Supporting Information for

Diversified access to polysubstituted alkenes via photoredox-catalysed E1cb-type elimination reactions of allyl ethers with N-centered radical precursors

Zhenjiang Ye, Binbin Fan, Li Zhang and Yewen Fang

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1. General information

1.1 Solvents, reagents, and starting materials

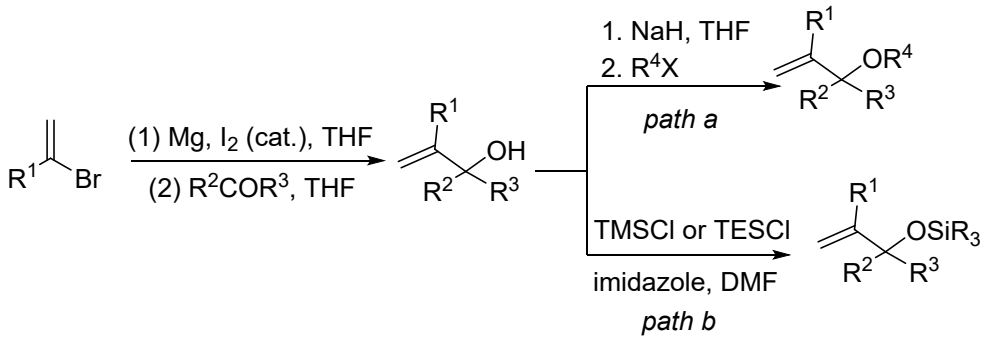
All reactions were carried out in glassware under inert (nitrogen) atmosphere unless otherwise noted. DMF and CH_2Cl_2 were dried from CaH, and THF was distilled after drying sodium wire. The dehydrated solvents DMSO, DMA, and CH_3CN were purchased from Energy Chemical Chemicals. Allyl ethers **1**[1] and MBH ester **5**[2] were prepared according to literature procedures and our previous report. All known cycloketone oxime esters **2**[3] and α-imino-oxy acids **4**[4] were prepared via reported procedures. Photocatalysts and all other chemicals were purchased from local vendors and used as supplied unless otherwise stated.

1.2 Instruments

NMR spectra were recorded on a Bruker Avance 500 spectrometer (500 MHz). Chemical shifts were reported in ppm downfield from tetramethylsilane, and calibrated using residue undeuterated solvent ($CHCl_3$ at 7.26 ppm ^1H NMR, 77.0 ppm ^{13}C NMR). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz) and integration. High resolution mass spectra (HRMS) were recorded on an ESI-Q-TOF spectrometer Agilent 6210 ESI/TOF. TLC analyses were performed on precoated GF_{254} silica gel plates and were visualized under UV254 nm light or by I_2 staining. Column chromatography was carried out using 300-400 mesh silica gel and eluted with petroleum/ethyl acetate unless otherwise noted.

1.3 Picture of a typical reaction setup

2. Preparation of allylic ethers



Under a nitrogen atmosphere, added magnesium turnings (546.8 mg, 22.5 mmol), two iodine pellets, and anhydrous tetrahydrofuran (THF, 5 mL) to a dry Schlenk tube. After stirring for several minutes, the reddish-brown color of the solution fades. Subsequently, a solution of α-bromostyrene (15 mmol) in anhydrous THF (30 mL) was added dropwise, with the rate controlled to maintain a gentle reflux in the reaction system. Upon completion of the addition, the reaction mixture was heated to 50 °C and stirred for an additional 1 hour to ensure complete formation of the Grignard reagent. The mixture was then allowed to stand for 1 hour before use.

Under a nitrogen atmosphere, a second dry Schlenk flask was charged with the ketone substrate (10 mmol) and anhydrous THF (10 mL). The flask was then immersed in an ice-water bath to cool

the reaction system. Subsequently, the pre-prepared Grignard reagent was added dropwise to the cooled ketone solution at a controlled rate. After complete addition of the Grignard reagent, the ice-water bath was removed, and the reaction mixture was stirred at room temperature for 2 hours. The system was then heated to 50 °C and stirred for an additional 3 hours to drive the reaction to completion. Upon completion of the reaction, the mixture was carefully quenched via slow addition of 20 mL saturated aqueous ammonium chloride, with the reaction tube maintained in an ice-water bath. The aqueous layer was extracted with ethyl acetate (4× 15 mL), and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude product was purified by column chromatography over silica gel with hexane/ethyl acetate as the eluent.

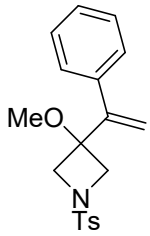
Path a:

To a dry Schlenk tube was added alcohol (5 mmol) prepared above and DMF or THF (10 mL) under nitrogen.The reaction mixture was then cooled in ice/water bath, followed by NaH (500 mg, 60 wt % dispersion in mineral oil, 12.5 mmol) was added in portions. The reaction mixture was stirred at 0 ^oC for 30 min. Then alkyl halide RX (20 mmol) was added dropwise in ice/water bath and the final solution was continued to stir for overnight at room temperature. After completion of the reaction, the mixture was carefully quenched via slow addition of 10 mL saturated aqueous ammonium chloride at 0 ^oC. Then, the aqueous layer was extracted with ethyl acetate (4 × 10 mL), and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude product was purified by column chromatography over silica gel with hexane/ethyl acetate as the eluent.

Path b:

Under a nitrogen atmosphere, the pre-synthesized alcohol (5 mmol) and anhydrous DMF (10 mL) were added sequentially to a flame-dried Schlenk tube. The reaction mixture was then immersed in an ice-water bath to cool to 0 °C, after which imidazole (851 mg, 12.5 mmol) was added portionwise. After stirring at 0 °C for 30 minutes, TMSCl or TESCl (7.5 mmol) was slowly added dropwise to the reaction mixture while maintaining ice-water bath cooling. After complete addition, the cooling bath was removed, and the mixture was allowed to warm to room temperature and stirred overnight. Upon reaction completion, the mixture was quenched by

addition of saturated aqueous ammonium chloride (10 mL). Then, the aqueous layer was extracted with ethyl acetate (4 × 10 mL), and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude product was purified by column chromatography over silica gel with hexane/ethyl acetate as the eluent.

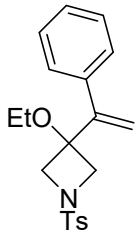


3-Methoxy-3-(1-phenylvinyl)-1-tosylazetidine (1a). White solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.1 Hz, 2H), 7.34 (d, *J* = 7.9 Hz, 2H), 7.33 – 7.22 (m, 5H), 5.62 (s, 1H), 5.20 (s, 1H), 3.96 (d, *J* = 9.3 Hz, 2H), 3.85 (d, *J* = 9.2 Hz, 2H), 3.04 (s, 3H), 2.46 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 144.1, 144.0, 137.2, 131.9, 129.7, 128.4, 128.3, 128.1, 126.6, 117.5, 76.5, 59.8, 50.6, 21.6.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{19}H_{21}NO_3NaS^+$: 366.1134, found 366.1143.

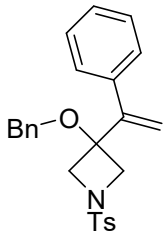


3-Ethoxy-3-(1-phenylvinyl)-1-tosylazetidine (1aa). White solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.33 – 7.29 (m, 2H), 7.29 – 7.26 (m, 3H), 5.58 (s, 1H), 5.17 (s, 1H), 3.96 (d, *J* = 9.6 Hz, 2H), 3.86 (d, *J* = 9.6 Hz, 2H), 3.19 (q, *J* = 7.0 Hz, 2H), 2.46 (s, 3H), 1.02 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 144.8, 144.0, 137.3, 131.8, 129.6, 128.4, 128.3, 128.0, 126.6, 117.0, 75.8, 60.3, 58.6, 21.6, 15.1.

HRMS (ESI) $[M+H]^+$: calculated for $C_{20}H_{24}NO_3S^+$: 358.1471, found 358.1479.

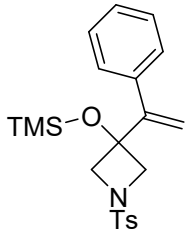


3-(Benzyloxy)-3-(1-phenylvinyl)-1-tosylazetidine (1ab). White solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.3 Hz, 2H), 7.39 – 7.34 (m, 4H), 7.32 – 7.28 (m, 3H), 7.27 – 7.25 (m, 3H), 7.07 – 6.99 (m, 2H), 5.72 (s, 1H), 5.33 (s, 1H), 4.17 (s, 2H), 4.06 (d, *J* = 9.7 Hz, 2H), 3.93 (d, *J* = 9.7 Hz, 2H), 2.46 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 144.6, 144.0, 137.1, 131.5, 129.7, 128.5, 128.3, 128.2, 127.7(5), 127.7(1), 126.7, 117.6, 76.2, 65.5, 60.5, 21.6.

HRMS (ESI) $[M+H]^+$: calculated for $C_{25}H_{26}NO_3S^+$: 420.1628, found 420.1634.

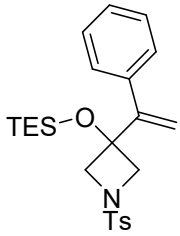


3-(1-Phenylvinyl)-1-tosyl-3-((trimethylsilyl)oxy)azetidine (1ac). Pale yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 – 7.63 (m, 2H), 7.38 – 7.33 (m, 2H), 7.32 – 7.25 (m, 5H), 5.49 (s, 1H), 5.29 (s, 1H), 4.14 – 4.00 (m, 2H), 3.89 – 3.80 (m, 2H), 2.45 (s, 3H), -0.10 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 148.8, 144.0, 137.5, 131.3, 129.6, 128.4, 128.1, 127.9, 127.1, 114.5,72.4, 63.8, 21.5, 1.1.

HRMS (ESI) $[M+H]^+$: calculated for $C_{21}H_{28}NO_3SSi^+$: 402.1554, found 402.1561.

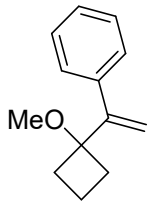


3-(1-Phenylvinyl)-1-tosyl-3-((triethylsilyl)oxy)azetidine (1ad). White solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 – 7.65 (m, 2H), 7.36 (d, *J* = 7.9 Hz, 2H), 7.34 – 7.29 (m, 2H), 7.29 – 7.24 (m, 3H), 5.50 (s, 1H), 5.32 (s, 1H), 4.16 – 4.00 (m, 2H), 3.85 – 3.72 (m, 2H), 2.45 (s, 3H), 0.71 (t, *J* = 8.0 Hz, 9H), 0.36 (q, *J* = 8.0 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 148.8, 144.1, 137.5, 130.9, 129.7, 128.5, 128.1, 127.8, 127.1, 114.3, 72.1, 63.8, 21.5, 6.6, 5.5.

HRMS (ESI) $[M+H]^+$: calculated for $C_{24}H_{34}NO_3SiS^+$: 444.2023, found 444.2028.

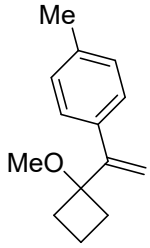


(1-(1-Methoxycyclobutyl)vinyl)benzene (1b). Pale yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 – 7.53 (m, 2H), 7.40 – 7.26 (m, 3H), 5.64 (s, 1H), 5.36 (s, 1H), 3.12 (s, 3H), 2.41 – 2.32 (m, 2H), 2.32 – 2.23 (m, 2H), 2.04 – 1.92 (m, 1H), 1.77 – 1.64 (m, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 147.9, 139.2, 127.9, 127.3, 127.2, 115.5, 82.8, 49.8, 32.1, 13.5.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{13}H_{16}ONa^+$: 211.1093, found 211.1087**.**

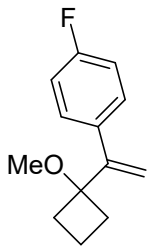


1-(1-(1-Methoxycyclobutyl)vinyl)-4-methylbenzene (1c). Pale yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 – 7.38 (m, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 5.62 (d, *J* = 1.2 Hz, 1H), 5.32 (d, *J* = 1.1 Hz, 1H), 3.11 (s, 3H), 2.39 (s, 3H), 2.38 – 2.32 (m, 2H), 2.32 – 2.24 (m, 2H), 2.03 – 1.92 (m, 1H), 1.75 – 1.64 (m, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 147.6, 136.9, 136.1, 128.6, 127.1, 114.7, 82.9, 49.7, 32.1, 21.0, 13.5.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{14}H_{18}ONa^+$: 225.1250, found 225.1250.



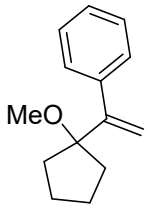
1-Fluoro-4-(1-(1-methoxycyclobutyl)vinyl)benzene (1d). Pale yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 – 7.42 (m, 2H), 7.09 – 6.90 (m, 2H), 5.55 (d, *J* = 0.9 Hz, 1H), 5.30 (d, *J* = 1.0 Hz, 1H), 3.06 (s, 3H), 2.37 – 2.17 (m, 4H), 2.00 – 1.85 (m, 1H), 1.71 – 1.57 (m, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 162.3 (d, *J* = 246.6 Hz), 146.8, 135.1 (d, *J* = 3.5 Hz), 128.9 (d, *J* = 7.8 Hz), 115.4, 114.7 (d, *J* = 21.0 Hz), 82.7, 49.7, 32.0, 13.4.

^{19}F NMR (471 MHz, $CDCl_3$) δ -115.4.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{13}H_{15}ONaF$: 229.0999, found 229.0997.

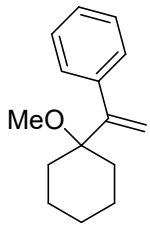


(1-(1-Methoxycyclopentyl)vinyl)benzene (1e). Pale yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.33 (m, 2H), 7.25 – 7.14 (m, 3H), 5.25 (d, *J* = 1.4 Hz, 1H), 5.20 (d, *J* = 1.5 Hz, 1H), 3.10 (s, 3H), 1.92 – 1.79 (m, 2H), 1.75 – 1.63 (m, 2H), 1.62 – 1.47 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 149.4, 141.9, 127.8, 127.0, 116.3, 89.1, 49.9, 35.3, 22.8.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{14}H_{18}ONa^+$: 225.1250, found 225.1261**.**

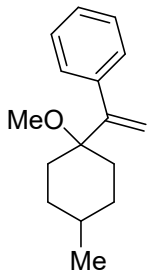


(1-(1-Methoxycyclohexyl)vinyl)benzene (1f). Pale yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.30 (m, 2H), 7.30 – 7.21 (m, 3H), 5.30 (d, *J* = 1.4 Hz, 1H), 5.29 (d, *J* = 1.5 Hz, 1H), 3.21 (s, 3H), 1.86 – 1.73 (m, 2H), 1.61 – 1.47 (m, 5H), 1.47 – 1.38 (m, 2H) , 1.30 – 1.19 (m, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 150.9, 141.7, 128.4, 127.7, 126.8, 117.0, 77.6, 48.9, 33.7, 25.8, 22.0.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{15}H_{20}ONa^+$: 239.1406, found 239.1408.

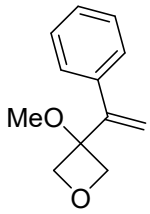


(1-(1-Methoxy-4-methylcyclohexyl)vinyl)benzene (1g). Pale yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.29 (m, 2H), 7.29 – 7.21 (m, 3H), 5.28 (d, *J* = 1.5 Hz, 1H), 5.22 (d, *J* = 1.5 Hz, 1H), 3.18 (s, 3H), 1.99 – 1.88 (m, 2H), 1.52 – 1.41 (m, 2H), 1.39 – 1.23 (m, 5H), 0.89 (d, *J* = 5.6 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 152.1, 141.8, 128.4, 127.6, 126.8, 116.0, 76.8, 49.0, 33.2, 31.9, 30.1, 22.3.

HRMS (ESI) $[M+H]^+$: calculated for $C_{16}H_{23}O^+$: 231.1743, found 231.1742.

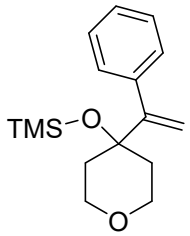


3-Methoxy-3-(1-phenylvinyl)oxetane (1h). Pale yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.39 (m, 2H), 7.38 – 7.27 (m, 3H), 5.80 (s, 1H), 5.33 (s, 1H), 4.81 – 4.76 (m, 4H), 3.19 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 144.4, 137.4, 128.3, 127.9, 126.3, 116.9, 81.9, 79.9, 50.6.

HRMS (ESI) $[M+H]^+$: calculated for $C_{12}H_{15}O_2^+$: 191.1067, found 191.1071.

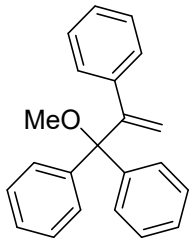


Trimethyl((4-(1-phenylvinyl)tetrahydro-2*H*-pyran-4-yl)oxy)silane (1i). White solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 – 7.36 (m, 2H), 7.34 – 7.18 (m, 3H), 5.37 (s, 1H), 5.24 (s, 1H), 3.84 – 3.71 (m, 2H), 3.70 – 3.58 (m, 2H), 1.89 – 1.71 (m, 4H), 0.14 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.0, 141.0, 128.8, 127.7, 127.1, 115.2, 73.9, 64.3, 38.1, 2.5.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{24}O_2SiNa^+$: 299.1438, found 299.1438.

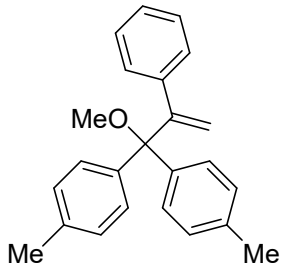


(1-Methoxyprop-2-ene-1,1,2-triyl)tribenzene (1j). White solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.43 (m, 4H), 7.25 – 7.15 (m, 6H), 7.13 – 7.02 (m, 5H), 5.69 (d, *J* = 1.3 Hz, 1H), 5.55 (d, *J* = 1.3 Hz, 1H), 3.18 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 149.8, 143.0, 141.1, 128.3, 128.2, 127.6, 127.4, 126.8, 126.6, 121.1, 87.8, 52.0.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{22}H_{20}ONa^+$: 323.1406, found 323.1415.

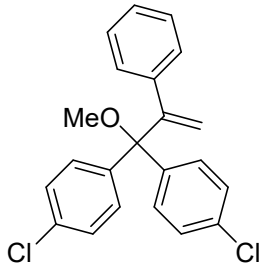


4,4'-(1-Methoxy-2-phenylprop-2-ene-1,1-diyl)bis(methylbenzene) (1k). White solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ7.34 (d, *J* = 8.3 Hz, 4H), 7.25 – 7.17 (m, 2H), 7.16 – 7.09 (m, 3H), 7.04 (d, *J* = 7.9 Hz, 4H), 5.66 (s, 1H), 5.55 (s, 1H), 3.16 (s, 3H), 2.28 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 150.3, 141.4, 140.1, 136.1, 128.4, 128.3, 128.2, 127.4, 126.7, 120.6, 87.7, 51.9, 21.0.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{24}H_{24}ONa^+$: 351.1719, found 351.1725.



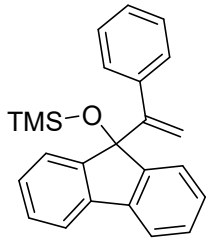
4,4'-(1-Methoxy-2-phenylprop-2-ene-1,1-diyl)bis(chlorobenzene) (1l). White solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.32 (m, 4H), 7.27 – 7.20 (m, 2H), 7.19 – 7.13 (m, 4H), 7.13 – 7.03 (m, 3H), 5.75 (d, *J* = 1.1 Hz, 1H), 5.51 (d, *J* = 1.1 Hz, 1H), 3.20 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 148.4, 141.7, 140.4, 132.6, 129.2, 128.1, 127.9, 127.7, 127.1,

122.0, 87.1, 52.0.

HRMS (ESI) $[M+H]^+$: calculated for $C_{22}H_{19}OCl_2^+$: 369.0807, found 369.0815.

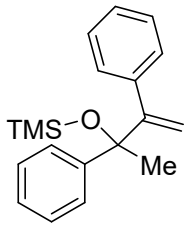


Trimethyl((9-(1-phenylvinyl)-9*H*-fluoren-9-yl)oxy)silane (1m). White solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.4 Hz, 2H), 7.40 (d, *J* = 7.4 Hz, 2H), 7.34 – 7.27 (m, 2H), 7.25 – 7.18 (m, 2H), 7.03 – 6.95 (m, 1H), 6.95 – 6.85 (m, 2H), 6.72 – 6.58 (m, 2H), 6.00 (d, *J* = 2.3 Hz, 1H), 5.24 (d, *J* = 2.3 Hz, 1H), -0.34 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 152.0, 147.9, 140.3, 140.2, 128.8, 128.5, 127.5, 126.9, 126.5, 125.4, 119.7, 114.8, 85.7, 1.2.

The characterization data were consistent with the literature.[1c]



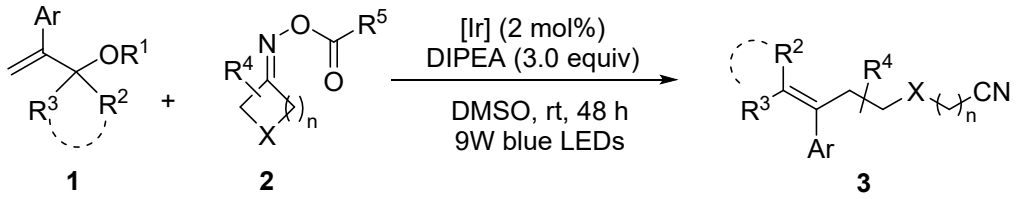
((2,3-Diphenylbut-3-en-2-yl)oxy)trimethylsilane (1n). Pale yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 – 7.54 (m, 2H), 7.48 – 7.40 (m, 2H), 7.40 – 7.32 (m, 1H), 7.31 – 7.21 (m, 3H), 7.18 – 7.07 (m, 2H), 5.81 – 5.61 (m, 1H), 5.50 – 5.43 (m, 1H), 1.99 – 1.82 (m, 3H), 0.23 – 0.06 (m, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 155.5, 147.2, 140.8, 128.7, 127.9, 127.2, 126.7, 126.6, 125.9, 113.6, 79.2, 30.1, 2.0.

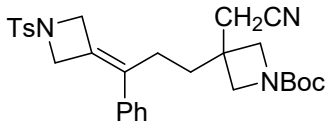
HRMS (ESI) $[M+H]^+$: calculated for $C_{19}H_{25}OSi^+$: 297.1669, found 297.1674.

3. General procedure of photocatalytic reaction of cycloketone oxime ester with allyl ether



1 **2** **3**

Under a nitrogen atmosphere, to an oven dried transparent 25 mL Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (4.5 mg, 0.004 mmol, 0.02 equiv), allyl ether **1** (0.2 mmol, 1.0 equiv), cycloketone oxime ester **2** (0.6 mmol, 3.0 equiv), DIPEA (0.6 mmol, 3.0 equiv) and degassed DMSO (4 mL) were added. The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 48 h (cooling with a fan). After the reaction was complete, the reaction solution was quenched by the addition of sodium bicarbonate solution (5 mL) and extracted with EtOAc (5 × 10 mL). The combined organic layer was washed with brine, dried over Na_2SO_4, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product.

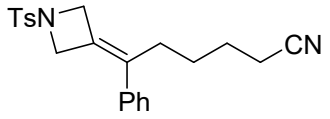


***tert*-Butyl 3-(cyanomethyl)-3-(3-phenyl-3-(1-tosylazetidin-3-ylidene)propyl)azetidine-1-carboxylate (3a).** Yellow solid (94.8 mg, 91% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.9 Hz, 2H), 7.37 (d, *J* = 7.9 Hz, 2H), 7.35 – 7.29 (m, 2H), 7.28 – 7.23 (m, 1H), 7.05 (d, *J* = 7.0 Hz, 2H), 4.51 (s, 2H), 4.46 (s, 2H), 3.77 – 3.42 (m, 4H), 2.60 (s, 2H), 2.45 (s, 3H), 2.20 (t, *J* = 8.4 Hz, 2H), 1.66 (t, *J* = 8.4 Hz, 2H), 1.42 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.0, 144.3, 136.7, 132.8, 131.9, 129.9, 128.8, 128.3, 127.8, 126.4, 123.3, 116.6, 80.1, 59.2, 58.3, 35.1, 34.7, 28.2, 26.0, 25.6, 21.6.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{29}H_{35}N_3O_4NaS^+$: 544.2240, found 544.2249.

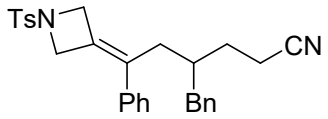


6-Phenyl-6-(1-tosylazetidin-3-ylidene)hexanenitrile (3b). Yellow oil (59.3 mg, 78% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.34 – 7.28 (m, 2H), 7.26 – 7.21 (m, 1H), 7.10 – 7.02 (m, 2H), 4.50 (s, 2H), 4.46 (s, 2H), 2.46 (s, 3H), 2.31 – 2.18 (m, 4H), 1.62 – 1.49 (m, 2H), 1.46 – 1.35 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 144.2, 137.1, 133.6, 131.9, 129.8, 128.6, 128.3, 127.5, 126.5, 122.7, 119.3, 59.2, 58.5, 30.5, 26.8, 24.8, 21.6, 16.9.

HRMS (ESI) [M+Na]$^+$: calculated for $C_{22}H_{24}N_2O_2NaS^+$: 403.1451, found 403.1460.

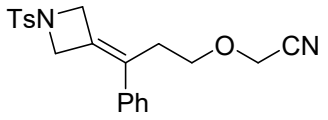


4-Benzyl-6-phenyl-6-(1-tosylazetidin-3-ylidene)hexanenitrile (3c). Yellow solid (74.3 mg, 79% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.32 – 7.27 (m, 2H), 7.26 – 7.21 (m, 3H), 7.21 – 7.16 (m, 1H), 6.99 – 6.87 (m, 4H), 4.45 (s, 4H), 2.56 – 2.41 (m, 1H), 2.39 (s, 3H), 2.41 – 2.31 (m, 1H), 2.23 – 2.08 (m, 4H), 1.75 – 1.64 (m, 1H), 1.57 – 1.45 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 144.3, 139.3, 136.9, 132.9, 131.8, 129.8, 128.8, 128.7, 128.5, 128.3, 127.7, 126.5, 126.4, 123.9, 119.3, 59.2, 58.7, 39.7, 37.6, 35.4, 29.1, 21.5, 14.8.

HRMS (ESI) [M+Na]$^+$: calculated for $C_{29}H_{30}N_2O_2NaS^+$: 493.1920, found 493.1928.

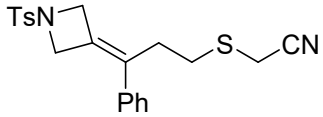


2-(3-Phenyl-3-(1-tosylazetidin-3-ylidene)propoxy)acetonitrile (3d). Yellow oil (59.7 mg, 78% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.33 – 7.28 (m, 2H), 7.26 – 7.22 (m, 1H), 7.10 – 7.03 (m, 2H), 4.53 (s, 2H), 4.46 (s, 2H), 4.10 (s, 2H), 3.47 (t, *J* = 6.6 Hz, 2H), 2.53 (t, *J* = 6.6 Hz, 2H), 2.46 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 144.2, 136.9, 132.1, 130.3, 129.8, 128.7, 128.4, 127.7, 126.6, 125.0, 115.7, 69.4, 59.2, 58.7, 56.2, 31.6, 21.6.

HRMS (ESI) [M+Na]$^+$: calculated for $C_{21}H_{22}N_2O_3NaS^+$: 405.1243, found 405.1251.

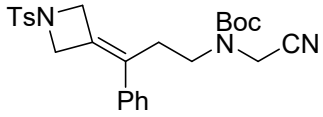


2-((3-Phenyl-3-(1-tosylazetidin-3-ylidene)propyl)thio)acetonitrile (3e). Yellow oil (44.6 mg, 56% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.35 – 7.29 (m, 2H), 7.28 – 7.23 (m, 1H), 7.09 – 7.03 (m, 2H), 4.56 (s, 2H), 4.47 (s, 2H), 3.20 (s, 2H), 2.66 (t, *J* = 6.6 Hz, 2H), 2.59 (t, *J* = 4.1 Hz, 2H), 2.46 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 144.3, 136.3, 132.0, 131.8, 129.9, 128.9, 128.4, 127.9, 126.6, 124.8, 116.3, 59.2, 58.6, 31.0, 30.9, 21.6, 17.3.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{21}H_{22}N_2O_2NaS_2^+$: 421.1015, found 421.1024.

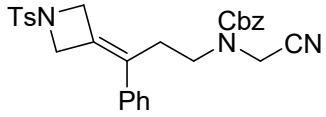


***tert*-Butyl (cyanomethyl)(3-phenyl-3-(1-tosylazetidin-3-ylidene)propyl)carbamate (3f).** Yellow oil (83.8 mg, 87% yield).

^{1}H NMR (500 MHz, $CDCl_3$) (mixture of rotamers) **δ** 7.76 (s, 0.95H), 7.75 (s, 1.05H), 7.38 (s, 1.05H), 7.36 (s, 0.95H), 7.35 – 7.29 (m, 3H), 7.27 – 7.24 (m, 1H), 7.10 (s, 1.05H), 7.09 (s, 0.95H), 4.51 (s, 2.10H), 4.50 (s, 1.90H), 3.95 (s, 1.05H), 3.81 (s, 0.95H), 3.21 (t, *J* = 7.2 Hz, 2H), 2.55 (t, *J* = 7.4 Hz, 2H), 2.45 (s, 3H), 1.45 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) (mixture of rotamers) **δ** 153.4, 144.2, 136.5, 131.8, 130.6, 129.9, 128.9, 128.3, 127.8, 126.3, 125.1, 115.9, 81.9, 59.4, 58.6, 46.6, 35.7, 29.6, 28.1, 21.6.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{26}H_{31}N_3O_4NaS^+$: 504.1927, found 504.1936.

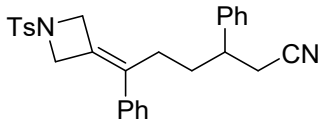


Benzyl (cyanomethyl)(3-phenyl-3-(1-tosylazetidin-3-ylidene)propyl)carbamate (3g). Yellow oil (95.9 mg, 93% yield).

^{1}H NMR (500 MHz, $CDCl_3$) (mixture of rotamers) **δ** 7.73 (s, 1.2 H), 7.72 (s, 0.8 H), 7.40 – 7.36 (m, 8H), 7.23 (s, 2H), 7.11 (s, 0.8 H), 6.99 (s, 1.2 H), 5.14 (s, 2H), 4.50 (br, 2H), 4.44 (s, 0.8 H), 4.30 (s, 1.2 H), 4.02 (s, 1.2 H), 3.92 (s, 0.8 H), 3.29 – 3.25 (m, 2H), 2.59 (s, 0.8 H), 2.51 (s, 1.2 H), 2.45 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) (mixture of rotamers) **δ** 155.2, 154.2, 144.2, 136.2, 135.4, 131.7, 130.2, 129.8, 128.8, 128.7, 128.5, 128.3, 128.1, 127.8, 126.2, 125.3, 115.6, 68.4, 59.3, 58.5, 47.6, 46.6, 36.3, 30.0, 21.5.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{29}H_{29}N_3O_4NaS^+$: 538.1771, found 538.1779.

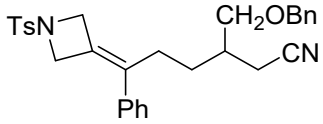


3,6-Diphenyl-6-(1-tosylazetidin-3-ylidene)hexanenitrile (3h). Yellow oil (74.0 mg, 81% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.2 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.36 – 7.32 (m, 2H), 7.32 – 7.27 (m, 3H), 7.26 – 7.21 (m, 1H), 7.11 (d, *J* = 6.8 Hz, 2H), 6.97 (d, *J* = 7.3 Hz, 2H), 4.47 (d, *J* = 13.0 Hz, 1H), 4.34 (d, *J* = 12.3 Hz, 1H), 4.25 (d, *J* = 12.8 Hz, 1H), 4.18 (d, *J* = 12.6 Hz, 1H), 2.89 – 2.70 (m, 1H), 2.50 – 2.40 (m, 2H) , 2.46 (s, 3H), 2.20 – 2.00 (m, 2H), 1.86 – 1.67 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 144.2, 140.6, 136.9, 133.3, 132.0, 129.8, 129.0, 128.6, 128.3, 127.7, 127.5, 127.2, 126.5, 122.9, 118.1, 59.2, 58.3, 41.5, 32.4, 28.5, 25.4, 21.6.

HRMS (ESI) $[M+H]^+$: calculated for $C_{28}H_{29}N_2O_2S^+$: 457.1944, found 457.1952.

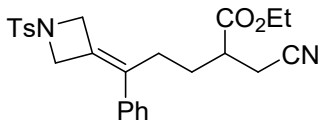


3-((Benzyloxy)methyl)-6-phenyl-6-(1-tosylazetidin-3-ylidene)hexanenitrile (3i). Yellow solid (41.1 mg, 41% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.39 – 7.34 (m, 4H), 7.34 – 7.30 (m, 2H), 7.29 – 7.27 (m, 3H), 7.25 – 7.21 (m, 1H), 7.06 – 7.00 (m, 2H), 4.51 – 4.39 (m, 6H), 3.44 – 3.38 (m, 1H), 3.34 – 3.28 (m, 1H), 2.45 (s, 3H), 2.39 (d, *J* = 6.1 Hz, 2H), 2.25 (t, *J* = 7.9 Hz, 2H), 1.92 – 1.85 (m, 1H), 1.49 – 1.32 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 144.2, 137.7, 137.0, 133.6, 132.1, 129.8, 128.7, 128.5, 128.3, 127.9, 127.7, 127.6, 126.5, 122.9, 118.3, 73.4, 70.6, 59.3, 58.5, 35.5, 28.7, 28.5, 21.6, 19.6.

HRMS (ESI) $[M+H]^+$: calculated for $C_{30}H_{33}N_2O_3S^+$: 501.2206, found 501.2214.

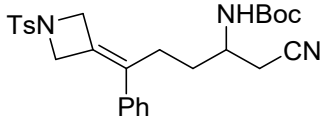


Ethyl 2-(cyanomethyl)-5-phenyl-5-(1-tosylazetidin-3-ylidene)pentanoate (3j). Yellow solid (75.1 mg, 83% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.9 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.34 – 7.27 (m, 2H), 7.26 – 7.20 (m, 1H), 7.04 (d, *J* = 7.7 Hz, 2H), 4.55 – 4.38 (m, 4H), 4.23 – 4.12 (m, 2H), 2.68 – 2.50 (m, 2H), 2.50 – 2.40 (m, 4H), 2.39 – 2.28 (m, 1H), 2.28 – 2.15 (m, 1H), 1.81 – 1.64 (m, 1H), 1.64 – 1.51 (m, 1H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.0, 144.2, 136.7, 132.8, 131.9, 129.8, 128.7, 128.3, 127.6, 126.4, 123.6, 117.3, 61.5, 59.2, 58.4, 40.7, 29.3, 28.2, 21.5, 19.5, 14.1.

HRMS (ESI) $[M+H]^+$: calculated for $C_{25}H_{29}N_2O_4S^+$: 453.1843, found 453.1852.

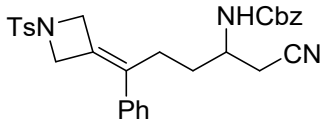


***tert*-Butyl (1-cyano-5-phenyl-5-(1-tosylazetidin-3-ylidene)pentan-2-yl)carbamate (3k).** Yellow solid (81.3 mg, 82% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.33 – 7.27 (m, 2H), 7.26 – 7.19 (m, 1H), 7.07 – 7.00 (m, 2H), 4.64 (d, *J* = 8.5 Hz, 1H), 4.55 – 4.33 (m, 4H), 3.75 – 3.63 (m, 1H), 2.70 – 2.54 (m, 1H), 2.45 (s, 3H), 2.42 – 2.31 (m, 2H), 2.30 – 2.20 (m, 1H), 1.59 – 1.48 (m, 2H), 1.44 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.9, 144.2, 136.8, 132.8, 132.0, 129.8, 128.8, 128.3, 127.7, 126.5, 123.5, 117.0, 80.3, 59.2, 58.4, 46.7, 31.4, 28.2, 27.6, 24.0, 21.6.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{27}H_{33}N_3O_4NaS^+$: 518.2084, found 518.2085.



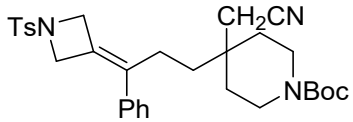
Benzyl (1-cyano-5-phenyl-5-(1-tosylazetidin-3-ylidene)pentan-2-yl)carbamate (3l). Yellow solid (92.1 mg, 87% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.0 Hz, 2H), 7.45 – 7.32 (m, 7H), 7.30 (t, *J* = 7.6 Hz,

2H), 7.24 (t, *J* = 7.3 Hz, 1H), 7.03 (d, *J* = 7.5 Hz, 2H), 5.09 (s, 2H), 4.99 (d, *J* = 8.6 Hz, 1H), 4.51 – 4.35 (m, 4H), 3.85 – 3.66 (m, 1H), 2.71 – 2.54 (m, 1H), 2.44 (s, 3H), 2.42 – 2.37 (m, 1H), 2.37 – 2.30 (m, 1H), 2.30 – 2.19 (m, 1H), 1.64 – 1.45 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 155.5, 144.2, 136.7, 135.9, 132.7, 131.9, 129.8, 128.8, 128.6, 128.3, 128.1, 127.7, 126.5, 123.6, 116.9, 67.1, 59.2, 58.4, 47.4, 31.4, 27.6, 23.9, 21.6.

HRMS (ESI) $[M+H]^+$: calculated for $C_{30}H_{32}N_3O_4S^+$: 530.2108, found 530.2115.

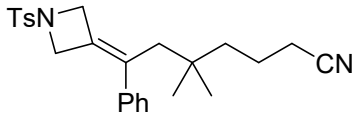


***tert*-Butyl 4-(cyanomethyl)-4-(3-phenyl-3-(1-tosylazetidin-3-ylidene)propyl)piperidine-1-carboxylate (3m).** Yellow solid (90.1 mg, 82% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.34 – 7.28 (m, 2H), 7.26 – 7.21 (m, 1H), 7.13 – 6.91 (m, 2H), 4.52 (s, 2H), 4.48 (s, 2H), 3.38 – 3.27 (m, 4H), 2.45 (s, 3H), 2.34 (s, 2H), 2.23 – 2.12 (m, 2H), 1.47 – 1.41 (m, 15H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.5, 144.2, 136.9, 133.3, 132.0, 129.9, 128.8, 128.4, 127.7, 126.4, 122.9, 117.2, 79.9, 59.3, 58.4, 35.2, 34.0, 28.3, 25.5, 24.8, 21.6.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{31}H_{39}N_3O_4NaS^+$: 572.2553, found 572.2561.

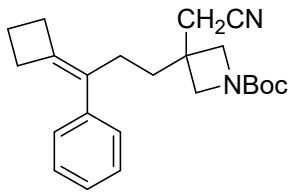


5,5-Dimethyl-7-phenyl-7-(1-tosylazetidin-3-ylidene)heptanenitrile (3n). Yellow oil (26.2 mg, 31% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 7.9 Hz, 2H), 7.30 – 7.25 (m, 2H), 7.24 – 7.18 (m, 1H), 7.06 (d, *J* = 7.1 Hz, 2H), 4.48 (s, 2H), 4.39 (s, 2H), 2.45 (s, 3H), 2.16 (s, 2H), 1.96 (t, *J* = 7.1 Hz, 2H), 1.46 – 1.38 (m, 2H), 1.12 – 1.06 (m, 2H), 0.69 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 144.2, 139.7, 133.6, 132.2, 129.8, 128.5, 128.3, 127.3, 126.7, 125.1, 119.5, 59.2, 59.0, 42.9, 41.2, 35.5, 27.3, 21.6, 20.4, 17.4.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{25}H_{30}N_2O_2NaS^+$: 445.1920, found 445.1929.

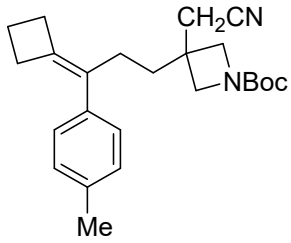


***tert*-Butyl 3-(cyanomethyl)-3-(3-cyclobutylidene-3-phenylpropyl)azetidine-1-carboxylate (3o).** Yellow oil (59.4 mg, 81% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.28 (m, 2H), 7.24 – 7.16 (m, 3H), 3.77 – 3.57 (m, 4H), 2.90 – 2.76 (m, 4H), 2.63 (s, 2H), 2.40 – 2.26 (m, 2H), 2.05 – 1.94 (m, 2H), 1.76 – 1.72 (m, 2H), 1.42 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.1, 139.4, 138.9, 129.0, 128.3, 127.0, 126.3, 116.9, 79.9, 35.5, 35.0, 31.9, 30.5, 28.3, 25.6, 25.3, 16.9.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{23}H_{30}N_2O_2Na^+$: 389.2199, found 389.2207.

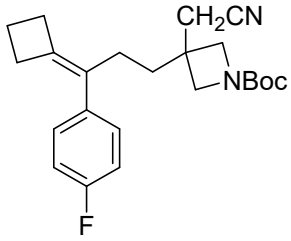


***tert*-Butyl 3-(cyanomethyl)-3-(3-cyclobutylidene-3-(*p*-tolyl)propyl)azetidine-1-carboxylate (3p).** Yellow oil (57.8 mg, 76% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17 – 7.06 (m, 4H), 3.80 – 3.58 (m, 4H), 2.86 – 2.75 (m, 4H), 2.63 (s, 2H), 2.33 (s, 3H), 2.33 – 2.26 (m, 2H), 2.06 – 1.93 (m, 2H), 1.79 – 1.71 (m, 2H), 1.42 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.1, 138.6, 136.0, 135.9, 129.0, 128.9, 126.9, 117.0, 80.0, 35.5, 35.1, 31.9, 30.5, 28.3, 25.7, 25.3, 21.1, 17.0.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{24}H_{32}N_2O_2Na^+$: 403.2356, found 403.2364.



