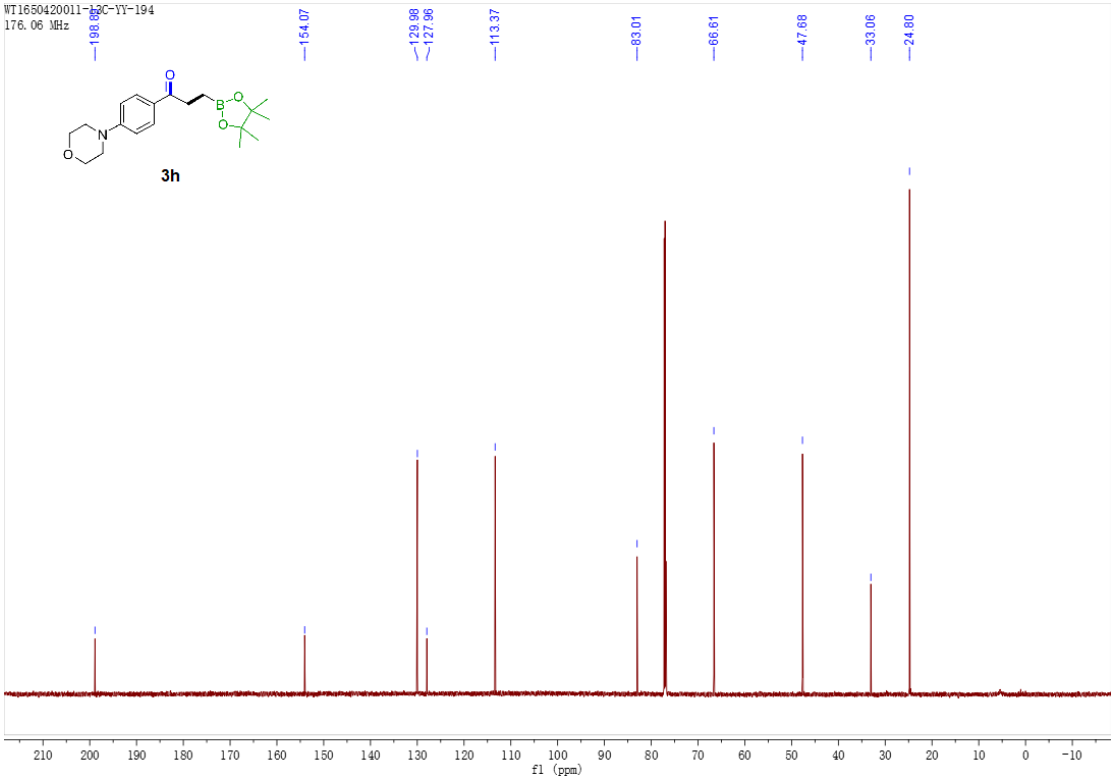

0680-YY-220
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 4
128.38 MHz
34.01
F
O
B
O
O
3f
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

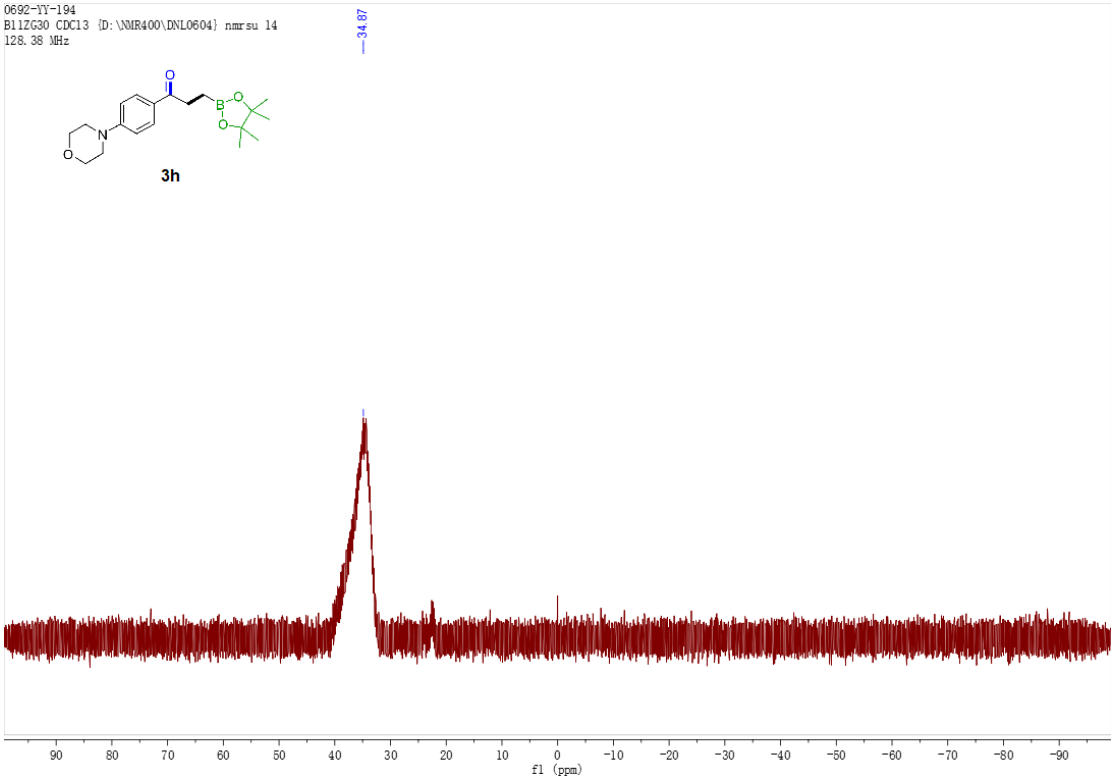

YY-184
700.17 MHz
7.81
7.80
7.68
7.67
7.26
3.11
3.10
3.09
1.08
1.07
1.06
3g
2.00
2.02
2.11
12.31
2.20
f1 (ppm)

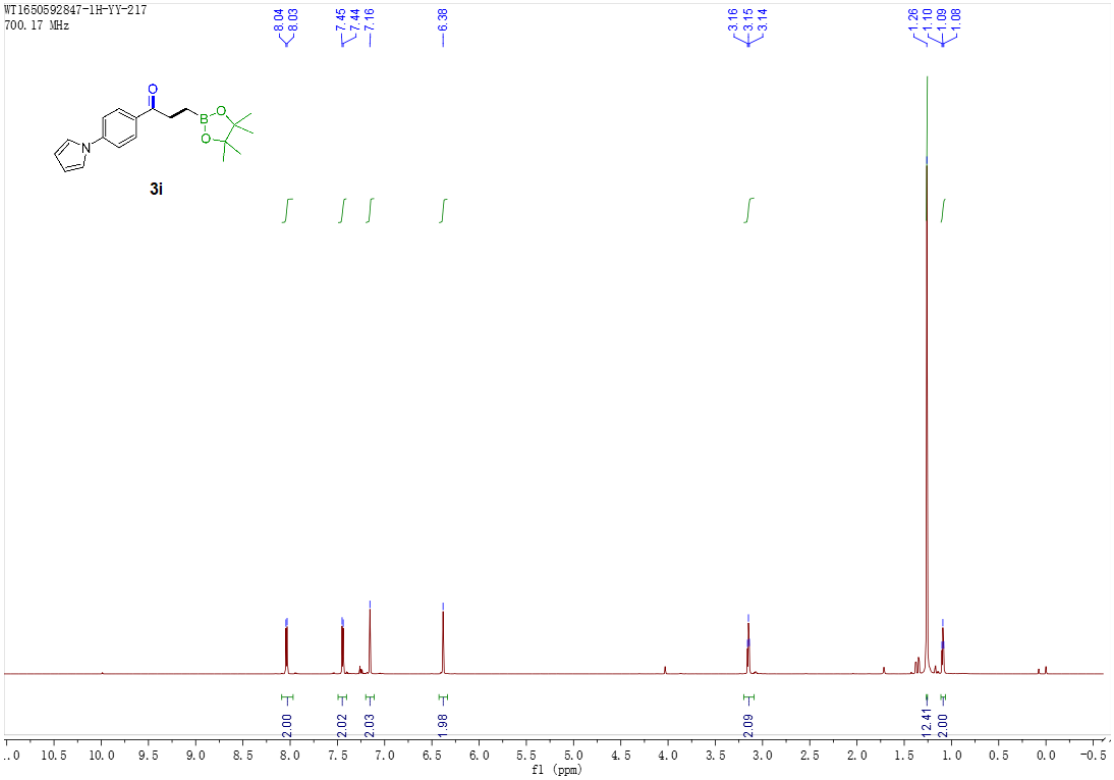

YY-184
176.06 MHz
199.86
137.78
136.22
129.48
100.58
83.18
33.58
24.78
3g
f1 (ppm)

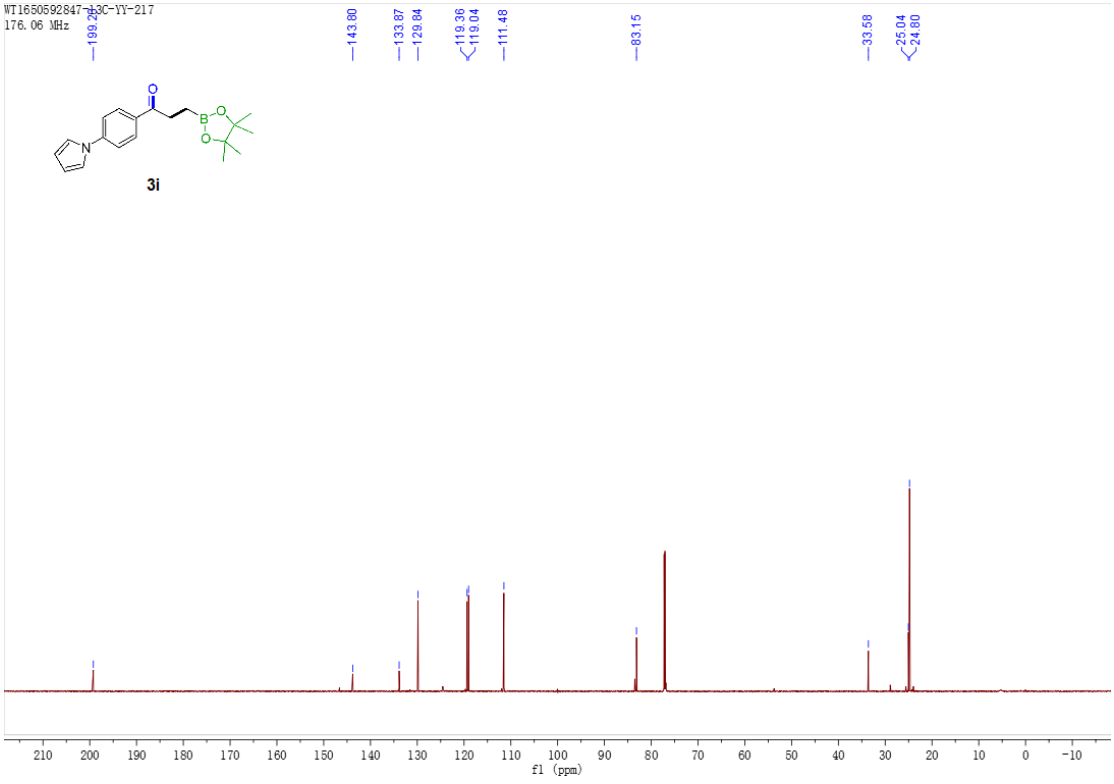

0692-YY-184
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 7
128.38 MHz
33.72
3g
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

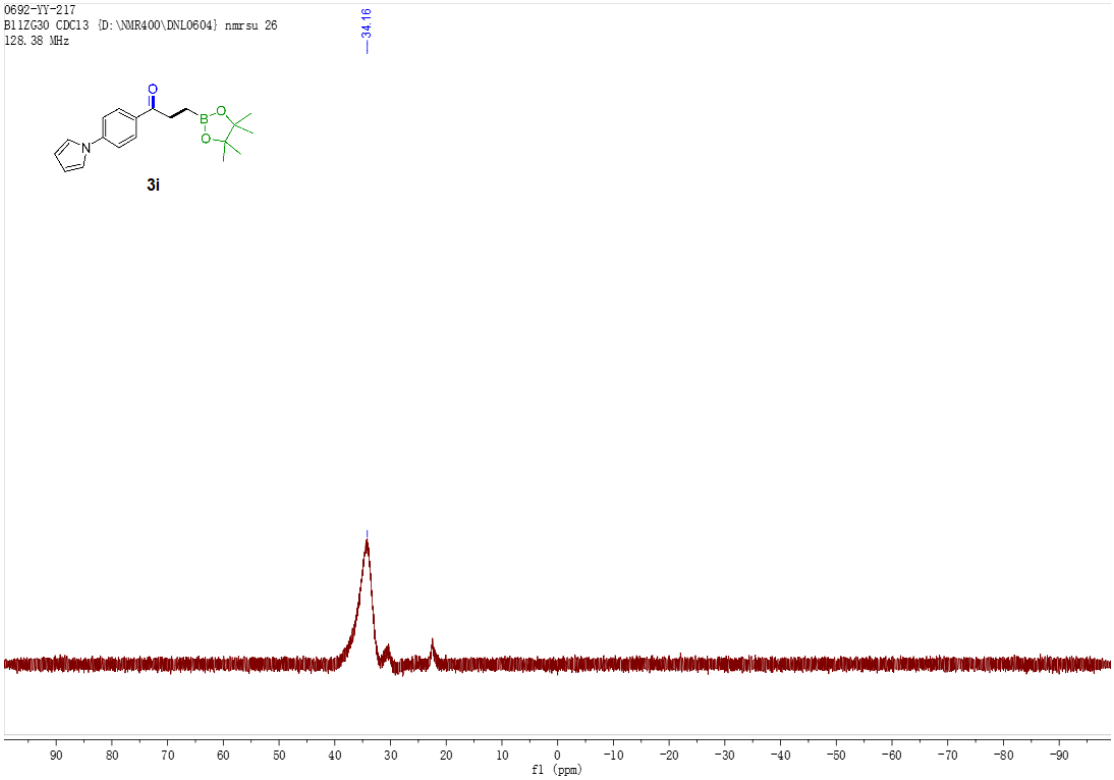

WT1650420011-1H-YY-194
700.17 MHz
7.91
7.89
7.26
6.86
6.84
3.29
3.28
3.27
3.08
3.07
3.06
1.25
1.05
1.04
1.03
3h
1.92
2.00
3.97
3.89
1.94
11.98
1.95
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

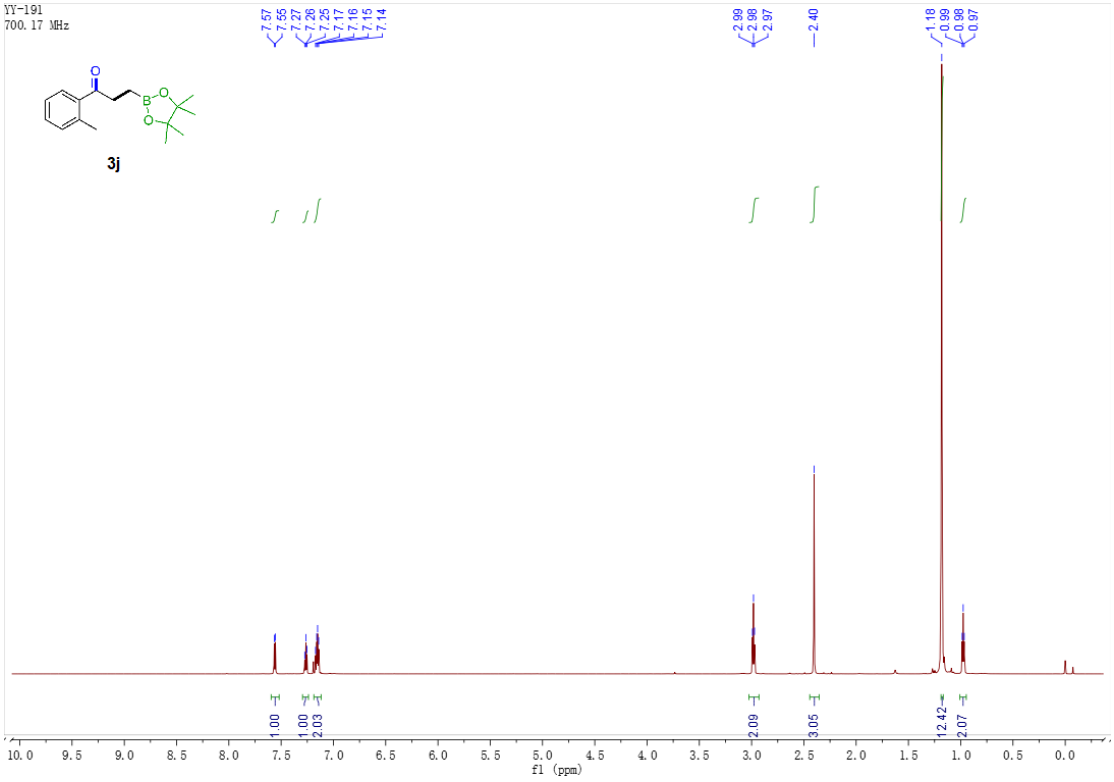

WT1650420011-13C-YY-194
176.06 MHz
198.88
154.07
129.98
127.96
113.37
83.01
66.61
47.68
33.06
24.80
3h
f1 (ppm)

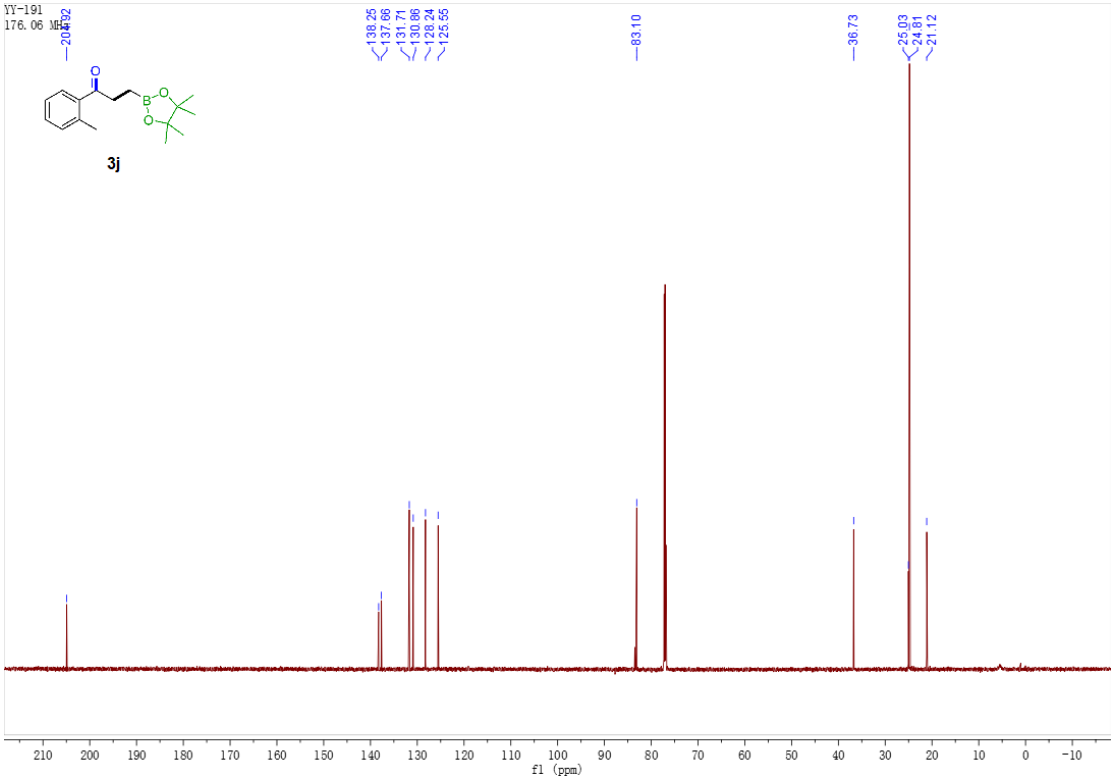

0692-YY-194
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 14
128.38 MHz
34.87
3h
f1 (ppm)

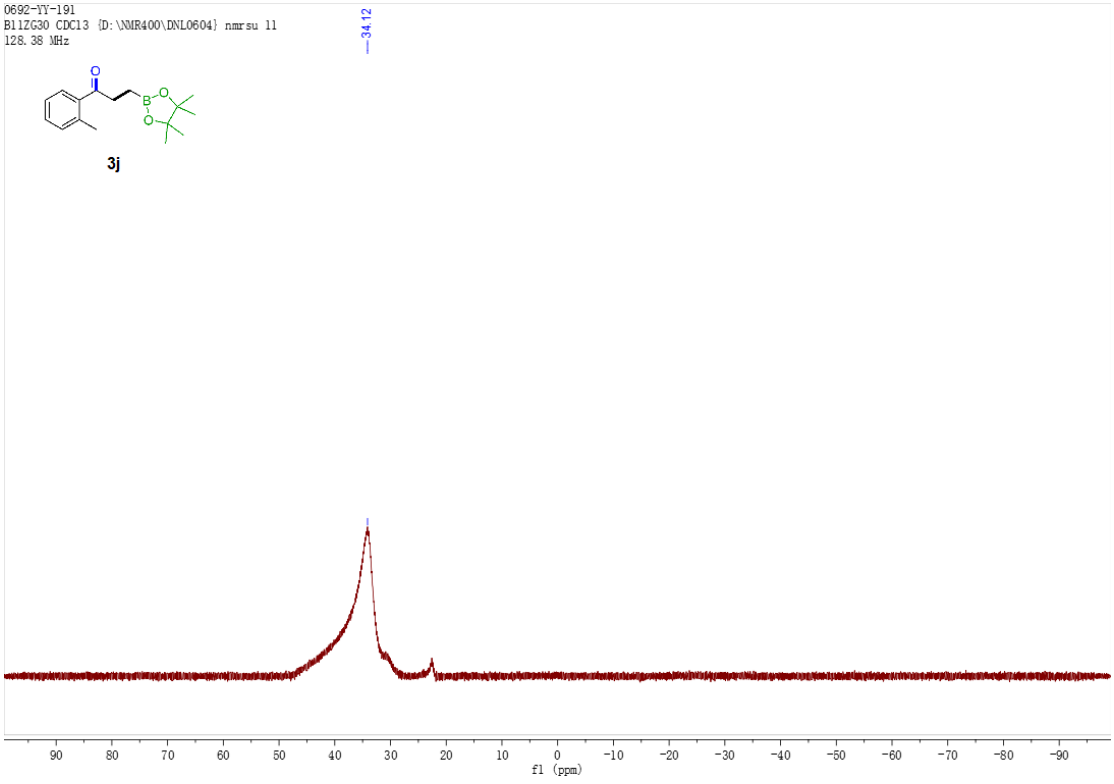

WT1650592847-1H-YY-217
700.17 MHz
8.04
8.03
7.45
7.44
7.16
6.38
3.16
3.15
3.14
1.26
1.10
1.09
1.08
3i
2.00
2.02
2.03
1.98
2.09
12.41
2.00
f1 (ppm)

