Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supplementary Information

Electrochemically-initiated intramolecular 1,2-amino oxygenation of alkynes: facile access to formyl- and acyl-substituted indolizines

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1. General Information

All the electrochemical oxidations were performed in an undivided cell equipped with two platinum electrodes (10 × 15 × 0.2 mm^3) unless otherwise noted. All commercial reagents were purchased from TCI, Sigma-Aldrich, Accela, Bidepharm and Adamas-beta of the highest purity grade and used without further purification. The conversion of starting materials was monitored by thin layer chromatography using silica gel plates, and components were visualized by observation under UV light (254 and 365 nm).

^{1}H NMR spectra was recorded at 400 MHz or 600 MHz. The ^{13}C NMR spectra were recorded at 100 or 150 MHz. The ^{19}F NMR spectra were recorded at 376 MHz or 565 MHz. Chemical shifts were expressed in parts per million (δ) downfield from the internal standard tetramethylsilane (TMS), and were reported as s (singlet), d (doublet), t (triplet), dd (doublets of doublet), dt (doublets of triplet), td (triplets of doublet), ddd (doublets of doublets of doublet), dddd (doublets of doublets of doublets of doublet), dddt (doublets of doublets of doublets of triplet) and m (multiplet). The residual solvent signals were used as references and the chemical shifts were converted to the TMS scale ($CDCl_3$: δ H = 7.26 ppm, δ C = 77.16 ppm). The coupling constants J were given in Hz. High resolution mass spectra (HRMS) were obtained via ESI mode by using an Agilent Q-TOF 6540 mass spectrometer. Unless otherwise noted, all other compounds have been reported in the literature or are commercially available.

2 Preparation of Substrate

2.1 Structures of Starting Materials

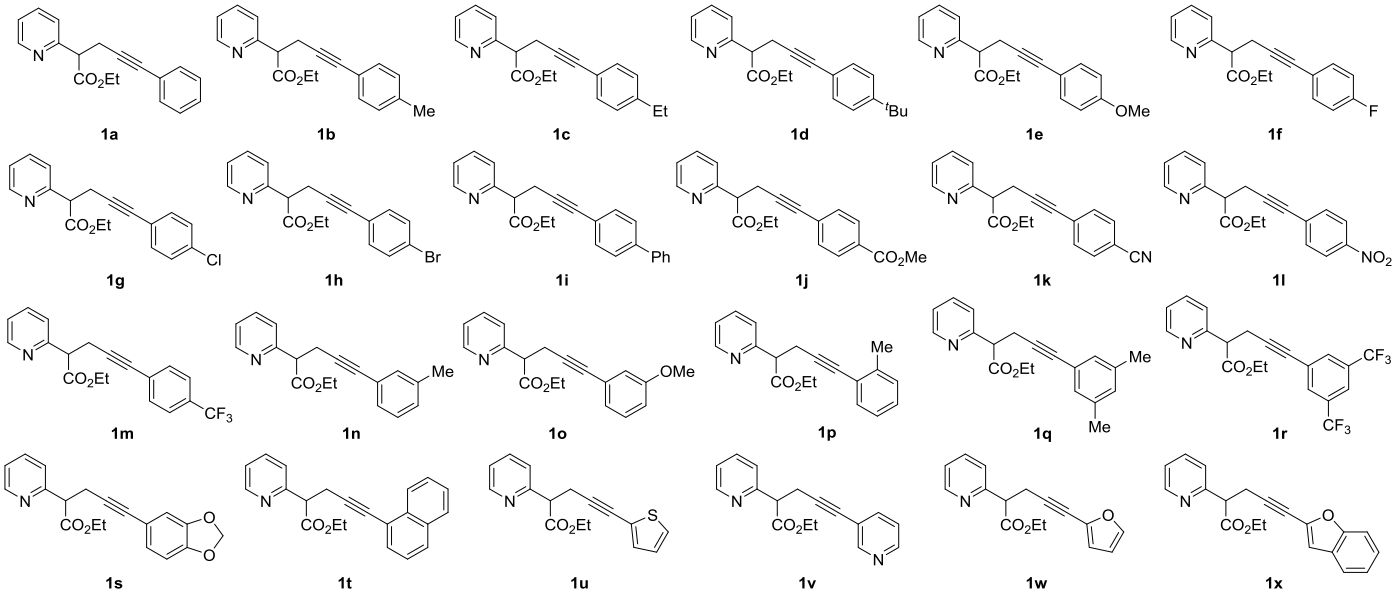


Figure S1: Scope of aromatic group

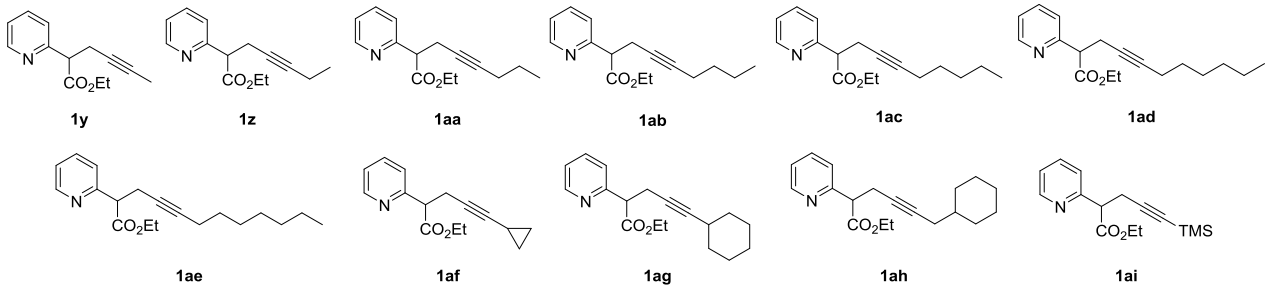


Figure S2: Scope of aliphatic group

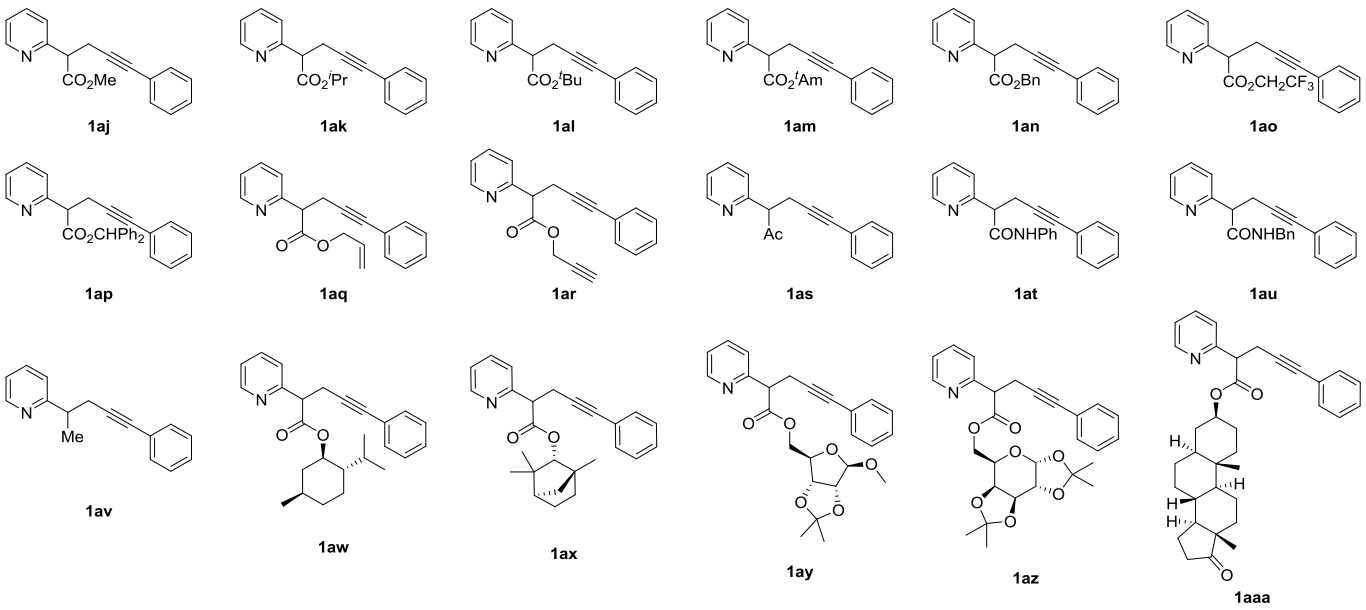


Figure S3: Scope of aliphatic group

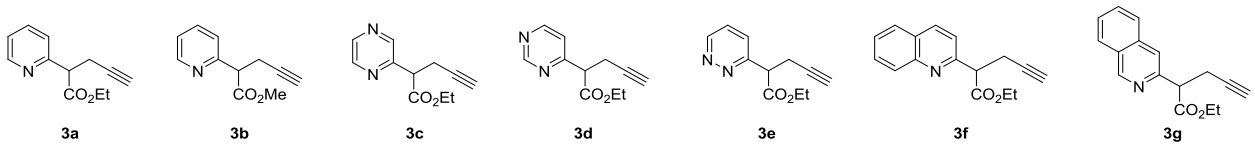
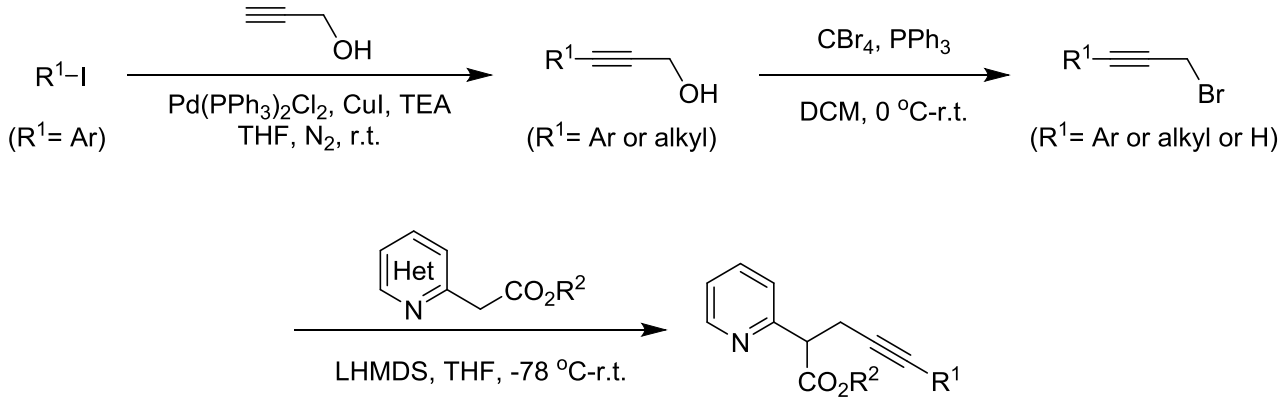


Figure S4: Scope of 6-Arylpurine Derivativesa

2.2 General Substrate Synthesis Steps:

All the starting substrates were prepared according to a protocol reported in the literature[1-9].



Step 1. Charge a 250 mL round bottom flask with stirring bar, $Pd(PPh_3)_2Cl_2$ (526.5 mg, 2.5 mol%) and CuI (286 mg, 5 mol%). Purge the sealed flask with argon for 5 minutes. Add THF (60 mL), triethylamine (12.5 mL, purged with argon) and aryl iodide (30 mmol, 1.0 equiv) to the mixture under argon atmosphere. Stir the mixture for 5 minutes at room temperature. Add propargyl alcohol (2.05 mL, 36 mmol, 1.2 equiv) dropwise to the mixture over 5 minutes. Stir the mixture for 16 hours at room temperature. Filter the reaction mixture through a pad of celite. Wash the solids with EtOAc. Concentrate the combined filtrates in vacuo. Purify the solid residue by flash column chromatography (PE/EA= 25/1 - 5/1) to afford aryl/alkyl propargyl alcohol.

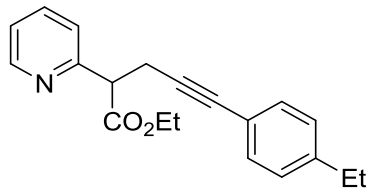
Step 2. In a 250 mL flask with stirring bar, add PPh_3 (6.3 g, 24 mmol, 1.2 equiv) slowly to a solution of aryl/alkyl propargyl alcohol (20 mmol, 1.0 equiv) and CBr_4 (7.96 g, 24 mmol,

1.2 equiv) in DCM (80 mL) at 0 ℃.Stir the reaction mixture at 25 ℃ for 10 hours. Evaporate the solvent under vacuum.Purify the mixture residue by flash column chromatography (only PE) to afford propargylic bromides.

Step 3. Charge a 250 mL round bottom flask with stirring bar, to a stirred solution of ethyl pyridine ester (10 mmol, 1.0 equiv) in THF (10 mL) was added LHMDS (11 mL, 1.0 M solution in THF, 1.1 equiv) at -78 ℃. After 15 min, a solution of the appropriate propargylic bromides (1.1 equiv) in THF (7 mL) was slowly added to this reaction mixture at -78 ℃. After being stirred for 16 h while slowly warmed up to rt, the reaction mixture was quenched with saturated aqueous NH_4Cl at 0 ℃. The organic layer was washed with brine and the aqueous layer was extracted with ethyl acetate one more time. The combined organic layers were dried over Na_2SO_4, filtered, and evaporated in vacuo. The resulting residue was purified by flash column chromatography (PE/EA= 20/1 - 4/1) to afford substrate.

2.3 Characterization Data for New Starting Materials

Ethyl 5-(4-ethylphenyl)-2-(pyridin-2-yl)pent-4-ynoate (1c)



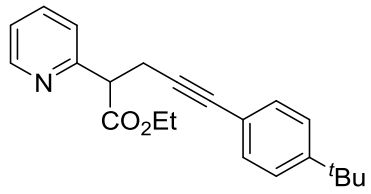
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1c** (2.18 g, 71%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (ddd, *J* = 5.2, 2.0, 0.8 Hz, 1H), 7.67 (tdd, *J* = 7.6, 2.0, 0.8 Hz, 1H), 7.37 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.23 – 7.18 (m, 3H), 7.10 – 7.05 (m, 2H), 4.27 – 4.14 (m, 2H), 4.08 (dd, *J* = 8.0, 7.2 Hz, 1H), 3.13 (ddd, *J* = 50.4, 16.8, 7.2 Hz, 2H), 2.60 (q, *J* = 7.6 Hz, 2H), 1.21 (dt, *J* = 10.4, 7.2 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.8, 157.5, 149.8, 144.2, 136.7, 131.6, 127.8, 123.4, 122.6, 120.8, 86.3, 82.5, 61.3, 53.3, 28.9, 22.6, 15.5, 14.3.

HRMS (ESI-TOF): exact mass calcd for $C_{20}H_{21}NO_2Na$ $(M+Na)^+$ requires m/z 330.1470, found m/z 330.1463.

Ethyl 5-(4-(tert-butyl)phenyl)-2-(pyridin-2-yl)pent-4-ynoate (1d)



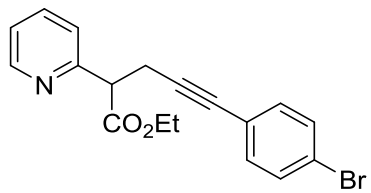
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1d** (2.31 g, 69%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (ddd, *J* = 4.8, 2.0, 1.2 Hz, 1H), 7.65 (td, *J* = 8.0, 2.0 Hz, 1H), 7.36 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.27 – 7.16 (m, 5H), 4.25 – 4.11 (m, 2H), 4.07 (dd, *J* = 8.0, 6.8 Hz, 1H), 3.12 (ddd, *J* = 48.4, 16.8, 7.2 Hz, 2H), 1.27 (s, 9H), 1.21 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 157.5, 151.1, 149.7, 136.7, 131.4, 125.3, 123.4, 122.6, 120.6, 86.4, 82.4, 61.3, 53.3, 34.8, 31.3, 22.6, 14.3.

HRMS (ESI-TOF): exact mass calcd for $C_{22}H_{25}NO_2Na$ $(M+Na)^+$ requires m/z 358.0783, found m/z 358.1779.

Ethyl 5-(4-bromophenyl)-2-(pyridin-2-yl)pent-4-ynoate (1h)



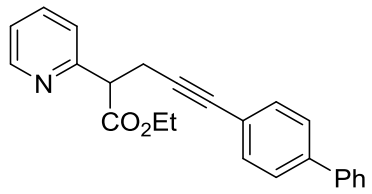
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 10/1) yielded **1h** (2.07 g, 58%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (ddd, *J* = 4.8, 2.0, 1.2 Hz, 1H), 7.67 (td, *J* = 7.6, 1.6 Hz, 1H), 7.38 – 7.34 (m, 3H), 7.20 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 7.16 – 7.11 (m, 2H), 4.26 – 4.13 (m, 2H), 4.07 (t, *J* = 7.6 Hz, 1H), 3.12 (ddd, *J* = 51.2, 16.8, 7.2 Hz, 2H), 1.21 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 157.3, 149.8, 136.7, 133.1, 131.5, 123.3, 122.7, 122.6, 122.0, 88.5, 81.3, 61.4, 53.0, 22.5, 14.3.

HRMS (ESI-TOF): exact mass calcd for $C_{18}H_{16}BrNO_2Na$ $(M+Na)^+$ requires m/z 380.0262, found m/z 380.0265.

Ethyl 5-([1,1'-biphenyl]-4-yl)-2-(pyridin-2-yl)pent-4-ynoate (1i)



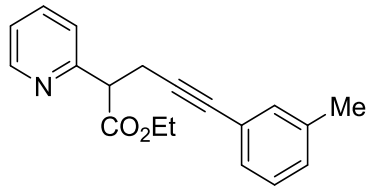
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1i** (2.34 g, 63%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (td, *J* = 7.6, 2.0 Hz, 1H), 7.59 – 7.54 (m, 2H), 7.51 – 7.48 (m, 2H), 7.46 – 7.32 (m, 6H), 7.22 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 4.28 – 4.18 (m, 2H), 4.12 (t, *J* = 7.6 Hz, 1H), 3.18 (ddd, *J* = 50.8, 17.2, 7.2 Hz, 1H), 1.24 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 157.4, 149.8, 140.6, 140.5, 136.7, 132.1, 128.9, 127.6, 127.1, 126.9, 123.3, 122.6, 122.6, 87.9, 82.2, 61.3, 53.2, 22.6, 14.3.

HRMS (ESI-TOF): exact mass calcd for $C_{24}H_{21}NO_2Na$ $(M+Na)^+$ requires m/z 378.1470, found m/z 378.1463.

Ethyl 2-(pyridin-2-yl)-5-(m-tolyl)pent-4-ynoate (1n)



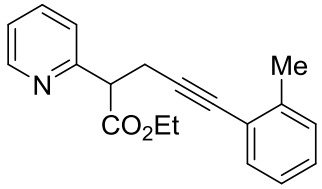
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1n** (2.02 g, 69%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (ddd, *J* = 4.8, 1.6, 0.6 Hz, 1H), 7.70 – 7.64 (m, 1H), 7.38 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.20 (ddt, *J* = 7.6, 4.0, 1.6 Hz, 1H), 7.15 – 7.04 (m, 4H), 4.27 – 4.14 (m, 2H), 4.08 (t, *J* = 7.6 Hz, 1H), 3.13 (ddd, *J* = 52.0, 16.8, 7.2 Hz, 2H), 2.28 (s, 3H), 1.22 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 157.5, 149.8, 137.9, 136.7, 132.3, 128.8, 128.7, 128.2, 123.4, 123.3, 122.6, 86.8, 82.5, 61.3, 53.2, 22.6, 21.3, 14.3.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{19}NO_2Na$ $(M+Na)^+$ requires m/z 316.1313, found m/z 316.1306.

Ethyl 2-(pyridin-2-yl)-5-(o-tolyl)pent-4-ynoate (1p)



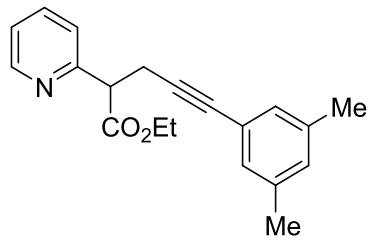
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1p** (1.93 g, 66%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (ddd, *J* = 4.8, 1.6, 0.8 Hz, 1H), 7.70 – 7.64 (m, 1H), 7.38 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.28 – 7.25 (m, 1H), 7.20 (ddt, *J* = 7.6, 4.8, 1.2 Hz, 1H), 7.17 – 7.10 (m, 2H), 7.09 – 7.03 (m, 1H), 4.26 – 4.15 (m, 2H), 4.11 (dd, *J* = 8.0, 6.8 Hz, 1H), 3.20 (ddd, *J* = 37.2, 17.2, 7.2 Hz, 2H), 2.24 (s, 3H), 1.22 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 157.4, 149.8, 140.2, 136.7, 131.9, 129.4, 127.9, 125.5, 123.4, 123.4, 122.6, 91.1, 81.2, 61.4, 53.4, 22.7, 20.6, 14.3.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{19}NO_2Na$ $(M+Na)^+$ requires m/z 316.313, found m/z 316.1307.

Ethyl 5-(3,5-dimethylphenyl)-2-(pyridin-2-yl)pent-4-ynoate (1q)



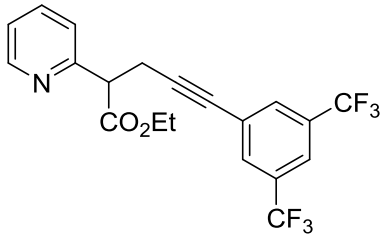
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1q** (2.18 g, 71%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.67 (td, *J* = 7.6, 1.6 Hz, 1H), 7.38 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.20 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 6.93 (s, 2H), 6.89 (s, 1H), 4.27 – 4.15 (m, 2H), 4.08 (dd, *J* = 8.0, 7.2 Hz, 1H), 3.12 (ddd, *J* = 53.2, 16.8, 7.2 Hz, 2H), 2.24 (s, 6H), 1.23 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 157.3, 149.8, 136.7, 133.1, 131.5, 123.3, 122.7, 122.6, 122.0, 88.5, 81.3, 61.4, 53.0, 22.5, 14.3.

HRMS (ESI-TOF): exact mass calcd for $C_{20}H_{21}NO_2Na$ $(M+Na)^+$ requires m/z 330.1470, found m/z 330.1461.

Ethyl 5-(3,5-bis(trifluoromethyl)phenyl)-2-(pyridin-2-yl)pent-4-ynoate (1r)



Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 5/1) yielded **1r** (2.62 g, 63%) as a brown oil.

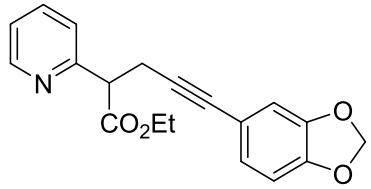
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.75 – 7.67 (m, 4H), 7.35 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.24 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 4.27 – 4.15 (m, 2H), 4.09 (t, *J* = 7.6 Hz, 1H), 3.18 (ddd, *J* = 48.4, 17.2, 7.6 Hz, 2H), 1.22 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 157.0, 149.9, 136.9, 132.0, 131.6 (m), 125.9, 123.8 (d, $J_{C\text{-}F}$ = 271.0 Hz), 123.3, 122.8, 121.3 (m), 91.5, 79.7, 61.6, 52.8, 22.3, 14.3.

^{19}F NMR (376 MHz, $CDCl_3$) δ -63.2.

HRMS (ESI-TOF): exact mass calcd for $C_{20}H_{15}F_6NO_2Na$ $(M+Na)^+$ requires m/z 438.0905, found m/z 438.0906.

Ethyl 5-(benzo[*d*][1,3]dioxol-5-yl)-2-(pyridin-2-yl)pent-4-ynoate (1s)



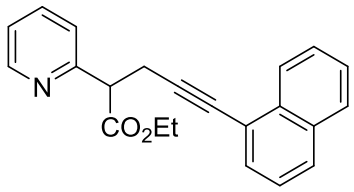
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 8/1) yielded **1s** (2.16 g, 67%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (ddd, *J* = 4.8, 1.6, 0.8 Hz, 1H), 7.67 (td, *J* = 7.6, 1.6 Hz, 1H), 7.36 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.20 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 6.81 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.73 (d, *J* = 1.6, 1H), 6.68 (d, *J* = 8.0, 1H), 5.93 (s, 2H), 4.27 – 4.13 (m, 2H), 4.06 (t, *J* = 8.0, 1H), 3.11 (ddd, *J* = 49.6, 16.8, 7.2 Hz, 2H), 1.22 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 157.5, 149.8, 147.6, 147.4, 136.7, 126.1, 123.3, 122.6, 116.9, 111.7, 108.4, 101.3, 85.4, 82.1, 61.3, 53.2, 22.5, 14.3.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{17}NO_4Na$ $(M+Na)^+$ requires m/z 346.1055, found m/z 346.1048.

Ethyl 5-(naphthalen-1-yl)-2-(pyridin-2-yl)pent-4-ynoate (1t)



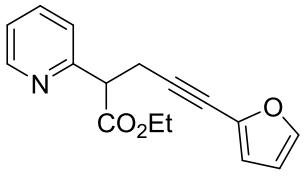
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1t** (2.07 g, 61%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (ddd, *J* = 5.2, 2.0, 1.2 Hz, 1H), 8.05 – 8.00 (m, 1H), 7.82 – 7.77 (m, 1H), 7.76 (dt, *J* = 8.4, 1.2 Hz, 1H), 7.69 (td, *J* = 7.6, 2.0 Hz, 1H), 7.53 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.48 – 7.42 (m, 3H), 7.36 (dd, *J* = 8.0, 7.2 Hz, 1H), 7.24 (ddd, *J* = 7.6, 5.2, 1.2 Hz, 1H), 4.30 – 4.17 (m, 3H), 3.11 (ddd, *J* = 31.6, 17.2, 7.2 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 157.5, 149.9, 136.8, 133.6, 133.2, 130.2, 128.3, 128.2, 126.6, 126.4, 126.3, 125.3, 123.5, 122.7, 121.3, 92.2, 80.4, 61.4, 53.3, 22.8, 14.3.

HRMS (ESI-TOF): exact mass calcd for $C_{22}H_{19}NO_2Na$ $(M+Na)^+$ requires m/z 352.1313, found m/z 352.1305.

Ethyl 5-(furan-2-yl)-2-(pyridin-2-yl)pent-4-ynoate (1w)



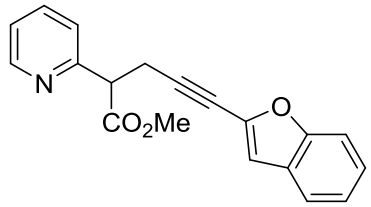
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1w** (1.51 g, 56%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (ddd, *J* = 4.8, 2.0, 1.2 Hz, 1H), 7.67 (td, *J* = 7.6, 1.6 Hz, 1H), 7.36 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.20 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 7.15 (dd, *J* = 5.2, 1.2 Hz, 1H), 7.04 (dd, *J* = 3.6, 1.2 Hz, 1H), 6.90 (dd, *J* = 5.2, 3.6 Hz, 1H), 4.27 – 4.13 (m, 2H), 4.08 (t, *J* = 7.6 Hz, 1H), 3.16 (ddd, *J* = 54.4, 16.8, 7.2 Hz, 2H), 1.22 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 157.3, 149.8, 136.7, 131.4, 126.9, 126.4, 123.7, 123.4, 122.6, 91.3, 75.5, 61.4, 53.0, 22.8, 14.3.

HRMS (ESI-TOF): exact mass calcd for $C_{16}H_{15}NO_3Na$ $(M+Na)^+$ requires m/z 292.0947, found m/z 292.0950.

Ethyl 5-(benzofuran-2-yl)-2-(pyridin-2-yl)pent-4-ynoate (1x)



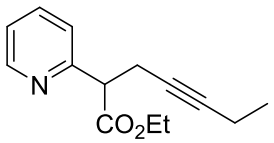
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1x** (2.11 g, 56%) as a brown solid, **M.P.** 66.4 - 67.7 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.67 (td, *J* = 8.0, 2.0 Hz, 1H), 7.51 – 7.48 (m, 1H), 7.41 – 7.34 (m, 2H), 7.31 – 7.25 (m, 1H), 7.23 – 7.17 (m, 2H), 6.77 (d, *J* = 0.8 Hz, 1H), 4.14 (dd, *J* = 8.0, 6.8 Hz, 1H), 3.73 (s, 3H), 3.25 (ddd, *J* = 56.0, 17.2, 7.2 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.8, 156.8, 154.6, 149.9, 138.8, 136.9, 127.7, 125.4, 123.4, 123.2, 122.8, 121.1, 111.2, 110.9, 93.7, 72.9, 52.6, 52.5, 22.5.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{15}NO_3Na$ $(M+Na)^+$ requires m/z 328.0950, found m/z 328.0944.

Ethyl 2-(pyridin-2-yl)hept-4-ynoate (1z)



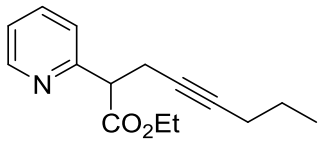
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 20/1) yielded **1z** (1.67 g, 72%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.62 (td, *J* = 7.6, 1.6 Hz, 1H), 7.30 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.15 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 4.22 – 4.07 (m, 2H), 3.94 (t, *J* = 7.6 Hz, 1H), 2.85 (dddt, *J* = 56.0, 16.4, 7.2, 2.4 Hz, 2H), 2.02 (tt, *J* = 7.2, 2.4 Hz, 2H), 1.38 (sext, *J* = 7.2 Hz, 2H), 1.19 (t, *J* = 7.2 Hz, 3H), 0.84 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 157.7, 149.6, 136.6, 123.2, 122.4, 83.7, 76.4, 61.1, 53.5, 21.9, 14.2, 14.2, 12.4.

HRMS (ESI-TOF): exact mass calcd for $C_{14}H_{17}NO_2Na$ $(M+Na)^+$ requires m/z 254.1157, found m/z 254.1160.

Ethyl 2-(pyridin-2-yl)oct-4-ynoate (1aa)



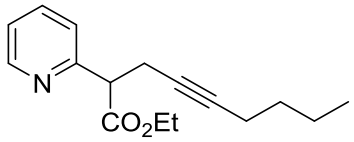
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 20/1) yielded **1aa** (1.79 g, 73%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (ddd, *J* = 0.8, 2.0, 4.8 Hz, 1H), 7.62 (td, *J* = 1.6, 7.6 Hz, 1H), 7.30 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.15 (ddd, *J* = 1.2, 4.8, 7.6 Hz, 1H), 4.22 – 4.07 (m, 2H), 3.94 (t, *J* = 7.6 Hz, 1H), 2.85 (dddt, *J* = 2.4, 7.2, 16.4, 56.0 Hz, 2H), 2.02 (tt, *J* = 2.4, 7.2 Hz, 2H), 1.38 (sext, *J* = 7.2 Hz, 2H), 1.19 (t, *J* = 7.2 Hz, 3H), 0.84 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 157.6, 149.6, 136.5, 123.1, 122.4, 82.1, 77.2, 61.1, 53.6, 22.3, 22.0, 20.7, 14.2, 13.4.

HRMS (ESI-TOF): exact mass calcd for $C_{15}H_{19}NO_2Na$ $(M+Na)^+$ requires m/z 268.1313, found m/z 268.1316.

Ethyl 2-(pyridin-2-yl)non-4-ynoate (1ab)



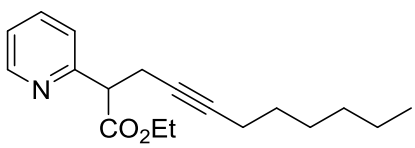
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 20/1) yielded **1ab** (1.95 g, 75%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.63 (td, *J* = 7.6, 2.0 Hz, 1H), 7.31 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.17 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 4.25 – 4.08 (m, 2H), 3.95 (t, *J* = 7.6 Hz, 1H), 2.85 (dddt, *J* = 53.2, 16.8, 7.6, 2.4 Hz, 2H), 2.05 (tt, *J* = 6.4, 2.4 Hz, 2H), 1.39 – 1.23 (m, 4H), 1.20 (t, *J* = 7.2 Hz, 3H), 0.83 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 157.7, 149.6, 136.5, 123.1, 122.4, 82.3, 77.0, 61.2, 53.6, 31.0, 22.0, 21.8, 18.4, 14.2, 13.7.

HRMS (ESI-TOF): exact mass calcd for $C_{16}H_{21}NO_2Na$ $(M+Na)^+$ requires m/z 282.1474, found m/z 282.1470.

Ethyl 2-(pyridin-2-yl)undec-4-ynoate (1ad)



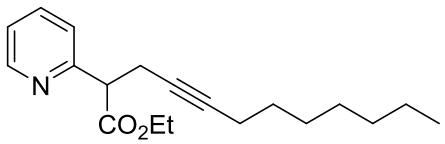
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 20/1) yielded **1ad** (2.04 g, 71%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.65 (td, *J* = 7.6, 2.0 Hz, 1H), 7.32 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.18 (ddd, *J* = 7.2, 4.8, 0.8 Hz, 1H), 4.24 – 4.11 (m, 2H), 3.96 (t, *J* = 7.6 Hz, 1H), 2.85 (dddt, *J* = 52.4, 16.4, 7.2, 2.4 Hz, 2H), 2.06 (tt, *J* = 6.8, 2.4 Hz, 2H), 1.37 (quint, *J* = 6.0 Hz, 2H), 1.32 – 1.16 (m, 10H), 0.87 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.0, 157.7, 149.7, 136.6, 123.2, 122.5, 82.4, 77.1, 61.2, 53.6, 31.5, 29.0, 28.5, 22.7, 22.0, 18.8, 14.3, 14.2.

HRMS (ESI-TOF): exact mass calcd for $C_{18}H_{26}NO_2$ $(M+Na)^+$ requires m/z 288.1958, found m/z 288.1953.

Ethyl 2-(pyridin-2-yl)dodec-4-ynoate (1ae)



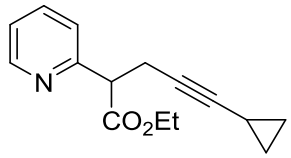
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 20/1) yielded **1ae** (2.35 g, 78%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 – 8.84 (m, 1H), 7.64 (td, *J* = 7.6, 1.6 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.20 – 7.14 (m, 1H), 4.22 – 4.12 (m, 2H), 3.96 (t, *J* = 8.0 Hz, 1H), 2.87 (dddt, *J* = 52.4, 16.4, 7.2, 2.4 Hz, 2H), 2.09 – 2.01 (m, 2H), 1.42 – 1.15 (m, 16H), 0.88 (t, *J* = 7.2 Hz, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 157.7, 149.7, 136.6, 123.2, 122.4, 82.4, 77.1, 61.2, 53.6, 31.9, 29.0, 28.9, 28.8, 22.8, 22.0, 18.8, 14.3, 14.2.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{28}NO_2$ $(M+H)^+$ requires m/z 302.2115, found m/z 302.2119.

Ethyl 5-cyclopropyl-2-(pyridin-2-yl)pent-4-ynoate (1af)



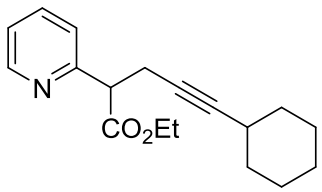
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 20/1) yielded **1af** (1.58 g, 65%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (ddd, *J* = 4.8, 2.0, 1.2 Hz, 1H), 7.64 (td, *J* = 7.6, 1.6 Hz, 1H), 7.29 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.17 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 4.23 – 4.09 (m, 2H), 3.92 (t, *J* = 7.6 Hz, 1H), 2.82 (dddd, *J* = 58.0, 16.8, 7.2, 2.0 Hz, 1H), 1.14 – 1.06 (m, 1H), 0.66 – 0.60 (m, 2H), 0.51 – 0.46 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 157.6, 149.6, 136.6, 123.2, 122.5, 85.4, 72.4, 61.2, 53.5, 22.0, 14.3, 8.1, 8.0.

HRMS (ESI-TOF): exact mass calcd for $C_{15}H_{18}NO_2$ $(M+H)^+$ requires m/z 244.1332, found m/z 244.1338.

Ethyl 5-cyclohexyl-2-(pyridin-2-yl)pent-4-ynoate (1ag)



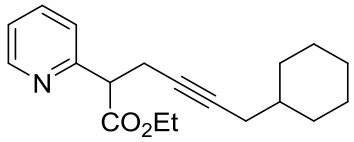
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 20/1) yielded **1ag** (2.17 g, 76%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (ddd, *J* = 4.8, 1.6, 0.8 Hz, 1H), 7.64 (td, *J* = 8.0, 2.0 Hz, 1H), 7.33 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.18 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 4.24 – 4.11 (m, 2H), 3.96 (t, *J* = 7.6 Hz, 1H), 2.87 (dddd, *J* = 48.8, 16.2, 7.6, 2.4 Hz, 1H), 2.30 – 2.21 (m, 1H), 1.69 – 1.54 (m, 4H), 1.48 – 1.41 (m, 1H), 1.33 – 1.19 (m, 8H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.0, 157.8, 149.7, 136.5, 123.3, 122.4, 86.7, 61.2, 53.7, 32.9, 29.0, 26.0, 24.8, 22.1, 14.3.

HRMS (ESI-TOF): exact mass calcd for $C_{18}H_{24}NO_2$ $(M+H)^+$ requires m/z 286.1802, found m/z 286.1801.