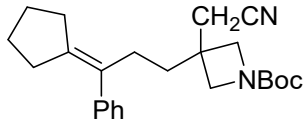
***tert*-Butyl 3-(cyanomethyl)-3-(3-cyclobutylidene-3-(4-fluorophenyl)propyl)azetidine-1-carboxylate (3q).** Yellow oil (65.4 mg, 85% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21 – 7.12 (m, 2H), 7.04 – 6.96 (m, 2H), 3.71 – 3.60 (m, 4H), 2.81 (t, *J* = 7.0 Hz, 2H), 2.76 (t, *J* = 7.2 Hz, 2H), 2.63 (s, 2H), 2.33 – 2.25 (m, 2H), 2.03 – 1.94 (m, 2H), 1.74 – 1.70 (m, 2H), 1.42 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 161.3 (d, *J* = 246.1 Hz), 156.1, 139.3, 134.9 (d, *J* = 3.2 Hz), 128.5 (d, *J* = 7.7 Hz), 128.2, 116.9, 115.2 (d, *J* = 21.3 Hz), 80.0, 35.5, 35.0, 31.7, 30.4, 28.3, 25.7, 25.5, 16.8.

^{19}F NMR (471 MHz, $CDCl_3$) δ -116.0.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{23}H_{29}N_2O_2NaF^+$: 407.2105, found 407.2112.

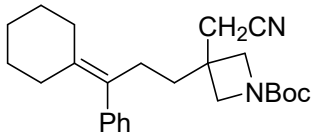


***tert*-Butyl 3-(cyanomethyl)-3-(3-cyclopentylidene-3-phenylpropyl)azetidine-1-carboxylate (3r).** Yellow oil (32.8 mg, 43% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (t, *J* = 7.6 Hz, 2H), 7.21 (t, *J* = 7.4 Hz, 1H), 7.17 – 7.11 (m, 2H), 3.73 – 3.56 (m, 4H), 2.60 (s, 2H), 2.45 – 2.24 (m, 4H), 2.15 (t, *J* = 7.2 Hz, 2H), 1.78 – 1.67 (m, 4H), 1.60 – 1.54 (m, 2H), 1.42 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.1, 142.7, 141.4, 129.8, 128.1(8), 128.1(9), 126.3, 117.0, 80.0, 35.4, 34.9, 32.6, 30.6, 29.6, 28.3, 26.9, 26.4, 25.7.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{24}H_{32}N_2O_2Na^+$: 403.2356, found 403.2364.

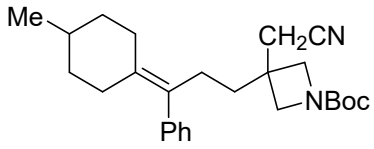


***tert*-Butyl 3-(cyanomethyl)-3-(3-cyclohexylidene-3-phenylpropyl)azetidine-1-carboxylate (3s).** Yellow oil (21.3 mg, 27% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 (t, *J* = 7.5 Hz, 2H), 7.22 (t, *J* = 7.4 Hz, 1H), 7.07 (d, *J* = 6.7 Hz, 2H), 3.64 (s, 4H), 2.57 (s, 2H), 2.37 – 2.24 (m, 4H), 1.95 (t, *J* = 6.1 Hz, 2H), 1.74 – 1.66 (m, 2H), 1.61 – 1.52 (m, 4H), 1.46 – 1.40 (m, 2H), 1.42 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.1, 143.0, 137.2, 130.2, 128.9, 128.2, 126.2, 116.9, 80.0, 35.6, 35.4, 32.2, 30.5, 28.5, 28.4, 28.3, 26.7, 25.8.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{25}H_{34}N_2O_2Na^+$: 417.2512, found 417.2519.

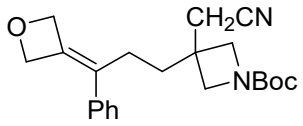


***tert*-Butyl 3-(cyanomethyl)-3-(3-(4-methylcyclohexylidene)-3-phenylpropyl)azetidine-1-carboxylate (3t).** Yellow oil (17.2 mg, 21% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 (t, *J* = 7.5 Hz, 2H), 7.22 (t, *J* = 7.4 Hz, 1H), 7.11 – 7.04 (m, 2H), 3.64 – 3.63 (m, 4H), 2.70 – 2.62 (m, 1H), 2.57 (s, 2H), 2.39 – 2.28 (m, 2H), 2.27 – 2.19 (m, 1H), 1.99 –1.83 (m, 2H), 1.74 – 1.63 (m, 6H), 1.42 (s, 9H), 1.07 – 1.02 (m, 1H), 0.90 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.2, 143.0, 136.9, 130.4, 128.9, 128.2, 126.3, 117.0, 80.0, 36.6(5), 36.6(3), 35.6, 35.4, 32.7, 31.5, 28.6, 28.3, 25.8, 22.0.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{26}H_{36}N_2O_2Na^+$: 431.2669, found 431.2678.

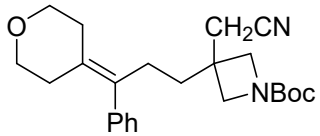


***tert*-Butyl 3-(cyanomethyl)-3-(3-(oxetan-3-ylidene)-3-phenylpropyl)azetidine-1-carboxylate (3u).** Yellow solid (45.0 mg, 61% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (t, *J* = 7.6 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 1H), 7.12 (d, *J* = 7.6 Hz, 2H), 5.37 (s, 2H), 5.35 (s, 2H), 3.69 (s, 4H), 2.64 (s, 2H), 2.34 – 2.15 (m, 2H), 1.87 – 1.72 (m, 2H), 1.43 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.1, 137.0, 132.8, 128.9, 128.8, 127.4, 126.3, 116.7, 80.2, 79.6, 78.8, 35.3, 34.9, 28.3, 25.7, 25.4.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{22}H_{28}N_2O_3Na^+$: 391.1992, found 391.1999.



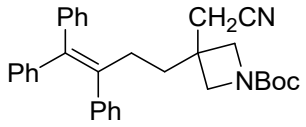
***tert*-Butyl 3-(cyanomethyl)-3-(3-phenyl-3-(tetrahydro-4*H*-pyran-4-ylidene)propyl)azetidine-1-carboxylate (3v).** Yellow solid (23.0 mg, 29% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.5 Hz, 2H), 7.27 – 7.22 (m, 1H), 7.12 – 7.01 (m, 2H), 3.76 (t, *J* = 5.4 Hz, 2H), 3.65 – 3.61 (m, 4H), 3.58 (t, *J* = 5.5 Hz, 2H), 2.58 (s, 2H), 2.44 (t, *J* = 5.4

Hz, 2H), 2.40 – 2.31 (m, 2H), 2.11 (t, *J* = 5.4 Hz, 2H), 1.73 – 1.66 (m, 2H), 1.42 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.1, 141.8, 132.5, 131.9, 128.8, 128.4, 126.7, 116.8, 80.1, 69.2, 68.9, 35.4, 35.3, 32.6, 31.0, 28.3, 28.2, 25.8.

HRMS (ESI) [M+Na]$^+$: calculated for $C_{24}H_{32}N_2O_3Na^+$: 419.2305, found 419.2314.

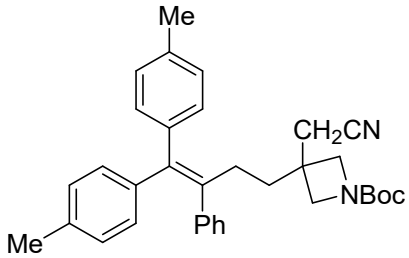


***tert*-Butyl 3-(cyanomethyl)-3-(3,4,4-triphenylbut-3-en-1-yl)azetidine-1-carboxylate (3w).** Yellow solid (43.1 mg, 45% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (t, *J* = 7.5 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H), 7.28 – 7.24 (m, 2H), 7.22 – 7.17 (m, 2H), 7.17 – 7.13 (m, 1H), 7.13 – 7.09 (m, 2H), 7.07 – 6.97 (m, 3H), 6.91 – 6.86 (m, 2H), 3.57 (d, *J* = 8.8 Hz, 2H), 3.51 (d, *J* = 8.9 Hz, 2H), 2.44 (s, 2H), 2.42 – 2.36 (m, 2H), 1.85 – 1.72 (m, 2H), 1.41 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.0, 142.9, 142.2, 141.4, 140.7, 138.3, 130.5, 129.4, 129.1, 128.5, 128.2, 127.5, 127.1, 126.7, 126.1, 116.8, 79.9, 35.6, 35.5, 30.6, 28.3, 25.4.

HRMS (ESI) [M+Na]$^+$: calculated for $C_{32}H_{34}N_2O_2Na^+$: 501.2512, found 501.2518.

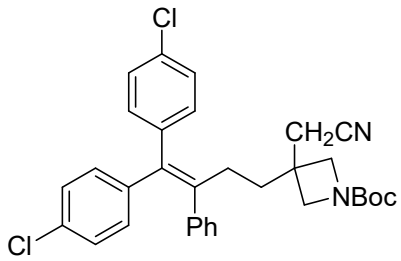


***tert*-Butyl 3-(cyanomethyl)-3-(3-phenyl-4,4-di-*p*-tolylbut-3-en-1-yl)azetidine-1-carboxylate (3x).** Yellow solid (53.7 mg, 53% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.23 – 7.17 (m, 4H), 7.17 – 7.10 (m, 5H), 6.82 (d, *J* = 8.0 Hz, 2H), 6.75 (d, *J* = 8.2 Hz, 2H), 3.69 – 3.46 (m, 4H), 2.46 (s, 2H), 2.44 – 2.39 (m, 2H), 2.37 (s, 3H), 2.19 (s, 3H), 1.81 – 1.72 (m, 2H), 1.42 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.0, 141.7, 140.5, 140.2, 139.5, 137.4, 136.7, 135.6, 130.5, 129.4, 129.1, 129.0, 128.2, 128.1, 126.5, 116.9, 79.9, 35.6, 35.5, 30.6, 28.3, 25.5, 21.2, 21.0.

HRMS (ESI) [M+Na]$^+$: calculated for $C_{34}H_{38}N_2O_2Na^+$: 529.2825, found 529.2833.

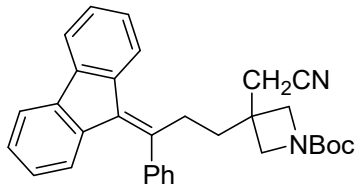


***tert*-Butyl 3-(4,4-bis(4-chlorophenyl)-3-phenylbut-3-en-1-yl)-3-(cyanomethyl)azetidine-1-carboxylate (3y).** Yellow solid (52.5 mg, 48% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.36 (m, 2H), 7.25 – 7.15 (m, 5H), 7.11 – 7.05 (m, 2H), 7.01 – 6.97 (m, 2H), 6.80 – 6.73 (m, 2H), 3.58 (d, *J* = 8.9 Hz, 2H), 3.52 (d, *J* = 8.9 Hz, 2H), 2.48 (s, 2H), 2.43 – 2.33 (m, 2H), 1.83 – 1.72 (m, 2H), 1.41 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.0, 140.8, 140.7, 140.3, 139.6, 138.2, 133.3, 132.2, 131.9, 130.6, 129.2, 128.9, 128.5, 127.8, 127.1, 116.8, 80.1, 35.5, 35.3, 30.6, 28.3, 25.5.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{32}H_{32}N_2O_2NaCl_2^+$: 569.1733, found 569.1735.

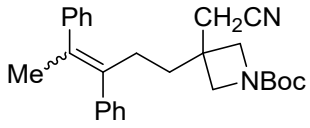


***tert*-Butyl 3-(3-(9*H*-fluoren-9-ylidene)-3-phenylpropyl)-3-(cyanomethyl)azetidine-1-carboxylate (3z).** Yellow solid (63.9 mg, 67% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 – 7.84 (m, 1H), 7.82 – 7.73 (m, 1H), 7.67 (d, *J* = 7.4 Hz, 1H), 7.56 – 7.44 (m, 3H), 7.44 – 7.36 (m, 2H), 7.33 – 7.28 (m, 2H), 7.19 (t, *J* = 7.0 Hz, 1H), 6.92 – 6.78 (m, 1H), 6.12 (d, *J* = 8.0 Hz, 1H), 3.79 – 3.65 (m, 4H), 3.13 – 3.01 (m, 2H), 2.70 (s, 2H), 2.15 – 2.05 (m, 2H), 1.44 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.1, 144.2, 143.0, 140.7, 139.8, 138.3, 137.9, 133.9, 129.3, 128.2, 128.1, 127.9, 127.3(3), 127.2(7), 126.6, 124.8, 124.3, 119.8, 119.1, 116.8, 80.2, 35.6, 33.1, 32.6, 28.3, 25.8.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{32}H_{32}N_2O_2Na^+$: 499.2356, found 499.2364.



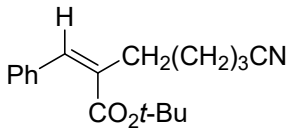
***tert*-Butyl (*Z*)-3-(cyanomethyl)-3-(3,4-diphenylpent-3-en-1-yl)azetidine-1-carboxylate (3aa).** Yellow oil (38.2 mg, 46% yield; *Z*/*E* = 10:1.6).

***Z*-3aa** (major isomer):

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.12 – 7.00 (m, 6H), 6.97 – 6.89 (m, 4H), 3.75 – 3.64 (m, 4H), 2.63 (s, 2H), 2.59 – 2.52 (m, 2H), 2.20 (s, 3H), 1.84 – 1.75 (m, 2H), 1.43 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.1, 144.0, 142.4, 135.9, 134.3, 129.6, 129.0, 127.8, 127.6, 126.1, 125.8, 116.9, 80.1, 35.4, 34.7, 29.6, 28.3, 25.8, 21.0.

HRMS (ESI) [M+Na]$^+$: calculated for $C_{27}H_{32}N_2O_2Na^+$: 439.2356, found 439.2365.

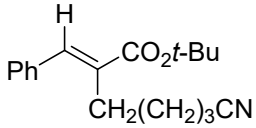


***tert*-Butyl (*Z*)-2-benzylidene-6-cyanohexanoate (*Z*-6).** Yellow oil (42.3 mg, 74% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (d, *J* = 6.9 Hz, 2H), 7.27 – 7.23 (m, 3H), 6.59 (s, 1H), 2.43 (t, *J* = 6.5 Hz, 2H), 2.39 (t, *J* = 6.7 Hz, 2H), 1.79 – 1.66 (m, 4H), 1.34 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.7, 136.4, 135.4, 132.5, 128.1, 128.0, 127.5, 119.5, 81.4, 34.7, 27.7, 27.3, 24.7, 17.0.

HRMS (ESI) [M+Na]$^+$: calculated for $C_{18}H_{23}NO_2Na^+$: 308.1621, found 308.1630.

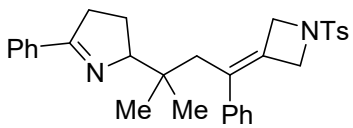


***tert*-Butyl (*E*)-2-benzylidene-6-cyanohexanoate (*E*-6).** Yellow oil (4.0 mg, 7% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (s, 1H), 7.39 (t, *J* = 7.2 Hz, 2H), 7.34 – 7.29 (m, 3H), 2.53 – 2.46 (m, 2H), 2.34 – 2.28 (m, 2H), 1.71 – 1.65 (m, 4H), 1.55 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 167.3, 138.7, 135.9, 134.0, 128.9, 128.5, 128.2, 119.6, 80.9, 28.2, 28.1, 26.5, 25.3, 16.8.

The characterization data were consistent with the literature.[3b]



2-(2-Methyl-4-phenyl-4-(1-tosylazetidin-3-ylidene)butan-2-yl)-5-phenyl-3,4-dihydro-2*H*-

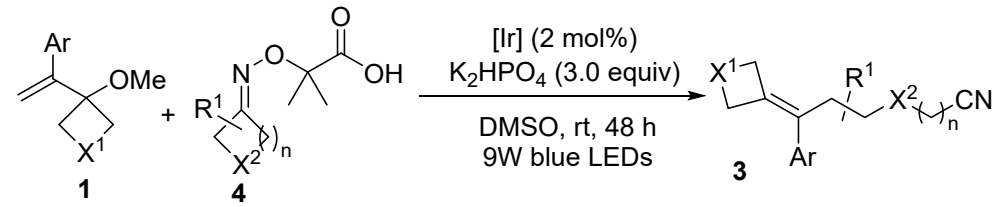
pyrrole (8). Yellow solid (97.7 mg, 98% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 – 7.76 (m, 2H), 7.68 (d, *J* = 8.2 Hz, 2H), 7.44 – 7.35 (m, 3H), 7.25 – 7.20 (m, 4H), 7.19 – 7.13 (m, 1H), 7.07 (d, *J* = 7.1 Hz, 2H), 4.61 – 4.45 (m, 3H), 4.33 – 4.31 (m, 1H), 3.80 (t, *J* = 7.9 Hz, 1H), 2.94 – 2.81 (m, 1H), 2.81 – 2.68 (m, 1H), 2.50 – 2.41 (m, 2H), 2.39 (s, 3H), 1.83 – 1.76 (m, 1H), 1.66 – 1.55 (m, 1H), 0.63 (s, 3H), 0.57 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.6, 143.9, 140.0, 134.7, 133.6, 131.9, 130.2, 129.6, 128.3(4), 128.3(1), 128.2(9), 127.5, 127.0, 126.7, 125.0, 80.7, 59.6, 59.4, 41.1, 39.4, 35.2, 24.6, 23.5, 23.2, 21.5.

HRMS (ESI) $[M+H]^+$: calculated for $C_{31}H_{35}N_2O_2S^+$: 499.2414, found 499.2421.

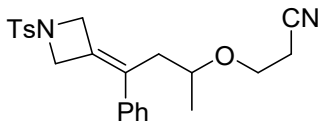
4. General procedure of photocatalytic reaction of α-imino-oxy acids with allyl ether



X^1 = NTs, CH_2 X^2 = CH_2, CHR^2, O, NR^3

n = 1, 2

Under a nitrogen atmosphere, to an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (4.5 mg, 0.004 mmol, 0.02 equiv), allyl ether **1** (0.2 mmol, 1.0 equiv), α-imino-oxy acids **4** (0.6 mmol, 3.0 equiv), K_2HPO_4 (0.6 mmol, 3.0 equiv) and degassed DMSO (4 mL) were added. The tube was evacuated and filled with nitrogen for 3 times. The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 48 h (cooling with a fan). After the reaction was complete, the reaction solution was quenched by the addition of sodium bicarbonate solution (5 mL) and extracted with EtOAc (5 × 10 mL). The combined organic layer was washed with brine, dried over Na_2SO_4, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product.



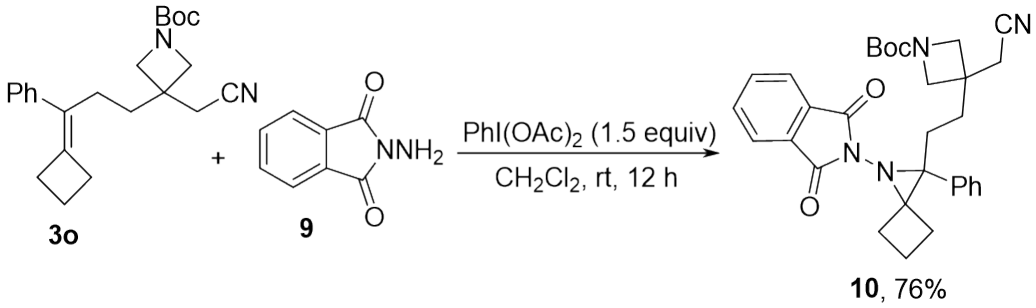
3-((4-Phenyl-4-(1-tosylazetidin-3-ylidene)butan-2-yl)oxy)propanenitrile (3ab). Yellow oil (22.2 mg, 27% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.2 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.33 – 7.27 (m, 2H), 7.25 – 7.20 (m, 1H), 7.11 – 7.05 (m, 2H), 4.56 – 4.41 (m, 4H), 3.60 – 3.47 (m, 1H), 3.39 – 3.26 (m, 2H), 2.45 (s, 3H), 2.53 – 2.40 (m, 1H), 2.38 – 2.23 (m, 3H), 1.04 (d, *J* = 6.1 Hz, 3H).

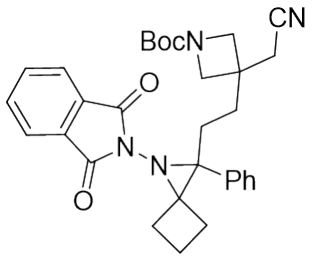
^{13}C NMR (126 MHz, $CDCl_3$) δ 144.2, 137.5, 132.1, 131.1, 129.8, 128.7, 128.4, 127.5, 126.6, 124.8, 117.7, 74.7, 63.3, 59.2, 59.0, 38.7, 21.6, 19.5, 19.0.

HRMS (ESI) $[M+H]^+$: calculated for $C_{23}H_{27}N_2O_3S$: 411.1737, found 411.1746.

5. Synthetic application



Under a nitrogen atmosphere, to an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, dimethyl phenyl-λ^3-iodanedicarboxylate (290 mg, 0.9 mmol, 1.5 equiv), *tert*-butyl 3-(cyanomethyl)-3-(3-cyclobutylidene-3-phenylpropyl)azetidine-1-carboxylate **3o** (220 mg, 0.6 mmol, 1.0 equiv), 2-aminoisoindoline-1,3-dione **9** (146 mg, 0.9 mmol, 1.5 equiv) and degassed DCM (5 mL) were added. The reaction was stirred at room temperature for 12 hours. After the reaction was complete, the reaction solution was quenched by the addition of sodium bicarbonate solution (4 mL) and extracted with EtOAc (4 x 10 mL). The combined organic layer was washed with brine, dried over Na_2SO_4, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product.



***tert*-butyl 3-(cyanomethyl)-3-(2-(1-(1,3-dioxoisoindolin-2-yl)-2-phenyl-1-azaspiro[2.3]hexan-2-yl)ethyl)azetidine-1-carboxylate (10).** Yellow solid (240 mg, 76% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 – 7.80 (m, 2H), 7.76 – 7.71 (m, 2H), 7.64 – 7.57 (m, 2H), 7.43 – 7.36 (m, 2H), 7.33 – 7.27 (m, 1H), 3.66 – 3.58 (m, 2H), 3.58 – 3.48 (m, 2H), 2.63 – 2.52 (m, 1H), 2.48 (s, 3H), 2.44 – 2.36 (m, 2H), 2.34 – 2.24 (m, 1H), 2.09 – 2.00 (m, 1H), 2.00 – 1.84 (m, 2H), 1.84 – 1.72 (m, 1H), 1.58 – 1.45 (m, 1H), 1.39 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 166.9, 156.0, 137.8, 134.2, 131.3, 128.9, 128.4, 127.5, 123.1, 116.7, 80.0, 56.5, 56.4, 35.2, 32.7, 28.9, 28.2, 26.5, 26.2, 25.6, 14.9.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{31}H_{34}N_4O_4Na^+$: 549.2472, found 549.2476.

6. References

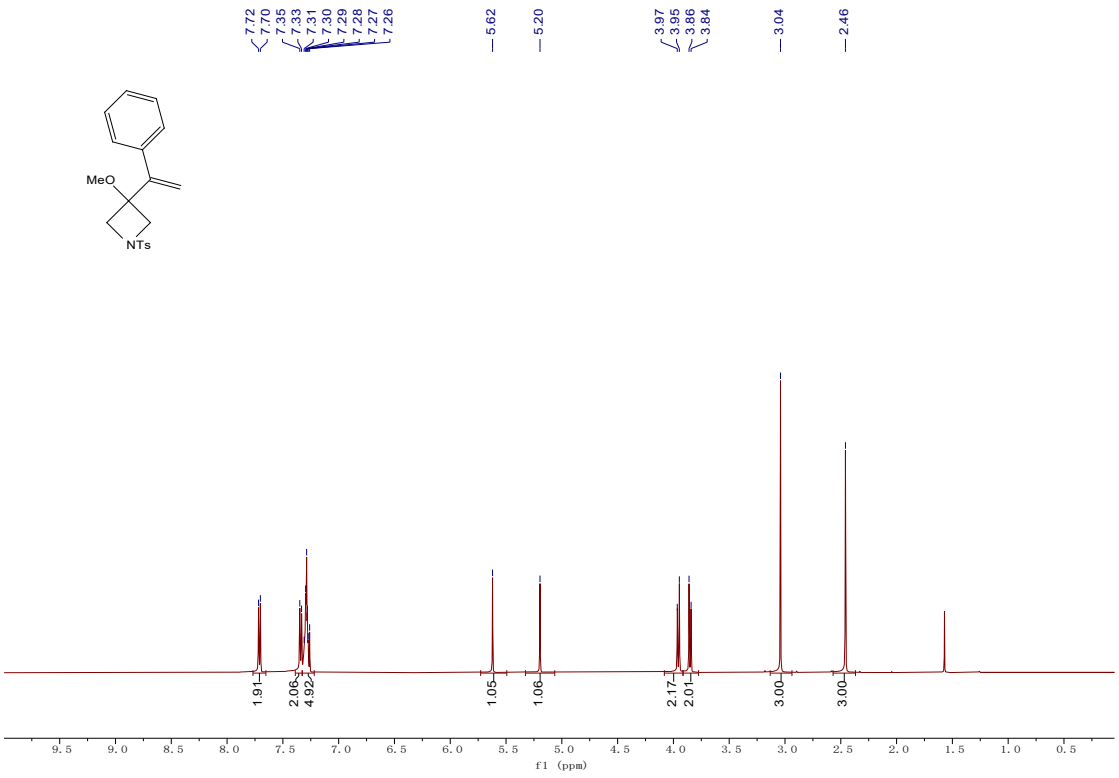
[1] (*a*) Q. Yin and S.-L. You, *Org. Lett.*, 2014, **16**, 1810; (*b*) J.-W. Dong, T. Ding, S.-Y. Zhang, Z.-M. Chen and Y.-Q. Tu, *Angew. Chem. Int. Ed.*, 2018, **57**, 13192; (*c*) H. I. Jung, Y. Kim and D. Y. Kim, *Org. Biomol. Chem.*, 2019, **17**, 3319; (*d*) S. Ma, Y. Guo, L. Liu, L. Shi, X. Lei, X. Duan and P. Jiao, *J. Org. Chem.*, 2023, **88**, 4743.

[2] Y. Lu, K. Yan, Y. Shi, C. Gao, Y. Zhu, Y. Zhang, T.-P. Loh and P. Xie, *Org. Chem. Front.*, 2025, **12**, 5947.

[3] (*a*) P.-Z. Wang, Y. Gao, J. Chen, X.-D. Huan, W.-J. Xiao and J.-R. Chen, *Nat. Commun.*, 2021, **12**, 1815; (*b*) Y. Bao, Z.-. Song, J.-L. Dai, S. Yan, Y. Zhang, J.-Y. Wang and G. Li, *Chin. J. Chem.*, 2024, **42**, 1399.

[4] (*a*) J. Davies, N. S. Sheikh and D. Leonori, *Angew. Chem. Int. Ed.*, 2017, **56**, 13361; (*b*) E. M. Dauncey, S. P. Morcillo, J. J. Douglas, N. S. Sheikh and D. Leonori, *Angew. Chem. Int. Ed.*, 2018, **57**, 744; (*c*) C. Tian and L. Shi, *Chin. J. Chem.*, 2025, **43**, 1988.

