Supplementary Information

Practical aziridination of α,β-unsaturated carbonyl compounds with a simple carbamate utilizing sodium hypochlorite pentahydrate

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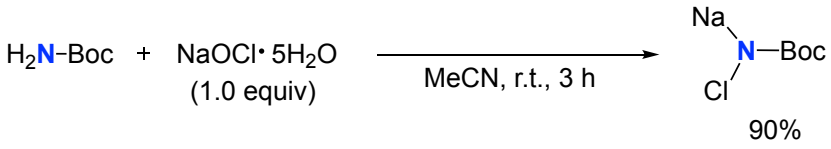
1. Materials and General

Unless otherwise noted, all reactions were performed in two-neck glass tubes under a nitrogen atmosphere. Concentration of solution was carried out by using a rotary evaporator and generally followed by removal of residual solvents on a vacuum line held at 0.1–1 torr. Products were purified by chromatography on silica gel BW-300 (Fuji Silysia Chemical Ltd.). Commercial reagents and solvents were purchased from Sigma Aldrich, TCI, FUJIFILM Wako Chemicals, nacalai tesque, Iharanikkei Chemical Industry and Kanto Chemical and used as received with the following exceptions: acetonitrile was used from a solvent purification system. Analytical thin-layer chromatography (TLC) was performed on pre-coated silica gel glass plates (Merck silica gel 60 F_{254}, 0.25 mm thickness). Compounds were visualized with UV lamp or treatment with an ethanolic solution of phosphomolybdic acid followed by heating or exposure to an iodine atmosphere. ^{1}H and ^{13}C NMR spectra were recorded on a JEOL JMTC-400/54/SS spectrometer (^{1}H NMR, 400 MHz; ^{13}C NMR, 100 MHz). ^{1}H NMR chemical shifts for $CDCl_3$ were determined relative to Me_4Si (0.0 ppm) as an internal standard. Infrared spectra were recorded on a SHIMADZU IRAffinity-1 FT-IR Spectrometer. High-resolution mass spectra were obtained on a JEOL JMS-DX303HF mass spectrometer. Melting points were determined on a Stanford Research Systems MPA100 OptiMelt Automated Melting Point System. Chiral-phase high-performance liquid chromatography (HPLC) was performed on a SHIMADZU prominence series instruments equipped with chiral columns.

2. Preparation of *tert*-butyl *N*-chloro-*N*-sodio-carbamate

Sodium hypochlorite pentahydrate (493.5 mg, 3 mmol) was added to a stirring solution of *tert*-butyl carbamate (351.5 mg, 3 mmol) at room temperature under a nitrogen atmosphere in MeCN (10 mL). The reaction mixture was stirred at room temperature for 3 h. After stirring, acetonitrile was removed from the resulting suspension by rotary evaporator. The residual solid was washed with Et_2O (40 mL). The resulting suspension was filtered by washing with ether and the solid was dried to give the pure product (466.7 mg; 90%).



3. Preparation of *electron deficient olefins*

Acrylates **1a**, **1e** and **1f** were prepared according to the literature procedures.[1] α,β-Unsaturated carbonyl compounds **1g**,[2] **1h**,[3] **1i**,[4] **1j**[5] and **1l** [6]were prepared by the known procedures.

4. Preparation of *chiral ammonium catalysts*

Ammonium catalysts **CN1** and **CD1** were synthesized by the literature procedures.[6,7]

5. Typical procudure for aziridination of acrylates

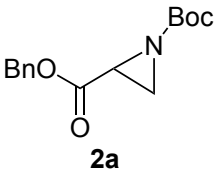
Potassium carbonate was ground in a mortar and then dried under reduced pressure at 200 ℃ prior to use. A heat-gun-dried 10 mL reaction flask containing a magnetic stir bar was charged with K_2CO_3 (4.5 mmol), $BnEt_3N^+Cl^-$ (0.025 mmol), *tert*-butyl carbamate (0.7 mmol), acrylate (0.5 mmol), and MeCN (2 mL). To the mixture, $NaOCl \cdot 5H_2O$ (0.7 mmol) was added, and the mixture was stirred at room temperature for the indicated time on the benchtop. The reaction mixture was quenched by $Na_2S_2O_3$ aq. (1 M, 10 mL), and extracted with CH_2Cl_2 (3 x 30 mL). Collected organic layers were dried over anhydrous Na_2SO_4. The solution was concentrated under reduced pressure to give the crude product. Purification by flash column chromatography on silica gel gave the pure product.

6. Typical procudure for aziridination of electron-deficient olefins

Potassium carbonate was ground in a mortar and then dried under reduced pressure at 200 ℃ prior to use. A heat-gun-dried 10 mL reaction flask containing a magnetic stir bar was charged with K_2CO_3 (4.5 mmol), $BnEt_3N^+Cl^-$ (0.025 mmol), *tert*-butyl carbamate (0.7 mmol), and MeCN (2 mL). To the mixture, $NaOCl \cdot 5H_2O$ (0.7 mmol) was added, and the mixture was stirred at room temperature. After stirring for 3 hours, electron-deficient olefin was added to the reaction mixture, followed by stirring at room temperature for the indicated time. The reaction mixture was quenched by $Na_2S_2O_3$ aq. (1 M, 10 mL), and extracted with CH_2Cl_2 (3 x 30 mL). Collected organic layers were dried over anhydrous Na_2SO_4. The solution was concentrated under reduced pressure to give the crude product. Purification by flash column chromatography on silica gel gave the pure product.

7. Product data

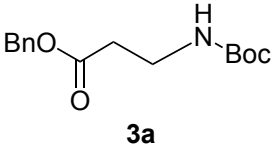
2-Benzyloxycarbonyl-1-*tert*-butoxycarbonylaziridine (**2a**).



2a

113.6 mg (82%); Colorless oil; R_f = 0.30 (hexane/EtOAc, 8:2, v/v, silica gel plate); HPLC (Daicel Chiralcel IA, hexane/2-propanol, 99:1, 0.2 mL/min, 254 nm, 30 °C) t = 56.8 and 61.7 min; IR (neat) 2980, 1744, 1724, 1152 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.43 (s, 9H), 2.40 (dd, 1H, J = 1.5, 5.4 Hz), 2.54 (dd, 1H, J = 1.5, 3.5 Hz), 3.07 (dd, 1H, J = 3.5, 5.4 Hz), 5.16 and 5.24 (1H and 1H, AB system, J =12.4 Hz), 7.34-7.38 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) 27.7, 31.3, 34.8, 67.3, 82.0, 128.3, 128.4, 128.5, 135.0, 159.4, 168.3; MS (FAB) m/z 278 ($[M+H]^+$, 20), 222 (34), 178 (80), 107 (5), 91 (100), 57 (60); HRMS (FAB) calcd for ($C_{15}H_{20}NO_4$) 278.1387 ($[M + H]^+$), found m/z 278.1391.

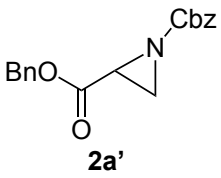
Benzyl 3-(*tert*-butoxycarbonylamino)propionate (3a).



3a

Colorless oil; R_f = 0.23 (hexane/EtOAc, 8:2, v/v, silica gel plate); IR (neat) 3375, 2978, 1732, 1713, 1163 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.43 (s, 9H), 2.57 (t, 2H, J = 6.0 Hz), 3.40 (q, 2H, J = 6.0 Hz), 5.12 (s, 3H, overlap with NH), 7.30-7.41 (m, 5 H); ^{13}C NMR (100 MHz, $CDCl_3$) 28.2, 34.4, 35.9, 66.3, 79.1, 128.0, 128.2, 128.4, 135.5, 155.6, 172.1; MS (CI) m/z 280 ($[M^+ + H]$, 3), 224 (100), 180 (33), 91 (9); HRMS (CI) calcd for ($C_{15}H_{22}NO_4$) 280.1543 ($[M+H]^+$), found m/z 280.1553.

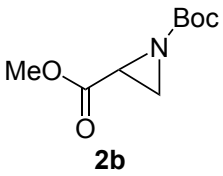
1,2-Bis(benzyloxycarbonyl)aziridine (2a').



2a'

51.6 mg (32%); Colorless oil; R_f = 0.40 (hexane/EtOAc, 7:3, v/v, silica gel plate); IR (neat) 3034, 1728, 1215, 1173 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.48 (dd, 1H, J = 1.3, 5.4 Hz), 2.62 (dd, 1H, J = 1.3, 3.1 Hz), 3.14 (dd, 1H, J = 3.1, 5.4 Hz), 5.06-5.17 (m, 4H), 7.32-7.39 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) 31.5, 34.9, 67.6, 68.6, 128.4, 128.5, 128.5, 128.5, 128.6, 128.7, 134.8, 135.3, 160.7, 168.1; MS (FAB) m/z 312 ($[M+H]^+$, 16), 178 (9), 107 (10), 91 (100), 77 (6); HRMS (FAB) calcd for ($C_{18}H_{18}NO_4$) 312.1230 ($[M+H]^+$), found m/z 312.1241.

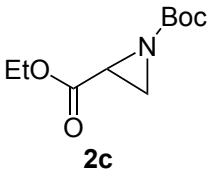
1-*tert*-Butoxycarbonyl-2-methoxycarbonylaziridine (2b).



2b

The analytical data for this compound were in excellent agreement with the reported data [8,9]. 67.7 mg (60%); Colorless oil; HPLC (Daicel Chiralcel IA, hexane/2-propanol, 95:5, 0.3 mL/min, RI detector, 30 °C) t = 17.7 and 19.3 min; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.46 (s, 9H), 2.42 (dd, 1H, J = 1.4, 5.4 Hz), 2.53 (dd, 1H, J = 1.4, 3.4 Hz), 3.04 (dd, 1H, J = 3.4, 5.4 Hz), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.8, 31.3, 34.8, 52.6, 82.1, 159.5, 168.9.

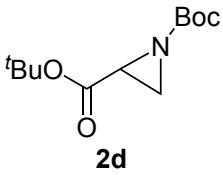
2-Ethoxycarbonyl-1-*tert*-butoxycarbonylaziridine (2c).



2c

60.1 mg (55%); Colorless oil; R_f = 0.38 (hexane/EtOAc, 8:2, v/v, silica gel plate); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.31 (t, 3H, J = 6.8 Hz), 1.46 (s, 9H), 2.40 (dd, 1H, J = 1.3, 5.3 Hz), 2.52 (dd, 1H, J = 1.3, 3.0 Hz), 3.02 (dd, 1H, J = 3.0, 5.3 Hz), 4.16-4.32 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 27.9, 31.2, 34.9, 61.6, 82.0, 159.5, 168.4; MS (FAB) m/z 216 ([M+H]$^+$, 16), 160 (34), 57 (100); HRMS (FAB) calcd for ($C_{10}H_{18}NO_4$) 216.1230 ([M+H]$^+$), found m/z 216.1238.

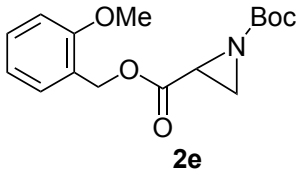
1,2-Bis(*tert*-butoxycarbonyl) aziridine (2d).



2d

42.2 mg (35%); Colorless oil; R_f = 0.29 (hexane/EtOAc, 8:2, v/v, silica gel plate); IR (neat) 2980, 1736, 1724, 1147 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.46 (s, 9H), 1.50 (s, 9H), 2.32 (dd, 1H, J = 1.6, 5.2 Hz), 2.47 (dd, 1H, J = 1.6, 3.0 Hz), 2.93 (dd, 1H, J = 3.0, 5.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.8, 27.9, 30.9, 35.7, 81.8, 82.4, 159.7, 167.4; MS (FAB) m/z 244 ([M+H]$^+$, 28), 188 (32), 57 (100); HRMS (FAB) calcd for ($C_{12}H_{22}NO_4$) 244.1543 ([M+H]$^+$), found m/z 244.1550.

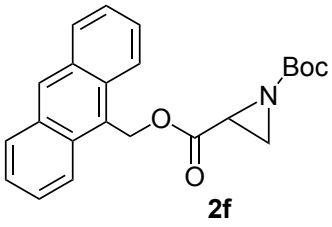
1-*tert*-Butoxycarbonyl-2-*o*-methoxybenzyloxycarbonylaziridine (2e).



2e

114.2 mg (75%); Colorless oil; R_f = 0.22 (hexane/EtOAc, 8:2, v/v, silica gel plate); IR (neat) 2978, 1742, 1724, 1248, 1152, 754 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.44 (s, 9H), 2.40 (dd, 1H, J = 1.1, 5.4 Hz), 2.54 (dd, 1H, J = 1.1, 3.3 Hz), 3.07 (dd, 1H, J = 3.3, 5.4 Hz), 3.84 (s, 3H), 5.23 and 5.29 (1H and 1H, AB system, J =12.2 Hz), 6.89-6.97 (m, 2H), 7.28-7.35 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) 27.7, 31.3, 34.8, 55.3, 62.8, 81.9, 110.4, 120.3, 123.3, 129.9, 130.0, 157.5, 159.5, 168.3; MS (FAB) m/z 328 ([M+Na]$^+$, 5), 308 ([M+H]$^+$, 4), 121 (100); HRMS (FAB) calcd for ($C_{16}H_{21}NO_5$) 307.1420 (M$^+$), found m/z 307.1414.

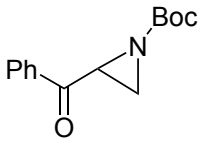
9[{1-*tert*-butoxycarbonylaziridin-2'-yl}carbonyloxymethyl]anthracene (2f).



2f

107.9 mg (57%); Pale yellow solid; mp 42.1–46.1 °C; R_f = 0.18 (hexane/EtOAc, 5:1, v/v, silica gel plate); HPLC (Daicel Chiralcel IB, hexane/2-propanol, 99:1, 1.0 mL/min, 254 nm, 30 °C) t = 19.8 and 22.7 min; IR (neat) 2976, 1732, 1304, 1192, 1157, 1144, 734 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.39 (s, 9H), 2.35 (dd, 1H, J = 1.6, 5.1 Hz), 2.54 (dd, 1H, J = 1.6, 3.4 Hz), 3.02 (dd, 1H, J = 3.4, 5.1 Hz), 6.24 and 6.29 (1H and 1H, AB system, J =12.6 Hz), 7.50 (t, 2H, J = 7.6 Hz), 7.59 (t, 2H, J = 7.8 Hz), 8.04 (d, 2H, J = 8.0 Hz), 8.35 (d, 2H, J = 8.8 Hz), 8.53 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) 27.8, 31.5, 34.9, 60.1, 82.1, 123.8, 125.2, 125.4, 126.8, 129.1, 129.5, 131.1, 131.4, 159.5, 168.7; MS (EI) *m*/*z* 377 (M^+, 20), 207 (20), 191 (100); HRMS (EI) calcd for ($C_{23}H_{23}NO_4$) 377.1627 (M^+), found *m*/*z* 377.1622.