Ethyl 6-cyclohexyl-2-(pyridin-2-yl)hex-4-ynoate (1ah)



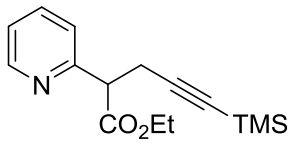
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1ah** (2.13 g, 71%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (ddd, *J* = 4.8, 1.6, 0.8 Hz, 1H), 7.61 (td, *J* = 7.6, 2.0 Hz, 1H), 7.30 (dt, *J* = 7.6, 0.8 Hz, 1H), 7.15 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 4.21 – 4.07 (m, 2H), 3.94 (t, *J* = 7.6 Hz, 1H), 2.85 (dddt, *J* = 44.0, 16.8, 7.2, 2.4 Hz, 1H), 1.92 (dt, *J* = 6.8, 2.4 Hz, 2H), 1.68 – 1.53 (m, 6H), 1.30 – 1.22 (m, 1H), 1.20 – 1.02 (m, 1H), 0.87 – 0.79 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.8, 157.6, 149.6, 136.5, 123.1, 122.4, 81.1, 77.9, 61.1, 53.6, 37.5, 32.5, 32.5, 26.5, 26.3, 21.9, 14.2.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{26}NO_2$ $(M+H)^+$ requires m/z 299.1885, found m/z 299.1888.

Ethyl 2-(pyridin-2-yl)-5-(trimethylsilyl)pent-4-ynoate (1ai)



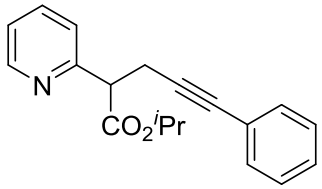
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 25/1) yielded **1ai** (2.12 g, 77%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (ddd, *J* = 4.8, 2.0, 1.2 Hz, 1H), 7.64 (td, *J* = 7.6, 1.6 Hz, 1H), 7.31 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.18 (ddd, *J* = 7.2, 4.8, 1.2 Hz, 1H), 4.24 – 4.10 (m, 2H), 3.98 (t, *J* = 8.0 Hz, 1H), 2.92 (ddd, *J* = 52.0, 17.2, 7.2 Hz, 1H), 1.21 (t, *J* = 7.2 Hz, 3H), 0.06 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 157.4, 149.7, 136.5, 123.4, 122.5, 104.1, 86.8, 61.3, 53.1, 23.0, 14.3, 0.1.

HRMS (ESI-TOF): exact mass calcd for $C_{15}H_{22}NO_2SiNa$ $(M+H)^+$ requires m/z 298.1239, found m/z 298.1230.

Isopropyl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1ak)



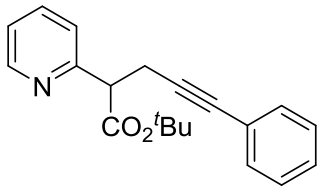
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1ak** (1.89 g, 63%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.66 (tt, *J* = 7.6, 1.6 Hz, 1H), 7.37 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.32 – 7.27 (m, 2H), 7.26 – 7.26 (m, 3H), 7.19 (ddt, *J* = 7.6, 4.8, 1.2 Hz, 1H), 5.09 (hept, *J* = 6.4 Hz, 1H), 4.06 (t, *J* = 7.6 Hz, 1H), 3.13 (ddd, *J* = 50.4, 16.8, 7.6 Hz, 1H), 1.24 (d, *J* = 6.0 Hz, 3H), 1.16 (d, *J* = 6.0 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 157.5, 149.7, 136.6, 131.7, 128.3, 127.9, 123.7, 123.2, 122.5, 87.3, 82.3, 68.8, 53.3, 22.5, 21.9, 21.7.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{20}NO_2$ $(M+H)^+$ requires m/z 293.1489, found m/z 293.1483.

Tert-butyl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1al)



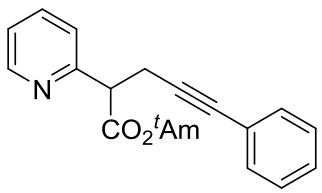
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1al** (2.00 g, 65%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.66 (td, *J* = 8.0, 2.0 Hz, 1H), 7.37 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.32 – 7.27 (m, 2H), 7.27 – 7.22 (m, 3H), 7.19 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 4.01 (t, *J* = 7.6 Hz, 1H), 3.09 (ddd, *J* = 43.2, 16.8, 7.2 Hz, 1H), 1.43 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 157.9, 149.6, 136.5, 131.6, 128.3, 127.8, 123.8, 123.1, 122.4, 87.5, 82.2, 81.5, 54.0, 28.1, 22.5.

HRMS (ESI-TOF): exact mass calcd for $C_{20}H_{22}NO_2$ $(M+H)^+$ requires m/z 308.1645, found m/z 308.1636.

Tert-pentyl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1am)



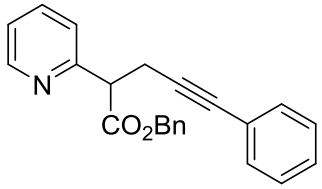
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1am** (2.02 g, 63%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.66 (td, *J* = 7.6, 1.6 Hz, 1H), 7.37 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.31 – 7.27 (m, 2H), 7.26 – 7.22 (m, 3H), 7.18 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 4.02 (t, *J* = 7.6 Hz, 1H), 3.10 (ddd, *J* = 47.2, 17.2, 7.6 Hz, 1H), 1.73 (qd, *J* = 7.6, 2.0 Hz, 2H), 1.40 (d, *J* = 4.8 Hz, 6H), 0.76 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 157.9, 149.6, 136.5, 131.6, 128.2, 127.8, 123.7, 123.2, 122.4, 87.6, 83.9, 82.2, 54.1, 33.6, 25.7, 25.4, 22.4, 8.1.

HRMS (ESI-TOF): exact mass calcd for $C_{21}H_{24}NO_2Na$ $(M+Na)^+$ requires m/z 344.1627, found m/z 344.1618.

Benzyl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1an)



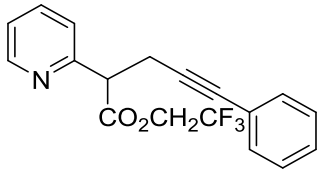
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1an** (2.01 g, 59%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (ddd, *J* = 4.8, 1.6, 0.8 Hz, 1H), 7.65 (td, *J* = 7.6, 1.6 Hz, 1H), 7.37 – 7.34 (m, 2H), 7.27 – 7.23 (m, 9H), 7.20 (ddd, *J* = 7.6, 5.2, 1.2 Hz, 1H), 5.19 (q, *J* = 12.4 Hz, 2H), 4.16 (t, *J* = 7.6 Hz, 1H), 3.17 (ddd, *J* = 55.2, 16.8, 7.2 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 157.2, 149.8, 136.7, 135.9, 131.7, 128.7, 128.6, 128.3, 128.2, 128.1, 127.9, 127.8, 127.1, 123.6, 123.4, 122.7, 87.1, 82.5, 66.9, 65.5, 53.2, 22.5.

HRMS (ESI-TOF): exact mass calcd for $C_{23}H_{20}NO_2$ $(M+H)^+$ requires m/z 342.1489, found m/z 342.1481.

2,2,2-trifluoroethyl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1ao)



Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 10/1) yielded **1ao** (2.20 g, 66%) as a brown oil.

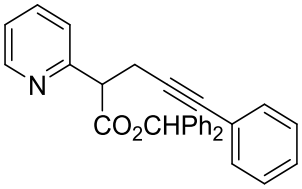
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.69 (td, *J* = 7.6, 2.0 Hz, 1H), 7.38 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.32 – 7.21 (m, 6H), 4.63 – 4.43 (m, 2H), 4.20 (t, *J* = 7.6 Hz, 1H), 3.17 (ddd, *J* = 57.6, 17.2, 7.6 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 156.4, 149.9, 136.9, 131.7, 128.3, 128.1, 124.3, 123.5, 123.4, 123.0, 121.5, 86.3, 82.8, 61.3 (q, $J_{C\text{-}F}$ = 3.6 Hz), 52.7, 22.5.

^{19}F NMR (376 MHz, $CDCl_3$) δ -73.7.

HRMS (ESI-TOF): exact mass calcd for $C_{18}H_{15}F_3NO_2$ $(M+H)^+$ requires m/z 334.1049, found m/z 334.1049.

Benzhydryl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1ap)



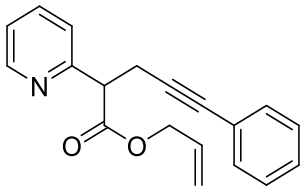
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1ap** (2.25 g, 54%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (ddd, *J* = 4.8, 1.6, 0.8 Hz, 1H), 7.63 (td, *J* = 7.6, 2.0 Hz, 1H), 7.34 – 7.28 (m, 3H), 7.29 – 7.18 (m, 13H), 7.17 – 7.11 (m, 2H), 6.93 (s, 1H), 4.23 (t, *J* = 7.6 Hz, 1H), 3.19 (ddd, *J* = 50.4, 17.2, 7.2 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 157.1, 149.7, 140.0, 139.9, 136.7, 131.7, 128.6, 128.4, 128.2, 128.0, 127.9, 127.3, 127.0, 123.5, 123.4, 122.6, 87.1, 82.5, 77.7, 53.4, 22.4.

HRMS (ESI-TOF): exact mass calcd for $C_{29}H_{23}NO_2Na$ $(M+Na)^+$ requires m/z 440.1621, found m/z 440.1618.

Allyl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1aq)



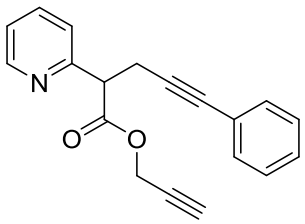
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1aq** (1.69 g, 58%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.68 (td, *J* = 7.6, 2.0 Hz, 1H), 7.38 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.31 – 7.19 (m, 6H), 5.87 (ddt, *J* = 17.2, 10.4, 5.6 Hz, 1H), 5.24 (dq, *J* = 17.2, 1.6 Hz, 1H), 5.17 (dq, *J* = 10.4, 1.2 Hz, 1H), 4.70 – 4.60 (m, 2H), 4.13 (dd, *J* = 8.0, 7.2 Hz, 1H), 3.16 (ddd, *J* = 51.6, 16.8, 7.2 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 157.3, 149.8, 136.7, 132.0, 131.7, 128.3, 127.9, 123.6, 123.4, 122.7, 118.3, 87.1, 82.5, 65.8, 53.2, 22.6.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{18}NO_2$ $(M+H)^+$ requires m/z 292.1332, found m/z 292.1327.

Prop-2-yn-1-yl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1ar)



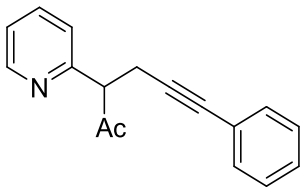
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1ar** (1.49 g, 51%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.71 – 7.65 (m, 1H), 7.38 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.32 – 7.19 (m, 6H), 4.74 (ddd, *J* = 44.0, 15.6, 2.4 Hz, 2H), 4.15 (dd, *J* = 8.0, 6.8 Hz, 1H), 3.16 (ddd, *J* = 50.0, 12.8, 6.8 Hz, 2H), 2.42 (t, *J* = 2.4 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 156.8, 149.9, 136.8, 131.7, 128.3, 127.9, 123.5, 123.5, 122.8, 86.8, 82.6, 75.2, 52.9, 52.7, 22.6.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{16}NO_2$ $(M+H)^+$ requires m/z 290.1176, found m/z 290.1176.

6-phenyl-3-(pyridin-2-yl)hex-5-yn-2-one (1as)



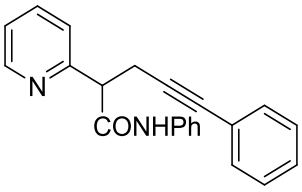
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1as** (1.82 g, 73%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (ddd, *J* = 4.8, 1.6, 0.8 Hz, 1H), 7.69 (td, *J* = 7.6, 1.6 Hz, 1H), 7.34 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.29 – 7.20 (m, 6H), 4.14 (dd, *J* = 8.4, 7.2 Hz, 1H), 3.06 (ddd, *J* = 94.0, 17.2, 6.8 Hz, 2H), 2.16 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 205.7, 157.6, 150.0, 137.0, 131.6, 128.3, 127.9, 123.7, 123.6, 122.7, 87.4, 82.4, 60.7, 29.2, 21.5.

HRMS (ESI-TOF): exact mass calcd for $C_{17}H_{16}NO$ $(M+H)^+$ requires m/z 250.1226, found m/z 250.1221.

***N*,5-diphenyl-2-(pyridin-2-yl)pent-4-ynamide (1at)**



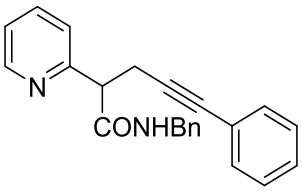
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 4/1) yielded **1at** (2.51 g, 77%) as a yellow solid, **M.P.** 173.4 - 175.3 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.74 (s, 1H), 8.65 (dt, *J* = 4.0, 1.6 Hz, 1H), 7.72 (td, *J* = 8.0, 2.0 Hz, 1H), 7.58 – 7.51 (m, 2H), 7.40 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.32 – 7.22 (m, 8H), 7.07 (tt, *J* = 7.6, 1.2 Hz, 1H), 4.05 (dd, *J* = 9.6, 6.0 Hz, 1H), 3.22 (ddd, *J* = 59.2, 16.8, 6.0 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 169.0, 157.9, 149.2, 138.2, 137.4, 131.7, 129.1, 128.3, 128.0, 124.3, 124.2, 123.5, 122.9, 120.0, 86.7, 83.1, 55.0, 24.7.

HRMS (ESI-TOF): exact mass calcd for $C_{22}H_{18}N_2O$ $(M+H)^+$ requires m/z 327.1492, found m/z 327.1494.

***N*-benzyl-5-phenyl-2-(pyridin-2-yl)pent-4-ynamide (1au)**



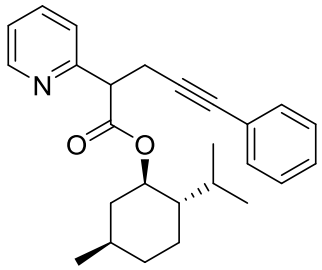
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 4/1) yielded **1au** (2.56 g, 75%) as a white solid, **M.P.** 124.7 - 125.2 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 – 8.49 (m, 1H), 7.66 (td, *J* = 7.6, 2.0 Hz, 2H), 7.38 (dt, *J* = 7.6, 1.2 Hz, 2H), 7.26 – 7.16 (m, 10H), 4.45 (d, *J* = 6.0 Hz, 2H), 3.98 (dd, *J* = 8.8, 6.8 Hz, 1H), 3.22 (ddd, *J* = 76.4, 16.8, 6.8 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0, 158.1, 149.2, 138.4, 137.1, 131.6, 128.6, 128.2, 127.9, 127.5, 127.3, 123.5, 123.5, 122.6, 87.1, 82.8, 54.4, 43.5, 24.1.

HRMS (ESI-TOF): exact mass calcd for $C_{23}H_{21}N_2O$ $(M+H)^+$ requires m/z 341.1648, found m/z 341.1647.

(1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1aw)



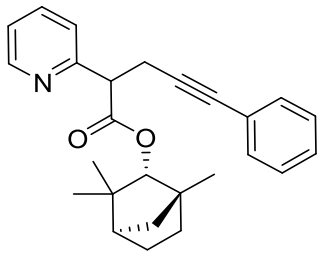
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1aw** (2.38 g, 61%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.39 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.85 – 7.80 (m, 3H), 7.60 – 7.55 (m, 1H), 7.54 – 7.48 (m, 2H), 7.45 (ddd, *J* = 8.8, 6.8, 0.8 Hz, 1H), 7.08 (td, *J* = 6.8, 1.2 Hz, 1H), 4.98 (td, *J* = 11.2, 4.4 Hz, 1H), 2.18 – 2.10 (m, 1H), 2.00 – 1.91 (m, 1H), 1.76 – 1.68 (m, 2H), 1.61 – 1.50 (m, 2H), 1.20 – 1.07 (m, 2H), 0.94 – 0.89 (m, 6H), 0.81 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 163.8, 140.1, 140.0, 131.6, 129.3, 129.2, 129.1, 128.5, 127.7, 122.6, 119.7, 115.3, 106.8, 74.0, 47.5, 41.5, 34.5, 31.6, 26.8, 24.0, 22.2, 20.8, 16.8.

HRMS (ESI-TOF): exact mass calcd for $C_{26}H_{31}NO_2Na$ $(M+Na)^+$ requires m/z 412.2247, found m/z 412.2241.

(1*R*,2*R*,4*R*)-1,3,3-trimethylbicyclo[2.2.1]heptan-2-yl5-phenyl-2-(pyridin-2-yl)pent -4-ynoate (1ax)



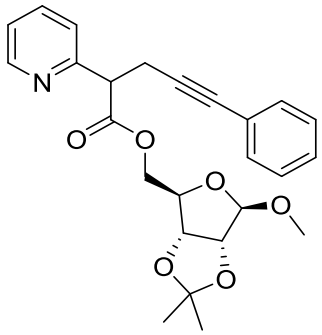
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 15/1) yielded **1ax** (2.44 g, 63%) as a brown gum.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 – 8.56 (m, 1H), 7.66 (tt, *J* = 7.6, 2.4 Hz, 1H), 7.39 (ddt, *J* = 10.0, 7.6, 1.2 Hz, 1H), 7.31 – 7.26 (m, 2H), 7.25 – 7.21 (m, 3H), 7.21 – 7.17 (m, 1H), 4.40 (dd, *J* = 18.8, 2.0 Hz, 1H), 4.15 (td, *J* = 7.2, 1.6 Hz, 1H), 3.31 – 3.07 (m, 2H), 1.69 – 1.51 (m, 4H), 1.42 – 1.34 (m, 1H), 1.13 (ddd, *J* = 9.6, 7.6, 1.2 Hz, 1H), 1.10 – 0.95 (m, 6H), 0.80 (d, *J* = 45.6 Hz, 3H), 0.51 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 171.8, 157.5, 157.4, 149.6, 149.5, 136.4, 131.6, 128.1, 127.7, 123.6, 123.6, 123.4, 123.2, 122.5, 122.4, 87.4, 87.3, 87.1, 86.9, 82.1, 53.4, 53.3, 48.5, 48.4, 48.3, 41.3, 41.3, 39.5, 39.4, 29.6, 29.6, 26.5, 26.5, 25.8, 25.7, 22.1, 22.0, 20.2, 19.9, 19.3, 19.2.

HRMS (ESI-TOF): exact mass calcd for $C_{26}H_{30}NO_2$ $(M+H)^+$ requires m/z 388.2271, found m/z 388.2267.

((3a*R*,4*R*,6*R*,6a*R*)-6-methoxy-2,2-dimethyltetrahydrofuro[3,4-*d*][1,3]dioxol-4-yl)methyl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1ay)



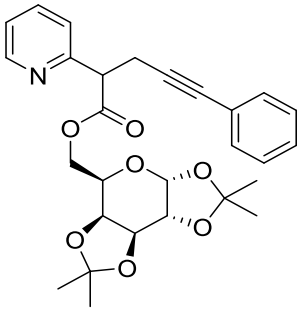
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 4/1) yielded **1ay** (2.62 g, 60%) as a yellow gum.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (dq, *J* = 4.8, 1.6 Hz, 1H), 7.67 (tt, *J* = 7.6, 2.0 Hz, 1H), 7.40 – 7.36 (m, 1H), 7.32 – 7.27 (m, 2H), 7.26 – 7.18 (m, 4H), 4.92 (d, *J* = 4.8 Hz, 1H), 4.59 – 4.48 (m, 2H), 4.38 – 4.31 (m, 1H), 4.26 – 4.08 (m, 4H), 3.26 – 3.19 (m, 5H), 3.08 (ddd, *J* = 16.8, 8.0, 3.6 Hz, 1H), 1.44 (s, 3H), 1.25 (d, *J* = 4.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 171.2, 157.2, 157.2, 149.8, 136.7, 131.7, 128.3, 127.9, 123.5, 123.5, 122.7, 122.7, 112.6, 109.6, 109.5, 85.3, 85.2, 84.1, 82.5, 81.9, 65.4, 65.3, 55.0, 54.9, 53.0, 53.0, 26.5, 26.5, 25.0, 22.5.

HRMS (ESI-TOF): exact mass calcd for $C_{25}H_{27}NO_6Na$ $(M+Na)^+$ requires m/z 460.1736, found m/z 460.1740.

((3a*R*,5*R*,5a*S*,8a*S*,8b*R*)-2,2,7,7-tetramethyltetrahydro-5*H*-bis([1,3]dioxolo)[4,5-*b*:4',5'-*d*] pyran-5-yl)methyl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1az)



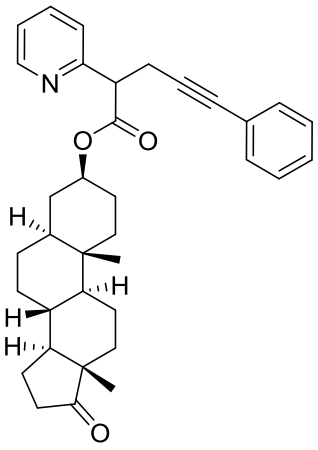
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 4/1) yielded **1az** (3.06 g, 60%) as a yellow gum.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 – 8.55 (m, 1H), 7.69 – 7.63 (m, 1H), 7.40 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.31 – 7.26 (m, 2H), 7.26 – 7.16 (m, 4H), 5.49 (t, *J* = 5.2 Hz, 1H), 4.53 (td, *J* = 8.4, 2.8 Hz, 1H), 4.41 – 4.22 (m, 3H), 4.19 – 4.08 (m, 2H), 4.04 – 3.95 (m, 1H), 3.25 – 3.07 (m, 2H), 1.44 – 1.37 (m, 6H), 1.29 (dd, *J* = 8.8, 3.2 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 171.4, 157.2, 157.2, 149.7, 136.7, 136.6, 131.7, 131.7, 128.2, 127.9, 123.6, 123.6, 123.6, 122.6, 122.5, 109.6, 108.9, 108.8, 96.4, 96.3, 87.1, 87.1, 82.4, 82.4, 71.0, 70.9, 70.7, 70.7, 70.6, 66.0, 65.8, 64.1, 63.8, 53.0, 52.9, 26.1, 26.0, 25.1, 24.5, 22.5, 22.4.

HRMS (ESI-TOF): exact mass calcd for $C_{28}H_{38}NO_7$ $(M+H)^+$ requires m/z 494.2173, found m/z 494.2174.

(3*S*,5*S*,8*R*,9*S*,10*S*,13*S*,14*S*)-10,13-dimethyl-17-oxohexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 5-phenyl-2-(pyridin-2-yl)pent-4-ynoate (1aaa)



Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 8/1) yielded **1aaa** (3.57 g, 67%) as a white solid, **M.P.** 127.8 - 131.2 ℃.

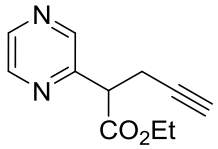
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (ddd, *J* = 4.8, 2.0, 1.2 Hz, 1H), 7.67 (td, *J* = 7.6, 2.0 Hz, 1H), 7.37 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.33 – 7.17 (m, 6H), 4.78 (hept, *J* = 4.8 Hz, 1H), 4.06 (t, *J* = 7.6 Hz, 1H), 3.12 (ddd, *J* = 51.6, 16.8, 7.6 Hz, 1H), 2.42 (dd, *J* = 19.2, 8.8 Hz, 1H), 2.06

(dt, *J* = 18.8, 9.2 Hz, 1H), 1.97 – 1.88 (m, 1H), 1.82 – 1.59 (m, 6H), 1.55 – 1.35 (m, 4H), 1.33 – 1.18 (m, 6H), 1.05 – 0.93 (m, 2H), 0.84 (s, 3H), 0.80 (s, 3H), 0.73 – 0.65 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 171.2, 157.5, 149.7, 136.6, 131.7, 128.2, 127.8, 123.7, 123.2, 123.2, 122.5, 87.3, 82.3, 74.5, 54.3, 53.3, 51.4, 47.9, 44.7, 44.7, 36.8, 36.7, 35.9, 35.7, 35.1, 34.0, 33.7, 31.6, 30.9, 28.3, 27.5, 27.2, 22.6, 22.6, 21.9, 20.5, 13.9, 12.3.

HRMS (ESI-TOF): exact mass calcd for $C_{35}H_{42}NO_3$ $(M+H)^+$ requires m/z 524.3159, found m/z 524.3152.

Ethyl 2-(pyrazin-2-yl)pent-4-ynoate (3c)



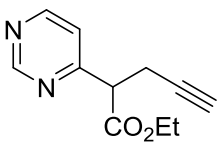
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 10/1) yielded **3c** (1.43 g, 70%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 (s, 1H), 8.50 (ddd, *J* = 18.0, 2.4, 1.2 Hz, 2H), 4.23 – 4.09 (m, 2H), 4.01 (ddd, *J* = 8.8, 6.4, 0.8 Hz, 1H), 2.94 (qddd, *J* = 42.4, 16.8, 6.4, 1.6 Hz, 2H), 1.92 (t, *J* = 2.8 Hz, 1H), 1.18 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 152.9, 145.1, 144.4, 143.7, 80.8, 70.7, 61.7, 50.2, 21.0, 14.1.

HRMS (ESI-TOF): exact mass calcd for $C_{11}H_{12}N_2O_2Na$ $(M+Na)^+$ requires m/z 227.0791, found m/z 227.0788.

Ethyl 2-(pyrimidin-4-yl)pent-4-ynoate (3d)



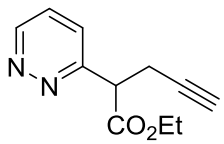
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 10/1) yielded **3d** (1.47 g, 72%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.14 (s, 1H), 8.68 (dt, *J* = 5.2, 1.2 Hz, 1H), 7.35 (dq, *J* = 5.2, 1.2 Hz, 1H), 4.23 – 4.10 (m, 2H), 3.92 (t, *J* = 7.6 Hz, 1H), 3.00 – 2.81 (m, 2H), 1.93 (t, *J* = 2.8 Hz, 1H), 1.18 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 165.3, 159.0, 157.3, 121.0, 80.5, 70.8, 61.8, 52.2, 20.8, 14.1.

HRMS (ESI-TOF): exact mass calcd for $C_{11}H_{12}N_2O_2Na$ $(M+Na)^+$ requires m/z 227.0791, found m/z 227.0790.

Ethyl 2-(pyridazin-3-yl)pent-4-ynoate (3e)



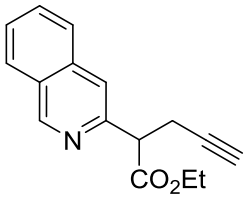
Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 8/1) yielded **3e** (1.41 g, 69%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.13 (dd, *J* = 4.8, 1.6 Hz, 1H), 7.60 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.47 (dd, *J* = 8.4, 4.8 Hz, 1H), 4.28 – 4.15 (m, 3H), 3.03 – 2.97 (m, 2H), 1.96 (t, *J* = 2.8 Hz, 1H), 1.22 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 159.9, 150.8, 126.7, 126.6, 80.5, 71.1, 61.8, 51.1, 21.6, 14.2.

HRMS (ESI-TOF): exact mass calcd for $C_{11}H_{13}N_2O_2$ $(M+H)^+$ requires m/z 205.0976, found m/z 205.0972.

Ethyl 2-(isoquinolin-3-yl)pent-4-ynoate (3g)



Follow the general procedure, product purification by column chromatography on silica gel (PE/EA = 10/1) yielded **3g** (1.90 g, 75%) as a yellow solid, **M.P.** 52.9 - 55.0 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 5.6 Hz, 1H), 8.28 (d, *J* = 8.8 Hz, 1H), 7.82 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.71 – 7.60 (m, 2H), 7.57 (dd, *J* = 5.6, 0.8 Hz, 1H), 4.87 (t, *J* = 7.2 Hz, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 3.14 (dddd, *J* = 110.8, 16.8, 8.0, 2.8 Hz, 2H), 1.90 (t, *J* = 2.8 Hz, 1H), 1.13 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 156.6, 142.1, 136.6, 130.1, 127.6, 127.6, 127.2, 124.9, 120.5, 82.0, 69.9, 61.4, 48.7, 21.1, 14.1.

HRMS (ESI-TOF): exact mass calcd for $C_{16}H_{16}NO_2$ $(M+H)^+$ requires m/z 254.1176, found m/z 254.1167.

3. Graphical Supporting Information for Electrochemistry enabled Intramolecular Dehydrogenative Aminooxygenation

Figure S5: (*Left*): General equipment for electrolysis. **(*Right*):** All reagents for this reaction.

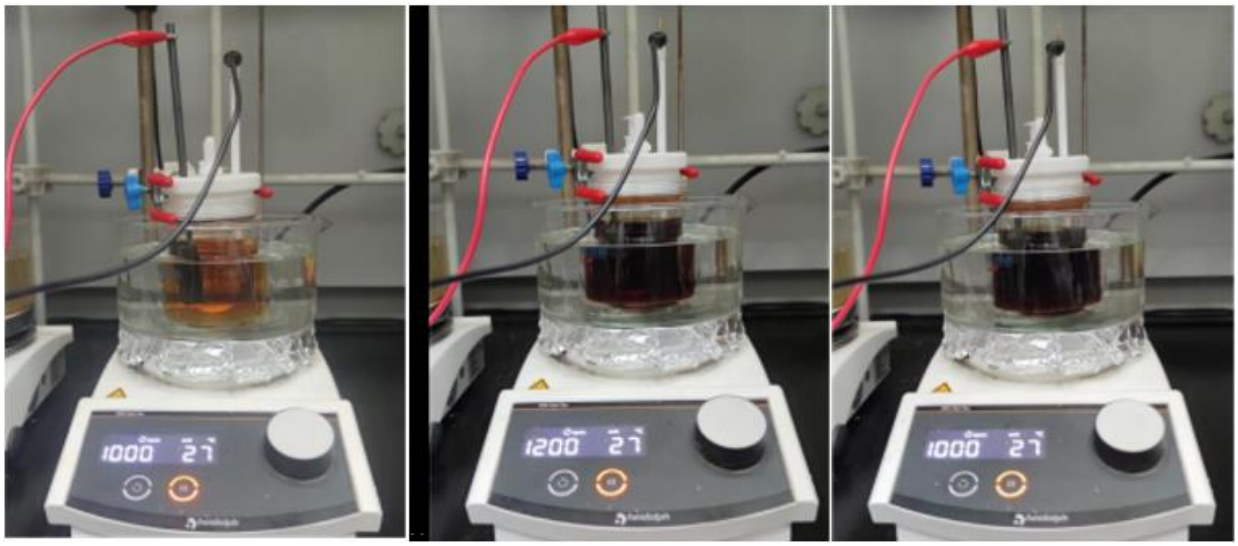


Figure S6: (*Left and Center*): The reaction mixture was subjected to constant current electrolysis (I = 20 mA). **(*Right*):** The reaction mixture after 36 h of electrolysis.

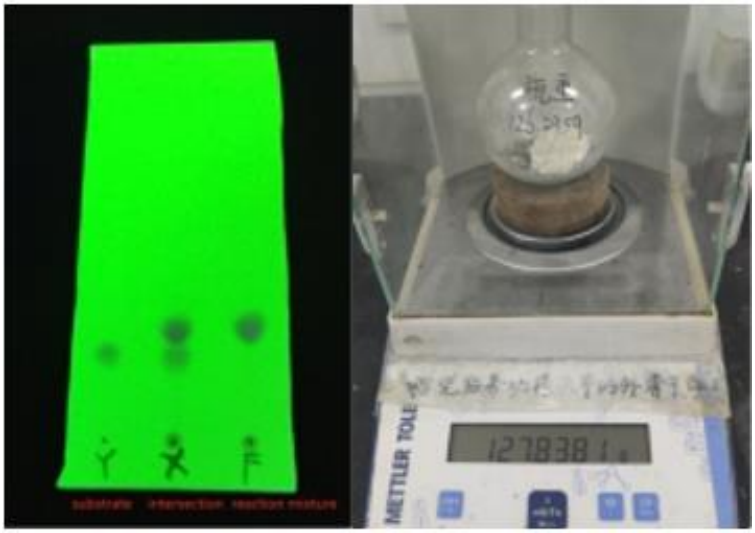
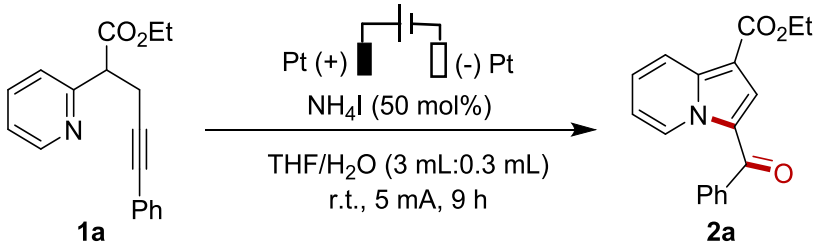


Figure S7: (*Left*): The reaction mixture after 36 h of TLC. **(*Right*):** Quality of separated products.

4. Optimization of the Reaction Conditions[a]



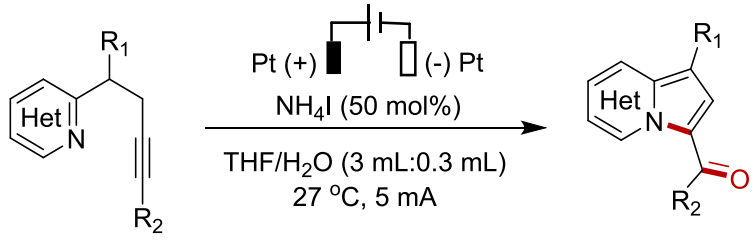
Entry	Variation from standard conditions above[a]	Yield (%)[b]
1	None	94(90)[c]
2	NH_4I (25 mol%)	75
3	Add $LiClO_4$ as electrolyte	37
4	Add $^nBu_4NPF_6$ as electrolyte	19
5	Without NH_4I	nr[d]
6	LiCl	<5
7	KBr	31
8	NaI	90
9	KI	91
10	DMMI	89
11	HI	80
12	MeOH (3 mL)	<5
13	MeCN (3 mL)	50
14	TFE (3 mL)	<5
15	HFIP (3 mL)	<5
16	DCE (3 mL)	<5
17	DMF (3 mL)	<5
18	THF (3 mL)	66
19	H_2O (3 mL)	16
20	THF/H_2O (2.5/0.5 mL)	75
21	THF/H_2O (1.5/1.5 mL)	56
22	4 mA, 10h	94

23	7 mA, 6h	94
24	graphite rod as anode	70
25	graphite felt as anode	76
26	Reticulated vitreous carbon as anode	85
27	Ni foam as cathode	20
28	Fe plate as cathode	26
29	graphite rod as cathode	30
30	Under N_2	92
31	No electric current	nr[d]

[a]Standard conditions: substrate **1a** (0.2 mmol), NH_4I (50 mol%), electrolyte (0.2 mmol) in THF/H_2O (3/0.3 mL), two platinum electrodes (each 15 × 10 × 0.2 mm^3), undivided cell, 27 ℃, 5 mA (j_{anode} = 1.67 mA·cm^{-2}), 9 h. [b]Yield determined by ^1H NMR analysis with CH_2Br_2 as an internal standard. [c]Isolated yield in parentheses. [d]nr: no reaction.

5. General procedure for Intramolecular Dehydrogenative Aminooxygenation

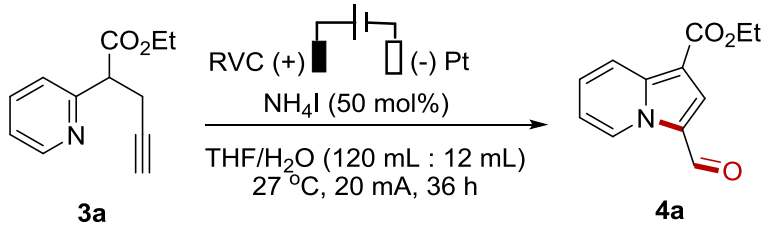
General Procedure A: Electrochemistry Enabled Intramolecular Dehydrogenative Aminooxygenation



The electrocatalysis was carried out in an undivided cell equipped with two platinum electrode (15 × 10 × 0.2 mm^3). The substrates **1** (0.2 mmol) and NH_4I (14.5 mg, 0.1 mmol, 50 mol%) were dissolved in the mixture solvent THF/H_2O (3/0.3 mL). The electrolysis was

carried out at 27 ℃ (oil bath temperature) using a constant current of 5.0 mA until complete consumption of the substrate (monitored by TLC or ^{1}H NMR analysis). After the reaction, the solvent was removed under reduced pressure. The resulting residue was chromatographed through silica gel eluting with PE/EA or DCM/MeOH to afford the corresponding product.

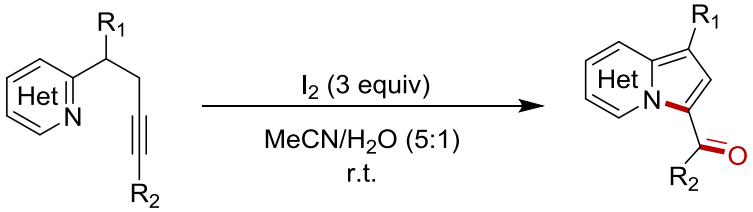
General Procedure for a Gram-Scale Experiment



3a **4a**

The gram scale reaction was conducted in a 125 mL Straight undivided five port electrolytic cell . The substrates **3a** (1.626 g, 8.0 mmol) and NH_4I (580 mg, 4.0 mmol, 0.5 equiv) were dissolved in the mixture solvent THF/H_2O (120/12 mL). The electrolysis was carried out at 27 ℃ (oil bath temperature) using a constant current of 20 mA for 36 hours (3.4 $F \cdot mol^{-1}$). After the reaction, the solvent was removed under reduced pressure. The residue was poured into a saturated aqueous solution of $Na_2S_2O_3$ and the product was then extracted with AcOEt (3 × 120 mL), dried over Na_2SO_4, and concentrated in vacuo. The resulting residue was chromatographed through silica gel eluting with PE/EA (15/1) to afford the corresponding product **4a** as white solid with 89% yield (1.54 g).