7. NMR Spectra of new compounds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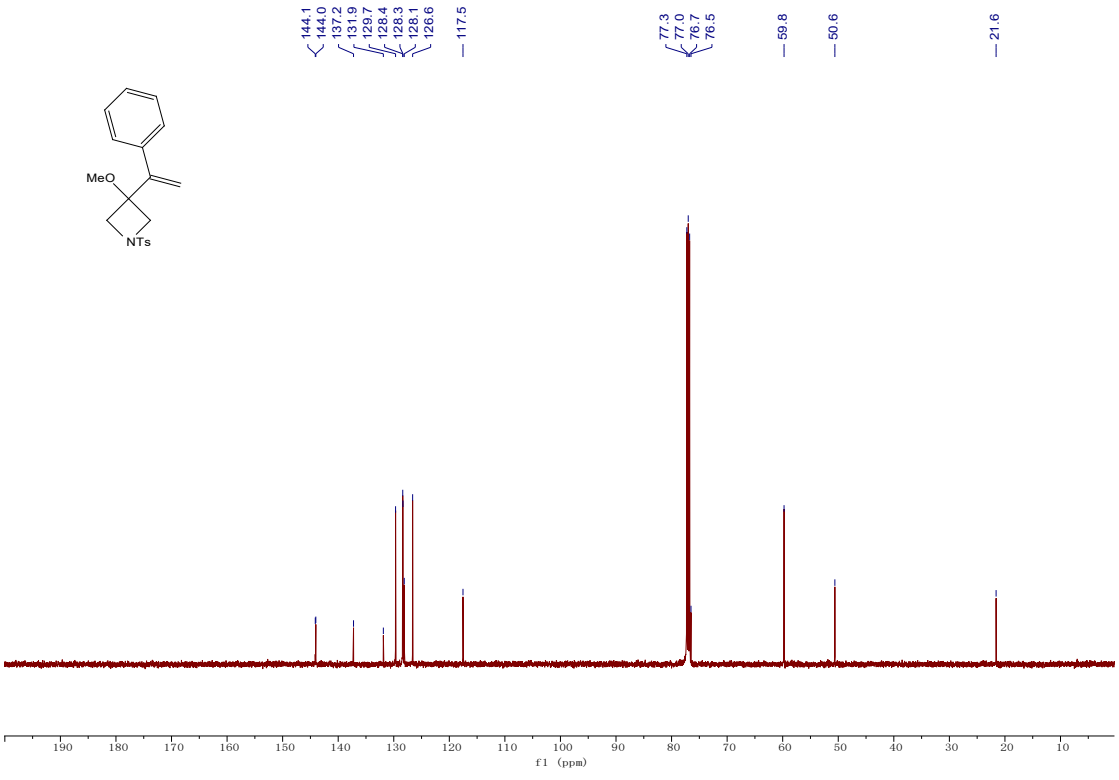
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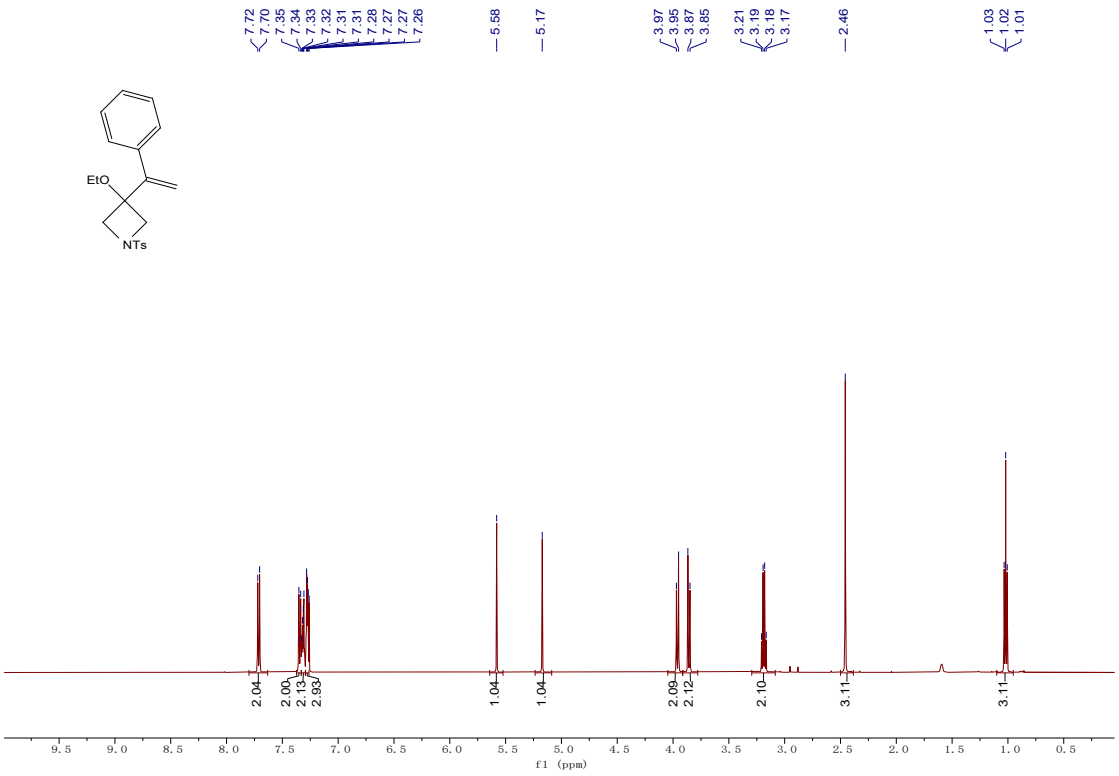
f1 (ppm)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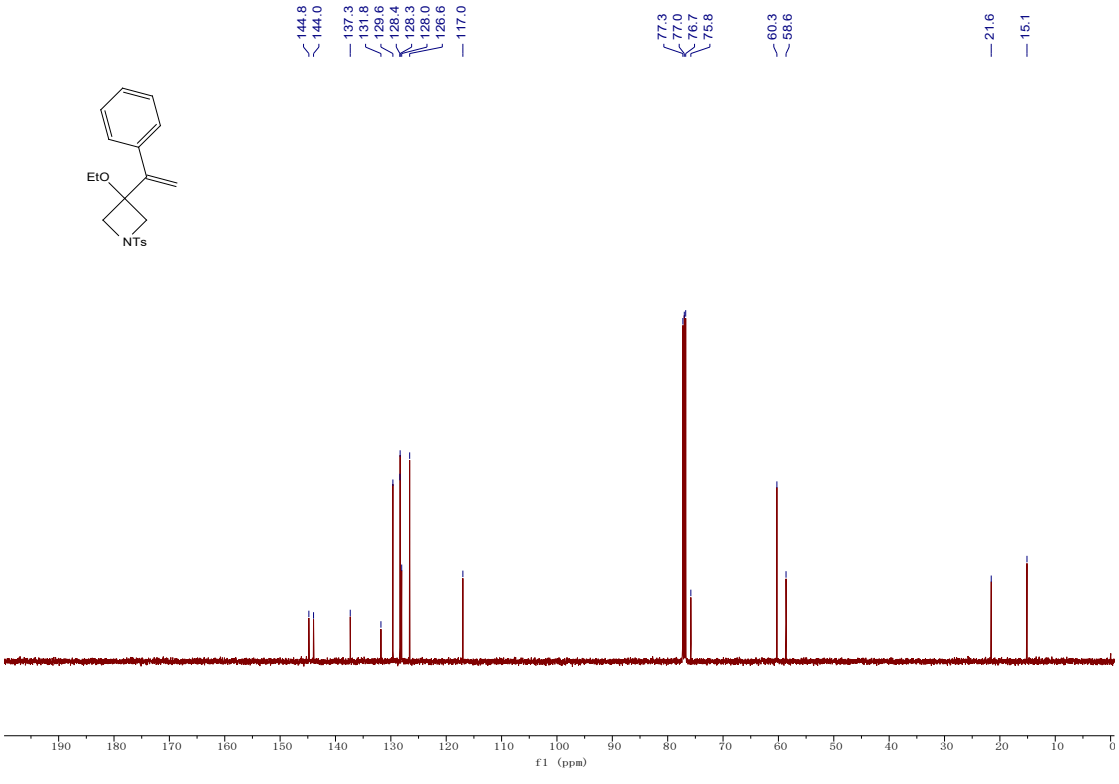
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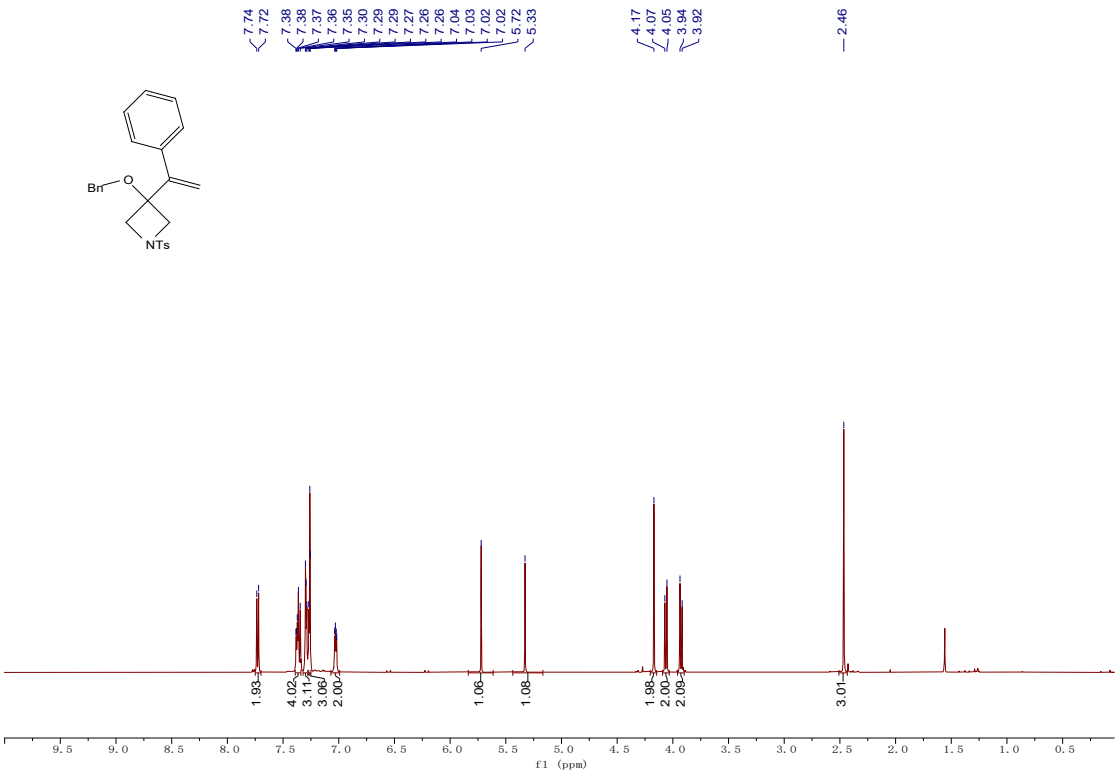
NTs
f1 (ppm)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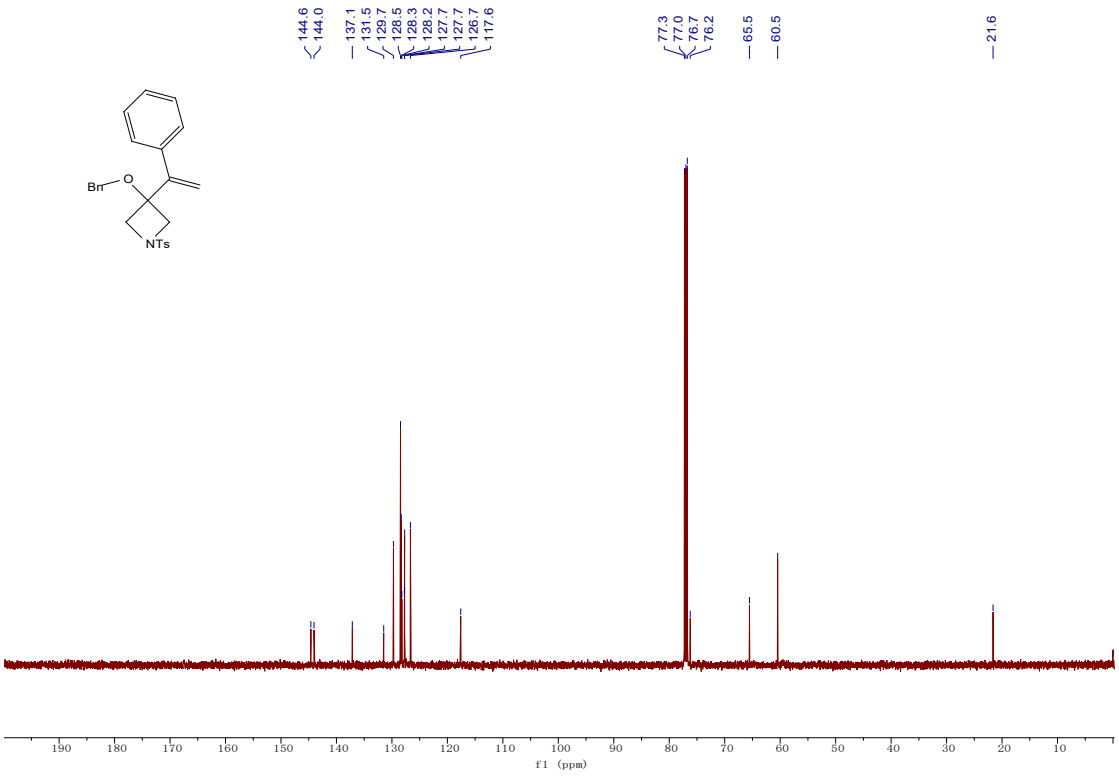
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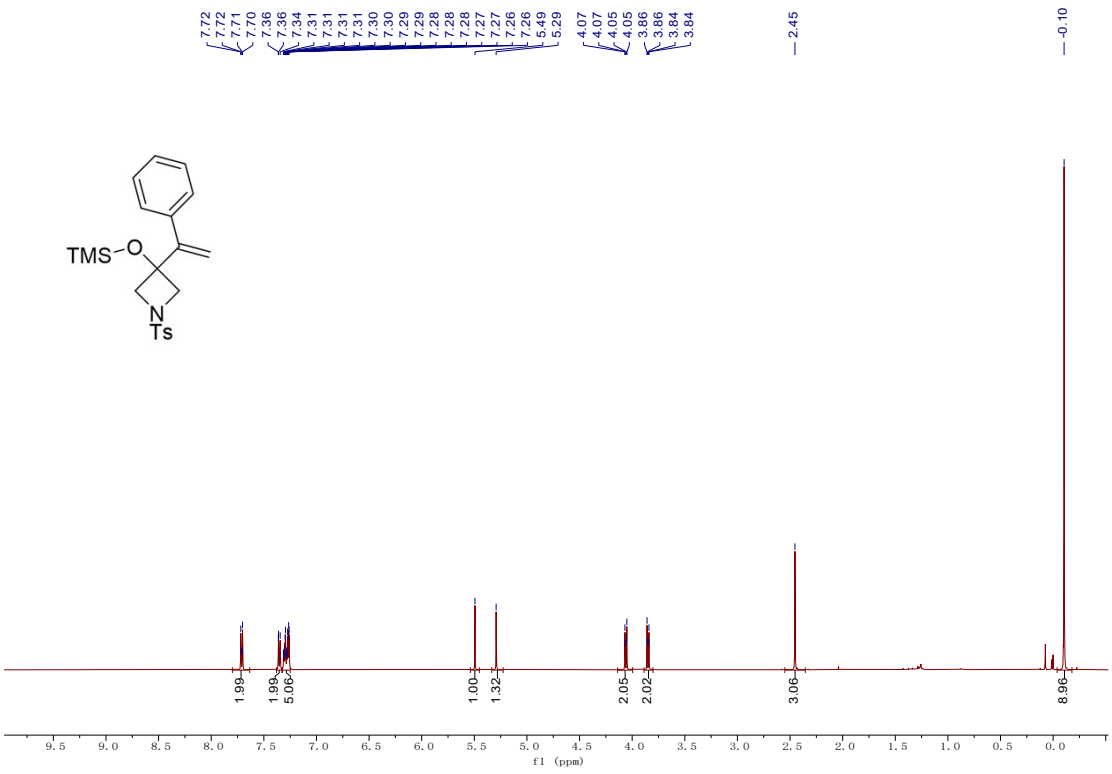
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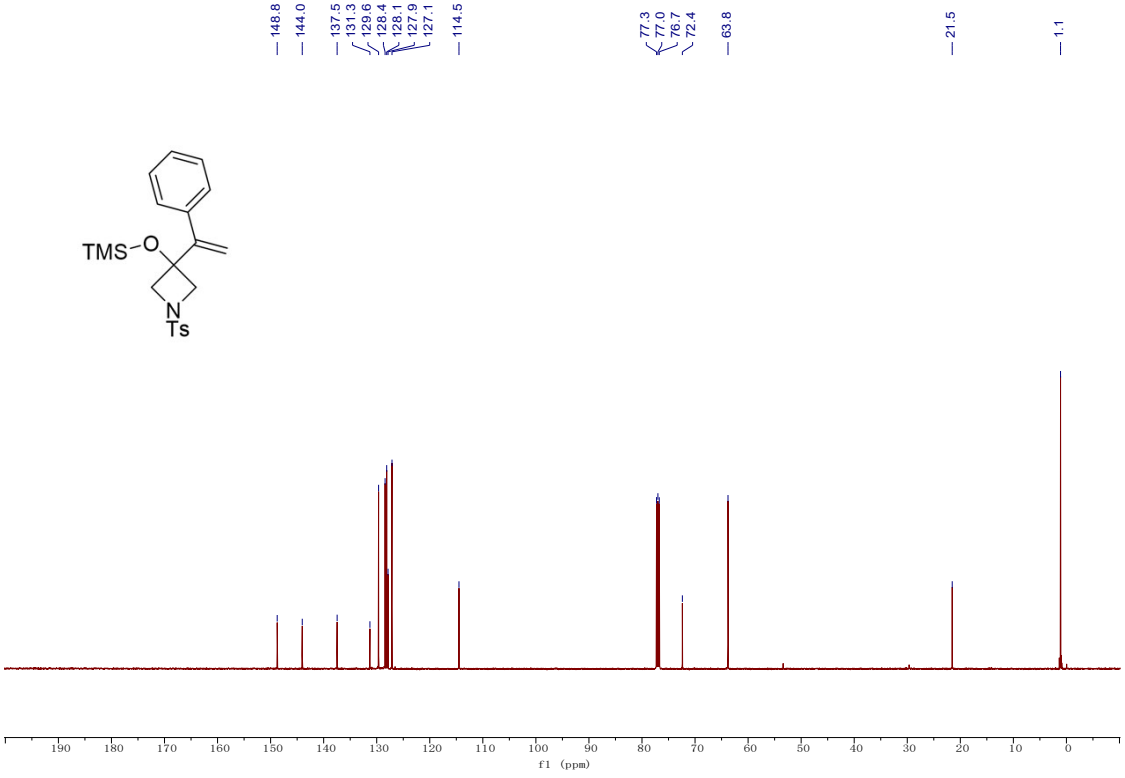
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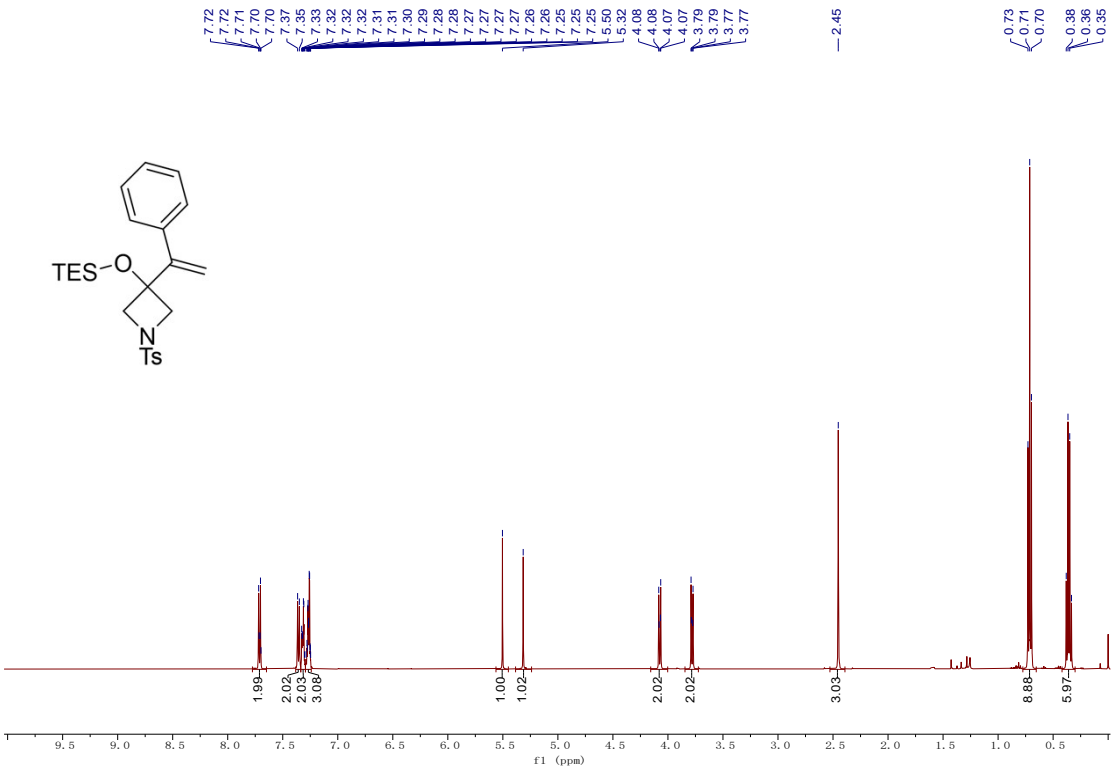
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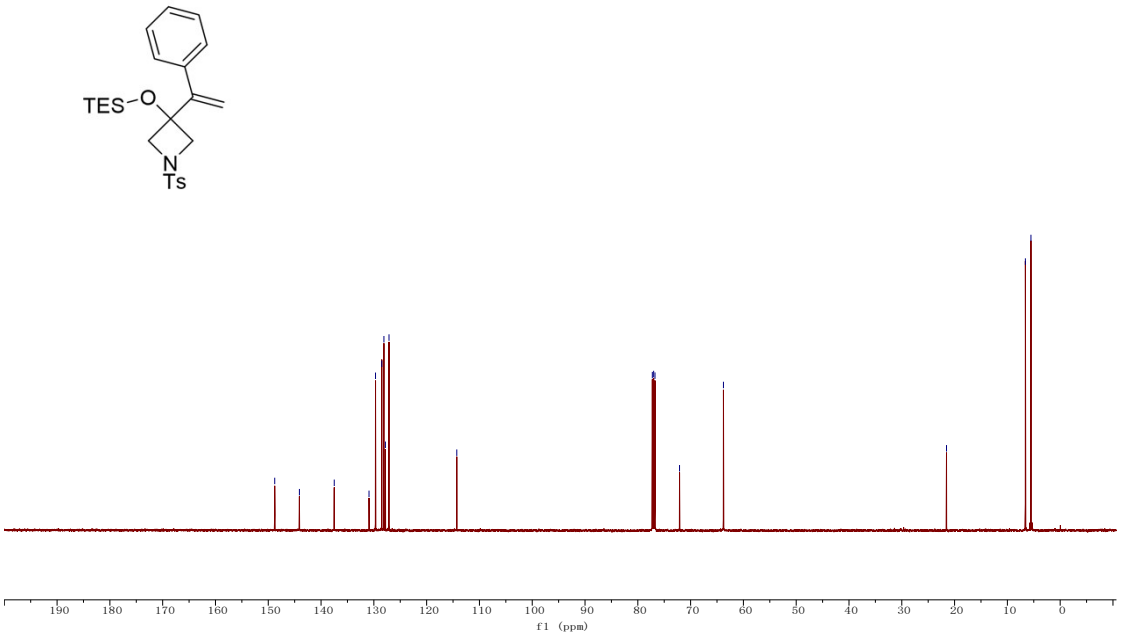
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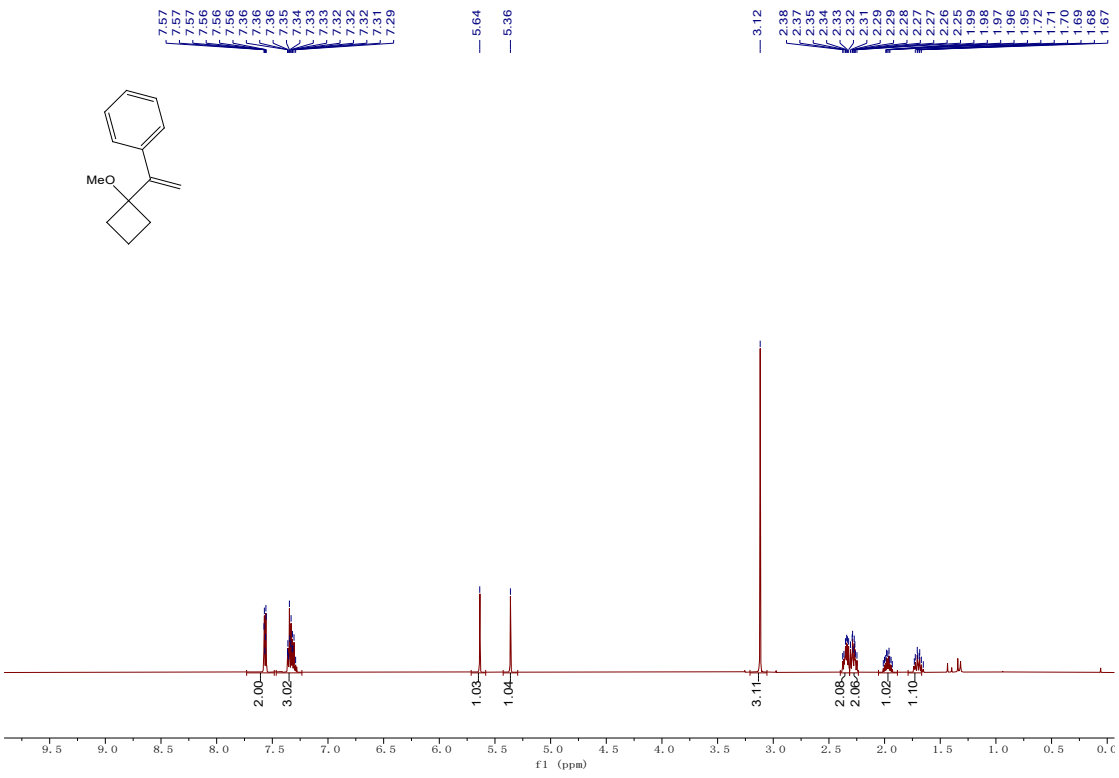
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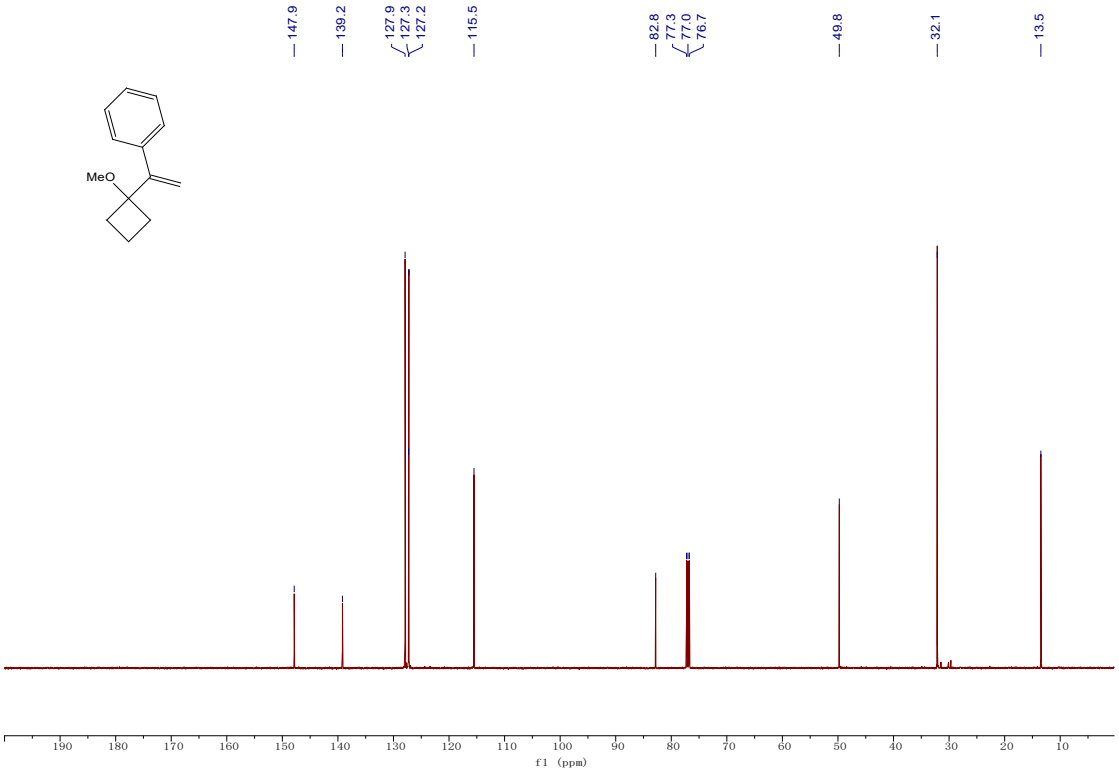
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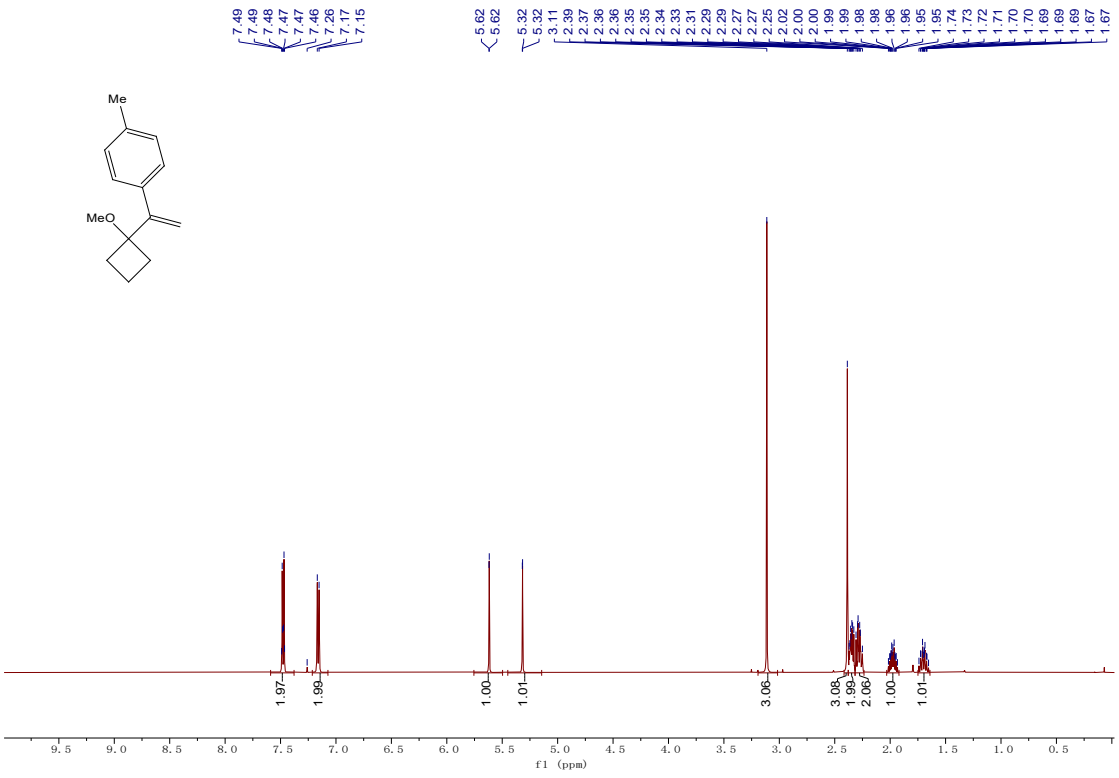
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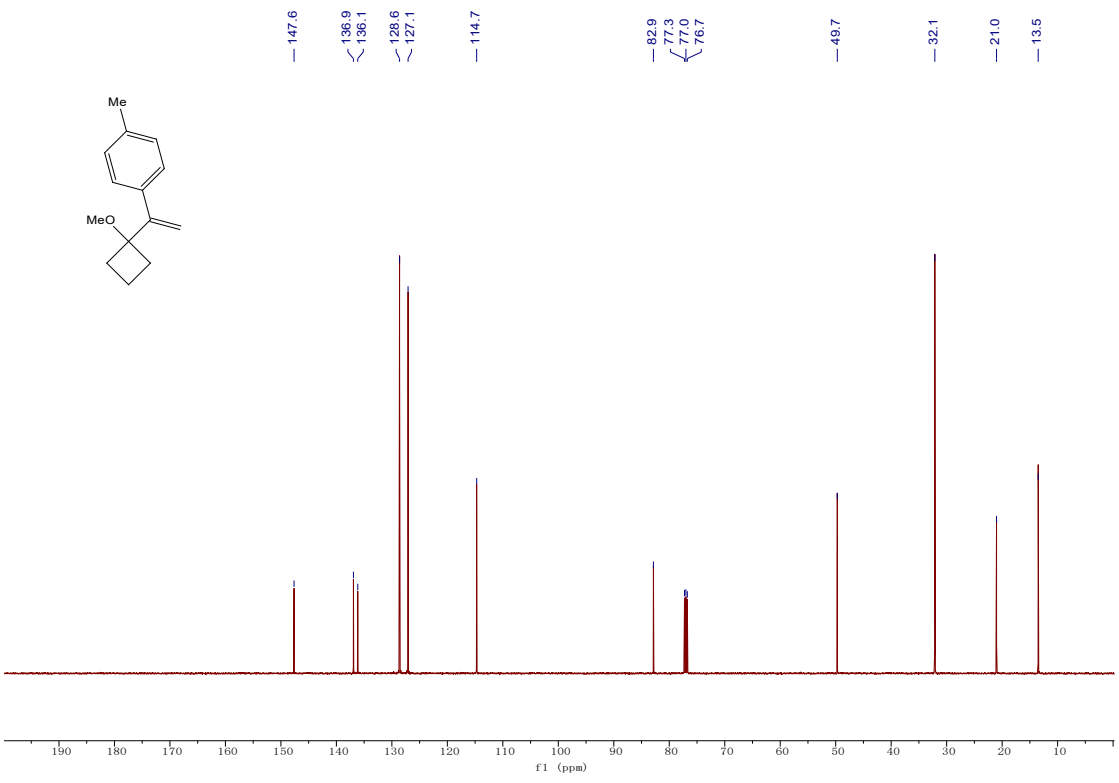
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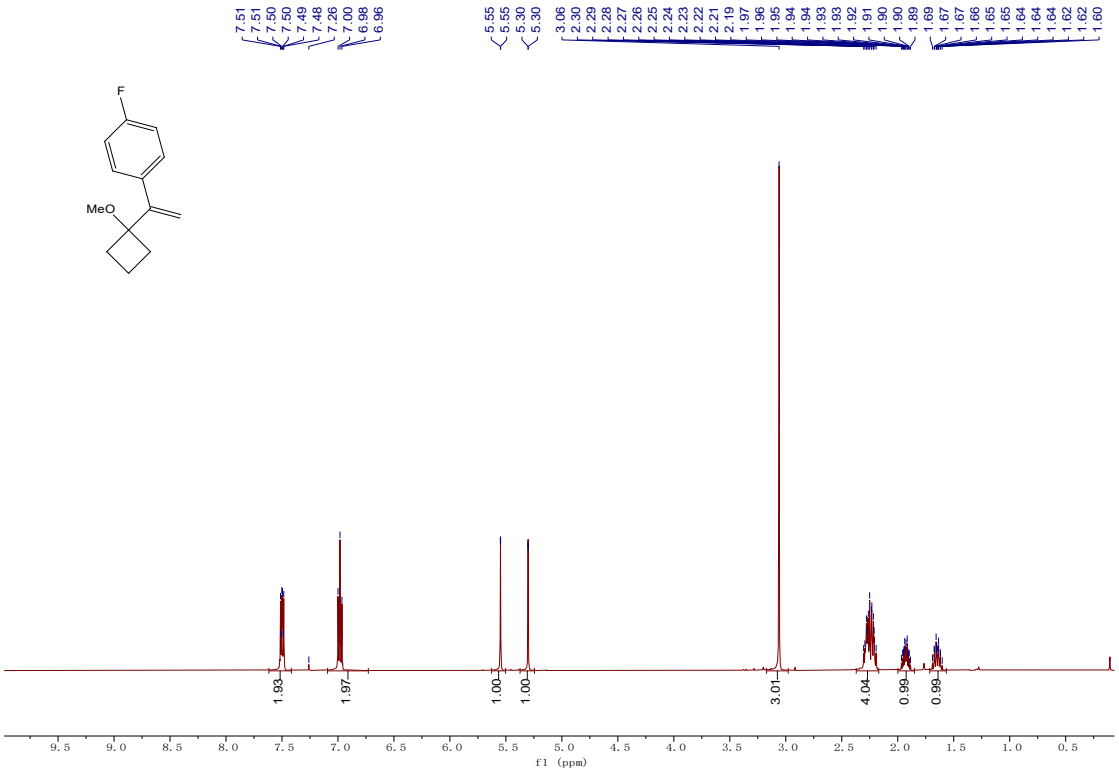
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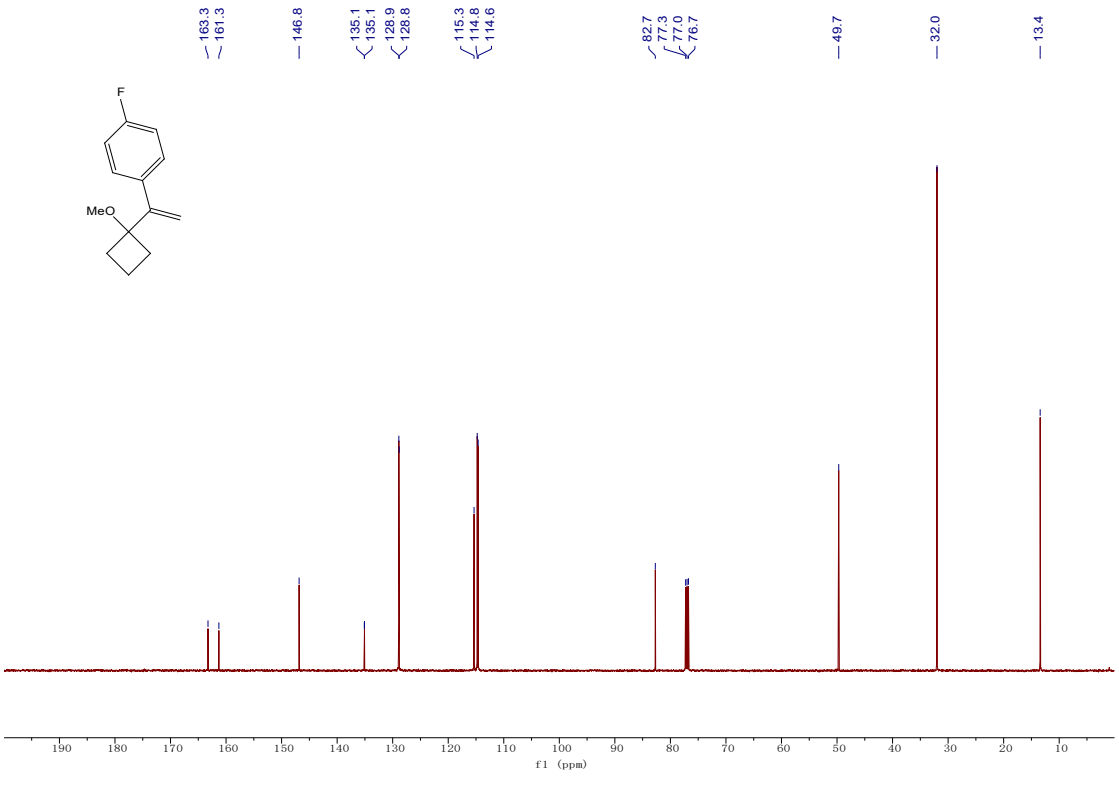
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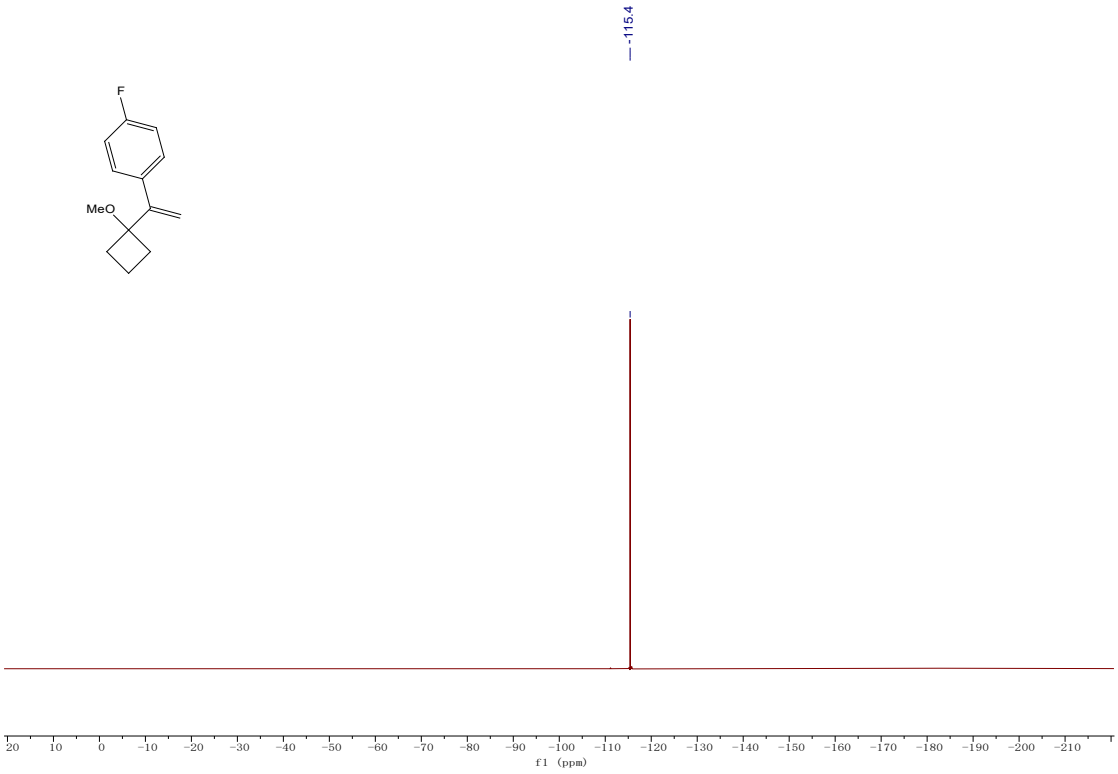
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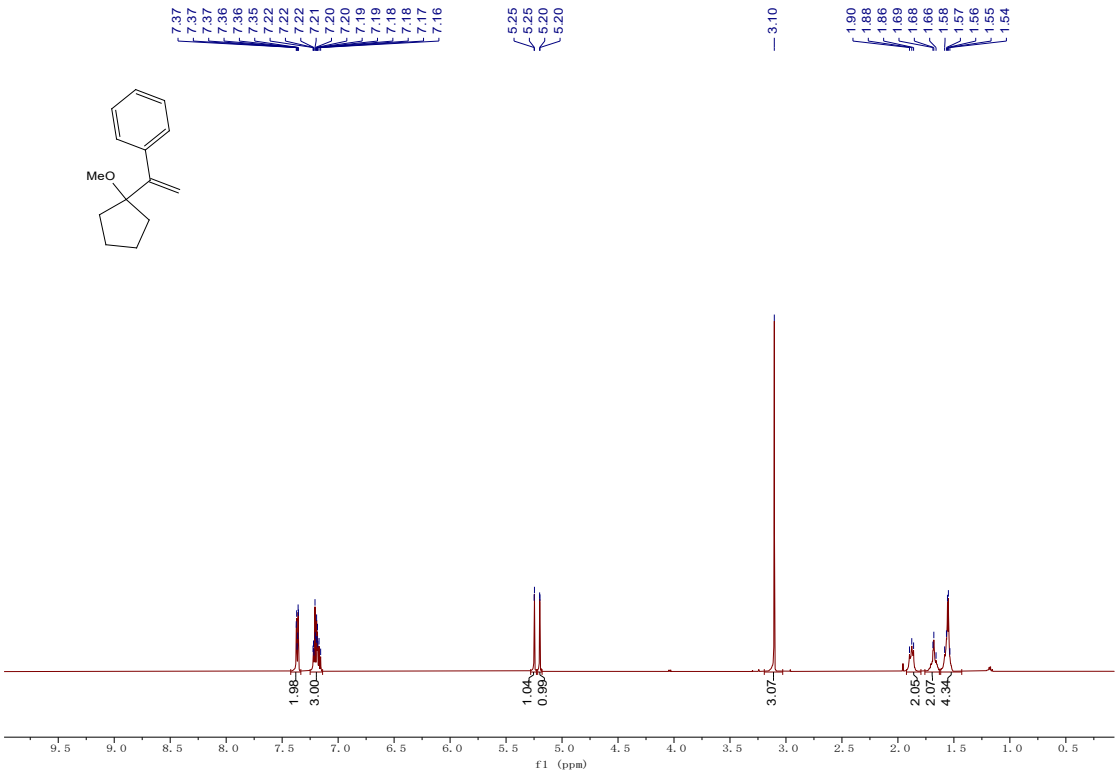
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1.55
1.54
MeO
1.98
3.00
1.04
0.99
3.07
2.05
2.07
4.34
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
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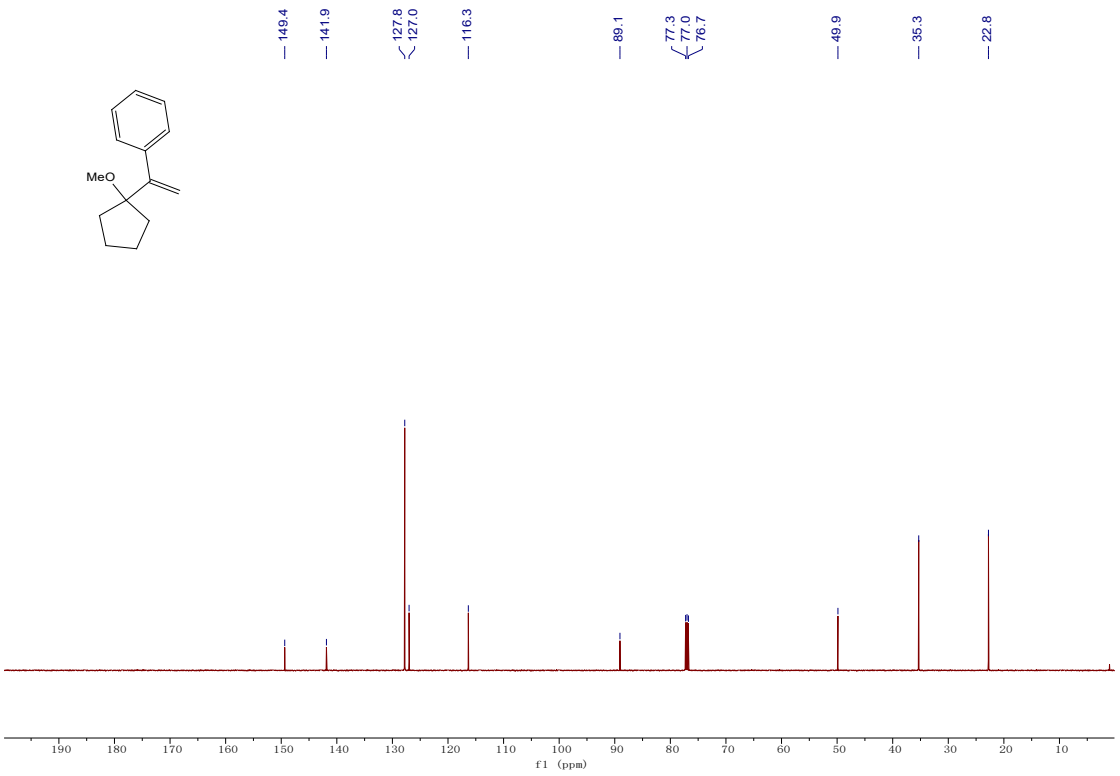
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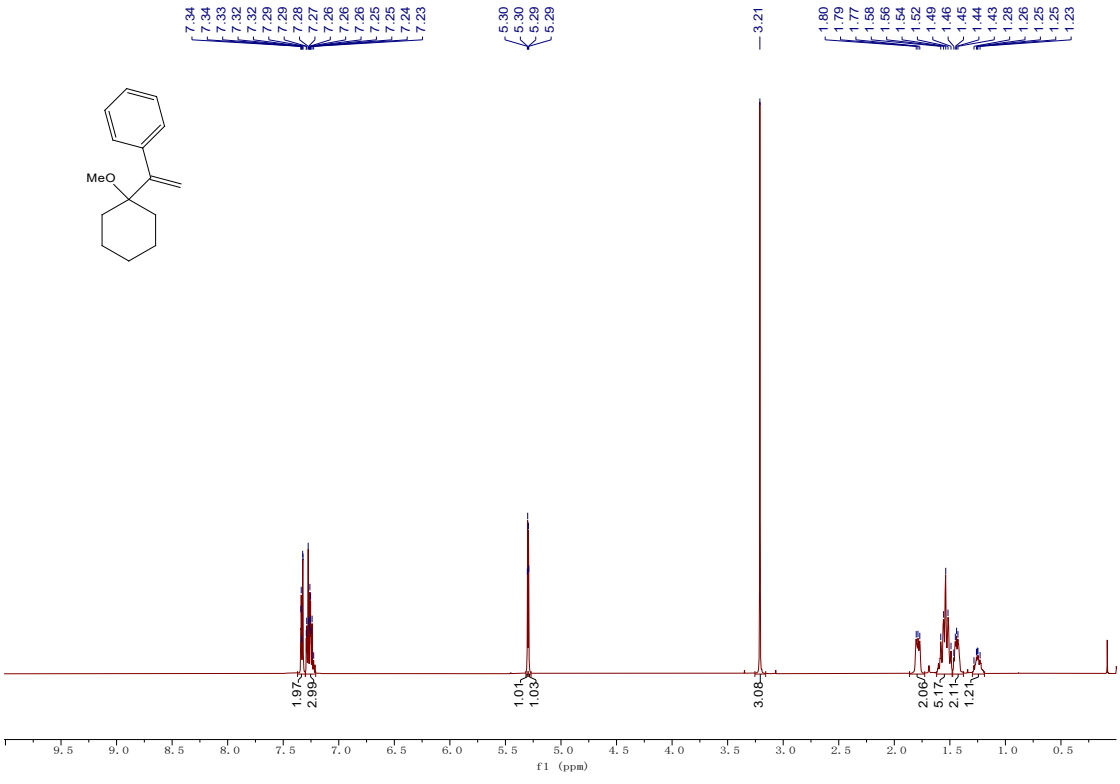
MeO
f1 (ppm)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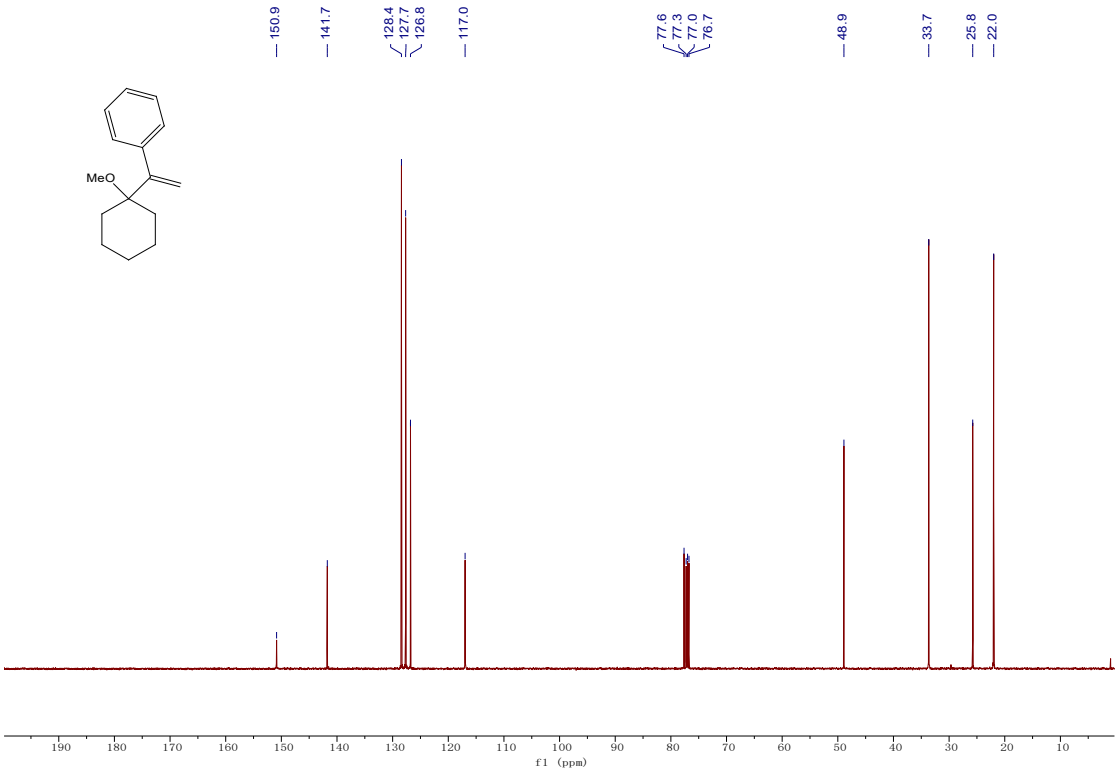
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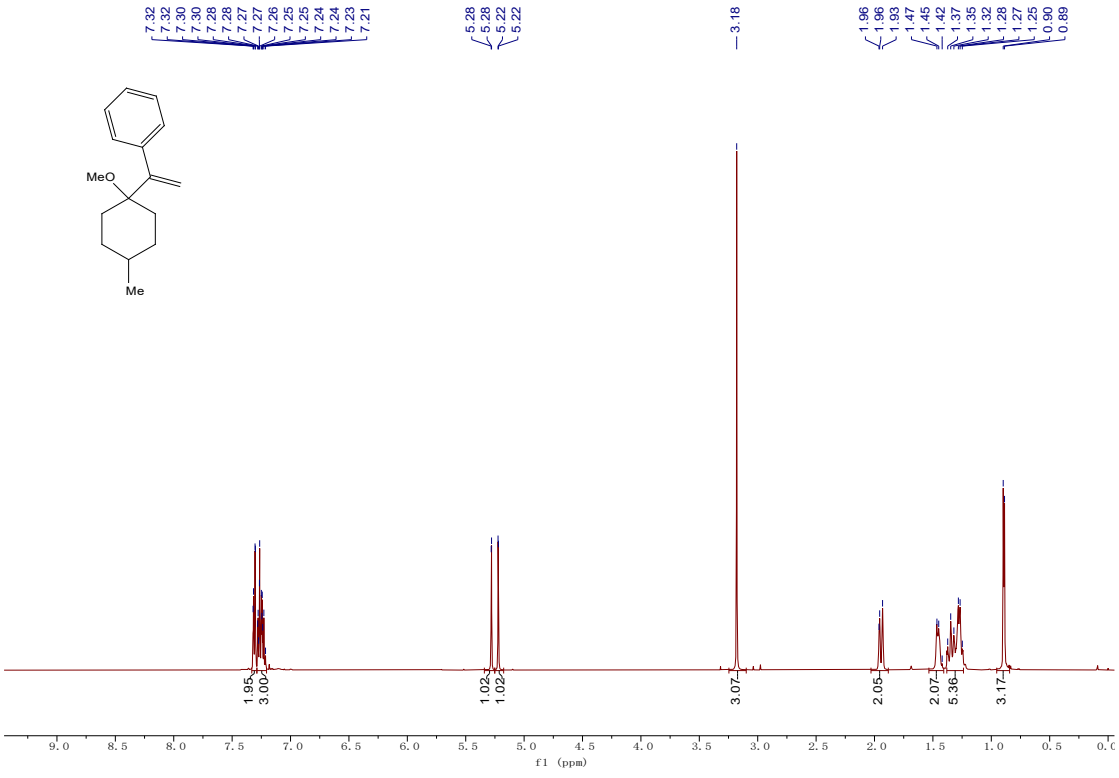
MeO
f1 (ppm)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0.90
0.89
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Me
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1.02
1.02
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2.07
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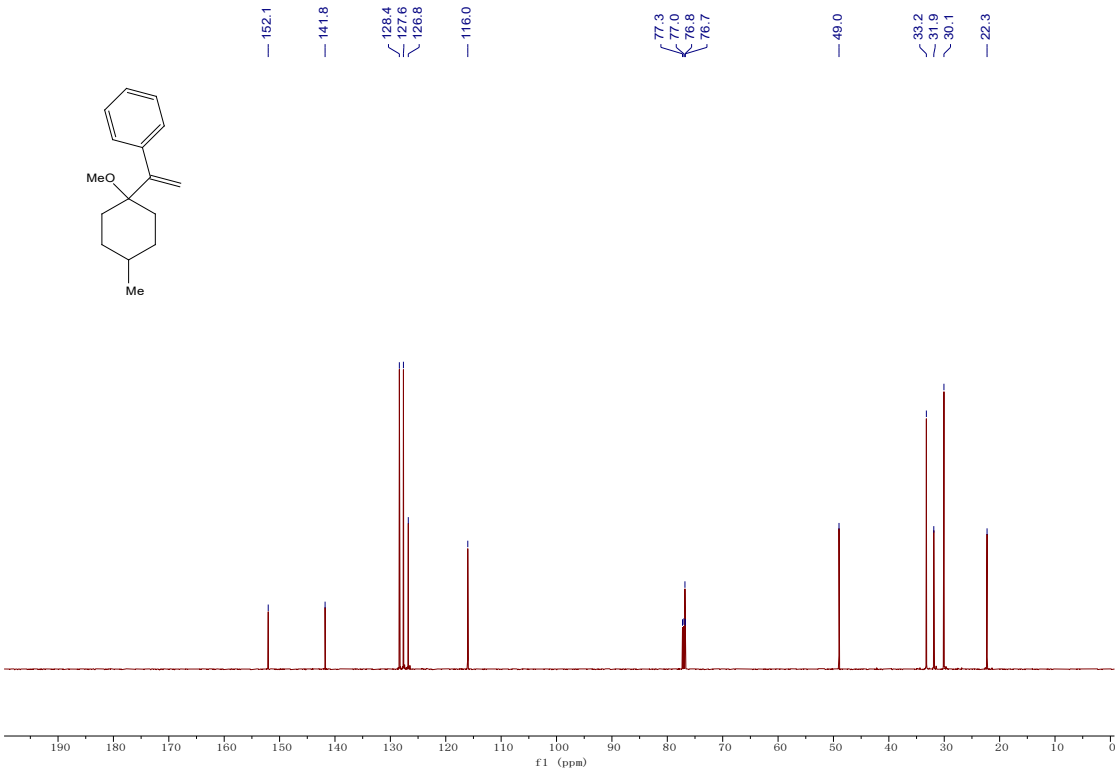
3.17
f1 (ppm)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22.3
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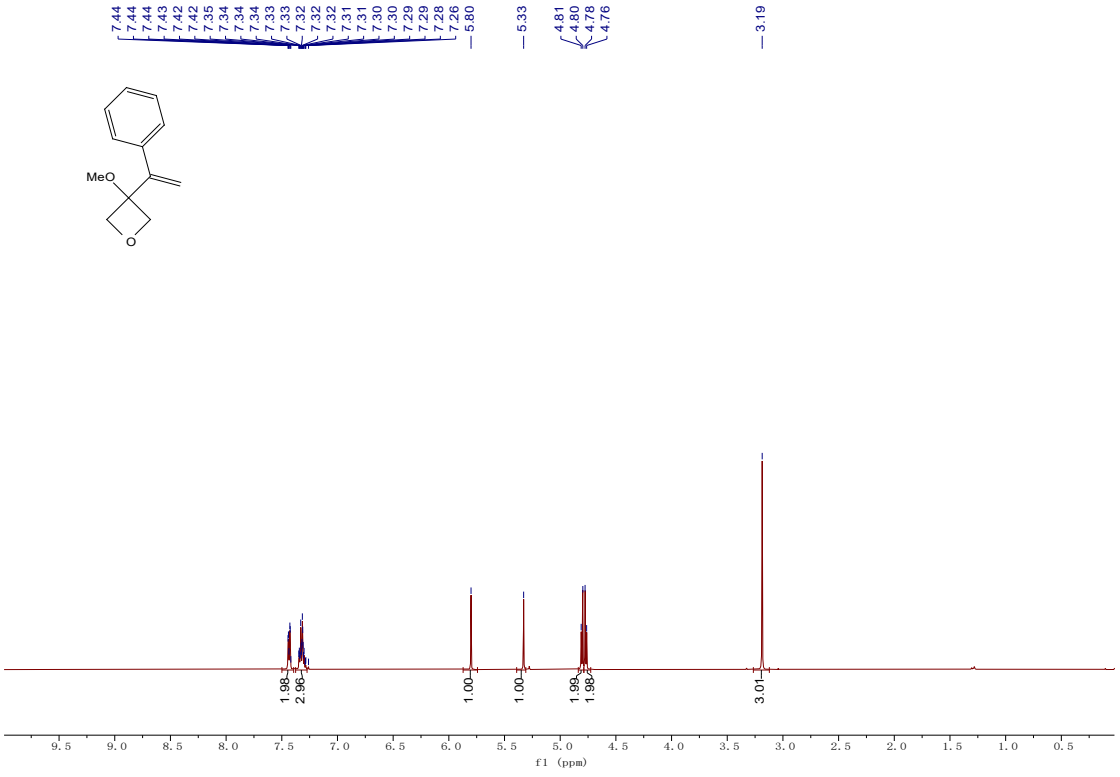
Me
f1 (ppm)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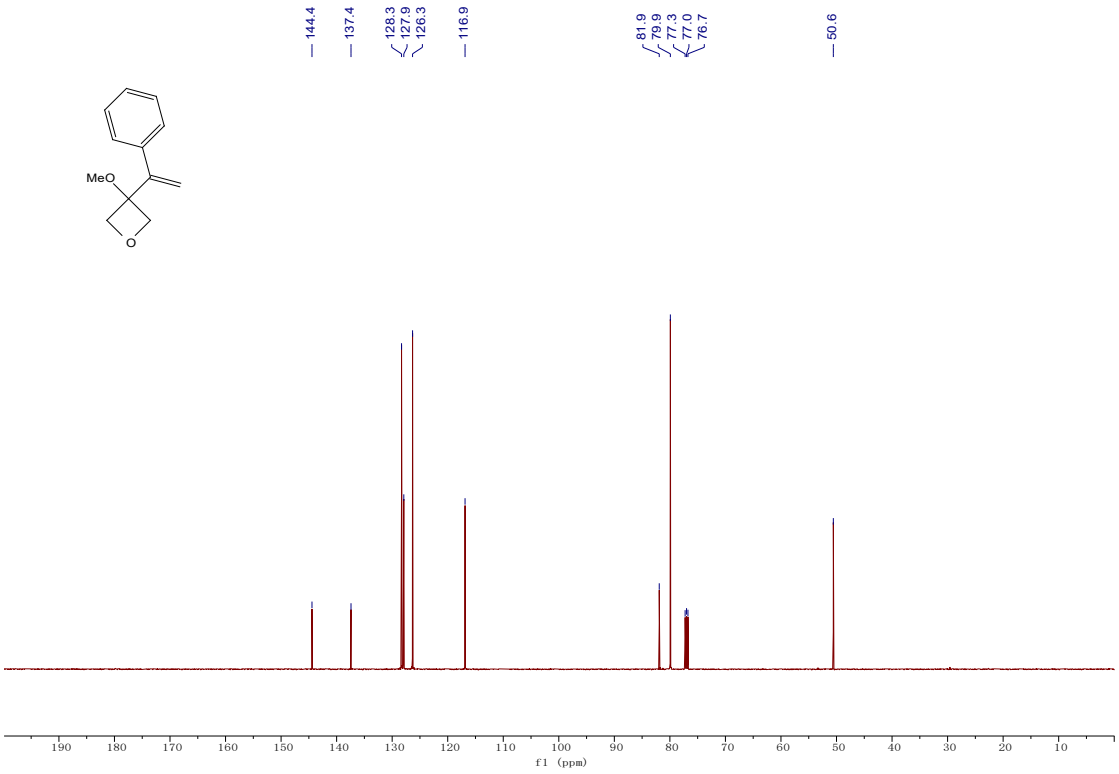
1.00
1.99
1.98
3.01
f1 (ppm)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76.7
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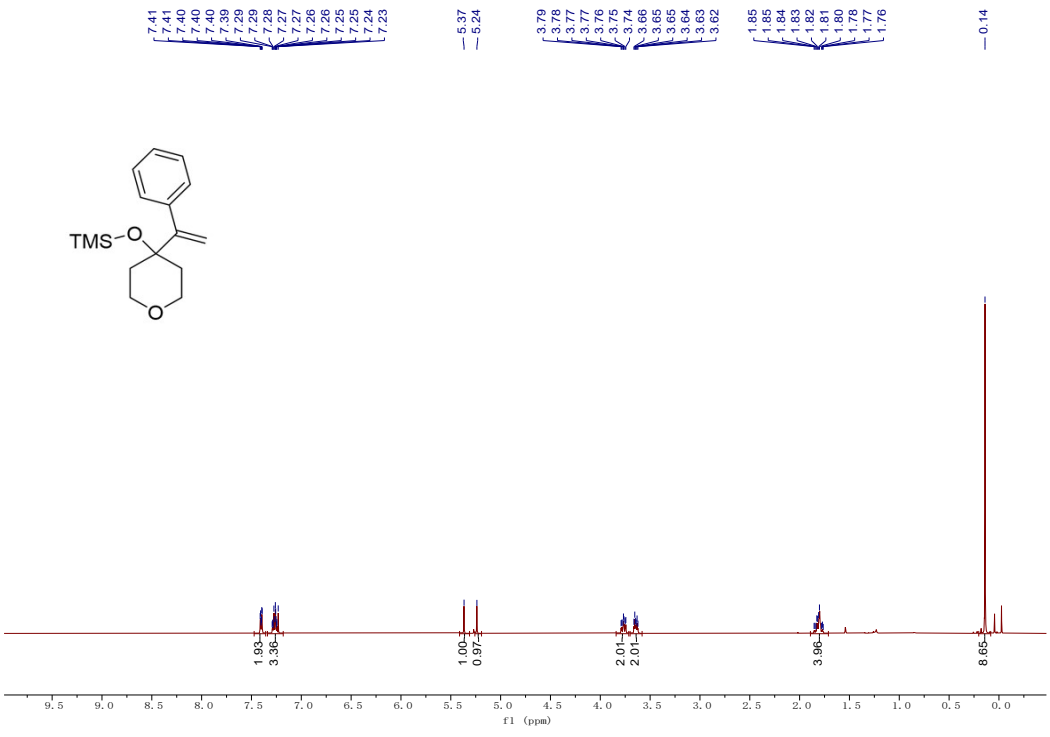
MeO
O
f1 (ppm)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3.78
3.77
3.76
3.75
3.74
3.66
3.65
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3.63
3.62
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1.83
1.82
1.81
1.80
1.78
1.77
1.76
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O
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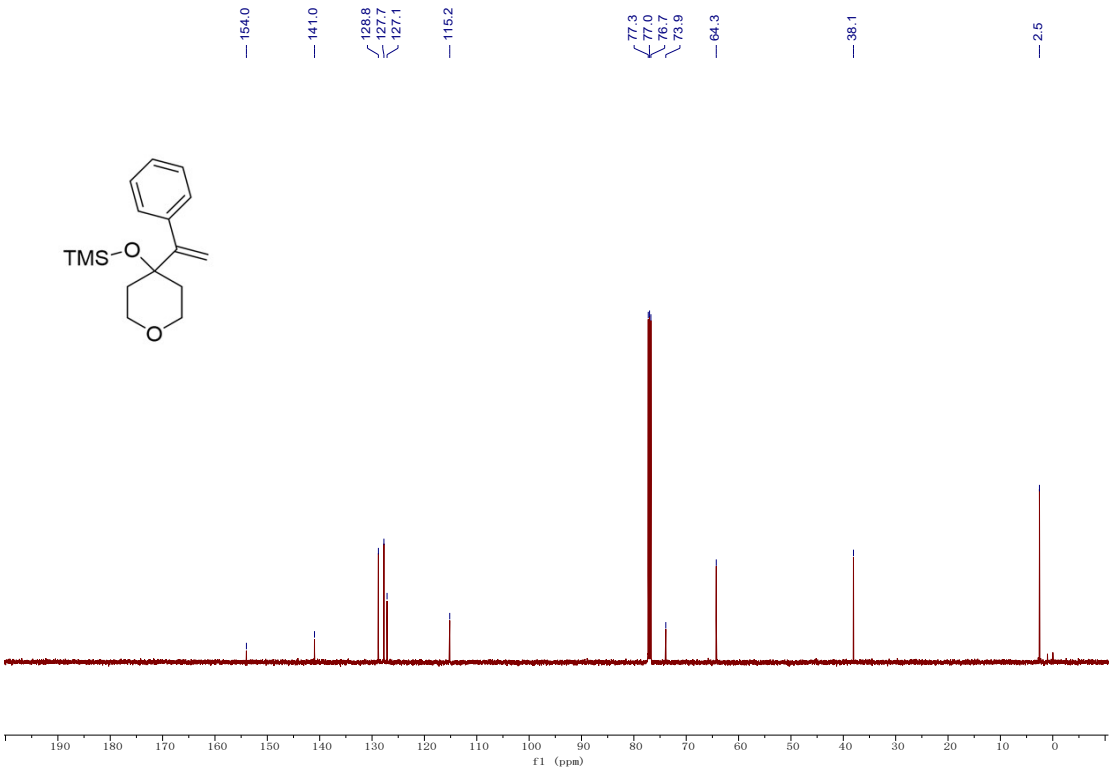
1.00
0.97
2.01
2.01
3.96
8.65
f1 (ppm)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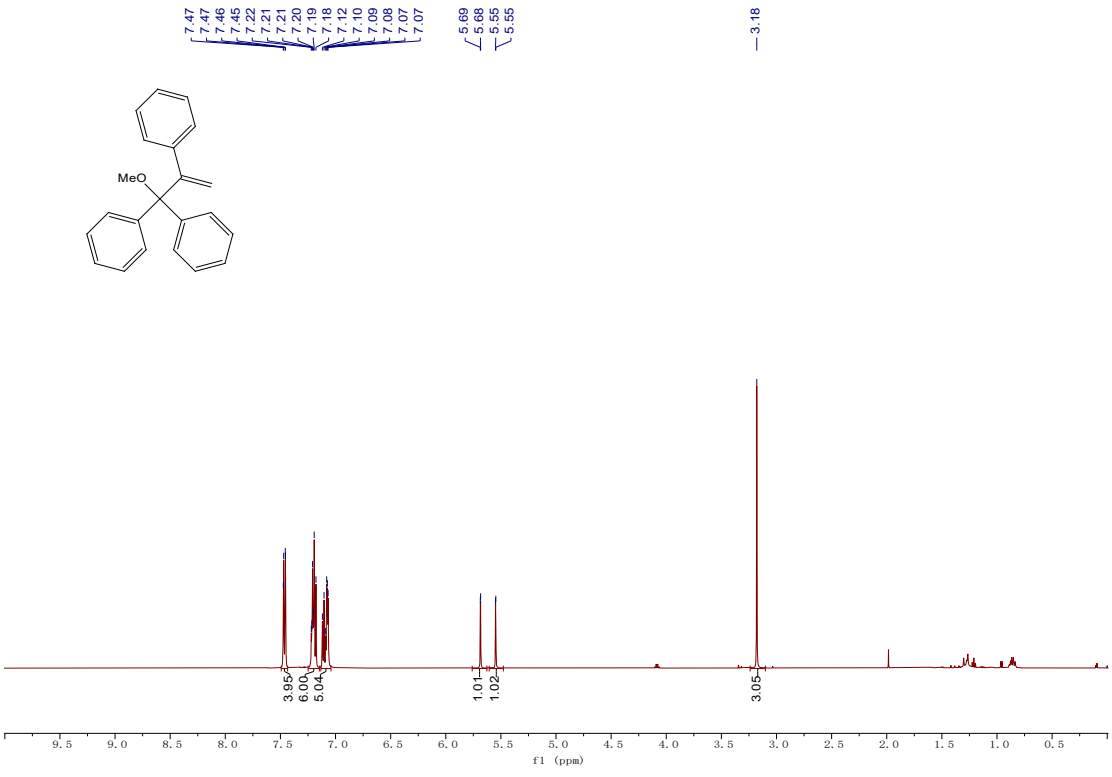
TMS
O
O
f1 (ppm)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7.07
7.07
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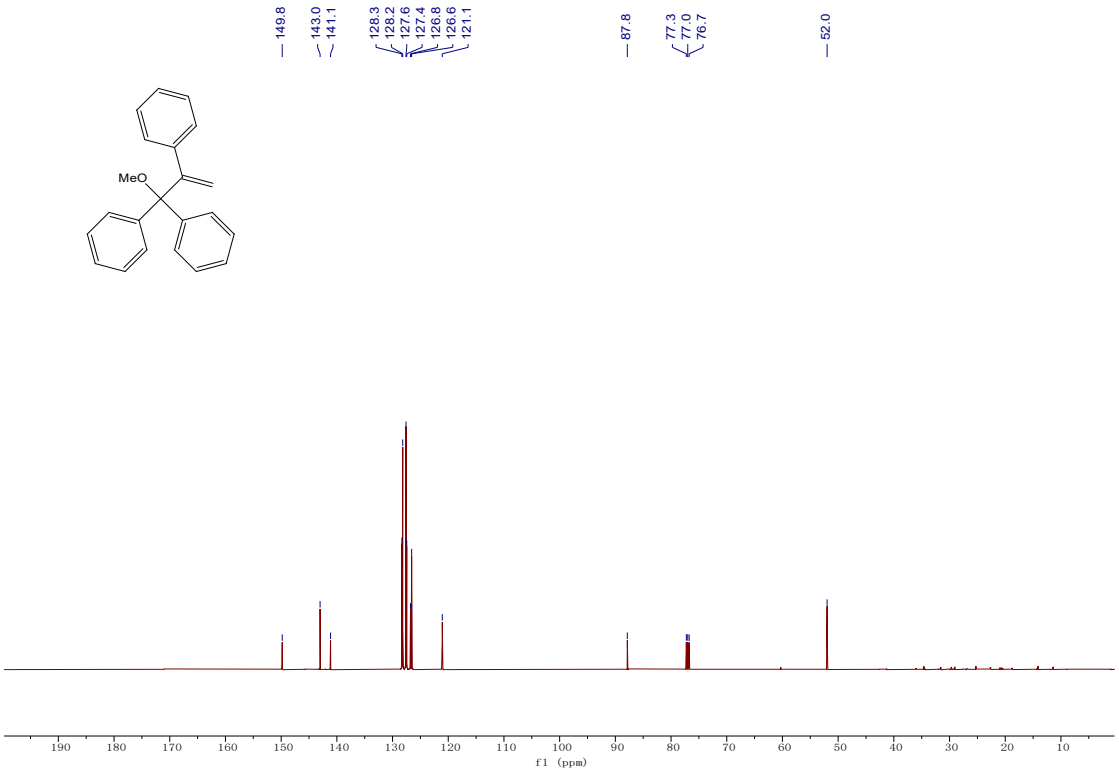
6.00
5.04
1.01
1.02
3.05
f1 (ppm)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77.3
77.0
76.7
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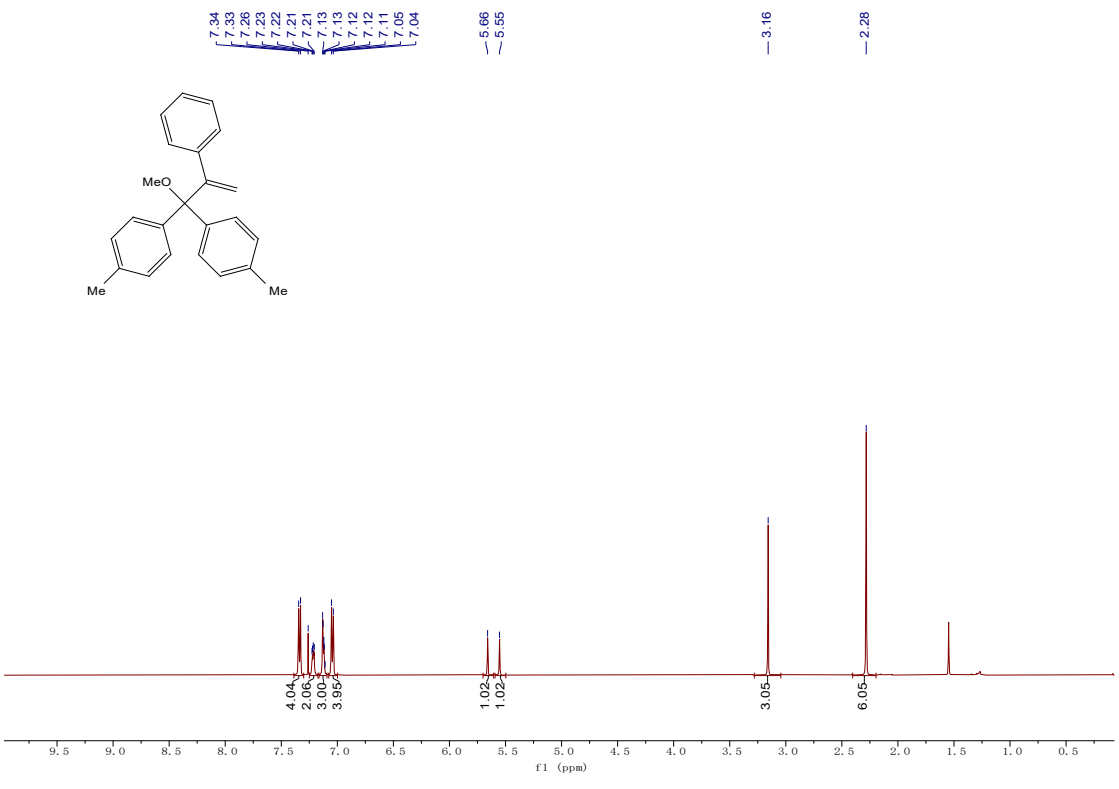
MeO
f1 (ppm)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5.55
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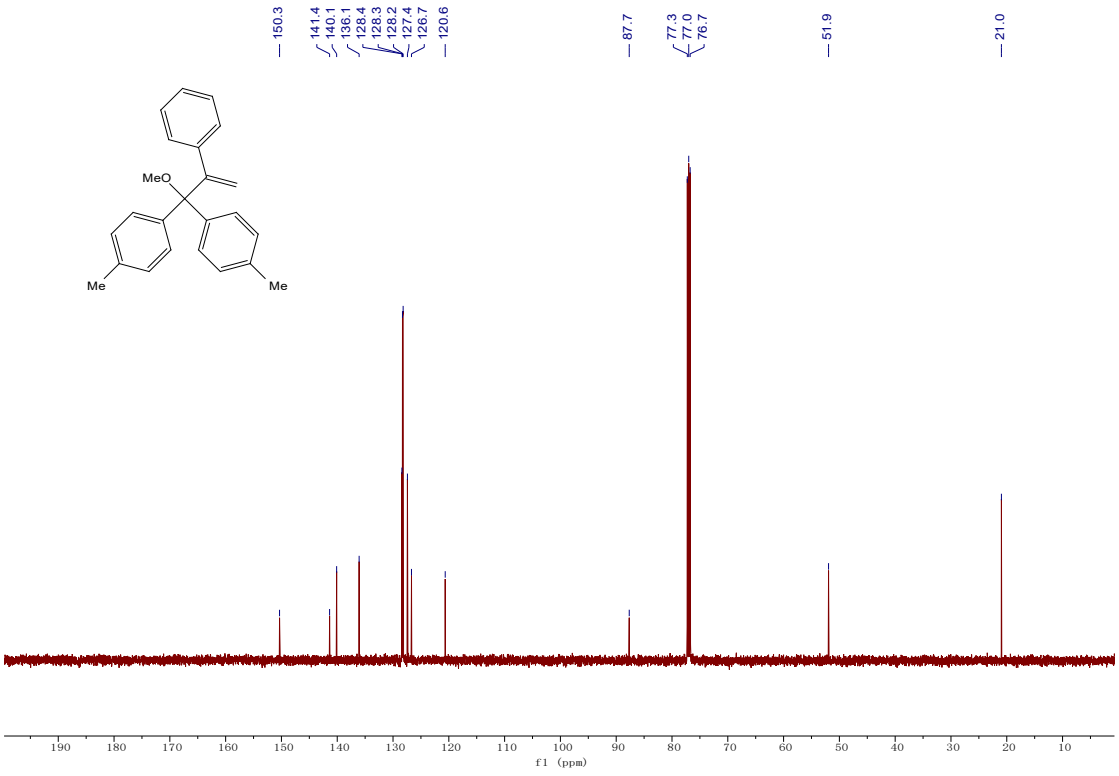
3.95
1.02
1.02
3.05
6.05
f1 (ppm)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21.0
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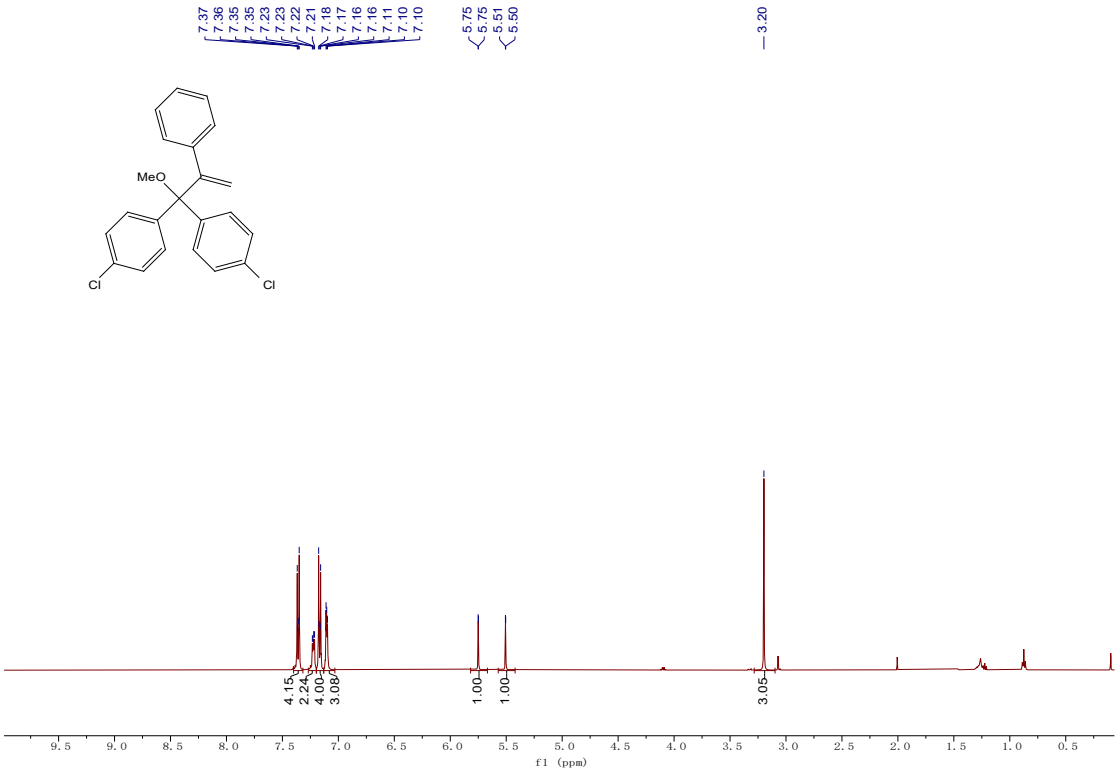
Me
Me
f1 (ppm)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7.11
7.10
7.10
5.75
5.75
5.51
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Cl
Cl
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4.00
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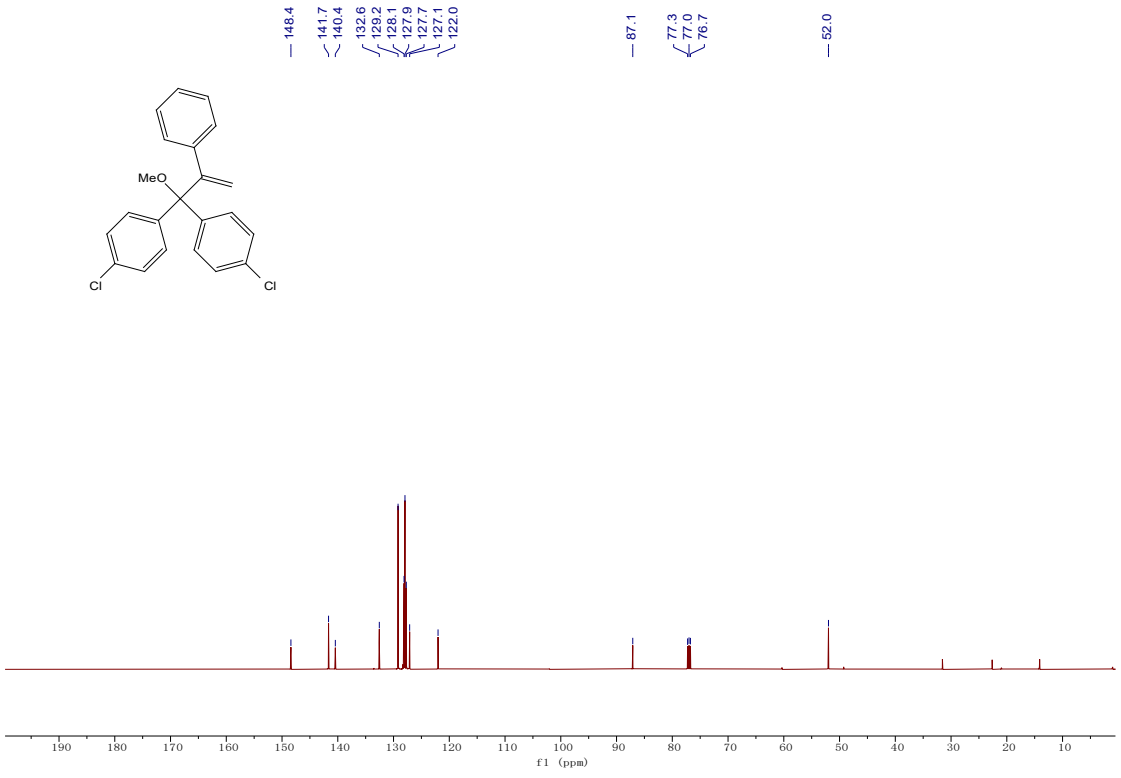
1.00
1.00
3.05
f1 (ppm)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77.0
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52.0
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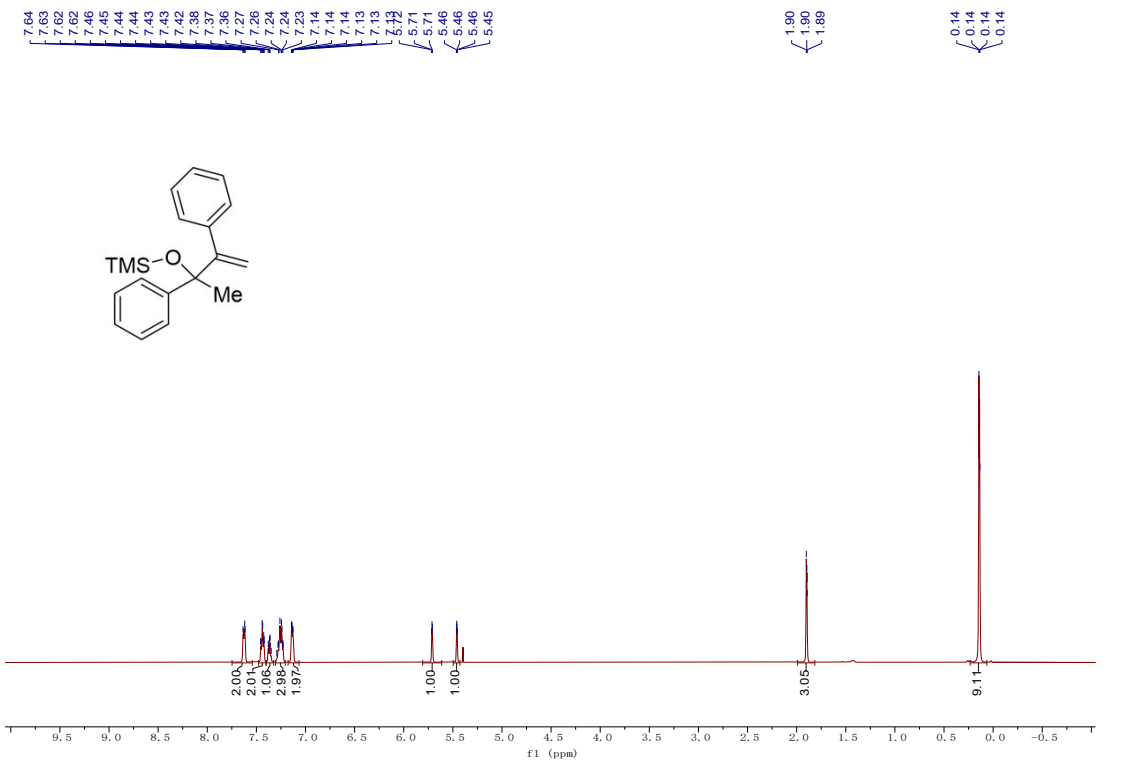
Cl
Cl
f1 (ppm)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1.90
1.89
0.14
0.14
0.14
0.14
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Me
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1.06
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1.00
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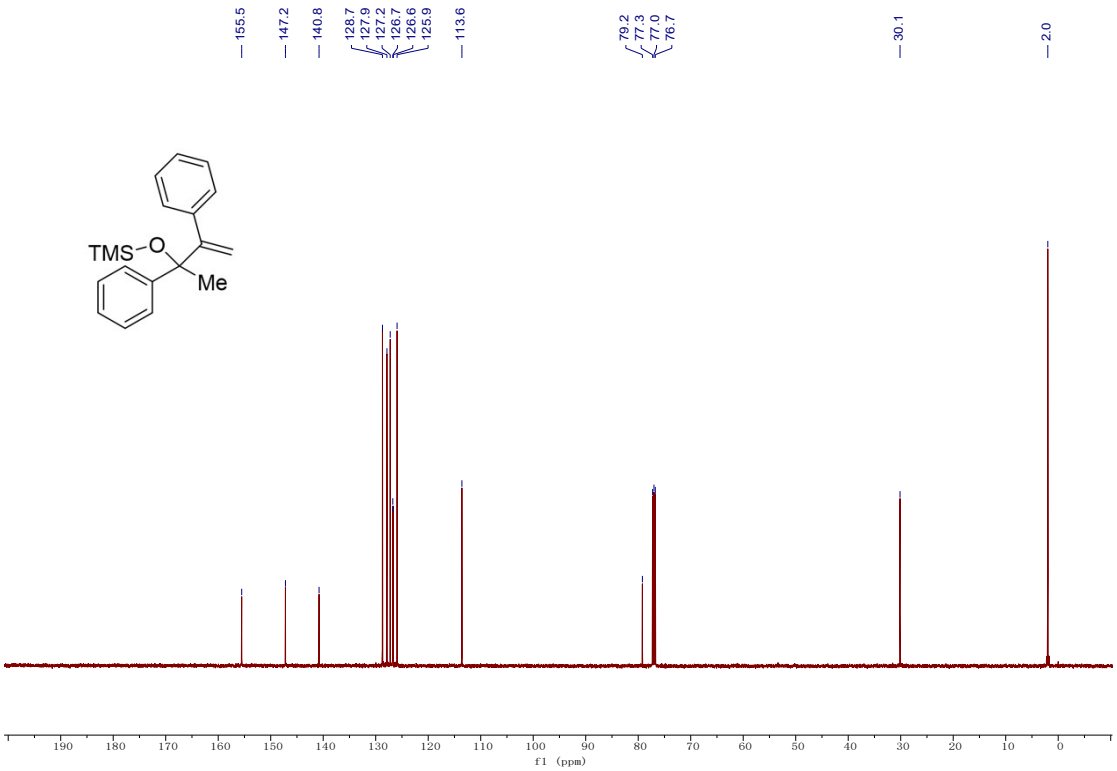
9.11
f1 (ppm)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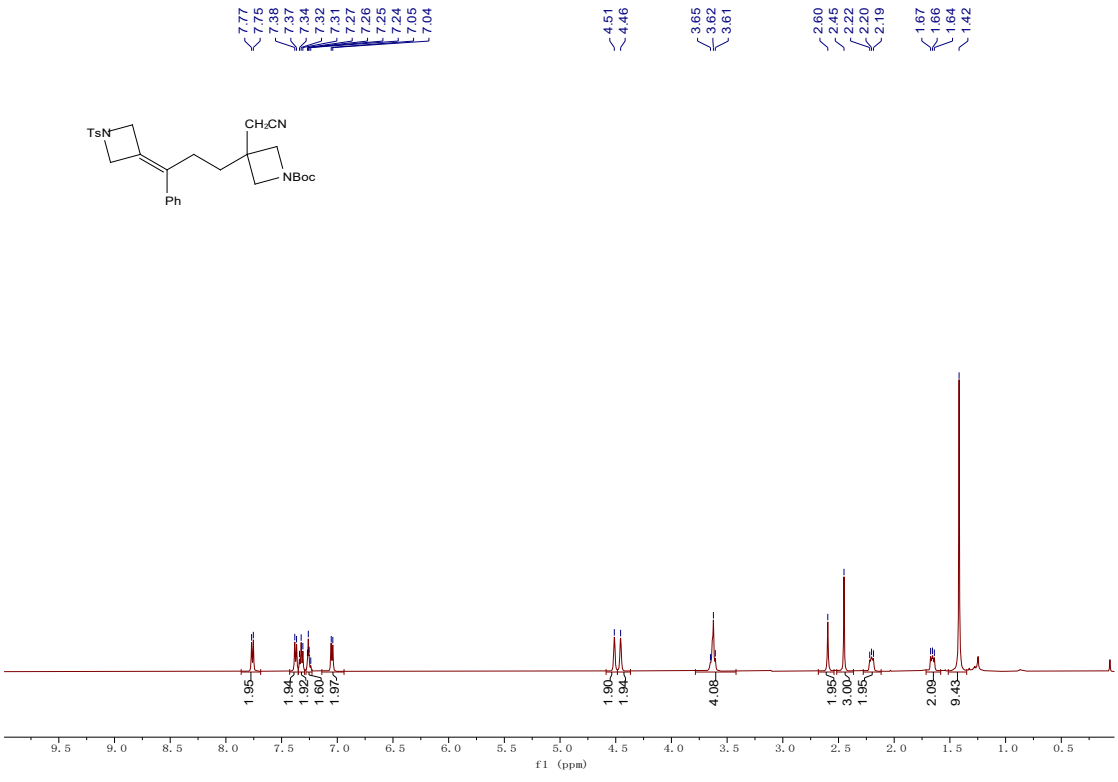
2.0
TMS
O
Me
f1 (ppm)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7.24
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7.04
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3.61
2.60
2.45
2.22
2.20
2.19
1.67
1.66
1.64
1.42
TsN
CH2CN
NBoc
Ph
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1.94
4.08
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9.43
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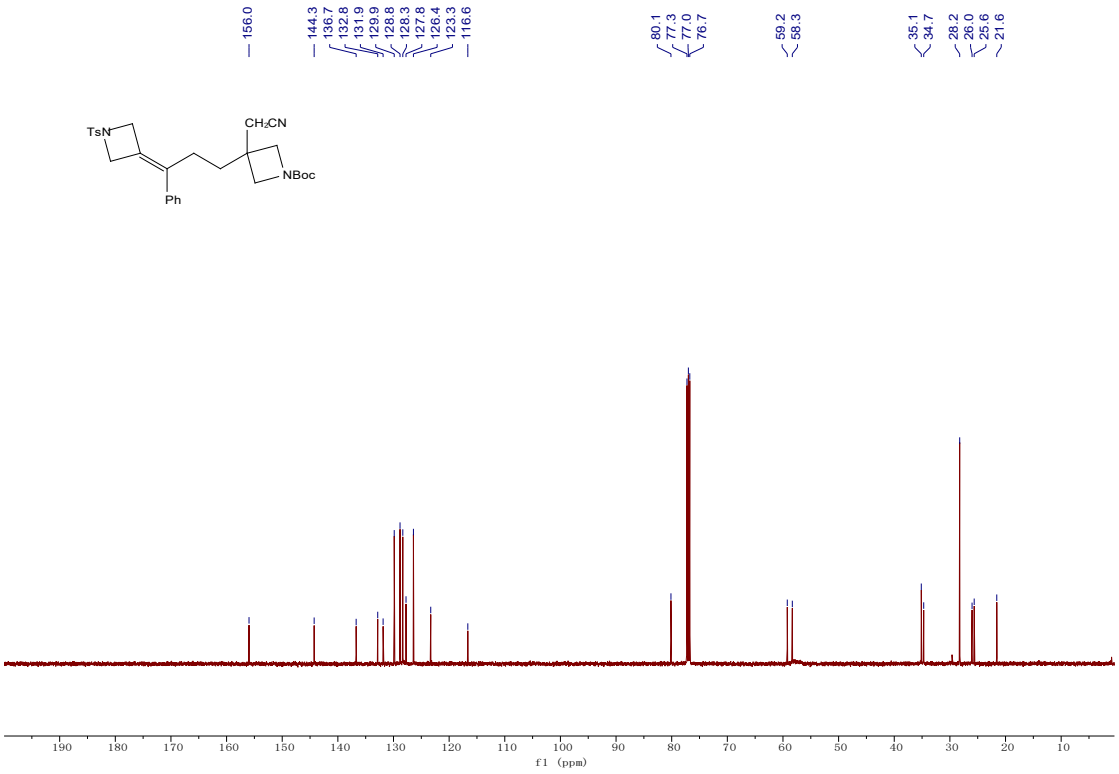
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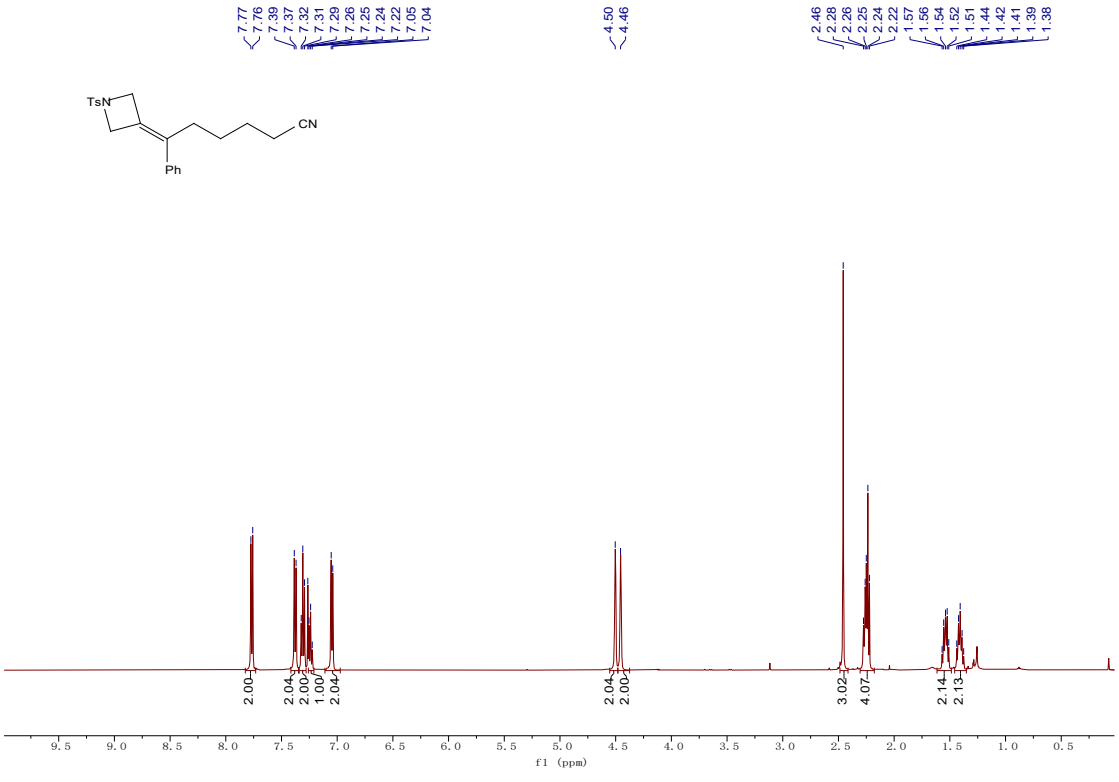
CH2CN
NBoc
Ph
f1 (ppm)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1.38
TsN
CN
Ph
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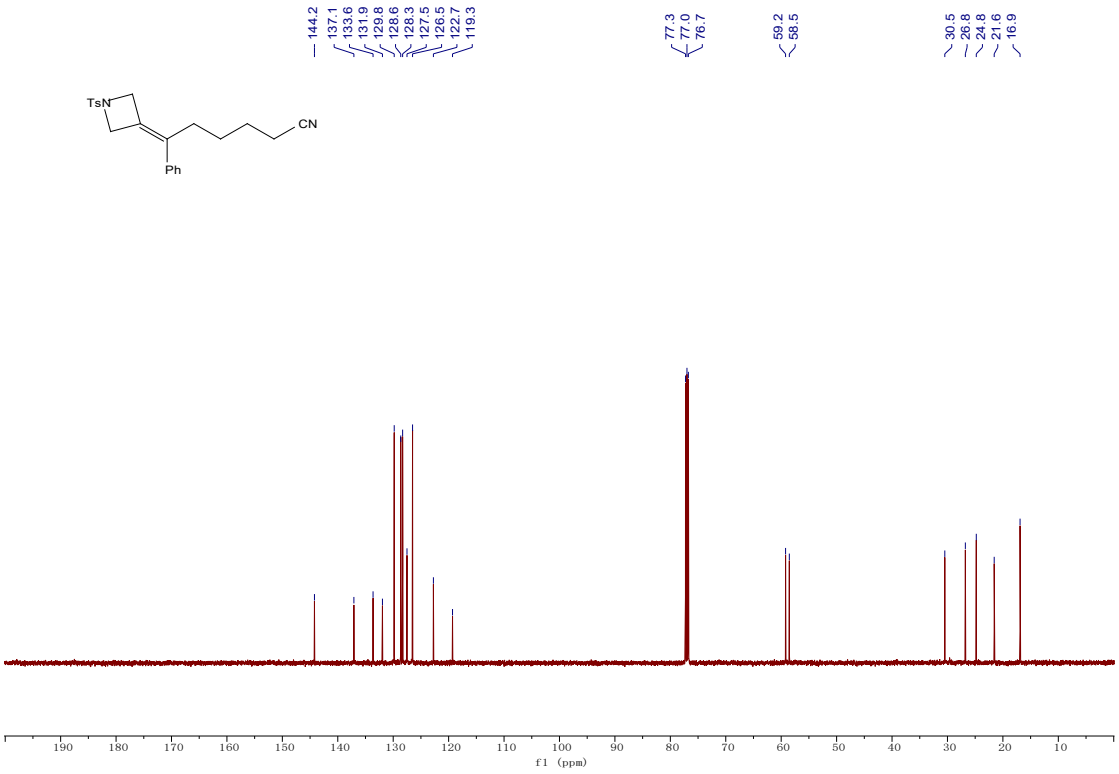