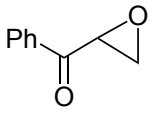
2-Benzoyl-1-*tert*-butoxycarbonylaziridine (2g).



2g

114.6 mg (87%); Colorless oil; R_f = 0.31 (hexane/EtOAc, 8:2, v/v, silica gel plate); IR (neat) 2978, 1722, 1682, 1227, 1153, 708 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.45 (s, 9H), 2.53 (dd, 1H, J = 1.5, 5.4 Hz), 2.70 (dd, 1H, J = 1.5, 3.3 Hz), 3.94 (dd, 1H, J = 3.3, 5.4 Hz), 7.52 (t, 2H, J = 7.4 Hz), 7.63 (t, 1H, J = 7.2 Hz), 8.09 (d, 2H, J = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) 27.8, 32.5, 37.3, 81.9, 128.5, 128.7, 133.7, 136.4, 160.2, 193.5; MS (FAB) *m*/*z* 248 ($[M+H]^+$, 39), 192 (70), 148 (100), 105 (53), 57 (70); HRMS (FAB) calcd for ($C_{14}H_{18}NO_3$) 248.1281 ($[M+H]^+$), found *m*/*z* 248.1288.

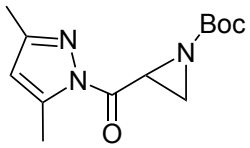
Oxirane-2-yl-(phenyl)-methanone (4g).



4g

Colorless solid; R_f = 0.25 (hexane/EtOAc, 8:2, v/v, silica gel plate); IR (neat) 1684 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.97 (dd, 1H, J = 2.7, 6.5 Hz), 3.13 (dd, 1H, J = 4.4, 6.5 Hz), 4.26 (dd, 1H, J = 2.7, 4.4 Hz), 7.51 (t, 2H, J = 7.8 Hz), 7.63 (t, 1H, J = 7.3 Hz), 8.04 (d, 2H, J = 7.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 47.5, 50.9, 128.2, 128.7, 133.9, 135.2, 194.6; MS (CI) *m*/*z* (relative intensity, %) 149 ($[M+H]^+$, 100), 133 (6), 105 (8); HRMS (CI) calcd for ($C_9H_9O_2$) 149.0597 ($[M+H]^+$), found *m*/*z* 149.0602.

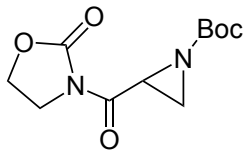
1-[{(1'-*tert*-Butoxycarbonyl)aziridin-2'-yl}carbonyl]-3,5-dimethylpyrazole (2h).



2h

113.1 mg (85%); Colorless oil; R_f = 0.39 (hexane/EtOAc, 8:2, v/v, silica gel plate); HPLC (Daicel Chiralcel OB-H, hexane/2-propanol, 99:1, 0.3 mL/min, 254 nm, 30 °C) t = 29.1 and 49.1 min; IR (neat) 2978, 1728, 1337, 1153 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.45 (s, 9H), 2.28 (s, 3H), 2.55 (s, 3H), 2.56-2.58 (m, 1H), 2.62 (dd, 1H, J = 1.4, 2.9 Hz), 4.53 (dd, 1H, J =2.9, 5.6 Hz), 6.04 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) 13.8, 14.3, 27.9, 32.6, 34.7, 82.0, 111.8, 144.5, 153.2, 160.0, 167.4; MS (EI) m/z 265 (M^+, 0.7), 164 (34), 57 (100); HRMS (EI) calcd for ($C_{13}H_{19}N_3O_3$) 265.1426 (M^+), found m/z 265.1427.

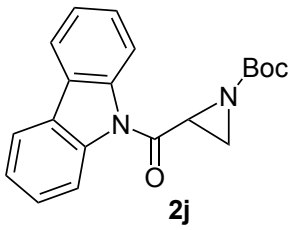
3-[{(1'-*tert*–butoxycarbonyl)aziridin-2'-yl}carbonyl]-2-oxazolidinone (2i).



2i

124.0 mg (96%); Colorless oil; R_f = 0.33 (hexane/EtOAc, 1:1, v/v, silica gel plate); IR (neat) 2982, 1778, 1721, 1703, 1219, 1153 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.46 (s, 9H), 2.53-2.56 (m, 2H), 4.02-4.13 (m, 2H), 4.45 (dd, 1H, J = 3.4, 5.8 Hz), 4.50 (t, 2H, J = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) 27.8, 32.6, 33.9, 42.7, 62.5, 82.1, 153.4. 159.7, 167.7 MS (FAB) m/z 257 ($[M+H]^+$, 10), 201 (100), 157 (65), 57(^{t}Bu, 58); HRMS (FAB) calcd for ($C_{11}H_{17}N_2O_5$) 257.1132 ($[M+H]^+$)$^+$, found m/z 257.1134.

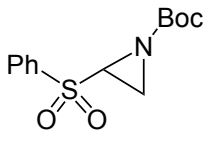
1-[{(1'-*tert*-Butoxycarbonyl)aziridin-2'-yl}carbonyl]carbazole (2j).



2j

125.2 mg (76%); Colorless solid; mp 106.1–113.2 °C; R_f = 0.21 (hexane/EtOAc, 9:1, v/v, silica gel plate); HPLC (Daicel Chiralcel IB, hexane/2-propanol, 9:1, 1.0 mL/min, 254 nm, 30 °C) t = 7.0 and 9.9 min.; IR (neat) 2974, 1730, 1680, 1294, 1144, 754 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.35 (s, 9H), 2.63 (d, 1H, J = 4.7 Hz), 3.13 (d, 1H, J = 3.4 Hz), 3.93 (dd, 1H, J =3.4, 4.7 Hz), 7.42 (t, 2H, J = 7.2 Hz), 7.50 (t, 2H, J = 7.8 Hz), 8.01 (d, 2H, J = 8.4 Hz), 8.30 (d, 2H, J = 8.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) 27.8, 32.2, 38.1, 82.6, 115.8, 120.0, 124.0, 126.3, 127.3, 138.3, 159.7, 166.9; MS (FAB) m/z 336 (M^+, 42), 281 (43), 237 (50), 167 (100), 57 (89); HRMS (FAB) calcd for ($C_{20}H_{20}N_2O_3$) 336.1474 (M^+), found m/z 336.1475.

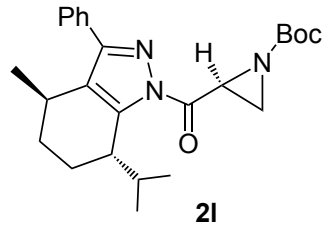
1-*tert*-Butoxycarbonyl-2-phenylsulfonylaziridine (2k).



2k

95.5 mg (66%); Colorless solid; mp 116.1–119.7 °C; R_f = 0.30 (hexane/EtOAc, 7:3, v/v, silica gel plate); IR (neat) 3026, 1726, 1287, 1146 cm^{-1};^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.26 (s, 9H), 2.68 (d, 1H, J = 5.8 Hz), 2.89 (d, 1H, J = 2.8 Hz), 3.73 (dd, 1H, J = 2.8, 5.8 Hz), 7.60 (t, 2H, J = 7.8 Hz), 7.70 (t, 1H, J = 7.4 Hz), 7.98 (d, 2H, J = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.4, 30.0, 50.9, 82.9, 128.7, 129.0, 134.1, 137.4, 158.4; MS (FAB) *m*/*z* 284 ([M+H]$^+$, 12), 228 (30), 184 (100) 154 (38), 57 (90); HRMS (FAB) calcd for $C_{13}H_{18}NO_4S$ 284.0951 ([M+H]$^+$), found *m*/*z* 284.0955.

(4*R*,7*S*)-1-[{(2'*S*)-(1'-*tert*-Butoxycarbonyl)aziridin-2'-yl}carbonyl]-4-methyl-3-phenyl-7-propyl-4,5,6,7-tetrahydroindazole (2l).



2l

The analytical data for this compound were in excellent agreement with the reported data.[6] 144.5 mg (66%); Colorless oil; R_f = 0.44 (hexane/EtOAc, 7:3, v/v, silica gel plate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89-0.97 (m, 9H), 1.46 (s, 10H overlap with 1H), 1.76-1.85 (m, 2H), 2.04-2.16 (m, 2H), 2.56 (dd, 1H, J = 1.6, 5.7 Hz), 2.68 (dd, 1H, J = 1.6, 3.2 Hz), 3.20-3.26 (m, 1H), 3.36-3.39 (m, 1H), 4.67 (dd, 1H, J = 3.2, 5.7 Hz), 7.39-7.47 (m, 3H), 7.81-7.84 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.5, 19.7, 20.6, 20.9, 25.6, 26.8, 27.8, 31.4, 32.3, 35.0, 37.7, 81.8, 125.2, 127.5, 128.5, 128.8, 132.7, 146.2, 153.5, 160.0, 167.0.

8. References

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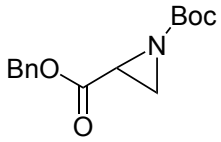
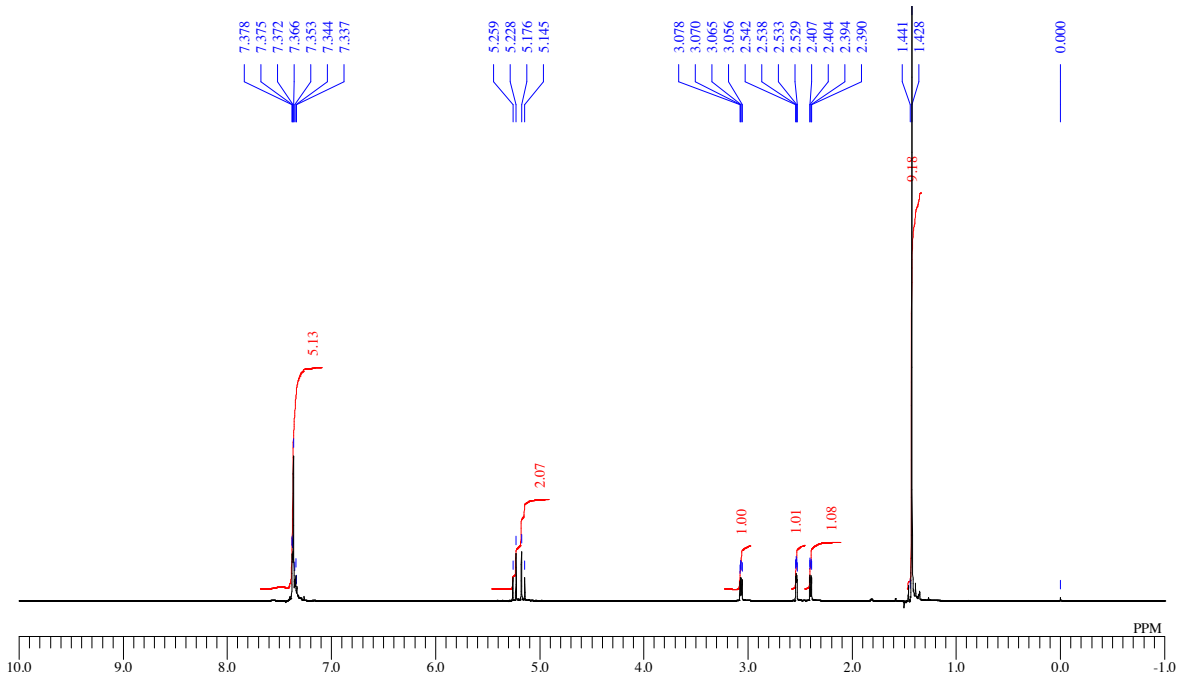
9. NMR & HPLC data

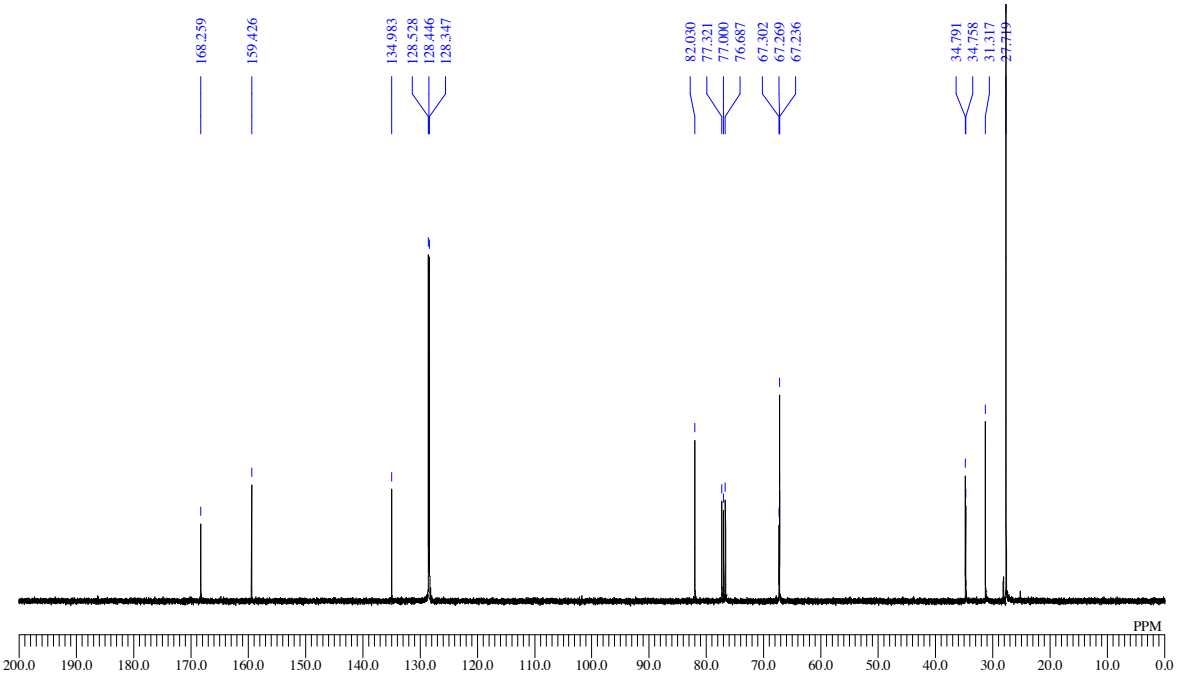


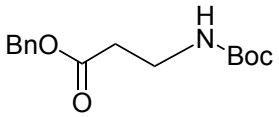
2a

^{1}H NMR (400 MHz, $CDCl_3$)