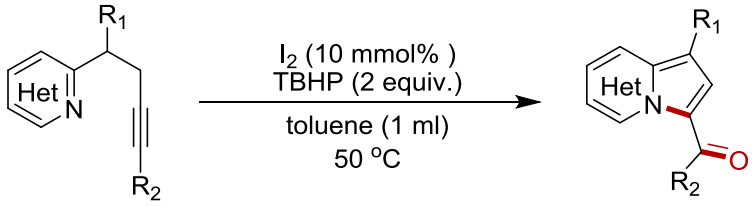
General Procedure B: I_2-Mediated Intramolecular Dehydrogenative Amino-oxygenation[5]



To a stirred solution of alkyne **1** (0.2 mmol, 1 equiv) in CH_3CN/ H_2O (2.5/0.5 mL) was

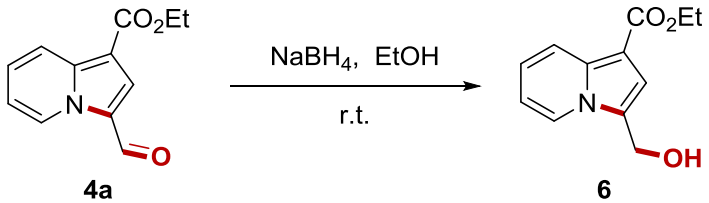
added iodine (152.3 mg, 0.6 mmol, 3.0 equiv) at room temperature for 12h. After the reaction was complete, the reaction mixture was concentrated under reduced pressure. The residue was diluted with dichloromethane, and washed with aqueous $Na_2S_2O_3$ and aqueous $NaHCO_3$. The aqueous layer was extracted with dichloromethane one more time. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuo. The resulting residue was chromatographed through silica gel eluting with PE/EA or DCM/MeOH to afford the corresponding product.

General Procedure C: I_2/TBHP Catalyzed Intramolecular Dehydrogenative Amino-oxygenation [12]



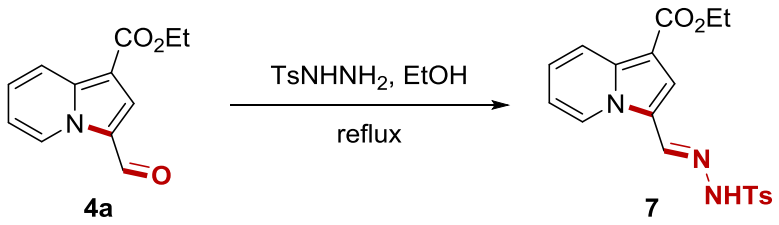
A 15 mL schlenk tube was equipped with a rubber septum and magnetic stir bar and was charged with a mixture of **1** (0.2 mmol), I_2 (5.1 mg, 0.02 mmol, 10 mol%) and TBHP (18.3 mg, 0.4 mmol, 2.0 equiv.) in toluene (1.0 mL) and was stirred at 50 °C for 12h. After the reaction was complete (as determined by TLC analysis), the reaction was cooled to room temperature and EtOAc (20 mL) was added to the solution and washed with brine, dried over Na_2SO_4, concentrated under reduced pressure. The resulting residue was chromatographed through silica gel eluting with PE/EA or DCM/MeOH to afford the corresponding product.

6. Experimental Procedure for the Transformations of the Product



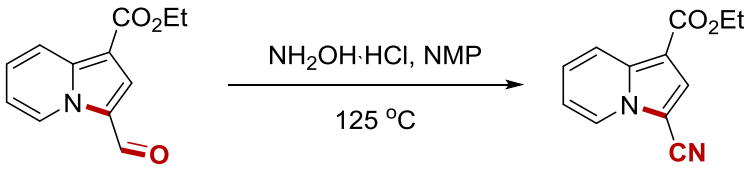
4a **6**

To a stirred solution of **4a** (43.5 mg, 0.2 mmol, 1.0 equiv) in EtOH (3 mL), $NaBH_4$ (7.6 mg, 0.2 mmol, 1.0 equiv) was added. Then the reaction was performed at r.t. for 0.5 h. The mixture was quenched with H_2O (2 mL), extracted with AcOEt (3 × 5mL). The organic layers were combined and dried over anhydrous Na_2SO_4. After filtration and removal of the solvents, the resulting residue was chromatographed through silica gel eluting with PE/EA (15/1) to afford the corresponding product **6** as white solid with 91% yield (39.9 mg).



4a 7

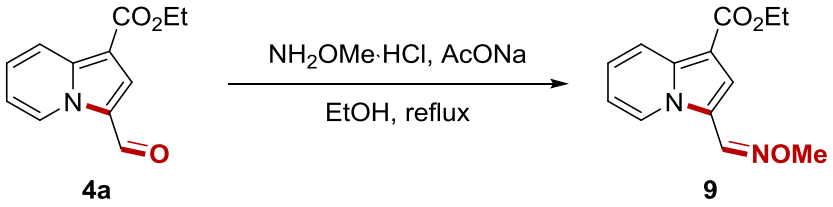
The reaction was performed in a 15 mL pressure tube, and **4a** (43.5 mg, 0.2 mmol, 1.0 equiv), p-methylbenzenesulfonyl hydrazine (41 mg, 0.22 mmol, 1.1 equiv) was dissolved in EtOH (3 mL), and the tube was sealed. The reaction mixture was stirred at 80 ℃ for 10 h (oil bath as the heat source). After cooling to room temperature, removal of the solvents, the resulting residue was chromatographed through silica gel eluting with DCM/MeOH (50/1) to afford the corresponding product **7** as brown solid with 85% yield (63.1 mg).



4a 8

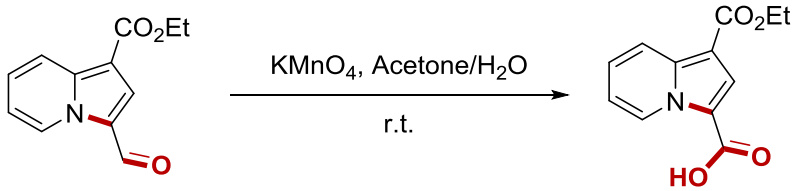
The reaction was performed in a 15 mL pressure tube, and **4a** (43.5 mg, 0.2 mmol, 1.0 equiv), hydroxylamine hydrochloride (16.7 mg, 0.22 mmol, 1.2 equiv) was dissolved in NMP (3 mL), and the tube was sealed. The reaction mixture was stirred at 125 ℃ for 16 h (oil bath as the heat source). After cooling to room temperature, the mixture was add with H_2O (2 mL), extracted with AcOEt (3 × 5 mL). The organic layers were combined and dried over

anhydrous Na_2SO_4. After filtration and removal of the solvents, the resulting residue was chromatographed through silica gel eluting with PE/EA (10/1) to afford the corresponding product **8** as white solid with 70% yield (30.0 mg).



4a **9**

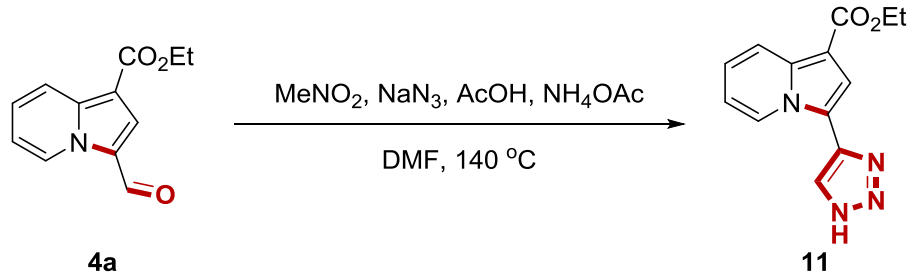
The reaction was performed in a 15 mL pressure tube, and **4a** (43.5 mg, 0.2 mmol, 1.0 equiv), methoxyamine hydrochloride (25.1 mg, 0.3 mmol, 1.5 equiv), sodium acetate anhydrous (26.3 mg, 0.32 mmol, 1.6 equiv) was dissolved in EtOH (3 mL), and the tube was sealed. The reaction mixture was stirred at 80 ℃ for 12 h (oil bath as the heat source). After cooling to room temperature, removal of the solvents, the resulting residue was chromatographed through silica gel eluting with PE/EA (10/1) to afford the corresponding product **9** as brown solid with 88% yield (43.3 mg).



4a **10**

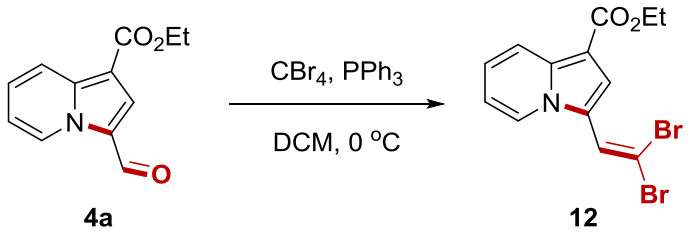
To a stirred solution of **4a** (43.5 mg, 0.2 mmol, 1.0 equiv) in Acetone (2 mL), slowly add Acetone/H_2O (2.5 mL/2.5 mL) solution of $KMnO_4$ (63.2 mg, 0.4 mmol, 2.0 equiv). Then the reaction was performed at r.t. for 12 h. The mixture was quenched with saturated $Na_2S_2O_3$ solution (2 ml), the generated MnO_2 is filtered with diatomite, and the filtrate is extracted with AcOEt (3 × 10 ml) extraction. The organic layers were combined and dried over anhydrous Na_2SO_4. After filtration and removal of the solvents, the resulting residue was chromatographed through silica gel eluting with DCM/MeOH (25/1) to afford the

corresponding product **10** as white solid with 83% yield (38.7 mg).



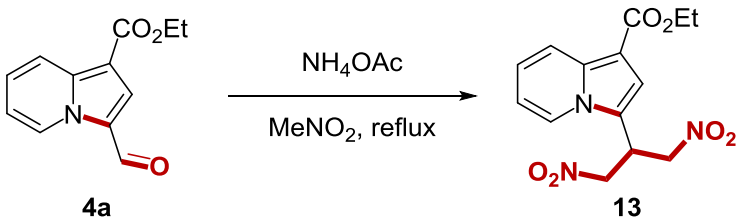
4a **11**

The reaction was performed in a 15 mL pressure tube, and **4a** (43.5 mg, 0.2 mmol, 1.0 equiv), sodium azide (26 mg, 0.4 mmol, 2.0 equiv), ammonium acetate (15.5 mg, 0.2 mmol, 1.0 equiv), nitromethane (21.7 μL, 0.4 mmol, 2.0 equiv), acetic acid (100.0 μL, 1.6 mmol, 8.0 equiv) was dissolved in DMF (3 mL), and the tube was sealed. The reaction mixture was stirred at 140 ℃ for 16 h (oil bath as the heat source). After cooling to room temperature, the mixture was add with H_2O (2 mL), extracted with AcOEt (3 × 5 mL). The organic layers were combined and dried over anhydrous Na_2SO_4. After filtration and removal of the solvents, the resulting residue was chromatographed through silica gel eluting with DCM/MeOH (50/1) to afford the corresponding product **11** as brown solid with 78% yield (40.0 mg).



4a **12**

To a stirred solution of **4a** (43.5 mg, 0.2 mmol, 1.0 equiv) in DCM (3 ml) at 0 ℃, slowly add CBr_4 (132.8 mg, 0.4 mmol, 2.0 equiv) and PPh_3 (210 mg, 0.8 mmol, 4.0 equiv). Then the reaction was performed at 0 ℃ for 2 h. After warming to room temperature, removal of the solvents, the resulting residue was chromatographed through neutral alumina eluting with PE/EA (20/1) to afford the corresponding product **12** as brown oil with 94% yield (70.1 mg).

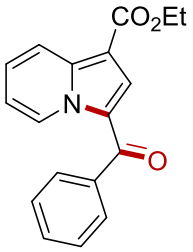


4a **13**

The reaction was performed in a 15 mL pressure tube, and **4a** (43.5 mg, 0.2 mmol, 1.0 equiv), ammonium acetate (23.2 mg, 0.3 mmol, 1.5 equiv) was dissolved in $MeNO_2$ (3 mL), and the tube was sealed. The reaction mixture was stirred at 105 ℃ for 10 h (oil bath as the heat source). After cooling to room temperature, the mixture was add with H_2O (2 mL), extracted with AcOEt (3 × 5 mL). The organic layers were combined and dried over anhydrous Na_2SO_4. After filtration and removal of the solvents, the resulting residue was chromatographed through silica gel eluting with DCM/MeOH (50/1) to afford the corresponding product **13** as brown solid with 72% yield (46.3 mg).

7. Characterization Data for Electrolysis Products

Ethyl 3-benzoylindolizine-1-carboxylate (2a)

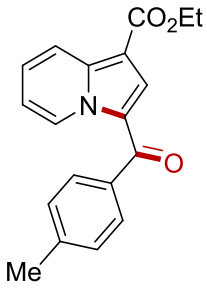


Following the general procedure A, the electrochemical reaction was carried out with **1a** (55.9 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2a** (52.8 mg, 90%) as a yellow solid. Spectral data matched those previously reported[4].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.37 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.90 – 7.73 (m, 3H), 7.59 – 7.53 (m, 1H), 7.52 – 7.46 (m, 2H), 7.42 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.06 (td, *J* = 7.2, 1.6 Hz, 1H), 4.36 (q, *J* = 6.8 Hz, 2H), 1.38 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.6, 164.1, 140.0, 139.9, 131.5, 129.2, 129.0, 128.5, 127.7, 122.6, 119.5, 115.3, 106.3, 60.2, 14.6.

Ethyl 3-(4-methylbenzoyl)indolizine-1-carboxylate (2b)

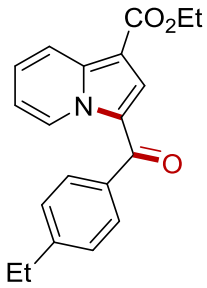


Following the general procedure A, the electrochemical reaction was carried out with **1b** (58.7 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2b** (43.6 mg, 71%) as a light yellow solid. Spectral data matched those previously reported[4].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.94 (dt, *J* = 1.2, 7.2 Hz, 1H), 8.38 (dt, *J* = 1.6, 8.8 Hz, 1H), 7.82 (s, 1H), 7.77 – 7.69 (m, 2H), 7.42 (ddd, *J* = 1.2, 6.8, 8.8 Hz, 1H), 7.35 – 7.28 (m, 2H), 7.06 (td, *J* = 1.6, 7.2 Hz, 1H), 4.37 (q, *J* = 7.2 Hz, 2H), 2.45 (s, 3H), 1.39 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.5, 164.2, 142.2, 139.9, 137.3, 129.3, 129.2, 128.8, 127.6, 122.7, 119.6, 115.3, 106.2, 60.2, 21.7, 14.7.

Ethyl 3-(4-ethylbenzoyl)indolizine-1-carboxylate (2c)



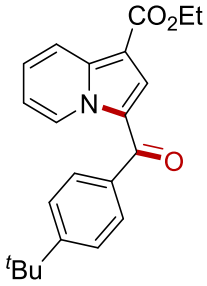
Following the general procedure A, the electrochemical reaction was carried out with **1c** (61.5 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2c** (51.4 mg, 80%) as a brown solid, **M.P.** 66.4 - 67.7 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.94 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.38 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.83 (s, 1H), 7.80 – 7.73 (m, 2H), 7.42 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.37 – 7.30 (m, 2H), 7.06 (td, *J* = 6.8, 1.6 Hz, 1H), 4.37 (q, *J* = 7.2 Hz, 2H), 2.75 (q, *J* = 7.6 Hz, 2H), 1.39 (t, *J* = 7.2 Hz, 3H), 1.30 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.6, 164.3, 148.4, 139.9, 137.5, 129.4, 129.3, 128.9, 128.0, 127.6, 122.8, 119.6, 115.2, 106.2, 60.2, 29.0, 15.4, 14.7.

HRMS (ESI-TOF): exact mass calcd for $C_{20}H_{19}NO_3Na$ $(M+Na)^+$ requires m/z 344.1263, found m/z 344.1257.

Ethyl 3-(4-(tert-butyl)benzoyl)indolizine-1-carboxylate (2d)



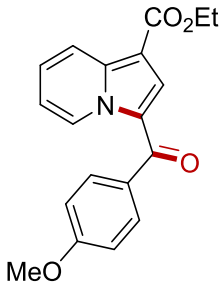
Following the general procedure A, the electrochemical reaction was carried out with **1d** (67.1 mg, 0.2 mmol) at 27 ℃ for 10 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2d** (51.7 mg, 74%) as a brown solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.38 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.86 (s, 1H), 7.57 – 7.49 (m, 2H), 7.42 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.06 (td, *J* = 7.2, 1.6 Hz, 1H), 4.38 (q, *J* = 7.2 Hz, 2H), 1.38 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.6, 164.3, 155.2, 139.9, 137.2, 129.3, 129.1, 128.9, 127.6, 125.5, 122.8, 119.6, 115.2, 106.2, 60.2, 35.1, 31.3, 14.7.

HRMS (ESI-TOF): exact mass calcd for $C_{22}H_{23}NO_3Na$ $(M+Na)^+$ requires m/z 372.1570, found m/z 372.1577.

Ethyl 3-(4-methoxybenzoyl)indolizine-1-carboxylate (2e)

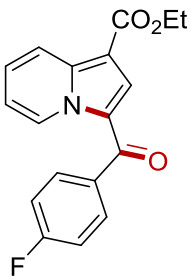


Following the general procedure A, the electrochemical reaction was carried out with **1e** (61.9 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 10/1) yielded **2e** (22.6 mg, 35%) as a yellow solid. Spectral data matched those previously reported[4].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.90 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.38 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.91 – 7.80 (m, 3H), 7.43 (ddd, *J* = 9.2, 7.2, 1.2 Hz, 1H), 7.06 (td, *J* = 7.2, 1.6 Hz, 1H), 7.04 – 7.00 (m, 2H), 4.39 (d, *J* = 7.2 Hz, 2H), 3.91 (s, 3H), 1.41 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 184.7, 164.3, 162.7, 139.8, 132.5, 131.4, 129.2, 128.5, 127.5, 122.8, 119.6, 115.2, 113.9, 106.1, 60.2, 55.6, 14.7.

Ethyl 3-(4-fluorobenzoyl)indolizine-1-carboxylate (2f)



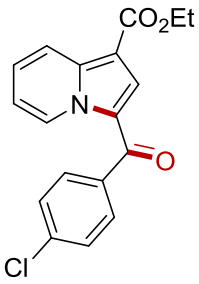
Following the general procedure A, the electrochemical reaction was carried out with **1f** (59.5 mg, 0.2 mmol) at 27 ℃ for 12 h. Purification by column chromatography on silica gel (PE/ EA = 10/1) yielded **2f** (52.3 mg, 84%) as a light yellow solid. Spectral data matched those previously reported[5].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.91 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.38 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.90 – 7.81 (m, 2H), 7.77 (s, 1H), 7.44 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.23 – 7.15 (m, 2H), 7.07 (td, *J* = 6.8, 1.2 Hz, 1H), 4.37 (q, *J* = 7.2 Hz, 2H), 1.39 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 184.1, 164.9 (d, $^{1}J_{C\text{-}F}$ = 251.0 Hz), 164.1, 140.0, 136.2, 136.1, 131.4 (d, $^{4}J_{C\text{-}F}$ = 4.0 Hz), 129.0 (d, $^{2}J_{C\text{-}F}$ = 42.0 Hz), 127.9, 122.4, 119.6, 115.7, 115.5 (d, $^{3}J_{C\text{-}F}$ = 6.0 Hz), 106.5, 60.3, 14.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ -107.7.

Ethyl 3-(4-chlorobenzoyl)indolizine-1-carboxylate (2g)

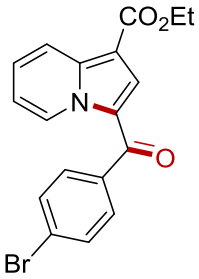


Following the general procedure A, the electrochemical reaction was carried out with **1g** (62.8 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 10/1) yielded **2g** (52.4 mg, 80%) as a yellow solid. Spectral data matched those previously reported[4].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.92 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.39 (dt, *J* = 9.0, 1.2 Hz, 1H), 7.80 – 7.73 (m, 3H), 7.53 – 7.42 (m, 3H), 7.09 (td, *J* = 7.2, 1.6 Hz, 1H), 4.37 (q, *J* = 7.2 Hz, 2H), 1.39 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 184.2, 164.1, 140.1, 138.3, 137.9, 130.5, 129.3, 128.9, 128.8, 128.0, 122.3, 119.7, 115.6, 106.6, 60.3, 14.7.

Ethyl 3-(4-bromobenzoyl)indolizine-1-carboxylate (2h)

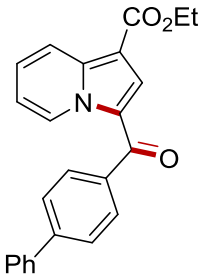


Following the general procedure A, the electrochemical reaction was carried out with **1h** (71.7 mg, 0.2 mmol) at 27 ℃ for 7 h. Purification by column chromatography on silica gel (PE/ EA = 10/1) yielded **2h** (64.8 mg, 87%) as a yellow solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.91 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.38 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.76 (s, 1H), 7.71 – 7.60 (m, 4H), 7.45 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.08 (td, *J* = 7.2, 1.6 Hz, 1H), 4.37 (q, *J* = 7.2 Hz, 2H), 1.39 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 184.2, 164.0, 140.1, 138.7, 131.8, 130.6, 129.3, 128.9, 128.0, 126.3, 122.2, 119.6, 115.6, 106.6, 60.3, 14.6.

Ethyl 3-([1,1'-biphenyl]-4-carbonyl)indolizine-1-carboxylate (2i)



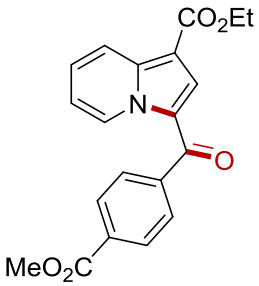
Following the general procedure A, the electrochemical reaction was carried out with **1i** (71.1 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel

(PE/ EA = 15/1) yielded **2i** (73.1 mg, 99%) as a yellow solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.40 (dt, *J* = 8.8, 1.6 Hz, 1H), 7.94 – 7.88 (m, 3H), 7.76 – 7.71 (m, 2H), 7.69 – 7.65 (m, 2H), 7.51 – 7.38 (m, 4H), 7.09 (td, *J* = 7.2, 1.2 Hz, 1H), 4.39 (q, *J* = 6.8 Hz, 2H), 1.41 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.2, 164.2, 144.4, 140.2, 140.0, 138.7, 129.7, 129.3, 129.0, 128.9, 128.1, 127.8, 127.4, 127.2, 122.7, 119.6, 115.4, 106.4, 60.2, 14.7.

Ethyl 3-(4-(methoxycarbonyl)benzoyl)indolizine-1-carboxylate (2j)

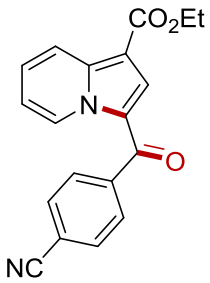


Following the general procedure A, the electrochemical reaction was carried out with **1j** (67.5 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 10/1) yielded **2j** (47.5 mg, 81%) as a yellow solid. Spectral data matched those previously reported[5].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.99 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.42 (dt, *J* = 8.8, 1.2 Hz, 1H), 8.21 – 8.16 (m, 2H), 7.88 – 7.84 (m, 2H), 7.77 (s, 1H), 7.49 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.13 (td, *J* = 6.8, 1.2 Hz, 1H), 4.38 (q, *J* = 7.2 Hz, 2H), 3.98 (s, 3H), 1.39 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 184.7, 166.6, 164.1, 143.9, 140.3, 132.7, 129.8, 129.4, 129.3, 129.0, 128.3, 122.4, 119.8, 115.8, 106.9, 60.4, 52.6, 14.7.

Ethyl 3-(4-cyanobenzoyl)indolizine-1-carboxylate (2k)

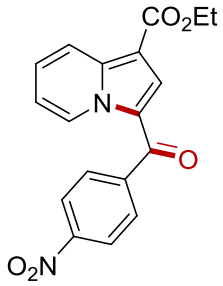


Following the general procedure A, the electrochemical reaction was carried out with **1k** (60.9 mg, 0.2 mmol) at 27 ℃ for 8 h. Purification by column chromatography on silica gel (PE/ EA = 10/1) yielded **2k** (43.3 mg, 68%) as a yellow solid. Spectral data matched those previously reported[5].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.41 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.91 – 7.86 (m, 2H), 7.83 – 7.79 (m, 2H), 7.72 (s, 1H), 7.50 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.14 (td, *J* = 6.8, 1.6 Hz, 1H), 4.37 (q, *J* = 7.2 Hz, 2H), 1.39 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 183.3, 163.8, 143.8, 140.4, 132.4, 129.4, 129.4, 129.2, 128.6, 121.9, 119.8, 118.3, 116.0, 114.9, 107.2, 60.4, 14.6.

Ethyl 3-(4-nitrobenzoyl)indolizine-1-carboxylate (2l)

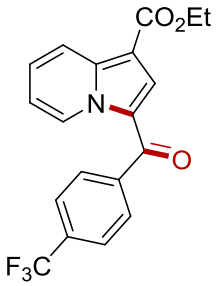


Following the general procedure A, the electrochemical reaction was carried out with **1l** (64.9 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 8/1) yielded **2l** (27.1 mg, 40%) as a yellow solid. Spectral data matched those previously reported[5].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.42 (dt, *J* = 8.8, 1.2 Hz, 1H), 8.39 – 8.33 (m, 2H), 7.97 – 7.91 (m, 2H), 7.72 (s, 1H), 7.52 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.15 (td, *J* = 6.8, 1.2 Hz, 1H), 4.37 (q, *J* = 7.2 Hz, 2H), 1.39 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 183.0, 163.8, 149.5, 145.4, 140.5, 129.8, 129.4, 129.3, 128.7, 123.8, 122.0, 119.8, 116.1, 107.3, 60.4, 14.6.

Ethyl 3-(4-(trifluoromethyl)benzoyl)indolizine-1-carboxylate (2m)



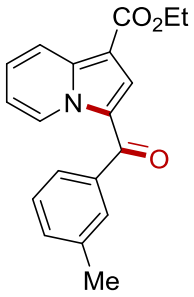
Following the general procedure A, the electrochemical reaction was carried out with **1m** (69.5 mg, 0.2 mmol) at 27 ℃ for 7 h. Purification by column chromatography on silica gel (PE/ EA = 8/1) yielded **2m** (59.3 mg, 82%) as a yellow solid. Spectral data matched those previously reported[12].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.40 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.93 – 7.87 (m, 2H), 7.81 – 7.72 (m, 3H), 7.48 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.11 (td, *J* = 6.8, 1.2 Hz, 1H), 4.37 (q, *J* = 7.2 Hz, 2H), 1.39 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 184.0, 163.9, 143.2, 140.3, 133.1 (q, $^{2}J_{C\text{-}F}$ = 32.0 Hz), 129.3 (q, $^{3}J_{C\text{-}F}$ = 15.0 Hz), 129.3, 128.3, 125.6 (q, $^{4}J_{C\text{-}F}$ = 4.0 Hz), 123.9 (q, $^{1}J_{C\text{-}F}$ = 271.0 Hz), 122.2, 119.7, 115.8, 107.0, 60.4, 14.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.9.

Ethyl 3-(3-methylbenzoyl)indolizine-1-carboxylate (2n)

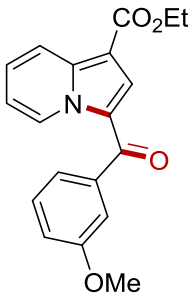


Following the general procedure A, the electrochemical reaction was carried out with **1n** (58.7 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2n** (53.5 mg, 87%) as a yellow solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.38 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.81 (s, 1H), 7.64 – 7.56 (m, 2H), 7.43 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.40 – 7.35 (m, 2H), 7.07 (td, *J* = 7.2, 1.6 Hz, 1H), 4.37 (q, *J* = 7.2 Hz, 2H), 2.44 (s, 3H), 1.39 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.9, 164.2, 140.1, 139.9, 138.4, 132.3, 129.5, 129.3, 129.1, 128.3, 127.7, 126.3, 122.7, 119.6, 115.3, 106.3, 60.2, 21.5, 14.6.

Ethyl 3-(3-methoxybenzoyl)indolizine-1-carboxylate (2o)



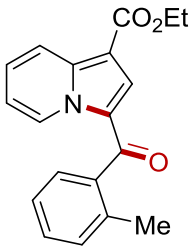
Following the general procedure A, the electrochemical reaction was carried out with **1o** (65.1 mg, 0.2 mmol) at 27 ℃ for 8.5 h. Purification by column chromatography on silica gel

(PE/ EA = 10/1) yielded **2o** (53.0 mg, 82%) as a light yellow solid. Spectral data matched those previously reported[4].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.40 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.84 (s, 1H), 7.48 – 7.36 (m, 3H), 7.34 (dd, *J* = 2.8, 1.2 Hz, 1H), 7.14 – 7.07 (m, 2H), 4.38 (q, *J* = 7.2 Hz, 2H), 3.88 (s, 3H), 1.40 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.5, 164.2, 159.7, 141.3, 140.1, 129.5, 129.4, 129.2, 127.9, 122.6, 121.7, 119.7, 117.8, 115.5, 113.8, 106.4, 60.3, 55.6, 14.7.

Ethyl 3-(2-methylbenzoyl)indolizine-1-carboxylate (2p)

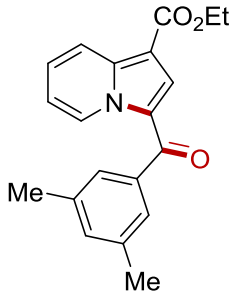


Following the general procedure A, the electrochemical reaction was carried out with **1p** (58.7 mg, 0.2 mmol) at 27 ℃ for 16 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2p** (32.0 mg, 52%) as a yellow solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.07 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.40 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.54 (s, 1H), 7.47 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.42 – 7.36 (m, 2H), 7.34 – 7.26 (m, 2H), 7.11 (td, *J* = 7.2, 1.6 Hz, 1H), 4.35 (q, *J* = 7.2 Hz, 2H), 2.38 (s, 3H), 1.37 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 187.7, 164.1, 140.2, 139.9, 136.1, 131.0, 129.9, 129.5, 129.5, 128.2, 128.0, 125.4, 123.5, 119.7, 115.6, 106.5, 60.2, 19.7, 14.7.

Ethyl 3-(3,5-dimethylbenzoyl)indolizine-1-carboxylate (2q)

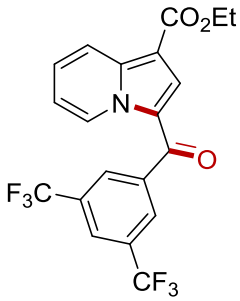


Following the general procedure A, the electrochemical reaction was carried out with **1q** (61.5 mg, 0.2 mmol) at 27 ℃ for 8.5 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2q** (51.4 mg, 80%) as a yellow solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.37 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.81 (s, 1H), 7.46 – 7.38 (m, 3H), 7.20 (s, 1H), 7.06 (td, *J* = 6.8, 1.6 Hz, 1H), 4.38 (q, *J* = 6.8 Hz, 2H), 2.40 (s, 6H), 1.40 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 186.2, 164.2, 140.2, 139.9, 138.1, 133.2, 129.3, 129.1, 127.6, 126.8, 122.8, 119.6, 115.3, 106.2, 60.2, 21.4, 14.6.

Ethyl 3-(3,5-bis(trifluoromethyl)benzoyl)indolizine-1-carboxylate (2r)



Following the general procedure A, the electrochemical reaction was carried out with **1r** (83.1 mg, 0.2 mmol) at 27 ℃ for 6.5 h. Purification by column chromatography on silica gel (PE/ EA = 8/1) yielded **2r** (70.4 mg, 82%) as a brown solid, **M.P.** 150.4 - 152.5 ℃.

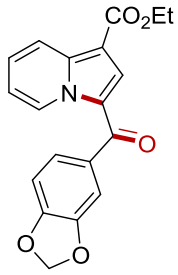
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.94 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.43 (dt, *J* = 9.2, 4.2 Hz, 1H), 8.27 – 8.22 (m, 2H), 8.14 – 8.04 (m, 1H), 7.70 (s, 1H), 7.53 (ddd, *J* = 10.4, 6.8, 1.2 Hz, 1H), 7.16 (td, *J* = 7.2, 1.6 Hz, 1H), 4.38 (q, *J* = 7.2 Hz, 2H), 1.39 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 181.8, 163.7, 141.9, 140.6, 132.1 (q, $^{2}J_{C\text{-}F}$ = 33.0 Hz), 129.4, 129.1, 129.0 (q, $^{3}J_{C\text{-}F}$ = 28.0 Hz), 128.8, 124.9 (quint, $^{4}J_{C\text{-}F}$ = 4.0 Hz), 123.1 (q, $^{1}J_{C\text{-}F}$ = 272.0 Hz), 121.6, 119.9, 116.2, 107.6, 60.5, 14.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ -107.7.

HRMS (ESI-TOF): exact mass calcd for $C_{20}H_{13}F_6NO_3Na$ $(M+Na)^+$ requires m/z 452.0692, found m/z 452.0684.

Ethyl 3-(benzo[*d*][1,3]dioxole-5-carbonyl)indolizine-1-carboxylate (2s)



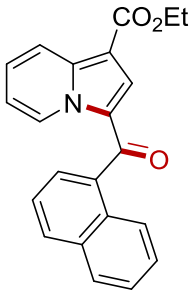
Following the general procedure A, the electrochemical reaction was carried out with **1s** (64.7 mg, 0.2 mmol) at 27 ℃ for 15 h. Purification by column chromatography on silica gel (PE/ EA = 8/1) yielded **2s** (39.1 mg, 58%) as a yellow solid, **M.P.** 118.8 - 120.9 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.38 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.83 (s, 1H), 7.46 – 7.39 (m, 2H), 7.33 (d, *J* = 1.6 Hz, 1H), 7.06 (td, *J* = 6.8, 1.6 Hz, 1H), 6.92 (d, *J* = 8 Hz, 1H), 6.07 (s, 2H), 4.38 (q, *J* = 7.2 Hz, 2H), 1.41 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 184.2, 164.3, 150.8, 148.0, 139.9, 134.2, 129.2, 128.5, 127.6, 124.9, 122.6, 119.6, 115.2, 109.4, 108.1, 106.2, 101.9, 60.2, 14.7.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{15}NO_5Na$ $(M+Na)^+$ requires m/z 360.0842, found m/z 360.0835.

Ethyl 3-(1-naphthoyl)indolizine-1-carboxylate (2t)

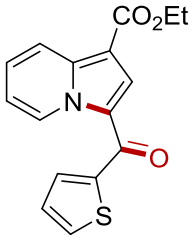


Following the general procedure A, the electrochemical reaction was carried out with **1t** (65.9 mg, 0.2 mmol) at 27 ℃ for 12 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2t** (43.3 mg, 63%) as a yellow solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.19 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.43 (dt, *J* = 9.2, 1.2 Hz, 1H), 8.16 – 8.08 (m, 1H), 8.00 (dt, *J* = 8.4, 1.2 Hz, 1H), 7.95 – 7.90 (m, 1H), 7.67 (dd, *J* = 6.8, 1.2 Hz, 1H), 7.61 – 7.47 (m, 5H), 7.16 (td, *J* = 6.8, 1.6 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 1.34 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 186.8, 164.1, 140.3, 137.6, 133.9, 130.9, 130.6, 129.7, 129.6, 128.5, 128.2, 127.2, 126.7, 126.5, 125.5, 124.6, 124.0, 119.7, 115.7, 106.6, 77.5, 77.2, 76.8, 60.2, 14.6.

Ethyl 3-(thiophene-2-carbonyl)indolizine-1-carboxylate (2u)

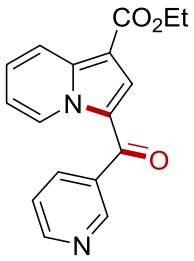


Following the general procedure A, the electrochemical reaction was carried out with **1u** (57.1 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2u** (44.3 mg, 74%) as a yellow solid. Spectral data matched those previously reported[4].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.84 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.38 (dt, *J* = 9.2, 1.2 Hz, 1H), 8.14 (s, 1H), 7.82 (dd, *J* = 3.6, 1.2 Hz, 1H), 7.67 (dd, *J* = 4.8, 1.2 Hz, 1H), 7.43 (ddd, *J* = 9.2, 6.8, 1.2 Hz, 1H), 7.20 (dd, *J* = 5.2, 4.0 Hz, 1H), 7.06 (td, *J* = 1.2, 6.8 Hz, 1H), 4.41 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 164.2, 144.4, 140.0, 132.2, 132.1, 129.1, 127.9, 127.7, 127.4, 122.3, 119.7, 115.3, 106.5, 60.3, 14.7.