2.13
f1 (ppm)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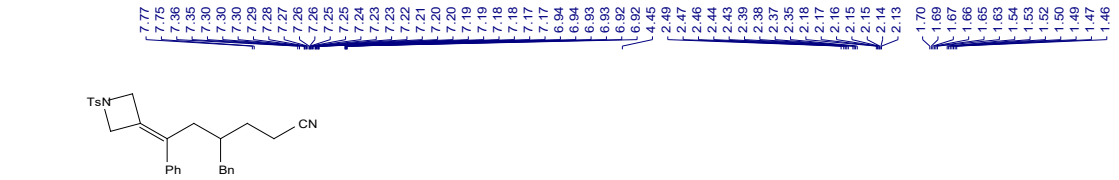
CN
Ph
f1 (ppm)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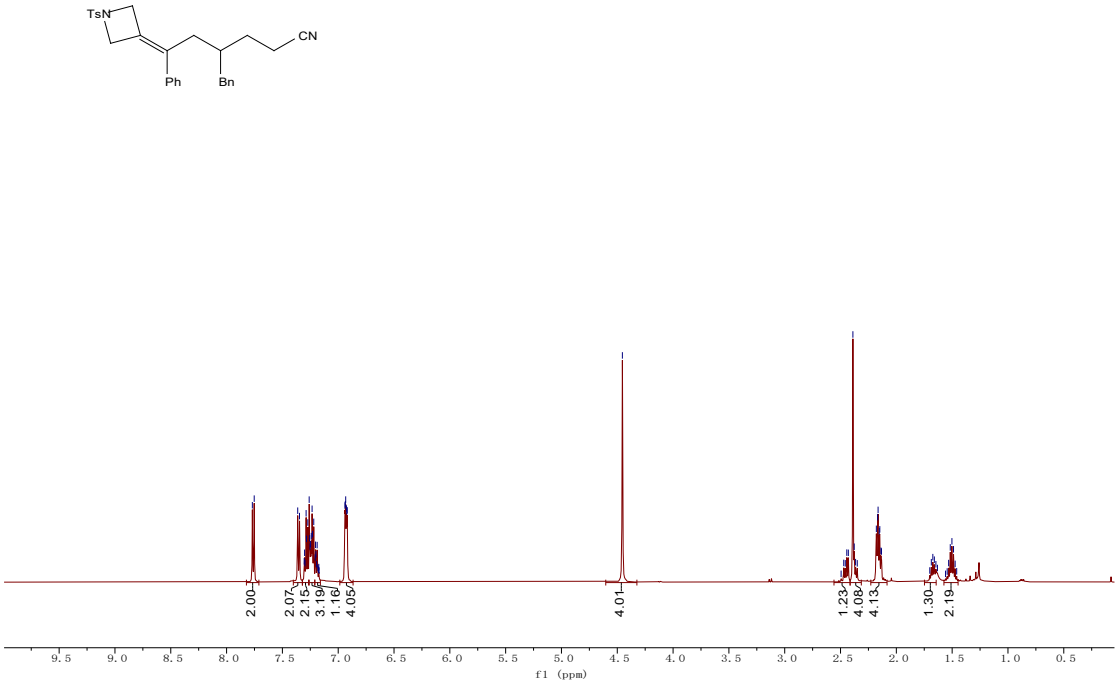
TsN
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4.13
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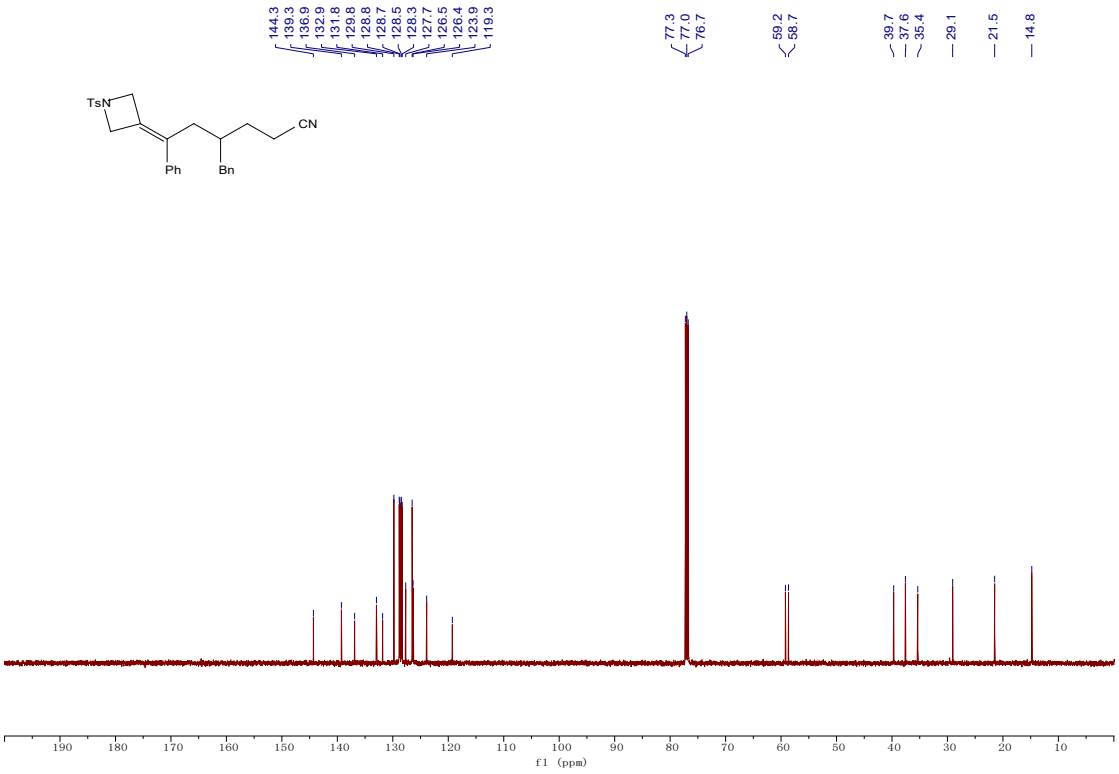
2.19
f1 (ppm)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14.8
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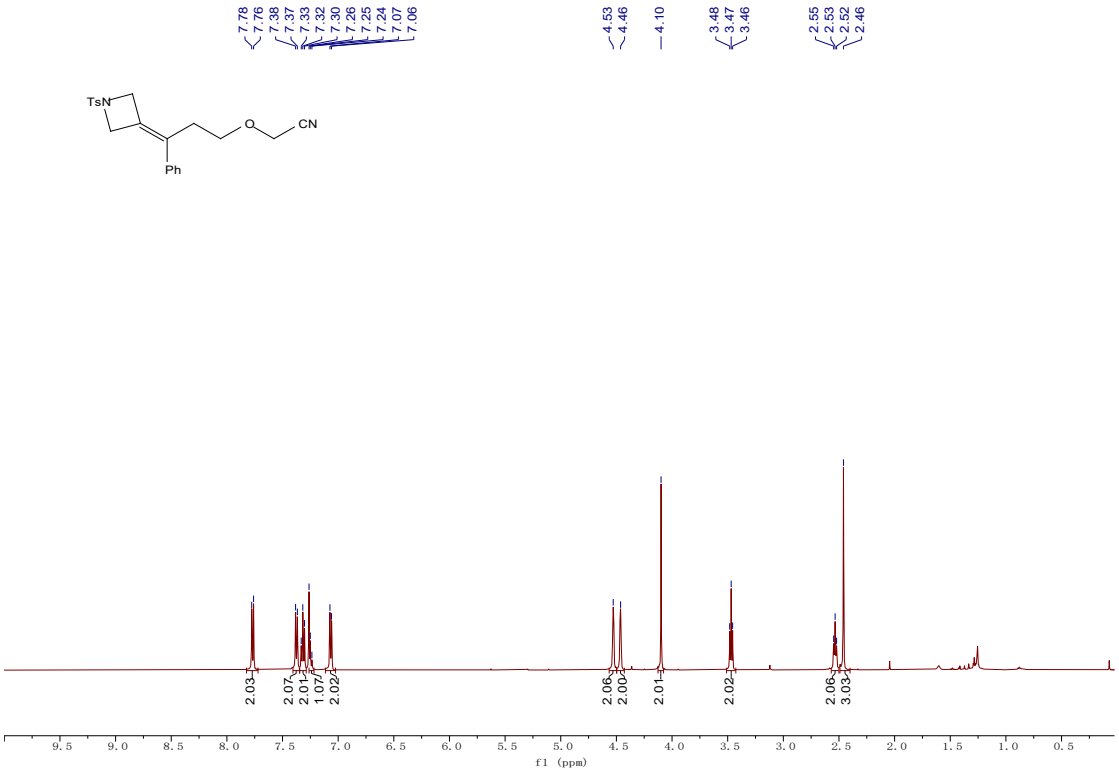
CN
Ph
Bn
f1 (ppm)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Ph
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2.06
3.03
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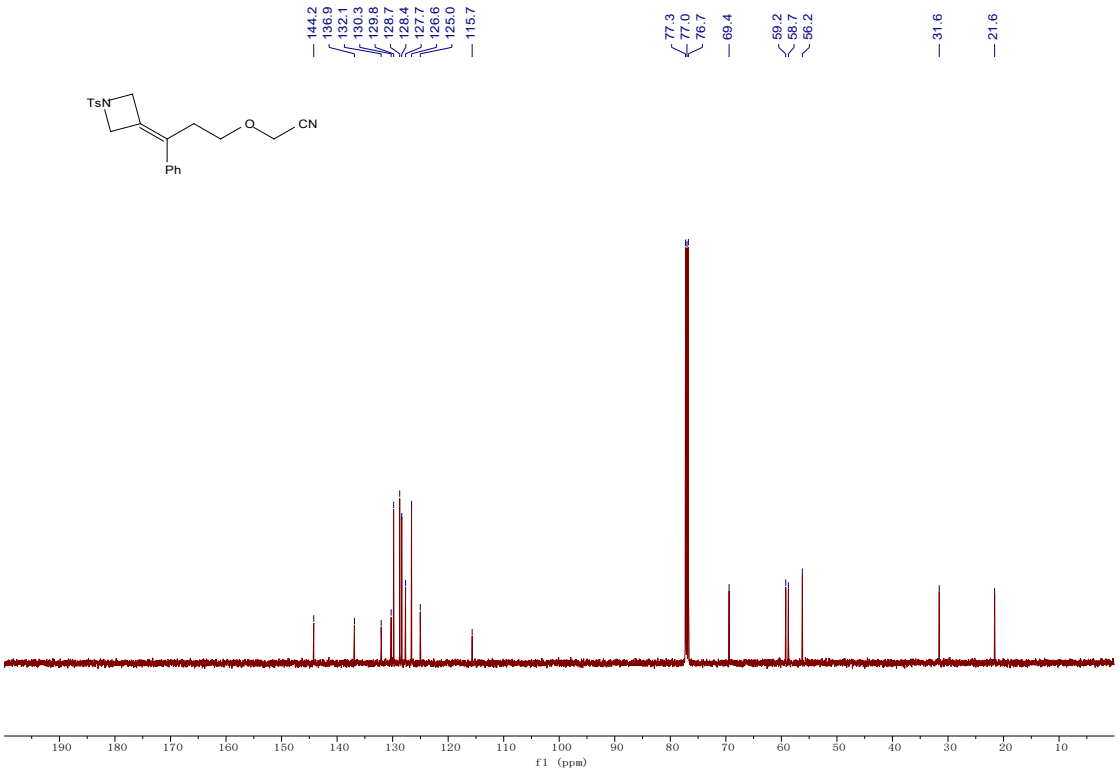
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56.2
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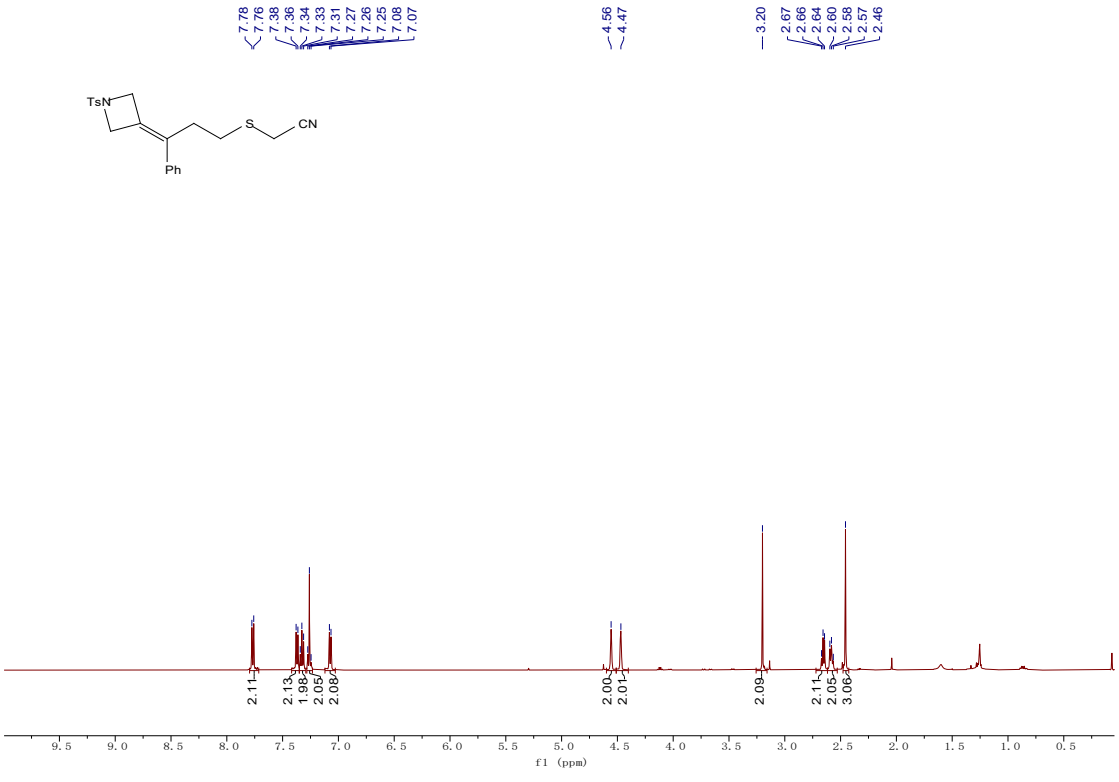
O
CN
Ph
f1 (ppm)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4.47
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TsN
S
CN
Ph
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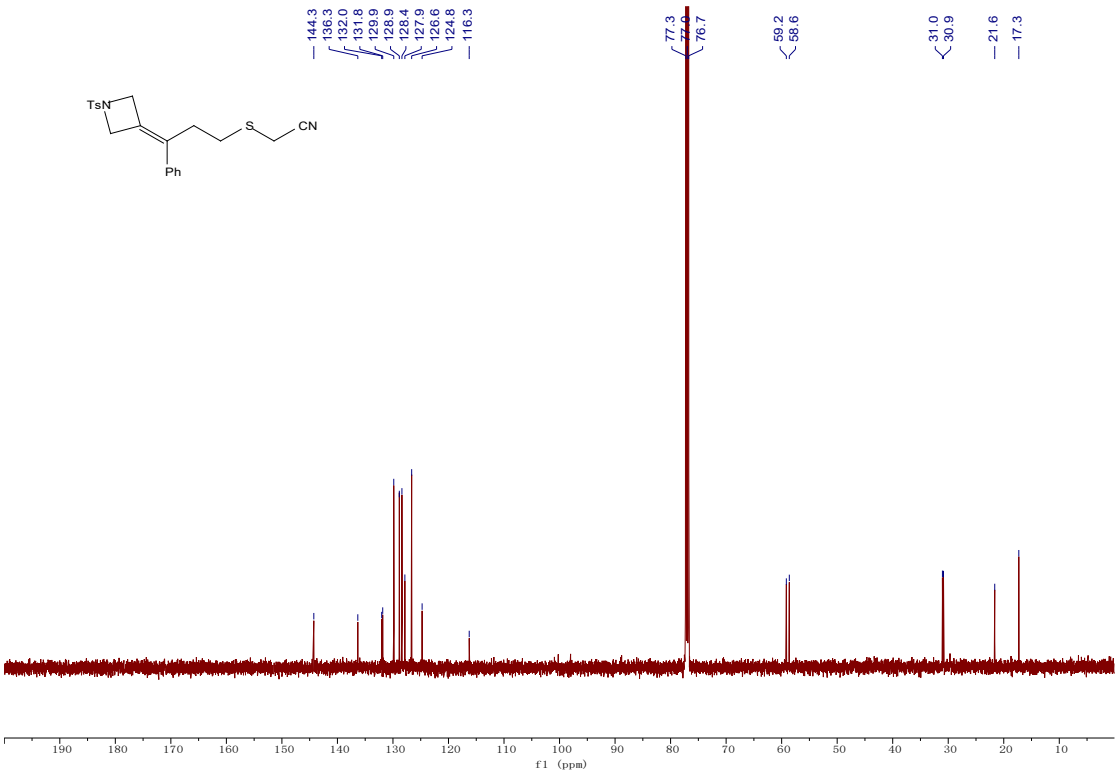
2.13
1.98
2.05
2.08
2.00
2.01
2.09
2.11
2.05
3.06
f1 (ppm)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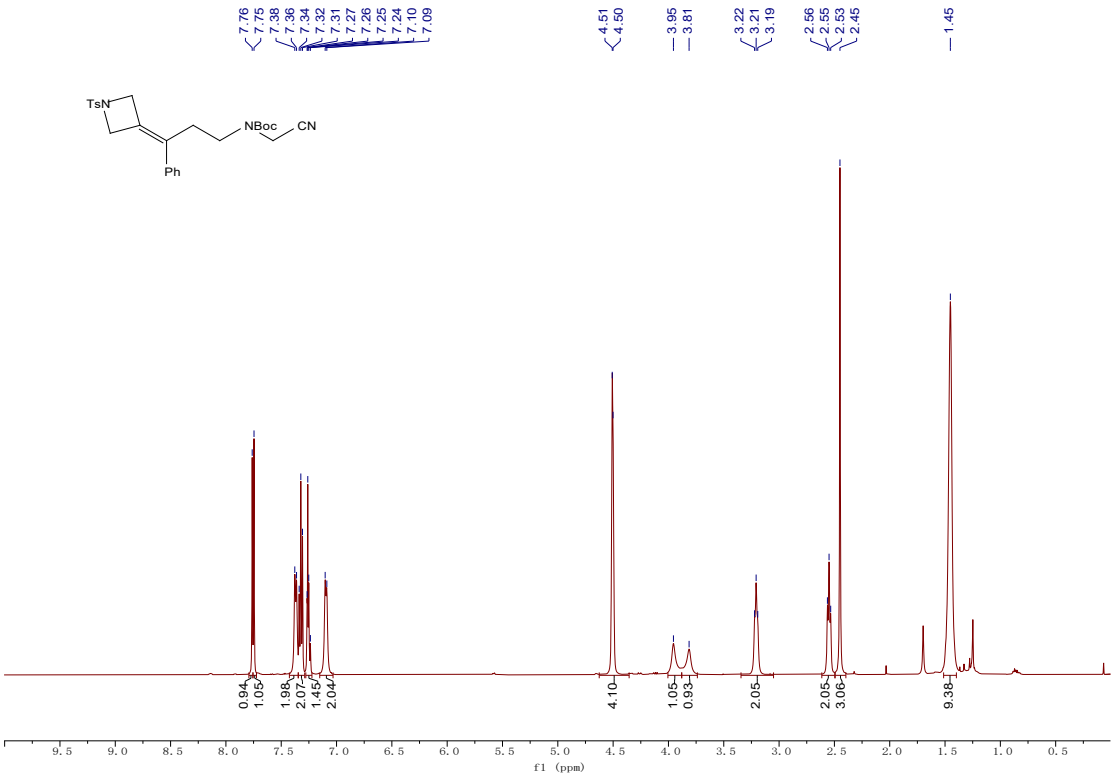
S
CN
Ph
f1 (ppm)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7.09
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4.50
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3.22
3.21
3.19
2.56
2.55
2.53
2.45
1.45
TsN
NBoc
CN
Ph
0.94
1.05
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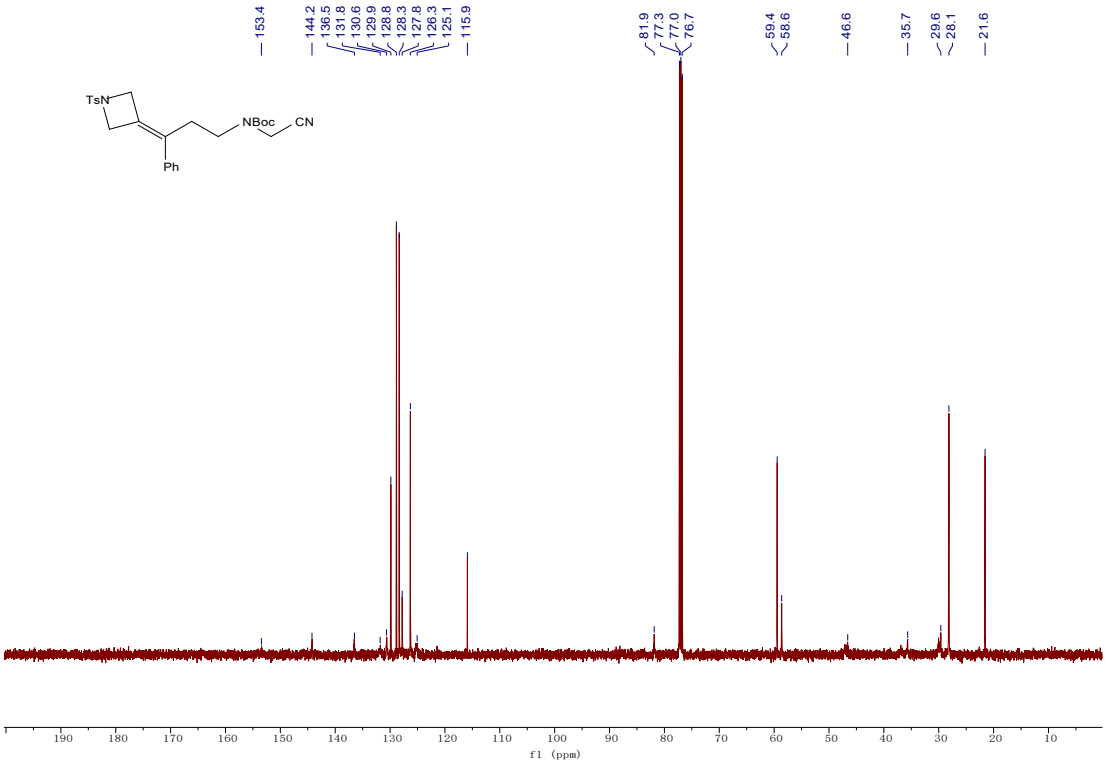
1.05
0.93
2.05
2.05
3.06
9.38
f1 (ppm)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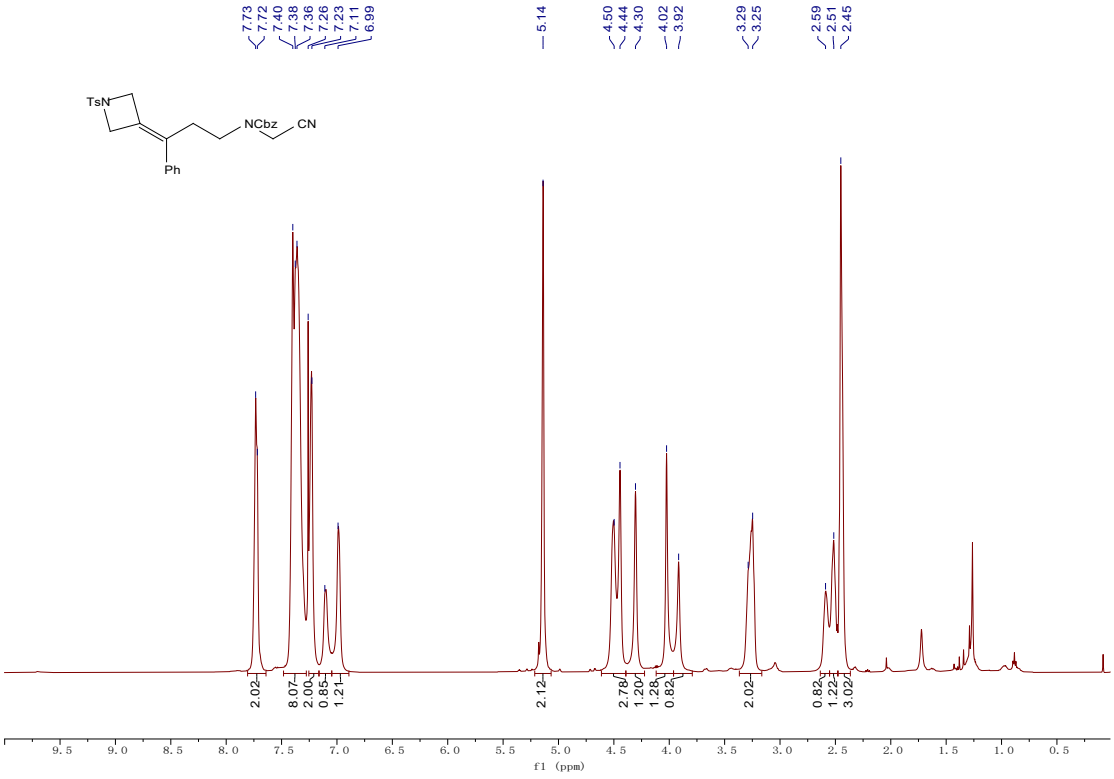
NBoc
CN
Ph
f1 (ppm)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3.25
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TsN
NCbz
CN
Ph
2.02
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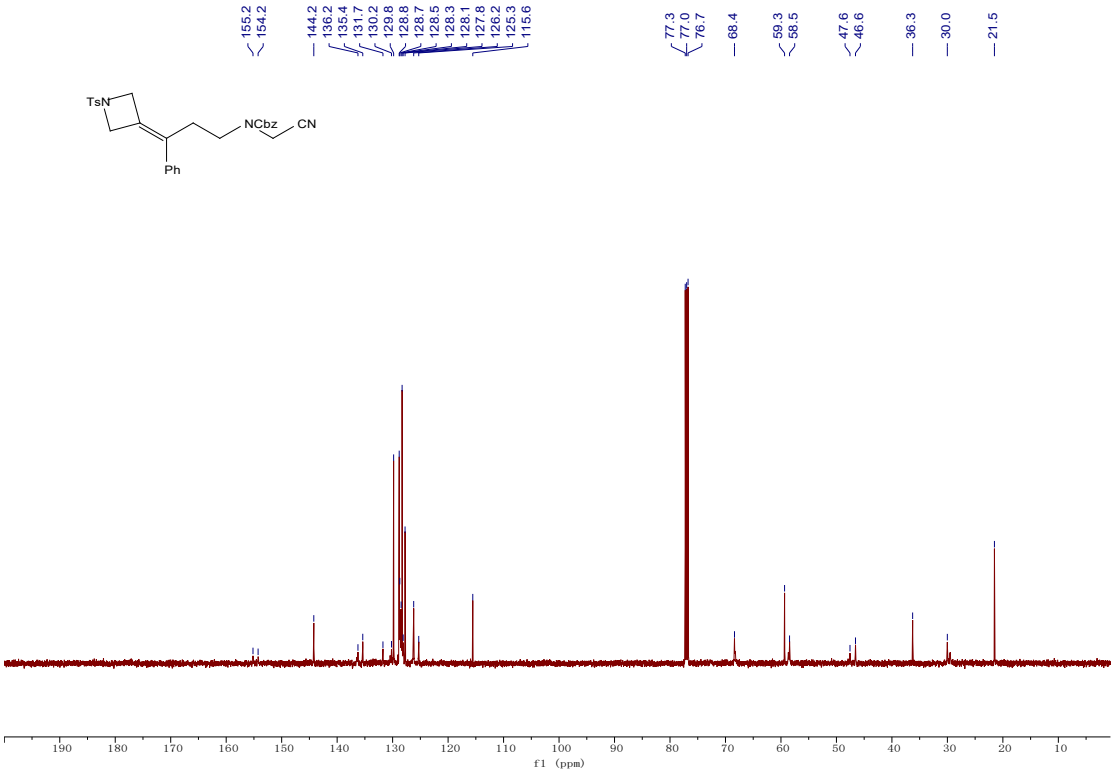
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126.2
125.3
115.6
77.3
77.0
76.7
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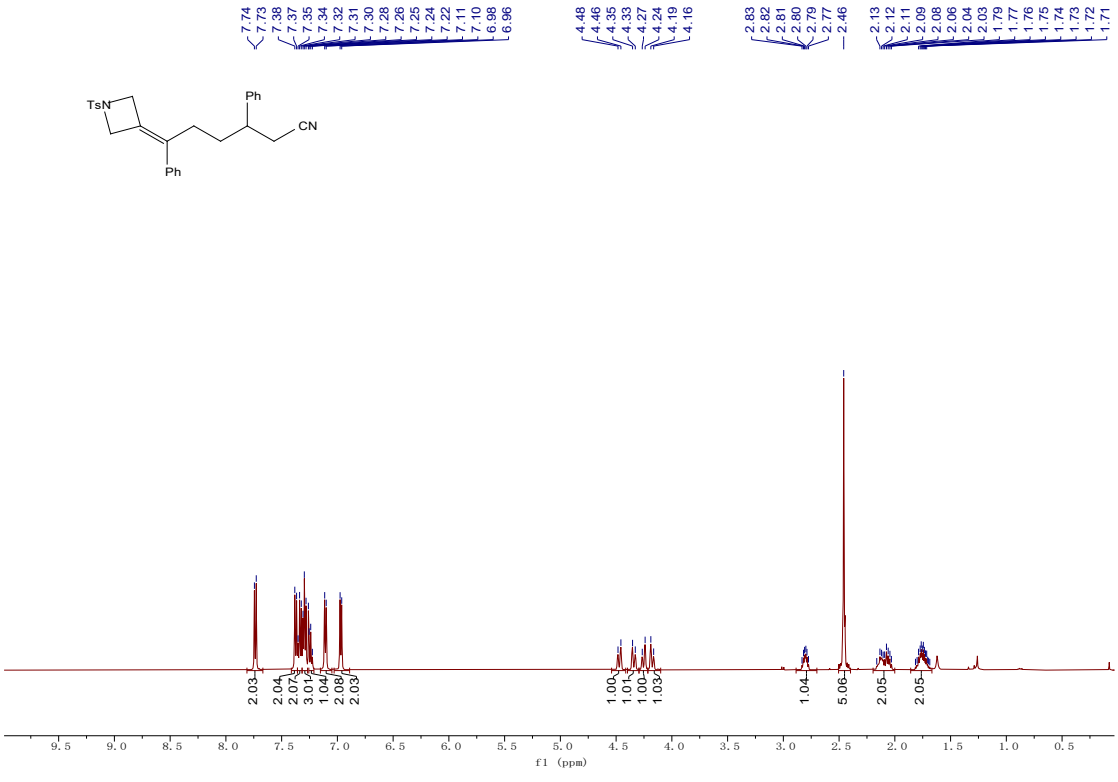
21.5
TsN
NCbz
CN
Ph
f1 (ppm)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TsN
Ph
CN
Ph
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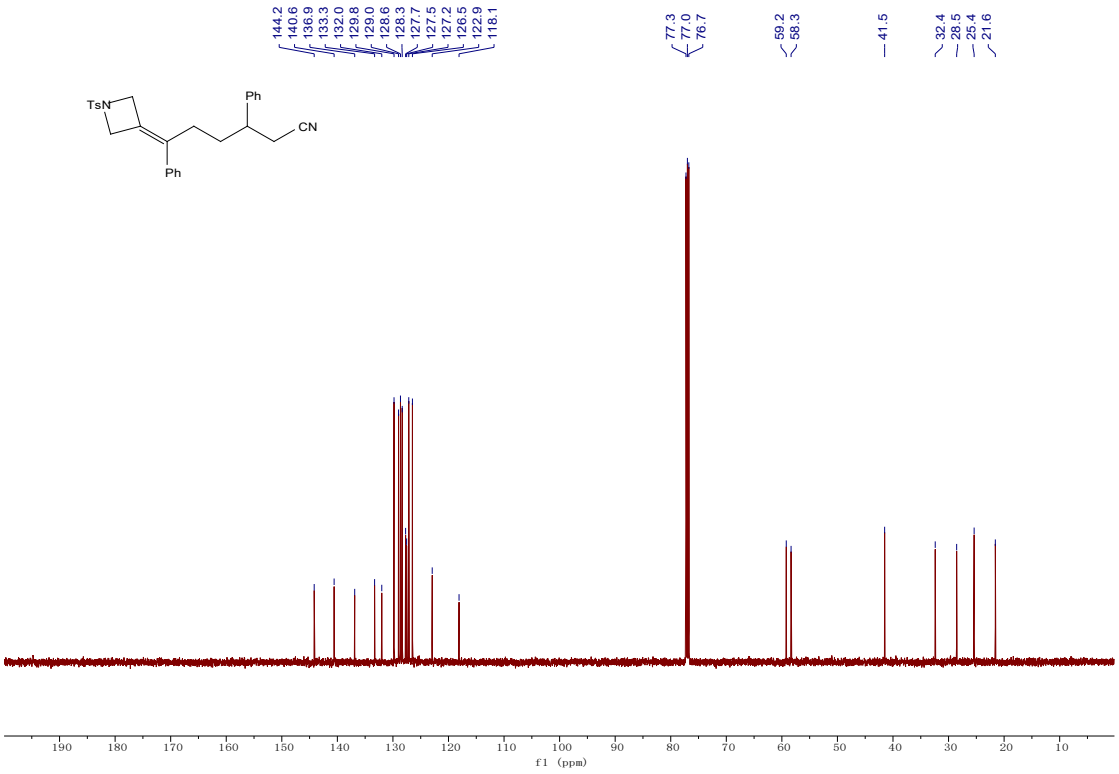
f1 (ppm)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77.0
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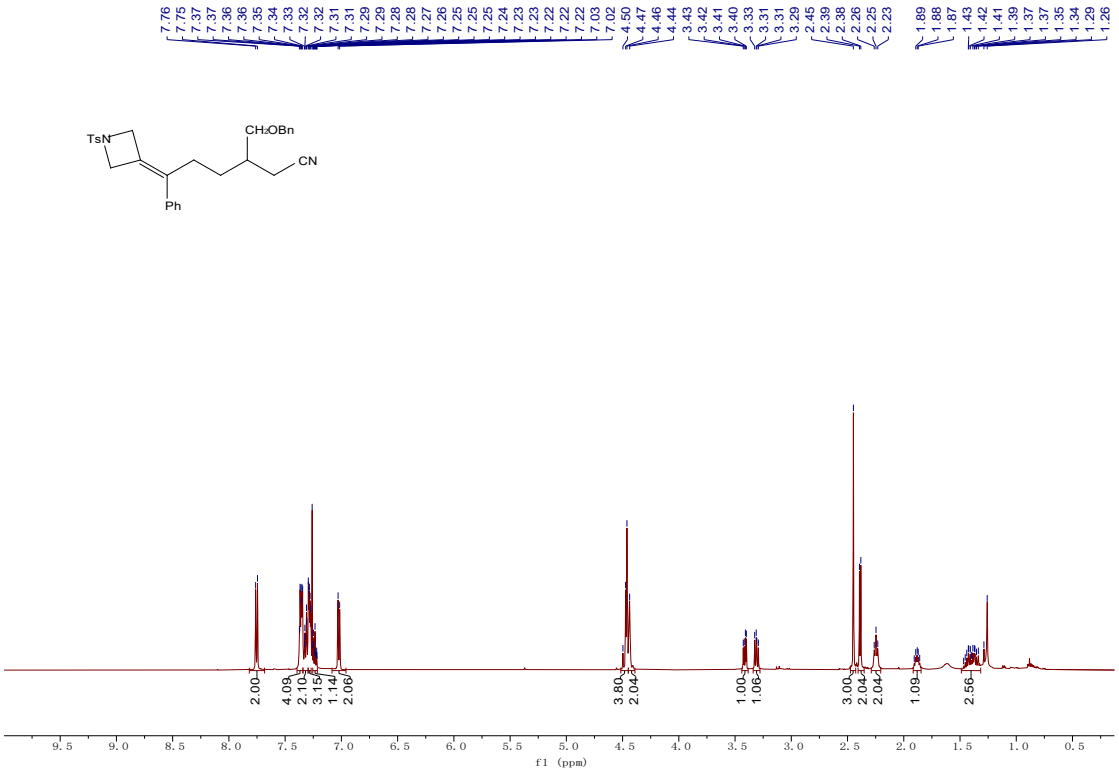
28.5
25.4
21.6
TsN
Ph
CN
Ph
f1 (ppm)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7.22
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4.46
4.44
3.43
3.42
3.41
3.40
3.33
3.31
3.31
3.29
2.45
2.39
2.38
2.26
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2.23
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1.41
1.39
1.37
1.37
1.35
1.34
1.29
1.26
TsN
CH2OBn
CN
Ph
2.00
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2.10
3.15
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1.06
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2.04
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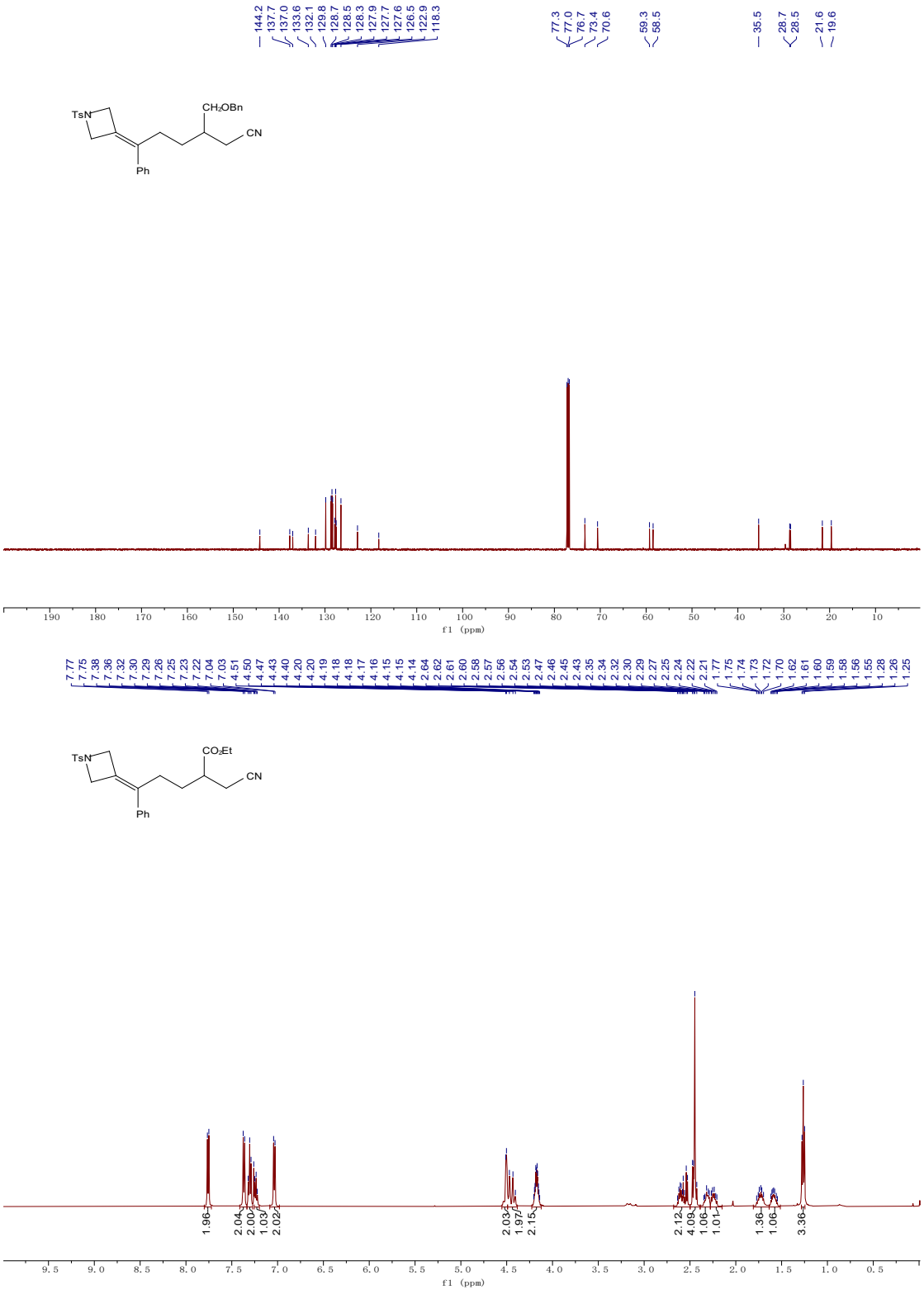
2.56
f1 (ppm)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122.9
118.3
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77.0
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73.4
70.6
59.3
58.5
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28.7
28.5
21.6
19.6
TsN
CH2OBn
CN
Ph
f1 (ppm)
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7.75
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7.29
7.26
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7.23
7.22
7.04
7.03
4.51
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4.20
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1.61
1.60
1.59
1.58
1.56
1.55
1.28
1.26
1.25
TsN
CO2Et
CN
Ph
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2.04
2.00
1.03
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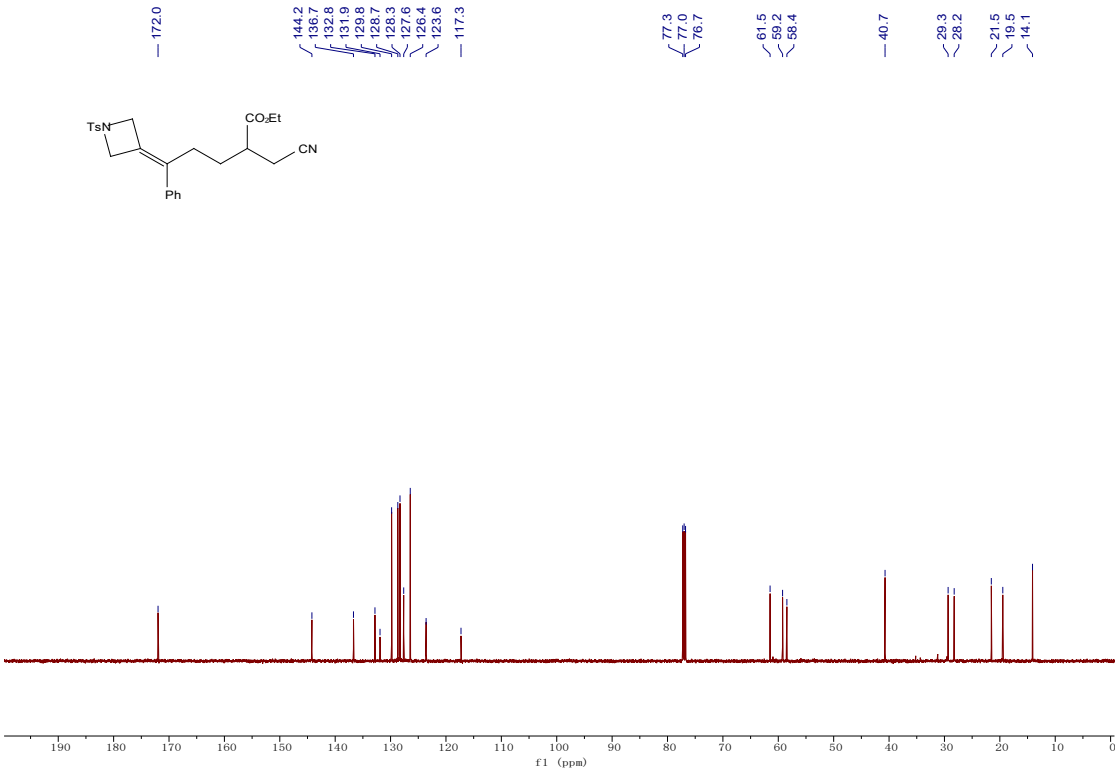
1.36
1.06
3.36
f1 (ppm)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123.6
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77.0
76.7
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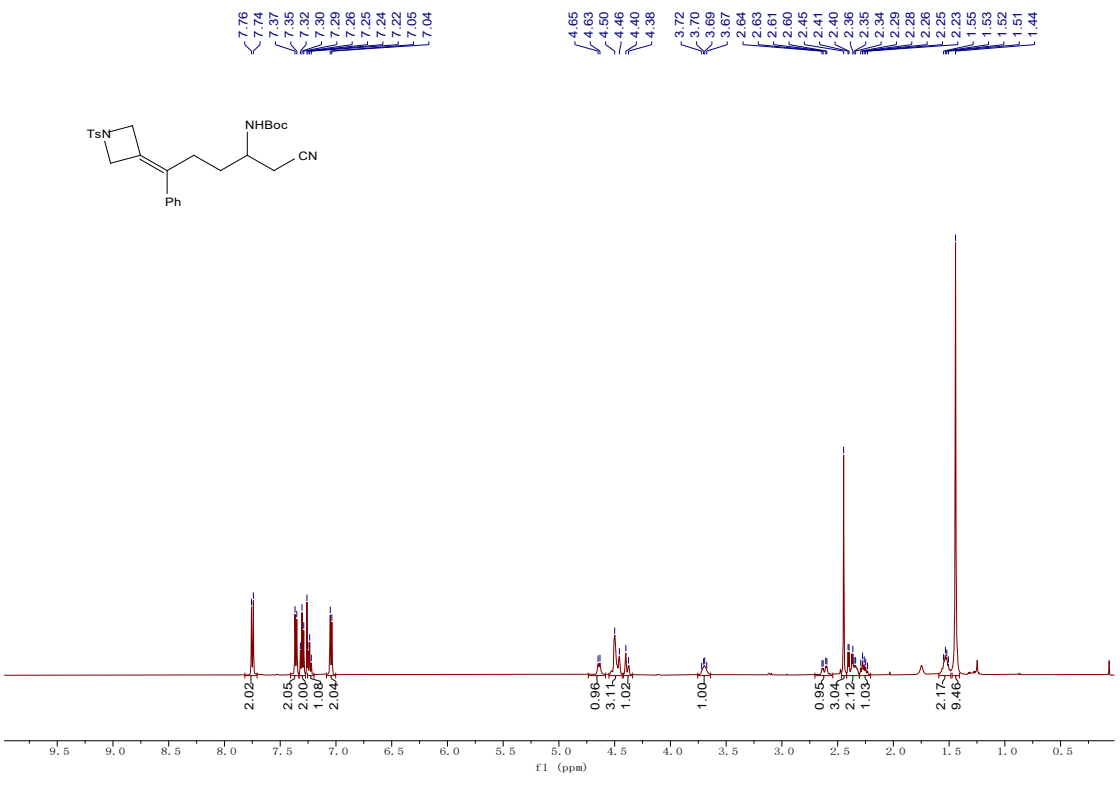
TsN
CO2Et
CN
Ph
f1 (ppm)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7.24
7.22
7.05
7.04
4.65
4.63
4.50
4.46
4.40
4.38
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3.69
3.67
2.64
2.63
2.61
2.60
2.45
2.41
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2.36
2.35
2.34
2.29
2.28
2.26
2.25
2.23
1.55
1.53
1.52
1.51
1.44
TsN
NHBoc
CN
Ph
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2.05
2.00
1.08
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1.02
1.00
0.95
3.04
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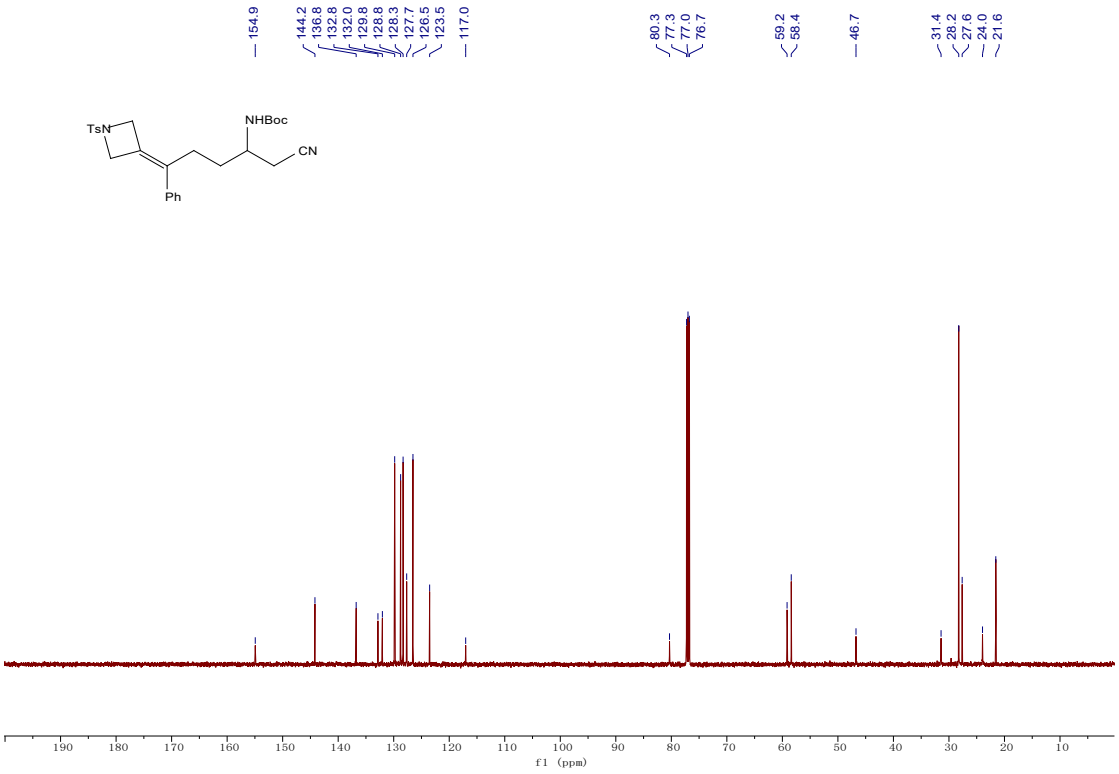
1.03
2.17
9.46
f1 (ppm)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77.0
76.7
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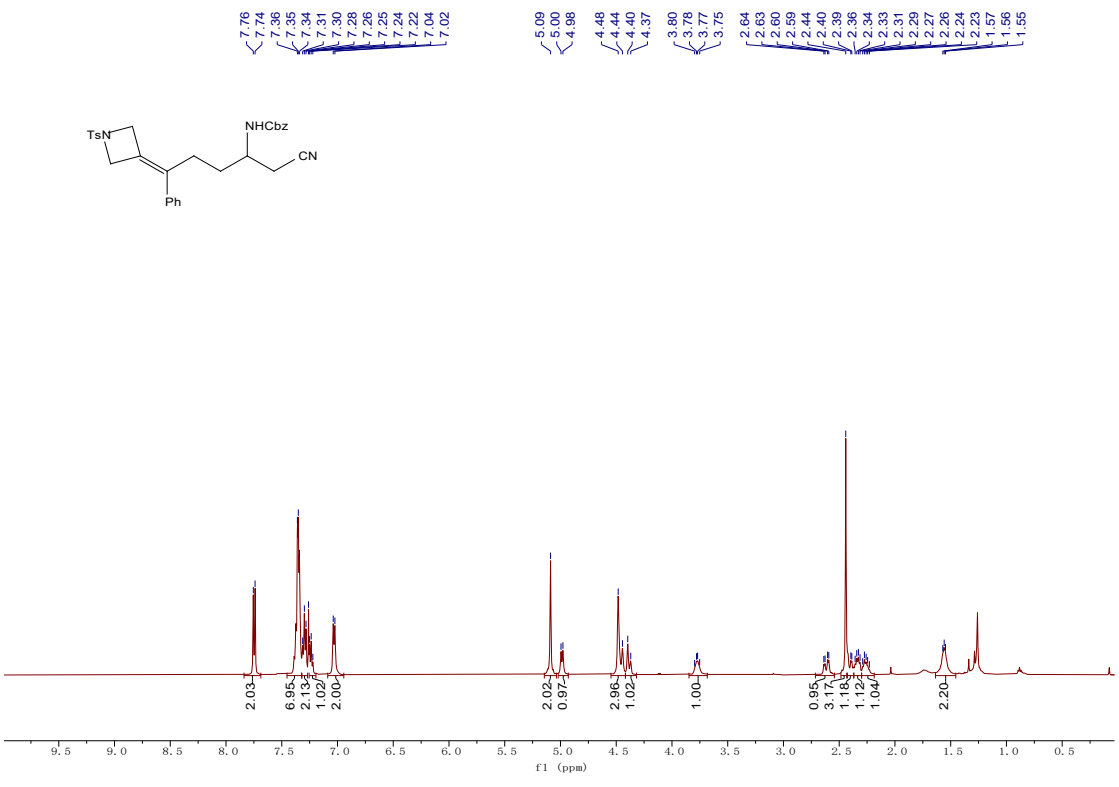
TsN
NHBoc
CN
Ph
f1 (ppm)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7.24
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7.02
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NHCbz
CN
Ph
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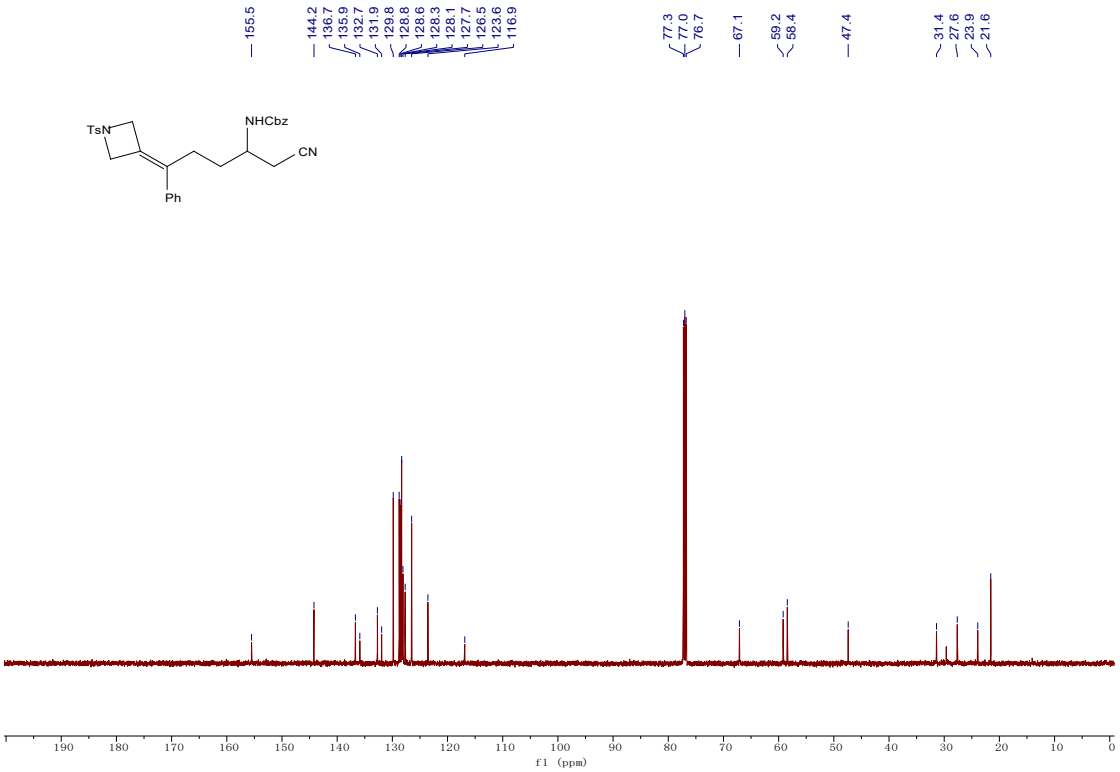
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116.9
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77.0
76.7
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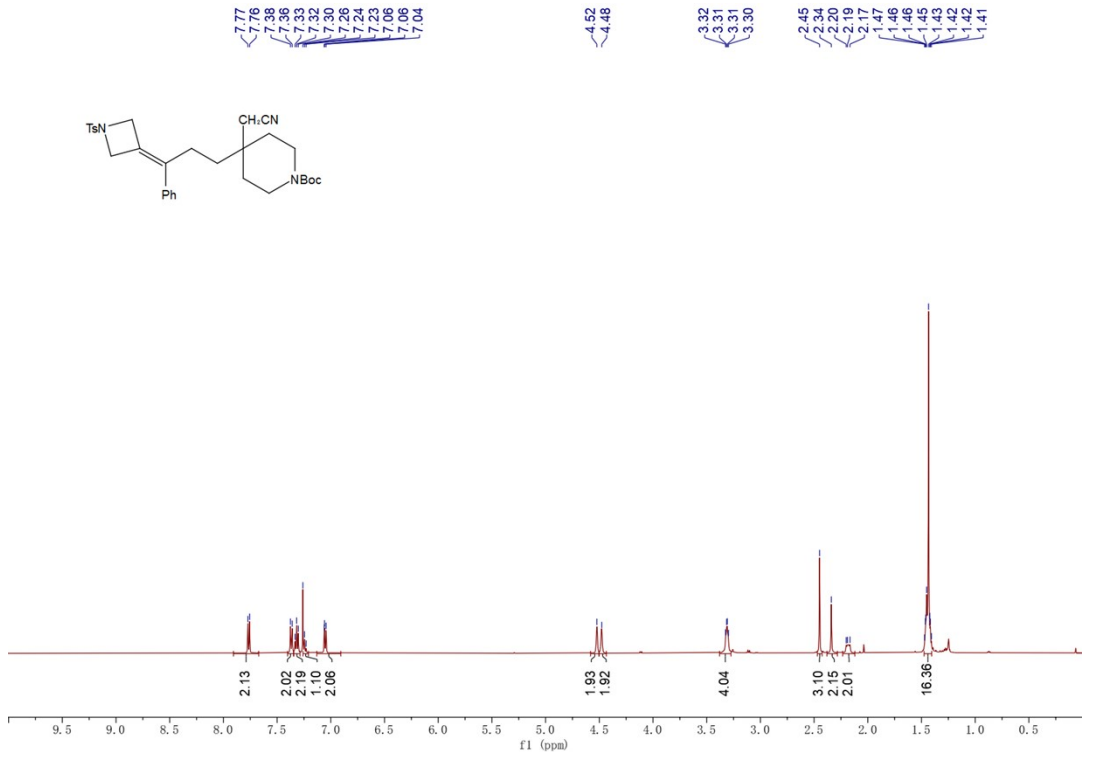
NHCbz
CN
Ph
f1 (ppm)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7.06
7.04
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3.30
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2.17
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1.46
1.46
1.45
1.43
1.42
1.42
1.41
TsN
CH2CN
NBoc
Ph