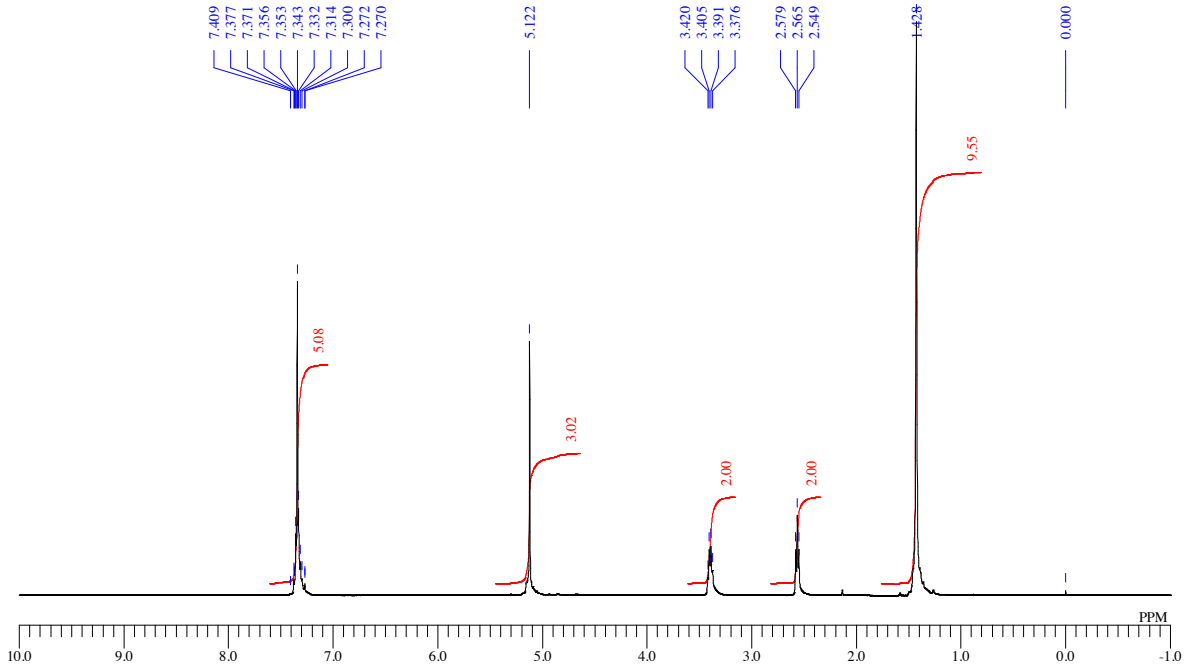
^{13}C NMR (100 MHz, $CDCl_3$)





3a

^{1}H NMR (400 MHz, $CDCl_3$)



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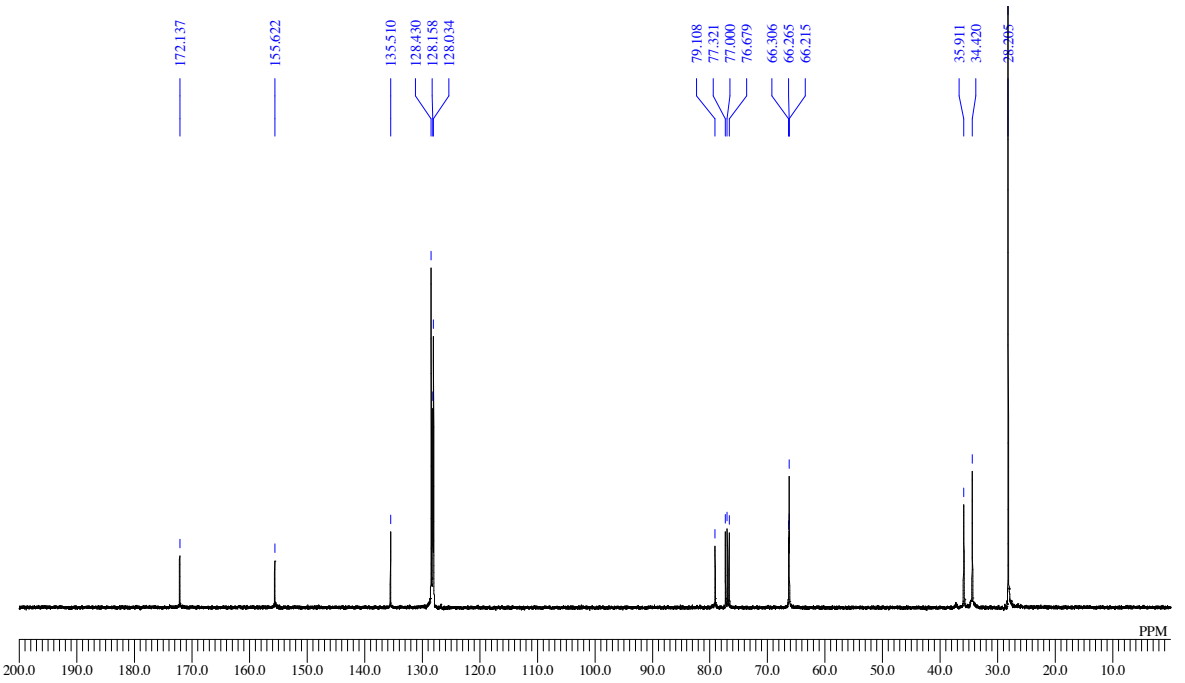
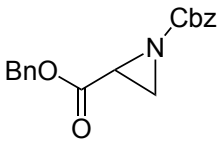
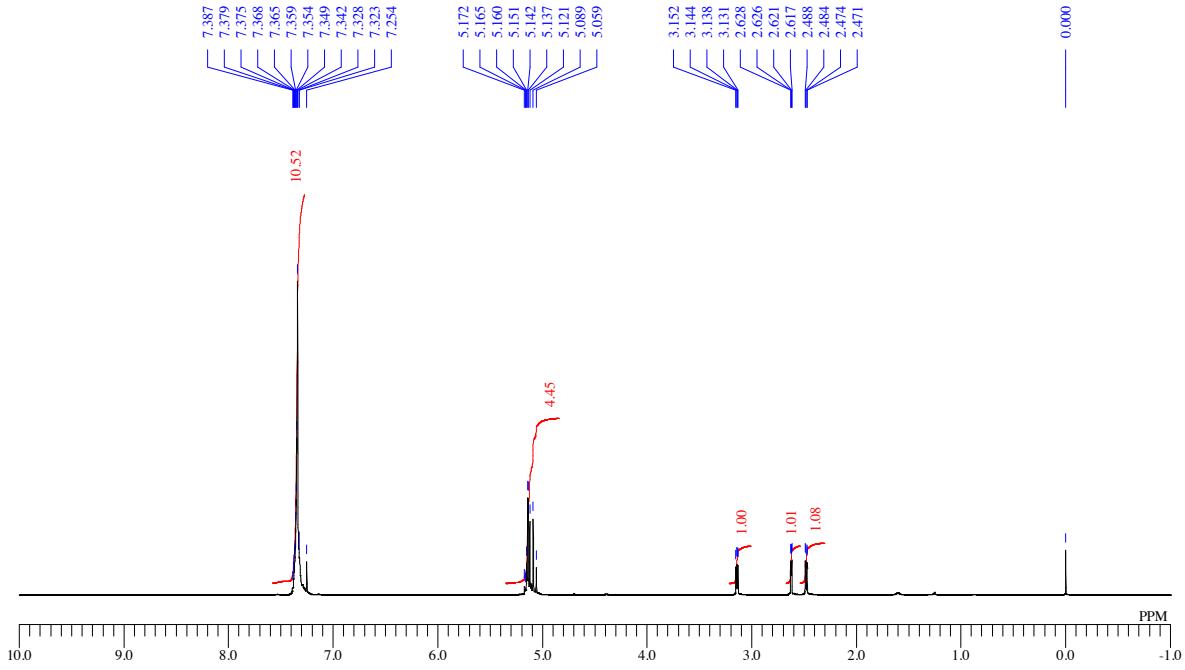


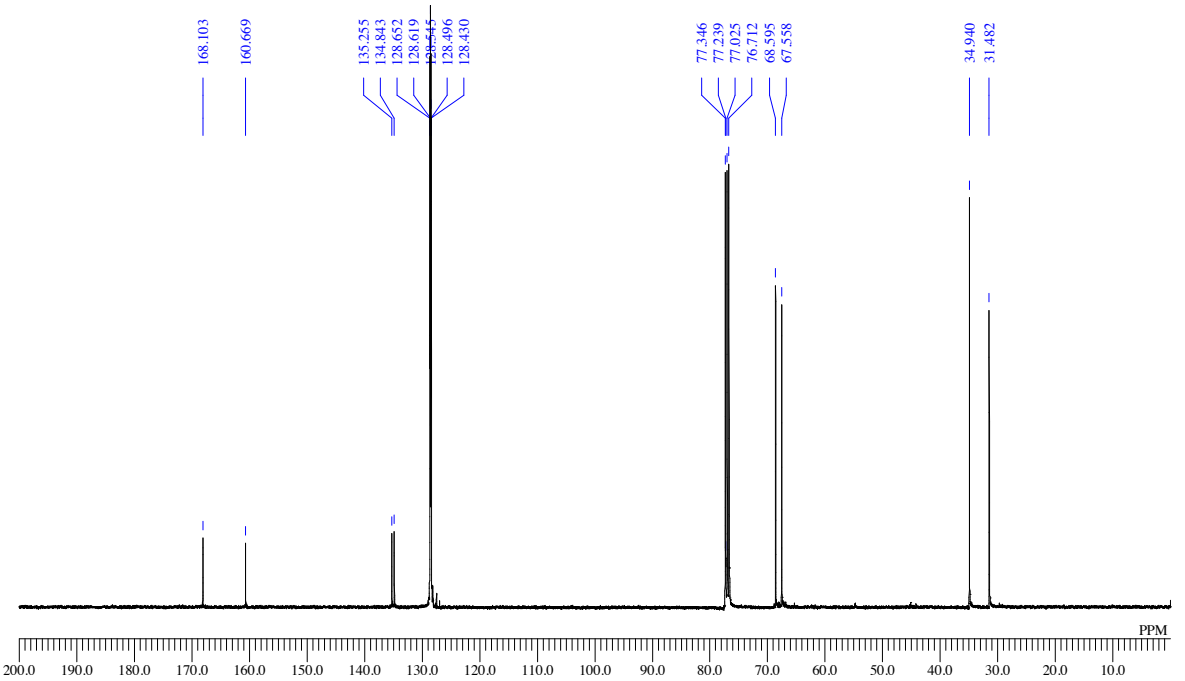


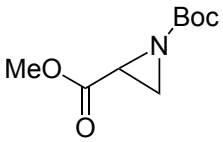
2a'

^{1}H NMR (400 MHz, $CDCl_3$)



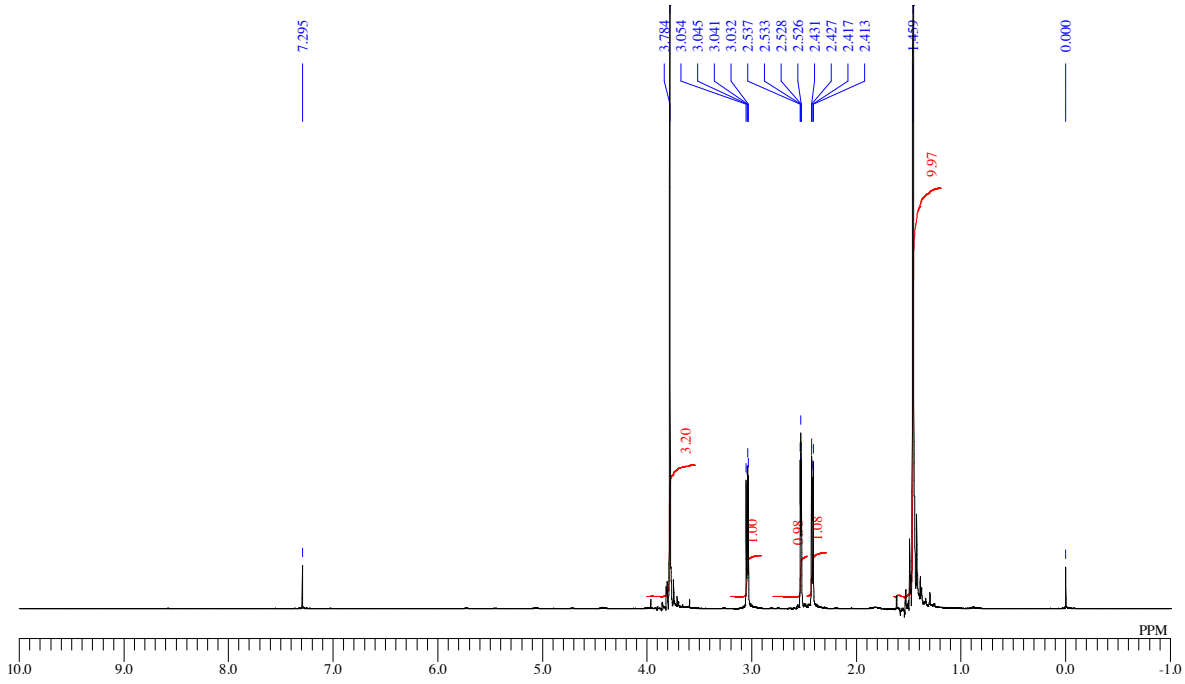
^{13}C NMR (100 MHz, $CDCl_3$)