Ethyl 3-nicotinoylindolizine-1-carboxylate (2v)

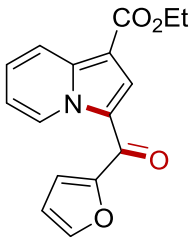


Following the general procedure A, the electrochemical reaction was carried out with **1v** (56.1 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 5/1) yielded **2v** (51.8 mg, 88%) as a brown solid. Spectral data matched those previously reported[5].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.93 (dd, *J* = 7.2, 1.2 Hz, 1H), 9.01 (d, *J* = 2.0 Hz, 1H), 8.77 (dd, *J* = 4.8, 1.6 Hz, 1H), 8.38 (dt, *J* = 8.8, 1.6 Hz, 1H), 8.08 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.77 (s, 1H), 7.40 – 7.50 (m, 2H), 7.09 (td, *J* = 6.8, 1.6 Hz, 1H), 4.34 (q, *J* = 7.2 Hz, 2H), 1.36 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 182.9, 163.8, 152.2, 149.8, 140.3, 136.4, 135.6, 129.3, 129.1, 128.4, 123.5, 122.2, 119.7, 115.8, 107.0, 60.3, 14.6.

Ethyl 3-(furan-2-carbonyl)indolizine-1-carboxylate (2w)



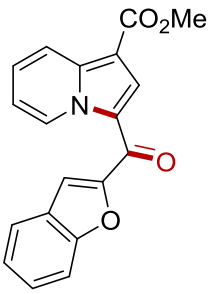
Following the general procedure A, the electrochemical reaction was carried out with **1w** (53.9 mg, 0.2 mmol) at 27 ℃ for 6.5 h. Purification by column chromatography on silica gel (PE/ EA = 10/1) yielded **2w** (41.4 mg, 73%) as a brown solid, **M.P.** 110.0 - 112.5 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.01 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.49 (s, 1H), 8.38 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.69 (dd, *J* = 2.0, 0.8 Hz, 1H), 7.42 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.34 (dd, *J* = 3.6, 0.8 Hz, 1H), 7.04 (td, *J* = 7.2, 1.6 Hz, 1H), 6.60 (q, *J* = 1.6 Hz, 1H), 4.41 (q, *J* = 7.2 Hz, 2H), 1.44 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 164.3, 153.4, 145.9, 139.8, 129.4, 127.8, 127.7, 121.5, 119.6, 117.7, 115.4, 112.2, 106.9, 60.3, 14.7.

HRMS (ESI-TOF): exact mass calcd for $C_{16}H_{13}NO_4Na$ $(M+Na)^+$ requires m/z 306.0737, found m/z 306.0732.

Ethyl 3-(benzofuran-2-carbonyl)indolizine-1-carboxylate (2x)

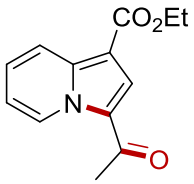


Following the general procedure A, the electrochemical reaction was carried out with **1x** (66.1 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2x** (51.1 mg, 80%) as a yellow solid. Spectral data matched those previously reported[13].

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.07 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.65 (s, 1H), 8.42 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.74 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.69 (dq, *J* = 8.4, 0.8 Hz, 1H), 7.66 (d, *J* = 0.8 Hz, 1H), 7.48 (dddd, *J* = 8.4, 7.2, 2.0, 1.2 Hz, 2H), 7.33 (ddd, *J* = 8.0, 7.2, 0.8 Hz, 1H), 7.11 (td, *J* = 7.2, 1.6 Hz, 1H), 3.97 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 164.6, 155.8, 153.8, 140.2, 129.6, 128.4, 128.3, 127.7, 127.3, 124.0, 123.0, 122.0, 119.7, 115.7, 113.5, 112.5, 106.9, 51.5.

Ethyl 3-acetylindolizine-1-carboxylate (2y)

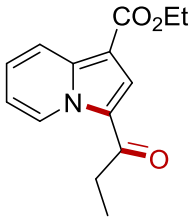


Following the general procedure A, the electrochemical reaction was carried out with **1y** (43.5 mg, 0.2 mmol) at 27 ℃ for 7 h. Purification by column chromatography on silica gel (PE/ EA = 20/1) yielded **2y** (32.8 mg, 71%) as a brown solid. Spectral data matched those previously reported[4].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.32 (dq, *J* = 9.2, 1.2 Hz, 1H), 7.98 (d, *J* = 2.0 Hz, 1H), 7.38 (ddt, *J* = 8.8, 7.2, 1.2 Hz, 1H), 7.00 (td, *J* = 6.8, 1.6 Hz, 1H), 4.39 (q, *J* = 7.2 Hz, 2H), 2.58 (s, 3H), 1.42 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 187.9, 164.2, 139.4, 129.2, 127.3, 126.3, 122.8, 119.5, 115.3, 105.8, 60.2, 27.4, 14.7.

Ethyl 3-propionylindolizine-1-carboxylate (2z)



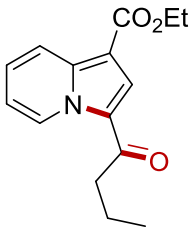
Following the general procedure A, the electrochemical reaction was carried out with **1z** (46.3 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 20/1) yielded **2z** (42.2 mg, 86%) as a brown solid, **M.P.** 112.8 - 115.1 ℃.

^{1}H NMR (600 MHz, $CDCl_3$) δ 9.91 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.32 (dt, *J* = 9.0, 1.2 Hz, 1H), 8.00 (s, 1H), 7.37 (ddd, *J* = 9.0, 7.2, 1.2 Hz, 1H), 7.00 (td, *J* = 6.6, 1.2 Hz, 1H), 4.39 (q, *J* = 7.2 Hz, 2H), 2.95 (q, *J* = 7.8 Hz, 2H), 1.42 (t, *J* = 7.2 Hz, 3H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$) δ 191.6, 164.3, 139.3, 129.2, 127.2, 125.5, 122.4, 119.5, 115.2, 105.7, 60.2, 32.6, 14.7, 9.4.

HRMS (ESI-TOF): exact mass calcd for $C_{14}H_{15}NO_3Na$ $(M+Na)^+$ requires m/z 268.0950, found m/z 268.0943.

Ethyl 3-butyrylindolizine-1-carboxylate (2aa)



Following the general procedure A, the electrochemical reaction was carried out with **1aa** (49.1 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 20/1) yielded **2aa** (42.5 mg, 82%) as a brown solid, **M.P.** 77.9 - 79.1 ℃.

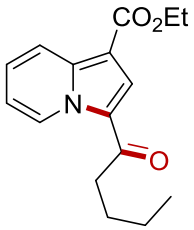
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.91 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.31 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.99 (s, 1H), 7.36 (ddd, *J* = 8.4, 6.8, 1.2 Hz, 1H), 6.99 (td, *J* = 6.8, 1.2 Hz, 1H), 4.38 (q, *J* =

7.2 Hz, 2H), 2.88 (t, *J* = 7.2 Hz, 2H), 1.80 (hept, *J* = 7.2 Hz, 2H), 1.42 (t, *J* = 7.2 Hz, 3H), 1.01 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 191.0, 164.2, 139.3, 129.2, 127.2, 125.6, 122.8, 119.5, 115.2, 105.7, 60.2, 41.4, 19.0, 14.7, 14.1.

HRMS (ESI-TOF): exact mass calcd for $C_{15}H_{17}NO_3Na$ $(M+Na)^+$ requires m/z 282.1106, found m/z 282.1104.

Ethyl 3-pentanoylindolizine-1-carboxylate (2ab)

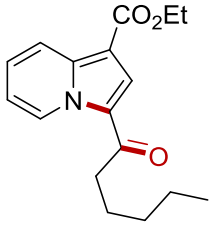


Following the general procedure A, the electrochemical reaction was carried out with **1ab** (49.1 mg, 0.2 mmol) at 27 ℃ for 10 h. Purification by column chromatography on silica gel (PE/ EA = 20/1) yielded **2ab** (49.2 mg, 90%) as a brown solid. Spectral data matched those previously reported[14].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.91 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.31 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.99 (s, 1H), 7.36 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 6.99 (td, *J* = 6.8, 1.6 Hz, 1H), 4.38 (q, *J* = 7.2 Hz, 2H), 2.93 – 2.87 (m, 2H), 1.79 – 1.70 (m, 2H), 1.47 – 1.37 (m, 5H), 0.95 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 191.2, 164.2, 139.3, 129.2, 127.2, 125.6, 122.7, 119.4, 115.2, 105.7, 60.2, 39.3, 27.7, 22.7, 14.7, 14.1.

Ethyl 3-hexanoylindolizine-1-carboxylate (2ac)



Following the general procedure A, the electrochemical reaction was carried out with **1ac** (54.7 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 20/1) yielded **2ac** (51.2 mg, 89%) as a brown solid. Spectral data matched those previously reported[4].

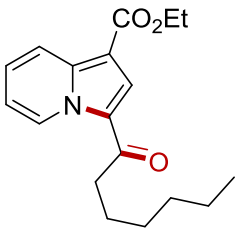
I_2/TBHP catalyzed intramolecular dehydrogenative aminooxygenation:

Following the general procedure C, the reaction yielded **2ac** (35.6 mg, 62%) as a brown solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.92 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.32 (dt, *J* = 9.2, 1.2 Hz, 1H), 8.00 (s, 1H), 7.37 (ddd, *J* = 9.2, 6.8, 1.2 Hz, 1H), 7.00 (td, *J* = 6.8, 1.2 Hz, 1H), 4.39 (q, *J* = 7.2 Hz, 2H), 2.90 (t, *J* = 7.2 Hz, 2H), 1.83 – 1.72 (m, 2H), 1.43 (t, *J* = 7.2 Hz, 3H), 1.40 – 1.32 (m, 4H), 0.93 – 0.88 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 191.3, 164.3, 139.4, 129.3, 127.2, 125.7, 122.8, 119.5, 115.2, 105.7, 60.2, 39.5, 31.8, 25.3, 22.7, 14.7, 14.1.

Ethyl 3-heptanoylindolizine-1-carboxylate (2ad)



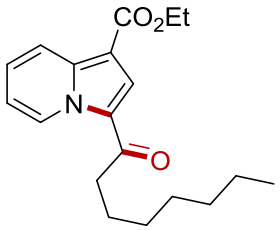
Following the general procedure A, the electrochemical reaction was carried out with **1ad** (57.5 mg, 0.2 mmol) at 27 ℃ for 8.5 h. Purification by column chromatography on silica gel (PE/ EA = 20/1) yielded **2ad** (54.3 mg, 90%) as a brown solid, **M.P.** 57.7 - 60.7 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.93 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.34 (dt, *J* = 9.2, 1.2 Hz, 1H), 8.01 (s, 1H), 7.38 (ddd, *J* = 8.4, 6.8, 1.2 Hz, 1H), 7.01 (td, *J* = 7.2, 1.6 Hz, 1H), 4.41 (q, *J* = 7.2 Hz, 2H), 2.91 (t, *J* = 7.6 Hz, 2H), 1.78 (quint, *J* = 7.4 Hz, 2H), 1.49 – 1.28 (m, 10H), 0.95 – 0.85 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 191.2, 164.2, 139.3, 129.2, 127.2, 125.6, 122.8, 119.5, 115.2, 105.7, 60.2, 39.6, 31.8, 29.3, 25.6, 22.6, 14.7, 14.2.

HRMS (ESI-TOF): exact mass calcd for $C_{18}H_{23}NO_3Na$ $(M+Na)^+$ requires m/z 324.1566, found m/z 324.1576.

Ethyl 3-octanoylindolizine-1-carboxylate (2ae)



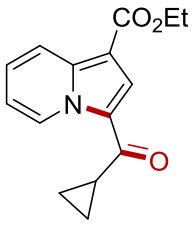
Following the general procedure A, the electrochemical reaction was carried out with **1ae** (60.3 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 20/1) yielded **2ae** (56.1 mg, 89%) as a brown solid, **M.P.** 54.8 - 56.7 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.91 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.31 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.98 (s, 1H), 7.36 (ddd, *J* = 9.2, 7.2, 1.2 Hz, 1H), 6.98 (td, *J* = 6.8, 1.2 Hz, 1H), 4.39 (q, *J* = 7.2 Hz, 2H), 2.89 (t, *J* = 7.6 Hz, 2H), 1.75 (quint, *J* = 6.4 Hz, 2H), 1.45 – 1.24 (m, 12H), 0.90 – 0.83 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 191.2, 164.2, 139.3, 129.2, 127.2, 125.6, 122.8, 119.5, 115.2, 105.7, 60.2, 39.6, 31.8, 29.5, 29.3, 25.6, 22.7, 14.7, 14.2.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{25}NO_3Na$ $(M+Na)^+$ requires m/z 338.1729, found m/z 338.1732.

Ethyl 3-(cyclopropanecarbonyl)indolizine-1-carboxylate (2af)



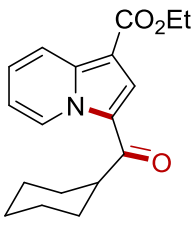
Following the general procedure A, the electrochemical reaction was carried out with **1af** (48.7 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 20/1) yielded **2af** (30.9 mg, 60%) as a light yellow solid, **M.P.** 94.8 - 98.0 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.90 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.34 (dt, *J* = 9.2, 1.2 Hz, 1H), 8.17 (s, 1H), 7.37 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 6.98 (td, *J* = 6.8, 1.2 Hz, 1H), 4.40 (q, *J* = 7.2 Hz, 2H), 2.63 – 2.55 (m, 1H), 1.43 (t, *J* = 7.2 Hz, 3H), 1.26 – 1.20 (m, 2H), 0.99 (dt, *J* = 8.0, 3.4 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 190.0, 164.3, 139.3, 129.2, 127.1, 125.6, 123.4, 119.5, 115.1, 105.9, 60.2, 18.2, 14.7, 10.4.

HRMS (ESI-TOF): exact mass calcd for $C_{15}H_{15}NO_3Na$ $(M+Na)^+$ requires m/z 280.0941, found m/z 280.0950.

Ethyl 3-(cyclohexanecarbonyl)indolizine-1-carboxylate (2ag)



Following the general procedure A, the electrochemical reaction was carried out with **1ag** (57.1 mg, 0.2 mmol) at 27 ℃ for 9.5 h. Purification by column chromatography on silica gel (PE/ EA = 20/1) yielded **2ag** (59.3 mg, 99%) as a light yellow solid, **M.P.** 42.6 - 46.0 ℃.

I_2-mediated intramolecular dehydrogenative aminooxygenation:

Following the general procedure B, the reaction yielded **2ag** (37.8 mg, 63%) as a light yellow solid.

I_2/TBHP catalyzed intramolecular dehydrogenative aminooxygenation:

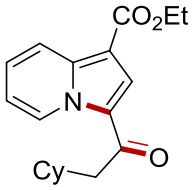
Following the general procedure C, the reaction yielded **2ag** (40.8 mg, 68%) as a light yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (d, *J* = 6.8 Hz, 1H), 8.32 (d, *J* = 9.2 Hz, 1H), 8.02 (s, 1H), 7.36 (dd, *J* = 6.8, 9.2 Hz, 1H), 6.99 (t, *J* = 6.8 Hz, 1H), 4.40 (q, *J* = 7.2 Hz, 2H), 3.16 (tt, *J* = 3.2, 11.6 Hz, 1H), 1.92 – 1.81 (m, 4H), 1.77 – 1.69 (m, 1H), 1.66 – 1.53 (m, 2H), 1.48 – 1.36 (m, 5H), 1.33 – 1.24 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 194.6, 164.3, 139.5, 129.4, 127.2, 125.3, 122.1, 119.4, 115.1, 105.7, 60.2, 47.2, 30.1, 26.0, 26.0, 14.7.

HRMS (ESI-TOF): exact mass calcd for $C_{18}H_{21}NO_3$ $(M+H)^+$ requires m/z 300.1585, found m/z 300.1594.

Ethyl 3-(2-cyclohexylacetyl)indolizine-1-carboxylate (2ah)



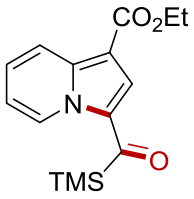
Following the general procedure A, the electrochemical reaction was carried out with **1ah** (59.9 mg, 0.2 mmol) at 27 ℃ for 10.5 h. Purification by column chromatography on silica gel (PE/ EA = 20/1) yielded **2ah** (58.9 mg, 94%) as a yellow solid, **M.P.** 45.1 - 48.8 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.94 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.31 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.97 (s, 1H), 7.36 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 6.99 (td, *J* = 6.8, 1.6 Hz, 1H), 4.39 (q, *J* = 7.2 Hz, 2H), 2.75 (d, *J* = 7.2 Hz, 2H), 2.04 – 1.92 (m, 1H), 1.79 – 1.60 (m, 5H), 1.42 (t, *J* = 7.2 Hz, 3H), 1.34 – 0.98 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 190.8, 164.2, 139.4, 129.2, 127.2, 125.9, 123.4, 119.5, 115.2, 105.7, 60.2, 47.3, 35.8, 33.6, 26.4, 26.3, 14.7.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{23}NO_3Na$ $(M+Na)^+$ requires m/z 336.1570, found m/z 336.1574.

Ethyl 3-((trimethylsilyl)carbonyl)indolizine-1-carboxylate (2ai)

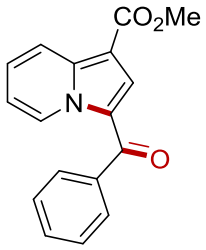


Following the general procedure A, the electrochemical reaction was carried out with **1ai** (55.1 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 20/1) yielded **2ai** (33.6 mg, 58%) as a brown solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.02 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.32 (dt, *J* = 8.8, 1.2 Hz, 1H), 8.01 (s, 1H), 7.40 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 6.99 (td, *J* = 6.8, 1.2 Hz, 1H), 4.40 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H), 0.41 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 164.3, 138.5, 129.3, 128.4, 128.1, 128.0, 119.4, 115.6, 106.7, 60.2, 14.7, -1.2.

Methyl 3-benzoylindolizine-1-carboxylate (2aj)

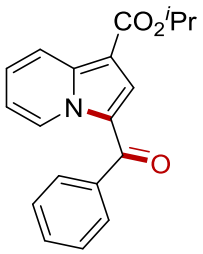


Following the general procedure A, the electrochemical reaction was carried out with **1aj** (53.1 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2aj** (44.7 mg, 80%) as a light yellow solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.38 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.89 – 7.74 (m, 3H), 7.60 – 7.54 (m, 1H), 7.53 – 7.48 (m, 2H), 7.45 (ddd, *J* = 9.2, 6.8, 1.2 Hz, 1H), 7.09 (td, *J* = 1.6, 7.2 Hz, 1H), 3.89 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 164.6, 140.0, 140.0, 131.6, 129.3, 129.1, 129.1, 128.5, 127.9, 122.7, 119.5, 115.5, 106.0, 51.4.

Isopropyl 3-benzoylindolizine-1-carboxylate (2ak)



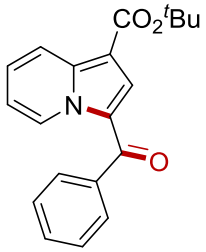
Following the general procedure A, the electrochemical reaction was carried out with **1ak** (58.7 mg, 0.2 mmol) at 27 ℃ for 8 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2ak** (53.5 mg, 87%) as a yellow solid, **M.P.** 108.3 - 110.3 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.97 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.38 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.86 – 7.78 (m, 3H), 7.60 – 7.55 (m, 1H), 7.54 – 7.49 (m, 2H), 7.44 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 5.28 (quint, *J* = 6.0 Hz, 1H), 1.37 (d, *J* = 6.4 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 163.8, 140.1, 139.9, 131.6, 129.3, 129.1, 129.1, 128.5, 127.7, 122.6, 119.7, 115.3, 106.9, 67.6, 22.3.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{17}NO_3Na$ $(M+Na)^+$ requires m/z 330.1108, found m/z 330.1116.

Tert-butyl 3-benzoylindolizine-1-carboxylate (2al)

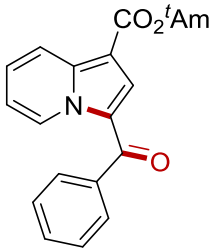


Following the general procedure A, the electrochemical reaction was carried out with **1al** (61.5 mg, 0.2 mmol) at 27 ℃ for 6 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2al** (53.3 mg, 83%) as a yellow solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.97 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.34 (dt, *J* = 8.8, 1.6 Hz, 1H), 7.84 – 7.79 (m, 2H), 7.78 (s, 1H), 7.59 – 7.54 (m, 1H), 7.53 – 7.47 (m, 2H), 7.42 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.06 (td, *J* = 6.8, 1.2 Hz, 1H), 1.61 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.6, 163.7, 140.1, 139.7, 131.5, 129.4, 129.3, 129.1, 128.5, 127.6, 122.4, 119.6, 115.2, 108.1, 80.8, 28.6.

Tert-pentyl 3-benzoylindolizine-1-carboxylate (2am)

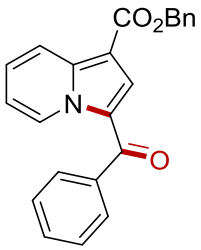


Following the general procedure A, the electrochemical reaction was carried out with **1am** (64.3 mg, 0.2 mmol) at 27 ℃ for 8 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2am** (63.7 mg, 95%) as a yellow solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.34 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.84 – 7.80 (m, 2H), 7.78 (s, 1H), 7.60 – 7.55 (m, 1H), 7.53 – 7.48 (m, 2H), 7.43 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.07 (td, *J* = 6.8, 1.6 Hz, 1H), 1.96 (q, *J* = 7.6 Hz, 2H), 1.58 (s, 6H), 0.96 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 163.7, 140.1, 139.7, 131.6, 129.5, 129.3, 129.1, 128.5, 127.6, 122.4, 119.7, 115.2, 108.1, 83.4, 33.8, 26.2, 8.5.

Benzyl 3-benzoylindolizine-1-carboxylate (2an)

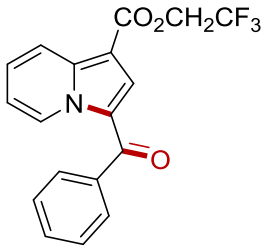


Following the general procedure A, the electrochemical reaction was carried out with **1an** (68.3 mg, 0.2 mmol) at 27 ℃ for 8 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2an** (57.6 mg, 81%) as a white solid. Spectral data matched those previously reported[15].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.40 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.86 (s, 1H), 7.84 – 7.80 (m, 2H), 7.60 – 7.55 (m, 1H), 7.53 – 7.48 (m, 2H), 7.47 – 7.30 (m, 6H), 7.09 (td, *J* = 7.2, 1.6 Hz, 1H), 5.39 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 164.0, 140.1, 139.9, 136.6, 131.6, 129.4, 129.2, 129.1, 128.7, 128.5, 128.3, 128.3, 128.0, 122.8, 119.6, 115.5, 105.9, 66.0.

2,2,2-trifluoroethyl 3-benzoylindolizine-1-carboxylate (2ao)



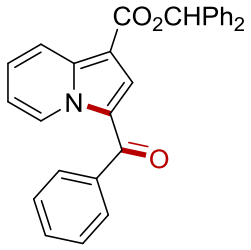
Following the general procedure A, the electrochemical reaction was carried out with **1ao** (66.7 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 10/1) yielded **2ao** (50.0 mg, 72%) as a brown solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.35 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.85 (s, 1H), 7.84 – 7.80 (m, 2H), 7.63 – 7.58 (m, 1H), 7.56 – 7.48 (m, 3H), 7.14 (td, *J* = 6.8, 1.2 Hz, 1H), 4.72 (q, *J* = 8.4 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.8, 162.0, 140.2, 139.6, 131.8, 129.5, 129.2, 129.0, 128.5, 124.7, 123.1, 121.9, 119.3, 115.7, 103.8, 59.9 (q, $J_{C\text{-}F}$ = 144.0 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -73.5.

Benzhydryl 3-benzoylindolizine-1-carboxylate (2ap)



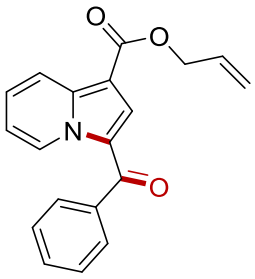
Following the general procedure A, the electrochemical reaction was carried out with **1ap** (83.5 mg, 0.2 mmol) at 27 ℃ for 8.5 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2ap** (61.3 mg, 71%) as a yellow solid, **M.P.** 133.6 - 136.3 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.99 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.45 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.94 (s, 1H), 7.88 – 7.82 (m, 2H), 7.62 – 7.57 (m, 1H), 7.56 – 7.50 (m, 2H), 7.49 – 7.42 (m, 5H), 7.39 – 7.34 (m, 4H), 7.33 – 7.28 (m, 2H), 7.20 (s, 1H), 7.10 (td, *J* = 6.8, 1.2 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 163.2, 140.6, 140.2, 139.9, 131.7, 129.4, 129.2, 129.1, 128.7, 128.6, 128.1, 128.0, 127.6, 127.4, 126.7, 122.9, 119.6, 115.5, 106.0, 76.8.

HRMS (ESI-TOF): exact mass calcd for $C_{29}H_{21}NO_3K$ $(M+K)^+$ requires m/z 470.1159, found m/z 470.1159.

Allyl 3-benzoylindolizine-1-carboxylate (2aq)

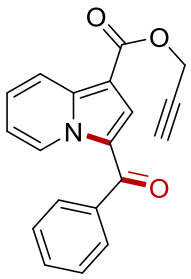


Following the general procedure A, the electrochemical reaction was carried out with **1aq** (58.3 mg, 0.2 mmol) at 27 ℃ for 8.5 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2aq** (47.0 mg, 77%) as a brown solid. Spectral data matched those previously reported[10].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.39 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.89 – 7.74 (m, 3H), 7.61 – 7.41 (m, 4H), 7.08 (td, *J* = 7.2, 1.6 Hz, 1H), 6.14 – 5.95 (m, 1H), 5.39 (dq, *J* = 17.2, 1.6 Hz, 1H), 5.27 (dq, *J* = 10.4, 1.2 Hz, 1H), 4.82 (dt, *J* = 6.0, 1.2 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 163.7, 140.1, 140.0, 132.8, 131.6, 129.3, 129.1, 129.1, 128.5, 127.9, 122.7, 119.6, 118.2, 115.5, 105.9, 64.9.

Prop-2-yn-1-yl 3-benzoylindolizine-1-carboxylate (2ar)

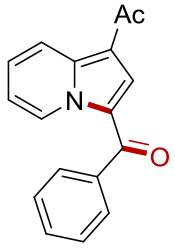


Following the general procedure A, the electrochemical reaction was carried out with **1ar** (57.9 mg, 0.2 mmol) at 27 ℃ for 7 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2ar** (51.6 mg, 85%) as a brown solid. Spectral data matched those previously reported[16].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.41 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.84 (s, 1H), 7.83 – 7.79 (m, 2H), 7.62 – 7.56 (m, 1H), 7.56 – 7.45 (m, 3H), 7.12 (td, *J* = 6.8, 1.2 Hz, 1H), 4.92 (d, *J* = 2.4 Hz, 2H), 2.51 (t, *J* = 2.4 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.8, 163.2, 140.3, 139.9, 131.7, 129.5, 129.3, 129.1, 128.6, 128.3, 122.9, 119.6, 115.7, 105.1, 78.3, 74.9, 51.7.

1-(3-benzoylindolizin-1-yl)ethan-1-one (2as)

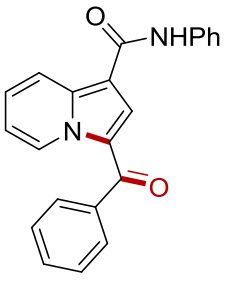


Following the general procedure A, the electrochemical reaction was carried out with **1as** (49.9 mg, 0.2 mmol) at 27 ℃ for 8.5 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2as** (31.6 mg, 60%) as a brown solid. Spectral data matched those previously reported[11].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.97 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.65 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.85 – 7.78 (m, 2H), 7.69 (s, 1H), 7.63 – 7.57 (m, 1H), 7.56 – 7.49 (m, 3H), 7.15 (td, *J* = 7.2, 1.6 Hz, 1H), 2.51 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.3, 185.7, 140.1, 139.7, 131.7, 129.2, 129.1, 129.1, 129.0, 128.6, 122.4, 120.6, 116.3, 114.9, 27.9.

3-benzoyl-N-phenylindolizine-1-carboxamide (2at)



Following the general procedure A, the electrochemical reaction was carried out with **1at** (65.3 mg, 0.2 mmol) at 27 ℃ for 11 h. Purification by column chromatography on silica gel (PE/ EA = 4/1) yielded **2at** (62.6 mg, 92%) as a yellow solid. Spectral data matched those previously reported[17].

I_2-mediated intramolecular dehydrogenative aminooxygenation:

Following the general procedure B, the reaction yielded **2at** (19.1 mg, 28%) as a yellow solid.

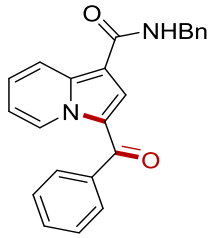
I_2/TBHP catalyzed intramolecular dehydrogenative aminooxygenation:

Following the general procedure C, the reaction yielded **2at** (63.9 mg, 94%) as a yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.84 (d, *J* = 6.8 Hz, 1H), 8.48 (d, *J* = 8.8 Hz, 1H), 8.15 (s, 1H), 7.73 – 7.66 (m, 2H), 7.61 (s, 1H), 7.59 – 7.54 (m, 2H), 7.46 – 7.32 (m, 4H), 7.29 – 7.22 (m, 2H), 7.09 – 6.98 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.3, 162.5, 139.9, 139.9, 138.2, 131.4, 129.0, 128.9, 128.8, 128.4, 127.5, 124.9, 124.2, 121.9, 120.5, 120.0, 115.7, 109.6.

3-benzoyl-N-benzylindolizine-1-carboxamide (2au)



Following the general procedure A, the electrochemical reaction was carried out with **1au** (71.3 mg, 0.2 mmol) at 27 ℃ for 11 h. Purification by column chromatography on silica gel

(PE/ EA = 4/1) yielded **2au** (67.3 mg, 95%) as a brown solid. Spectral data matched those previously reported[11].

I_2-mediated intramolecular dehydrogenative aminooxygenation:

Following the general procedure B, the reaction yielded **2au** (34.7 mg, 49%) as a brown solid.

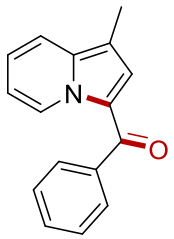
I_2/TBHP catalyzed intramolecular dehydrogenative aminooxygenation:

Following the general procedure C, the reaction yielded **2au** (65.2 mg, 92%) as a brown solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.88 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.56 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.72 – 7.67 (m, 2H), 7.53 (s, 1H), 7.47 – 7.33 (m, 4H), 7.33 – 7.18 (m, 5H), 7.03 (td, *J* = 6.8, 1.2 Hz, 1H), 6.64 (t, *J* = 5.6 Hz, 1H), 4.55 (d, *J* = 5.6 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.3, 164.1, 140.1, 139.8, 138.7, 131.3, 128.9, 128.8, 128.7, 128.4, 127.9, 127.5, 127.3, 124.6, 121.9, 120.1, 115.5, 109.2, 43.4.

(1-methylindolizin-3-yl)(phenyl)methanone (2av)



Following the general procedure A, the electrochemical reaction was carried out with **1av** (44.3 mg, 0.2 mmol) at 27 ℃ for 9.5 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2av** (26.4 mg, 56%) as a yellow oil.

I_2-mediated intramolecular dehydrogenative aminooxygenation:

Following the general procedure B, the reaction only a trace amount of **2av** was detected.

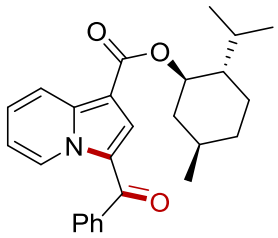
I_2/TBHP catalyzed intramolecular dehydrogenative aminooxygenation:

Following the general procedure C, the reaction yielded **2av** (25.5 mg, 54%) as a yellow oil.

^{1}H NMR (600 MHz, $CDCl_3$) δ 9.97 (dt, *J* = 7.2, 1.2 Hz, 1H), 7.79 (dt, *J* = 6.0, 1.2 Hz, 2H), 7.54 – 7.46 (m, 4H), 7.19 – 7.16 (m, 1H), 7.15 (d, *J* = 1.2 Hz, 1H), 6.92 (td, *J* = 7.2, 1.8 Hz, 1H), 2.33 (s, 3H).

^{13}C NMR (150 MHz, $CDCl_3$) δ 182.8, 140.1, 137.0, 129.5, 127.9, 127.8, 127.1, 125.5, 122.6, 120.2, 115.7, 112.7, 110.6, 9.5.

(1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl 3-benzoylindolizine-1-carboxylate (2aw)



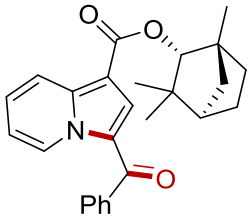
Following the general procedure A, the electrochemical reaction was carried out with **1aw** (77.9 mg, 0.2 mmol) at 27 ℃ for 6.5 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2aw** (56.5 mg, 70%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.39 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.85 – 7.79 (m, 3H), 7.61 – 7.41 (m, 4H), 7.08 (td, *J* = 6.8, 1.2 Hz, 1H), 4.98 (td, *J* = 11.2, 4.4 Hz, 1H), 2.18 – 2.11 (m, 1H), 2.00 – 1.91 (m, 1H), 1.77 – 1.68 (m, 2H), 1.61 – 1.50 (m, 2H), 1.21 – 1.07 (m, 2H), 1.03 – 0.94 (m, 1H), 0.94 – 0.89 (m, 6H), 0.81 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 163.8, 140.1, 140.0, 131.6, 129.3, 129.2, 129.1, 128.5, 127.7, 122.6, 119.7, 115.3, 106.8, 74.0, 47.5, 41.5, 34.5, 31.6, 26.8, 24.0, 22.2, 20.8, 16.8.

HRMS (ESI-TOF): exact mass calcd for $C_{26}H_{29}NO_3Na$ $(M+Na)^+$ requires m/z 426.2045, found m/z 426.2051.

(1*R*,2*R*,4*R*)-1,3,3-trimethylbicyclo[2.2.1]heptan-2-yl 3-benzoylindolizine-1-carboxylate (2ax)



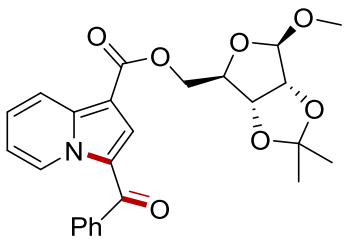
Following the general procedure A, the electrochemical reaction was carried out with **1ax** (77.5 mg, 0.2 mmol) at 27 ℃ for 6.5 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2ax** (69.9 mg, 87%) as a brown solid, **M.P.** 109.4 - 111.0 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.99 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.40 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.86 (s, 1H), 7.85 – 7.81 (m, 2H), 7.63 – 7.41 (m, 4H), 7.09 (td, *J* = 6.8, 1.2 Hz, 1H), 4.66 (d, *J* = 2.0 Hz, 1H), 1.94 – 1.83 (m, 1H), 1.83 – 1.74 (m, 2H), 1.67 (dt, *J* = 10.4, 2.4 Hz, 1H), 1.57 – 1.47 (m, 1H), 1.28 – 1.22 (m, 2H), 1.20 (s, 3H), 1.13 (s, 3H), 0.85 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.6, 164.7, 140.0, 139.7, 131.7, 129.4, 129.3, 129.1, 128.5, 127.8, 122.7, 119.6, 115.3, 106.7, 86.4, 48.6, 48.5, 41.6, 39.8, 29.8, 27.4, 26.1, 20.5, 19.7.

HRMS (ESI-TOF): exact mass calcd for $C_{26}H_{27}NO_3Na$ $(M+Na)^+$ requires m/z 424.1889, found m/z 424.1887.

((3a*R*,4*R*,6*R*,6a*R*)-6-methoxy-2,2-dimethyltetrahydrofuro[3,4-*d*][1,3]dioxol-4-yl)methyl 3-benzoylindolizine-1-carboxylate (2ay)



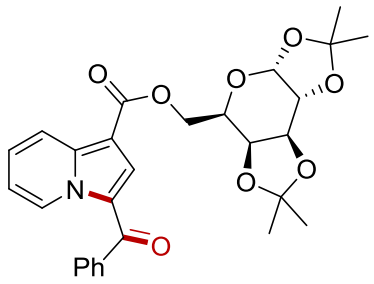
Following the general procedure A, the electrochemical reaction was carried out with **1ay** (87.5 mg, 0.2 mmol) at 27 ℃ for 8.5 h. Purification by column chromatography on silica gel (PE/ EA = 5/1) yielded **2ay** (78.6 mg, 87%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.40 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.85 (s, 1H), 7.81 (dt, *J* = 6.8, 1.2 Hz, 2H), 7.57 (tt, *J* = 7.6, 1.2 Hz, 1H), 7.54 – 7.43 (m, 4H), 7.09 (td, *J* = 6.8, 1.6 Hz, 1H), 5.01 (s, 1H), 4.75 (dd, *J* = 6.0, 0.8 Hz, 1H), 4.63 (d, *J* = 6.0 Hz, 1H), 4.53 (td, *J* = 7.2, 1.2 Hz, 1H), 4.35 (qd, *J* = 11.3, 7.2 Hz, 2H), 3.31 (s, 3H), 1.48 (s, 3H), 1.31 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 163.6, 140.1, 139.9, 131.7, 129.4, 129.2, 129.1, 128.5, 128.1, 122.8, 119.6, 115.5, 112.7, 109.6, 105.6, 85.4, 84.6, 82.0, 64.5, 55.0, 26.6, 25.1.