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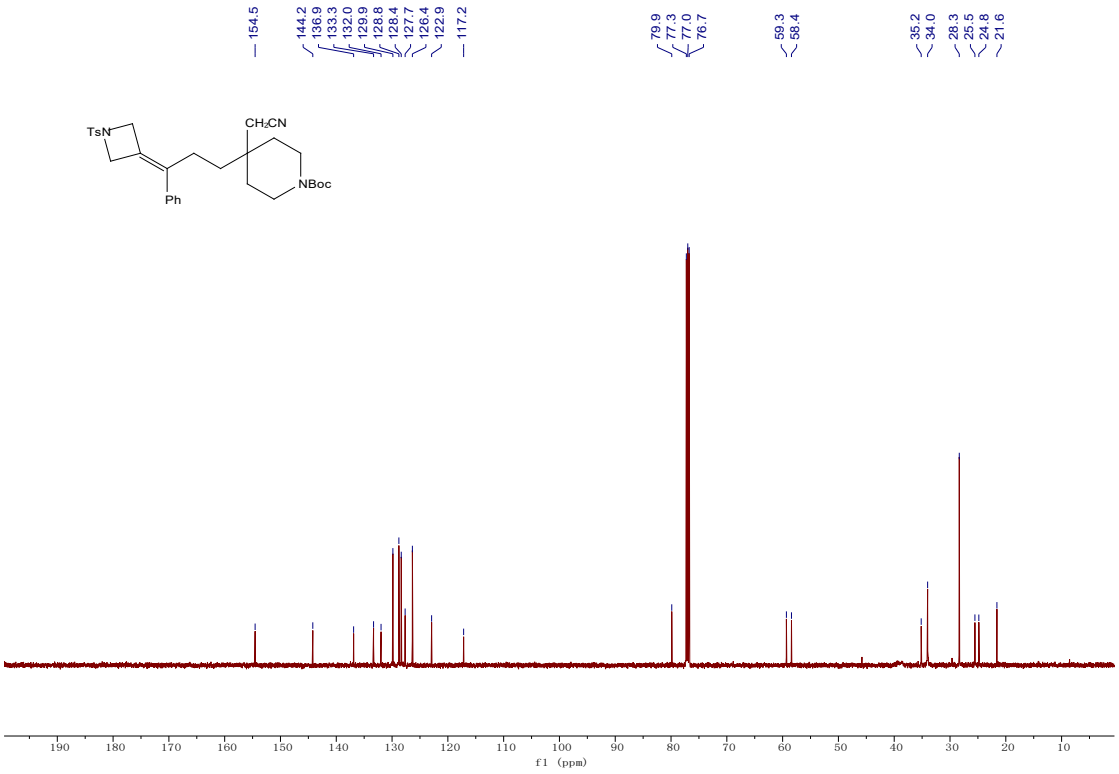
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117.2
79.9
77.3
77.0
76.7
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28.3
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24.8
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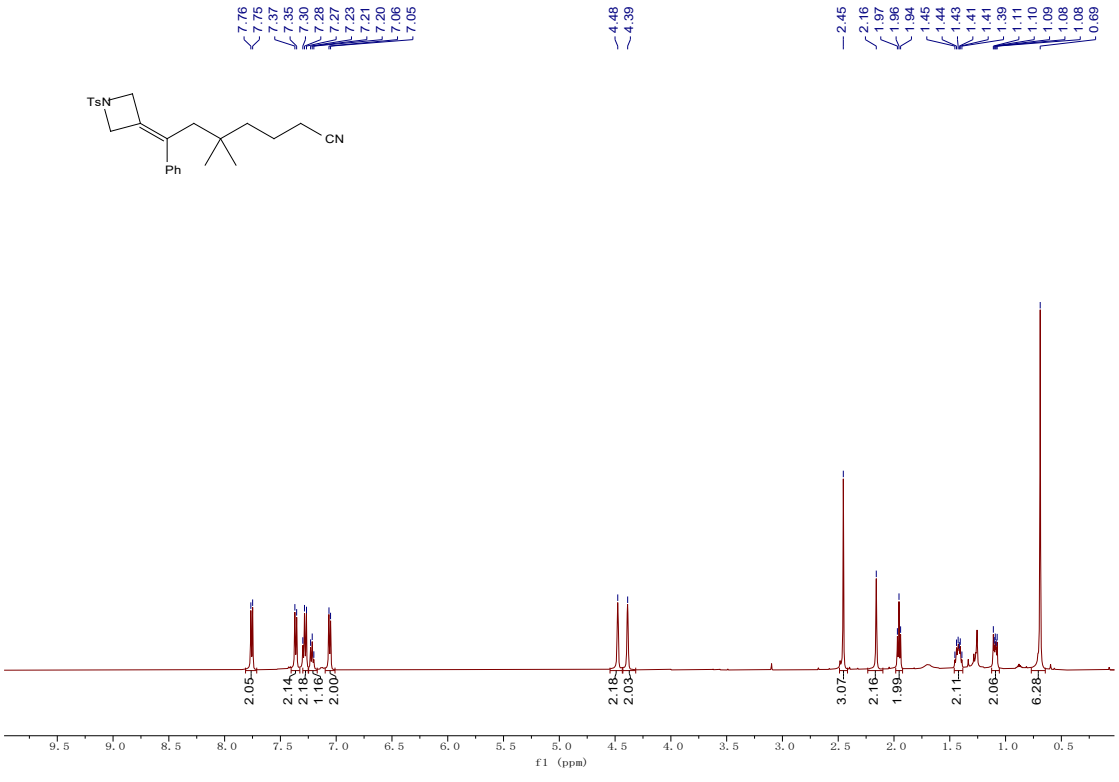
TsN
CH2CN
NBoc
Ph
f1 (ppm)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7.06
7.05
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4.39
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2.16
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1.96
1.94
1.45
1.44
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1.41
1.41
1.39
1.11
1.10
1.09
1.08
1.08
0.69
TsN
CN
Ph
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2.14
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2.03
3.07
2.16
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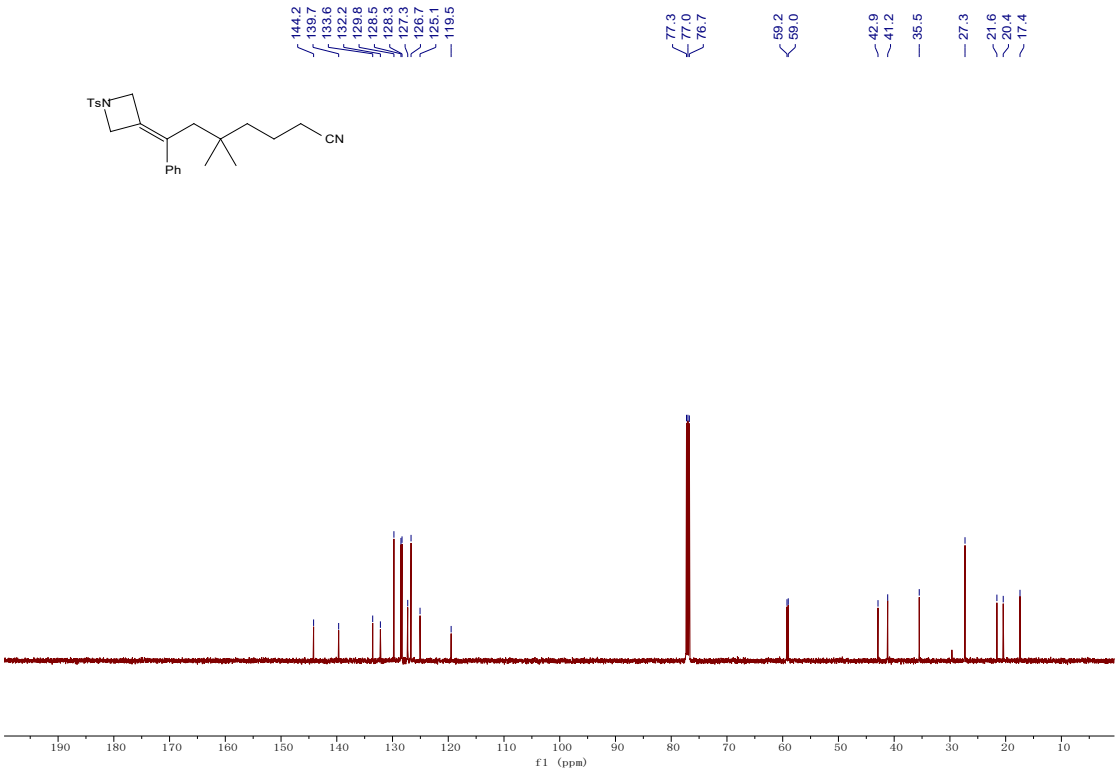
2.11
2.06
6.28
f1 (ppm)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41.2
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27.3
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20.4
17.4
TsN
CN
Ph
190
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120
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100
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80
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60
50
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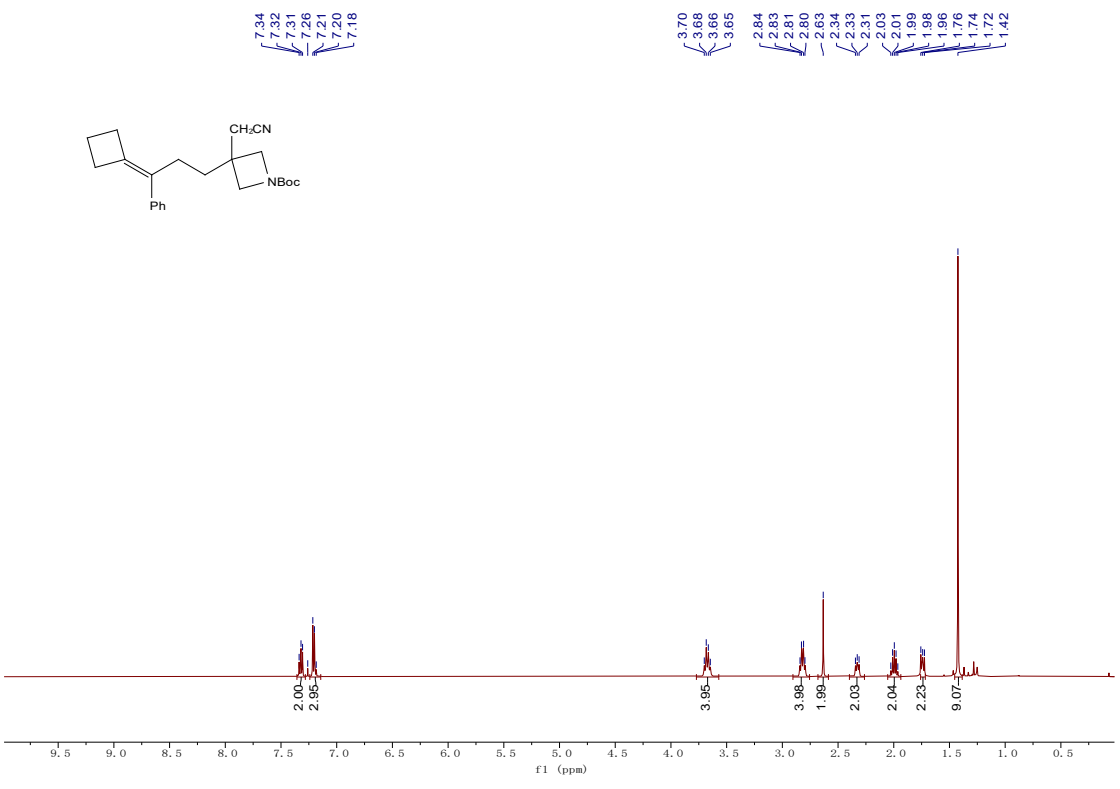
20
10
f1 (ppm)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2.33
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2.03
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1.96
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1.74
1.72
1.42
CH2CN
NBoc
Ph
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1.99
2.03
2.04
2.23
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9.5
9.0
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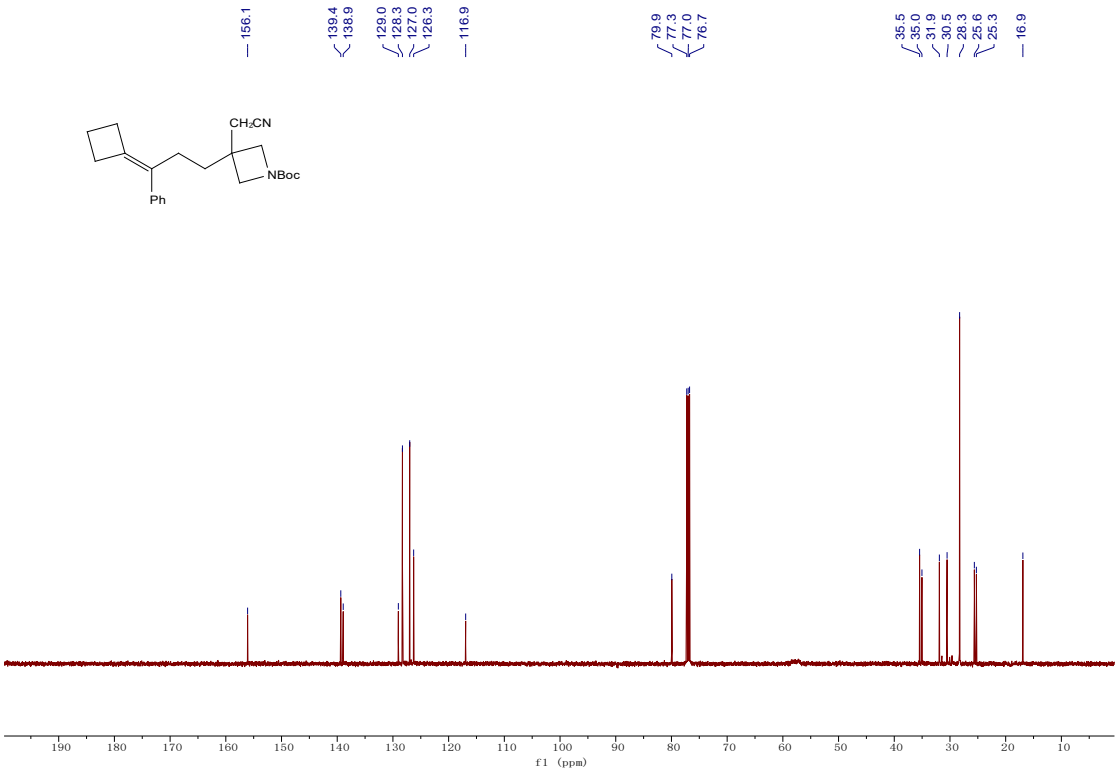
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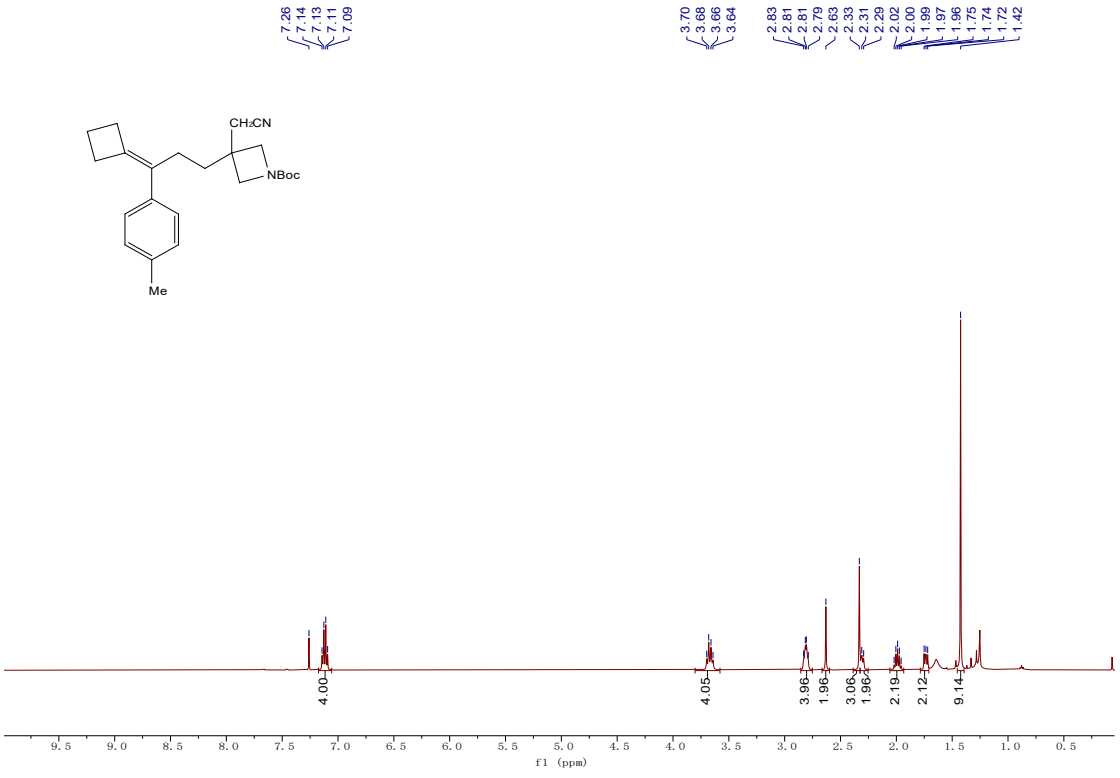
16.9
CH2CN
NBoc
Ph
f1 (ppm)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2.81
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2.79
2.63
2.33
2.31
2.29
2.02
2.00
1.99
1.97
1.96
1.75
1.74
1.72
1.42
CH2CN
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Me
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3.06
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2.19
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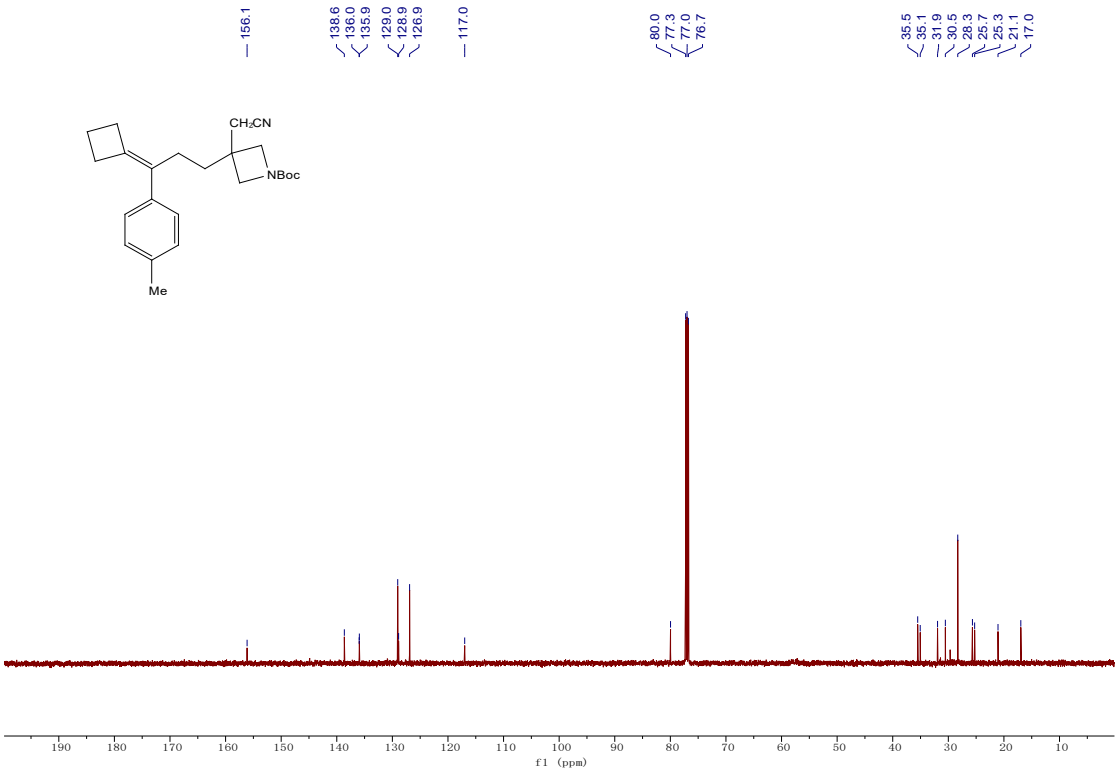
9.14
f1 (ppm)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77.0
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35.5
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17.0
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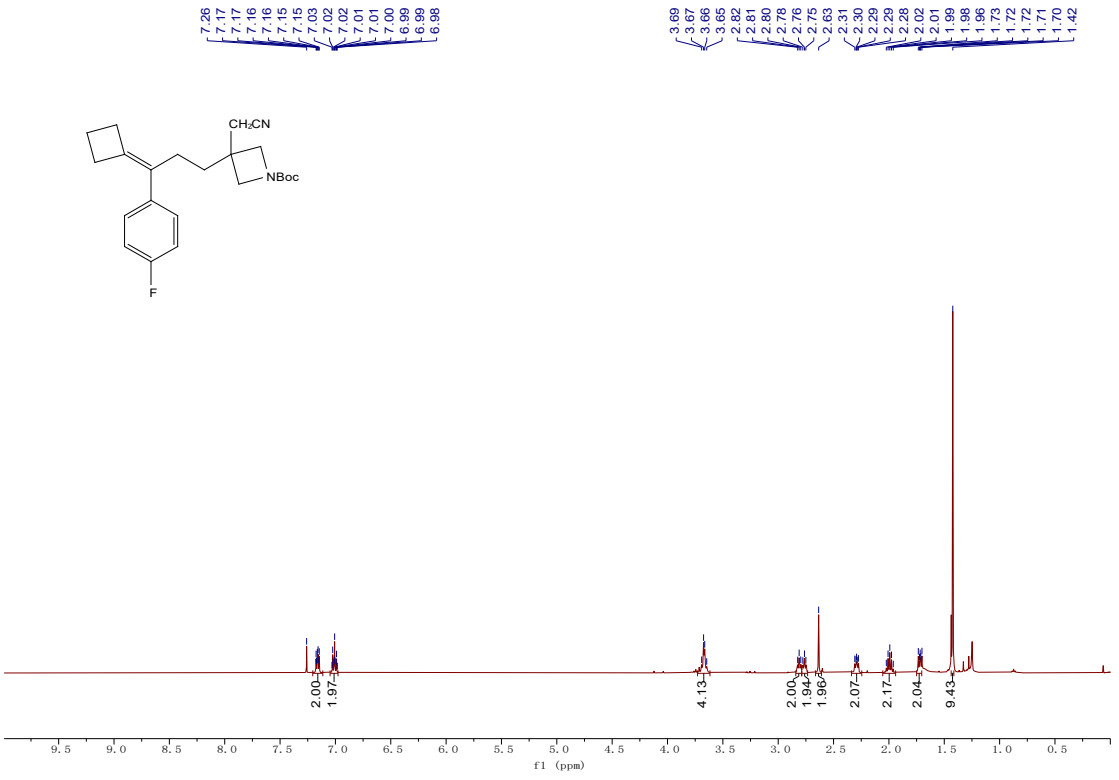
NBoc
Me
f1 (ppm)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6.99
6.98
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1.42
CH2CN
NBoc
F
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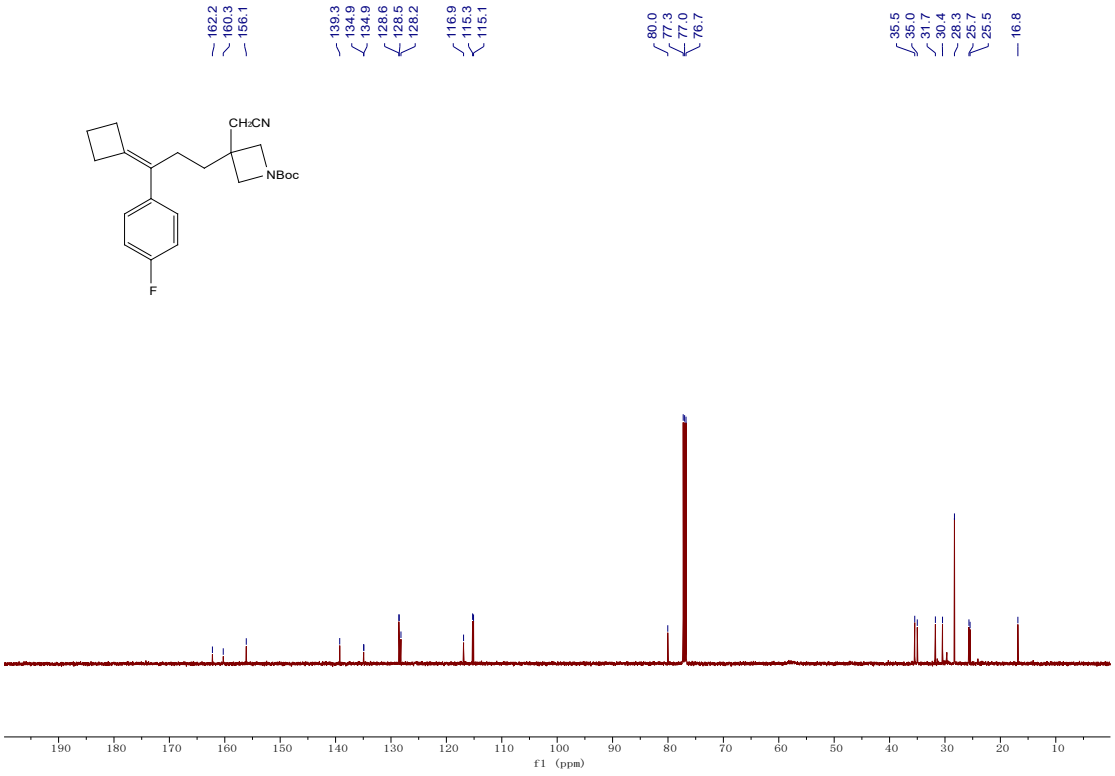
2.17
2.04
9.43
f1 (ppm)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77.0
76.7
35.5
35.0
31.7
30.4
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25.5
16.8
CH2CN
NBoc
F
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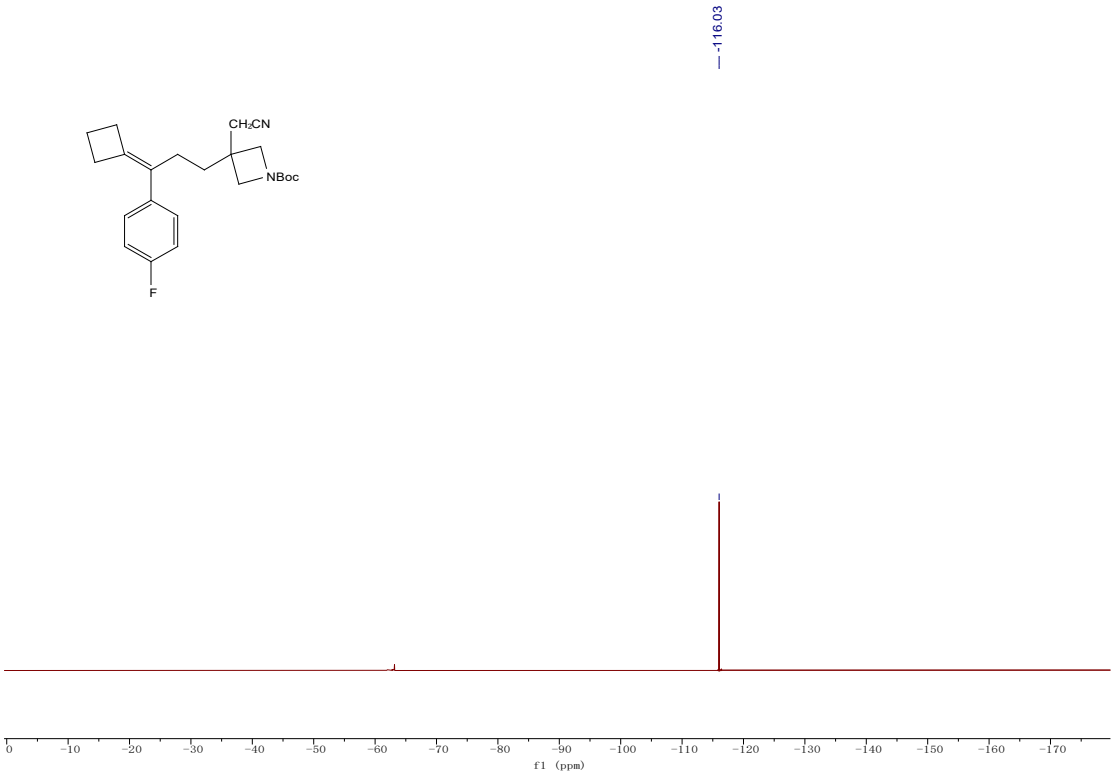
f1 (ppm)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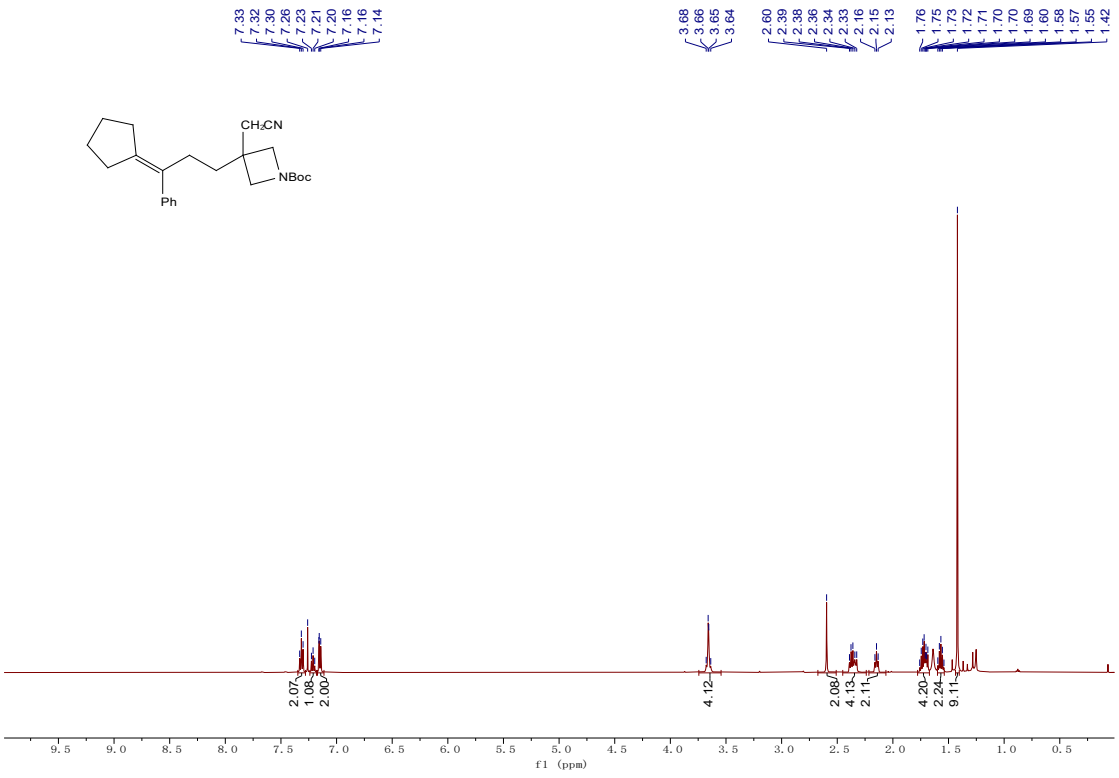
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2.33
2.16
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2.13
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1.70
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1.60
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1.57
1.55
1.42
CH2CN
NBoc
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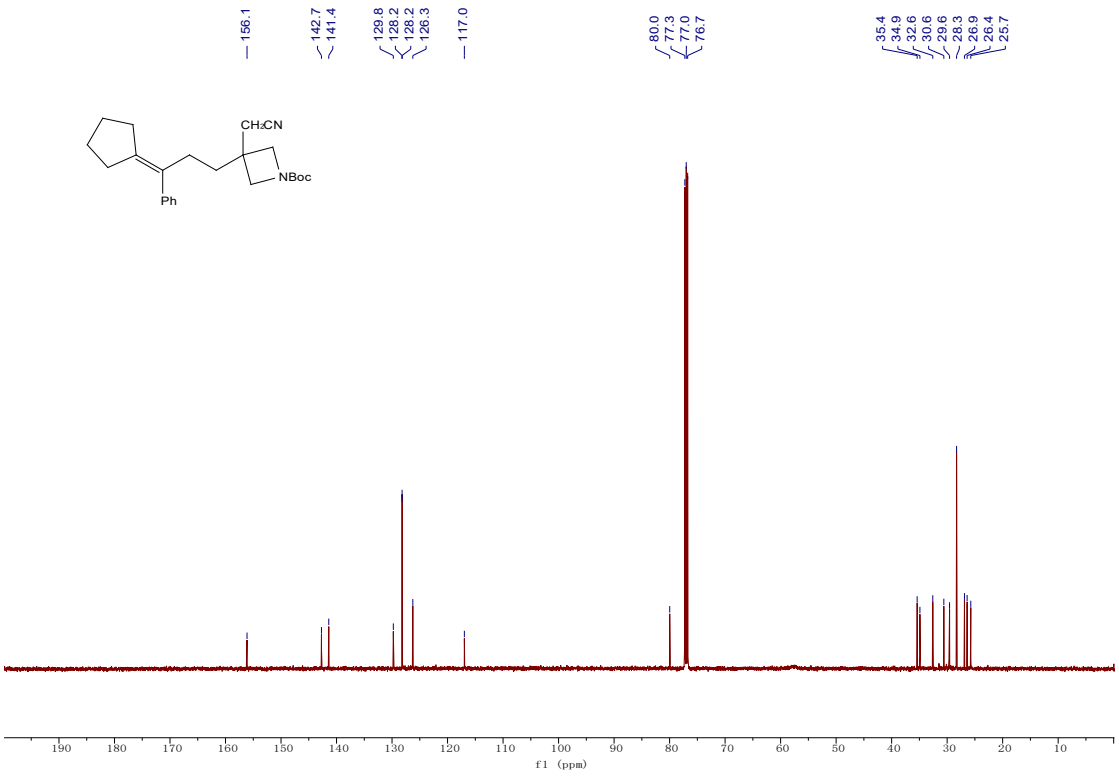
f1 (ppm)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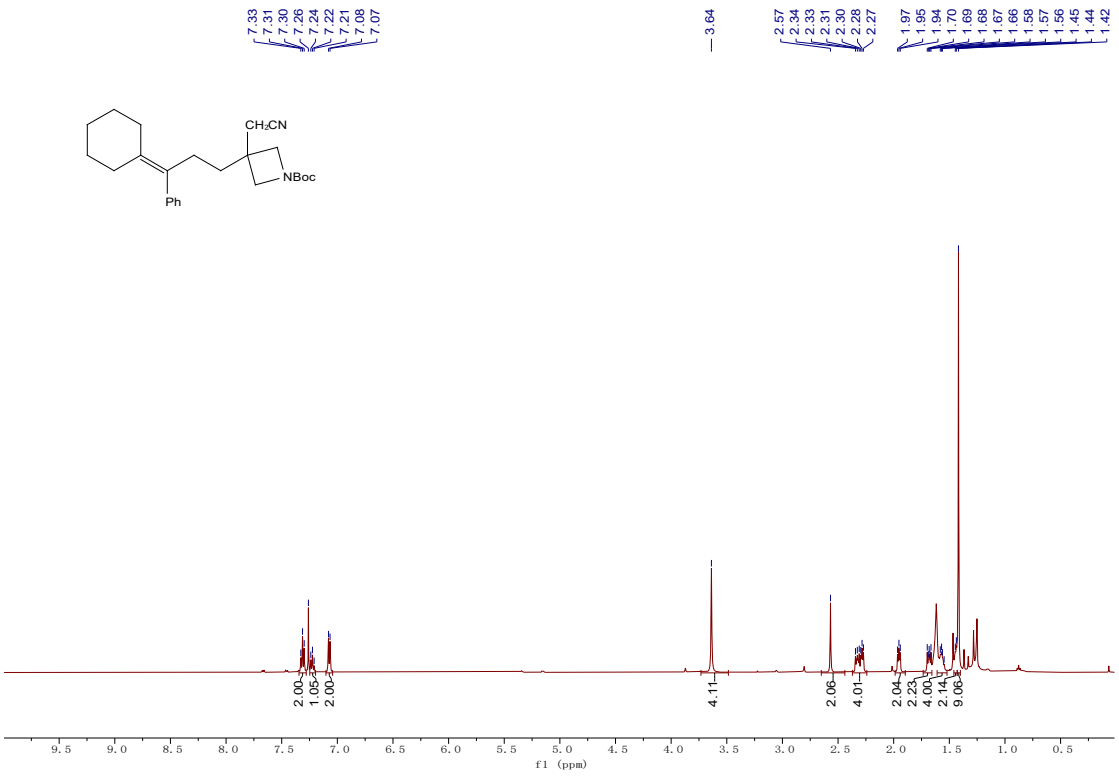
CH2CN
NBoc
Ph
f1 (ppm)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2.30
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1.56
1.45
1.44
1.42
CH2CN
NBoc
Ph
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4.01
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4.00
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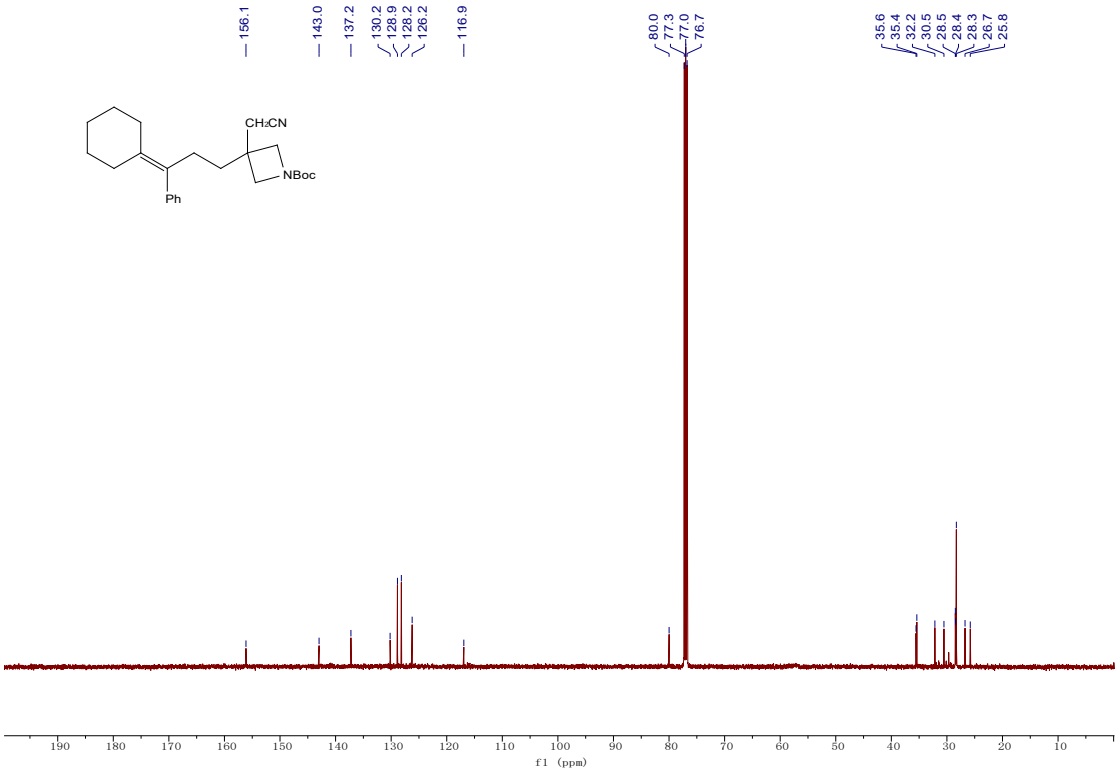
9.06
f1 (ppm)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28.4
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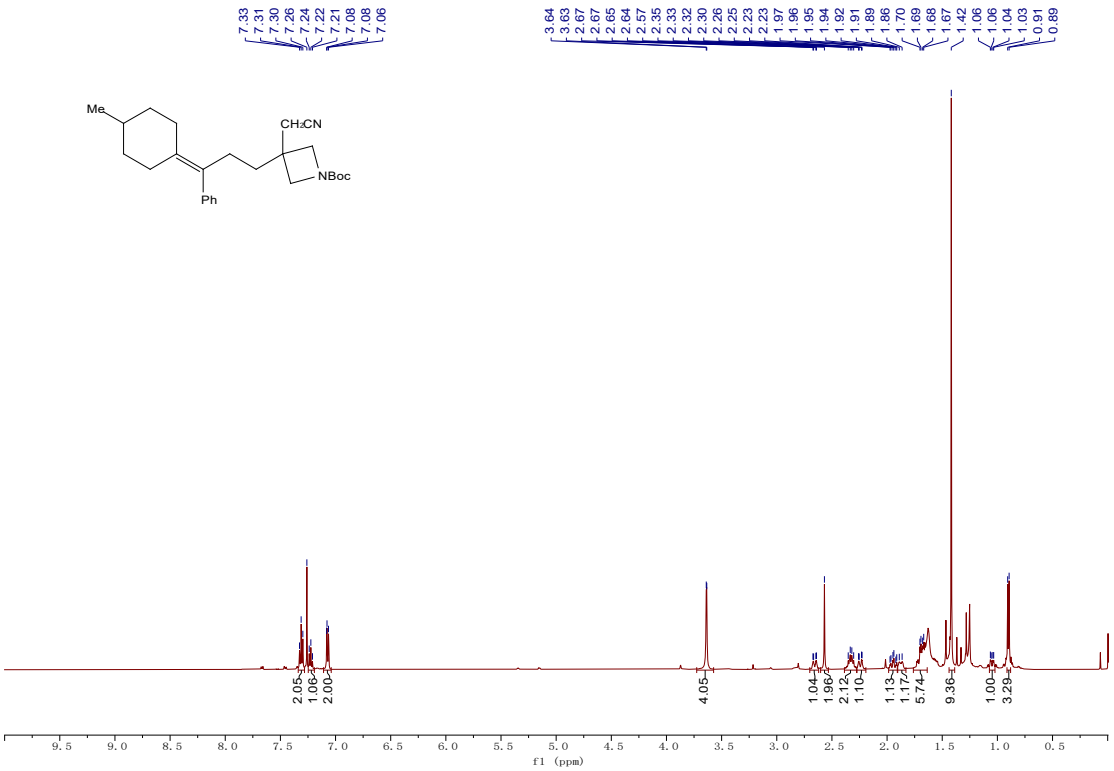
25.8
CH2CN
NBoc
Ph
f1 (ppm)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2.64
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1.06
1.04
1.03
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Me
CH2CN
NBoc
Ph
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2.00
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1.13
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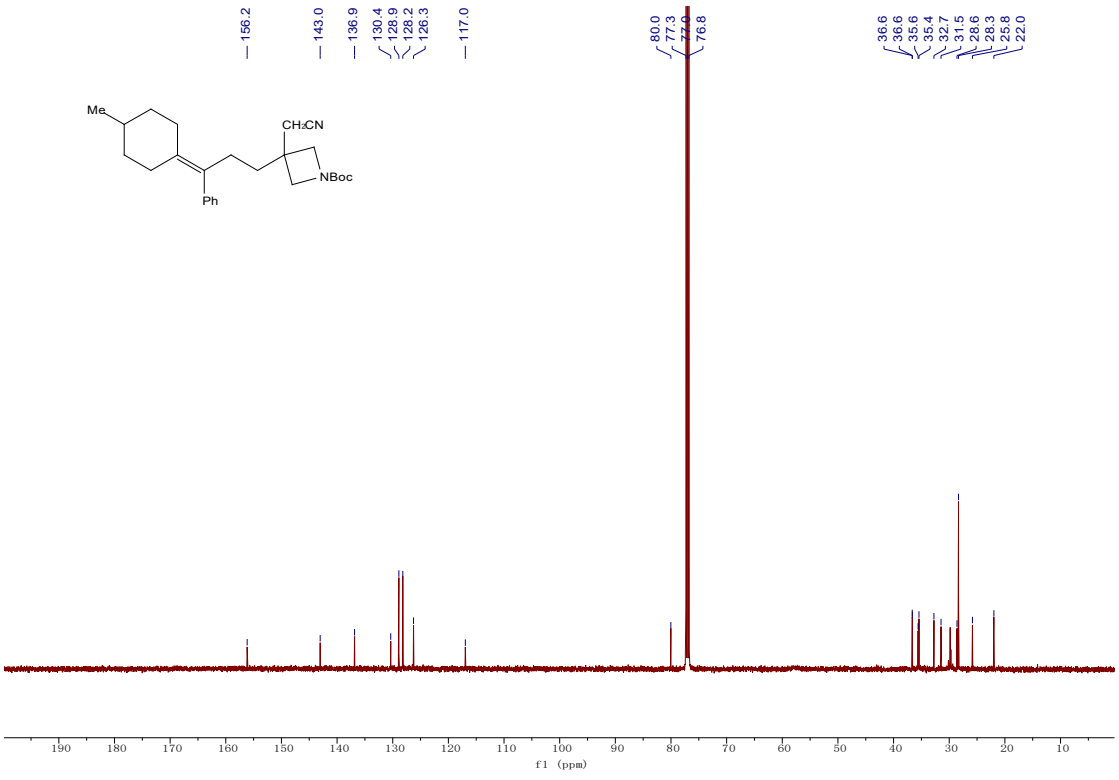
f1 (ppm)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36.6
36.6
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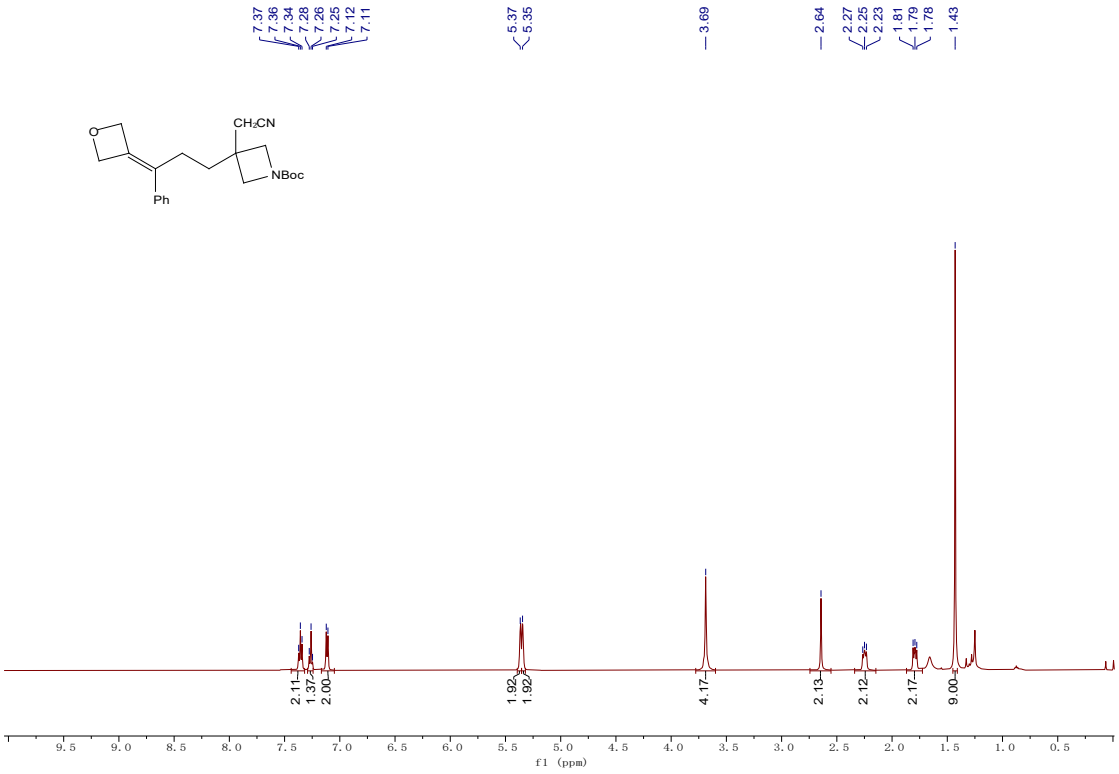
22.0
Me
CH2CN
NBoc
Ph
f1 (ppm)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2.27
2.25
2.23
1.81
1.79
1.78
1.43
CH2CN
O
NBoc
Ph
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2.12
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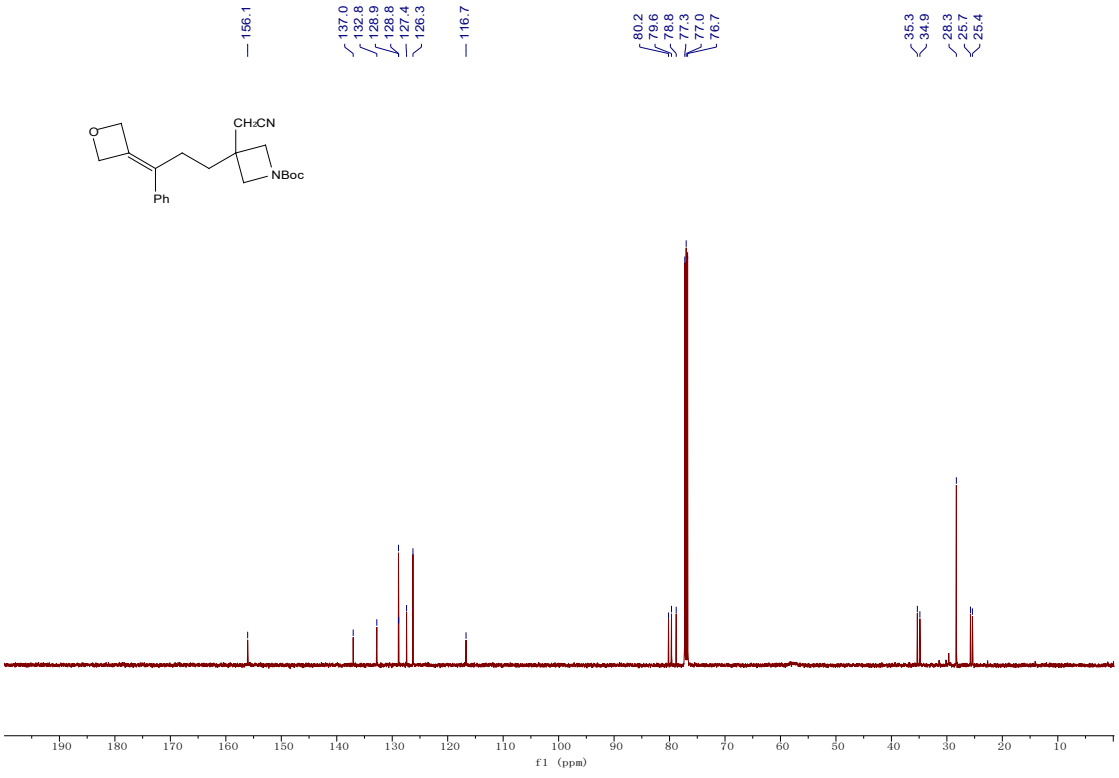
9.00
f1 (ppm)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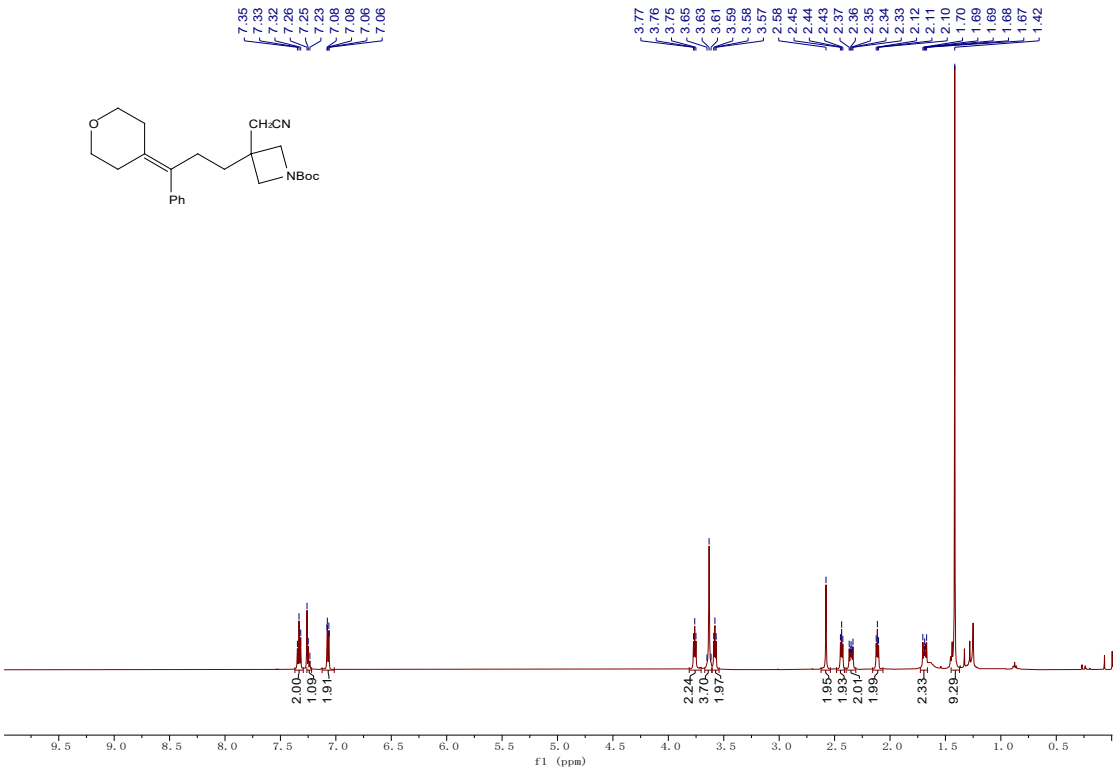
25.4
CH2CN
O
NBoc
Ph
f1 (ppm)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3.58
3.57
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2.34
2.33
2.12
2.11
2.10
1.70
1.69
1.69
1.68
1.67
1.42
O
CH2CN
NBoc
Ph
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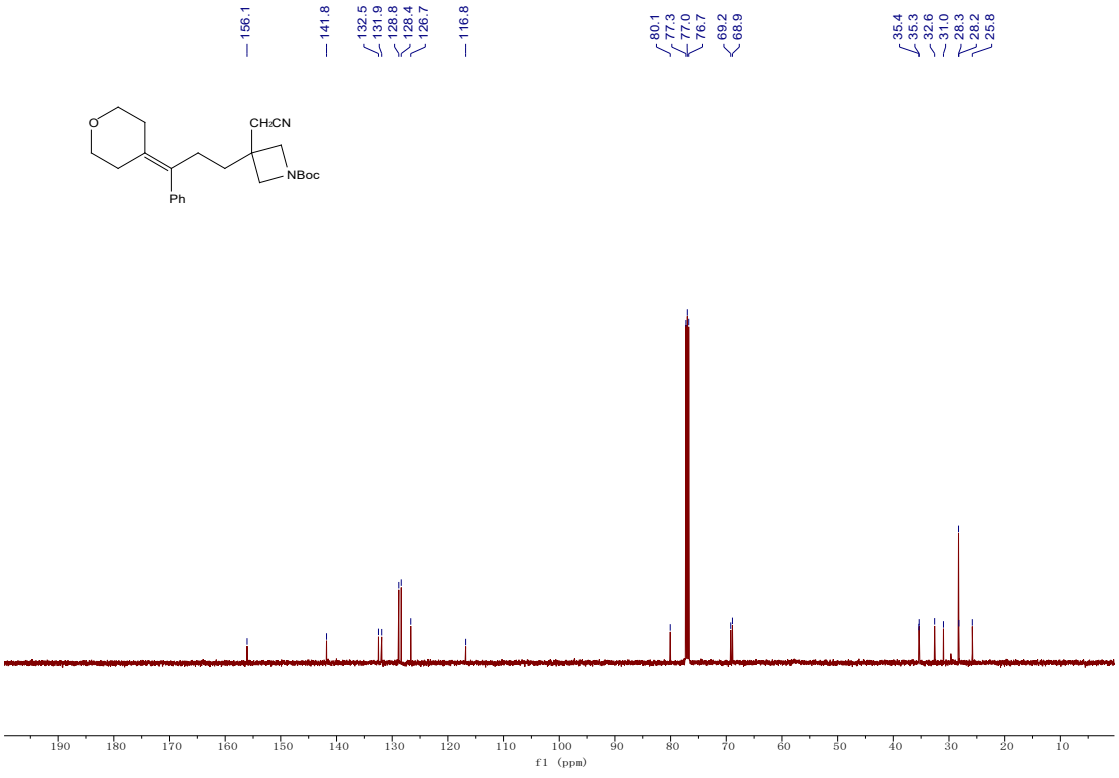
1.93
2.01
1.99
2.33
9.29
f1 (ppm)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68.9
35.4
35.3
32.6
31.0
28.3
28.2
25.8
O
CH2CN
NBoc
Ph
f1 (ppm)