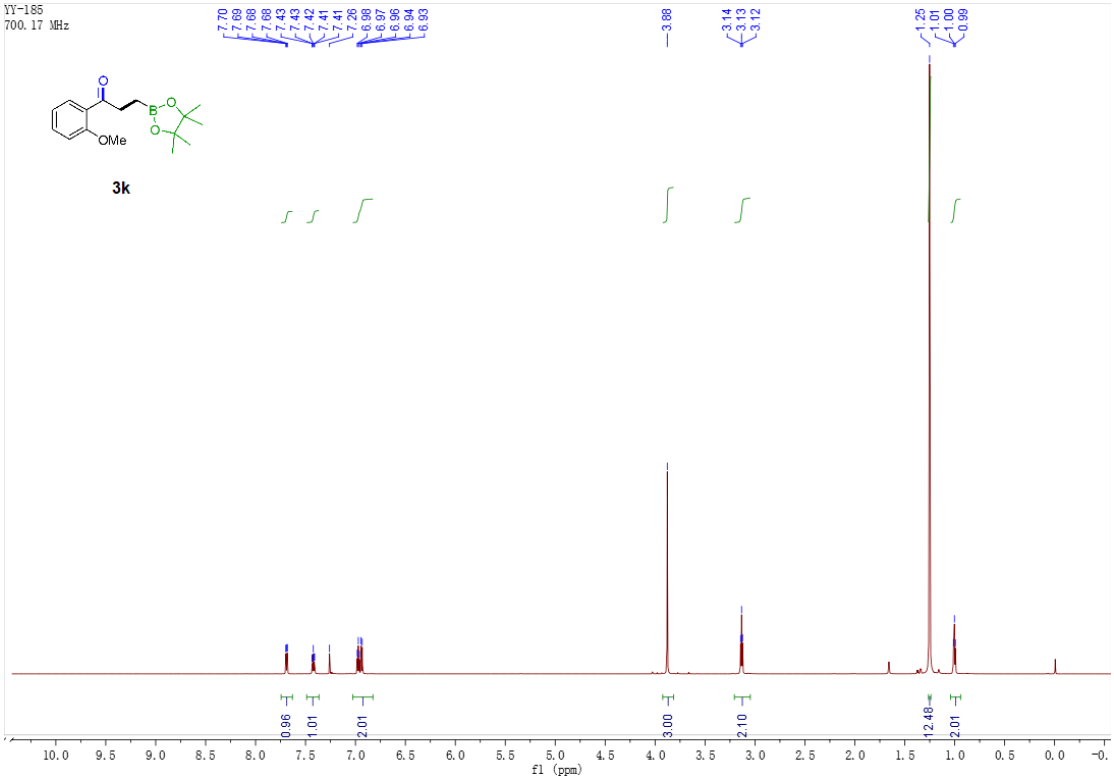

WT1650592847-13C-YY-217
176.06 MHz
199.25
143.80
133.87
129.84
119.36
119.04
111.48
83.15
33.58
25.04
24.80
3i
f1 (ppm)

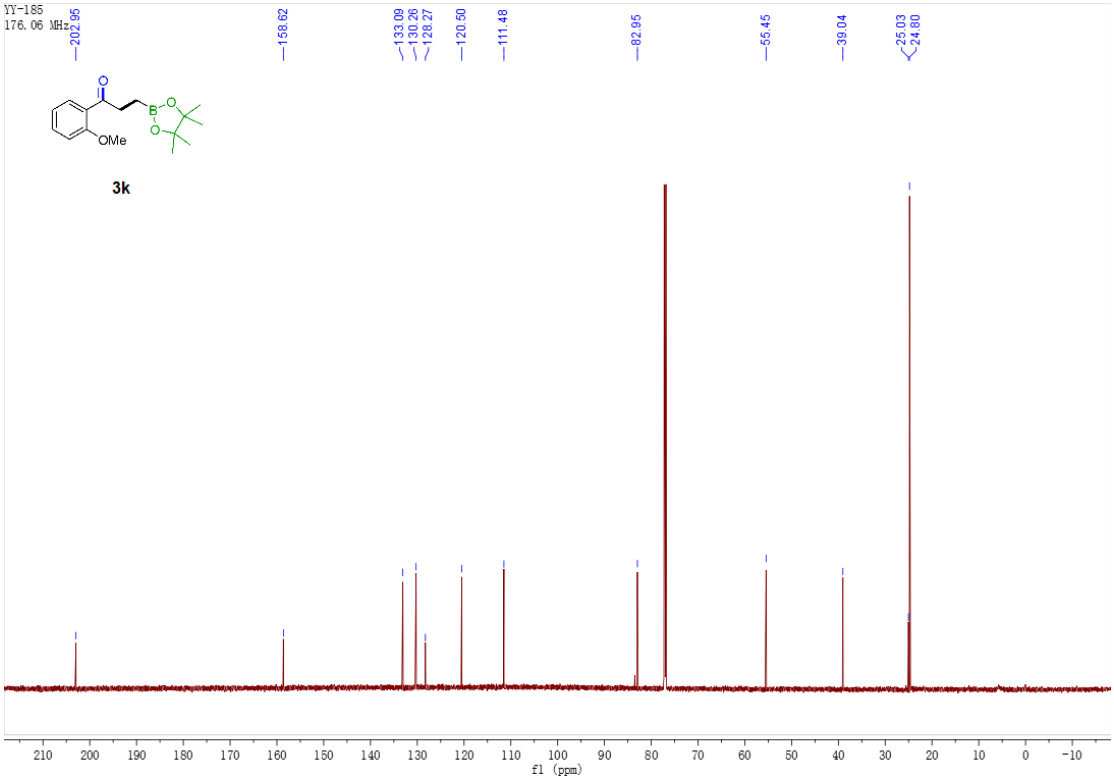

0692-YY-217
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 26
128.38 MHz
34.16
3i
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

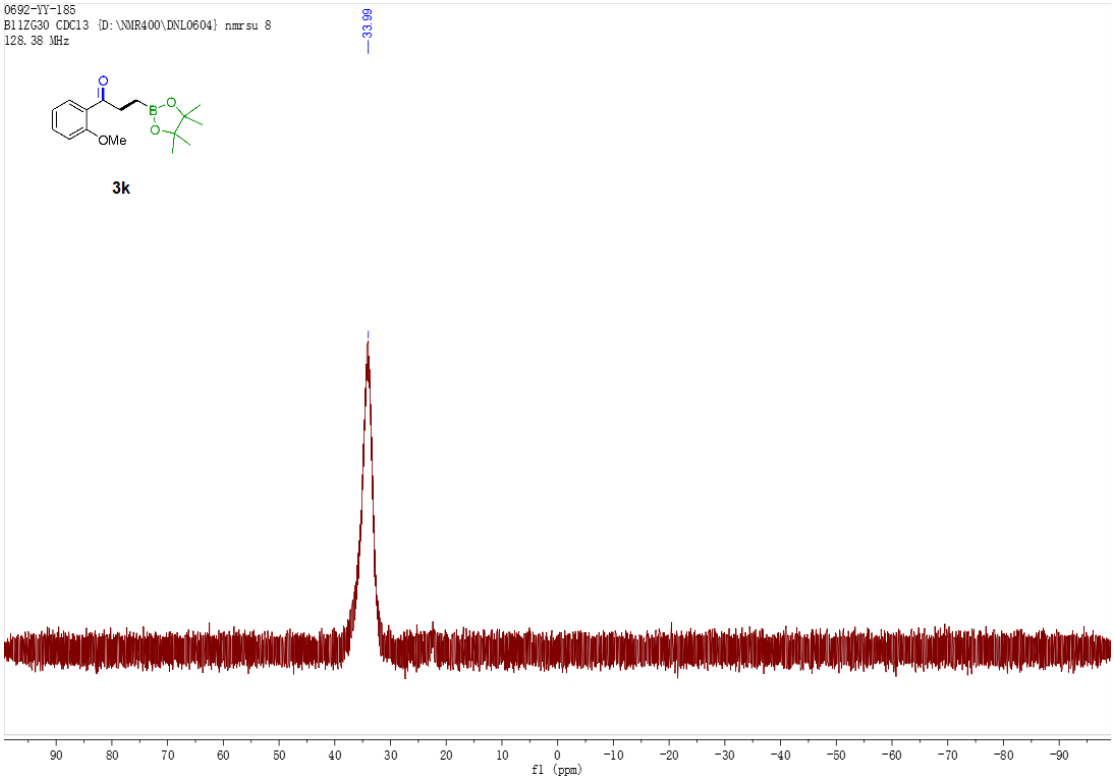

YY-191
700.17 MHz
7.57
7.55
7.27
7.26
7.25
7.17
7.16
7.15
7.14
2.99
2.98
2.97
2.40
1.18
0.99
0.98
0.97
3j
1.00
1.00
2.03
2.09
3.05
12.42
2.07
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

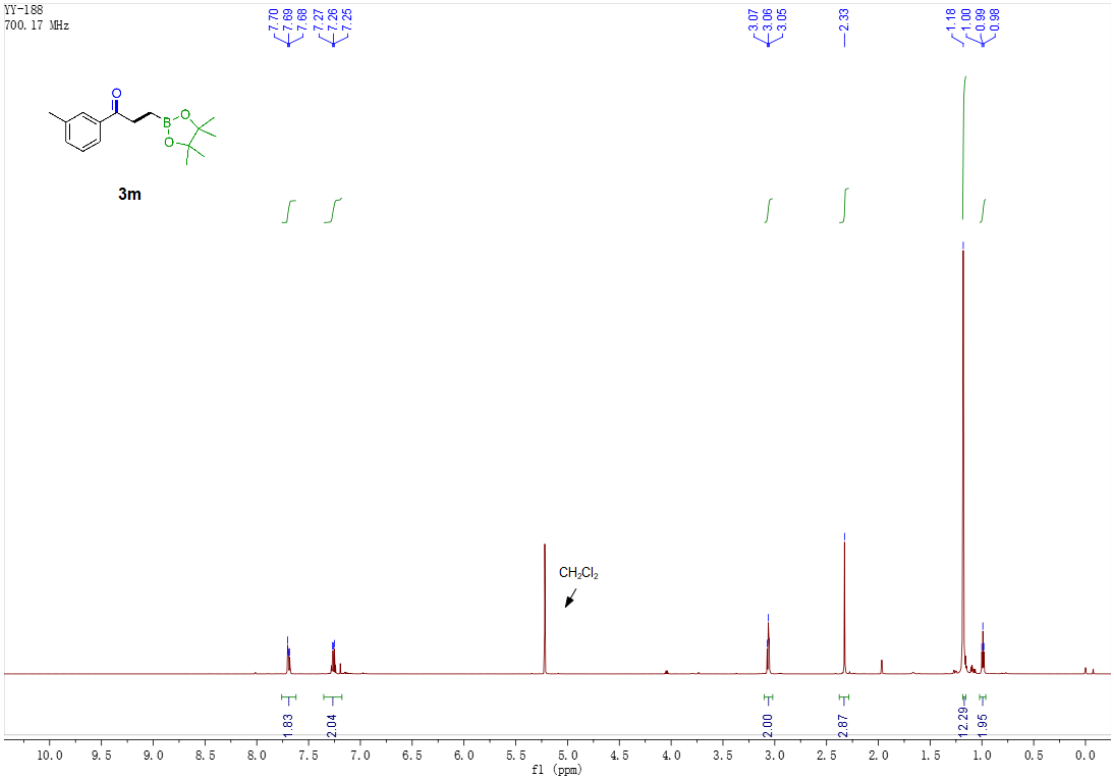

YY-191
176.06 MHz
204.32
138.25
137.66
131.71
130.86
128.24
125.55
83.10
36.73
25.03
24.81
21.12
3j
f1 (ppm)

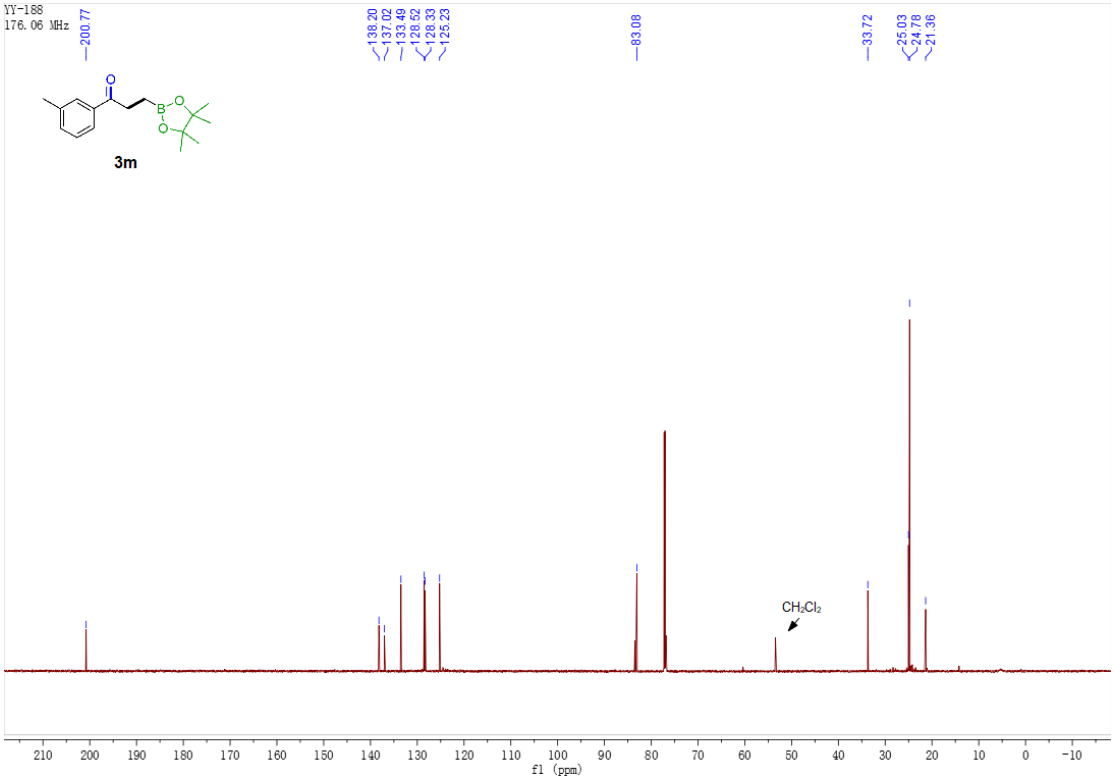

0692-YY-191
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 11
128.38 MHz
34.12
3j
f1 (ppm)

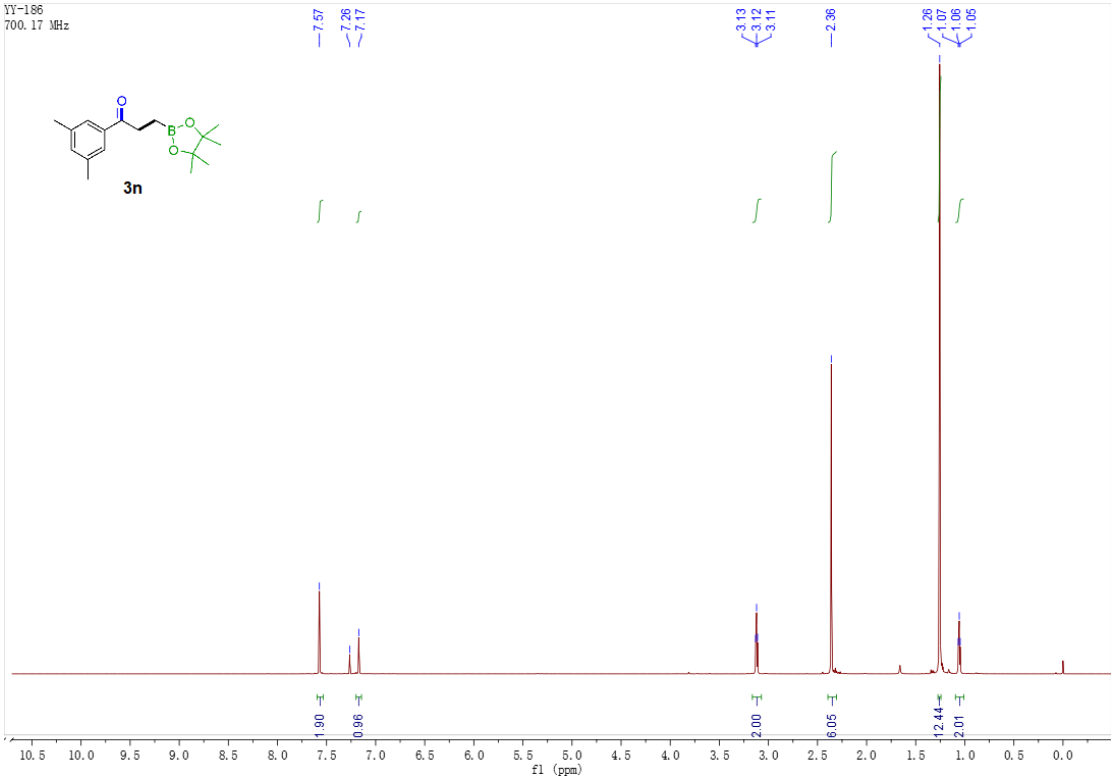

YY-185
700.17 MHz
7.70
7.69
7.68
7.68
7.43
7.43
7.42
7.41
7.41
7.28
6.98
6.97
6.96
6.94
6.93
3.88
3.14
3.13
3.12
1.25
1.01
1.00
0.99
OMe
3k
0.96
1.01
2.01
3.00
2.10
12.48
2.01
f1 (ppm)

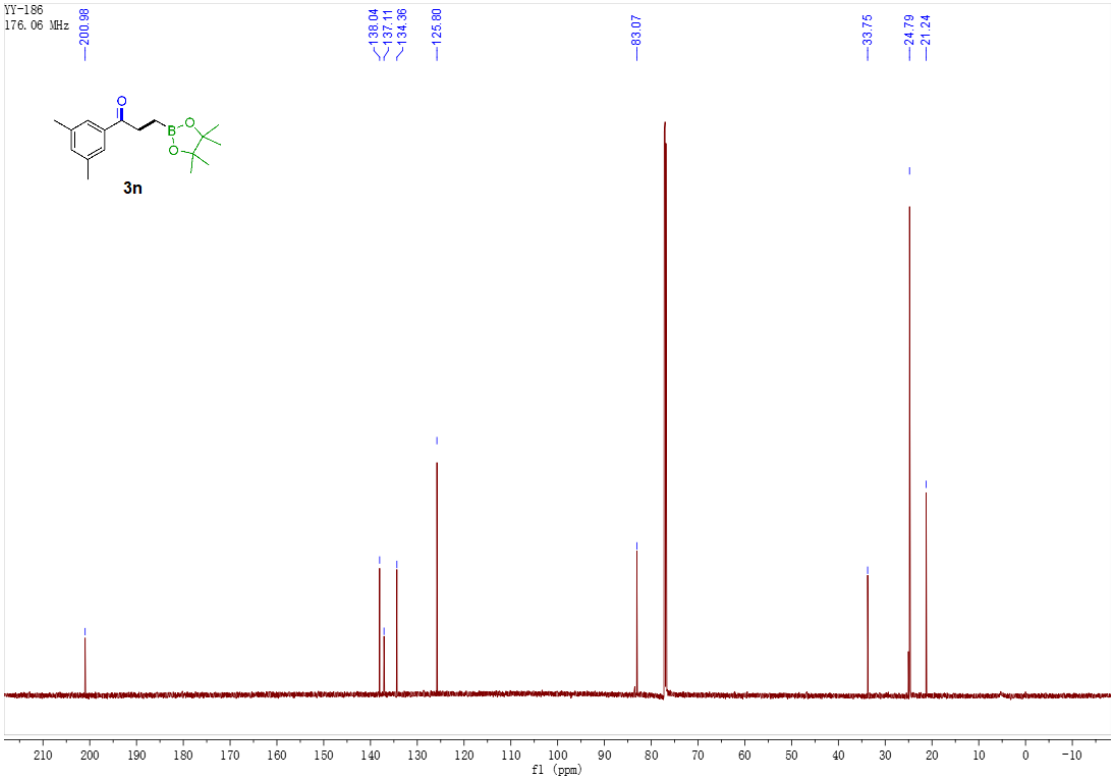

YY-185
176.06 MHz
202.95
158.62
133.09
130.26
128.27
120.50
111.48
82.95
55.45
39.04
25.03
24.80
OMe
3k
f1 (ppm)

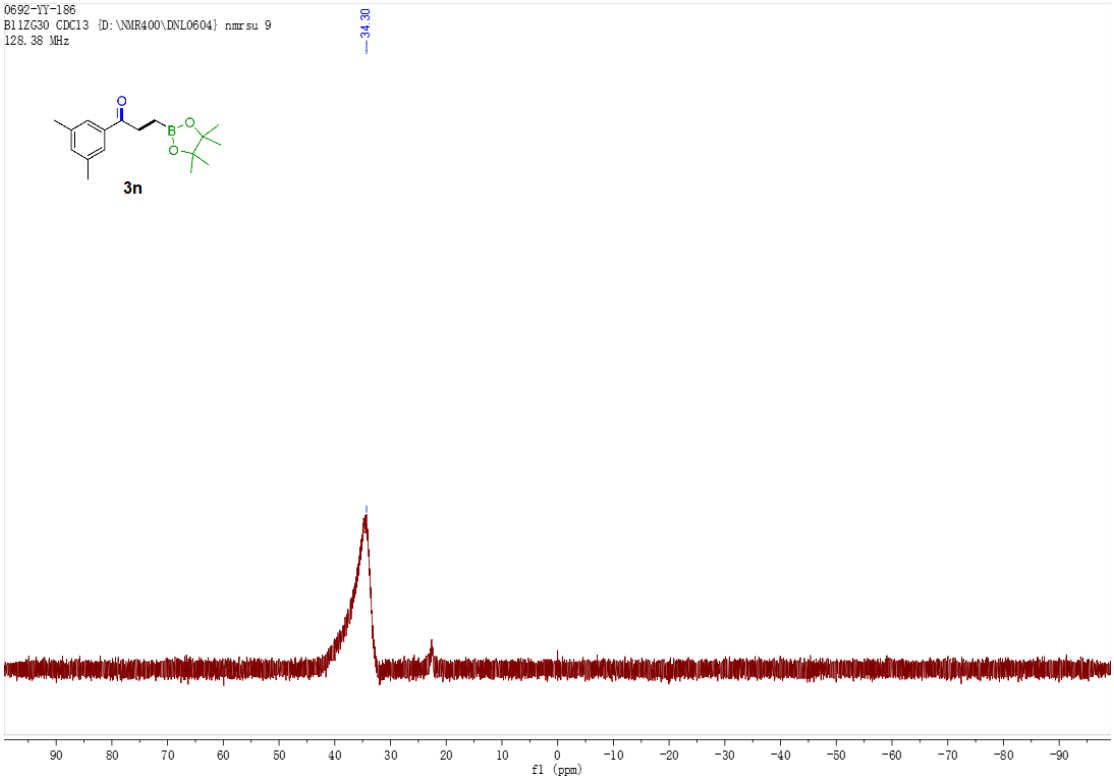

0692-YY-185
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 8
128.38 MHz
33.99
OMe
3k
f1 (ppm)