HRMS (ESI-TOF): exact mass calcd for $C_{25}H_{25}NO_7Na$ $(M+Na)^+$ requires m/z 474.1523, found m/z 474.1528.

((3a*R*,5*R*,5a*S*,8a*S*,8b*R*)-2,2,7,7-tetramethyltetrahydro-5*H*-bis([1,3]dioxolo)[4,5-*b*:4',5'-*d*] pyran-5-yl)methyl 3-benzoylindolizine-1-carboxylate (2az)



Following the general procedure A, the electrochemical reaction was carried out with **1az** (98.7 mg, 0.2 mmol) at 27 ℃ for 8.5 h. Purification by column chromatography on silica gel (PE/ EA = 2/1) yielded **2az** (81.2 mg, 80%) as a brown solid, **M.P.** 72.1 - 73.9 ℃.

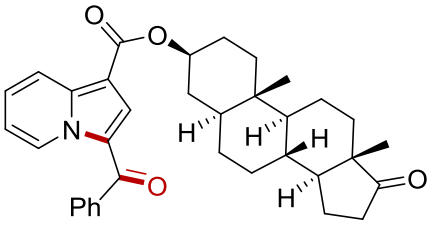
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.42 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.85 (s, 1H), 7.83 – 7.77 (m, 2H), 7.59 – 7.53 (m, 1H), 7.53 – 7.40 (m, 3H), 7.08 (td, *J* = 7.2,

1.2 Hz, 1H), 5.57 (d, *J* = 4.8 Hz, 1H), 4.64 (dd, *J* = 7.6, 2.4 Hz, 1H), 4.52 (dd, *J* = 11.6, 4.0 Hz, 1H), 4.43 (dd, *J* = 11.6, 8.0 Hz, 1H), 4.34 (dd, *J* = 4.8, 2.4 Hz, 1H), 4.31 (dd, *J* = 8.0, 2.0 Hz, 1H), 4.21 (ddd, *J* = 8.0, 4.0, 2.0 Hz, 1H), 1.48 (d, *J* = 5.6 Hz, 6H), 1.33 (d, *J* = 6.8 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 163.9, 140.0, 140.0, 131.6, 129.5, 129.3, 129.1, 128.5, 127.9, 122.8, 119.9, 115.4, 109.8, 108.9, 106.1, 96.5, 71.3, 70.9, 70.7, 66.6, 63.6, 26.1, 26.1, 25.1, 24.5.

HRMS (ESI-TOF): exact mass calcd for $C_{28}H_{29}NO_8Na$ $(M+Na)^+$ requires m/z 530.1791, found m/z 530.1785.

(3*S*,5*S*,8*R*,9*S*,10*S*,13*S*,14*S*)-10,13-dimethyl-17-oxohexadecahydro-1H-cyclopenta[*a*]phenanthren-3-yl 3-benzoylindolizine-1-carboxylate (2aaa)



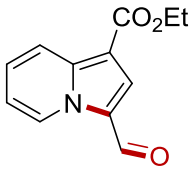
Following the general procedure A, the electrochemical reaction was carried out with **1aaa** (106.6 mg, 0.2 mmol) at 27 ℃ for 8 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **2aaa** (89.3 mg, 83%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.97 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.38 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.84 – 7.77 (m, 3H), 7.61 – 7.55 (m, 1H), 7.55 – 7.49 (m, 2H), 7.45 (ddd, *J* = 8.8, 7.2, 1.2 Hz, 1H), 7.09 (td, *J* = 7.2, 1.6 Hz, 1H), 4.96 (hept, *J* = 4.8 Hz, 1H), 2.43 (dd, *J* = 19.2, 8.8 Hz, 1H), 2.12 – 1.89 (m, 3H), 1.84 – 1.74 (m, 4H), 1.72 – 1.45 (m, 6H), 1.36 – 1.23 (m, 6H), 1.17 – 0.95 (m, 2H), 0.90 (s, 3H), 0.86 (s, 3H), 0.80 – 0.70 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 163.8, 140.1, 140.0, 131.6, 129.3, 129.2, 129.1, 128.5, 127.8, 122.6, 119.7, 115.4, 106.7, 73.5, 54.5, 51.5, 47.9, 44.9, 36.9, 36.0, 35.9, 35.2, 34.4, 31.7, 30.9, 28.4, 27.9, 21.9, 20.6, 13.9, 12.6.

HRMS (ESI-TOF): exact mass calcd for $C_{35}H_{39}NO_4Na$ $(M+Na)^+$ requires m/z 560.2777, found m/z 560.2768.

Ethyl 3-formylindolizine-1-carboxylate (4a)

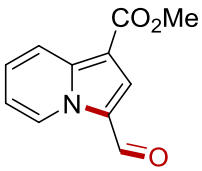


Following the general procedure A, the electrochemical reaction was carried out with **3a** (40.7 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **4a** (36.1 mg, 83%) as a white solid. Spectral data matched those previously reported[4].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.76 (s, 1H), 9.72 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.37 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.93 (s, 1H), 7.46 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.07 (td, *J* = 7.2, 1.6 Hz, 1H), 4.40 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.6, 163.9, 140.2, 129.6, 129.0, 128.4, 124.0, 119.7, 115.7, 107.5, 60.3, 14.6.

Methyl 3-formylindolizine-1-carboxylate (4b)

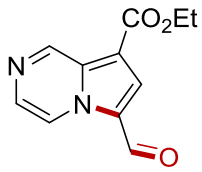


Following the general procedure A, the electrochemical reaction was carried out with **3b** (37.9 mg, 0.2 mmol) at 27 ℃ for 9 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **4b** (37.4 mg, 92%) as a white solid. Spectral data matched those previously reported[18].

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.73 (s, 1H), 9.68 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.33 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.88 (s, 1H), 7.44 (td, *J* = 6.8, 1.2 Hz, 1H), 7.05 (td, *J* = 6.8, 1.6 Hz, 1H), 3.91 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.6, 164.2, 140.2, 129.5, 128.9, 128.5, 124.0, 119.7, 115.7, 107.1, 51.5.

Ethyl 6-formylpyrrolo[1,2-*a*]pyrazine-8-carboxylate (4c)



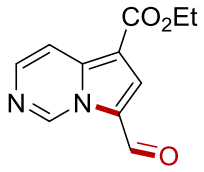
Following the general procedure A, the electrochemical reaction was carried out with **3c** (40.9 mg, 0.2 mmol) at 27 ℃ for 10.5 h. Purification by column chromatography on silica gel (PE/ EA = 10/1) yielded **4c** (32.7 mg, 75%) as a light yellow solid, **M.P.** 105.4 - 109.4 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.91 (s, 1H), 9.73 (d, *J* = 1.6 Hz, 1H), 9.44 (dd, *J* = 4.4, 1.6 Hz, 1H), 8.13 (d, *J* = 4.4 Hz, 1H), 7.97 (s, 1H), 4.43 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 180.2, 162.9, 146.1, 133.2, 132.1, 128.0, 124.4, 120.4, 110.1, 61.0, 14.5.

HRMS (ESI-TOF): exact mass calcd for $C_{11}H_{10}N_2O_3Na$ $(M+Na)^+$ requires m/z 241.0589, found m/z 241.0583.

Ethyl 7-formylpyrrolo[1,2-*c*]pyrimidine-5-carboxylate (4d)



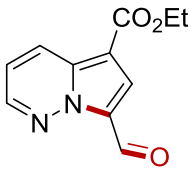
Following the general procedure A, the electrochemical reaction was carried out with **3d** (40.9 mg, 0.2 mmol) at 27 ℃ for 21 h. Purification by column chromatography on silica gel (PE/ EA = 8/1) yielded **4d** (10.9 mg, 25%) as a brown solid, **M.P.** 99.0 - 102.5 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.38 (d, *J* = 1.2 Hz, 1H), 9.84 (s, 1H), 8.19 (dd, *J* = 6.4, 1.6 Hz, 1H), 8.14 (d, *J* = 6.0 Hz, 1H), 8.00 (s, 1H), 4.42 (d, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 179.0, 163.3, 142.2, 141.1, 130.3, 123.7, 114.6, 108.2, 60.8, 14.6.

HRMS (ESI-TOF): exact mass calcd for $C_{11}H_{11}N_2O_3$ $(M+H)^+$ requires m/z 219.0764, found m/z 219.0773.

Ethyl 7-formylpyrrolo[1,2-*b*]pyridazine-5-carboxylate (4e)



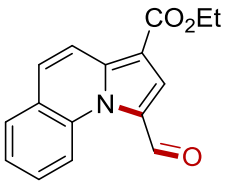
Following the general procedure A, the electrochemical reaction was carried out with **3e** (40.9 mg, 0.2 mmol) at 27 ℃ for 8 h. Purification by column chromatography on silica gel (PE/ EA = 8/1) yielded **4e** (26.2 mg, 60%) as a yellow solid, **M.P.** 96.5 - 99.0 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.35 (s, 1H), 8.61 (dd, *J* = 9.2, 1.6 Hz, 1H), 8.43 (dd, *J* = 4.4, 2.0 Hz, 1H), 7.97 (s, 1H), 7.16 (dd, *J* = 9.2, 4.8 Hz, 1H), 4.38 (q, *J* = 7.2 Hz, 2H), 1.40 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 179.2, 163.5, 144.2, 133.0, 128.5, 128.2, 121.6, 118.4, 107.1, 60.6, 14.5.

HRMS (ESI-TOF): exact mass calcd for $C_{11}H_{10}N_2O_3K$ $(M+K)^+$ requires m/z 257.0328, found m/z 257.0328.

Ethyl 1-formylpyrrolo[1,2-*a*]quinoline-3-carboxylate (4f)



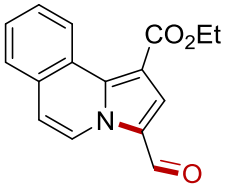
Following the general procedure A, the electrochemical reaction was carried out with **3f** (50.7 mg, 0.2 mmol) at 27 ℃ for 11.5 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **4f** (42.2 mg, 79%) as a yellow solid, **M.P.** 84.5 - 87.6 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.73 (s, 1H), 9.36 (d, *J* = 8.8 Hz, 1H), 8.30 (d, *J* = 9.2 Hz, 1H), 8.00 (s, 1H), 7.76 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.72 – 7.65 (m, 2H), 7.51 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 4.41 (q, *J* = 7.2 Hz, 2H), 1.44 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 163.7, 141.5, 134.9, 134.5, 130.0, 129.8, 129.3, 128.7, 126.1, 125.2, 121.1, 117.6, 109.4, 60.5, 14.6.

HRMS (ESI-TOF): exact mass calcd for $C_{16}H_{13}NO_3K$ $(M+K)^+$ requires m/z 306.0526, found m/z 306.0533.

Ethyl 3-formylpyrrolo[2,1-*a*]isoquinoline-1-carboxylate (4g)



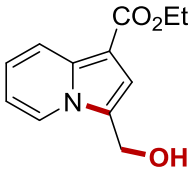
Following the general procedure A, the electrochemical reaction was carried out with **3g** (50.7 mg, 0.2 mmol) at 27 ℃ for 9.5 h. Purification by column chromatography on silica gel (PE/ EA = 15/1) yielded **4g** (48.1 mg, 90%) as a light yellow solid, **M.P.** 113.3 - 115.7 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.86 – 9.79 (m, 1H), 9.76 (s, 1H), 9.42 (d, *J* = 7.2 Hz, 1H), 7.95 (s, 1H), 7.73 – 7.69 (m, 1H), 7.68 – 7.60 (m, 2H), 7.20 (d, *J* = 7.6 Hz, 1H), 4.42 (q, *J* = 7.2 Hz, 2H), 1.45 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 179.2, 164.3, 137.4, 131.3, 130.9, 129.7, 128.5, 128.1, 126.9, 124.7, 124.6, 124.5, 116.1, 111.6, 60.8, 14.6.

HRMS (ESI-TOF): exact mass calcd for $C_{16}H_{13}NO_3Na$ $(M+Na)^+$ requires m/z 290.0785, found m/z 290.0793.

Ethyl 3-(hydroxymethyl)indolizine-1-carboxylate (6)



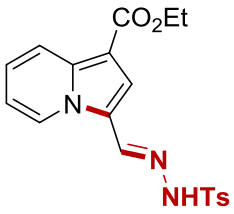
White solid, **M.P.** 157.4 - 160.0 ℃, 39.9 mg, 91% yield.

^{1}H NMR (400 MHz, CD_2Cl_2) δ 8.22 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.11 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.12 – 7.06 (m, 2H), 6.78 (td, *J* = 6.8, 1.2 Hz, 1H), 4.88 (s, 2H), 4.29 (q, *J* = 7.2 Hz, 2H), 2.25 (br, 1H), 1.37 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, CD_2Cl_2) δ 165.3, 137.0, 125.0, 124.7, 123.0, 119.9, 116.2, 112.8, 103.1, 59.9, 56.7, 14.8.

HRMS (ESI-TOF): exact mass calcd for $C_{12}H_{13}NO_3Na$ $(M+Na)^+$ requires m/z 242.0793, found m/z 242.0795.

Ethyl (*E*)-3-((2-tosylhydrazineylidene)methyl)indolizine-1-carboxylate (7)



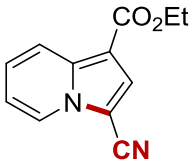
Brown solid, **M.P.** 217.4 - 219.6 ℃, 63.1 mg, 85% yield.

^{1}H NMR (400 MHz, DMSO-d_6) δ 11.32 (br, 1H), 9.27 (d, J = 7.2 Hz, 1H), 8.21 – 8.24 (m, 2H), 7.84 (d, J = 8.4 Hz, 1H), 7.56 (s, 1H), 7.44 – 7.35 (m, 3H), 7.17 (t, J = 6.8 Hz, 1H), 4.25 (q, J = 7.2 Hz, 2H), 2.32 (s, 3H), 1.30 (t, J = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, DMSO-d_6) δ 163.2, 143.6, 140.6, 137.6, 135.8, 129.7, 127.7, 127.4, 125.5, 122.7, 118.9, 118.8, 114.6, 104.5, 59.4, 21.0, 14.4.

HRMS (ESI-TOF): exact mass calcd for $C_{19}H_{20}NO_3$ $(M+H)^+$ requires m/z 386.1175, found m/z 386.1184.

Ethyl 3-cyanoindolizine-1-carboxylate (8)



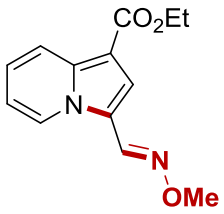
White solid, **M.P.** 93.6 - 95.6 ℃, 43.3 mg, 70% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 – 8.26 (m, 2H), 7.76 (s, 1H), 7.31 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 7.02 (td, *J* = 7.2, 1.2 Hz, 1H), 4.36 (q, *J* = 7.2 Hz, 2H), 1.39 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 163.3, 137.8, 126.1, 125.8, 125.2, 120.5, 115.1, 112.7, 106.1, 96.7, 60.3, 14.6.

HRMS (ESI-TOF): exact mass calcd for $C_{12}H_{11}N_2O_2$ $(M+H)^+$ requires m/z 215.0821, found m/z 215.0820.

Ethyl (*E*)-3-((methoxyimino)methyl)indolizine-1-carboxylate (9)



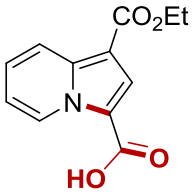
Brown solid, **M.P.** 198.7 - 202.5 ℃, 43.3 mg, 88% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.29 (dt, *J* = 6.8, 1.2 Hz, 1H), 8.29 (dt, *J* = 9.2, 1.2 Hz, 1H), 8.25 (s, 1H), 7.40 (s, 1H), 7.23 (ddd, *J* = 9.2, 6.8, 1.2 Hz, 1H), 6.92 (td, *J* = 6.8, 1.2 Hz, 1H), 4.37 (q, *J* = 7.2 Hz, 2H), 4.00 (s, 3H), 1.40 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 164.5, 141.8, 138.5, 128.9, 124.5, 122.4, 119.6, 117.4, 113.9, 105.5, 62.3, 59.9, 14.7.

HRMS (ESI-TOF): exact mass calcd for $C_{13}H_{14}N_2O_3Na$ $(M+Na)^+$ requires m/z 269.0902, found m/z 269.0909.

1-(ethoxycarbonyl)indolizine-3-carboxylic acid (10)



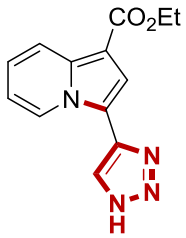
White solid, **M.P.** 150.1 - 152.3 ℃, 38.7 mg, 83% yield.

^{1}H NMR (400 MHz, DMSO-d_6) δ 12.86 (br, 1H), 9.50 (dt, J = 7.2, 1.2 Hz, 1H), 8.23 (dt, J = 9.2, 1.2 Hz, 1H), 7.78 (s, 1H), 7.47 (ddd, J = 8.8, 6.4, 1.2 Hz, 1H), 7.18 (td, J = 1.4, 1.2 Hz, 1H), 4.29 (q, J = 6.8 Hz, 2H), 1.33 (t, J = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, DMSO-d_6) δ 163.2, 161.9, 138.1, 127.8, 126.3, 123.2, 118.8, 115.1, 114.9, 103.9, 59.5, 14.4.

HRMS (ESI-TOF): exact mass calcd for $C_{12}H_{11}NO_4Na$ $(M+Na)^+$ requires m/z 256.0586, found m/z 256.0584.

Ethyl 3-(1*H*-1,2,3-triazol-4-yl)indolizine-1-carboxylate (11)



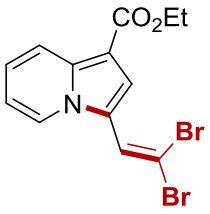
Brown solid, **M.P.** 199.8 - 201.0 ℃, 40.0 mg, 78% yield.

^{1}H NMR (400 MHz, DMSO-d_6) δ 15.28 (br, 1H), 9.37 (d, J = 6.4 Hz, 1H), 8.51 (s, 1H), 8.19 (dt, J = 8.8, 1.2 Hz, 1H), 7.72 (s, 1H), 7.30 (ddd, J = 8.8, 6.4, 1.2 Hz, 1H), 7.07 (td, J = 6.8, 1.2 Hz, 1H), 4.30 (q, J = 7.2 Hz, 2H), 1.34 (t, J = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, DMSO-d_6) δ 163.6, 135.8, 126.3, 123.4, 118.9, 116.5, 115.7, 113.7, 103.5, 59.2, 14.5.

HRMS (ESI-TOF): exact mass calcd for $C_{13}H_{12}N_4O_2Na$ $(M+Na)^+$ requires m/z 279.0858, found m/z 279.0866.

Ethyl 3-(2,2-dibromovinyl)indolizine-1-carboxylate (12)



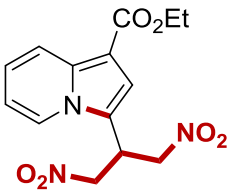
Brown oil, 70.1 mg, 94% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (dt, *J* = 8.8, 1.2 Hz, 1H), 7.98 (dt, *J* = 7.2, 1.2 Hz, 1H), 7.92 (s, 1H), 7.52 (s, 1H), 7.14 (ddd, *J* = 9.2, 6.8, 1.2 Hz, 1H), 6.82 (td, *J* = 6.8, 1.2 Hz, 1H), 4.38 (q, *J* = 7.2 Hz, 2H), 1.41 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 164.7, 136.2, 123.9, 123.5, 122.9, 120.5, 120.4, 118.1, 113.5, 105.3, 89.0, 59.9, 14.7.

HRMS (ESI-TOF): exact mass calcd for $C_{13}H_{12}Br_2NO_2K$ $(M+K)^+$ requires m/z 409.8785, found m/z 409.8794.

Ethyl 3-(1,3-dinitropropan-2-yl)indolizine-1-carboxylate (13)



Brown solid, **M.P.** 156.3 - 159.1 ℃, 46.3 mg, 72% yield.

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.81 (d, *J* = 6.8 Hz, 1H), 7.25 (dt, *J* = 9.2, 1.2 Hz, 1H), 6.53 (s, 1H), 6.40 (ddd, *J* = 8.8, 6.8, 1.2 Hz, 1H), 6.16 (td, *J* = 6.8, 1.6 Hz, 1H), 4.38 – 4.24 (m, 4H), 4.14 – 4.04 (m, 1H), 3.42 (q, *J* = 7.2 Hz, 2H), 0.48 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, DMSO-d_6) δ 163.6, 135.5, 124.5, 123.2, 120.8, 118.8, 114.4, 113.0, 102.7, 75.5, 59.1, 31.8, 14.5.

HRMS (ESI-TOF): exact mass calcd for $C_{14}H_{15}N_3O_6Na$ $(M+Na)^+$ requires m/z 344.0859, found m/z 344.0865.

8 Preliminary Mechanistic Studies

8.1 Radical Scavenger Addition Experiments

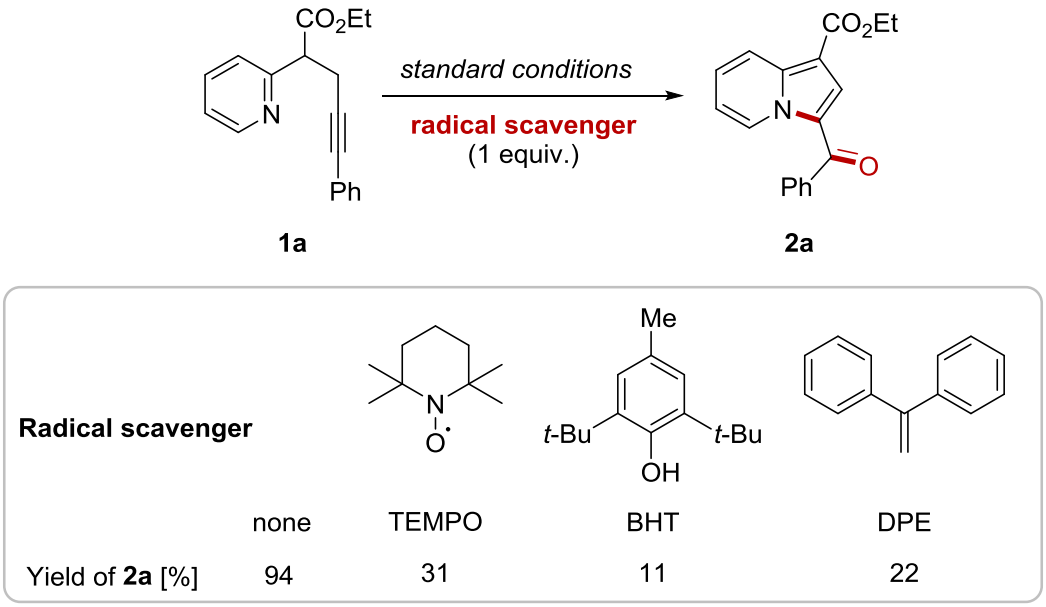
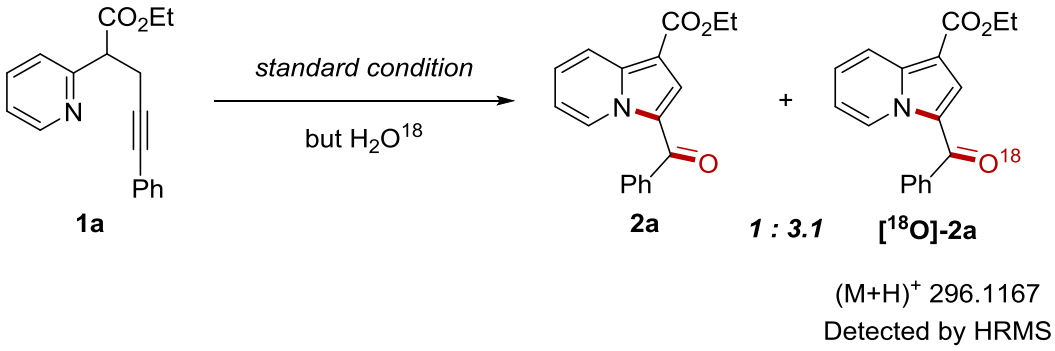


Figure S8: Radical scavenger addition experiments

Three parallel radical scavenger addition experiments were conducted under standard conditions using **1a** (55.9 mg, 0.2 mmol) as substrates with 1 equiv TEMPO (31.3 mg, 0.2 mmol), BHT (44.1 mg, 0.2 mmol), and 1,1-diphenylethylene (36.1 mg, 0.2 mmol) respectively. The electrochemistry enabled intramolecular dehydrogenative aminooxygenation was not completely inhibited but led to a significant decrease in the yield of **2a**, it indicates that the reaction may not be a free radical pathway.

8.2 Isotope labeling Experiment



An isotope labeling reaction was carried out by treatment of **1a** in the presence of $H_2{}^{18}O$ under standard conditions, leading to a mixture of **2a** and [^{18}O]-**2a** (1:3.1) in 81% yield. The HMRS spectra of the mixture of **2a** and [^{18}O]-**2a** was listed as bellow.

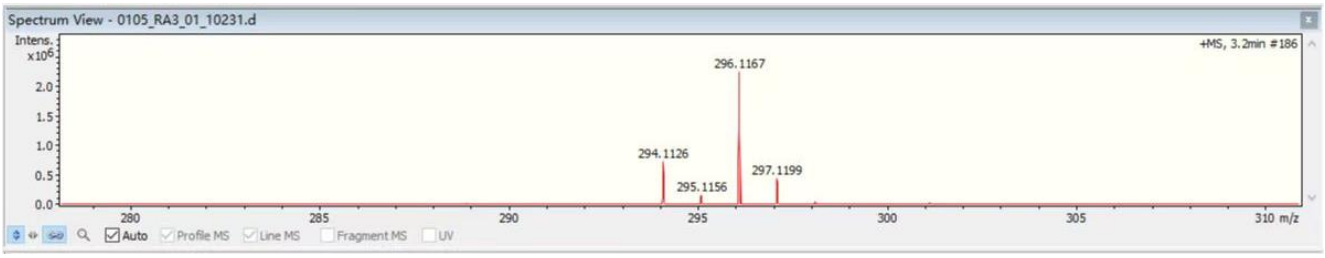
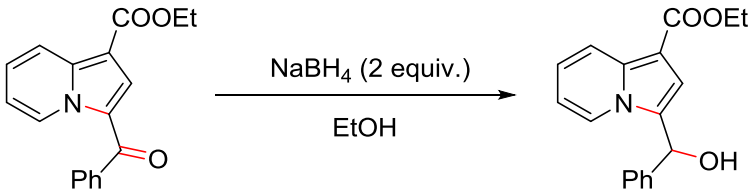


Figure S9: HMRS spectra of the mixture of **2a** and [^{18}O]-**2a**

8.3 Examination of Possible Intermediate

General Procedure for the Preparation of ethyl 3-(hydroxy(phenyl)methyl)indolizine -1-carboxylate (5)



2a **5**

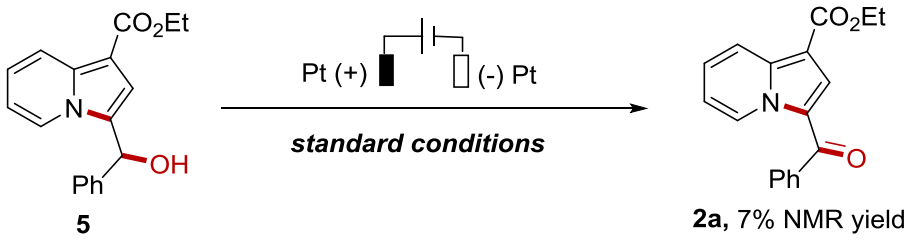
To a stirred solution of **2a** (293.3 mg, 1.0 mmol, 1.0 equiv) in EtOH (5 mL), $NaBH_4$ (76.0 mg, 2.0 mmol, 2.0 equiv) was added. Then the reaction was performed at r.t. for 1.0 h. The mixture was quenched with H_2O (5 mL), extracted with AcOEt (3 × 5mL). The organic layers were combined and dried over anhydrous Na_2SO_4. After filtration and removal of the solvents, the resulting residue was chromatographed through silica gel eluting with PE/EA (15/1) to afford the corresponding product **5** as white solid with 85% yield (251.0 mg).

White solid, **M.P.** 93.5 – 95.9 ℃, 251.0 mg, 85% yield.

^{1}H NMR (400 MHz, CD_2Cl_2) δ 8.13 (dt, *J* = 7.2, 1.2 Hz, 1H), 8.06 (dt, *J* = 9.2, 1.2 Hz, 1H), 7.39 – 7.24 (m, 5H), 7.02 – 6.97 (m, 1H), 6.74 (s, 1H), 6.64 (td, *J* = 6.8, 1.2 Hz, 1H), 6.06 (d, *J* = 3.6 Hz, 1H), 4.18 (q, *J* = 7.2 Hz, 2H), 2.64 (d, *J* = 4.8 Hz, 1H), 1.25 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, CD_2Cl_2) δ 164.7, 140.6, 136.9, 128.6, 128.0, 126.5, 126.4, 125.0, 122.6, 119.6, 116.2, 112.3, 102.8, 69.1, 59.4, 14.4.

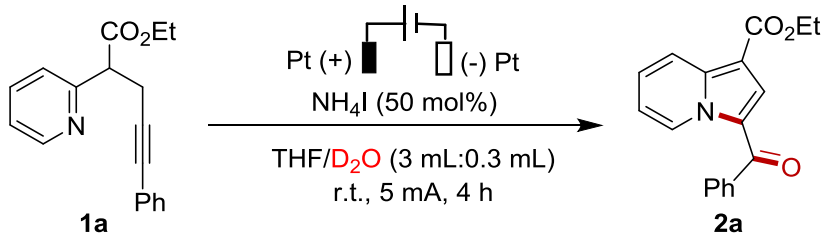
HRMS (ESI-TOF): exact mass calcd for $C_{18}H_{17}NO_3Na$ $(M+Na)^+$ requires m/z 318.1101, found m/z 318.1106.



5 **2a**, 7% NMR yield

According to procedure A, The electrocatalysis was carried out in an undivided cell equipped with two platinum electrode (15 × 10 × 0.2 mm^3). The substrates **5** (59.1 mg, 0.2 mmol) and NH_4I (14.5 mg, 0.1 mmol, 50 mol%) were dissolved in the mixture solvent THF/H_2O (3/0.3 mL). The electrolysis was carried out at 27 ℃ (oil bath temperature) using a constant current of 5.0 mA for 9h. The reaction only 7% yield of 2a was observed by crude ^{1}H NMR.

9 Proposed Mechanism with ESI-Mass (m/z) Analysis



1a **2a**

According to procedure A, The electrocatalysis was carried out in an undivided cell

equipped with two platinum electrode (15 × 10 × 0.2 mm^3). The substrates **1a** (55.9 mg, 0.2 mmol) and NH_4I (14.5 mg, 0.1 mmol, 50 mol%) were dissolved in the mixture solvent THF/D_2O (3/0.3 mL). The electrolysis was carried out at 27 ℃ (oil bath temperature) using a constant current of 5.0 mA for 4h.

The mass spectrum of reaction mixture showed molecular ion peak at m/z = 280.1334, 406.0297, 404.0144, 424.0372, 423.0311 and 316.0944, which matched the structure of substrate **1a** ($[M+H]^+$, calcd mass: 280.1332), intermediate **C** ($[M+H]^+$, calcd mass: 406.0298), intermediate **F** or **G** ($[M]^+$, calcd mass: 404.0142), intermediate **H** ($[M]^+$, calcd mass: 424.0373), intermediate **I** ($[M+H]^+$, calcd mass: 423.0310), and product **2a** ($[M+Na]^+$, calcd mass: 316.0944) respectively.

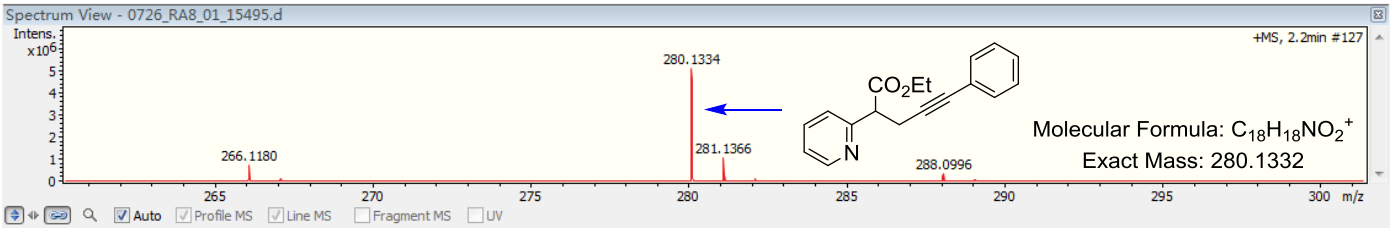


Figure S10: ESI-HMRS of $[M+H]^+$ for **1a**

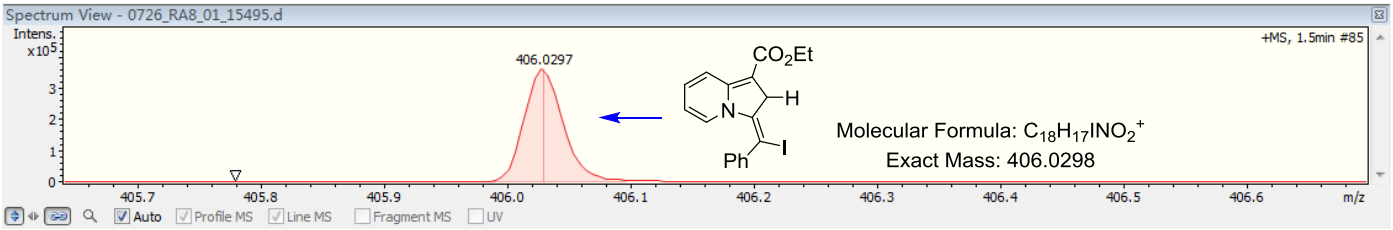


Figure S11: ESI-HMRS of $[M+H]^+$ for intermediate (**C**)

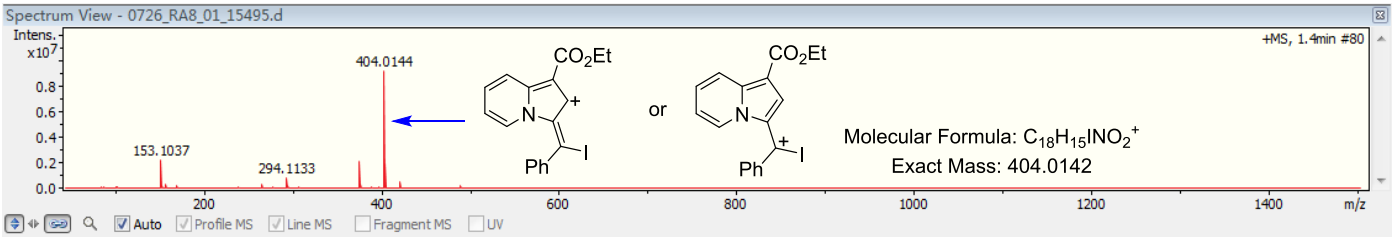


Figure S12: ESI-HMRS of $[M]^+$ for intermediate (**F** or **G**)

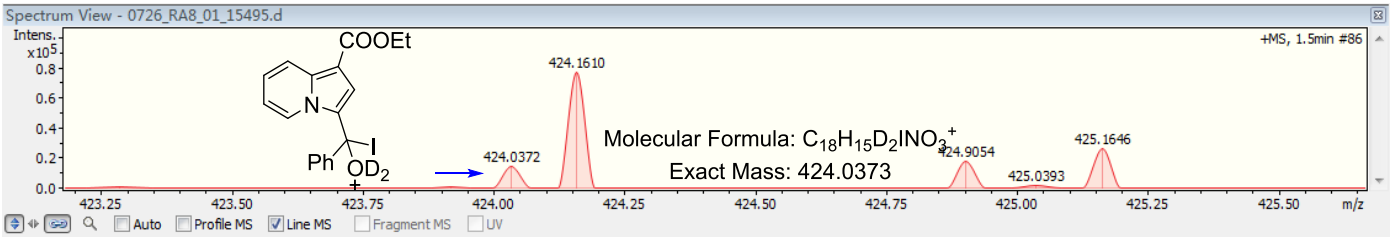


Figure S13: ESI-HMRS of $[M]^+$ for intermediate (**H**)

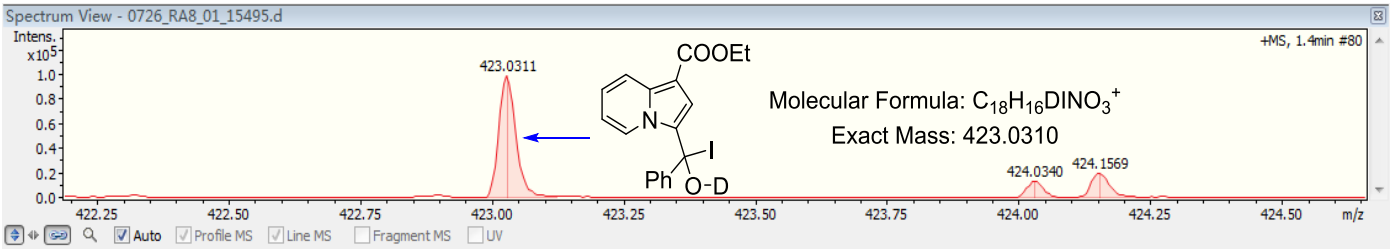


Figure S14: ESI-HMRS of $[M+H]^+$ for intermediate (**I**)

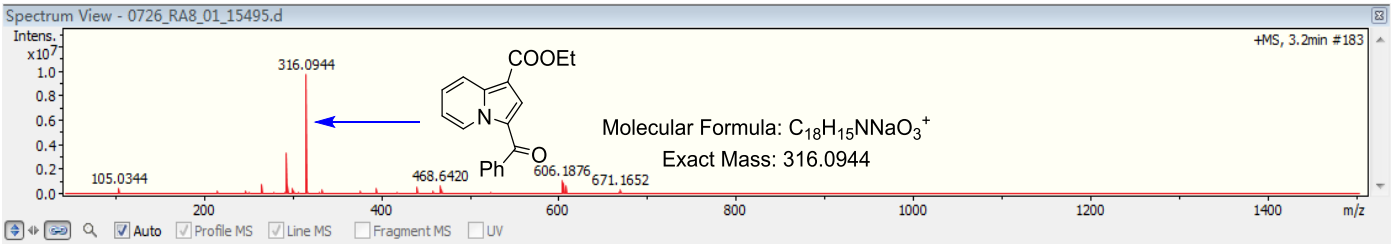


Figure S15: ESI-HMRS of $[M+Na]^+$ for **2a**

10 Cyclic Voltammetry Studies

Cyclic voltammograms were recorded with a CHI660E potentiostat at room temperature in MeCN. $^{n}Bu_4NPF_6$ (0.1 M) was used as the supporting electrolyte, and a Pt electrode (area = 0.03 cm^2) was used as the working electrode. The auxiliary electrode was a Pt sheet. All potentials are referenced against the SCE redox couple.