7.41
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7.11
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7.04
7.03
7.01
7.01
7.00
7.00
6.99
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6.89
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6.88
6.87


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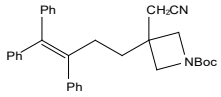
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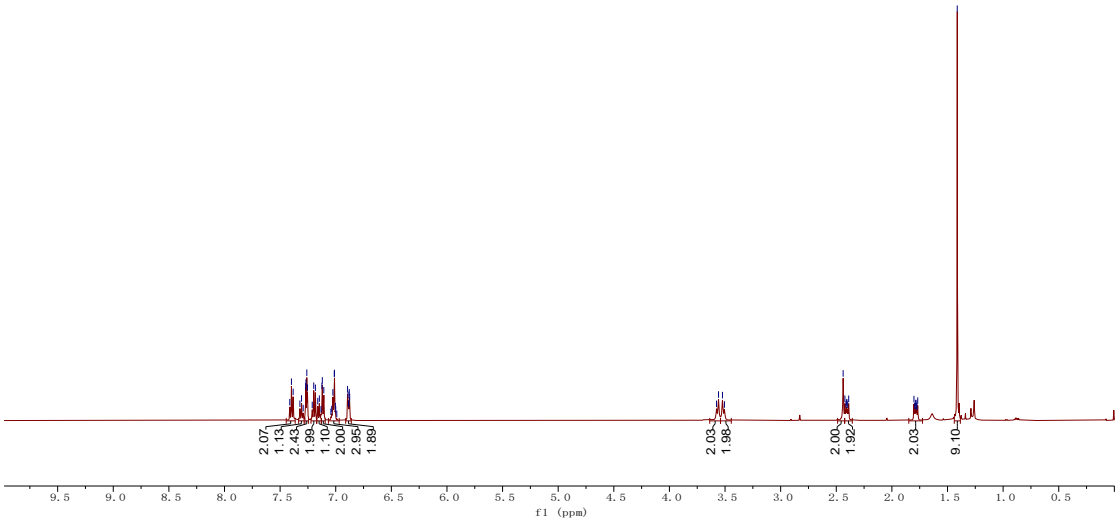
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9.0
8.5
8.0
7.5
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3.0
2.5
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1.0
0.5
f1 (ppm)