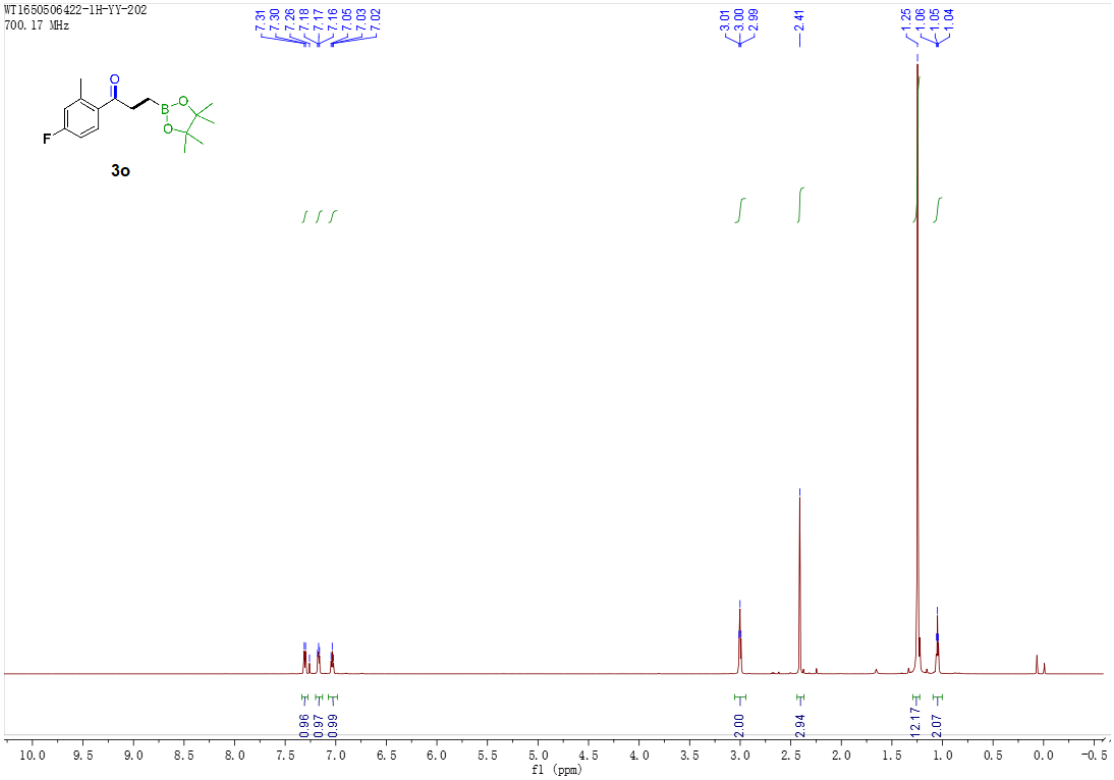

YY-188
700.17 MHz
7.70
7.69
7.68
7.27
7.26
7.25
3.07
3.06
3.05
2.33
1.18
1.00
0.99
0.98
3m
CH2Cl2
1.83
2.04
2.00
2.87
12.29
1.95
f1 (ppm)

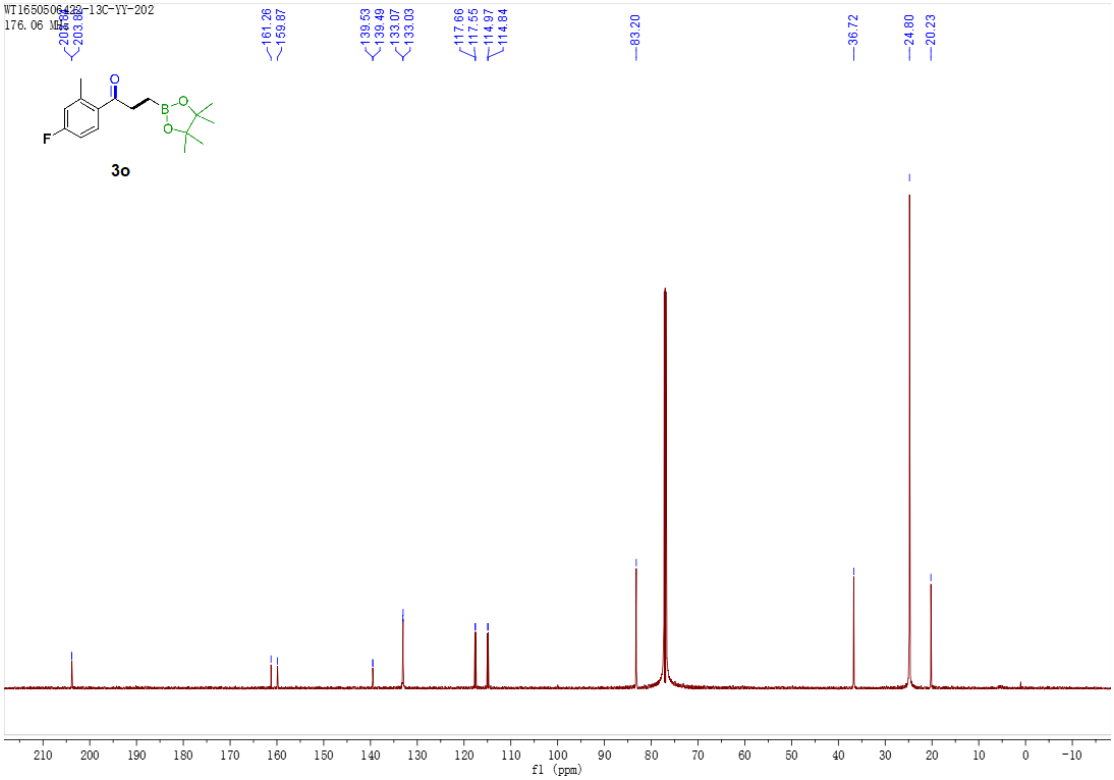

YY-188
176.06 MHz
200.77
138.20
137.02
133.49
128.52
128.33
125.23
83.08
33.72
25.03
24.78
21.36
3m
CH2Cl2
f1 (ppm)

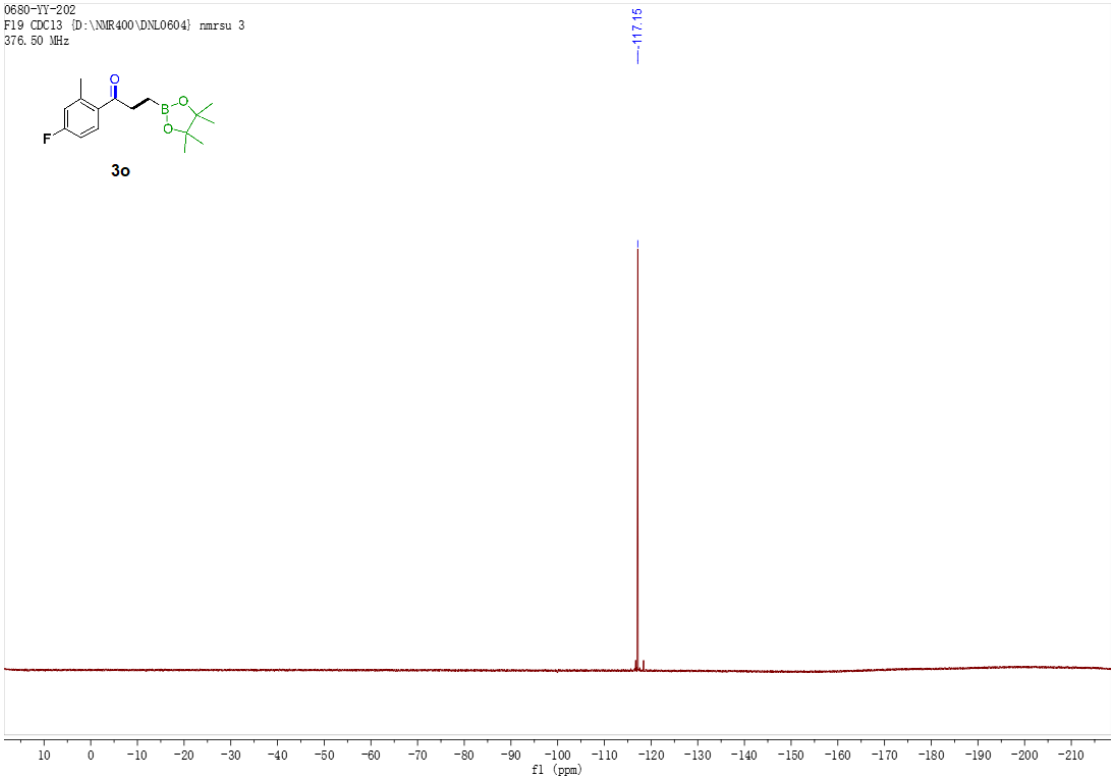

YY-186
700.17 MHz
7.57
7.26
7.17
3.13
3.12
3.11
2.36
1.26
1.07
1.06
1.05
3n
1.90
0.96
2.00
6.05
12.44
2.01
f1 (ppm)

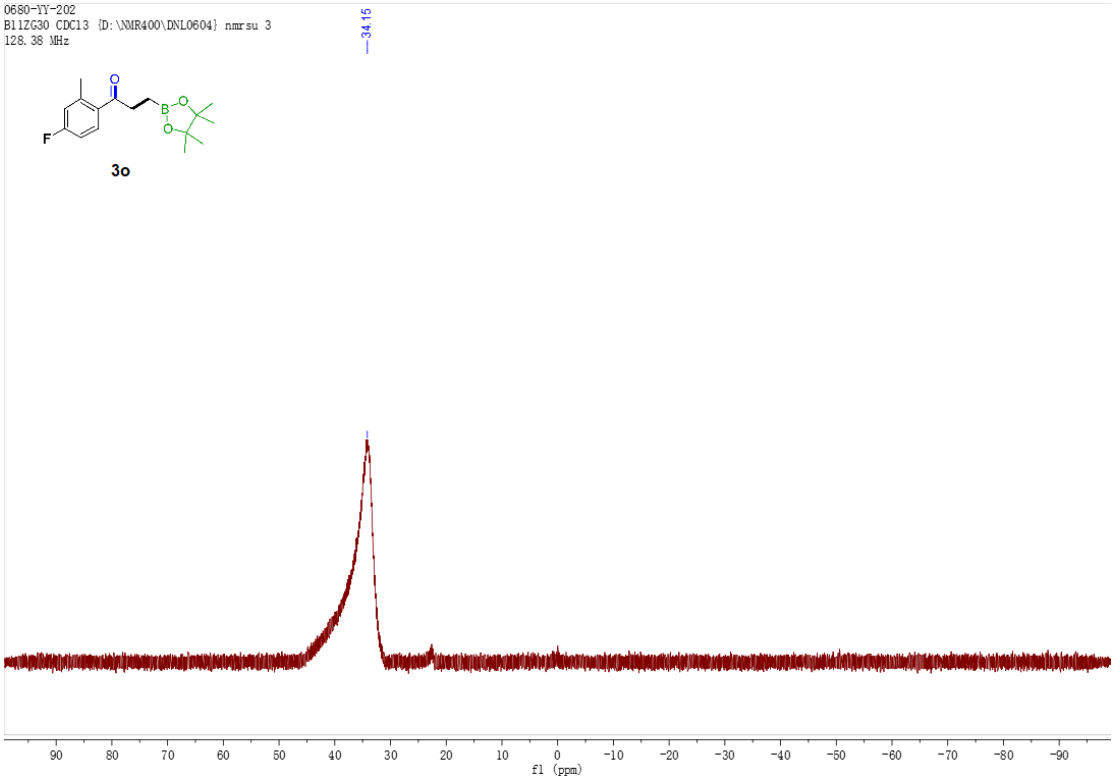

YY-186
176.06 MHz
200.98
138.04
137.11
134.36
125.80
83.07
33.75
24.79
21.24
3n
f1 (ppm)

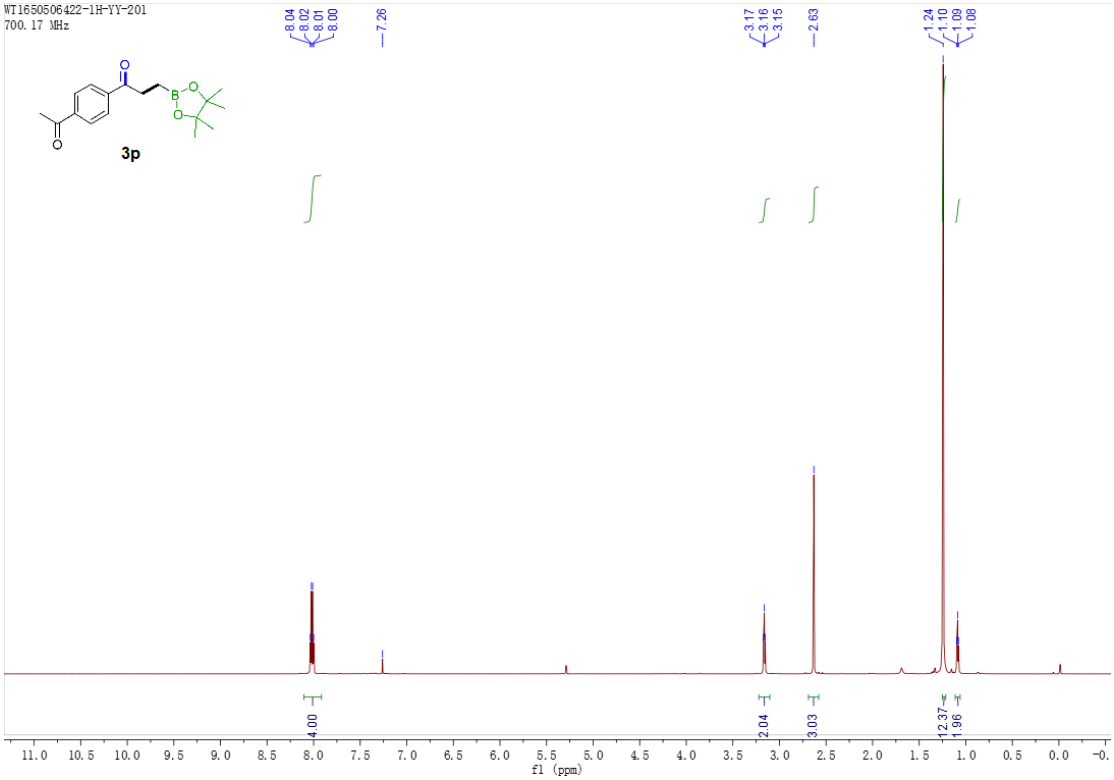

0692-YY-186
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 9
128.38 MHz
34.30
3n
f1 (ppm)

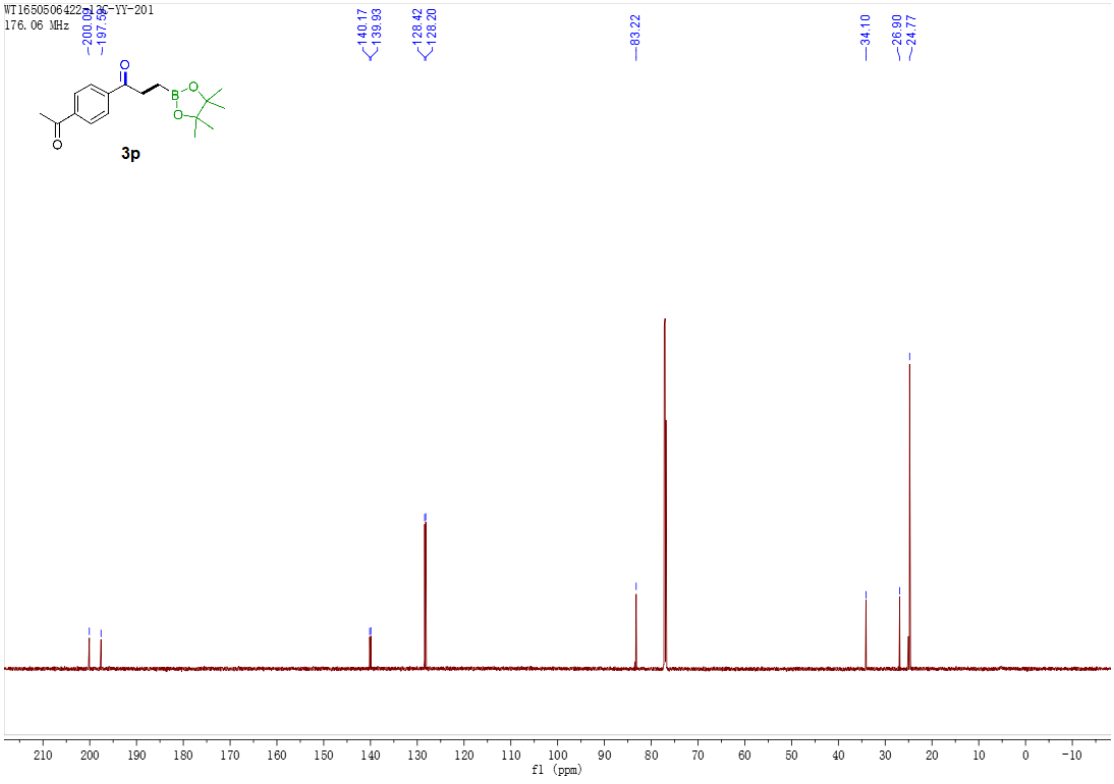

WT1650506422-1H-YY-202
700.17 MHz
7.31
7.30
7.26
7.18
7.17
7.16
7.05
7.03
7.02
3.01
3.00
2.99
2.41
1.25
1.06
1.05
1.04
3o
0.96
0.97
0.99
2.00
2.94
12.17
2.07
f1 (ppm)

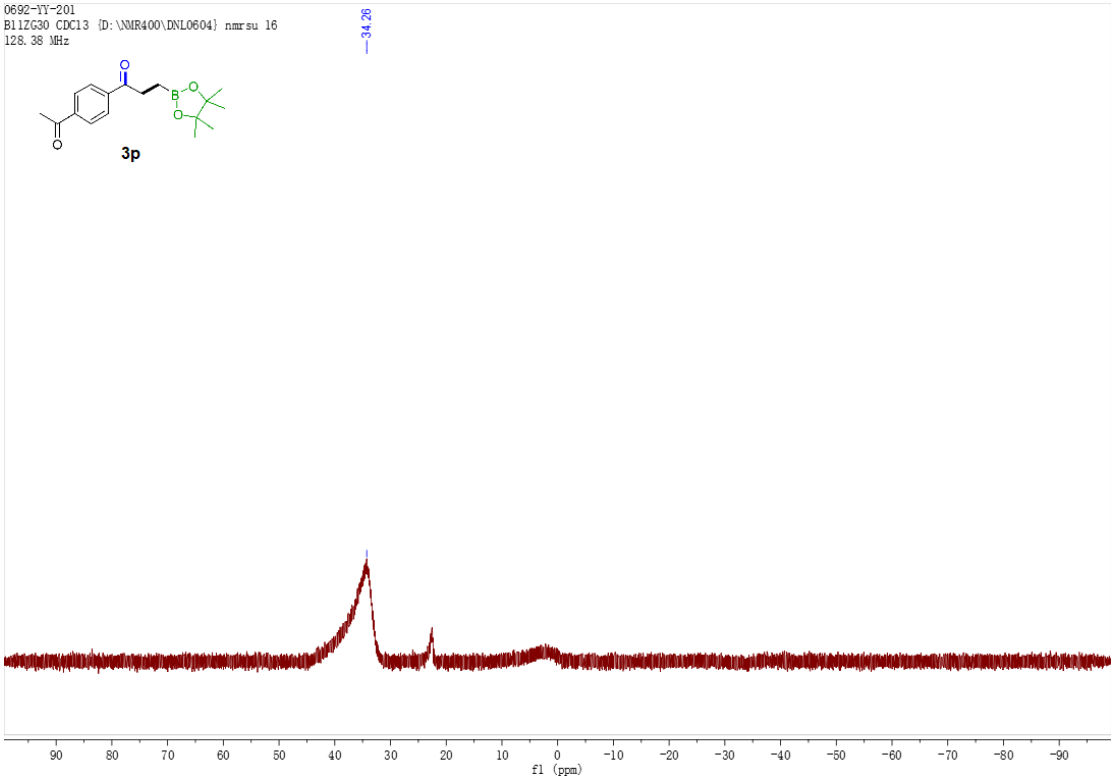

WT1650506422-13C-YY-202
176.06 MHz
161.26
159.87
139.53
139.49
133.07
133.03
117.66
117.55
114.97
114.84
83.20
36.72
24.80
20.23
3o
f1 (ppm)