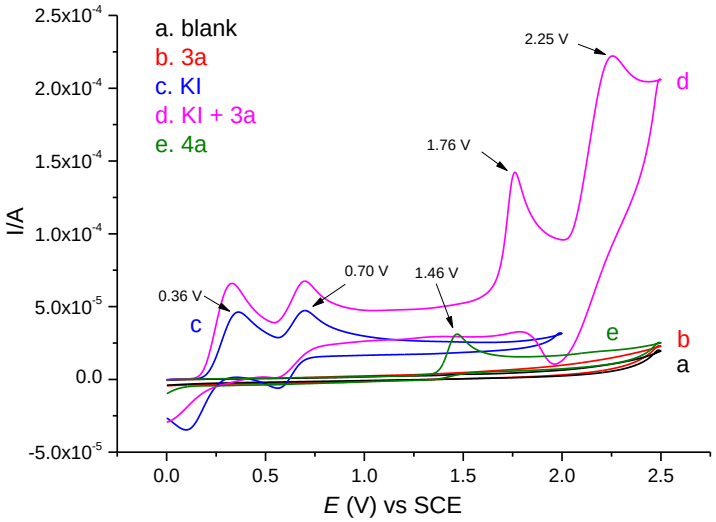


Figure S16: Cyclic voltammograms obtained in 0.1 M $^{n}Bu_4NPF_6$/MeCN using glass carbon (diameter, 3 mm) as the working electrode, Pt wire, and saturated calomel electrode (SCE) as the auxiliary and reference electrode, respectively, at a scan rate of 0.1 $V \cdot s^{-1}$: (a) background, (b) 5 mM **3a**, (c) 5 mM KI, (d) 5 mM 3**a** + 5 mM KI, (e) 5 mM **4a**.

11 X-ray Crystallographic Data

General Procedure for Compound 2a Crystal Preparation:

Compound **2a** (around 20 mg) were dissolved in PE-EtOAc (1:1, 10 mL). The single crystals were grown by slow evaporation of solvents at room temperature.

Compound **2a** was collected at 100 K on a Rigaku Oxford Diffraction Supernova Dual Source, Cu at Zero equipped with an AtlasS2 CCD. The data were collected and processed using CrysAlisPro. The structures were solved by direct methods using Olex2 software, and the non-hydrogen atoms were located from the trial structure and S26 then refined anisotropically with SHELXL-2018 using a full-matrix least squares procedure based on F^2 . The weighted R factor, wR and goodness-of-fit S values were obtained based on F^2. The hydrogen atom positions were fixed geometrically at the calculated distances and allowed to ride on their parent atoms. Crystallographic data for the structure reported in this paper have

been deposited at the Cambridge Crystallographic Data Center and allocated with the deposition number: CCDC 2123276 for compound **2a**, basic information pertaining to crystal parameters and structure refinement are summarized in Figure S16.

Compound 2a (CCDC 2123276)

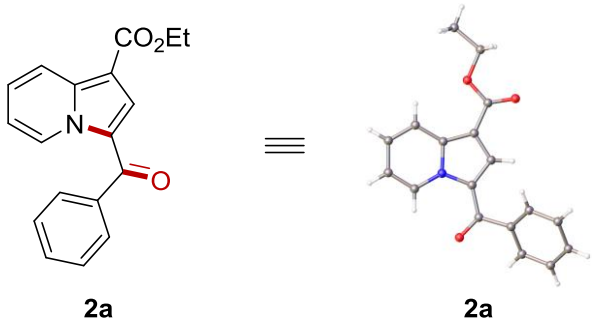


Figure S16 X-ray structure of **2a** with 50% ellipsoid probability

Empirical formula	$C_{18}H_{15}NO_3$
Formula weight	293.31
Temperature/K	150.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	7.8781(8)
b/Å	10.0442(7)
c/Å	18.5161(9)
α/°	97.017(5)
β/°	97.461(6)
γ/°	94.103(7)
Volume/Å^3	1436.18(19)
Z	4
ρ_{calc}g/cm^3	1.357
μ/mm^{-1}	0.093
F(000)	616.0
Crystal size/mm^3	0.15 × 0.13 × 0.12
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.102 to 59.208
Index ranges	-10 ≤ h ≤ 10, -13 ≤ k ≤ 13, -18 ≤ l ≤ 25
Reflections collected	12267
Independent reflections	6702 [R_{int} = 0.0408, R_{sigma} = 0.0686]
Data/restraints/parameters	6702/0/410
Goodness-of-fit on F^2	1.041
Final R indexes [I>=2σ (I)]	R_1 = 0.0597, wR_2 = 0.1406
Final R indexes [all data]	R_1 = 0.0899, wR_2 = 0.1646
Largest diff. peak/hole / e Å^{-3}	0.27/-0.28

General Procedure for Compound 2z Crystal Preparation:

Compound **2z** (around 20 mg) were dissolved in PE-EtOAc (1:1, 10 mL). The single crystals were grown by slow evaporation of solvents at room temperature.

Compound **2z** was collected at 100 K on a Rigaku Oxford Diffraction Supernova Dual Source, Cu at Zero equipped with an AtlasS2 CCD. The data were collected and processed using CrysAlisPro. The structures were solved by direct methods using Olex2 software, and the non-hydrogen atoms were located from the trial structure and S26 then refined anisotropically with SHELXL-2018 using a full-matrix least squares procedure based on F^2. The weighted R factor, wR and goodness-of-fit S values were obtained based on F^2. The hydrogen atom positions were fixed geometrically at the calculated distances and allowed to ride on their parent atoms. Crystallographic data for the structure reported in this paper have been deposited at the Cambridge Crystallographic Data Center and allocated with the deposition number: CCDC 2123277 for compound **2z**, basic information pertaining to crystal parameters and structure refinement are summarized in Figure S17.

Compound 2z (CCDC 2123277)

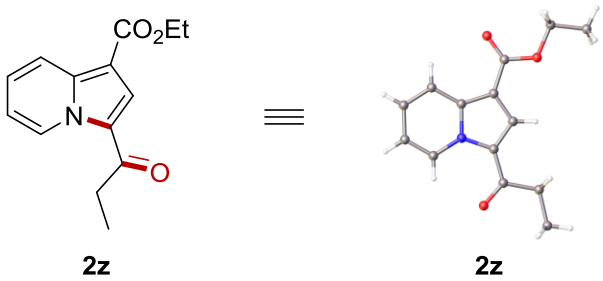


Figure S17 X-ray structure of **2z** with 50% ellipsoid probability

Empirical formula	$C_{14}H_{15}NO_3$
Formula weight	245.27
Temperature/K	150.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	7.4629(9)
b/Å	8.7979(11)

c/Å	10.1086(11)
α/°	100.975(10)
β/°	95.517(9)
γ/°	109.597(11)
Volume/Å^3	604.57(13)
Z	2
ρ_{calc}g/cm^3	1.347
μ/mm^{-1}	0.095
F(000)	260.0
Crystal size/mm^3	0.14 × 0.13 × 0.12
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.168 to 49.992
Index ranges	-8 ≤ h ≤ 8, -10 ≤ k ≤ 9, -10 ≤ l ≤ 12
Reflections collected	3842
Independent reflections	2132 [R_{int} = 0.0296, R_{sigma} = 0.0472]
Data/restraints/parameters	2132/0/165
Goodness-of-fit on F^2	1.097
Final R indexes [I>=2σ (I)]	R_1 = 0.0516, wR_2 = 0.1297
Final R indexes [all data]	R_1 = 0.0686, wR_2 = 0.1428
Largest diff. peak/hole / e Å^{-3}	0.21/-0.21

General Procedure for Compound 4c Crystal Preparation:

Compound **4c** (around 20 mg) were dissolved in PE-EtOAc (1:1, 10 mL). The single crystals were grown by slow evaporation of solvents at room temperature.

Compound **4c** was collected at 100 K on a Rigaku Oxford Diffraction Supernova Dual Source, Cu at Zero equipped with an AtlasS2 CCD. The data were collected and processed using CrysAlisPro. The structures were solved by direct methods using Olex2 software, and the non-hydrogen atoms were located from the trial structure and S26 then refined anisotropically with SHELXL-2018 using a full-matrix least squares procedure based on F^2. The weighted R factor, wR and goodness-of-fit S values were obtained based on F^2. The hydrogen atom positions were fixed geometrically at the calculated distances and allowed to ride on their parent atoms. Crystallographic data for the structure reported in this paper have been deposited at the Cambridge Crystallographic Data Center and allocated with the

deposition number: CCDC 2169509 for compound **4c**, basic information pertaining to crystal parameters and structure refinement are summarized in Figure S18.

Compound 4c (CCDC 2169509)

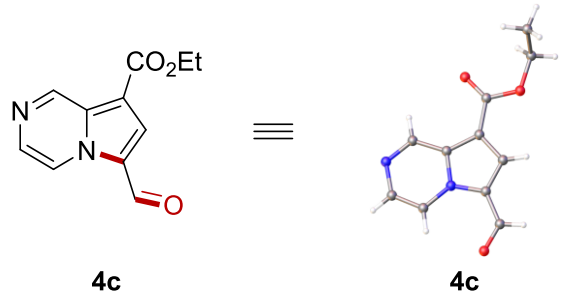


Figure S18: X-ray structure of **4c** with 50% ellipsoid probability

Empirical formula	$C_{11}H_{10}N_2O_3$
Formula weight	218.21
Temperature/K	169.99(10)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	4.2133(13)
b/Å	12.152(4)
c/Å	19.952(7)
α/°	90
β/°	93.32(3)
γ/°	90
Volume/Å^3	1019.8(6)
Z	4
ρ_{calc}g/cm^3	1.421
μ/mm^{-1}	0.106
F(000)	456.0
Crystal size/mm^3	0.14 × 0.13 × 0.12
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.09 to 49.976
Index ranges	$-5 \le h \le 4$, $-14 \le k \le 9$, $-20 \le l \le 23$
Reflections collected	4179
Independent reflections	1772 [R_{int} = 0.0545, R_{sigma} = 0.0742]
Data/restraints/parameters	1772/0/146
Goodness-of-fit on F^2	1.057
Final R indexes [I>=2σ (I)]	R_1 = 0.0552, wR_2 = 0.1140
Final R indexes [all data]	R_1 = 0.0842, wR_2 = 0.1330
Largest diff. peak/hole / e Å^{-3}	0.25/-0.26

12. Reference

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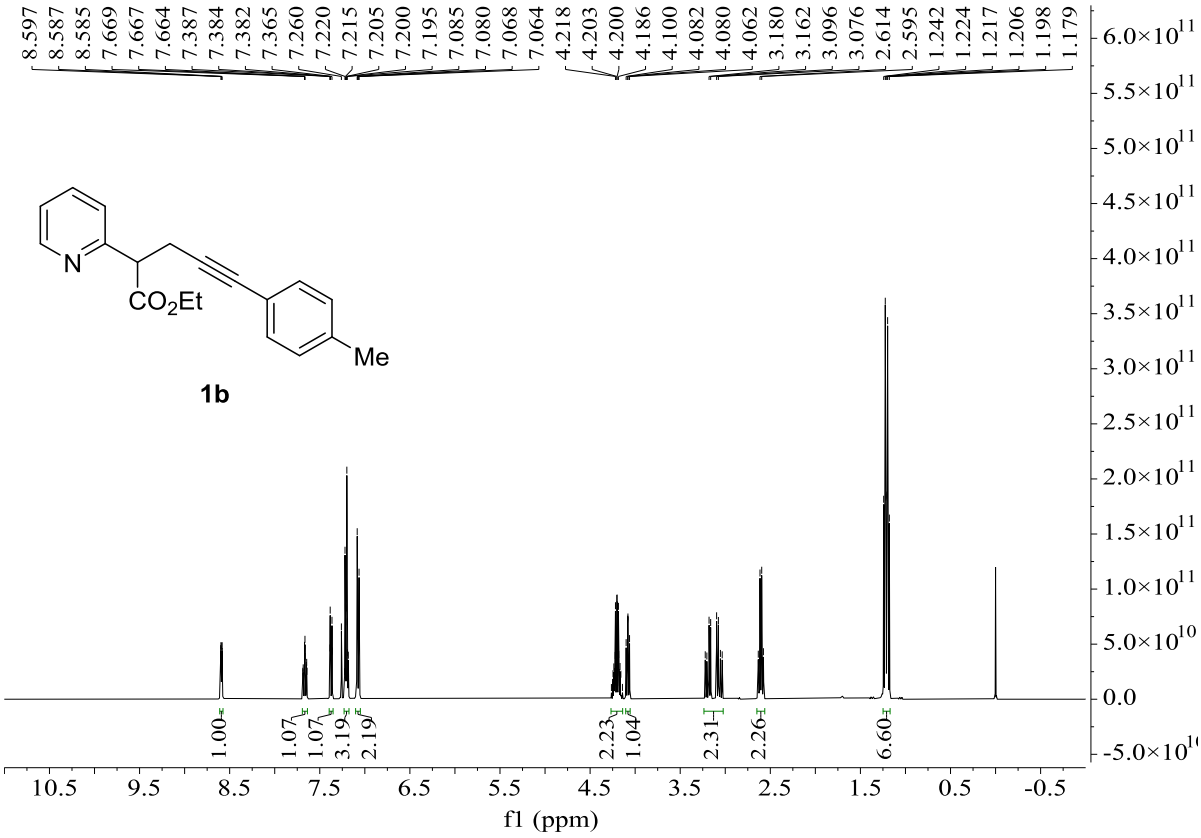
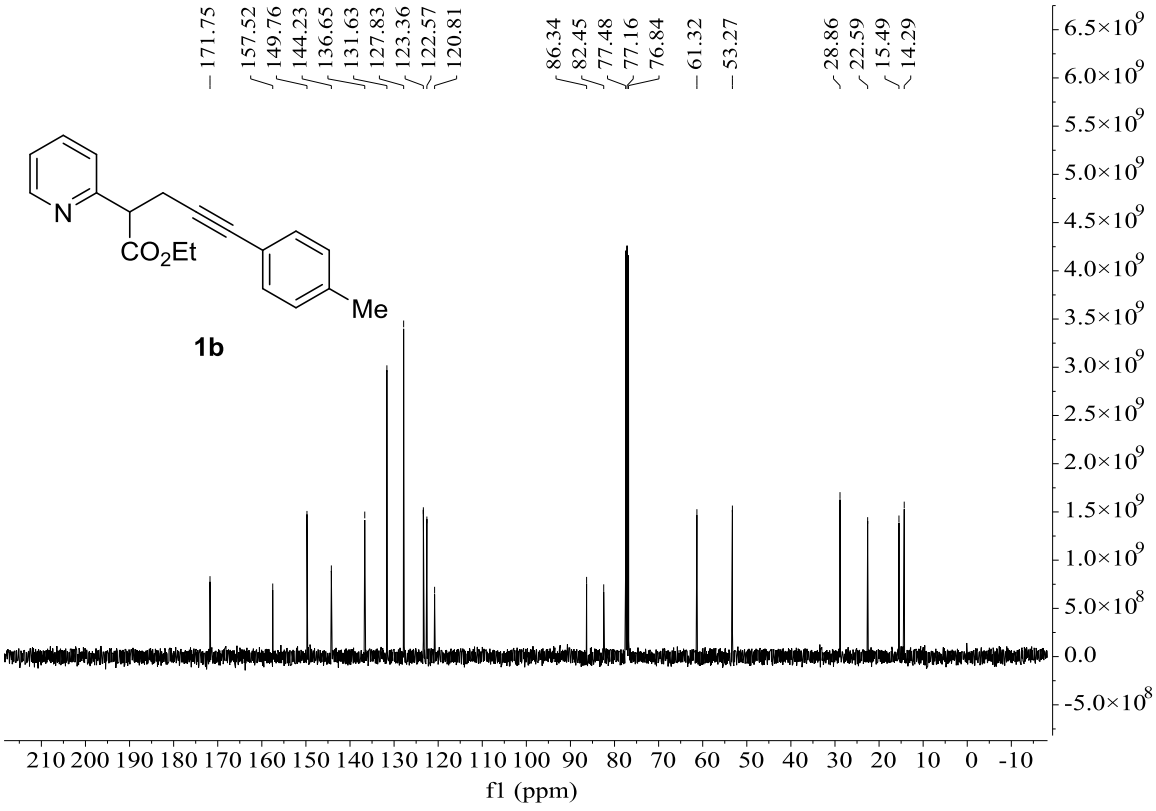
13 NMR Spectra

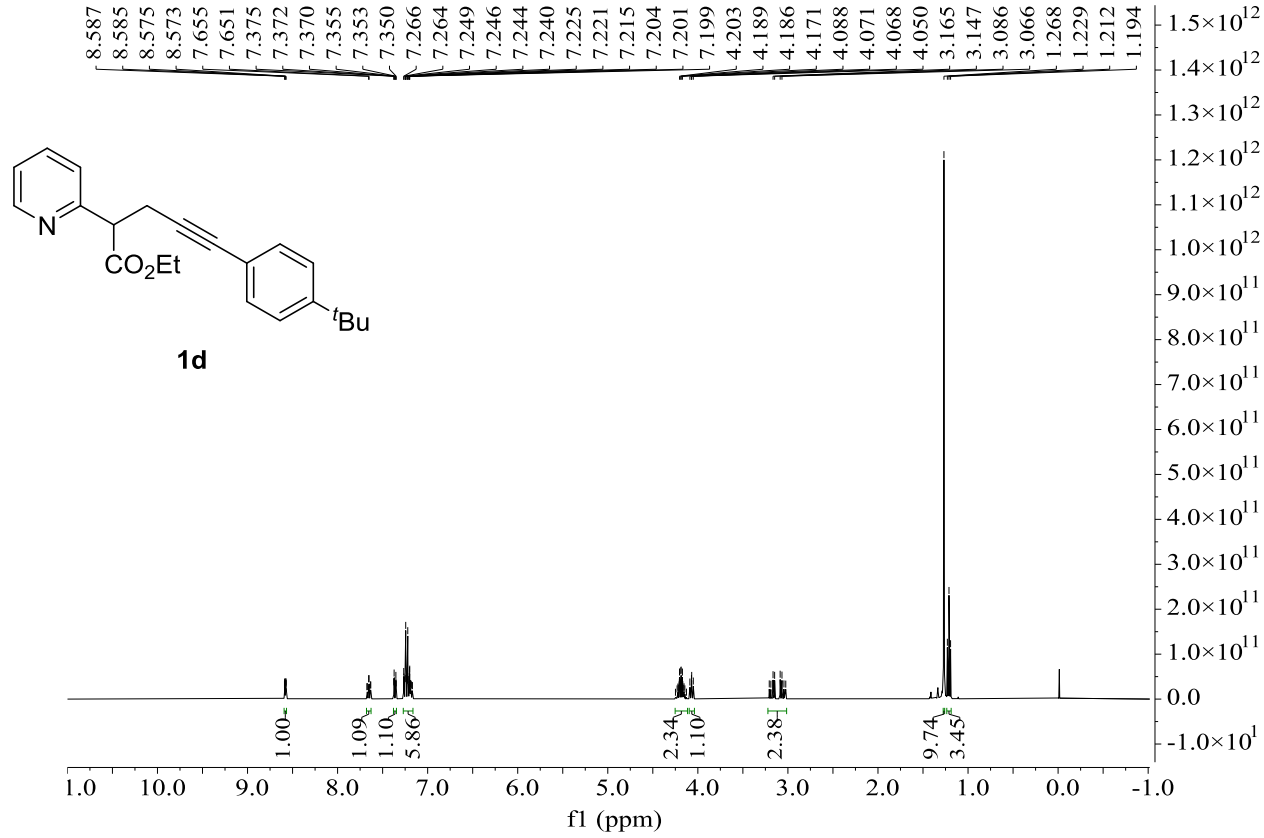
^{1}H NMR Spectrum of 1b at 25 °C ($CDCl_3$, 400 MHz)



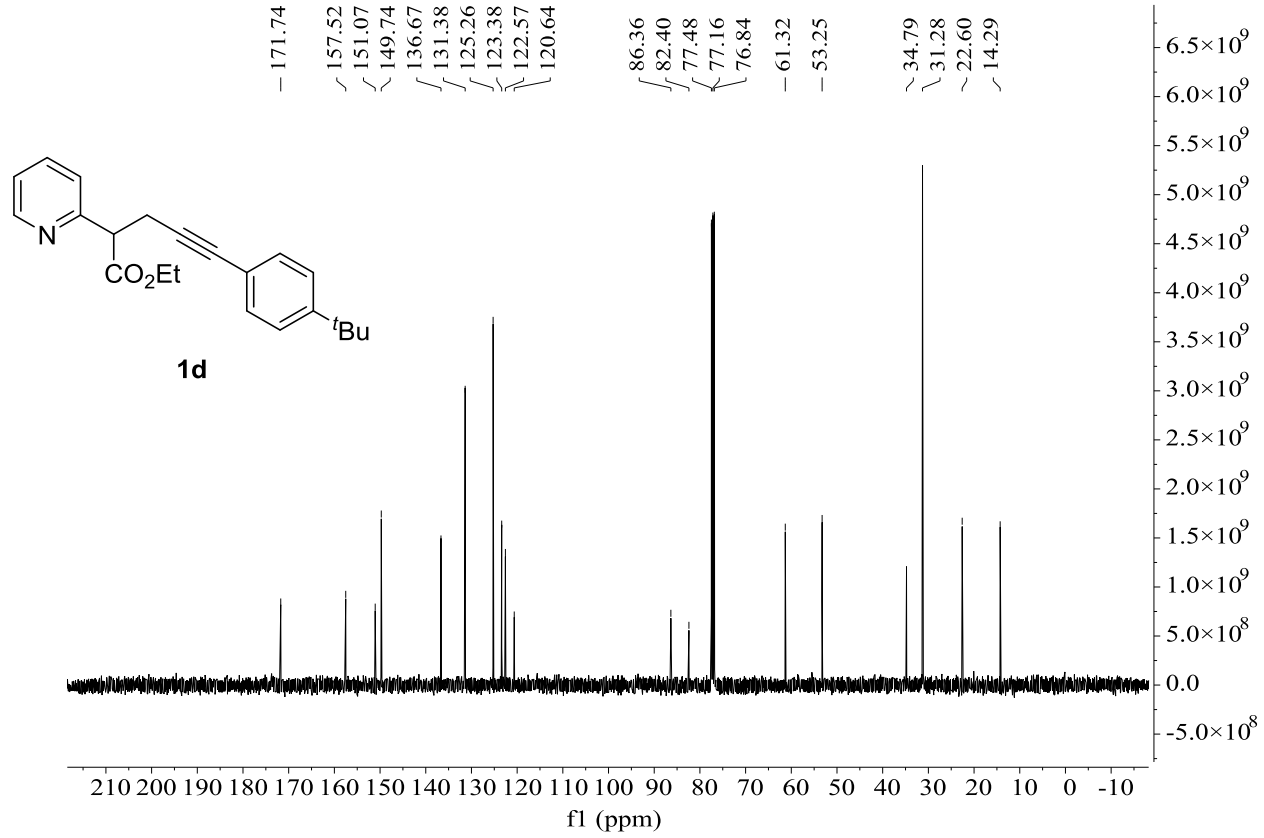
^{13}C NMR Spectrum of 1b at 25 °C ($CDCl_3$, 100 MHz)



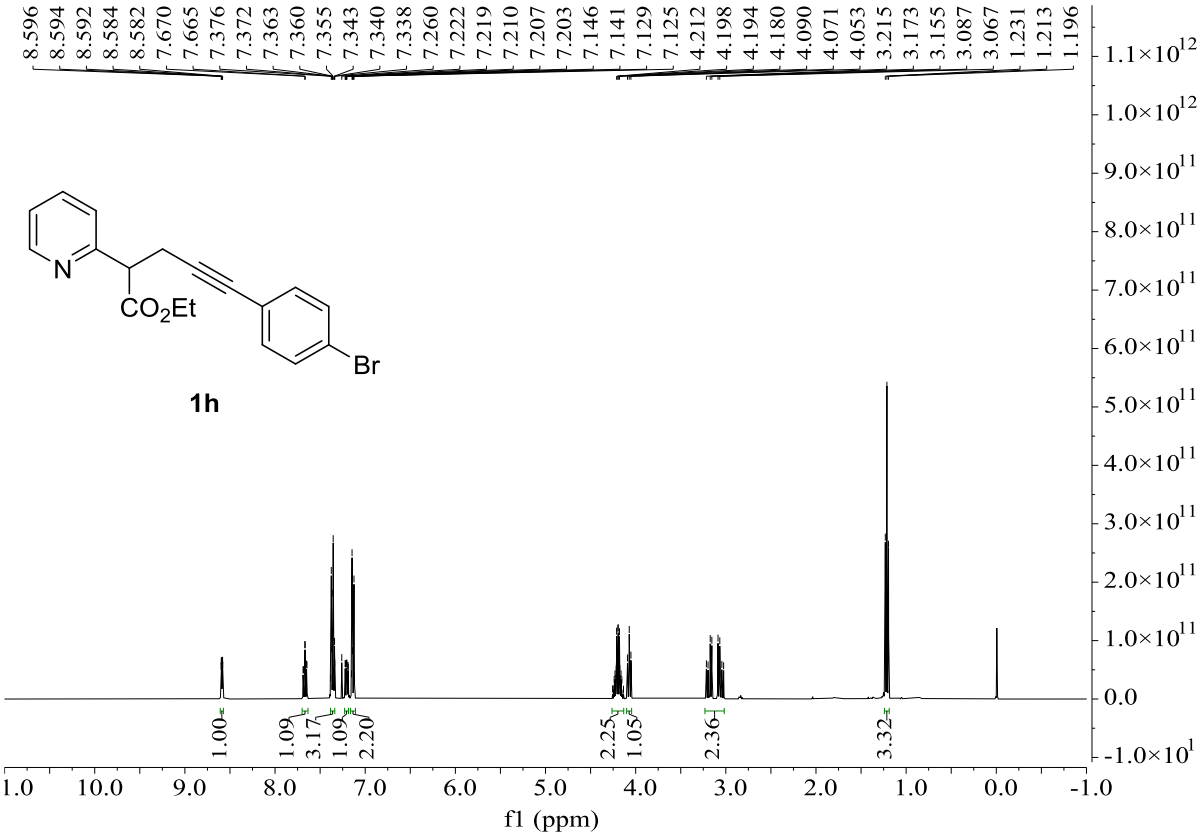
^{1}H NMR Spectrum of 1d at 25 °C ($CDCl_3$, 400 MHz)



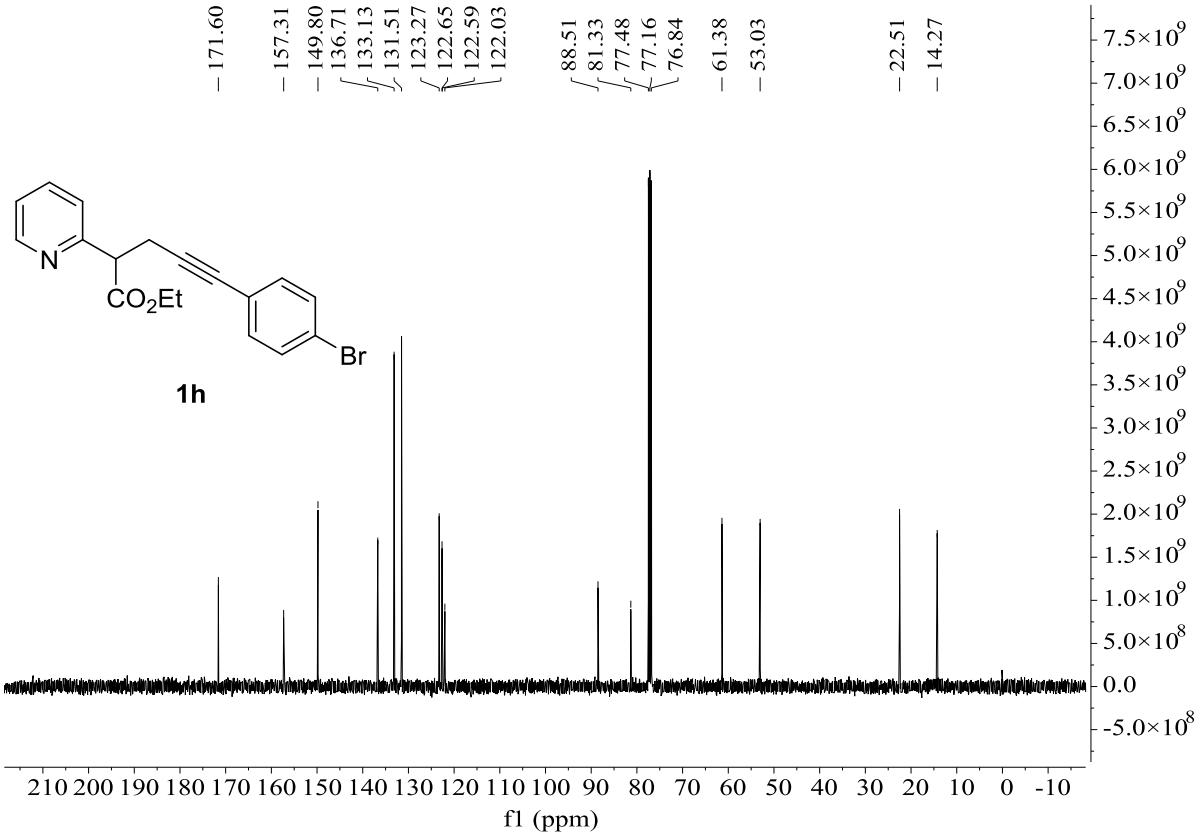
^{13}C NMR Spectrum of 1d at 25 °C ($CDCl_3$, 100 MHz)



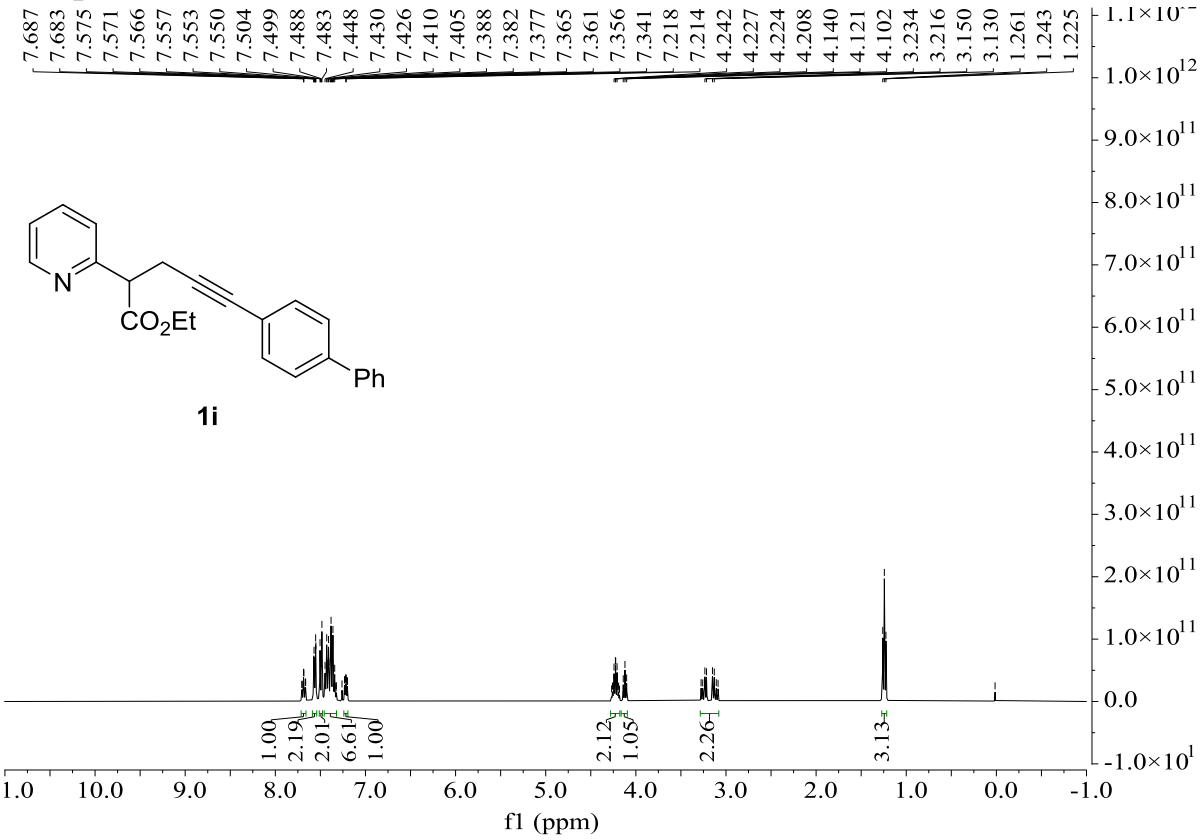
^{1}H NMR Spectrum of 1h at 25 °C ($CDCl_3$, 400 MHz)



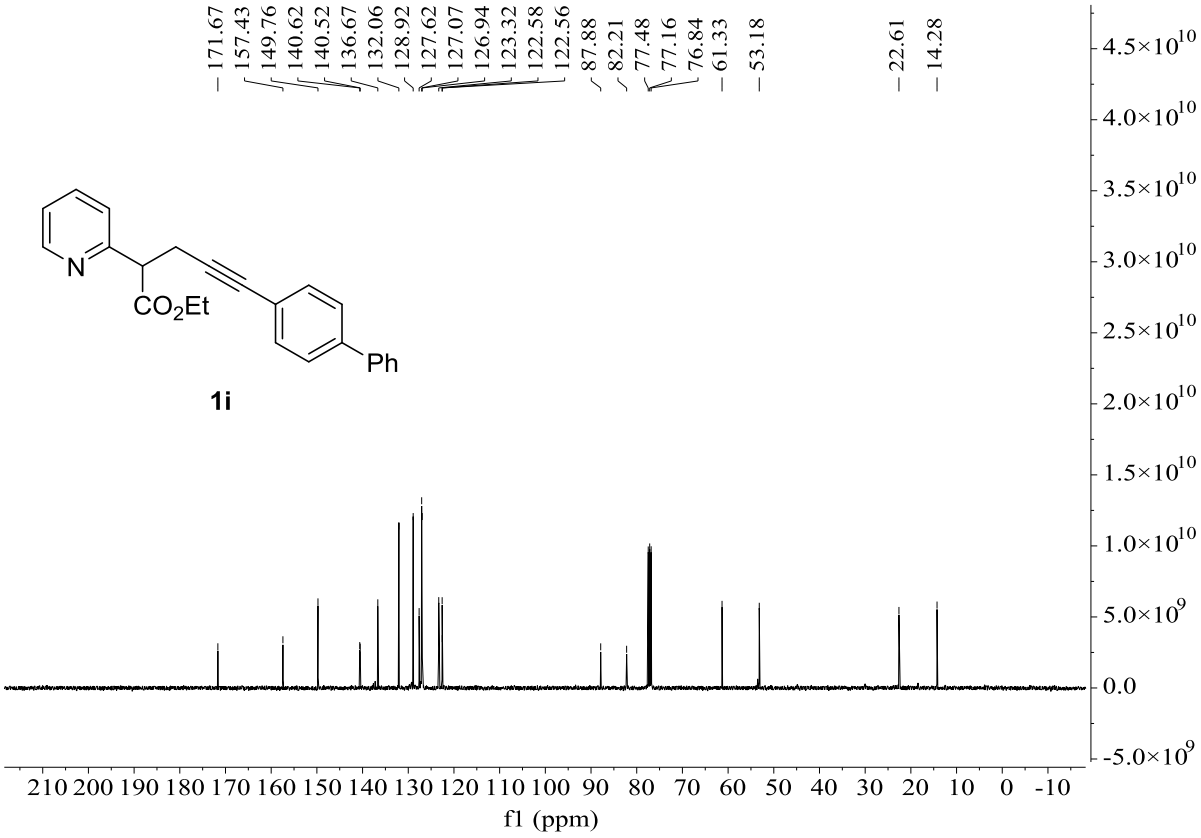
^{13}C NMR Spectrum of 1h at 25 °C ($CDCl_3$, 100 MHz)