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129.4
129.1
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128.2
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127.1
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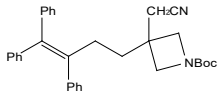
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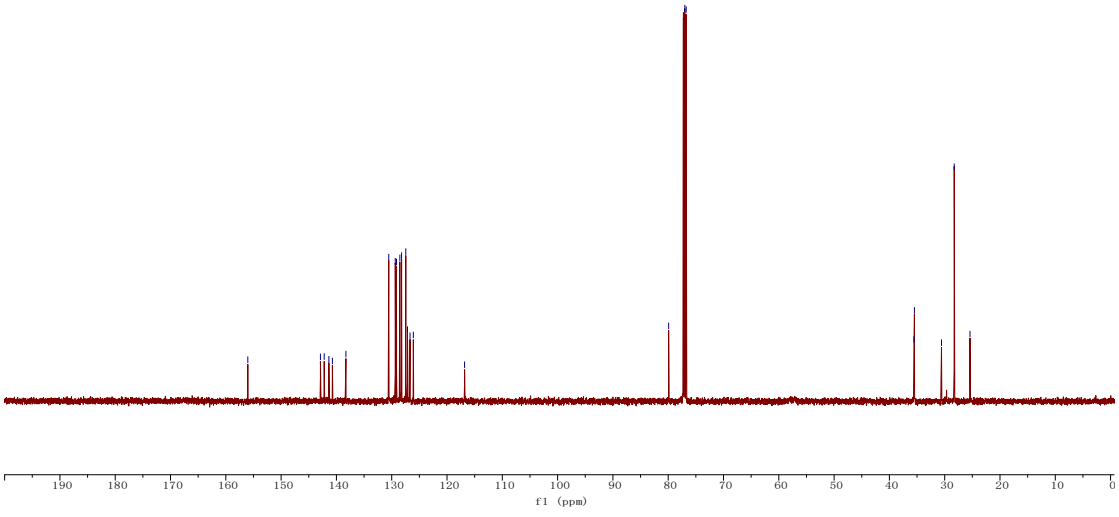
NBoc
Ph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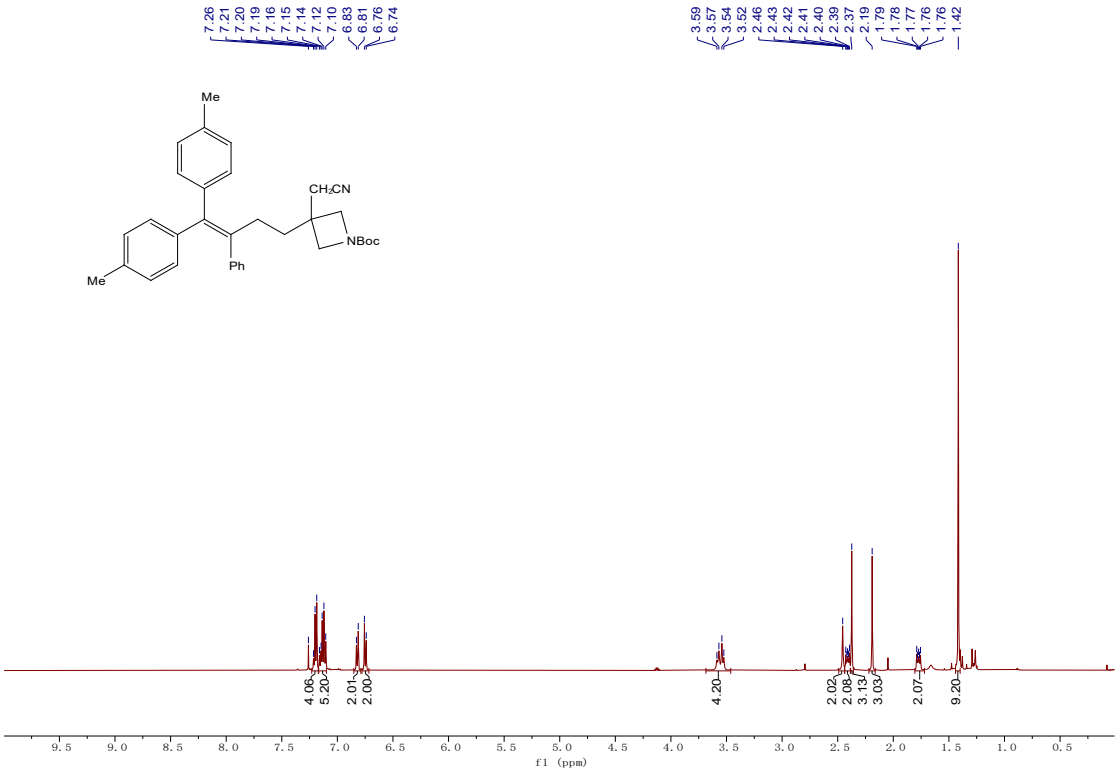
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1.77
1.76
1.76
1.42
Me
CH2CN
NBoc
Ph
Me
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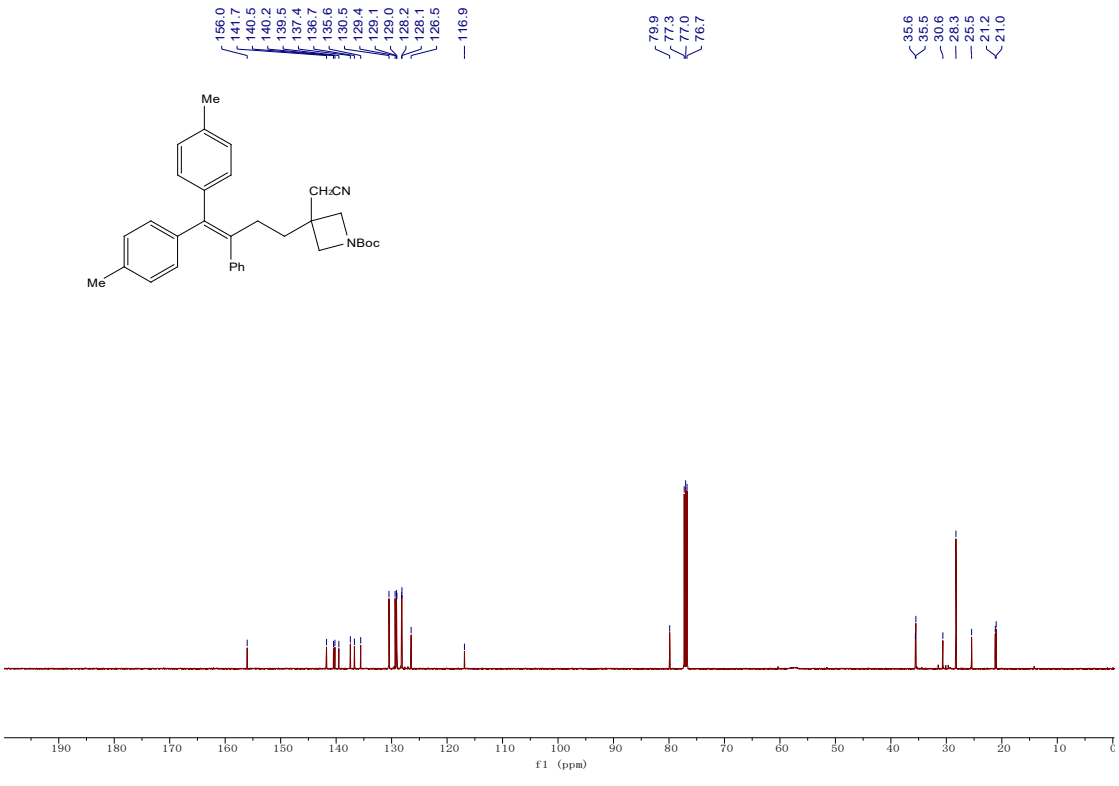
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126.5
116.9
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77.0
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NBoc
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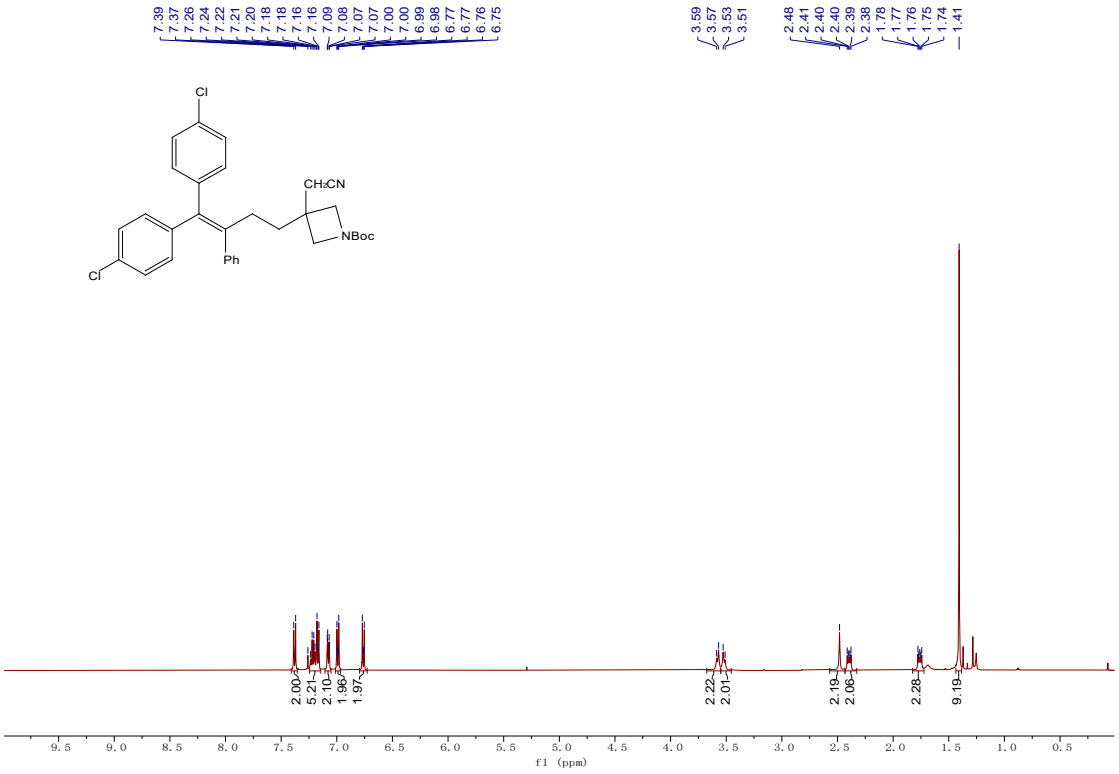
Me
f1 (ppm)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7.07
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3.53
3.51
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2.39
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1.77
1.76
1.75
1.74
1.41
Cl
CH2CN
NBoc
Ph
Cl
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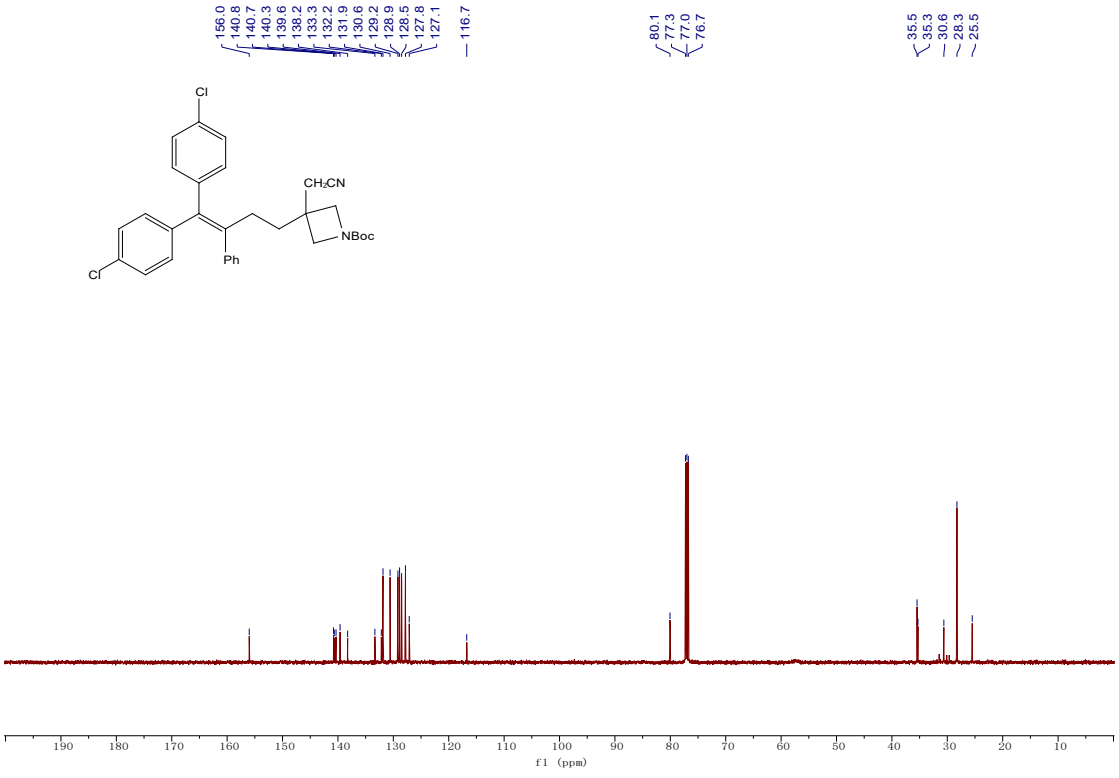
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127.1
116.7
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77.3
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Cl
CH2CN
NBoc
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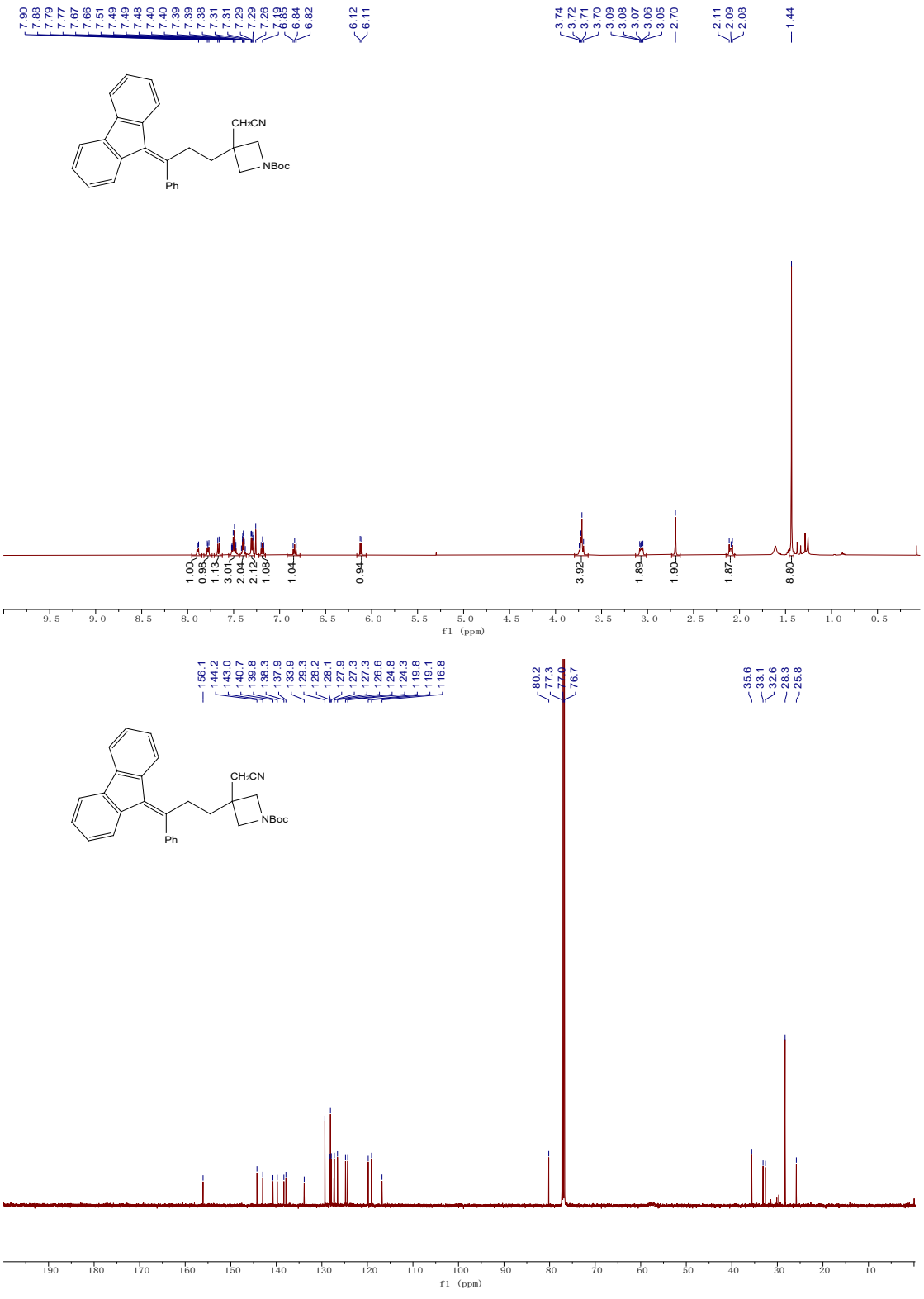
Cl
f1 (ppm)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f1 (ppm)
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128.1
127.9
127.3
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124.3
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119.1
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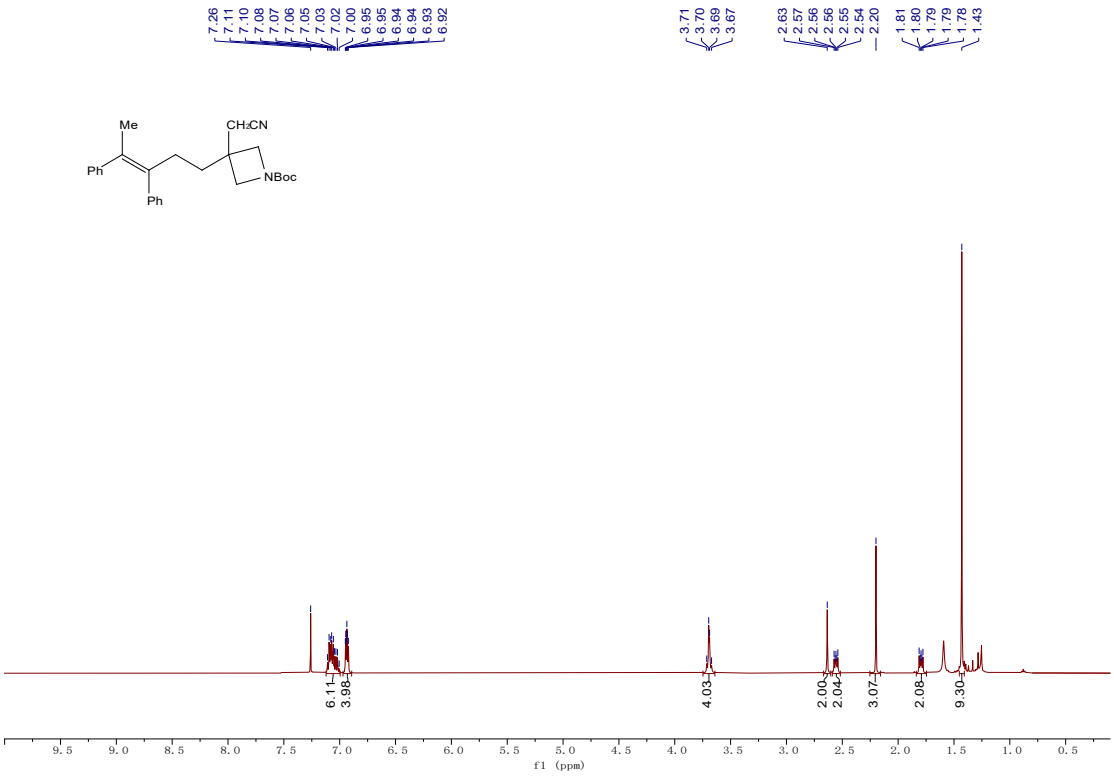
Ph
f1 (ppm)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1.43
Me
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NBoc
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9.30
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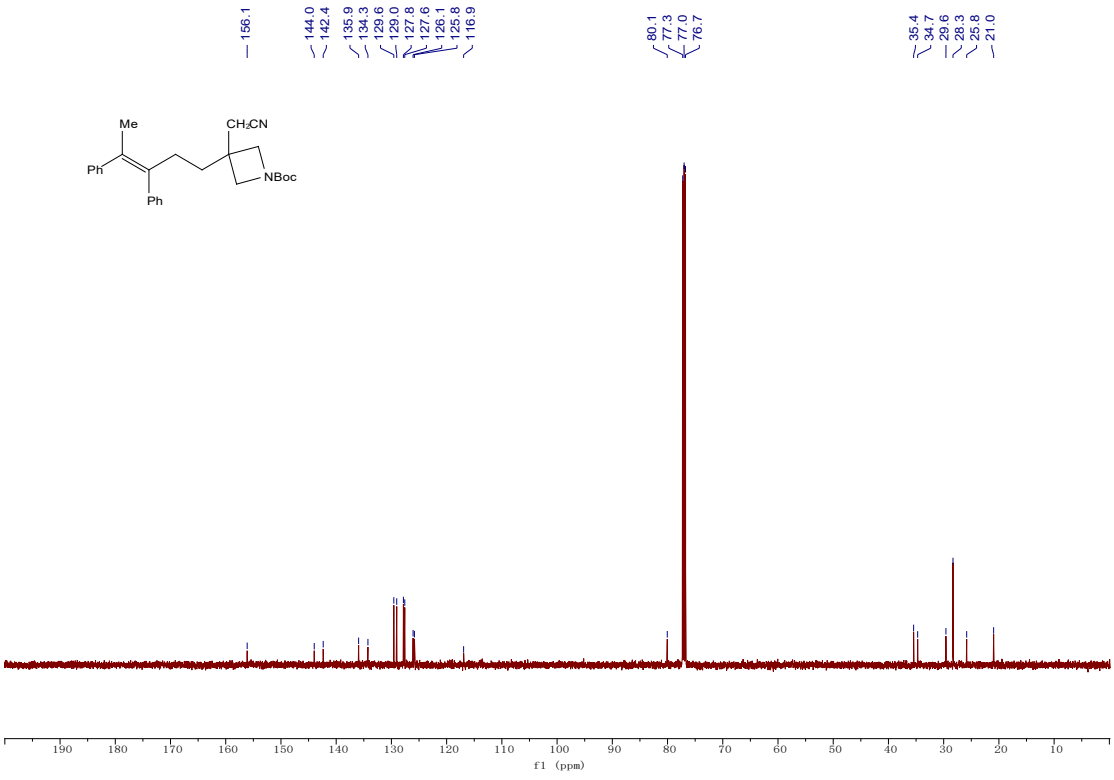
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77.0
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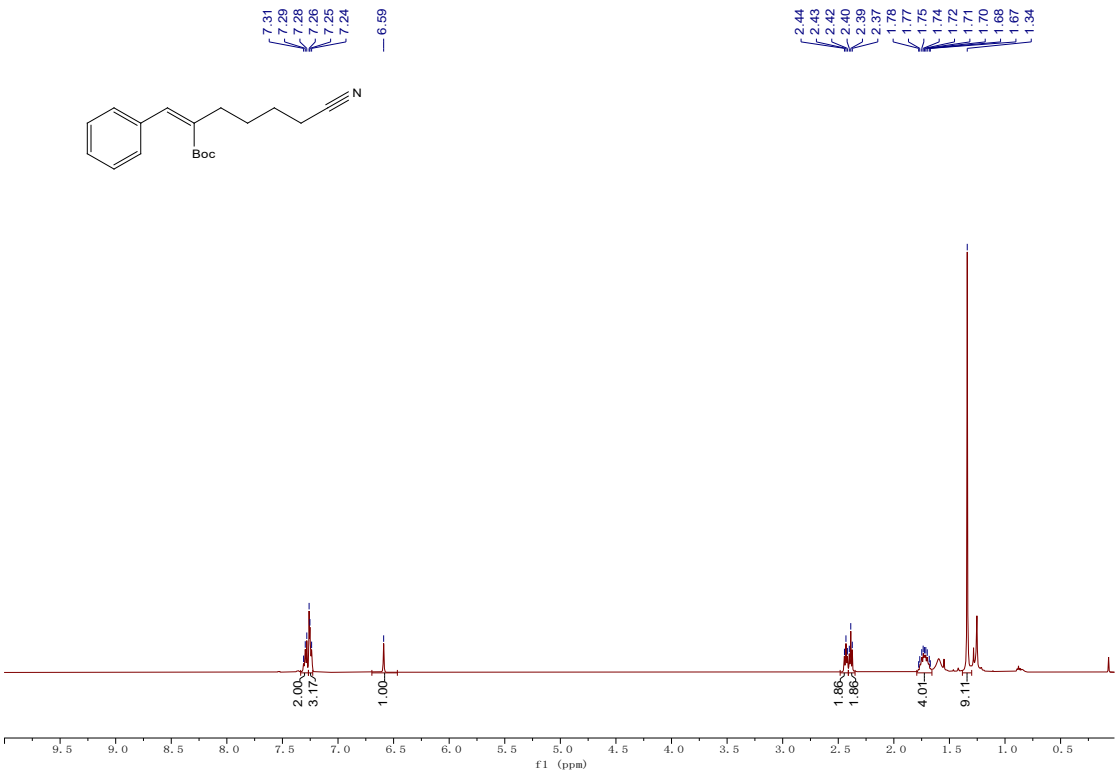
CH2CN
Ph
NBoc
Ph
f1 (ppm)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1.77
1.75
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Boc
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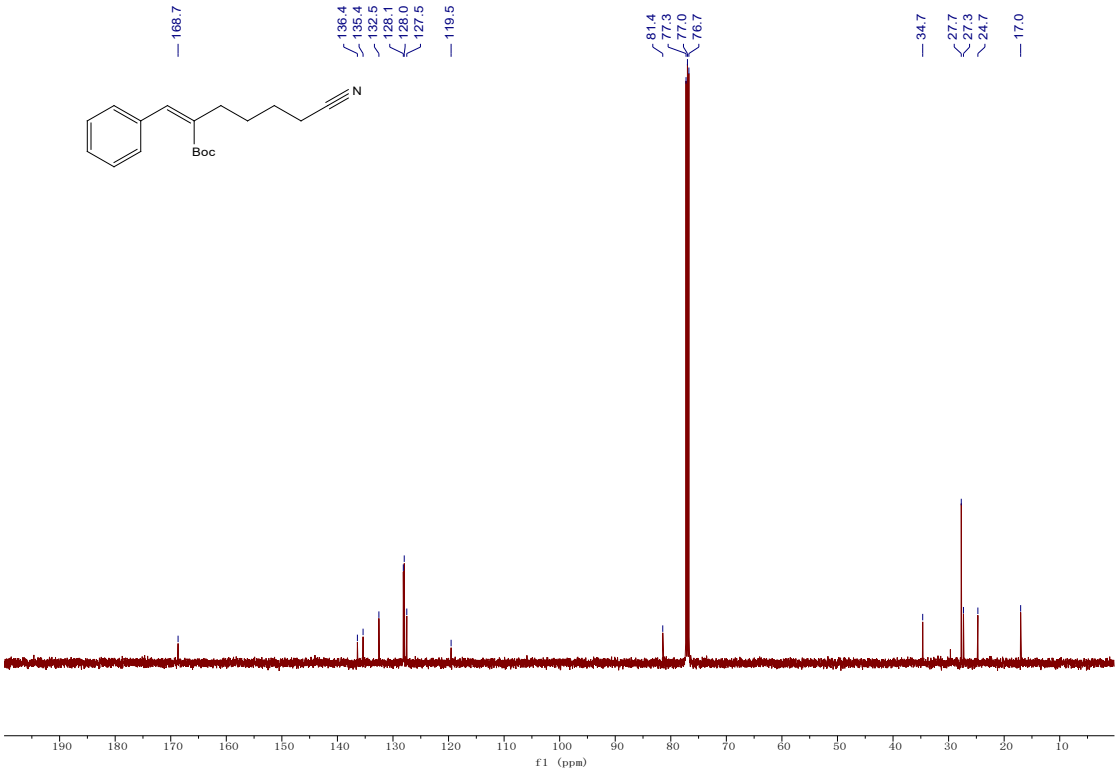
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24.7
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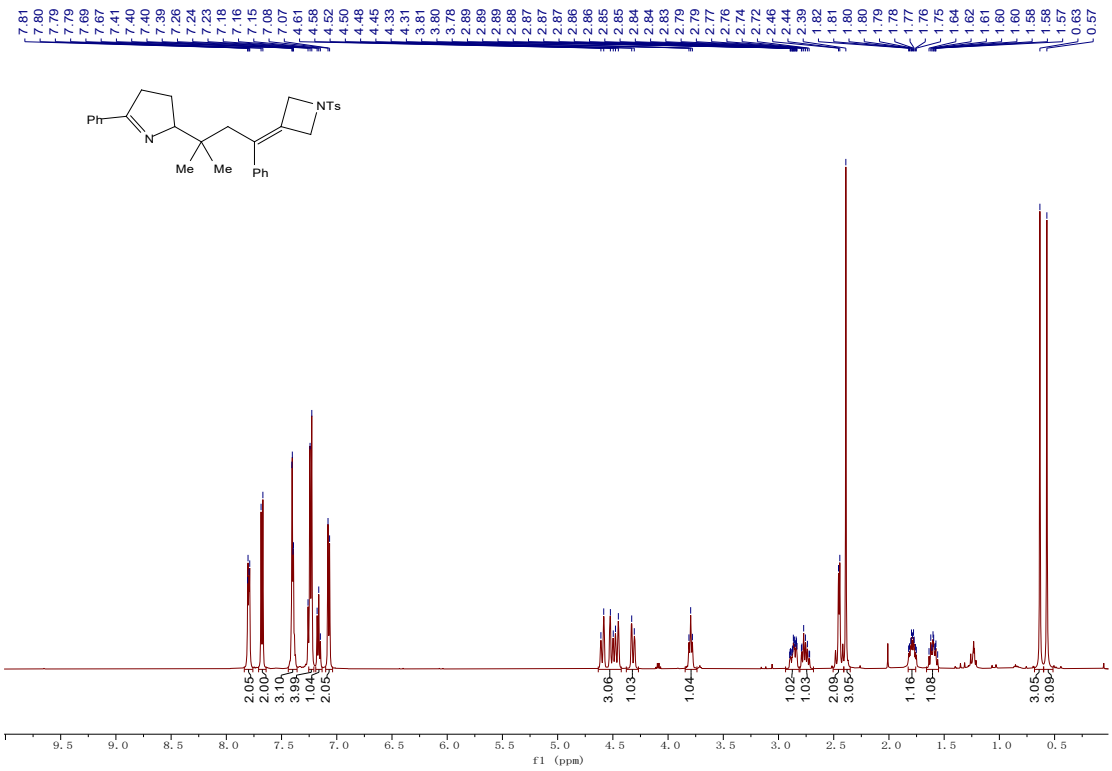
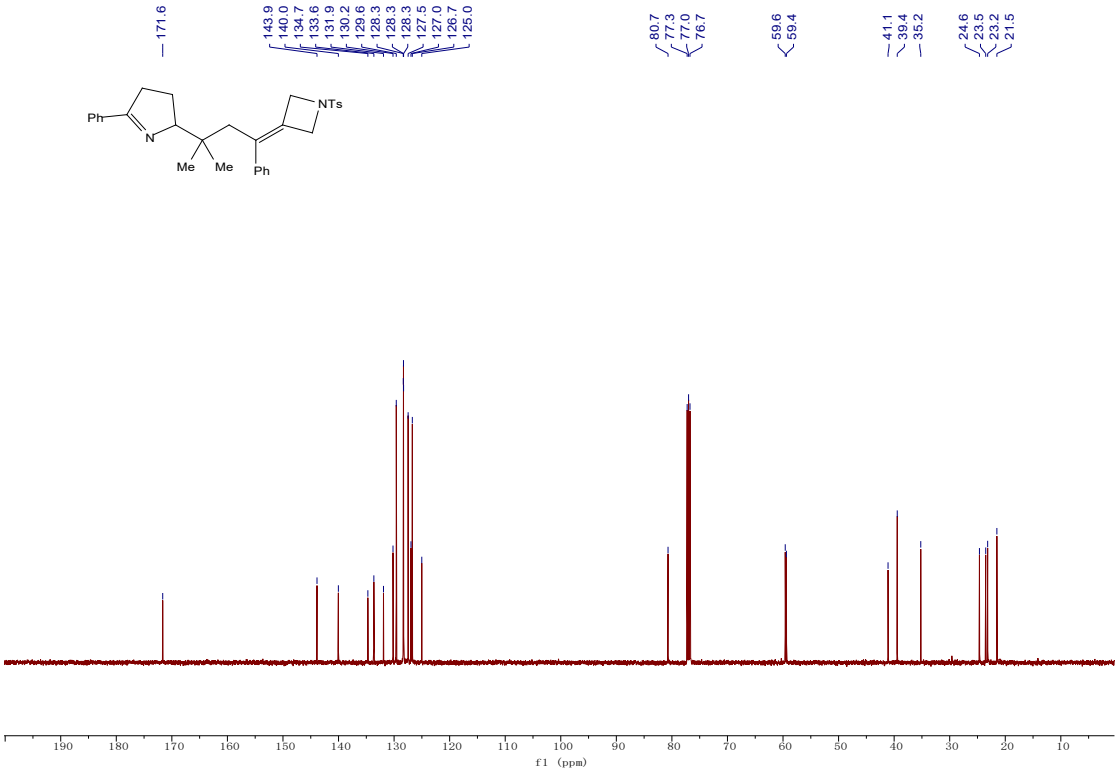
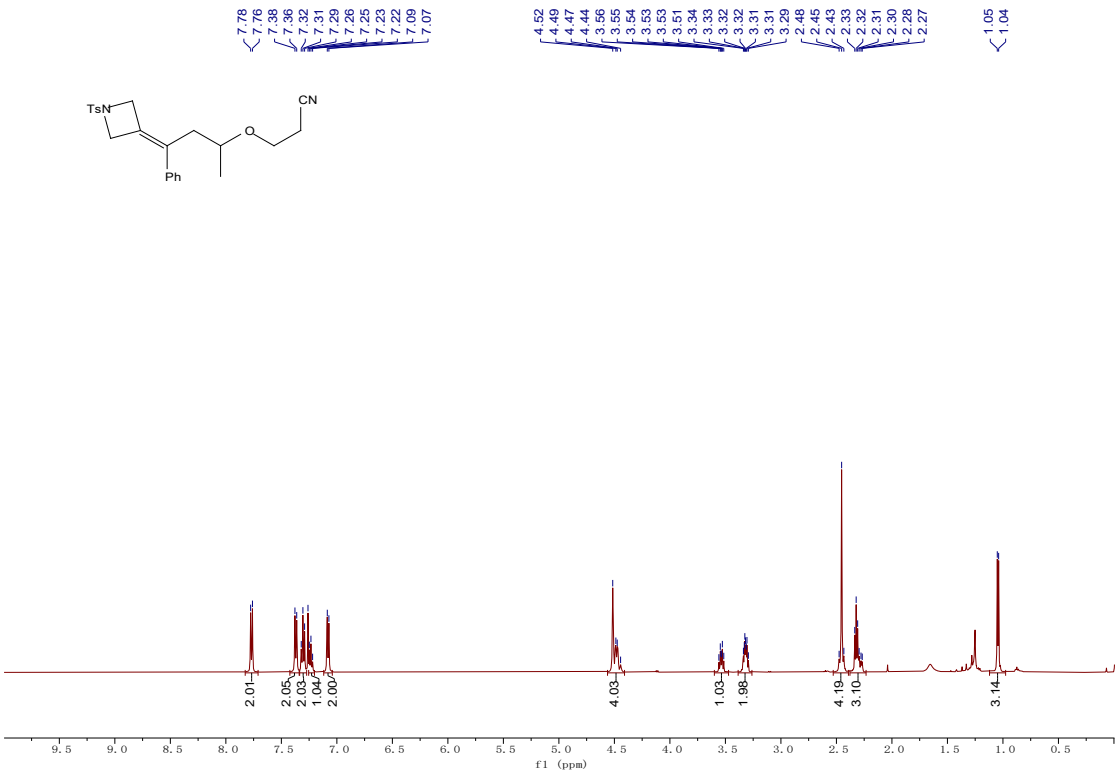
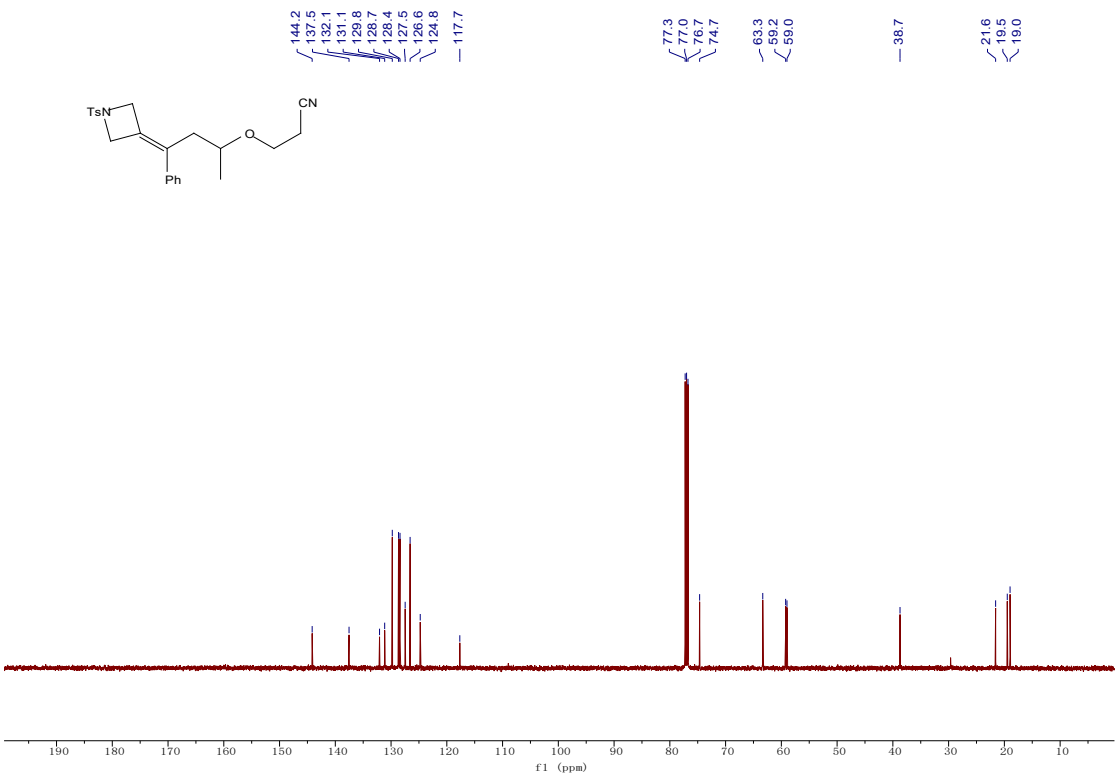
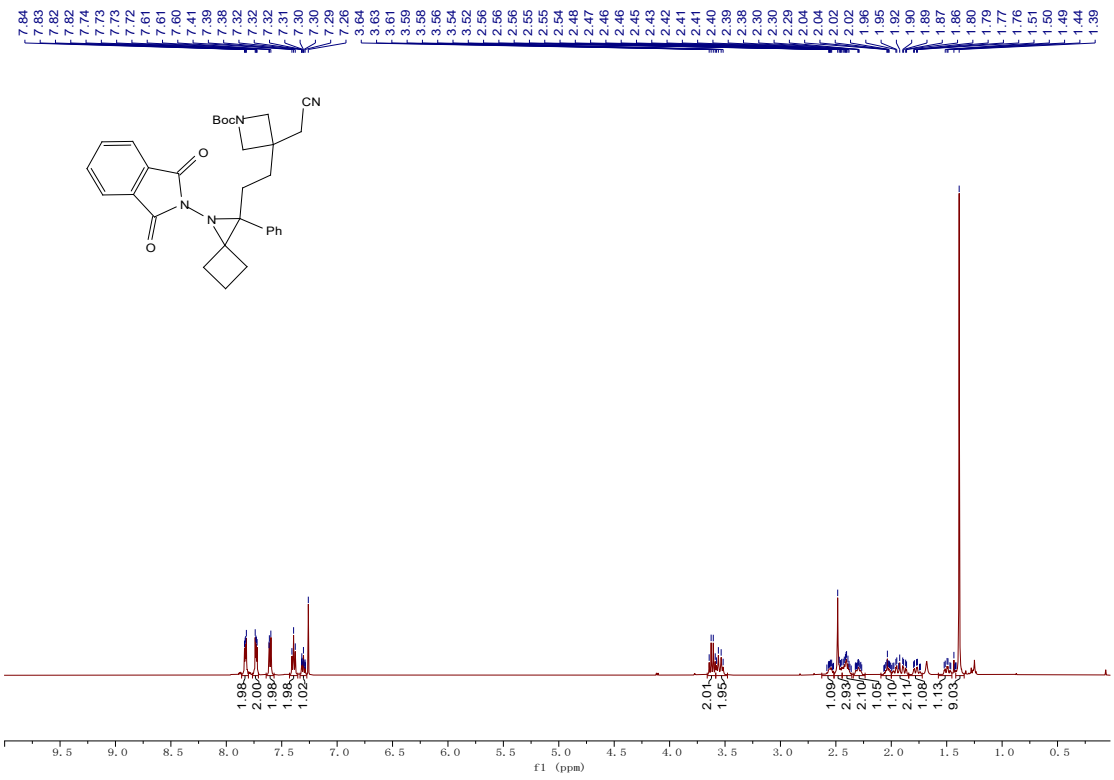
N
Boc
f1 (ppm)