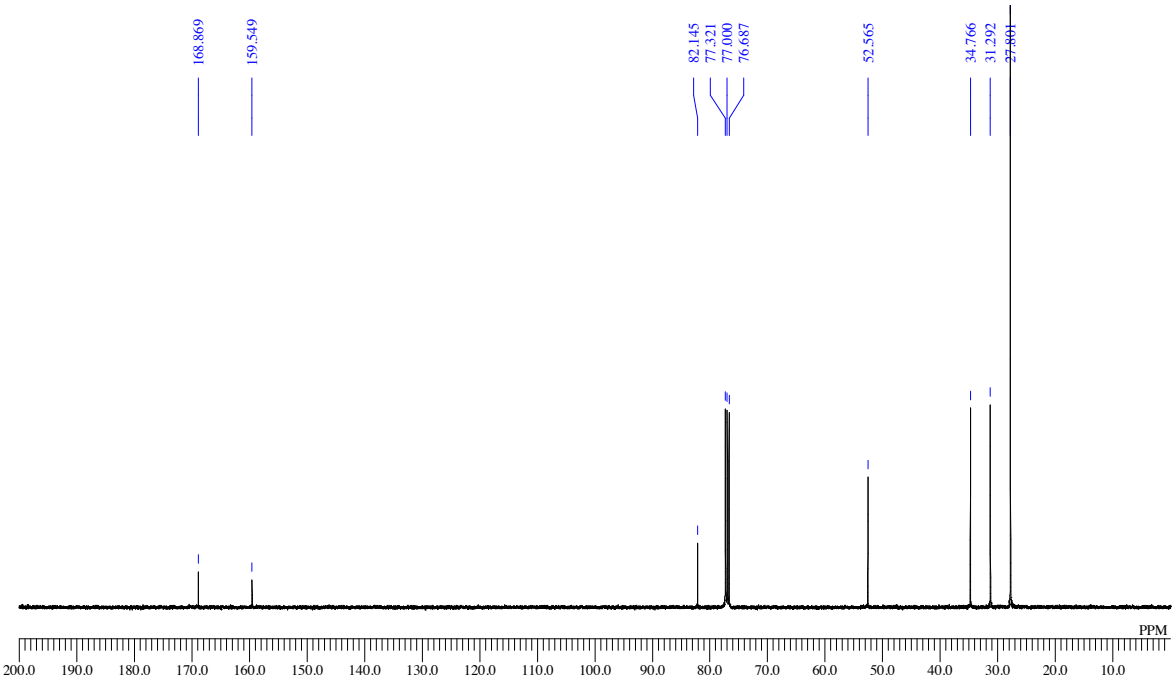


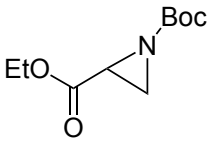
2b

^{1}H NMR (400 MHz, $CDCl_3$)



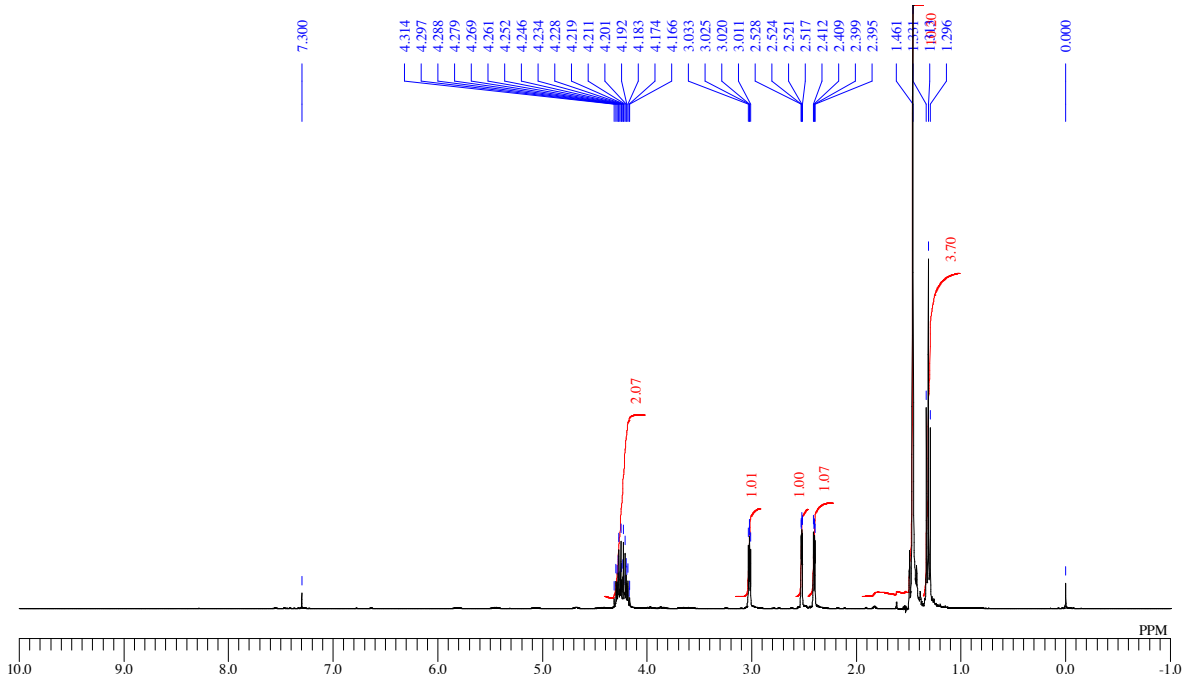
^{13}C NMR (100 MHz, $CDCl_3$)



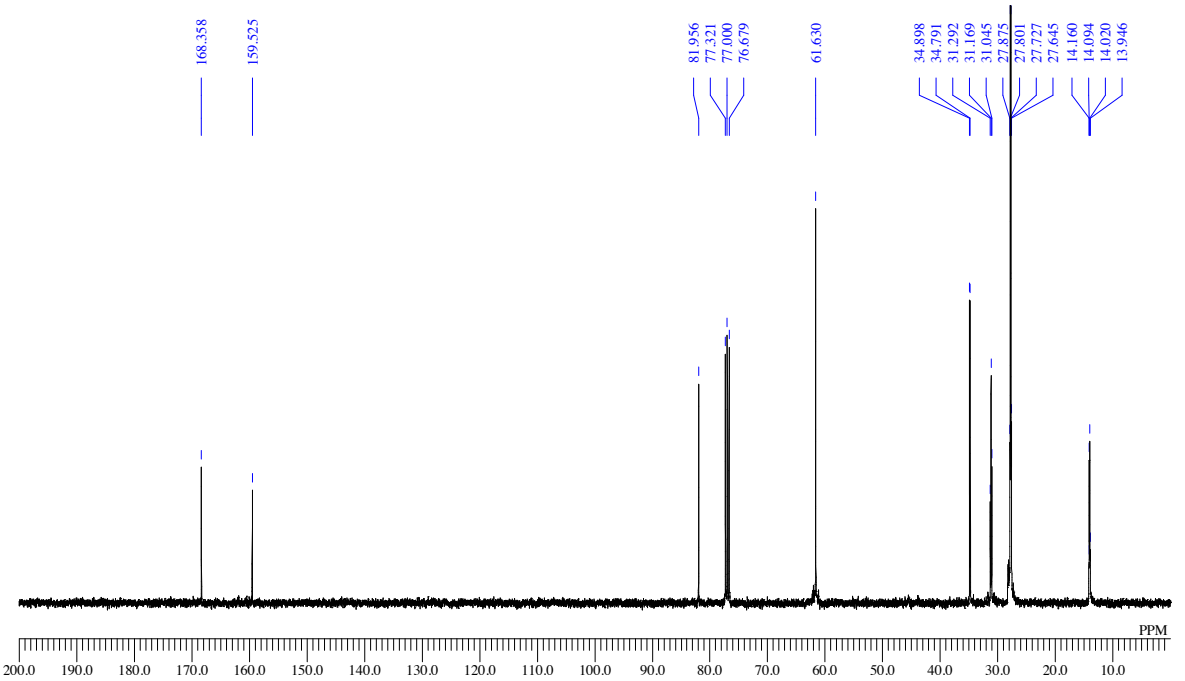


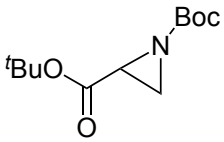
2c

^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)





2d

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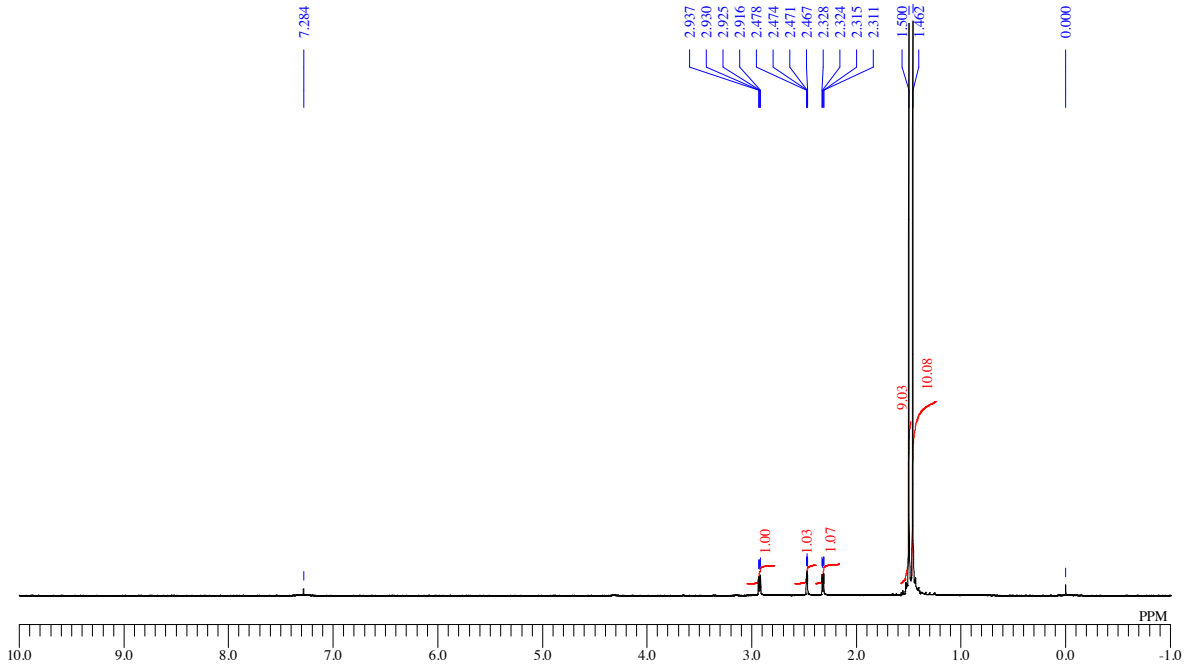
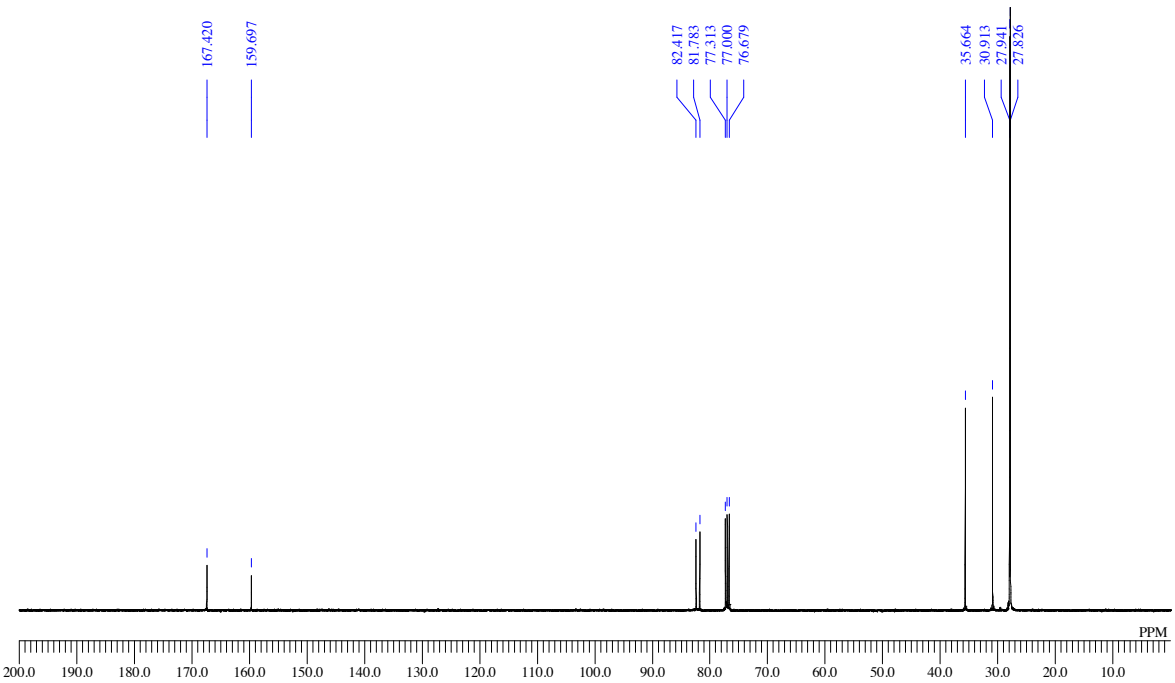
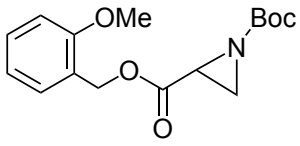


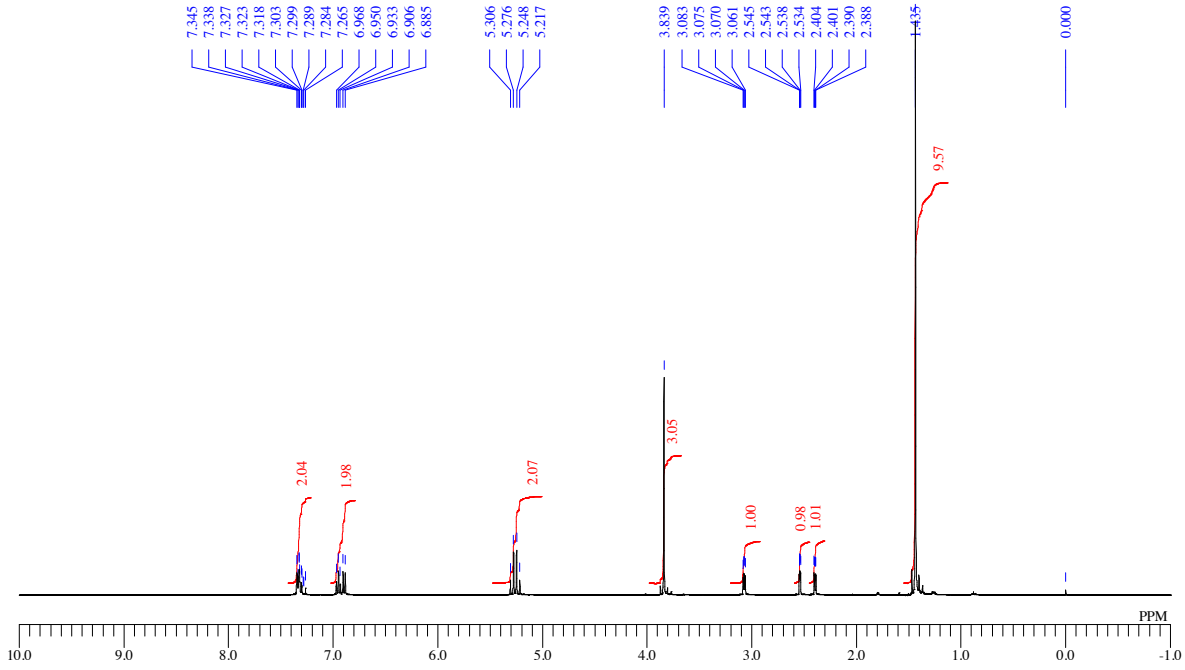
^{13}C NMR (100 MHz, $CDCl_3$)