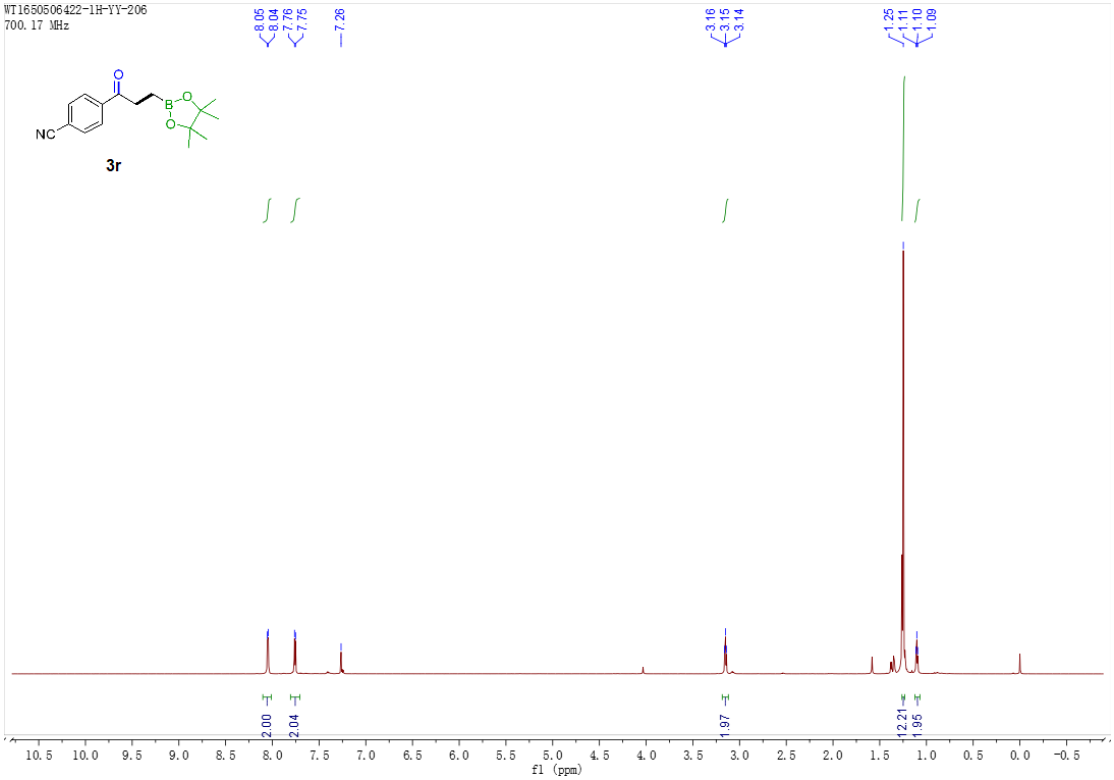

0680-YY-202
F19 CDCl3 {D:\NMR400\DNL0604} nmrsu 3
376.50 MHz
-117.15
3o
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

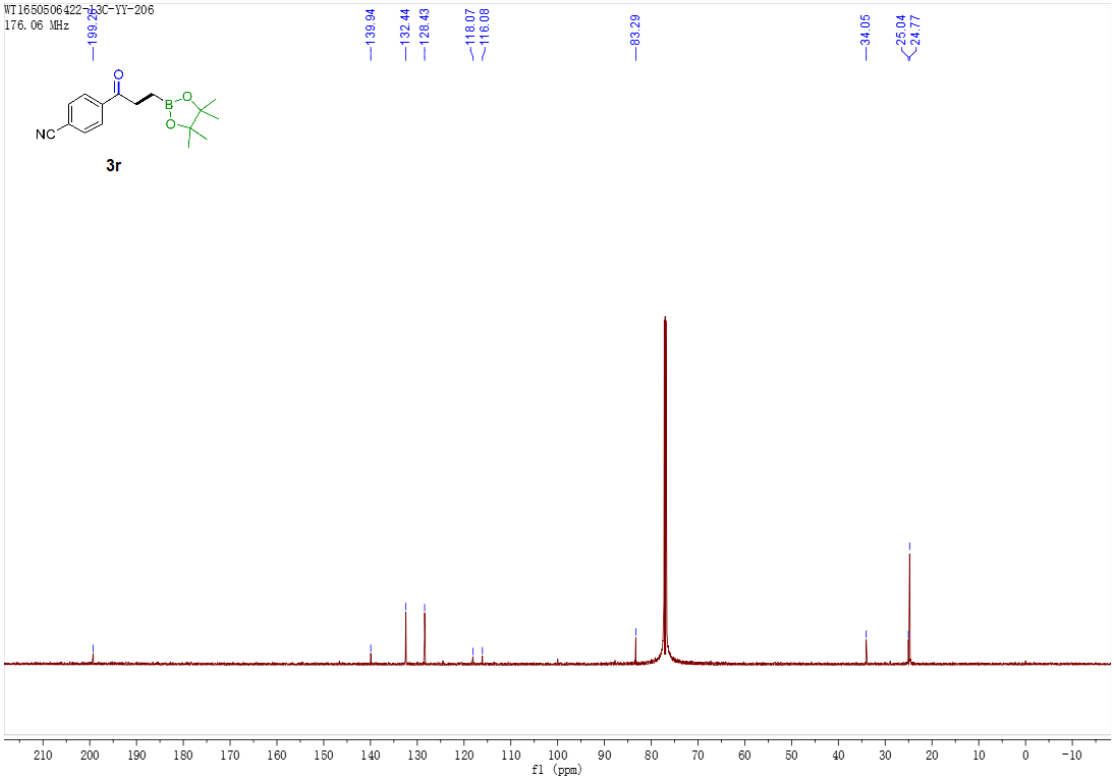

0680-YY-202
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 3
128.38 MHz
34.15
3o
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

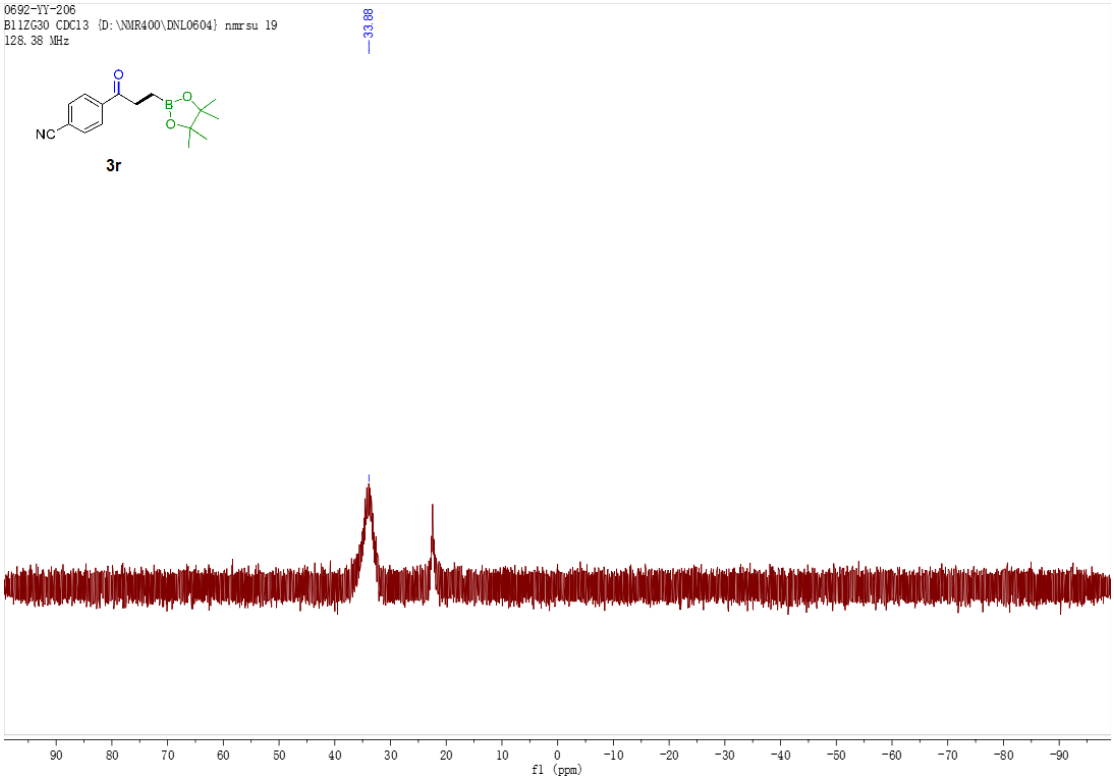

WT1650506422-1H-YY-201
700.17 MHz
8.04
8.02
8.01
8.00
7.26
3.17
3.16
3.15
2.63
1.24
1.10
1.09
1.08
3p
4.00
2.04
3.03
12.37
1.96
f1 (ppm)

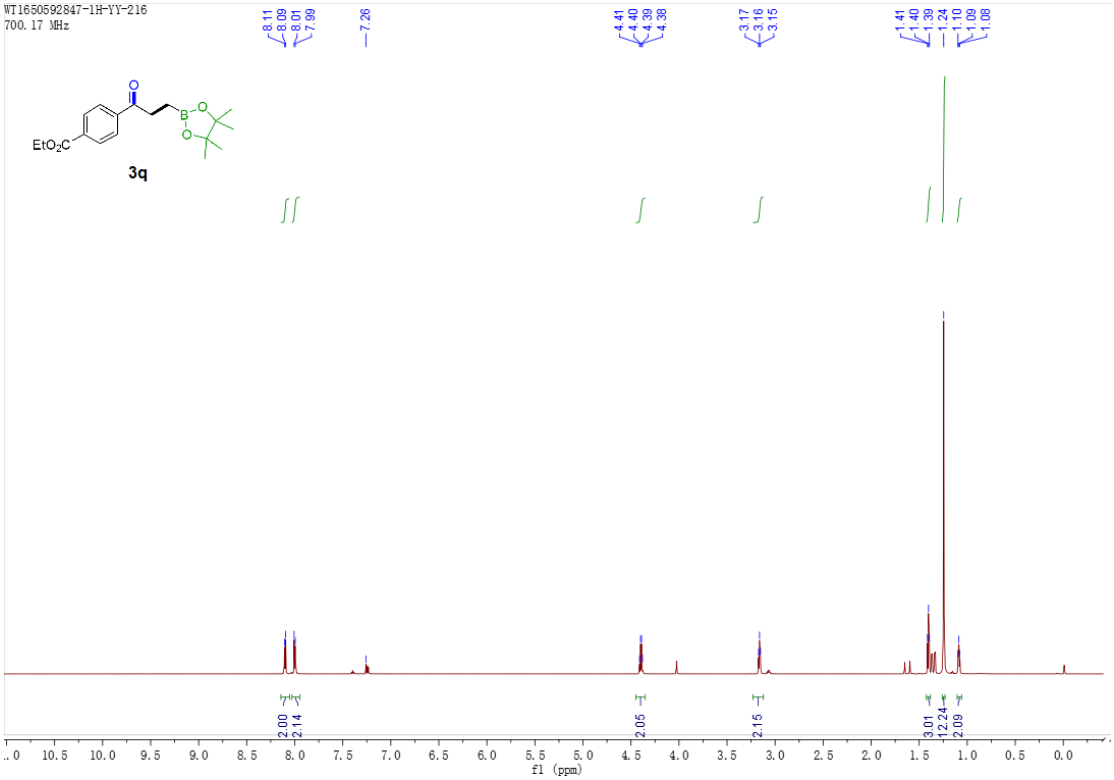

WT1650506422-13C-YY-201
176.06 MHz
200.02
197.36
140.17
139.93
128.42
128.20
83.22
34.10
26.90
24.77
3p
f1 (ppm)

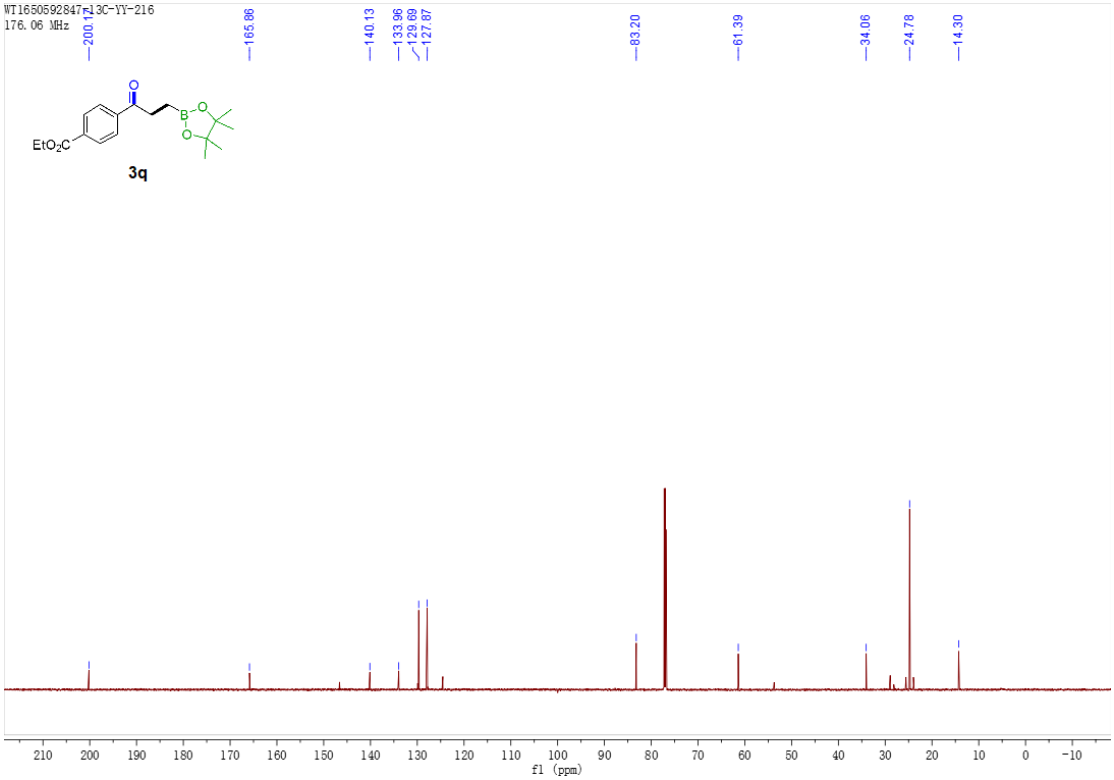

0692-YY-201
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 16
128.38 MHz
34.26
3p
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

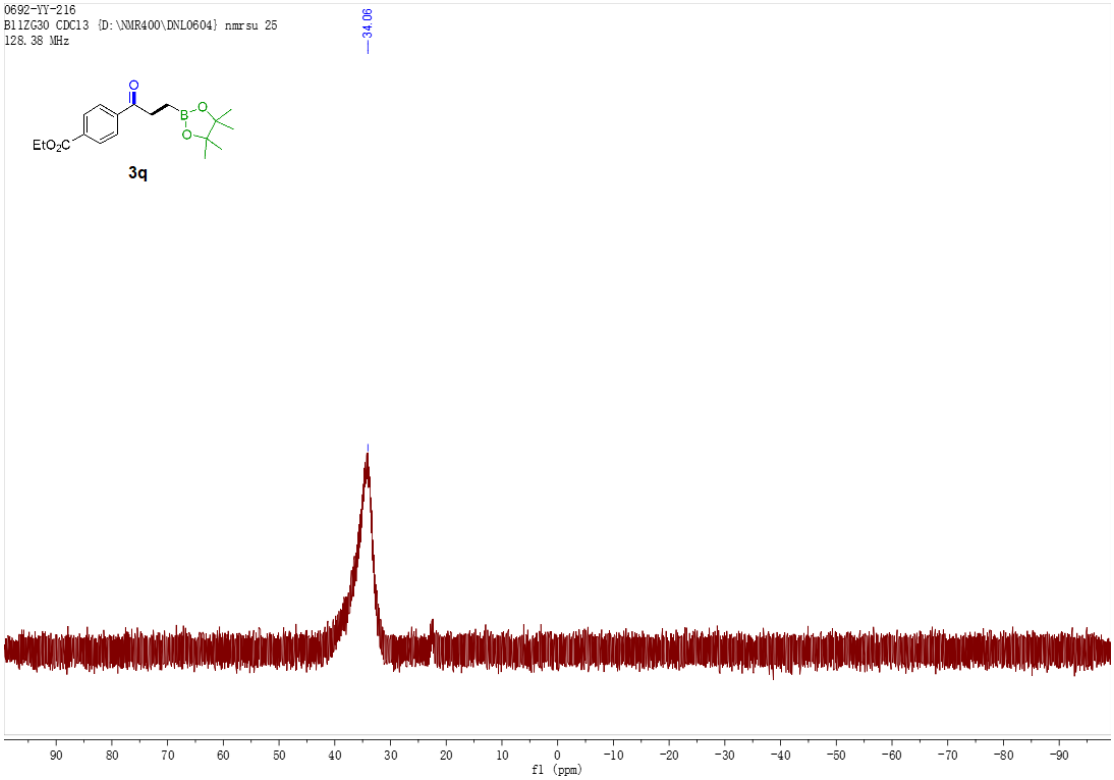

WT1650506422-1H-YY-206
700.17 MHz
8.05
8.04
7.76
7.75
7.26
3.16
3.15
3.14
1.25
1.11
1.10
1.09
NC
3r
2.00
2.04
1.97
12.21
1.95
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

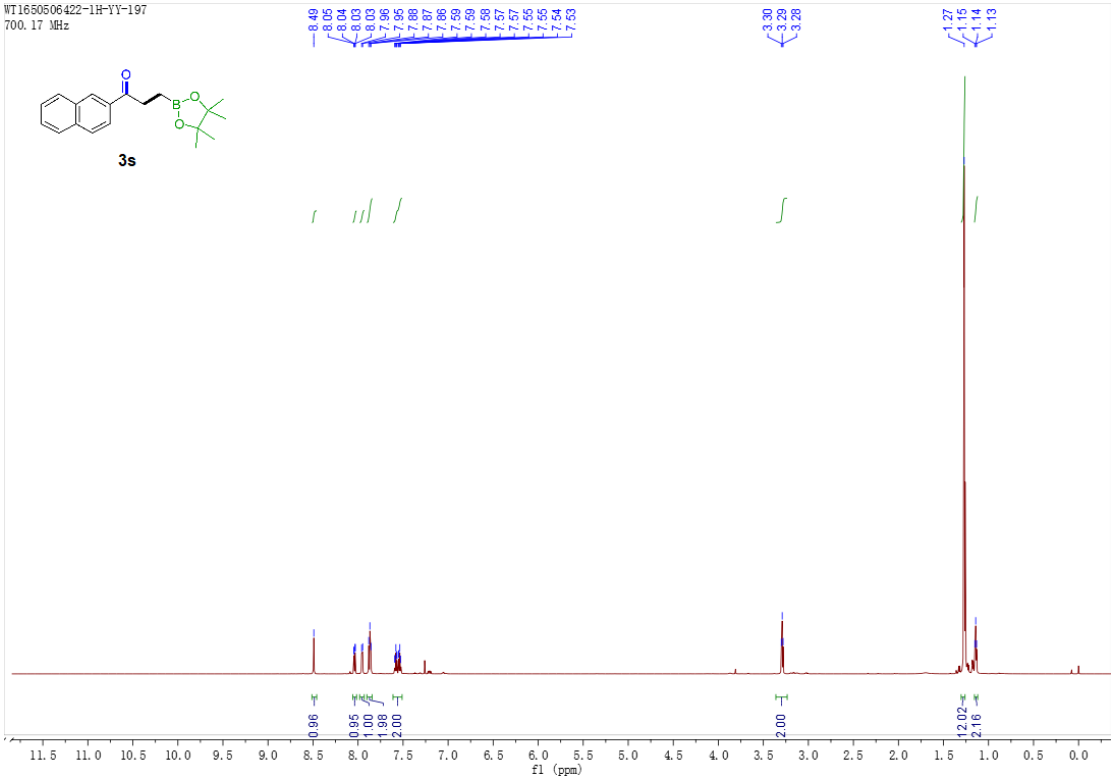

WT1650506422-13C-YY-206
176.06 MHz
199.29
139.94
132.44
128.43
118.07
116.08
83.29
34.05
25.04
24.77
NC
3r
f1 (ppm)

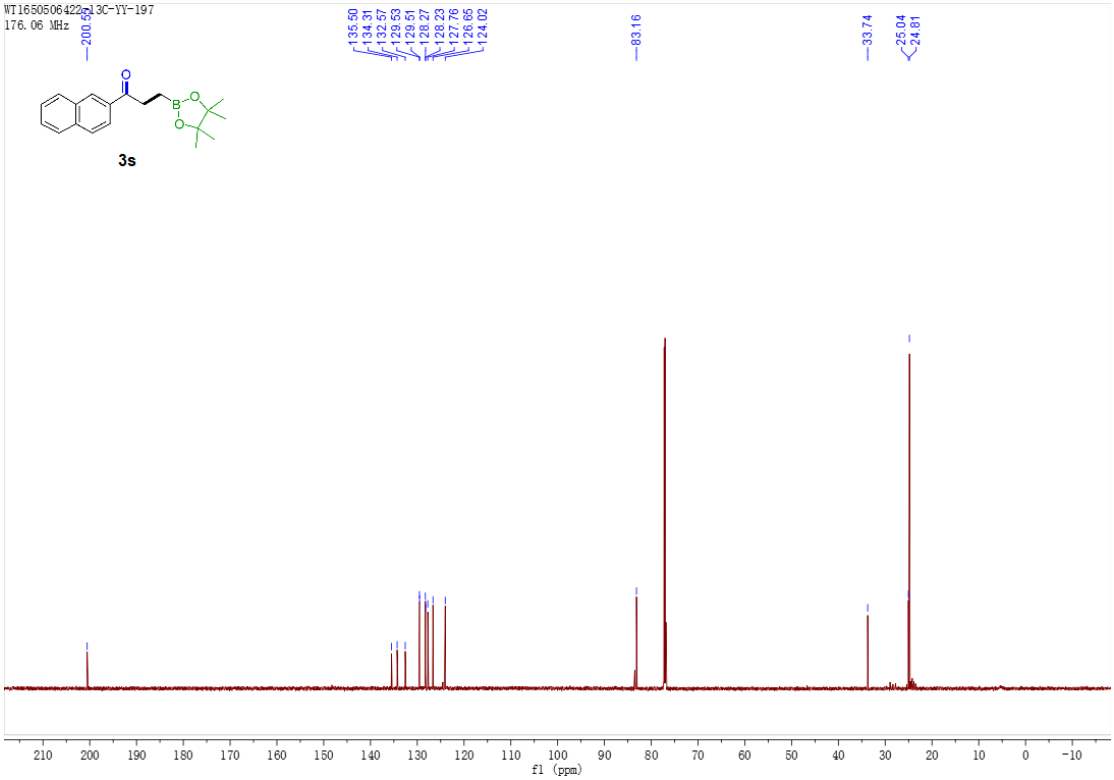

0692-YY-206
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 19
128.38 MHz
33.88
NC
3r
f1 (ppm)