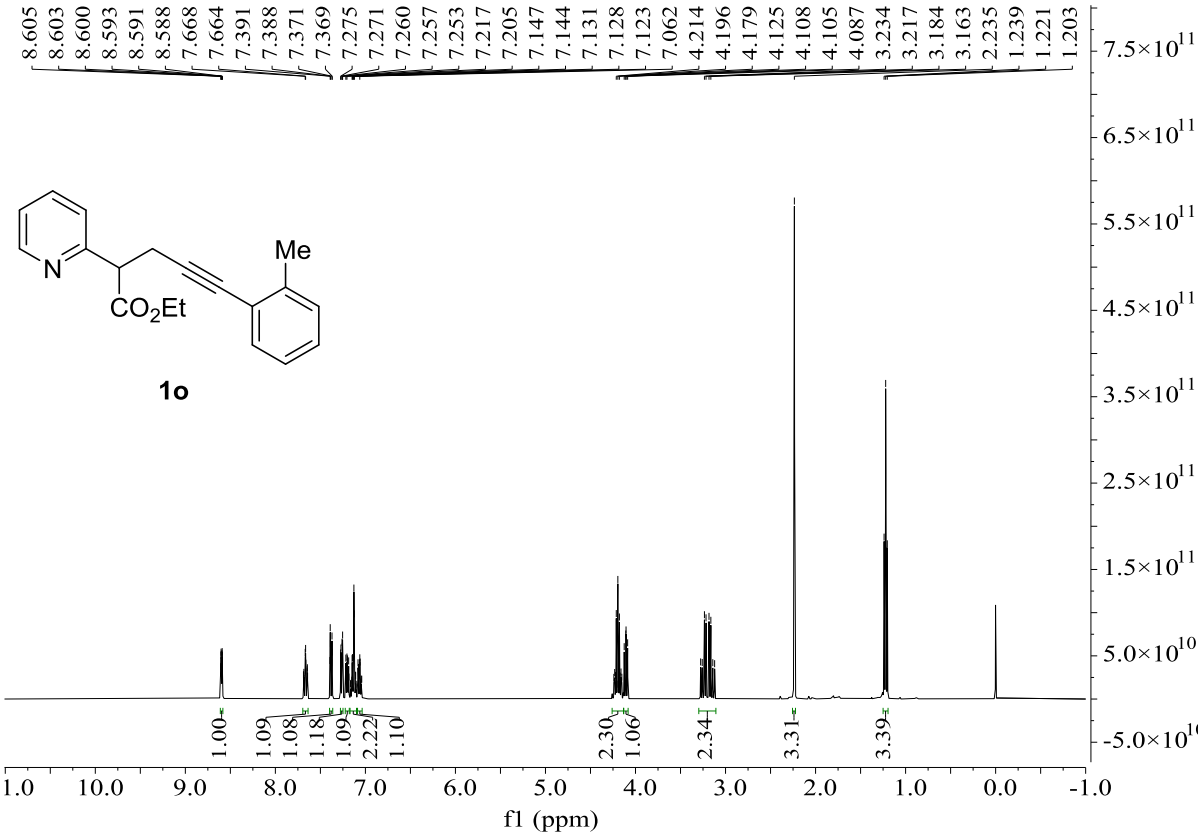
^{1}H NMR Spectrum of 1i at 25 °C ($CDCl_3$, 400 MHz)



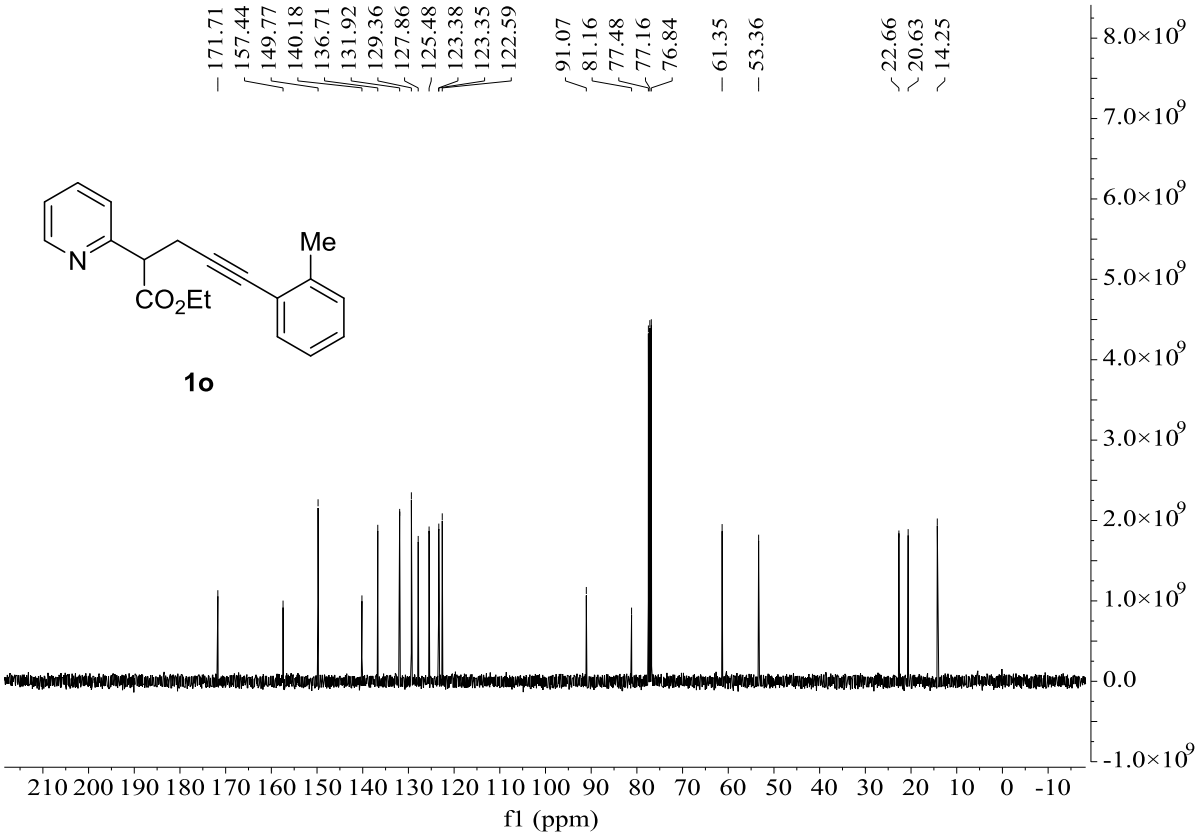
^{13}C NMR Spectrum of 1i at 25 °C ($CDCl_3$, 100 MHz)



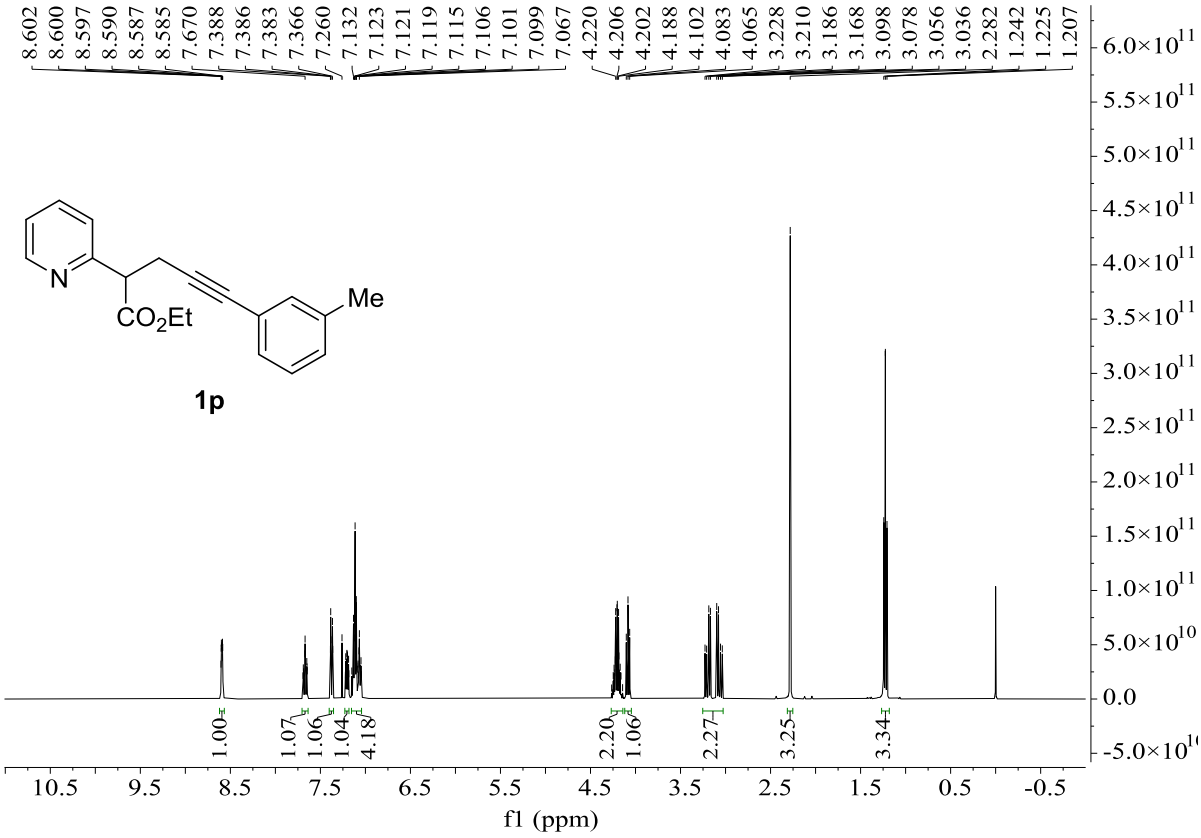
^{1}H NMR Spectrum of 1o at 25 °C ($CDCl_3$, 400 MHz)



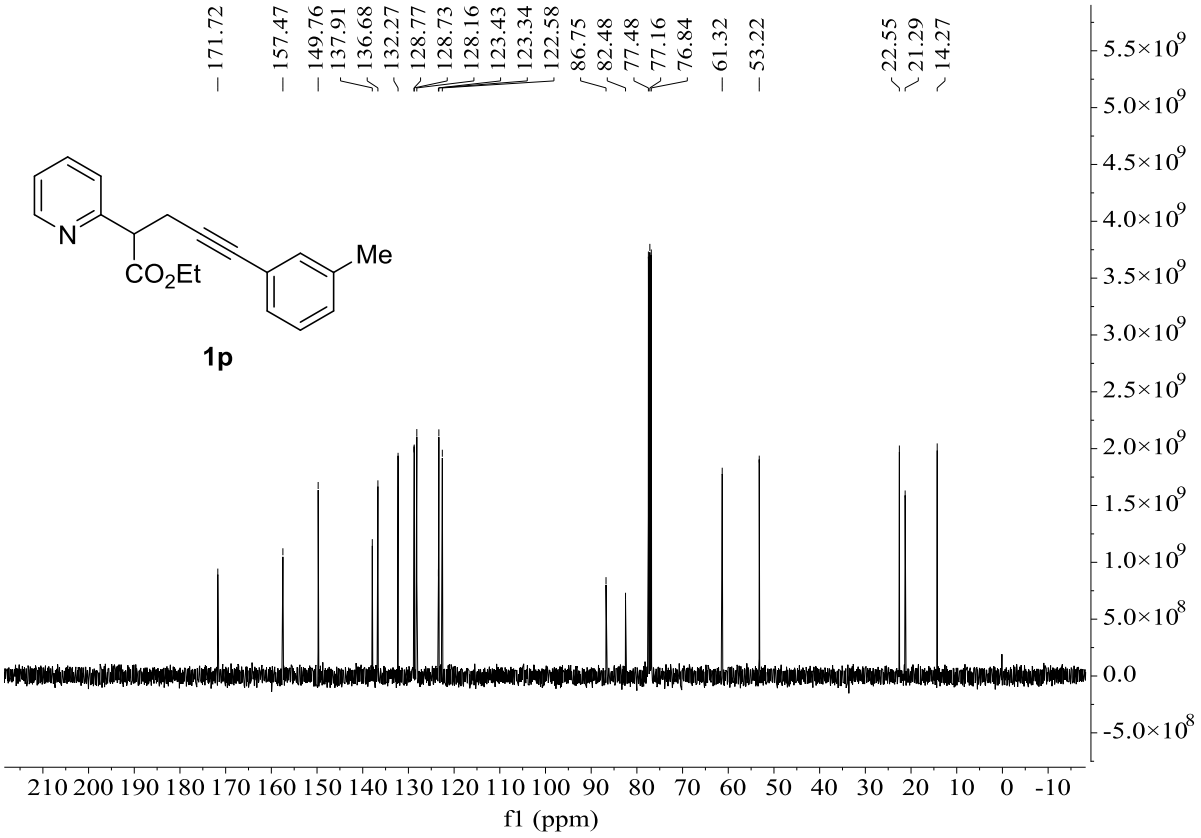
^{13}C NMR Spectrum of 1o at 25 °C ($CDCl_3$, 100 MHz)



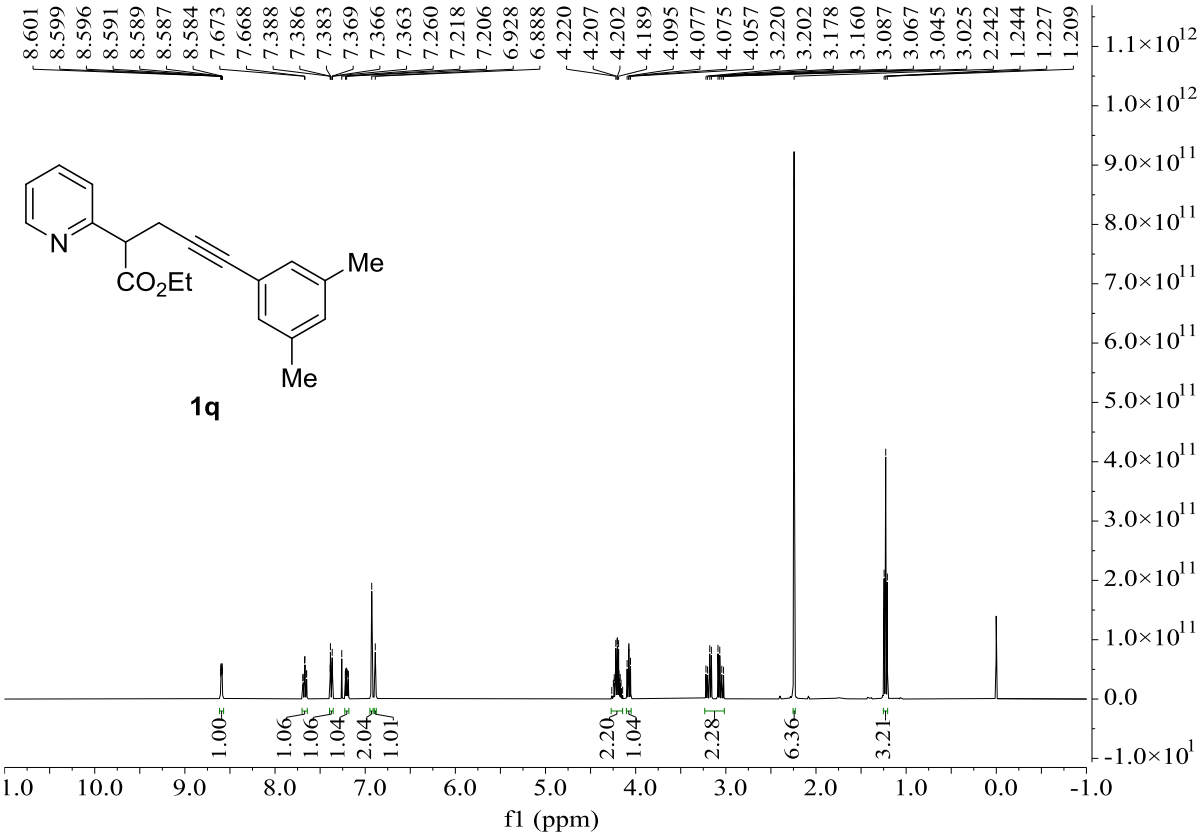
^{1}H NMR Spectrum of 1p at 25 °C ($CDCl_3$, 400 MHz)



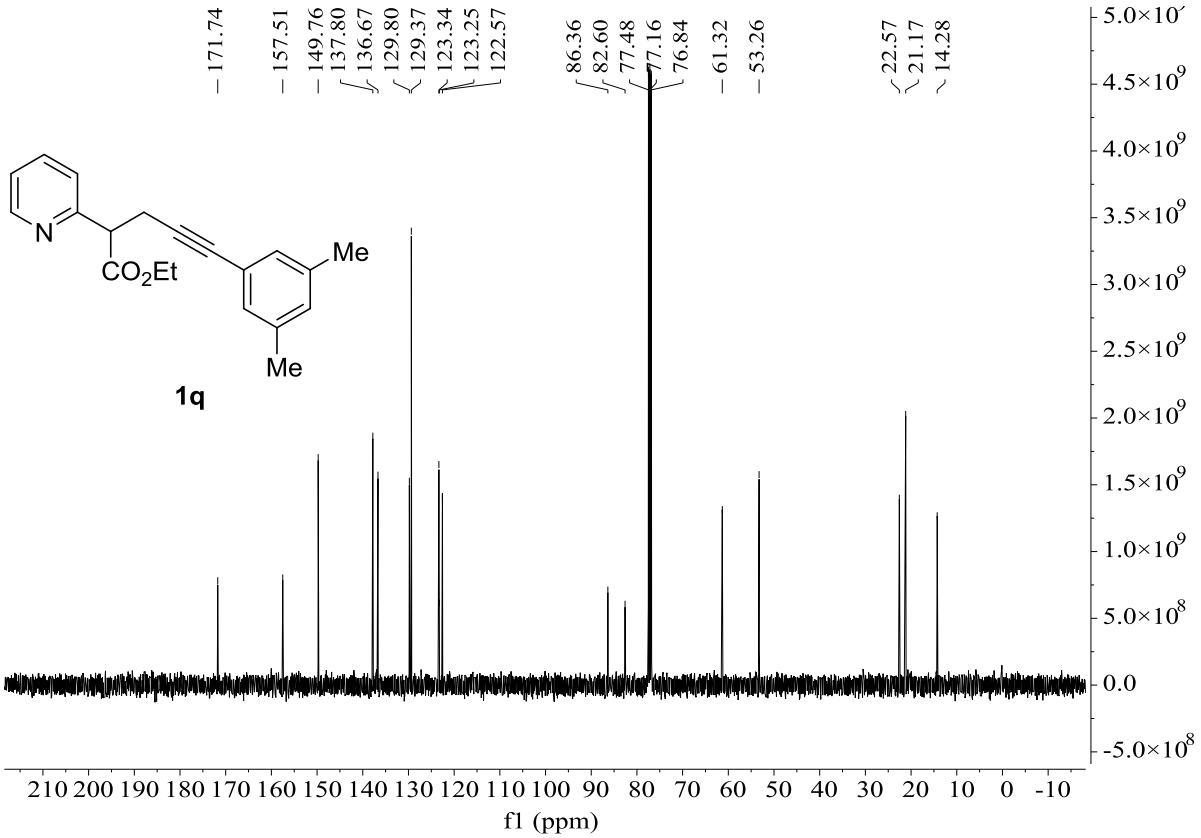
^{13}C NMR Spectrum of 1p at 25 °C ($CDCl_3$, 100 MHz)



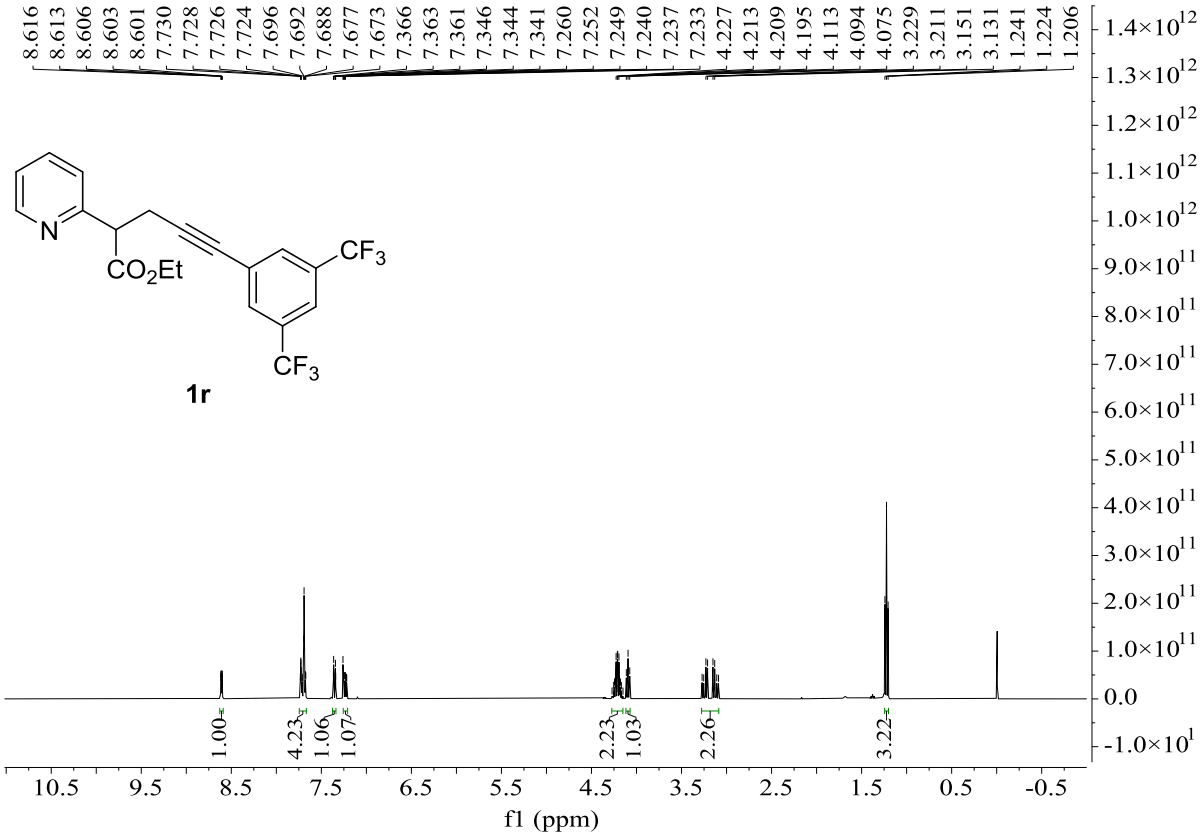
^{1}H NMR Spectrum of 1q at 25 °C ($CDCl_3$, 400 MHz)



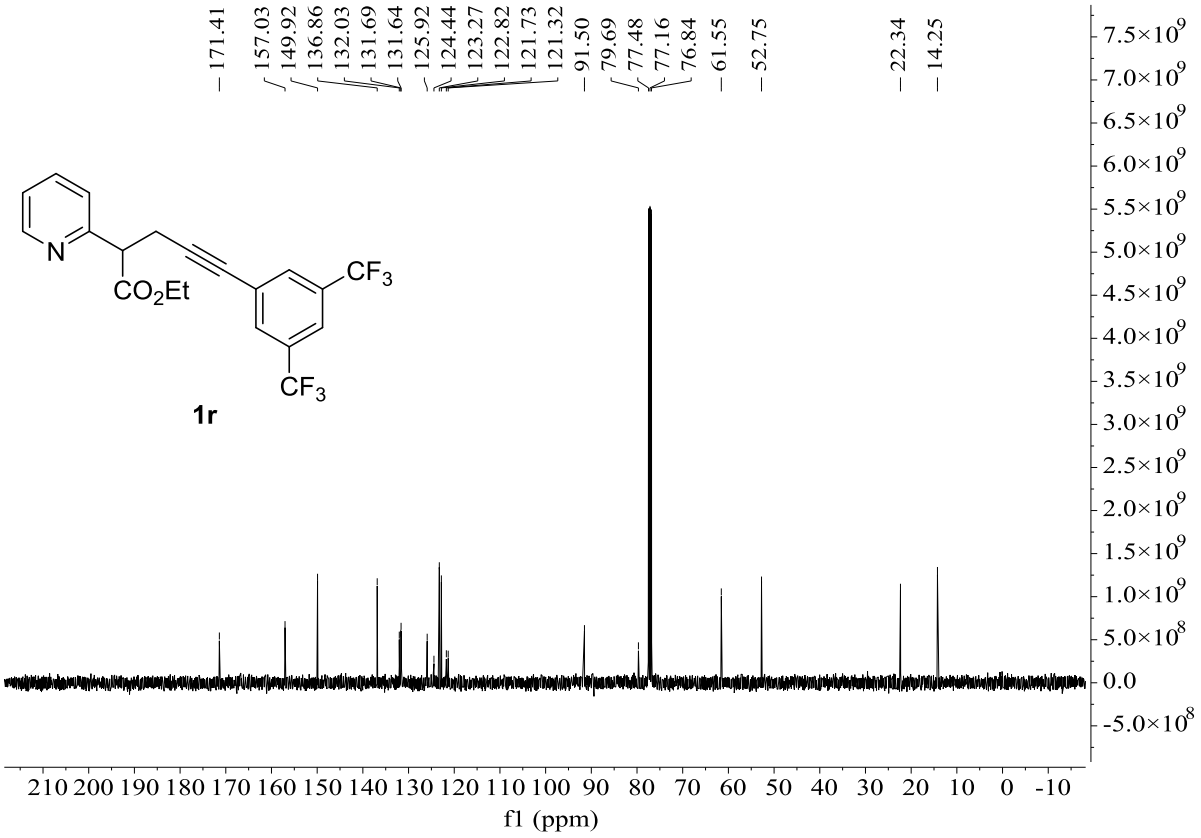
^{13}C NMR Spectrum of 1q at 25 °C ($CDCl_3$, 100 MHz)



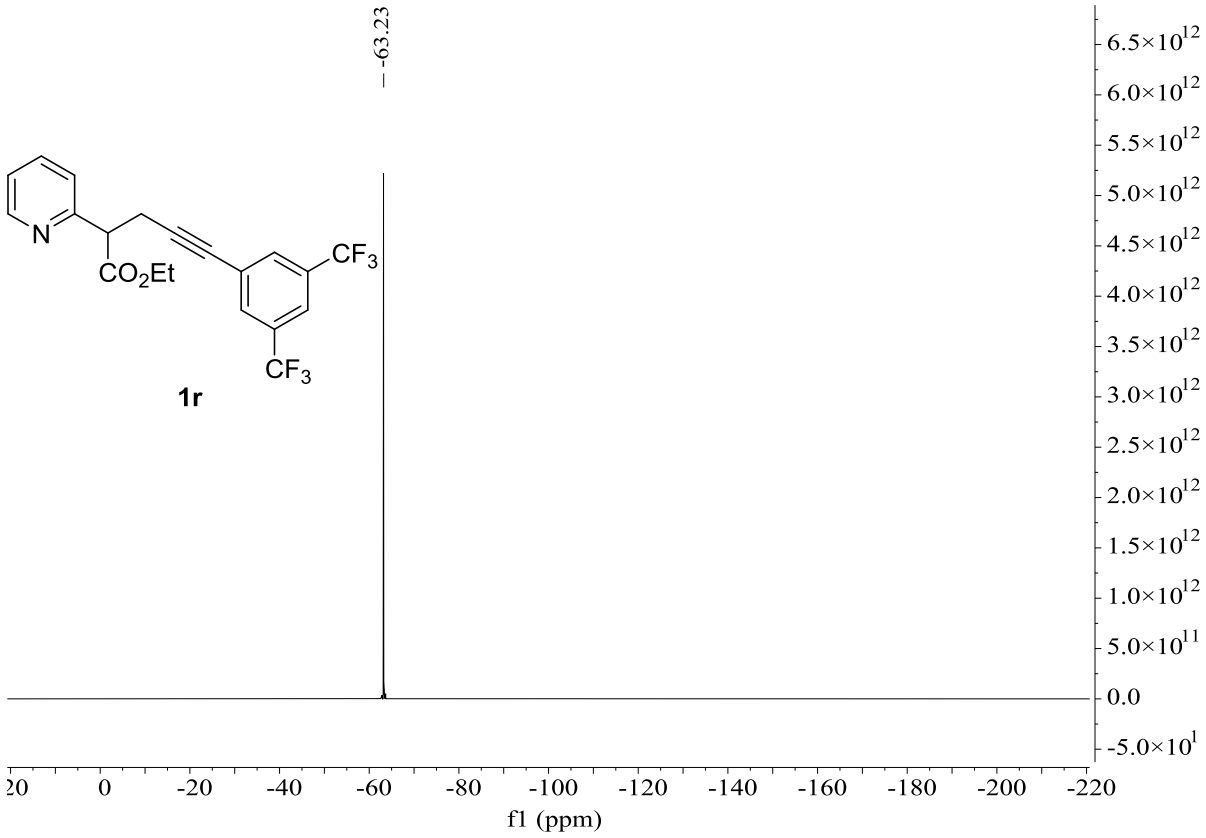
^{1}H NMR Spectrum of 1r at 25 °C ($CDCl_3$, 400 MHz)



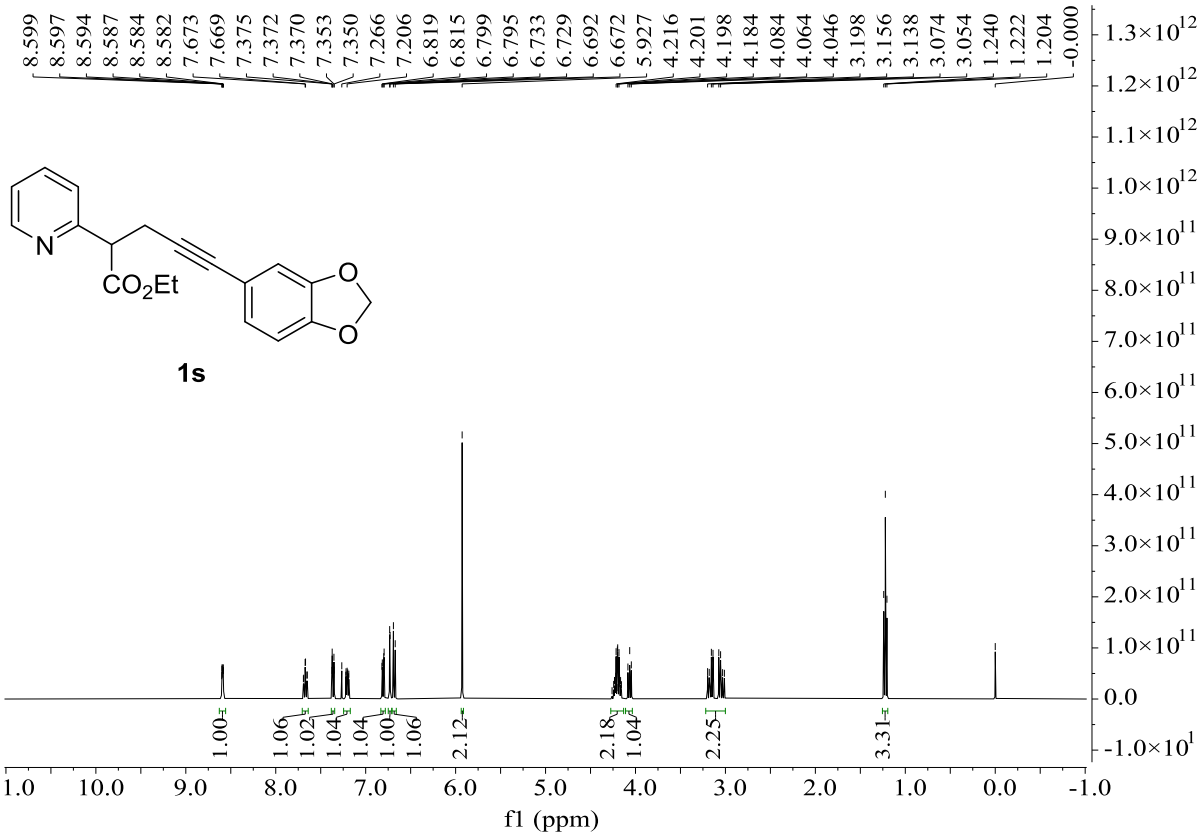
^{13}C NMR Spectrum of 1r at 25 °C ($CDCl_3$, 100 MHz)



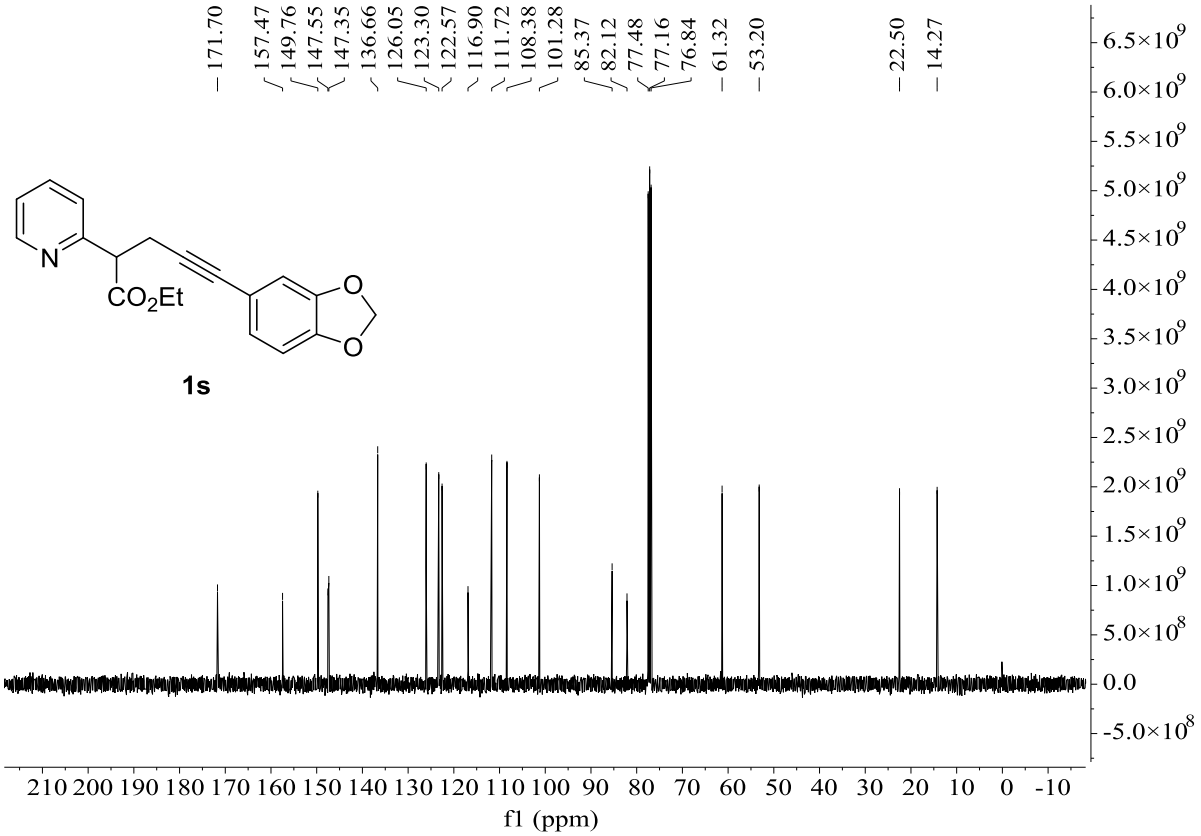
^{19}F NMR Spectrum of 1r at 25 °C ($CDCl_3$, 376 MHz)