2e

^{1}H NMR (400 MHz, $CDCl_3$)



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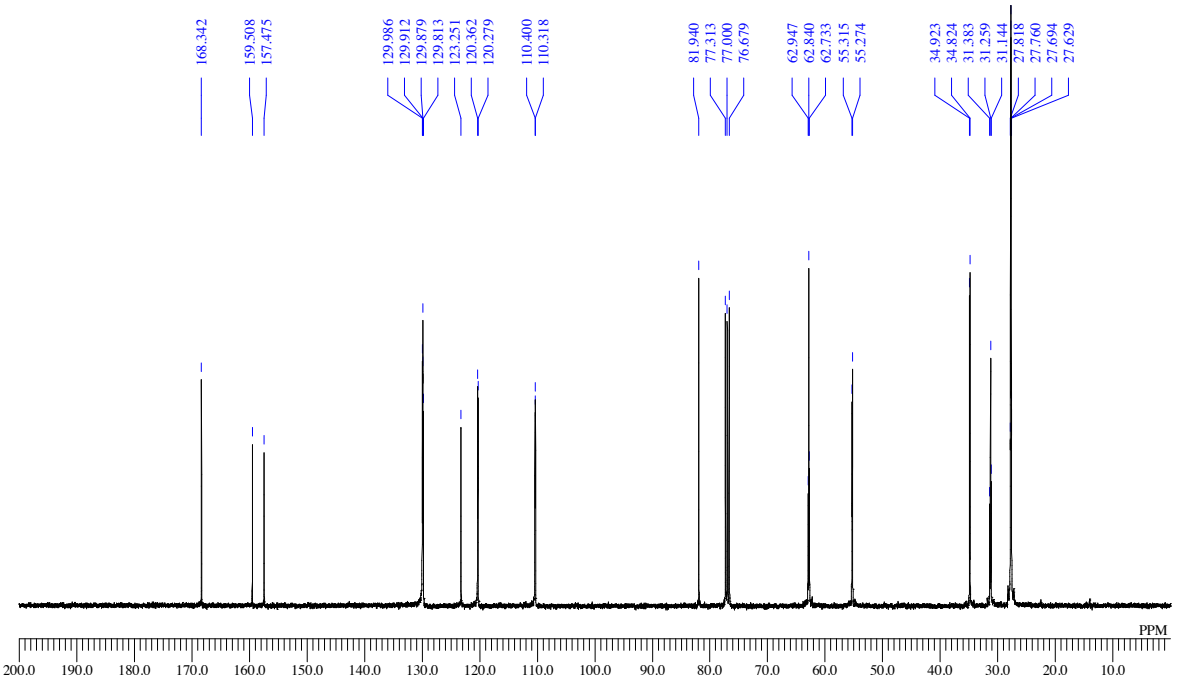

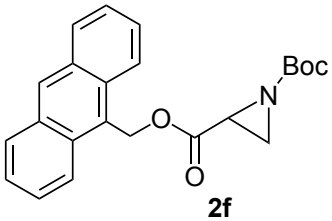

2f

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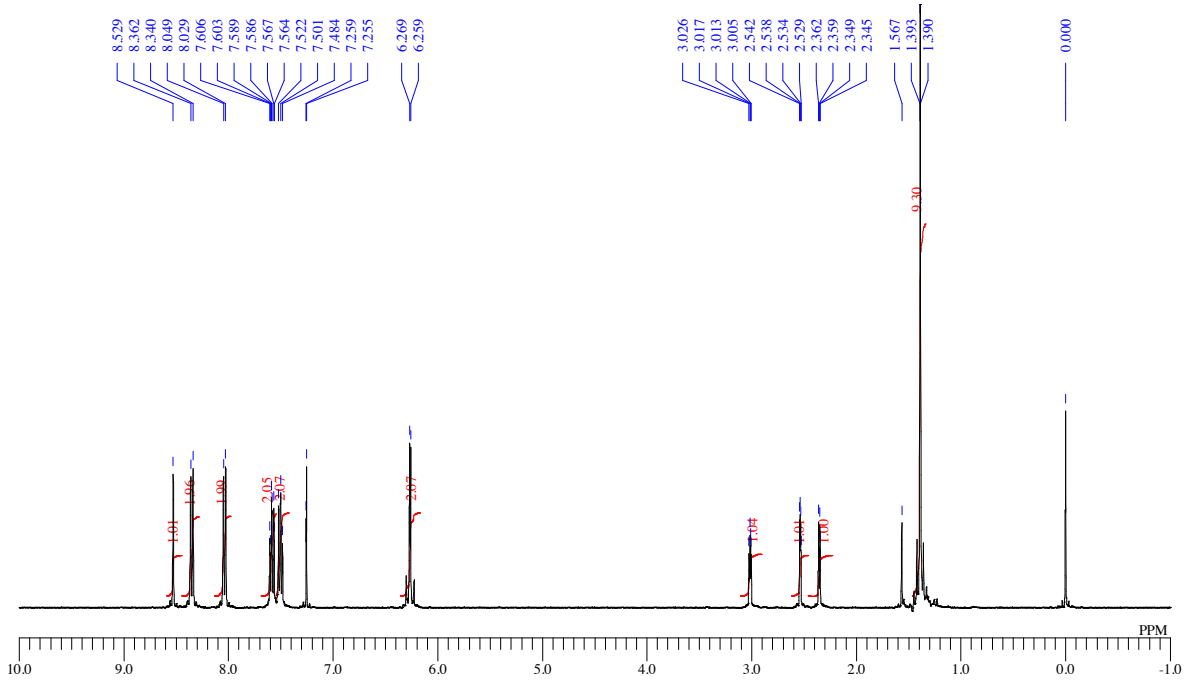

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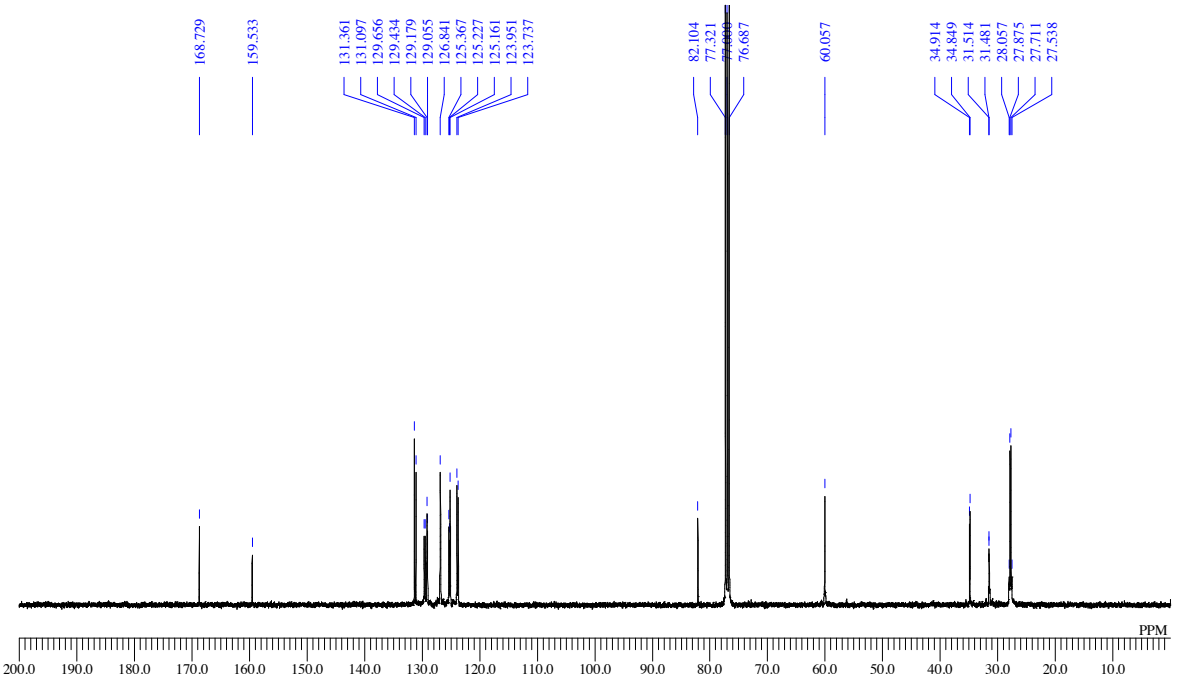
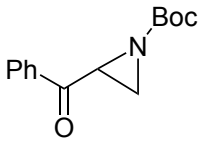
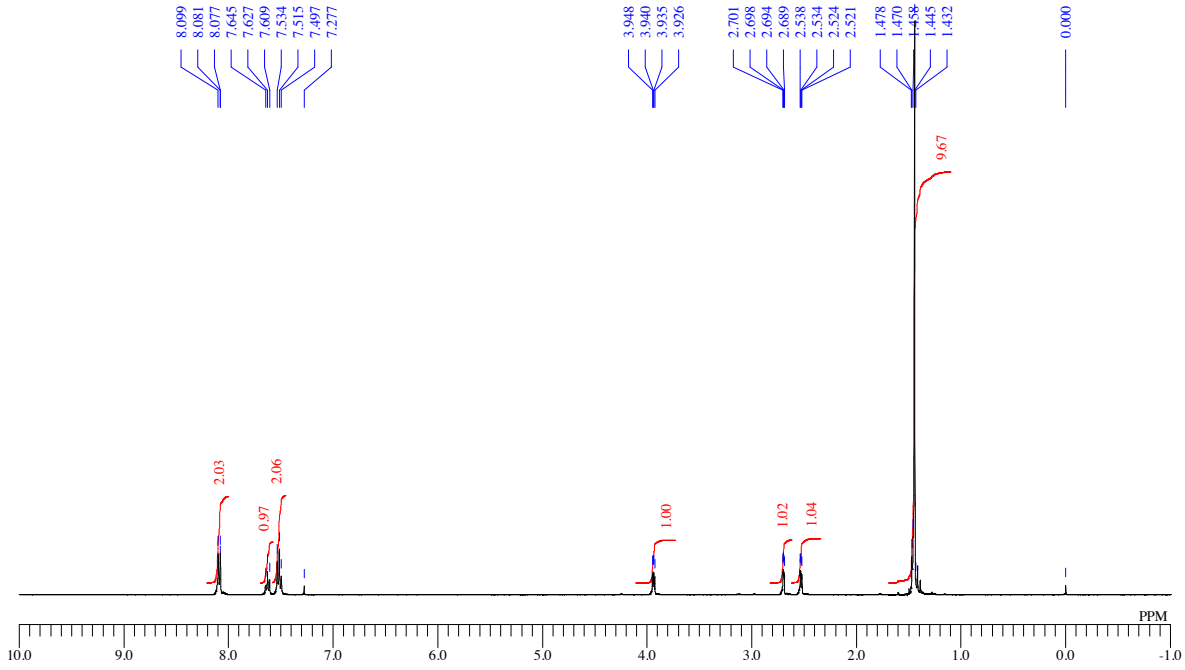




2g

^{1}H NMR (400 MHz, $CDCl_3$)



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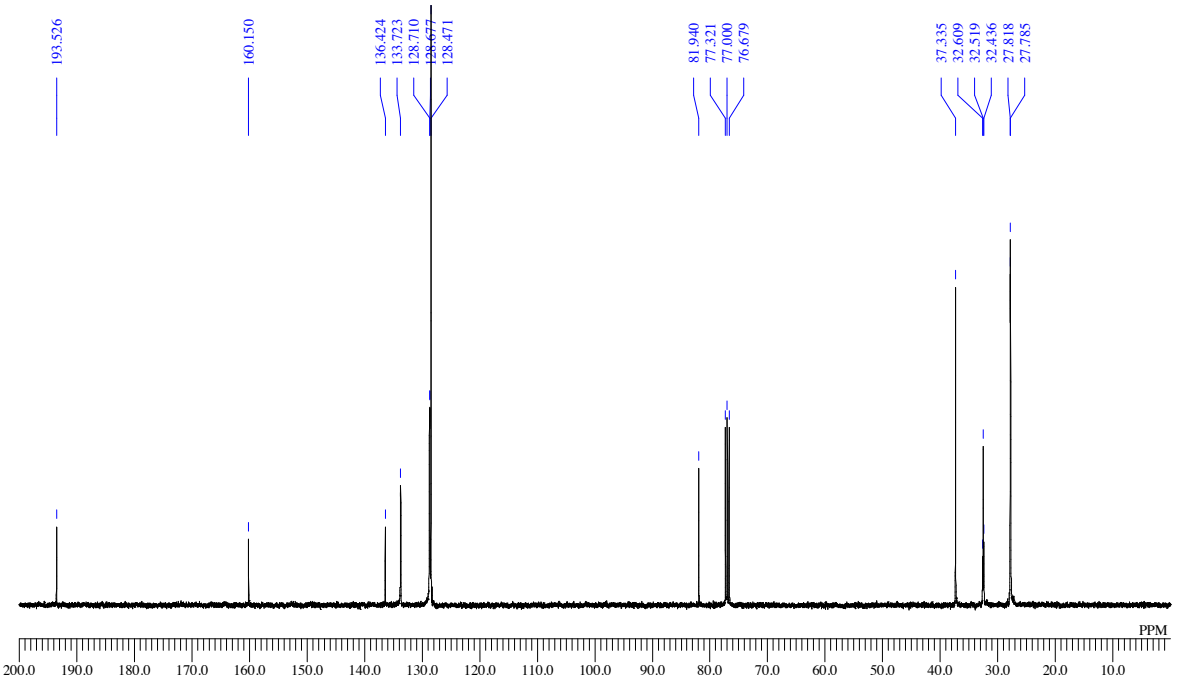