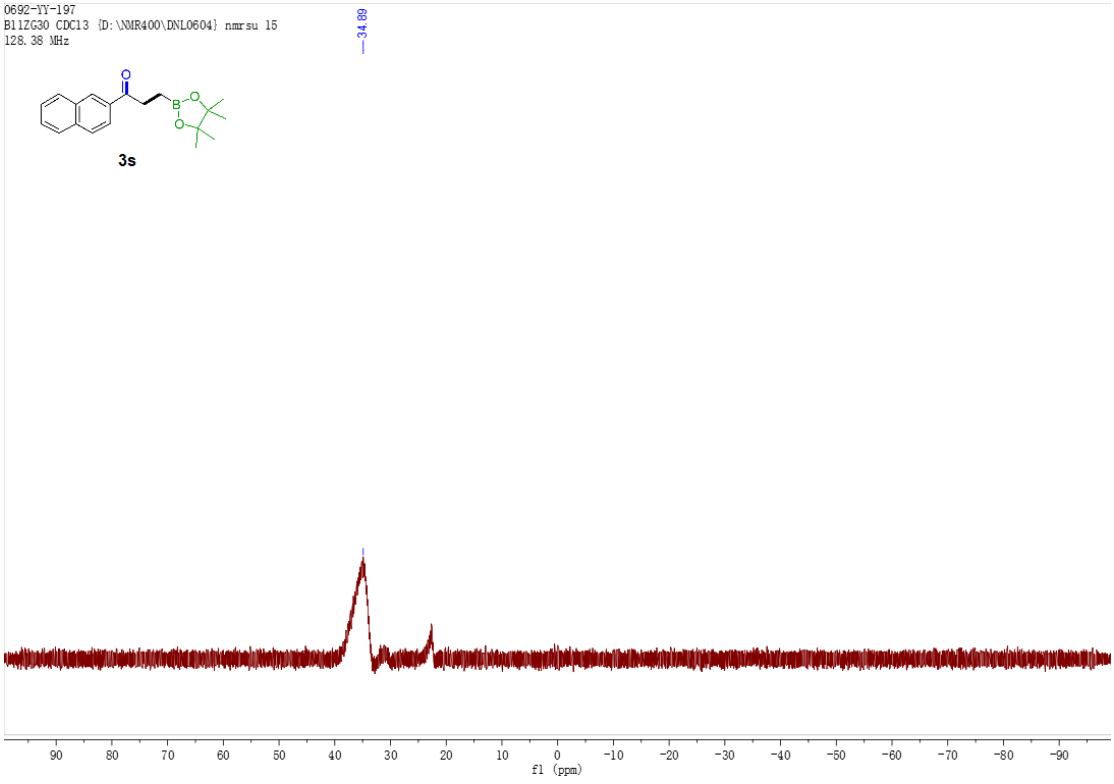

WT1650592847-1H-YY-216
700.17 MHz
8.11
8.09
8.01
7.99
7.26
4.41
4.40
4.39
4.38
3.17
3.16
3.15
1.41
1.40
1.39
1.24
1.10
1.09
1.08
EtO2C
3q
2.00
2.14
2.05
2.15
3.01
12.24
2.09
f1 (ppm)

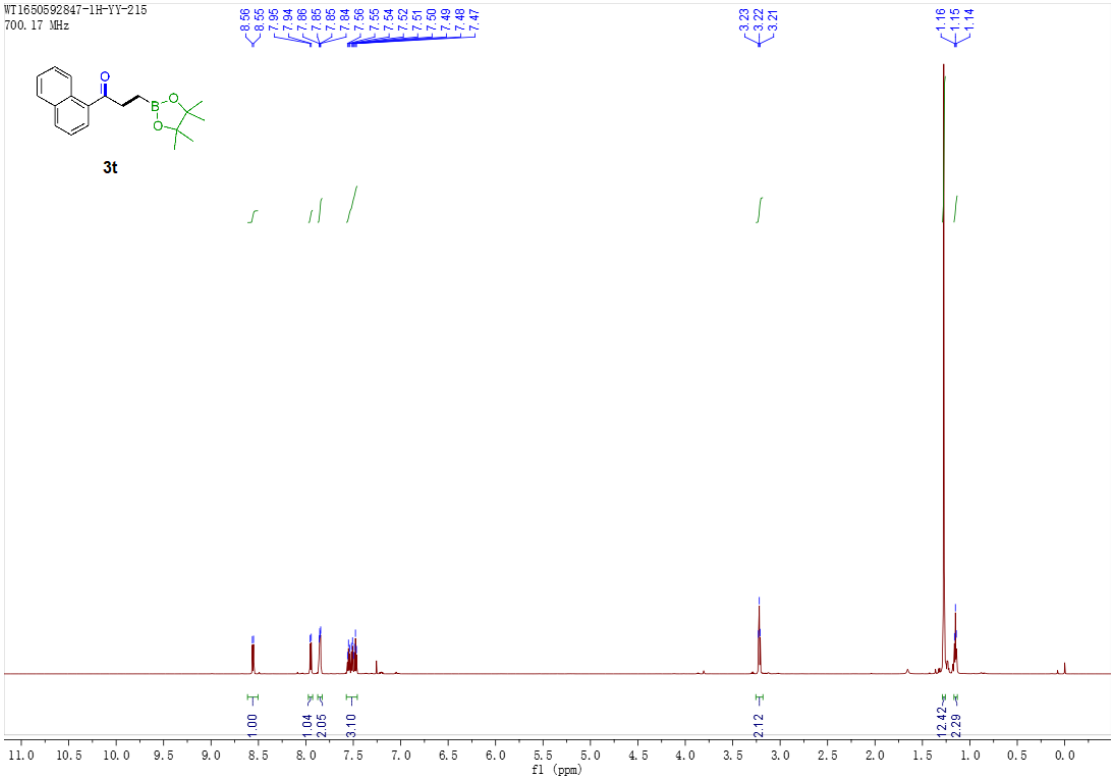

WT1650592847-13C-YY-216
176.06 MHz
200.17
165.86
140.13
133.96
129.69
127.87
83.20
61.39
34.06
24.78
14.30
EtO2C
3q
f1 (ppm)

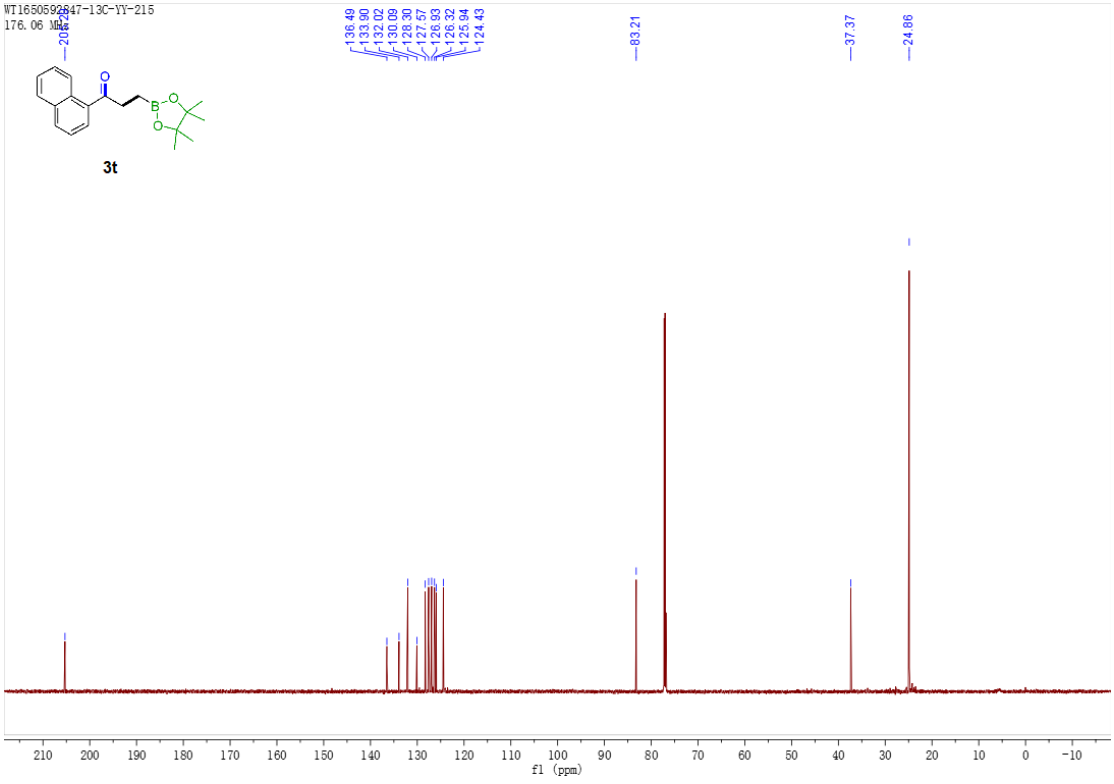

0692-YY-216
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 25
128.38 MHz
34.06
EtO2C
3q
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

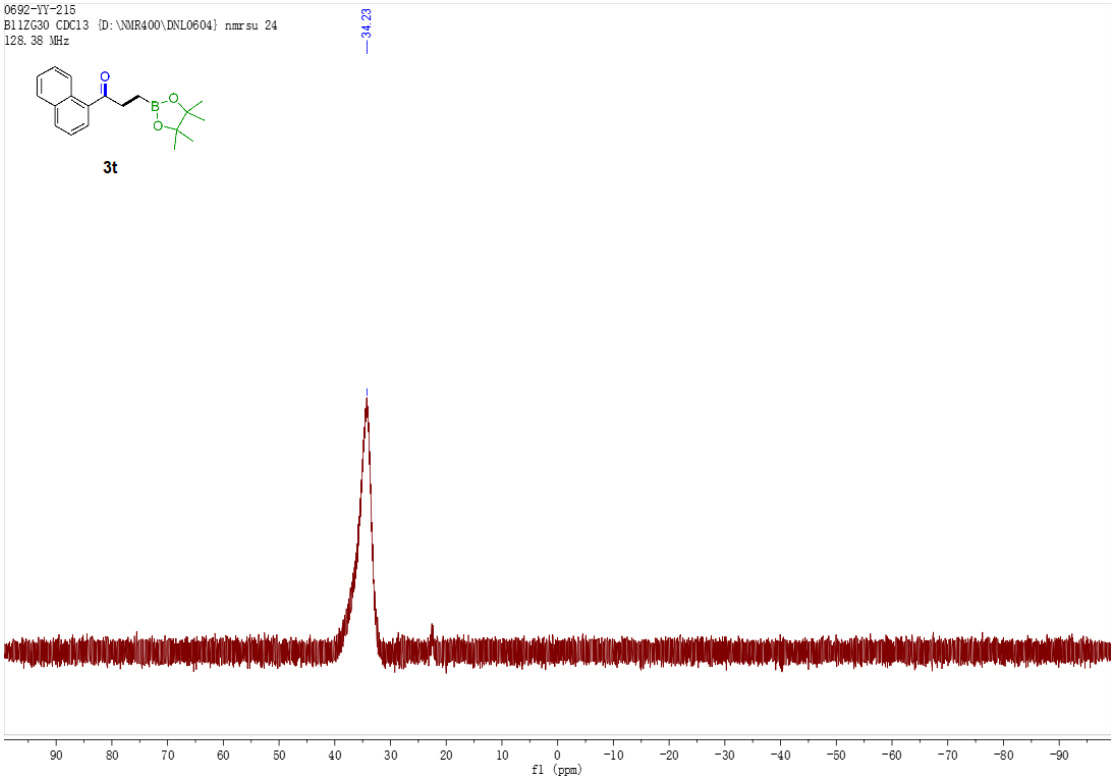

WT1650506422-1H-YY-197
700.17 MHz
8.49
8.05
8.04
8.03
8.03
7.96
7.95
7.88
7.87
7.86
7.59
7.59
7.58
7.57
7.57
7.55
7.55
7.54
7.53
3.30
3.29
3.28
1.27
1.15
1.14
1.13
3s
0.96
0.95
1.00
1.98
2.00
2.00
12.02
2.16
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

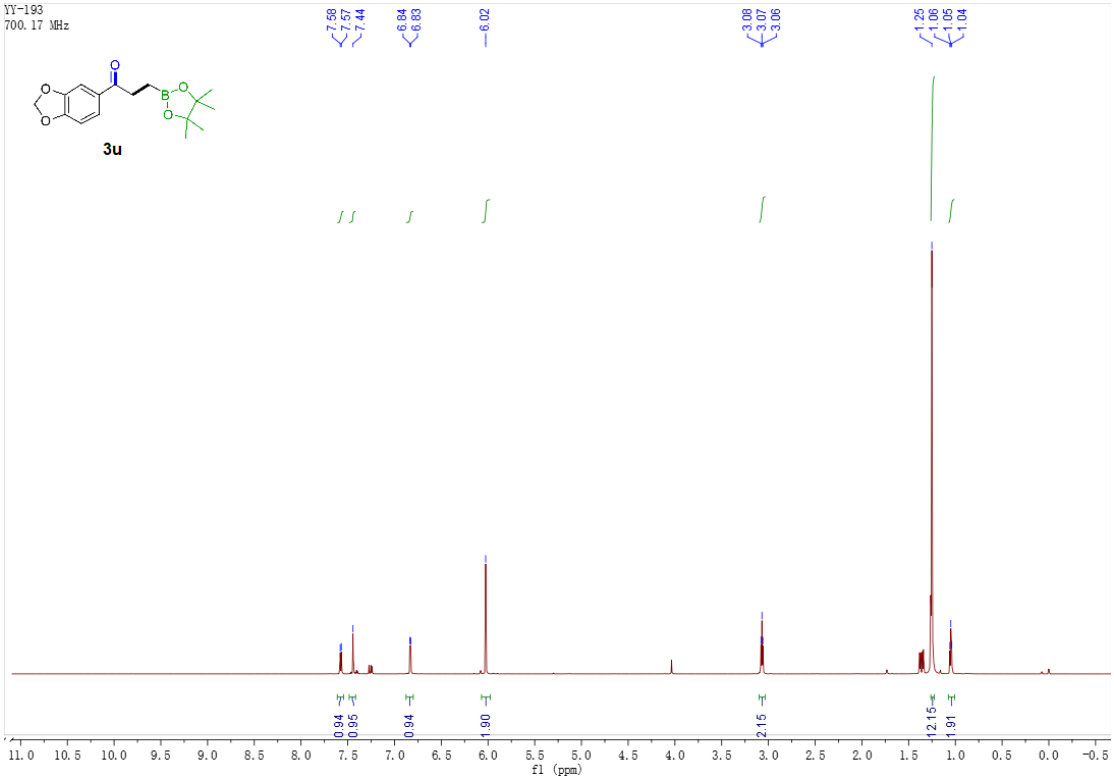

WT1650506422-13C-YY-197
176.06 MHz
200.65
135.50
134.31
132.57
129.53
129.51
128.27
128.23
127.76
126.65
124.02
83.16
33.74
25.04
24.81
3s
f1 (ppm)

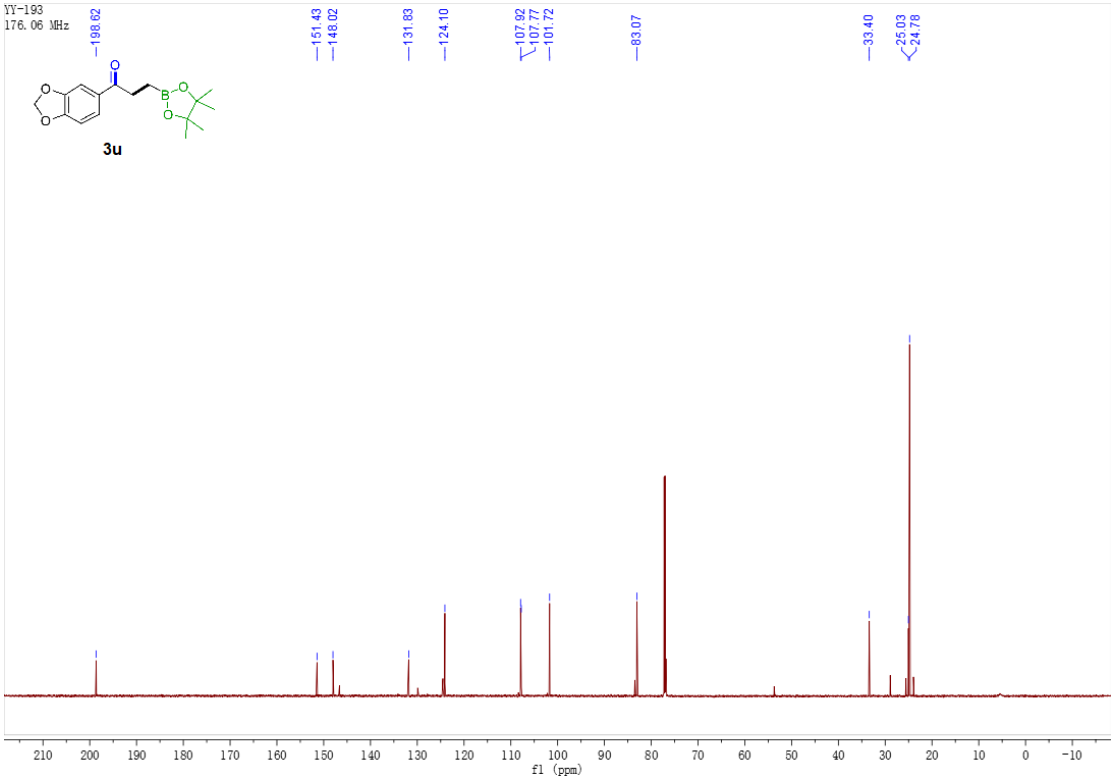

0692-YY-197
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 15
128.38 MHz
34.89
3s
f1 (ppm)

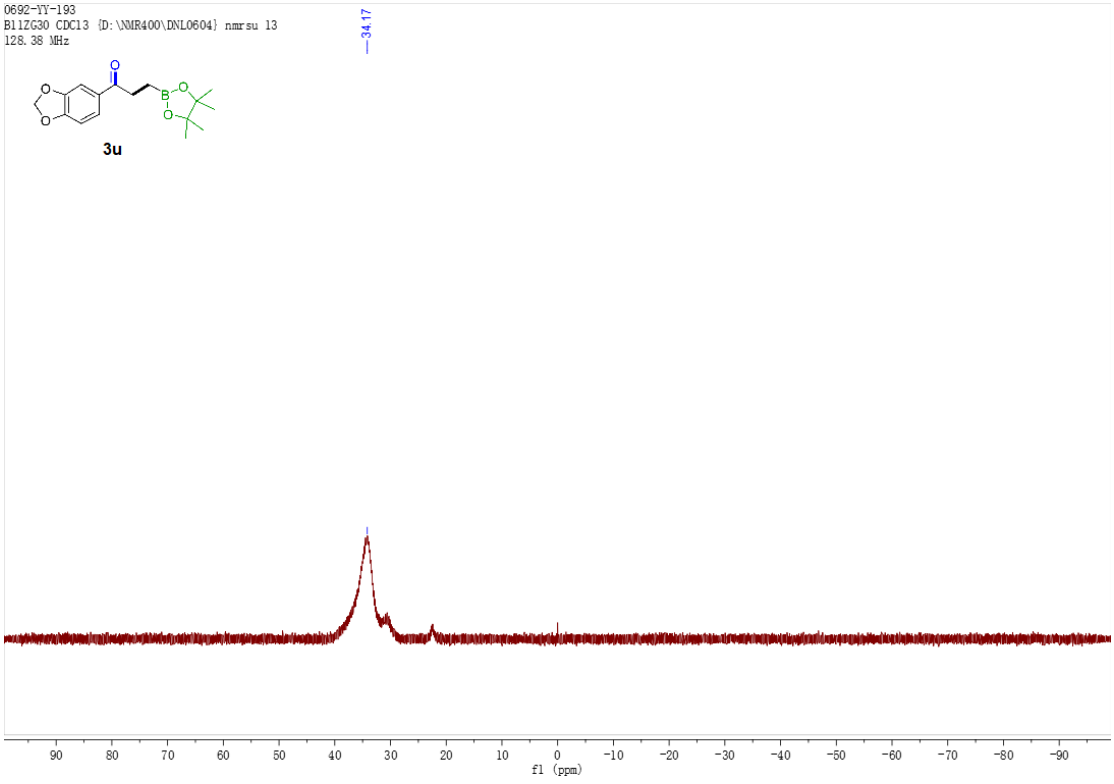

WT1650592847-1H-YY-215
700.17 MHz
8.56
8.55
7.95
7.94
7.86
7.85
7.85
7.84
7.56
7.55
7.54
7.52
7.51
7.50
7.49
7.48
7.47
3.23
3.22
3.21
1.16
1.15
1.14
3t
1.00
1.04
2.05
3.10
2.12
12.42
2.29
f1 (ppm)

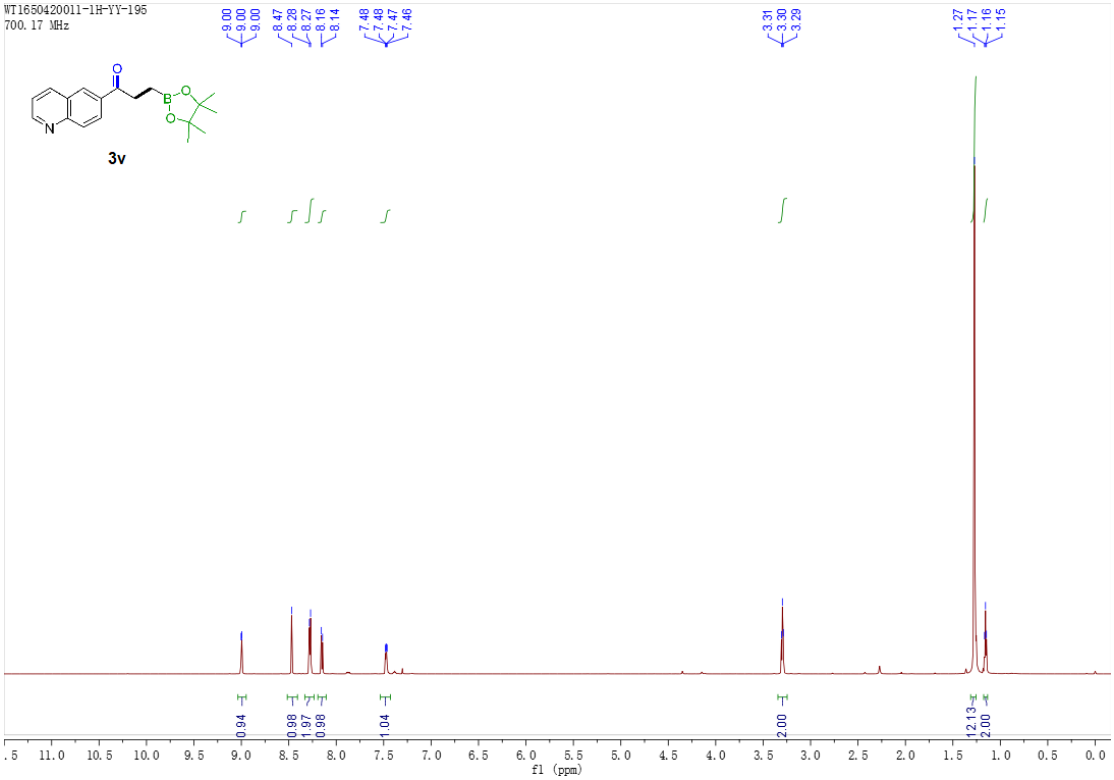

WT1650592847-13C-YY-215
176.06 MHz
136.49
133.90
132.02
130.09
128.30
127.67
126.93
126.32
125.94
124.43
83.21
37.37
24.86
3t
f1 (ppm)