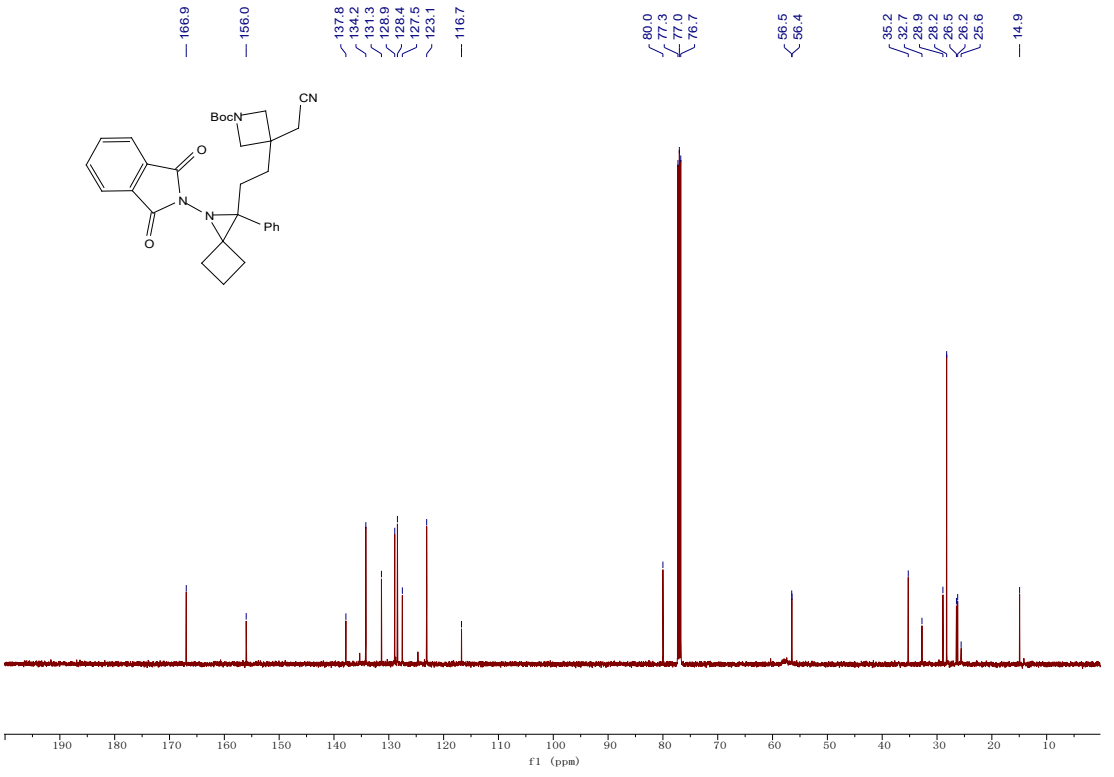

7.81 7.80 7.79 7.79 7.69 7.67 7.41 7.40 7.40 7.39 7.26 7.24 7.23 7.18 7.16 7.15 7.08 7.07 4.61 4.58 4.52 4.50 4.48 4.45 4.33 4.31 3.81 3.80 3.78 2.89 2.89 2.88 2.87 2.87 2.87 2.86 2.86 2.85 2.85 2.84 2.84 2.83 2.79 2.79 2.77 2.76 2.74 2.72 2.46 2.44 2.39 1.82 1.81 1.80 1.80 1.79 1.78 1.77 1.76 1.75 1.64 1.62 1.61 1.60 1.60 1.58 1.58 1.57 0.63 0.57
Ph
N
NTs
Me
Me
Ph
2.05 2.00 3.10 3.99 1.04 2.05 3.06 1.03 1.04 1.02 1.03 2.09 3.05 1.16 1.08 3.05 3.09
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
f1 (ppm)


171.6
143.9 140.0 134.7 133.6 131.9 130.2 129.6 128.3 128.3 128.3 127.5 127.0 126.7 125.0
80.7 77.3 77.0 76.7
59.6 59.4
41.1 39.4 35.2
24.6 23.5 23.2 21.5
Ph
N
NTs
Me
Me
Ph
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


7.78
7.76
7.38
7.36
7.32
7.31
7.29
7.26
7.25
7.23
7.22
7.09
7.07
4.52
4.49
4.47
4.44
3.56
3.55
3.54
3.53
3.53
3.51
3.34
3.33
3.32
3.32
3.31
3.31
3.29
2.48
2.45
2.43
2.33
2.32
2.31
2.30
2.28
2.27
1.05
1.04
TsN
CN
O
Ph
2.01
2.05
2.03
1.04
2.00
4.03
1.03
1.98
4.19
3.10
3.14
f1 (ppm)


144.2
137.5
132.1
131.1
129.8
128.7
128.4
127.5
126.6
124.8
117.7
77.3
77.0
76.7
74.7
63.3
59.2
59.0
38.7
21.6
19.5
19.0
TsN
CN
O
Ph
f1 (ppm)


7.84 7.83 7.82 7.82 7.74 7.73 7.73 7.72 7.61 7.61 7.60 7.41 7.39 7.38 7.32 7.32 7.31 7.30 7.30 7.29 7.26
3.64 3.63 3.61 3.59 3.58 3.56 3.54 3.52 2.56 2.56 2.56 2.55 2.55 2.54 2.48 2.47 2.46 2.46 2.45 2.43 2.42 2.41 2.41 2.40 2.39 2.38 2.30 2.30 2.29 2.04 2.04 2.02 2.02 1.96 1.95 1.92 1.90 1.89 1.87 1.86 1.80 1.79 1.77 1.76 1.51 1.50 1.49 1.44 1.39
BocN
CN
O
N
N
Ph
O
1.98 2.00 1.98 1.99 1.02
2.01 1.95
1.09 2.93 2.10 1.05 1.10 2.11 1.08 1.13 9.03
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
f1 (ppm)


166.9 156.0 137.8 134.2 131.3 128.9 128.4 127.5 123.1 116.7 80.0 77.3 77.0 76.7 56.5 56.4 35.2 32.7 28.9 28.2 26.5 26.2 25.6 14.9
BocN
CN
O
N
N
Ph
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)