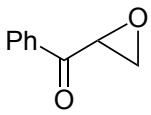

4g

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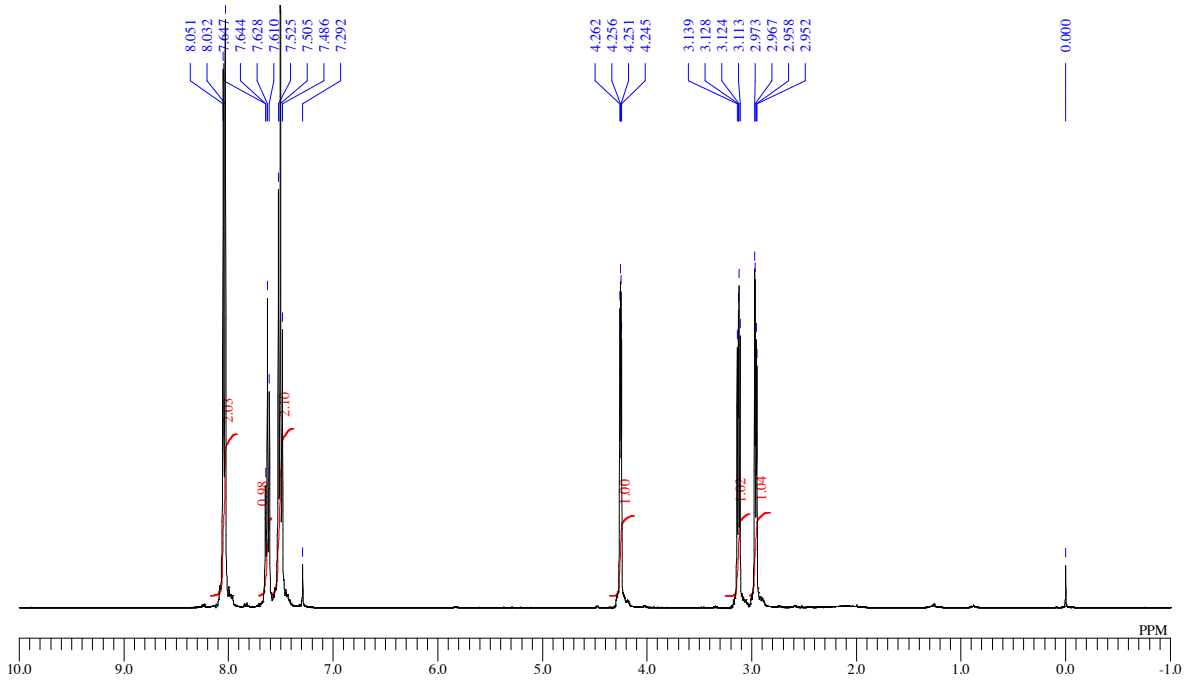

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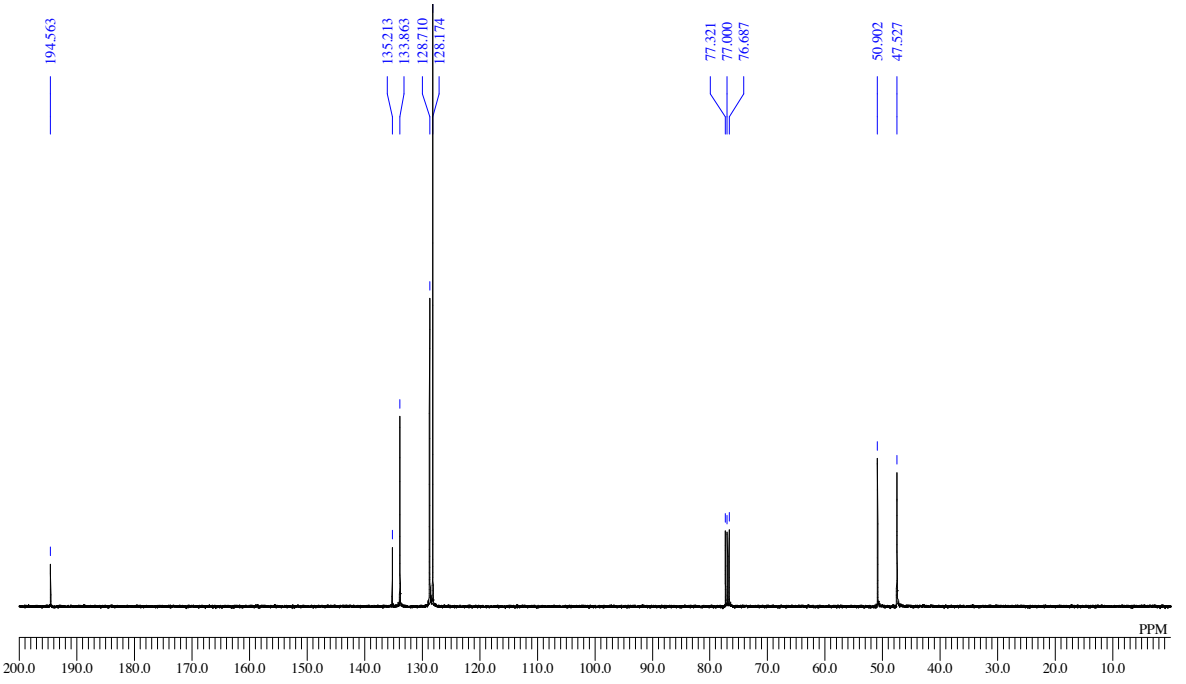
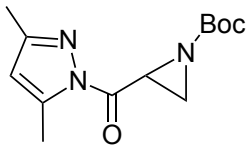
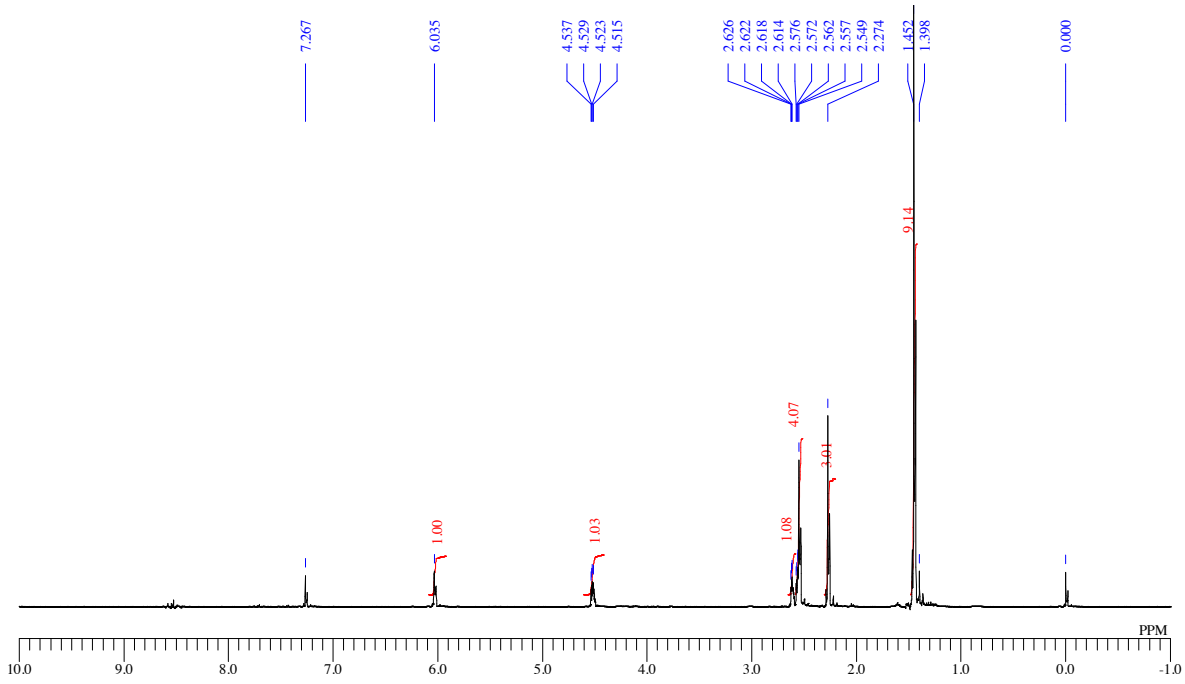


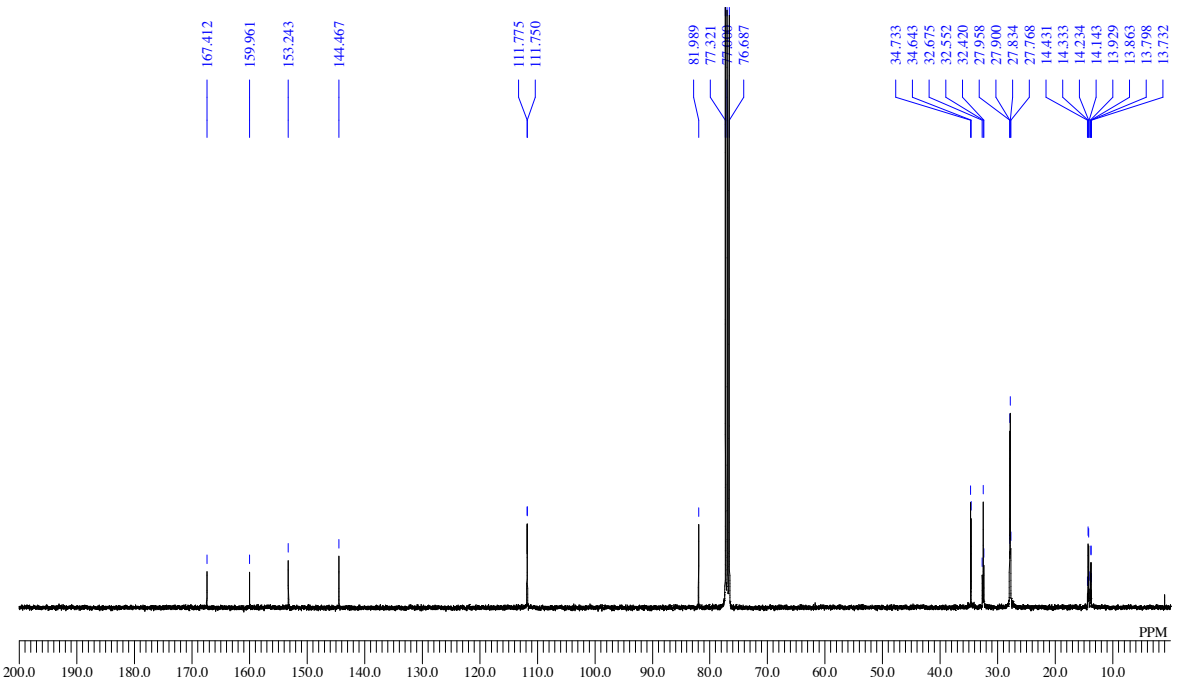


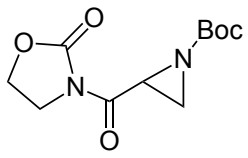
2h

^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)





2i

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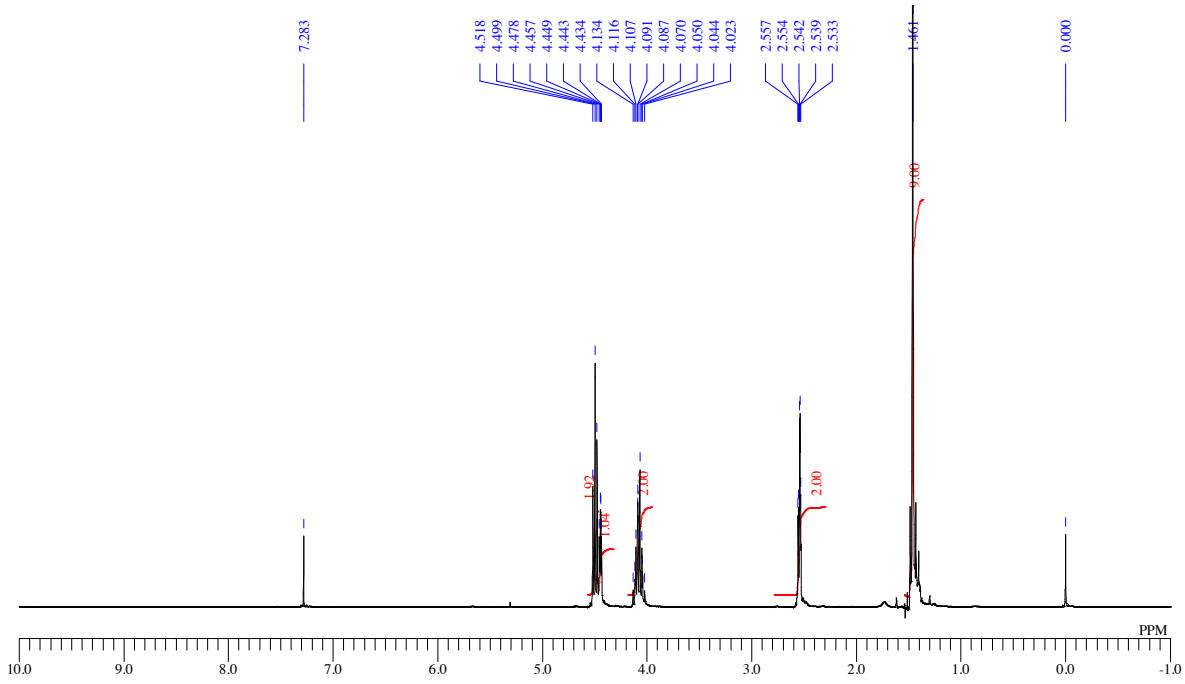
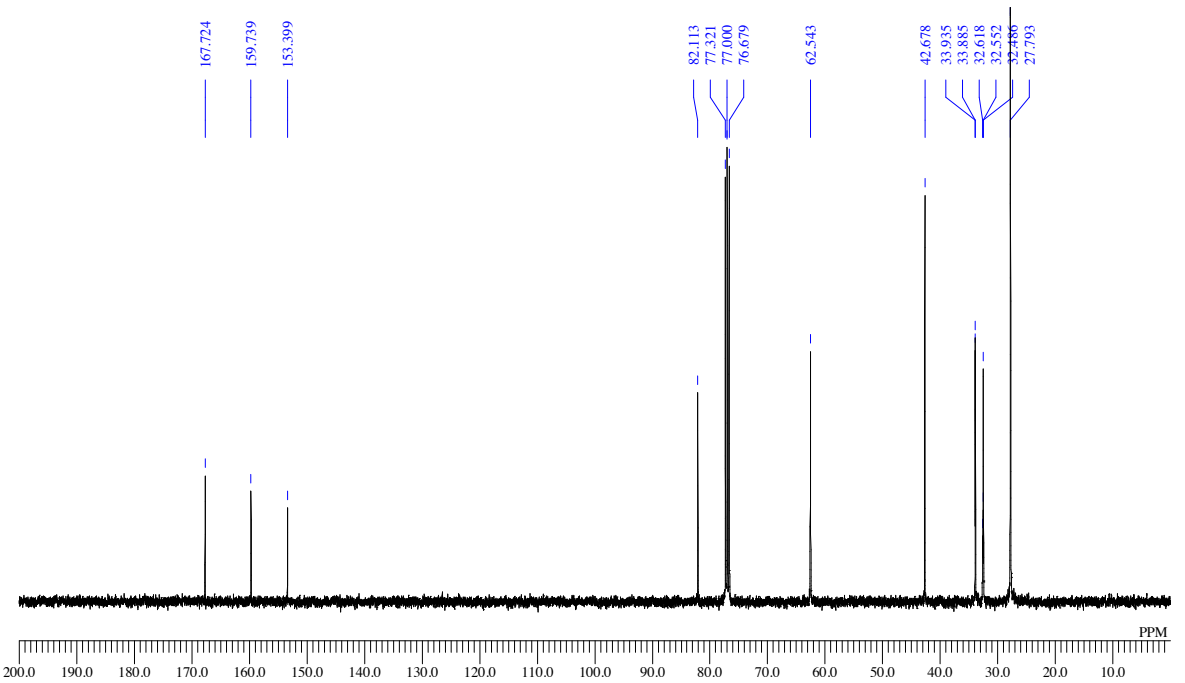
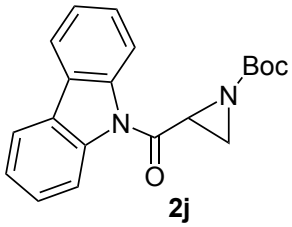