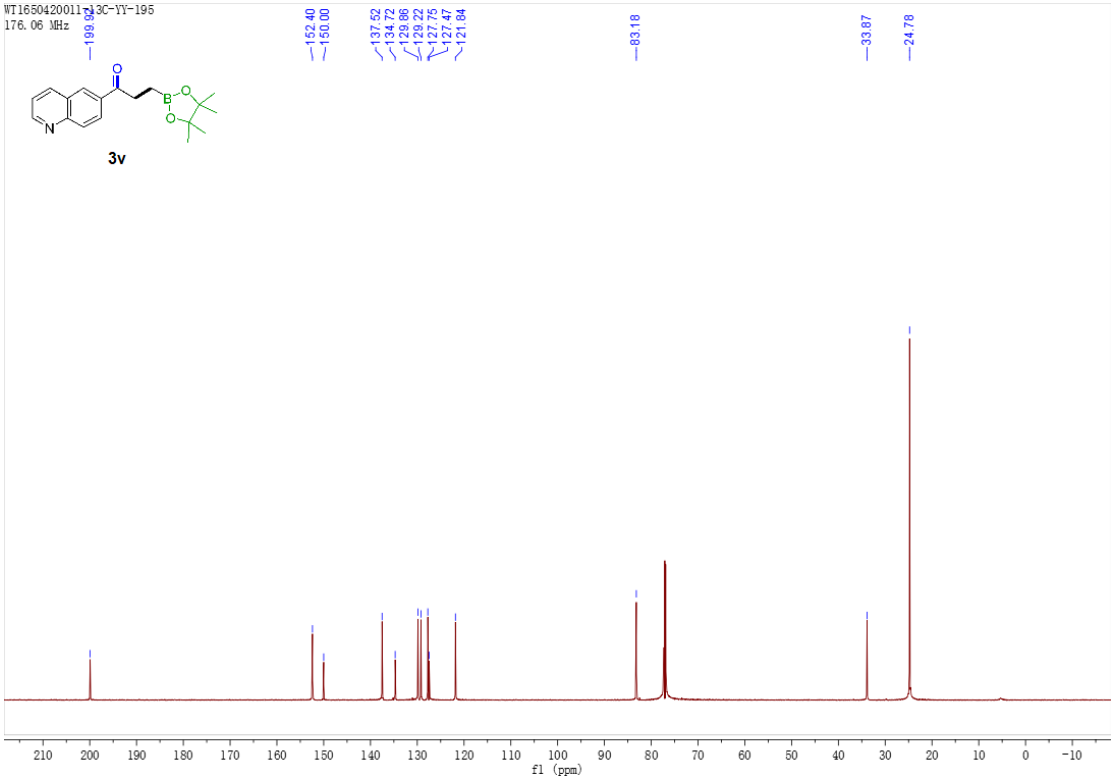

0692-YY-215
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 24
128.38 MHz
34.23
3t
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

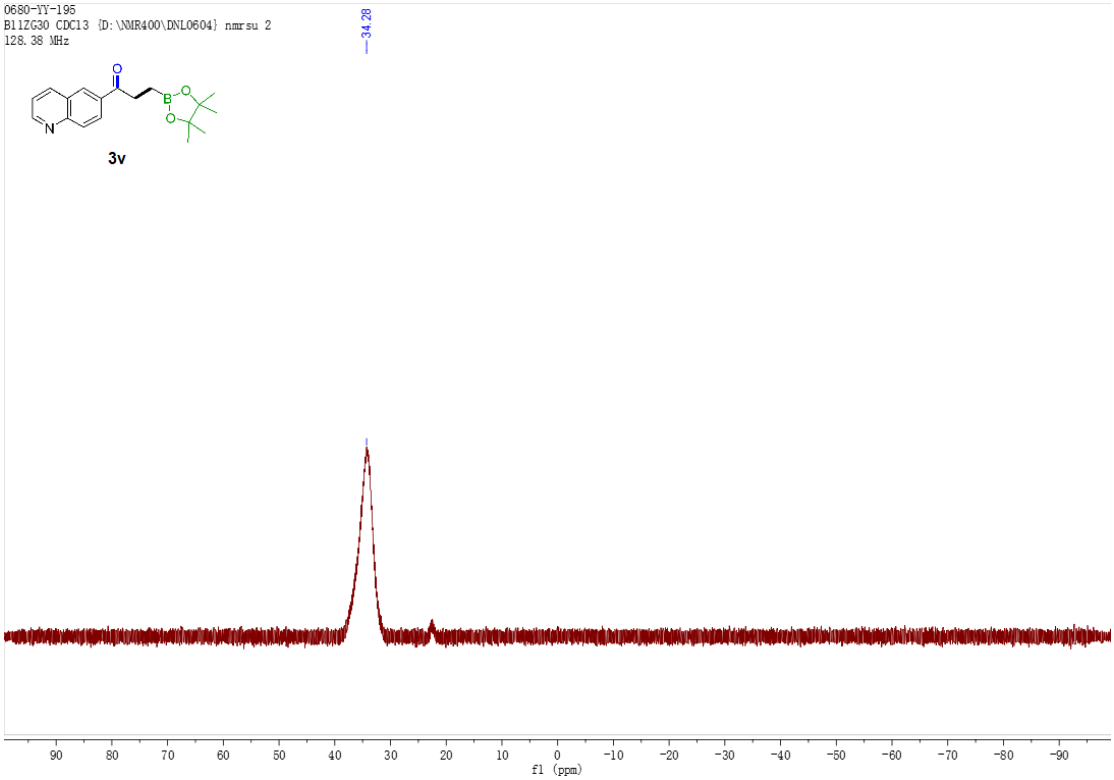

YY-193
700.17 MHz
7.58
7.57
7.44
6.84
6.83
6.02
3.08
3.07
3.06
1.25
1.06
1.05
1.04
3u
0.94
0.95
0.94
1.90
2.15
12.15
1.91
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

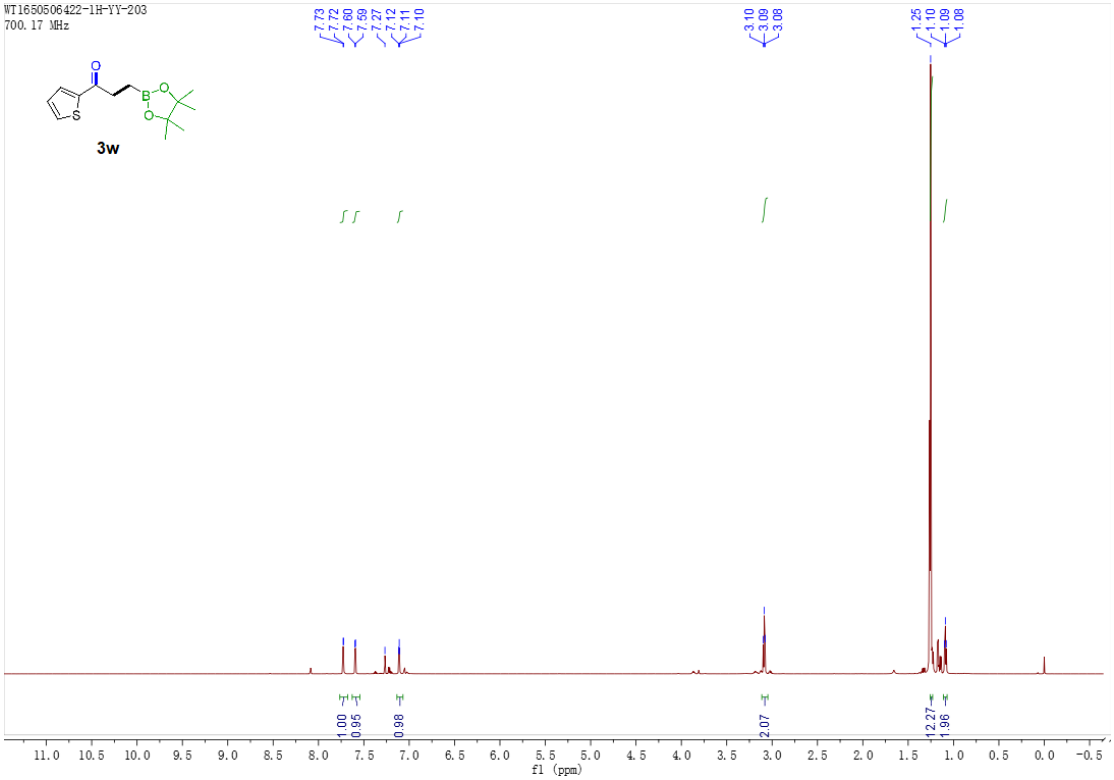

YY-193
176.06 MHz
198.62
151.43
148.02
131.83
124.10
107.92
107.77
101.72
83.07
33.40
25.03
24.78
3u
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

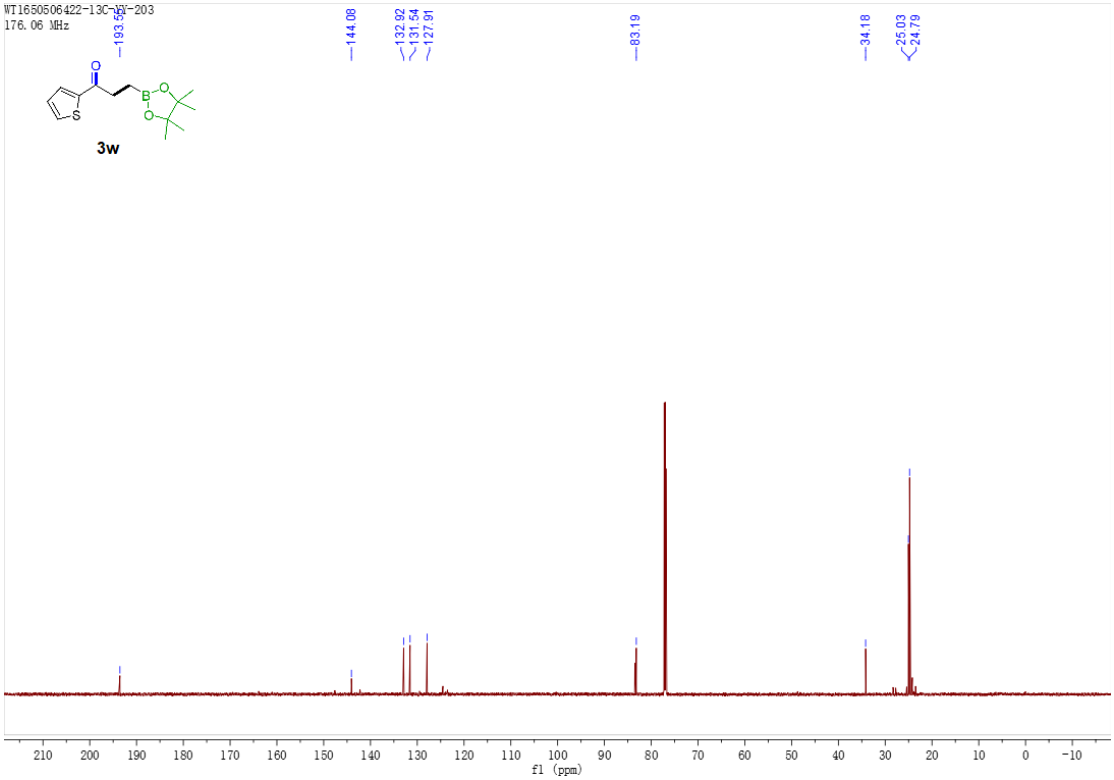

0692-YY-193
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 13
128.38 MHz
34.17
3u
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

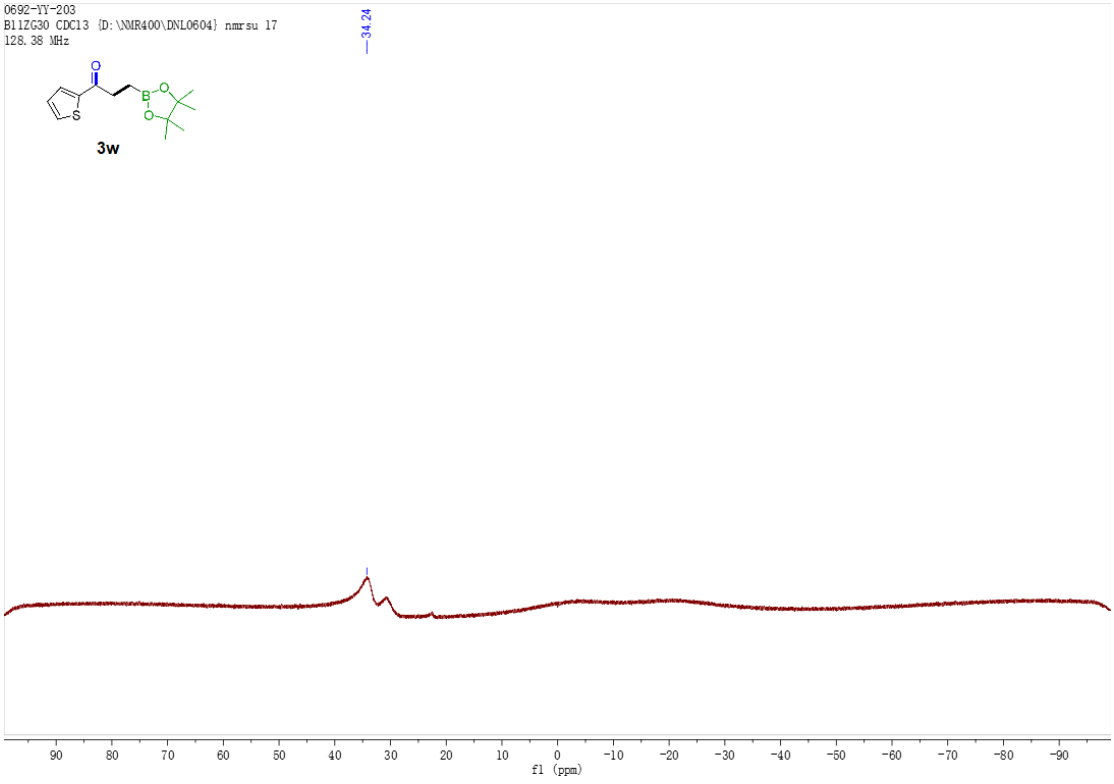

WT1650420011-1H-YY-195
700.17 MHz
9.00
9.00
9.00
8.47
8.28
8.27
8.16
8.14
7.48
7.48
7.47
7.46
3.31
3.30
3.29
1.27
1.17
1.16
1.15
3v
0.94
0.98
1.97
0.98
1.04
2.00
12.13
2.00
f1 (ppm)

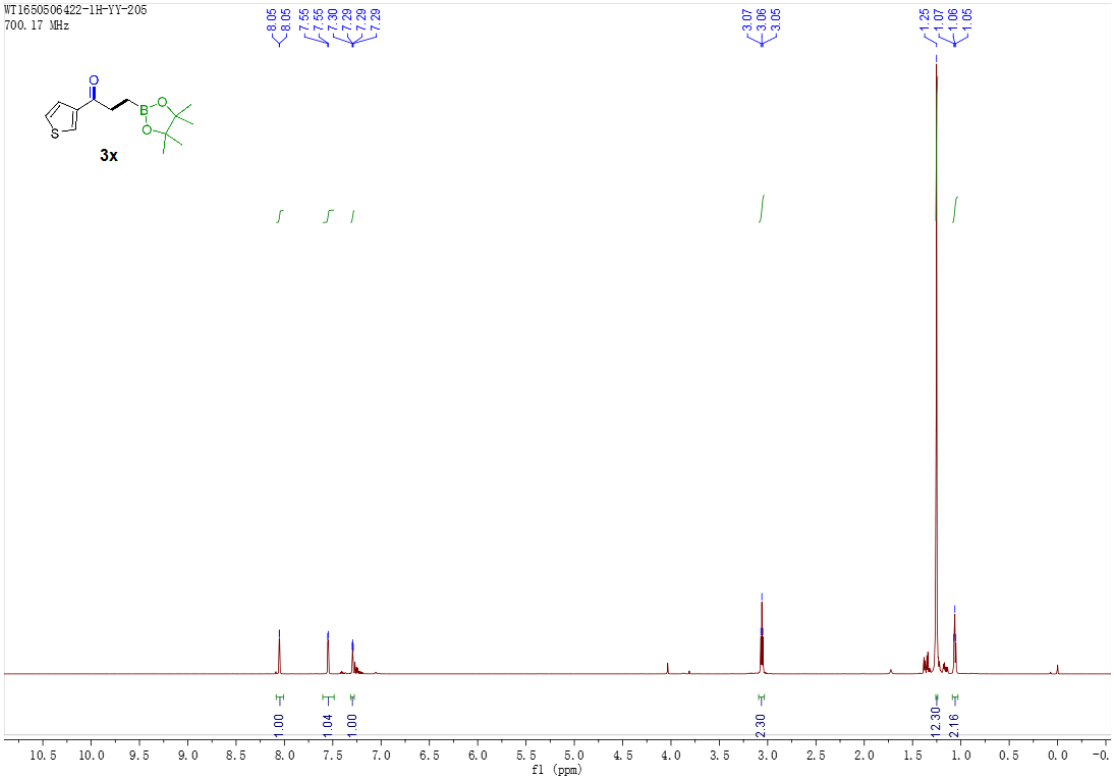

WT1650420011-13C-YY-195
176.06 MHz
199.59
152.40
150.00
137.52
134.72
129.86
129.22
127.75
127.47
121.84
83.18
33.87
24.78
3v
f1 (ppm)

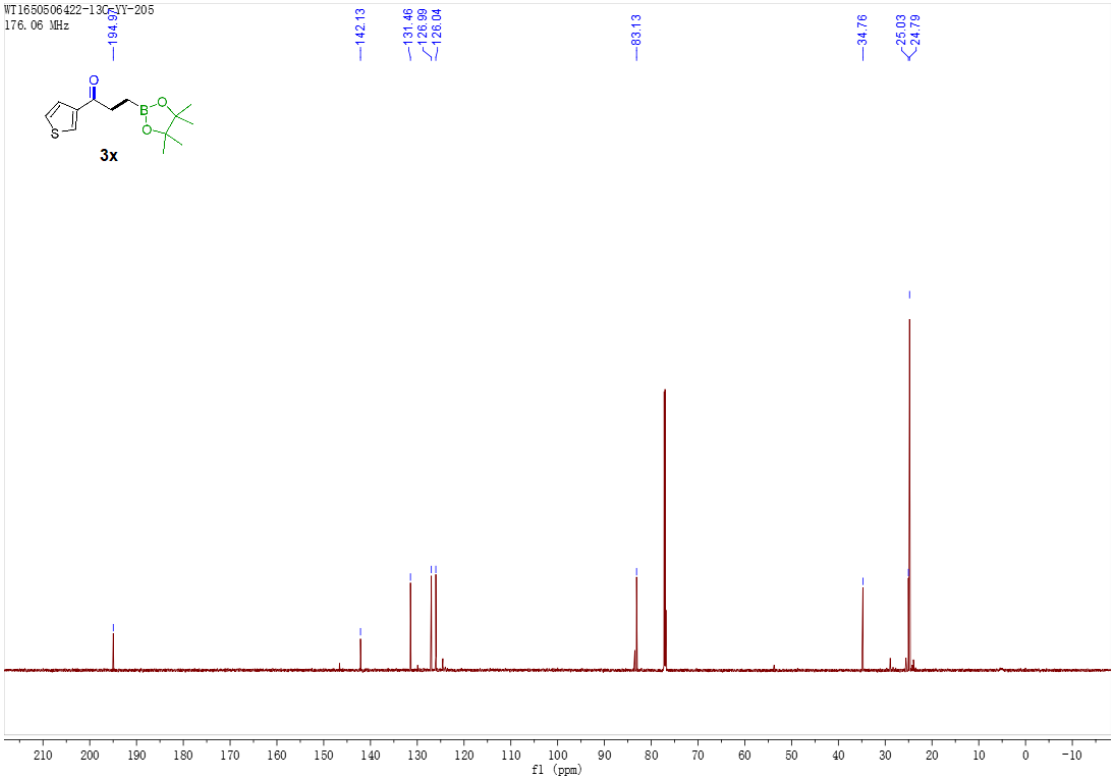

0680-YY-195
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 2
128.38 MHz
34.28
3v
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

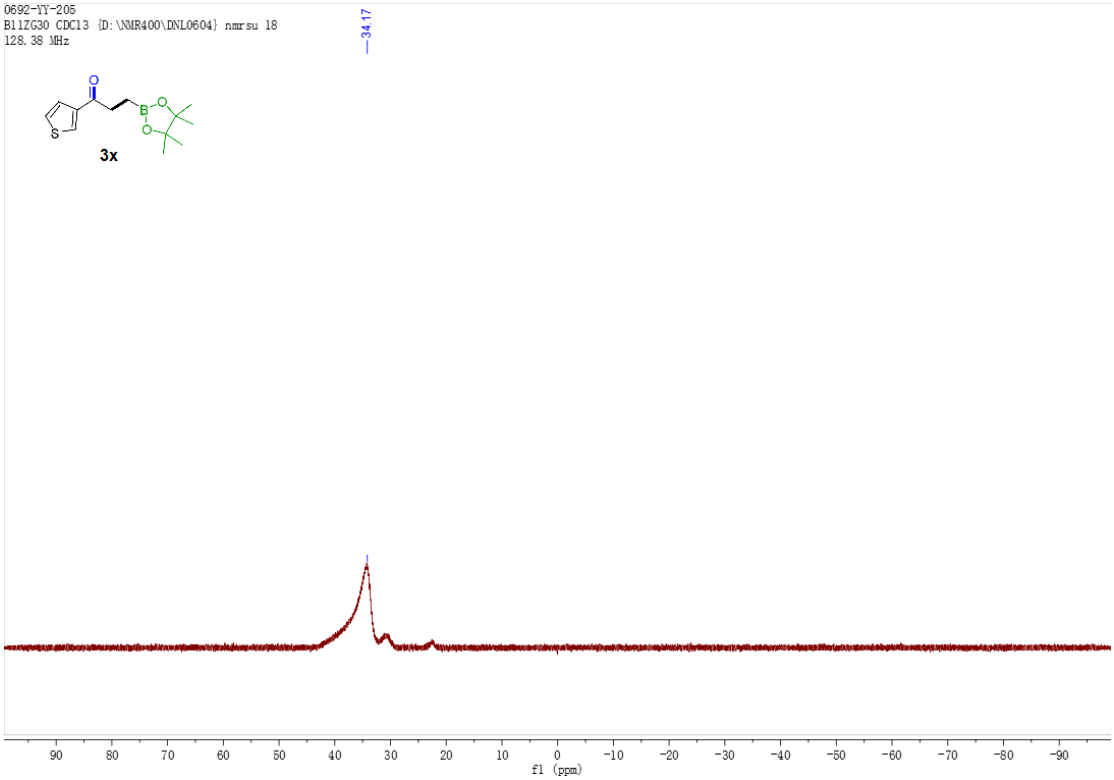

WT1650506422-1H-YY-203
700.17 MHz
7.73
7.72
7.60
7.59
7.27
7.12
7.11
7.10
3.10
3.09
3.08
1.25
1.10
1.09
1.08
3w
1.00
0.95
0.98
2.07
12.27
1.96
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