^{13}C NMR (100 MHz, $CDCl_3$)





2j

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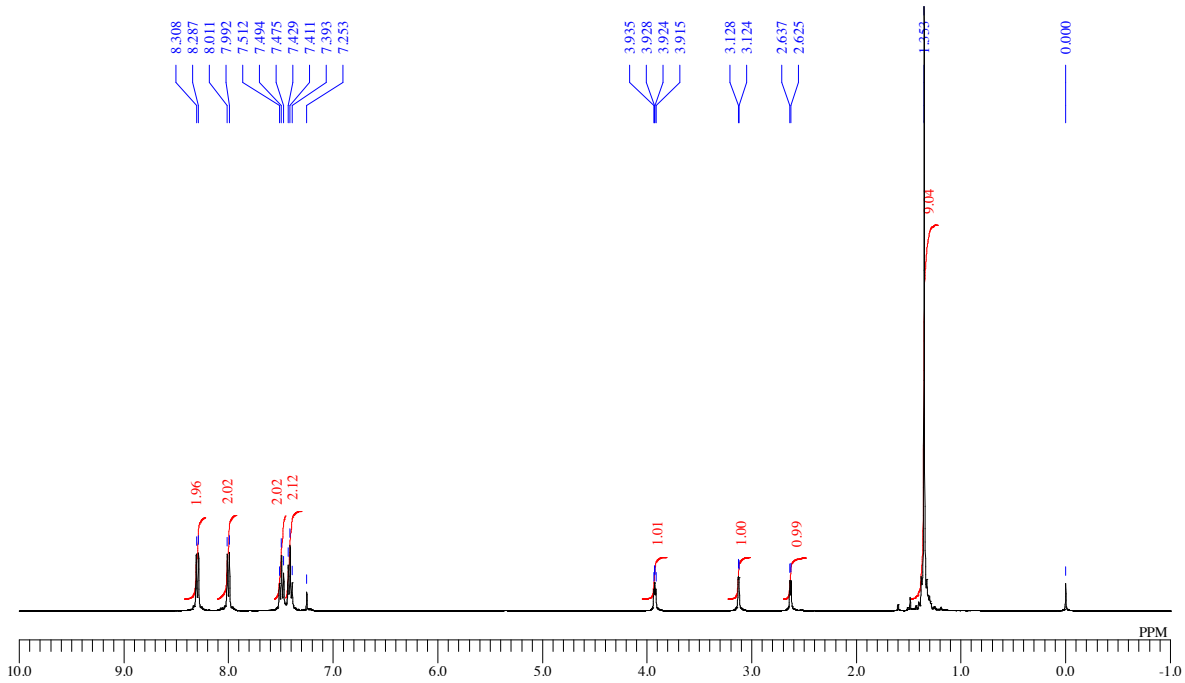
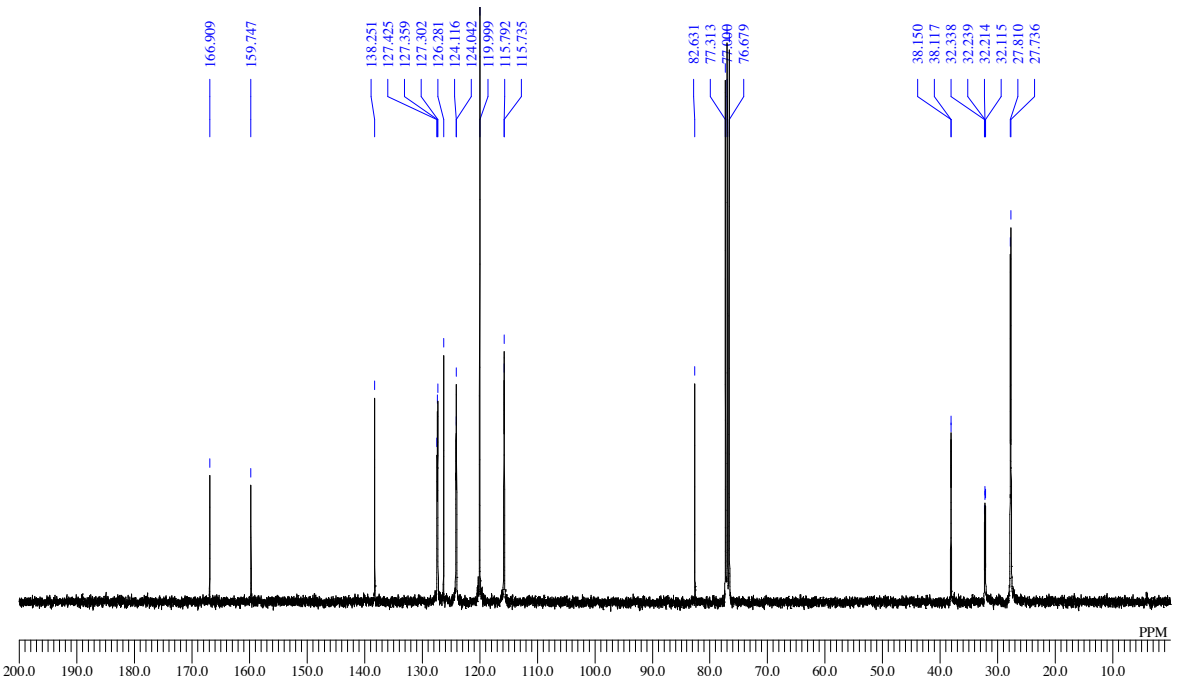
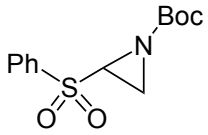


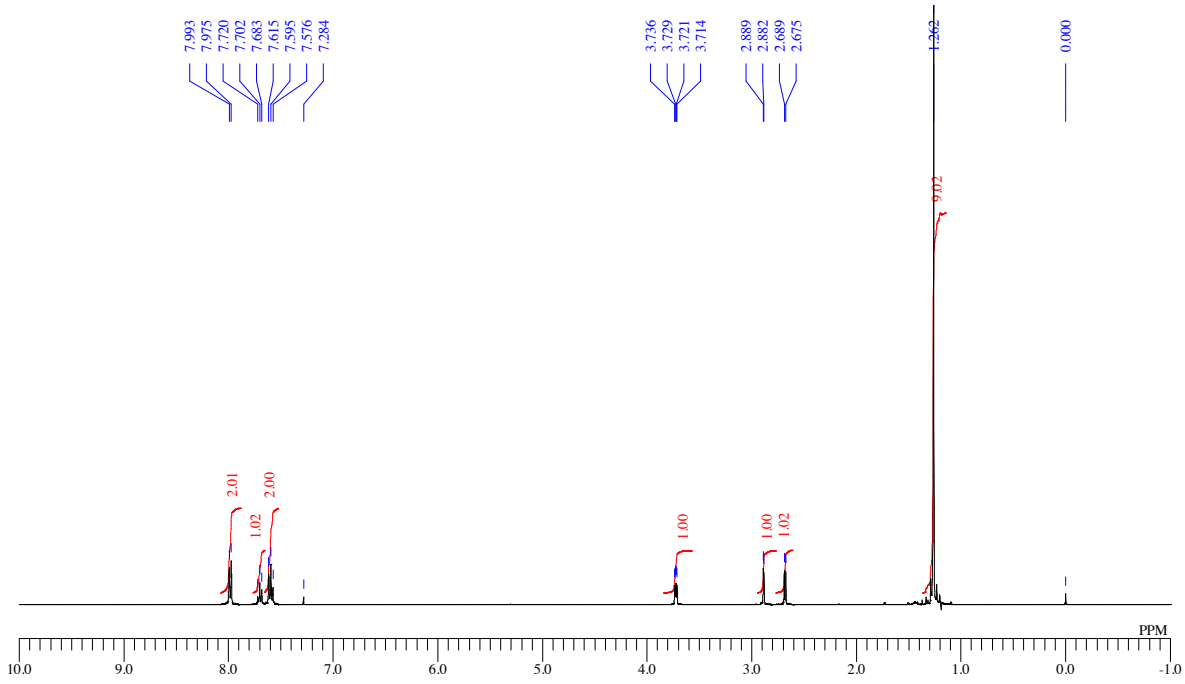
^{13}C NMR (100 MHz, $CDCl_3$)



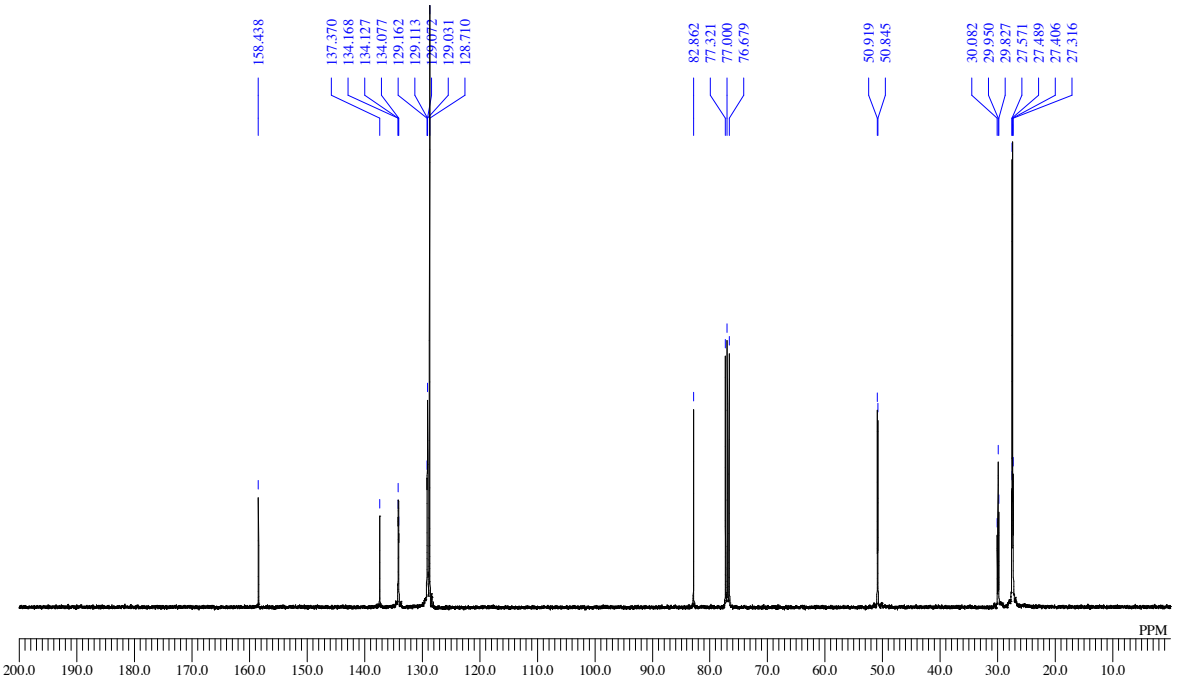


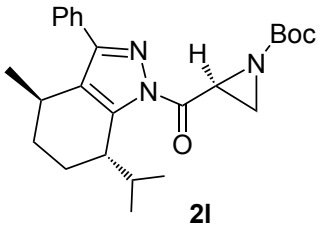
2k

^{1}H NMR (400 MHz, $CDCl_3$)



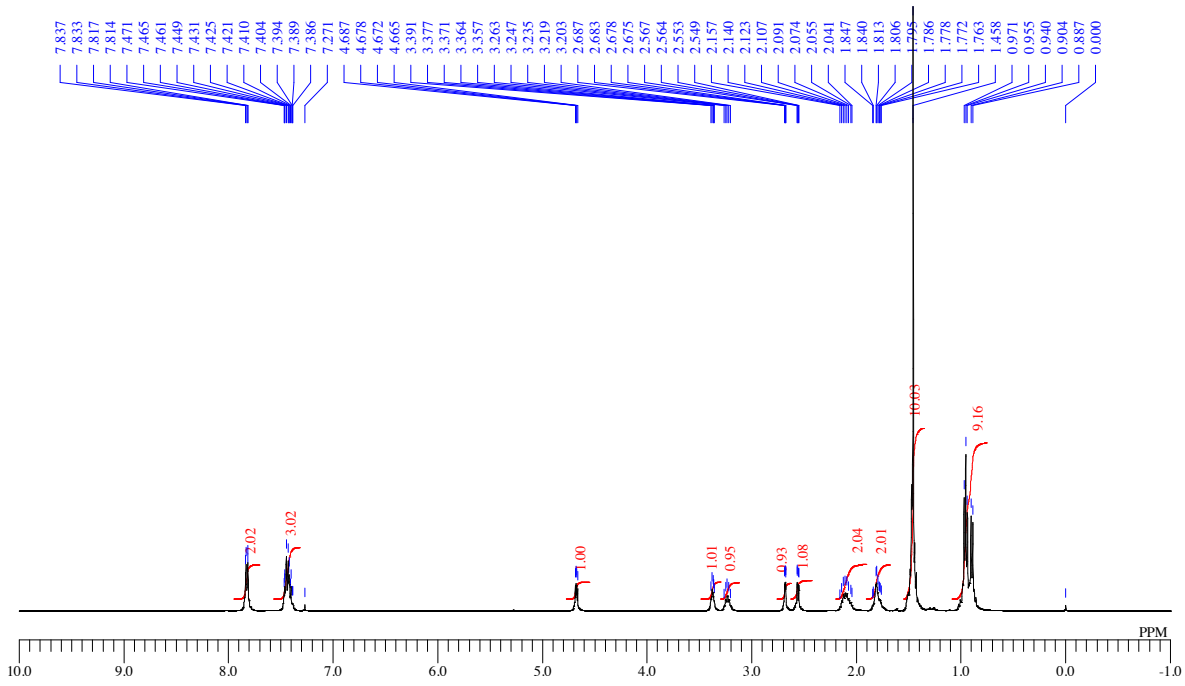
^{13}C NMR (100 MHz, $CDCl_3$)