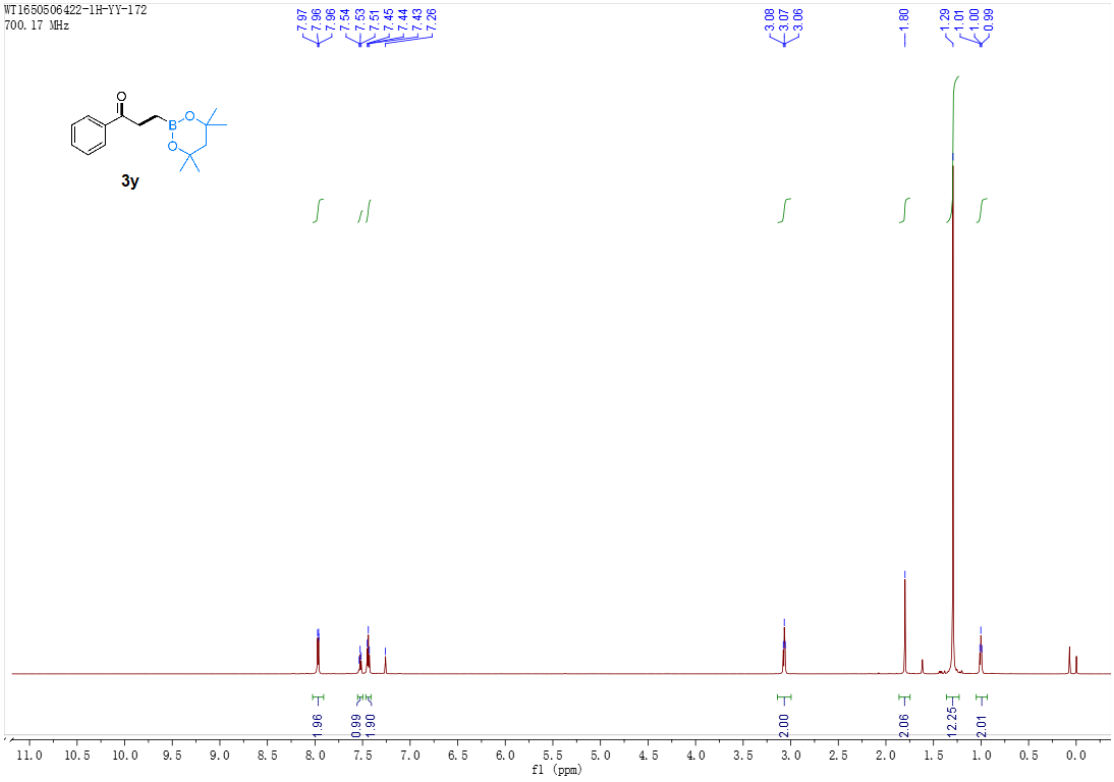

WT1650506422-13C-YY-203
176.06 MHz
193.52
144.08
132.92
131.54
127.91
83.19
34.18
25.03
24.79
3w
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

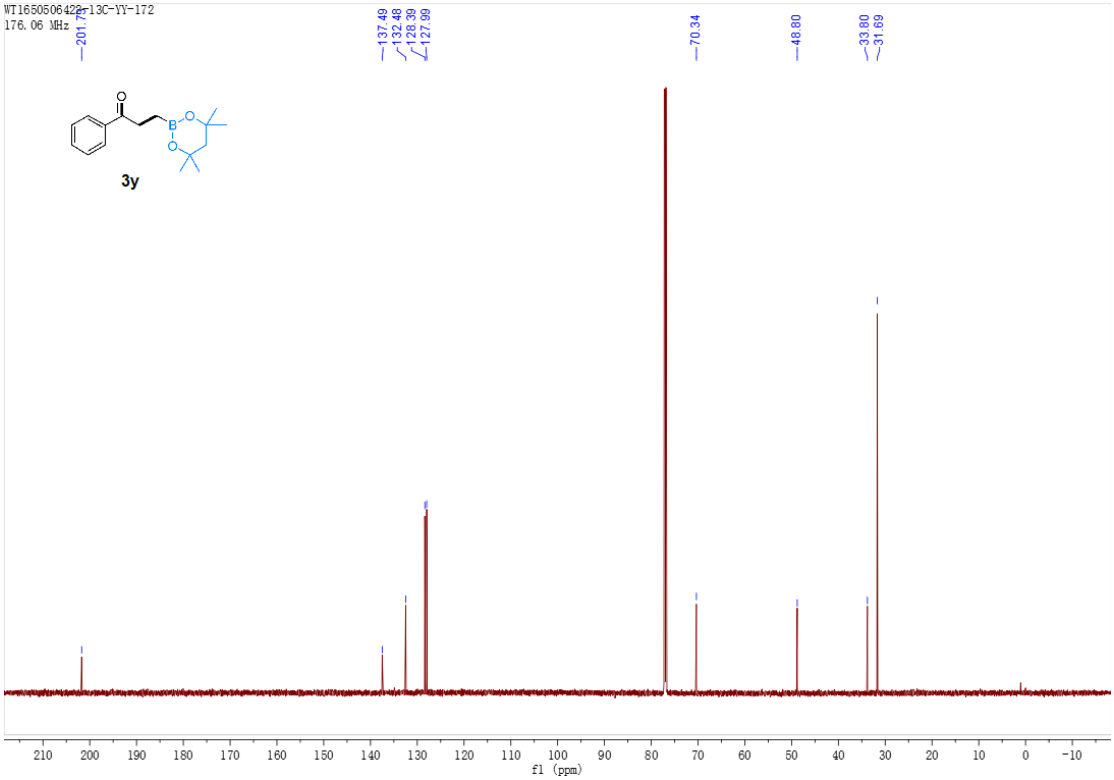

0692-YY-203
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 17
128.38 MHz
34.24
3w
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

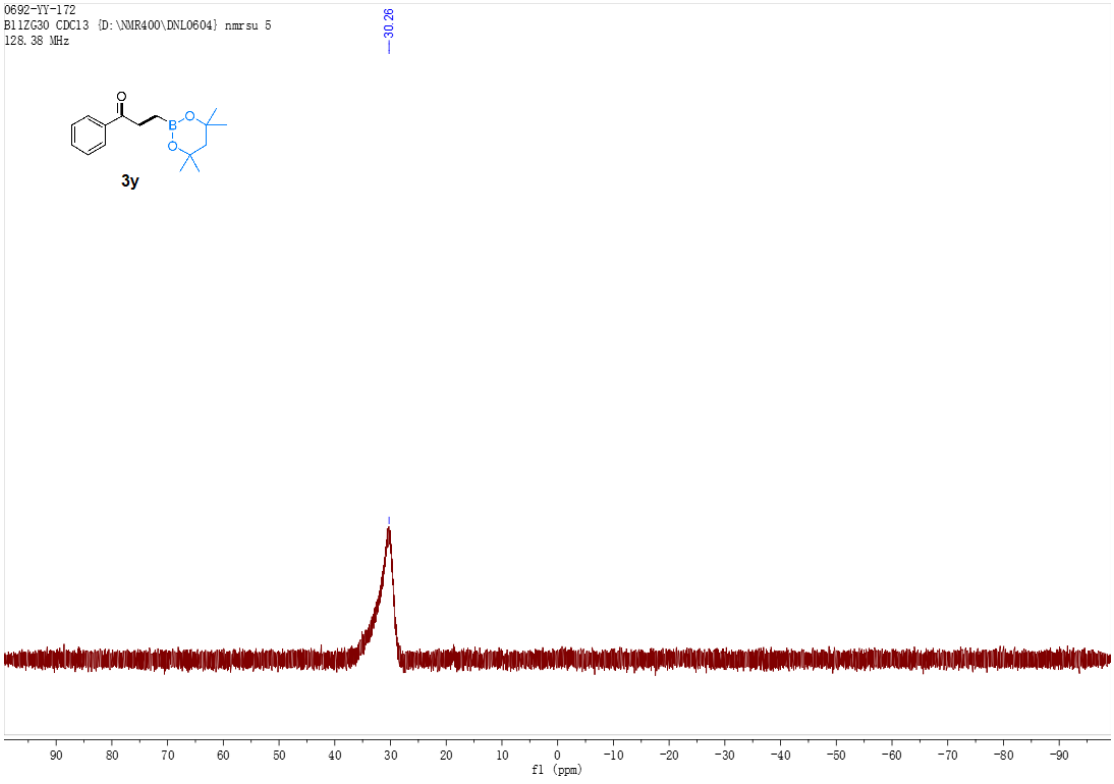

WT1650506422-1H-YY-205
700.17 MHz
8.05
8.05
7.55
7.55
7.30
7.29
7.29
7.29
3.07
3.06
3.05
1.25
1.07
1.06
1.05
3x
1.00
1.04
1.00
2.30
12.30
2.16
f1 (ppm)

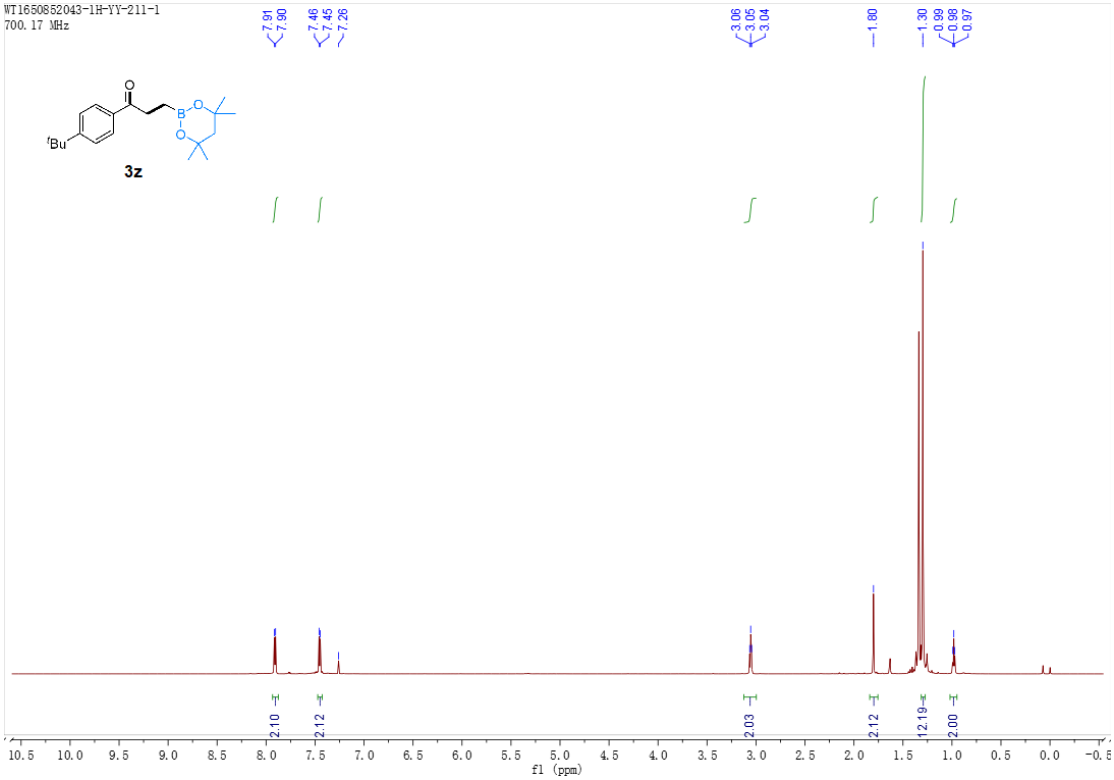

WT1650506422-13C-YY-205
176.06 MHz
194.95
142.13
131.46
128.89
128.04
83.13
34.76
25.03
24.79
3x
f1 (ppm)

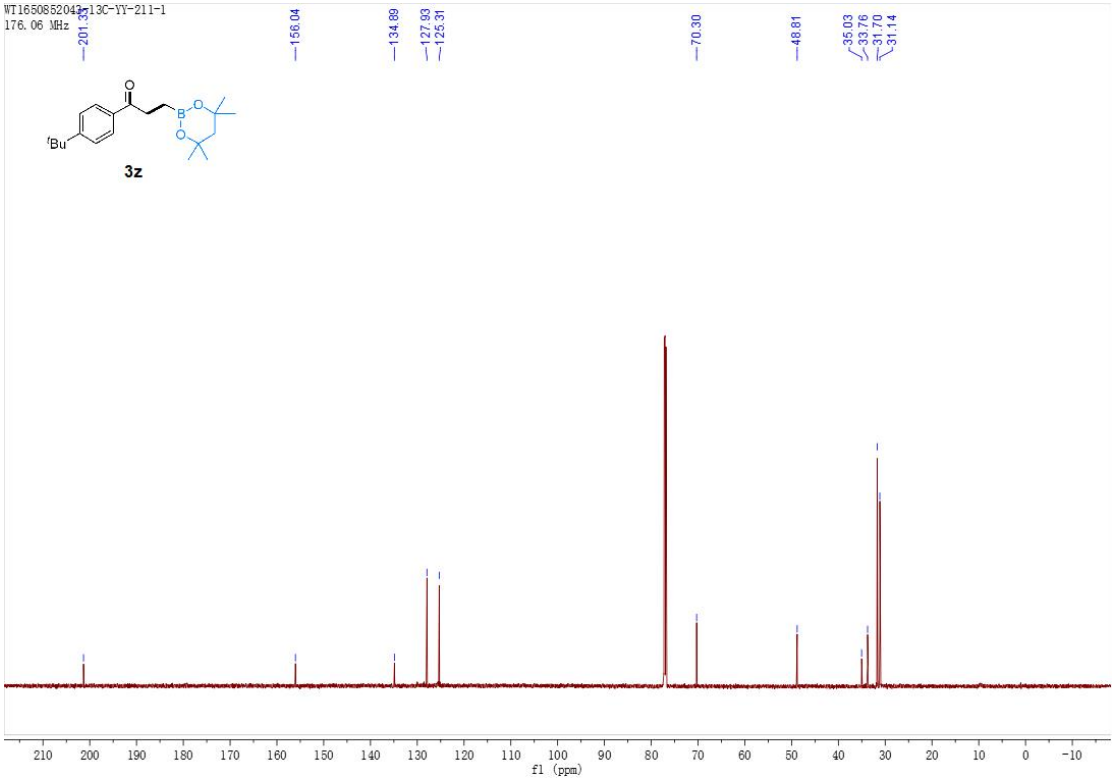

0692-YY-205
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 18
128.38 MHz
34.17
3x
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

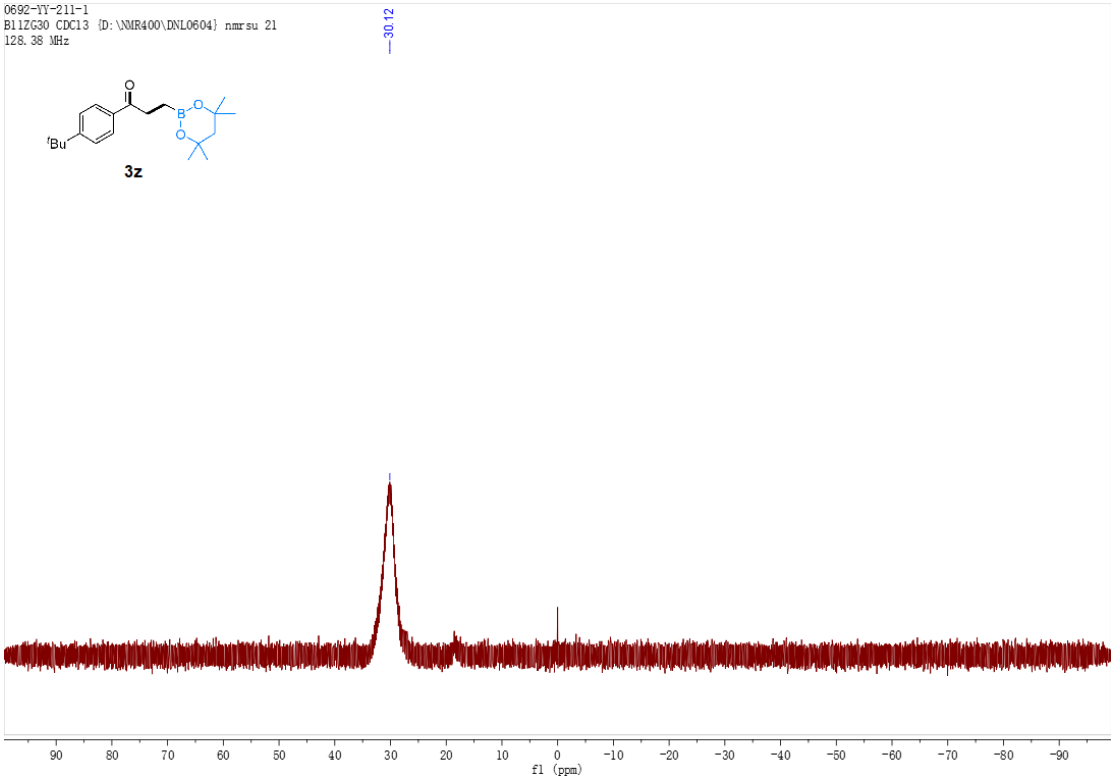

WT1650506422-1H-YY-172
700.17 MHz
7.97
7.96
7.96
7.54
7.53
7.51
7.45
7.44
7.43
7.26
3.08
3.07
3.06
1.80
1.29
1.01
1.00
0.99
3y
1.96
0.99
1.90
2.00
2.06
12.25
2.01
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

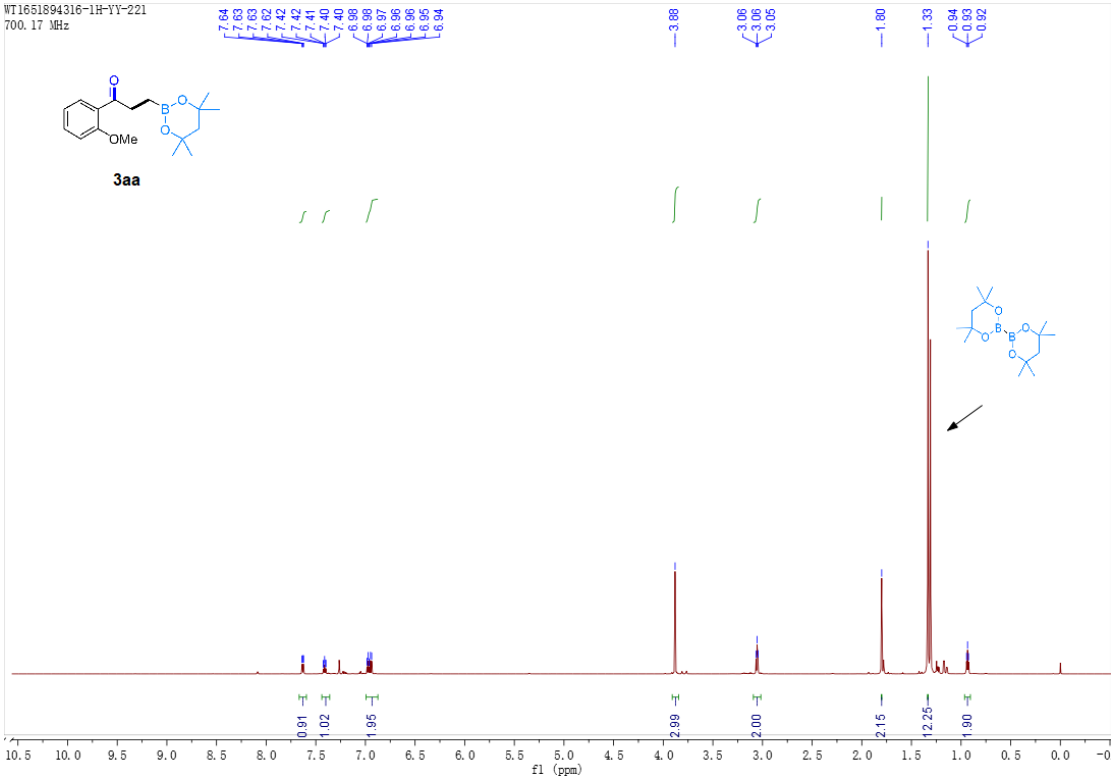

WT1650506422-13C-YY-172
176.06 MHz
201.75
137.49
132.48
128.39
127.99
70.34
48.80
33.80
31.69
3y
f1 (ppm)

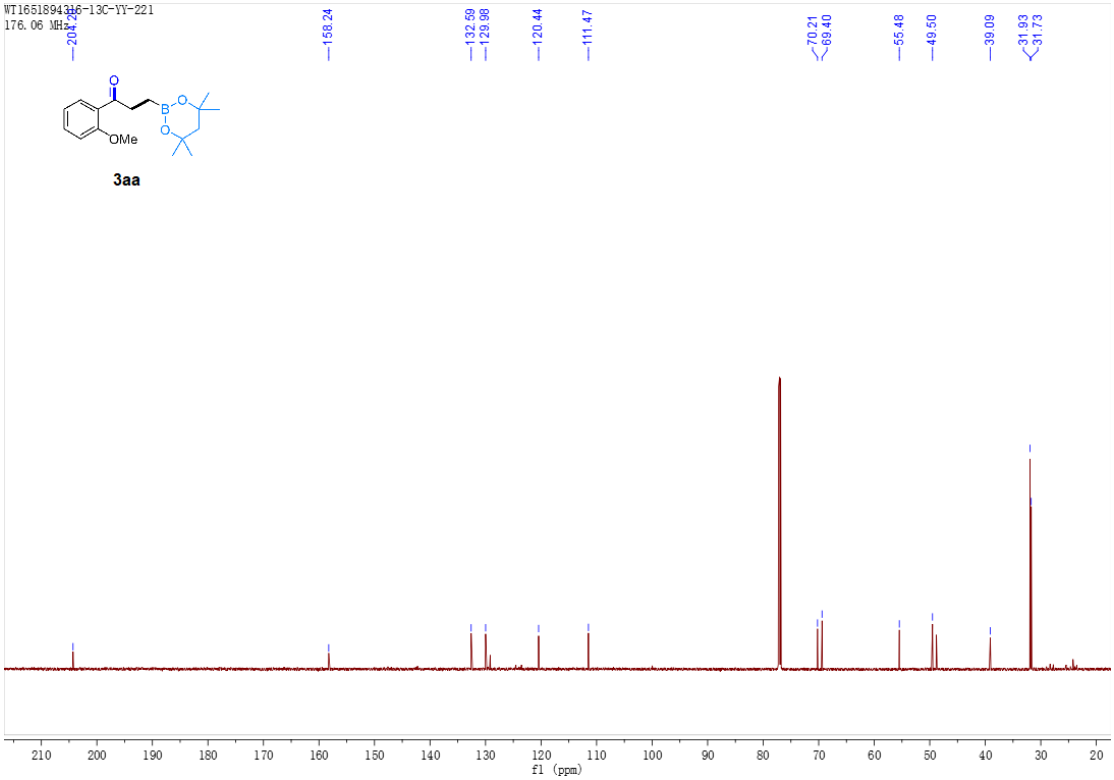

0692-YY-172
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 5
128.38 MHz
30.26
3y
f1 (ppm)

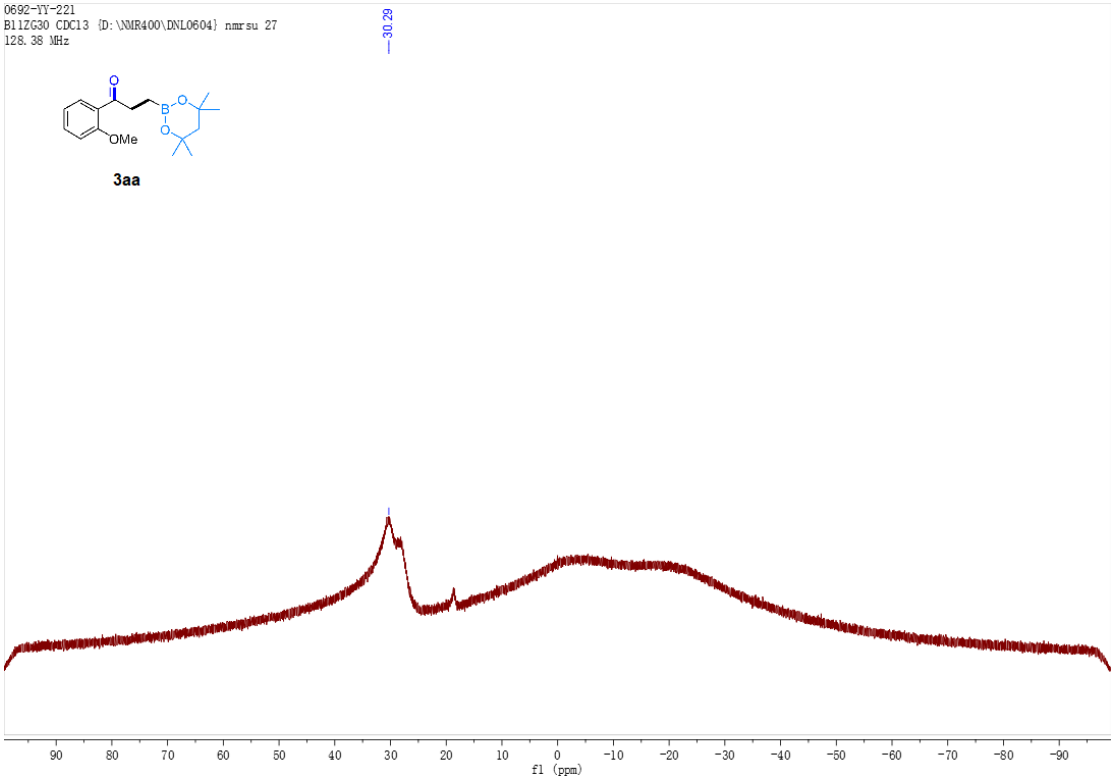

WT1650852043-1H-YY-211-1
700.17 MHz
7.91
7.90
7.46
7.45
7.26
3.06
3.05
3.04
1.80
1.30
0.99
0.98
0.97
tBu
3z
2.10
2.12
2.03
2.12
12.19
2.00
f1 (ppm)

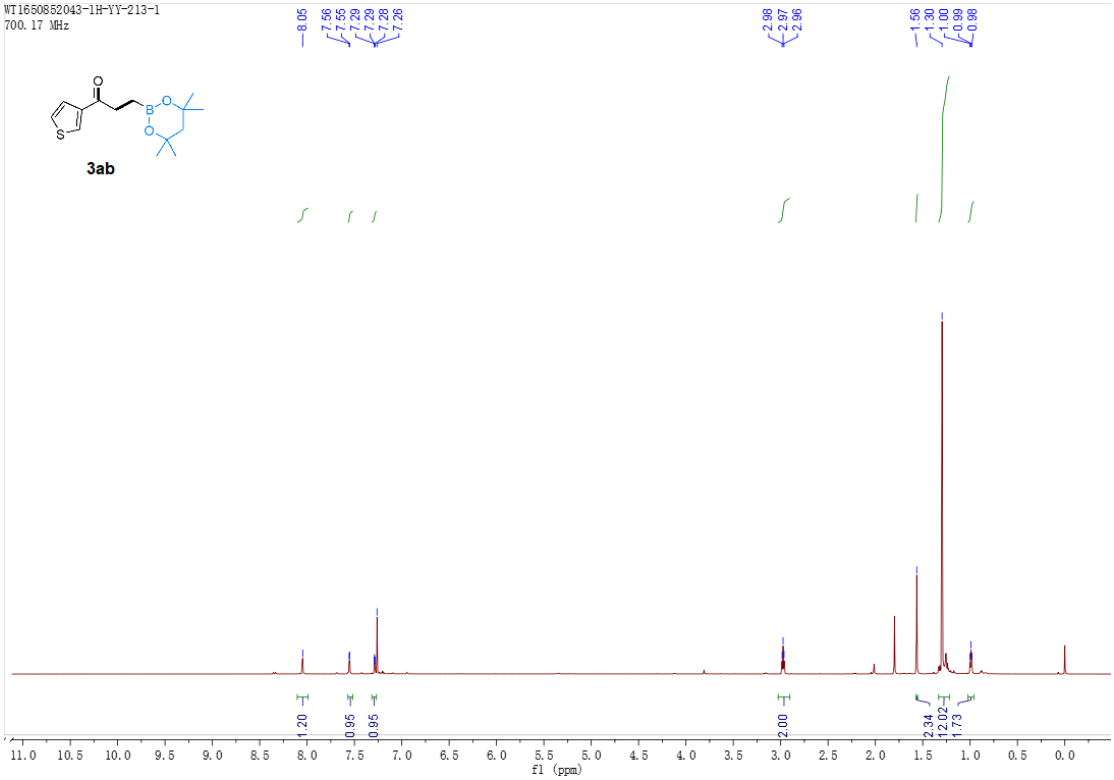

WT1650852043-13C-YY-211-1
176.06 MHz
201.39
156.04
134.89
127.93
125.31
70.30
48.81
35.03
33.76
31.70
31.14
tBu
3z
f1 (ppm)

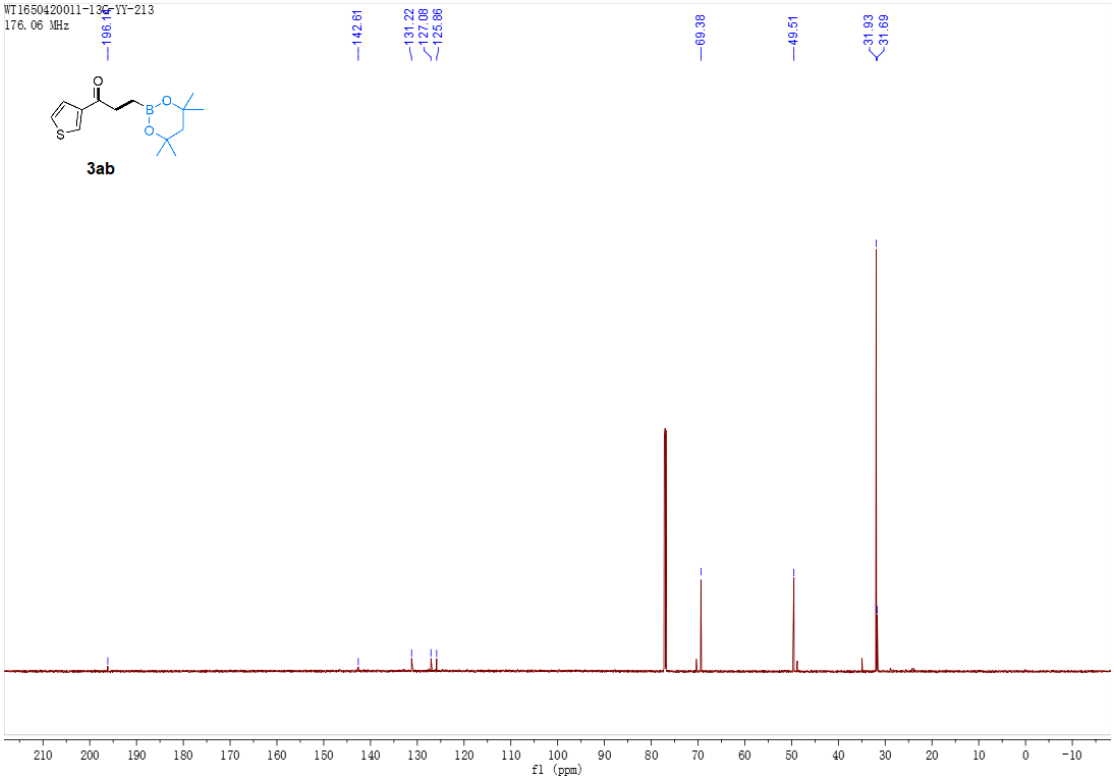

0692-YY-211-1
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 21
128.38 MHz
30.12
tBu
3z
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

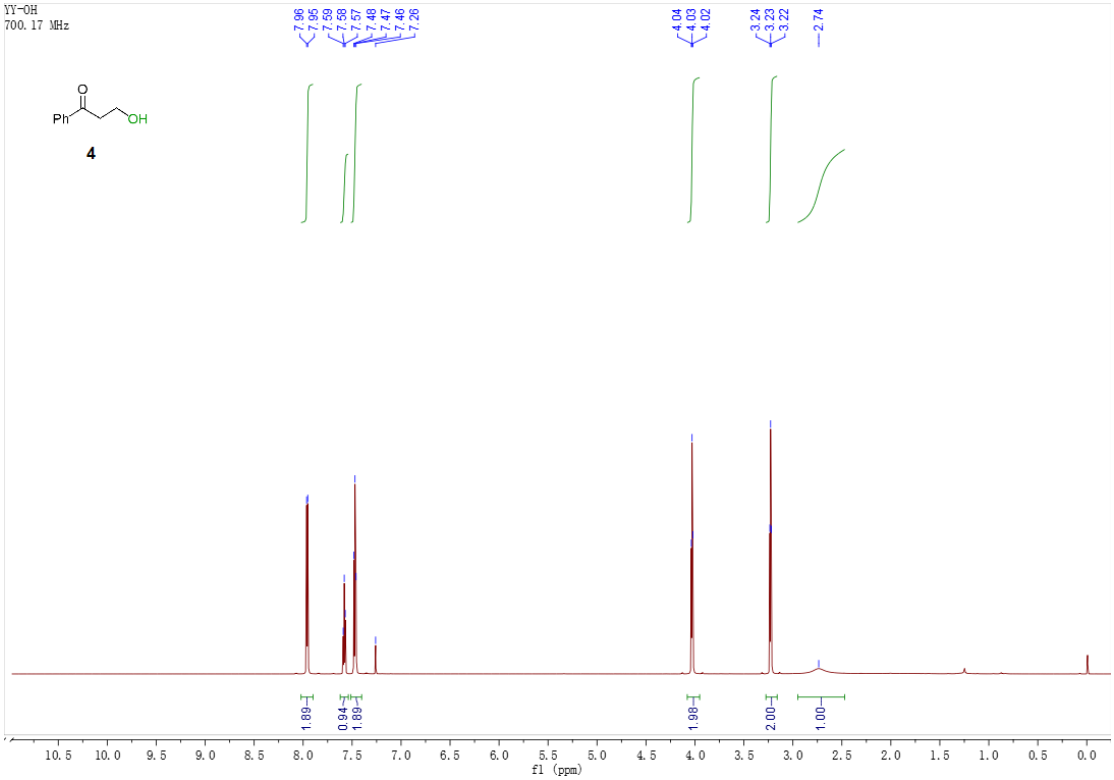

WT1651894316-1H-YY-221
700.17 MHz
7.64 7.63 7.63 7.62 7.42 7.41 7.40 7.40 6.98 6.98 6.97 6.96 6.96 6.95 6.94
3.88
3.06 3.06 3.05
1.80
1.33
0.94 0.93 0.92
OMe
3aa
0.91 1.02 1.95 2.99 2.00 2.15 12.25 1.90
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

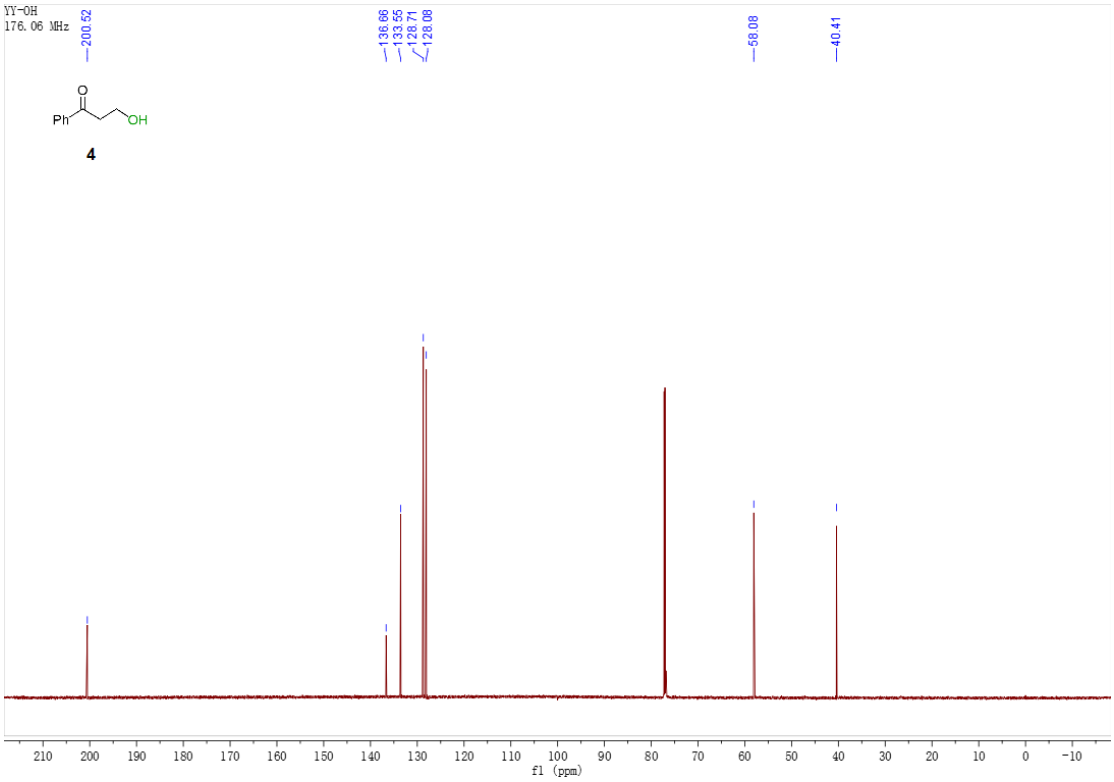

WT1651894316-13C-YY-221
176.06 MHz
204.26
158.24
132.59
129.98
120.44
111.47
70.21
69.40
55.48
49.50
39.09
31.93
31.73
OMe
3aa
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)

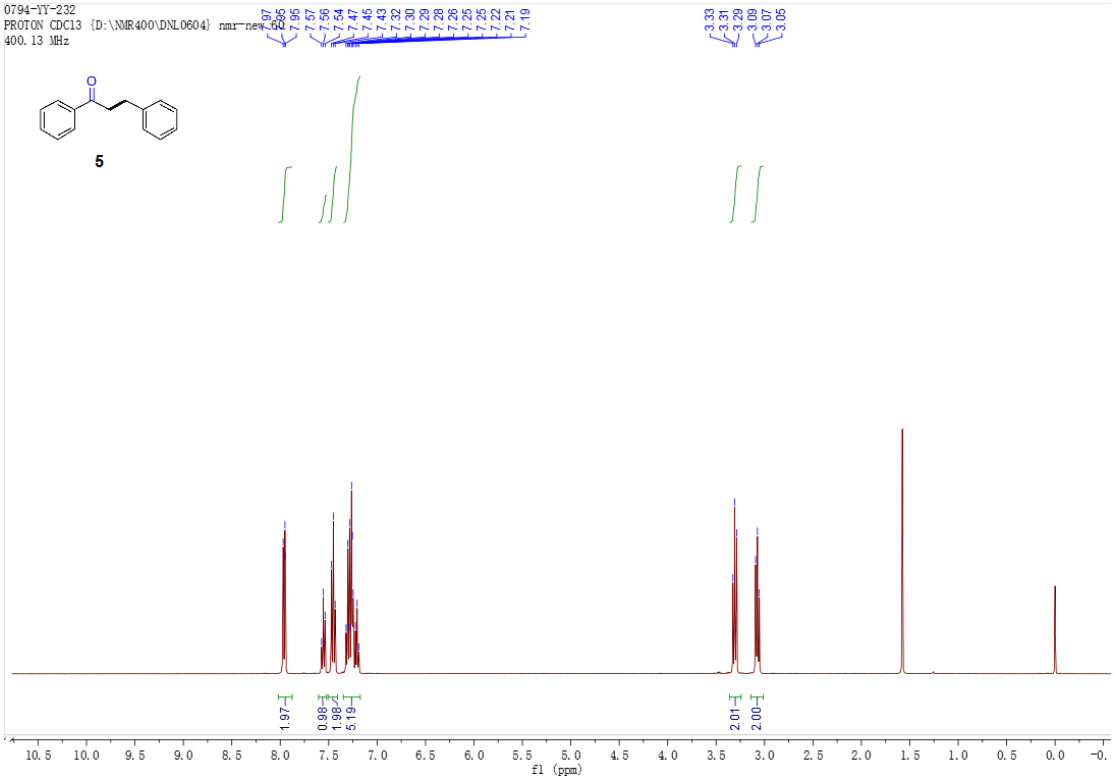

0692-YY-221
B11ZG30 CDCl3 {D:\NMR400\DNL0604} nmrsu 27
128.38 MHz
30.29
OMe
3aa
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

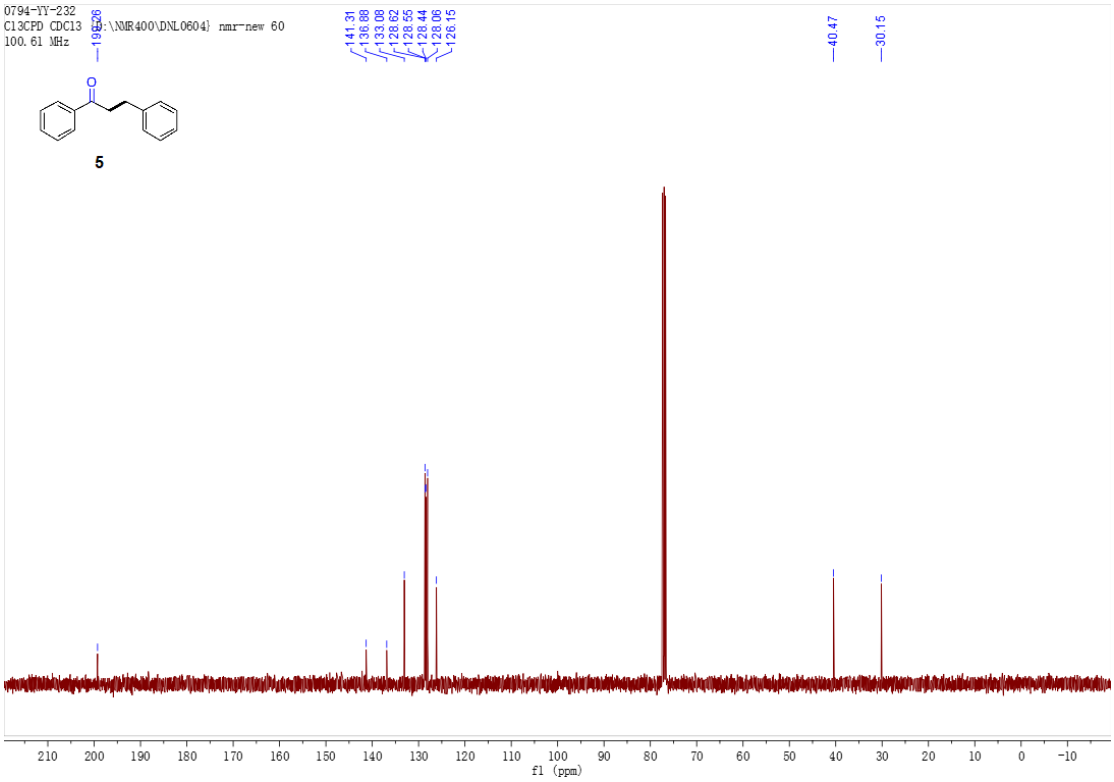

WT1650852043-1H-YY-213-1
700.17 MHz
8.05
7.56
7.55
7.29
7.29
7.28
7.26
2.98
2.97
2.96
1.56
1.30
1.00
0.99
0.98
3ab
1.20
0.95
0.95
2.00
2.34
12.02
1.73
f1 (ppm)


WT1650420011-13C-YY-213
176.06 MHz
196.16
142.61
131.22
127.08
125.86
69.38
49.51
31.93
31.69
3ab
f1 (ppm)


YY-OH
700.17 MHz
7.96
7.95
7.59
7.58
7.57
7.48
7.47
7.46
7.26
4.04
4.03
4.02
3.24
3.23
3.22
2.74
Ph
OH
4
1.89
0.94
1.89
1.98
2.00
1.00
f1 (ppm)


YY-OH
176.06 MHz
200.52
136.66
133.55
128.71
128.08
58.08
40.41
Ph
OH
4
f1 (ppm)


0794-YY-232
PROTON CDCl3 {D:\NMR400\DNL0604} nmr-new 60
400.13 MHz
7.97
7.95
7.67
7.56
7.54
7.47
7.45
7.43
7.32
7.30
7.29
7.28
7.26
7.25
7.25
7.22
7.21
7.19
3.33
3.31
3.29
3.09
3.07
3.05
O
5
1.97
0.98
1.98
5.19
2.01
2.00
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)


0794-YY-232
C13CPD CDCl3 {D:\NMR400\DNL0604} nmr-new 60
100.61 MHz
198.26
141.31
136.88
133.08
128.62
128.55
128.44
128.06
126.15
40.47
30.15
O
5
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)