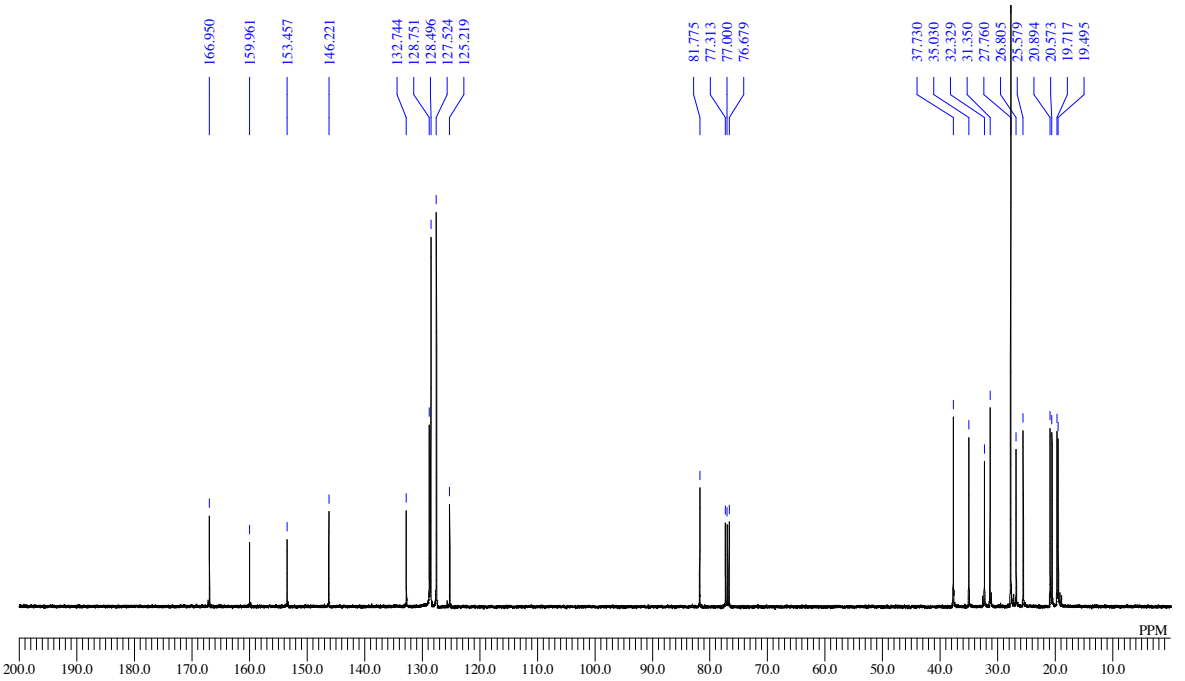


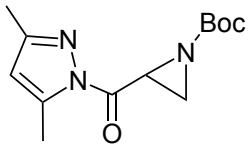
2l

^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)





2h (rac)

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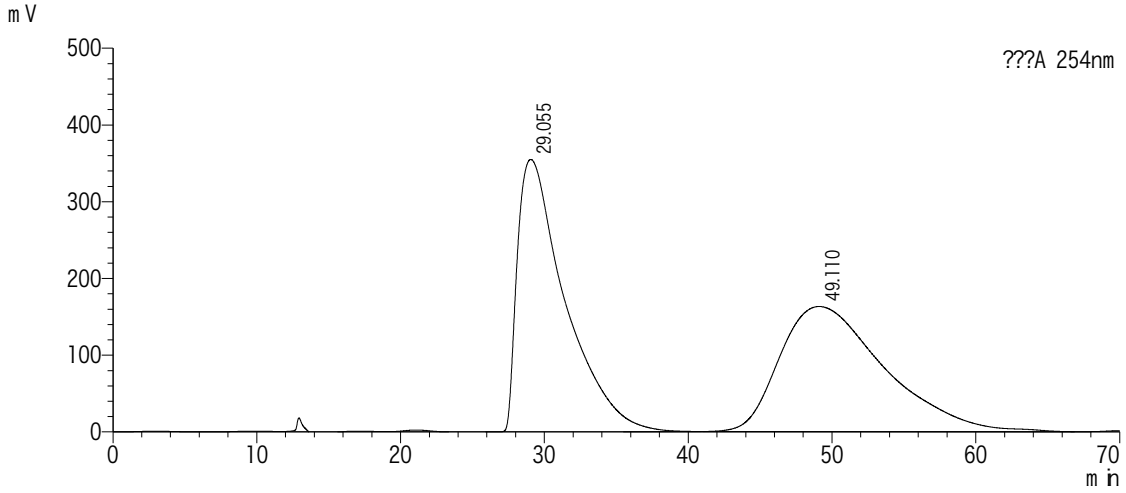
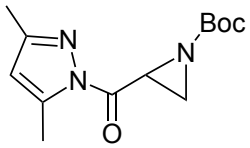


<peak report>

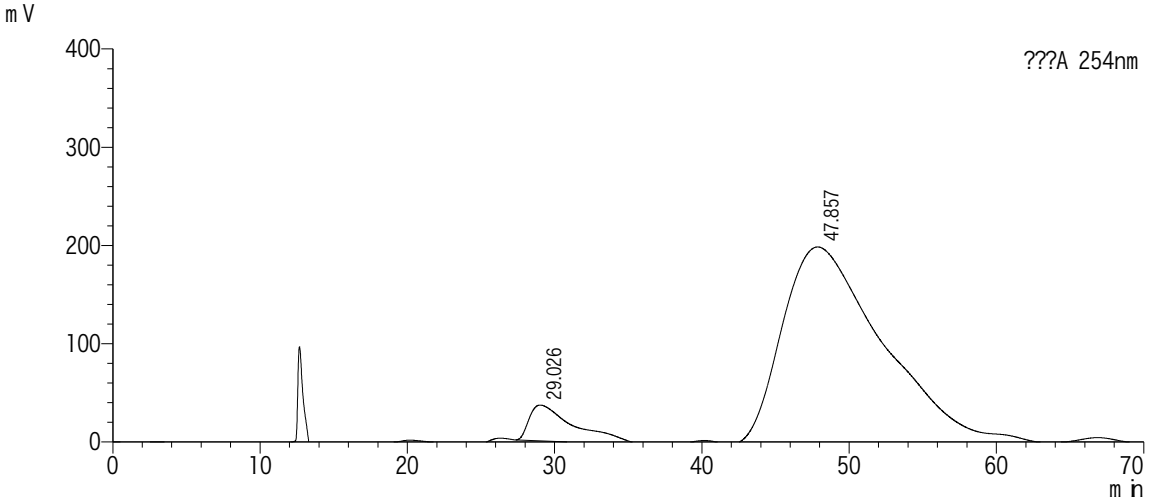
???A 254nm

peak#	retention time	area	height	area%
1	29.055	82106538	355604	49.905
2	49.110	82420254	162977	50.095



2h (84% ee)

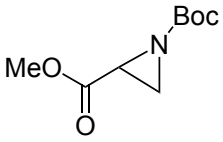
<chromatogram>



<peak report>

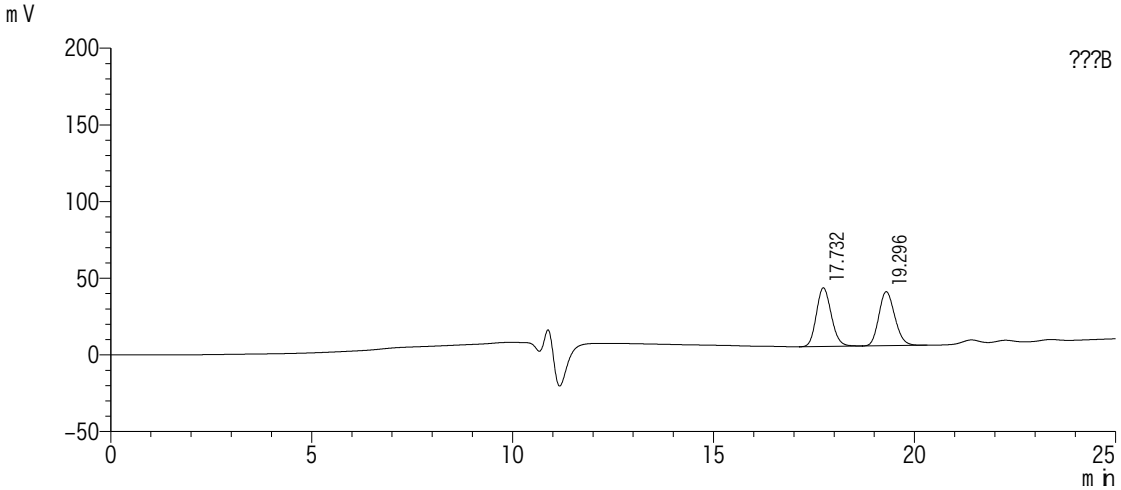
???A 254nm

peak#	retention time	area	height	area%
1	29.026	8053097	36504	7.791
2	47.857	95310013	200361	92.209



2b (rec)

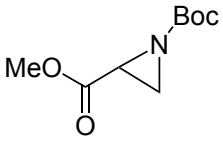
<chromatogram>



<peak report>

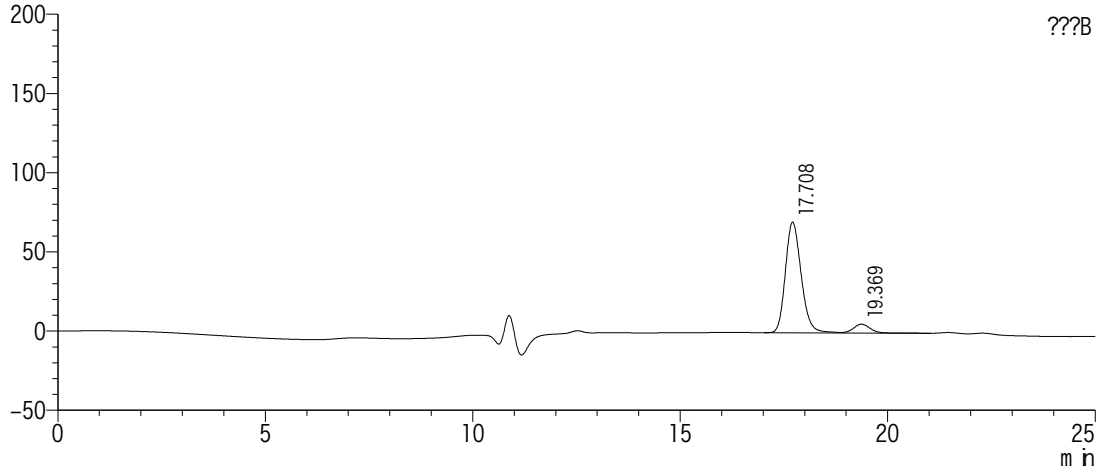
???B

peak#	retention time	area	height	area%
1	17.732	987115	38304	50.302
2	19.296	975268	35280	49.698



2b (84% ee)

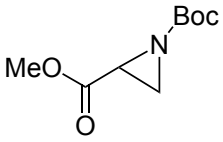
<chromatogram>



<peak report>

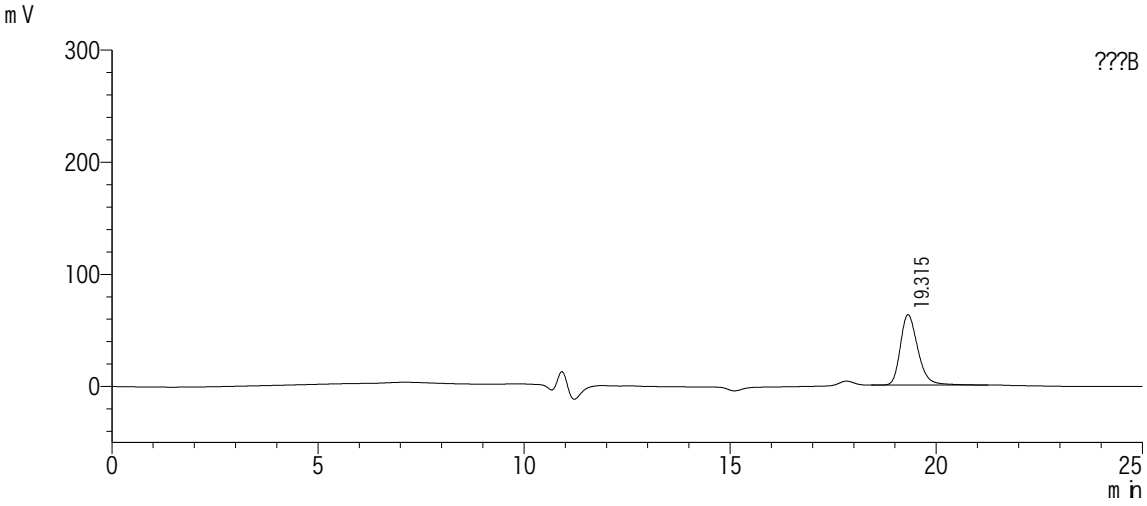
???B

peak#	retention time	area	height	area%
1	17.708	1845669	69949	92.132
2	19.369	157623	5575	7.868



2b (100% ee)

<chromatogram>



<peak report>

???B

peak#	retention time	area	height	area%
1	19.315	1858897	62891	100.000