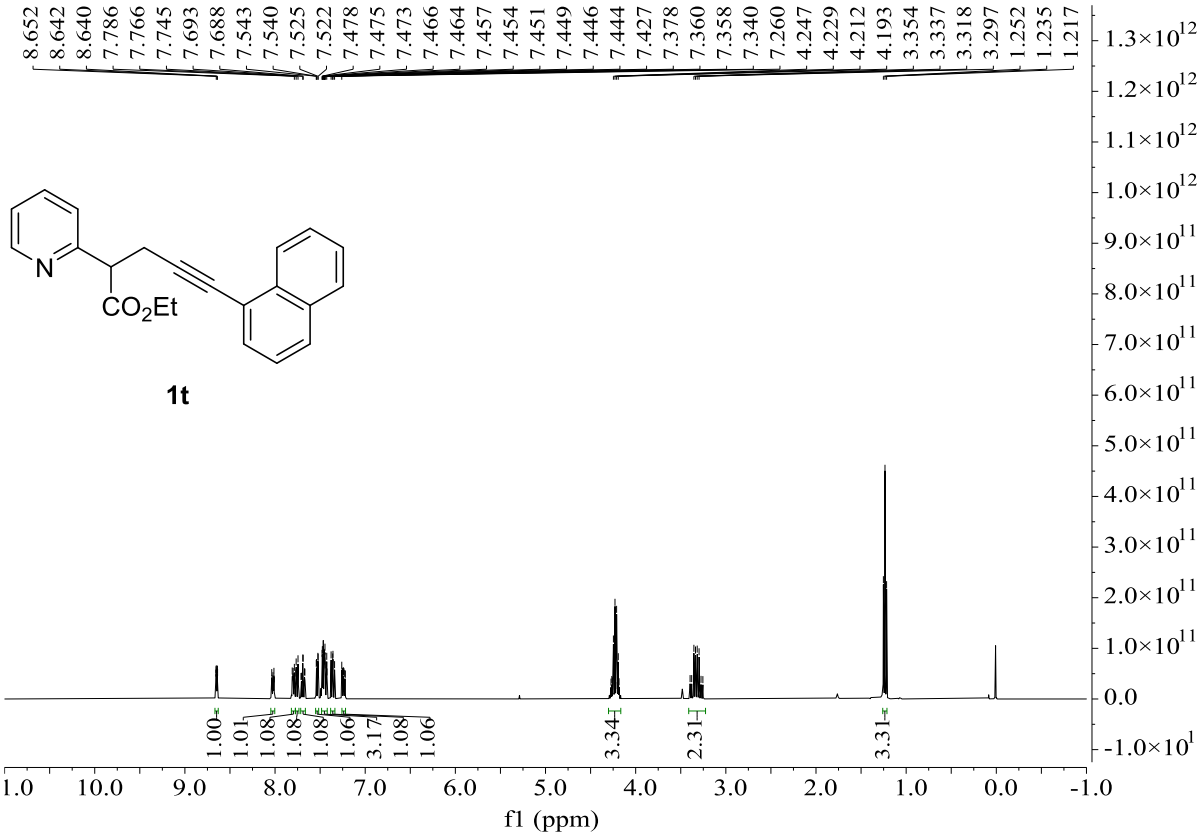
^{1}H NMR Spectrum of 1s at 25 °C ($CDCl_3$, 400 MHz)



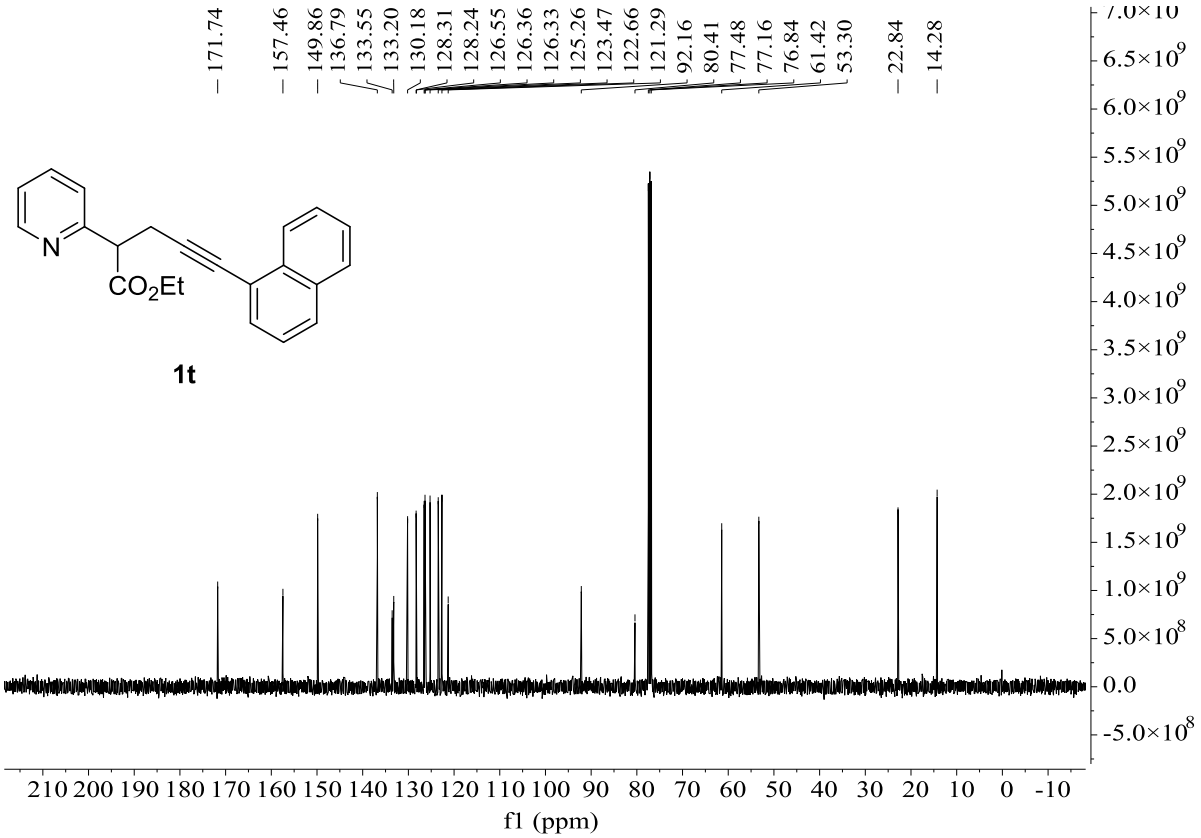
^{13}C NMR Spectrum of 1s at 25 °C ($CDCl_3$, 100 MHz)



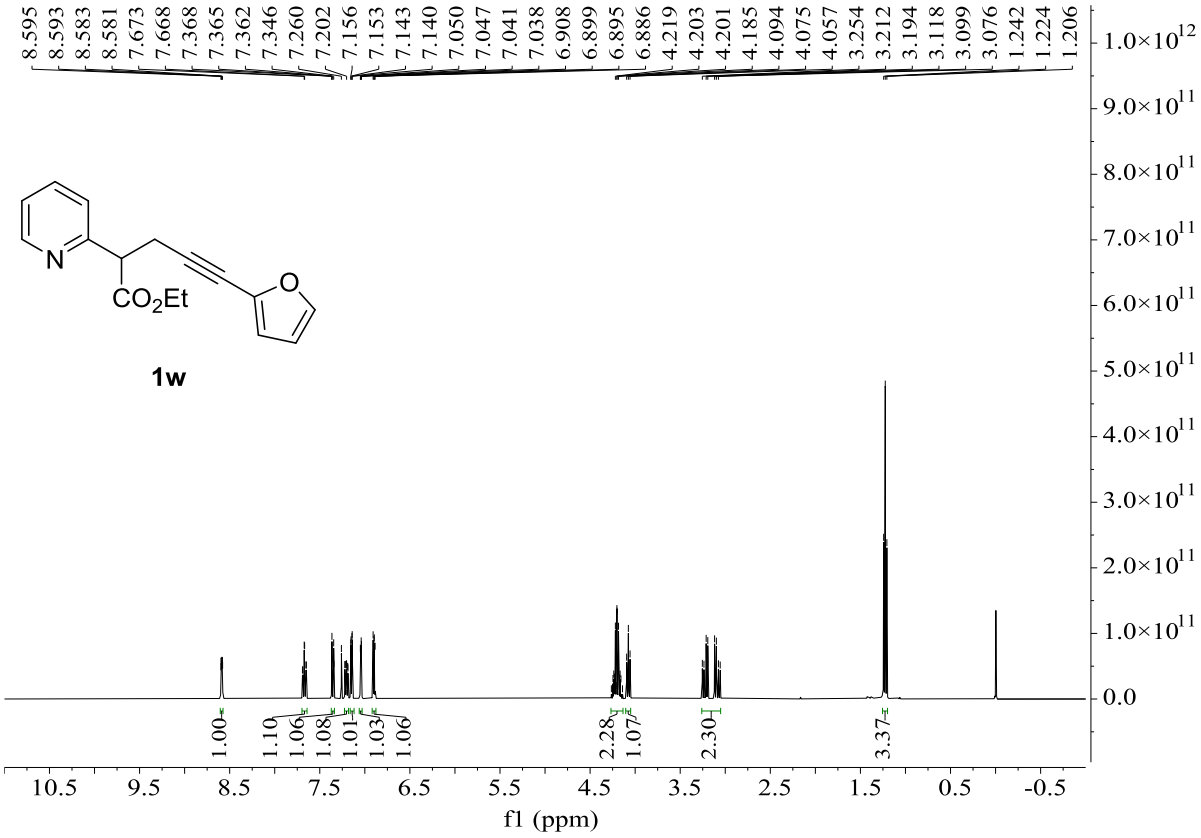
^{1}H NMR Spectrum of 1t at 25 °C ($CDCl_3$, 400 MHz)



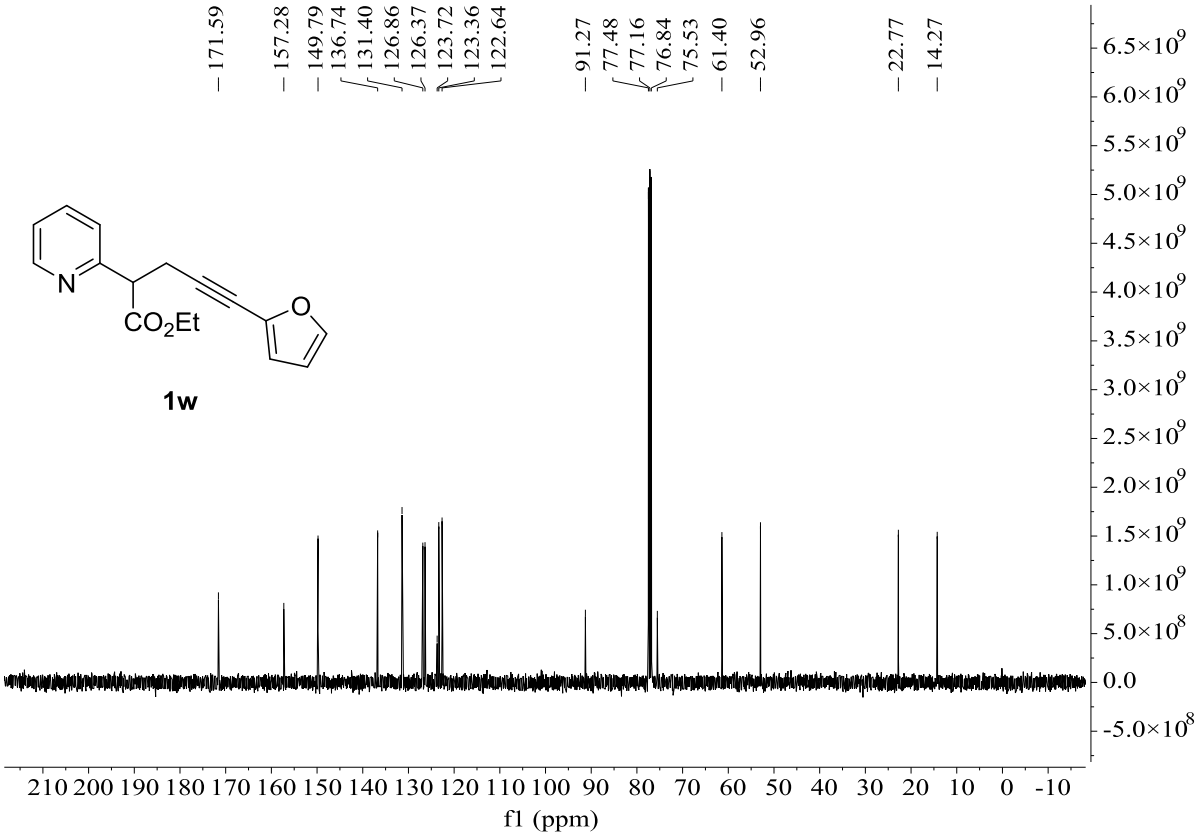
^{13}C NMR Spectrum of 1t at 25 °C ($CDCl_3$, 100 MHz)



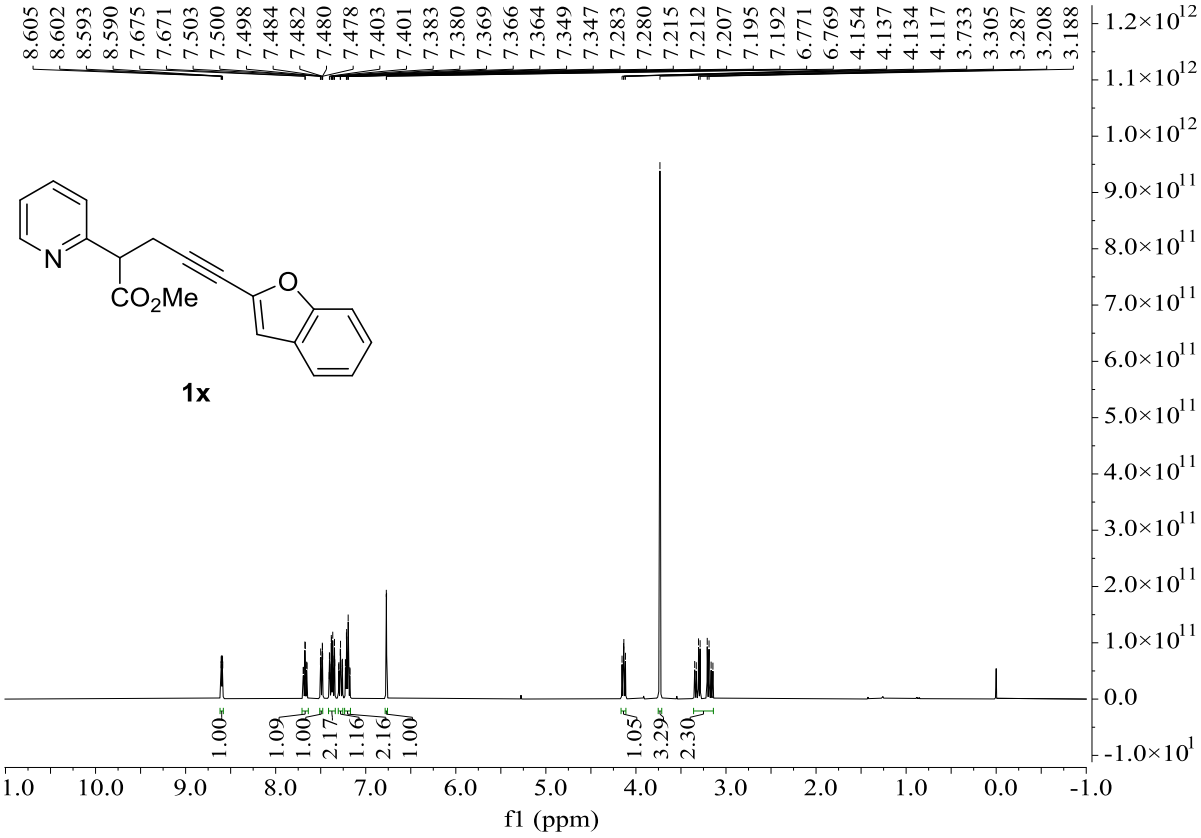
^{1}H NMR Spectrum of 1w at 25 °C ($CDCl_3$, 400 MHz)



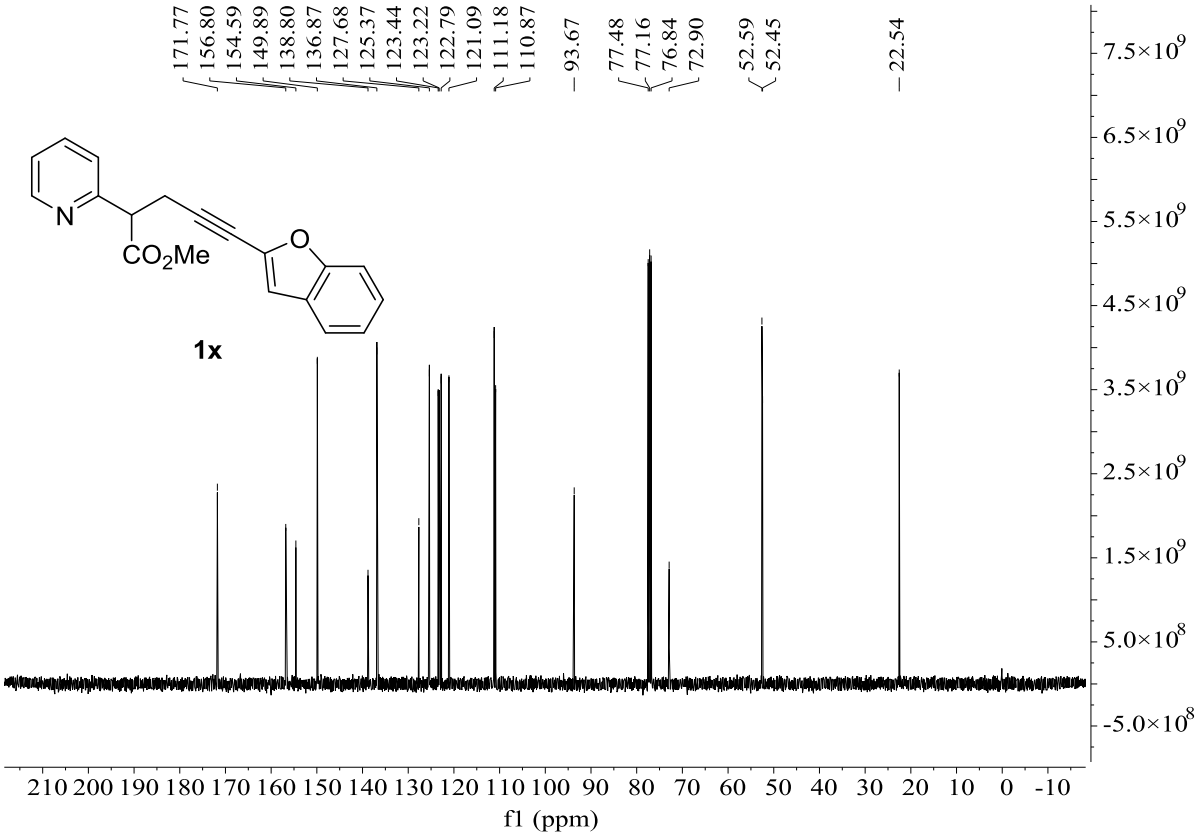
^{13}C NMR Spectrum of 1w at 25 °C ($CDCl_3$, 100 MHz)



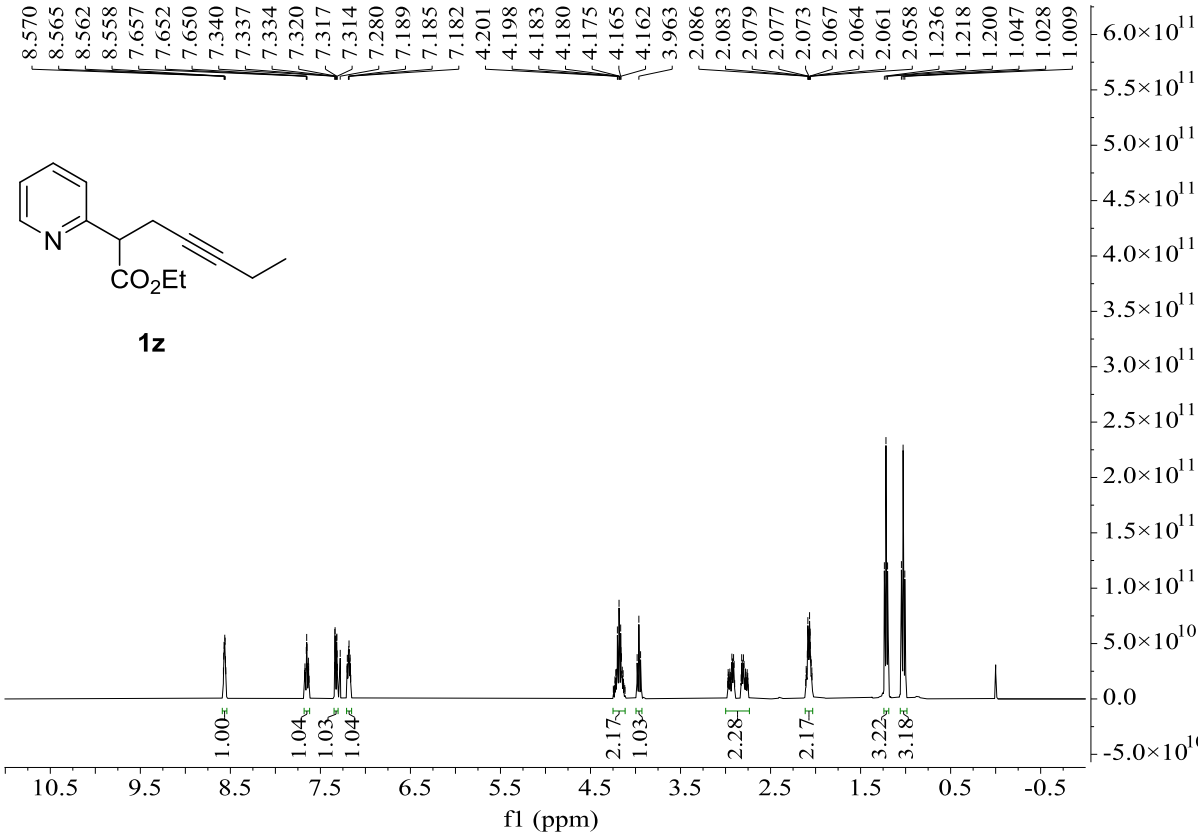
^{1}H NMR Spectrum of 1x at 25 °C ($CDCl_3$, 400 MHz)



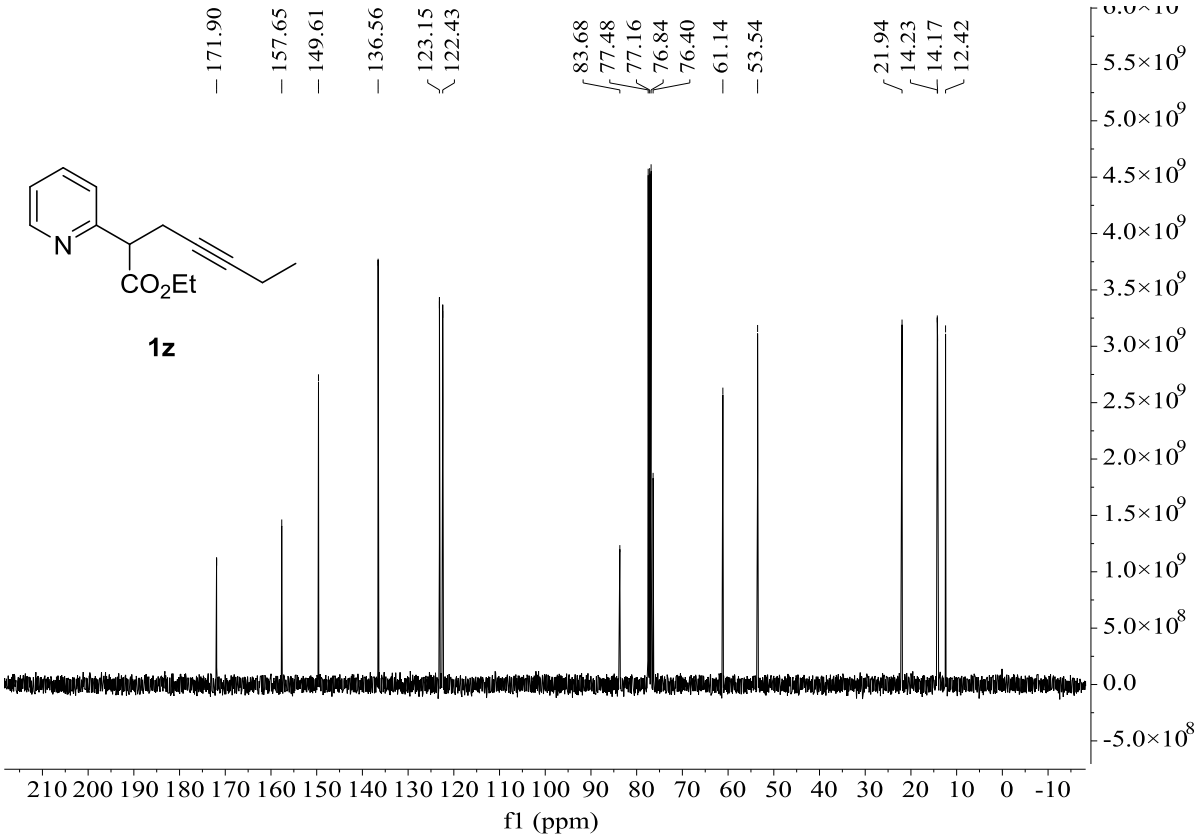
^{13}C NMR Spectrum of 1x at 25 °C ($CDCl_3$, 100 MHz)



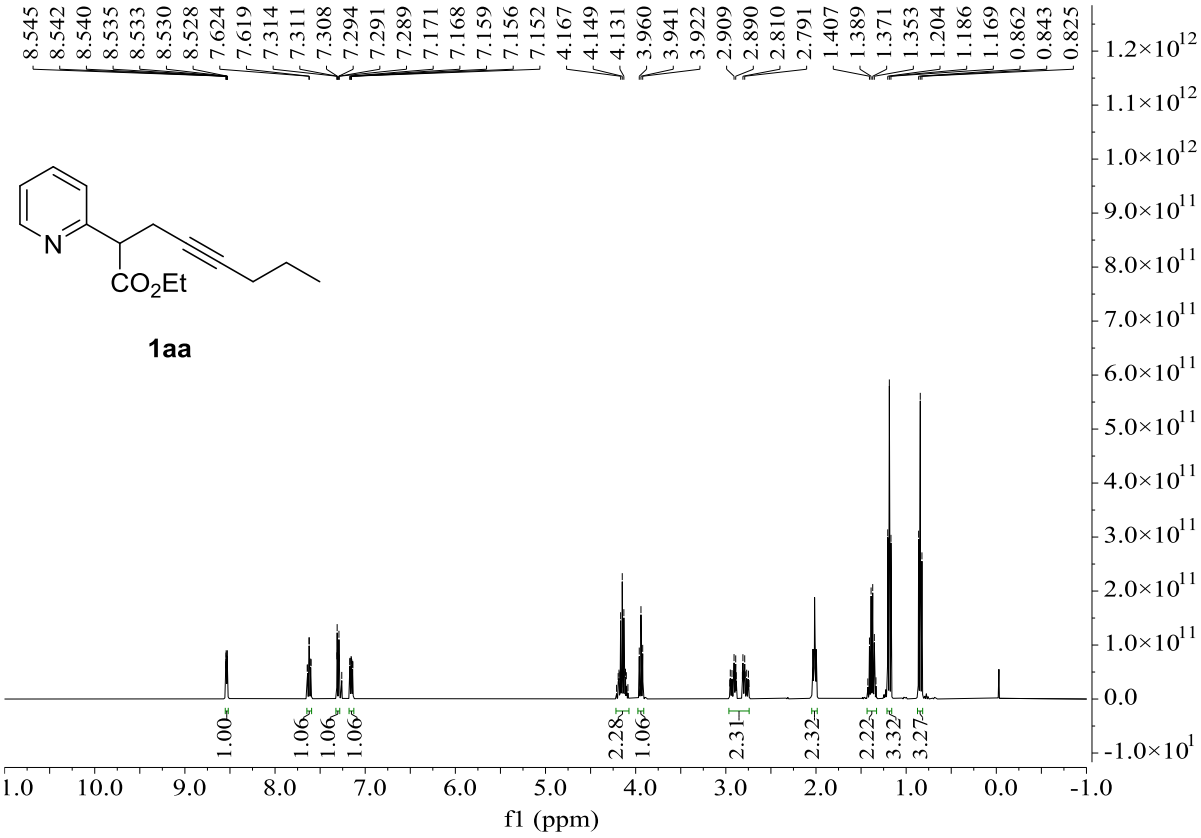
^{1}H NMR Spectrum of 1z at 25 °C ($CDCl_3$, 400 MHz)



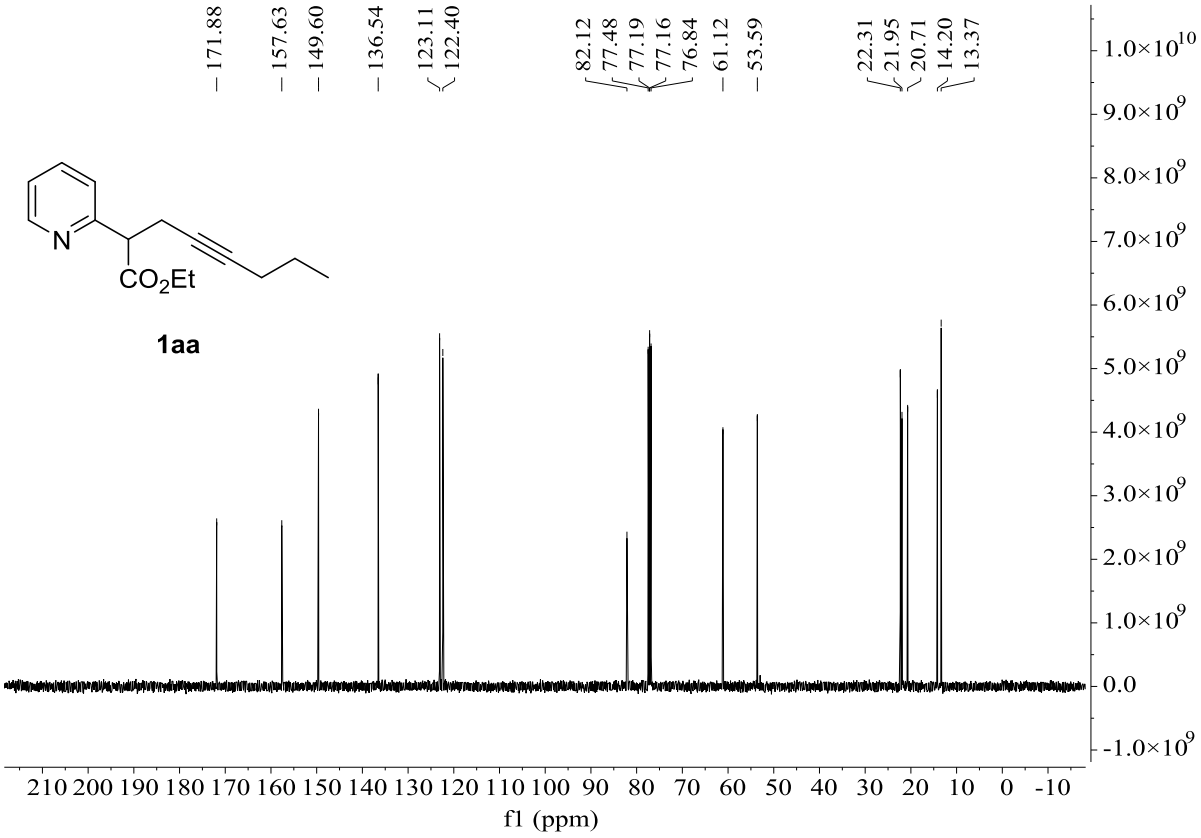
^{13}C NMR Spectrum of 1z at 25 °C ($CDCl_3$, 100 MHz)



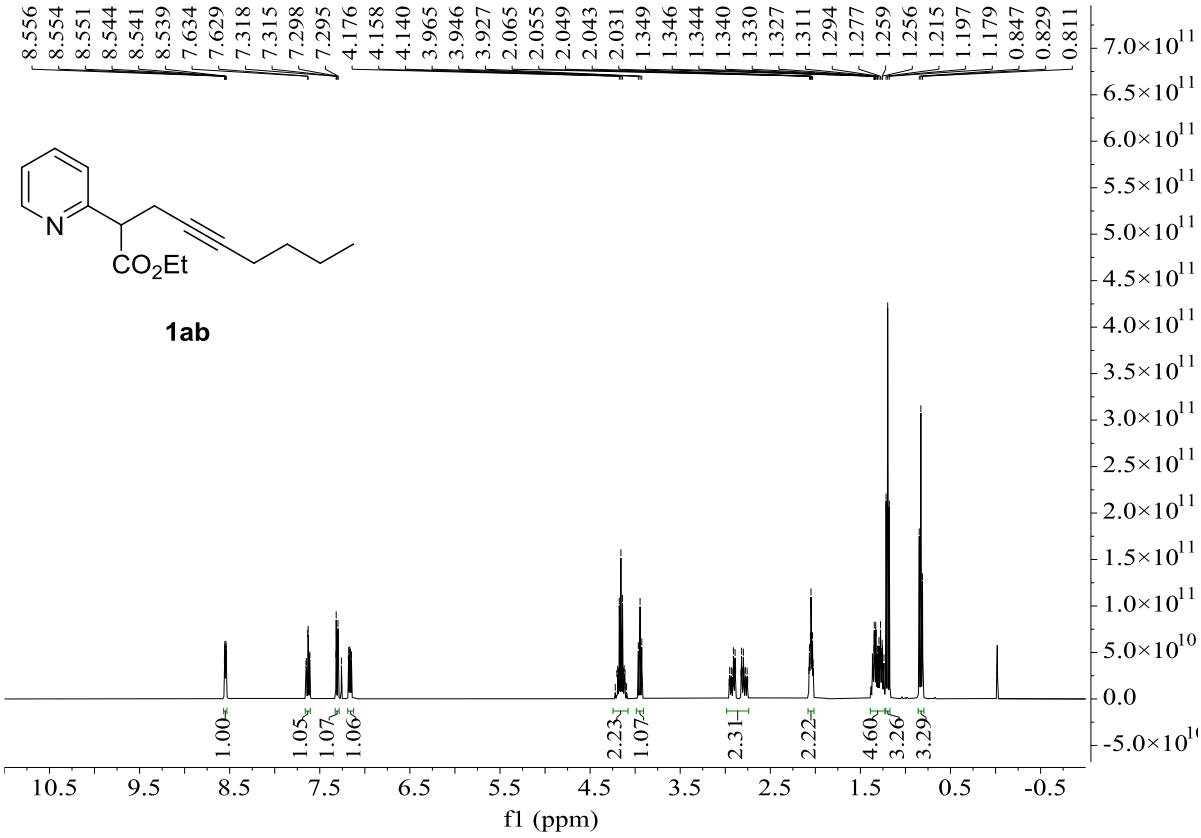
^{1}H NMR Spectrum of 1aa at 25 °C ($CDCl_3$, 400 MHz)



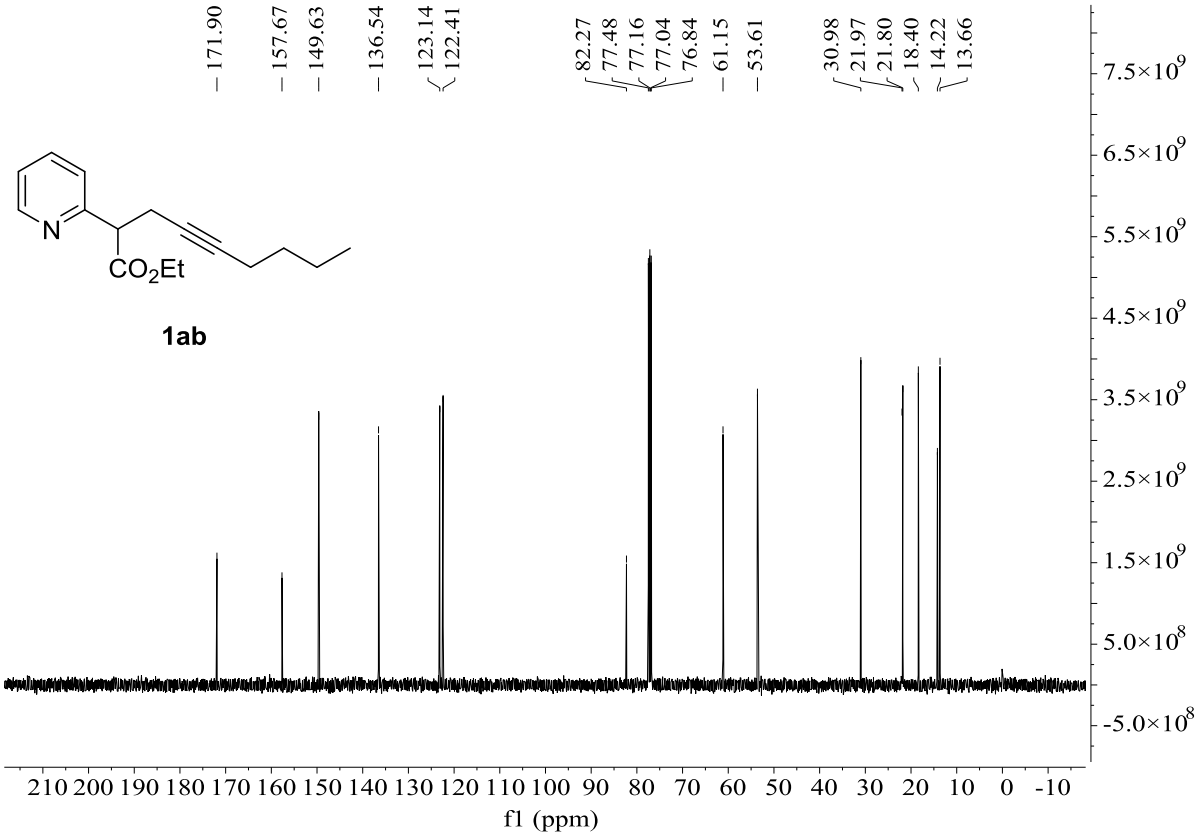
^{13}C NMR Spectrum of 1aa at 25 °C ($CDCl_3$, 100 MHz)



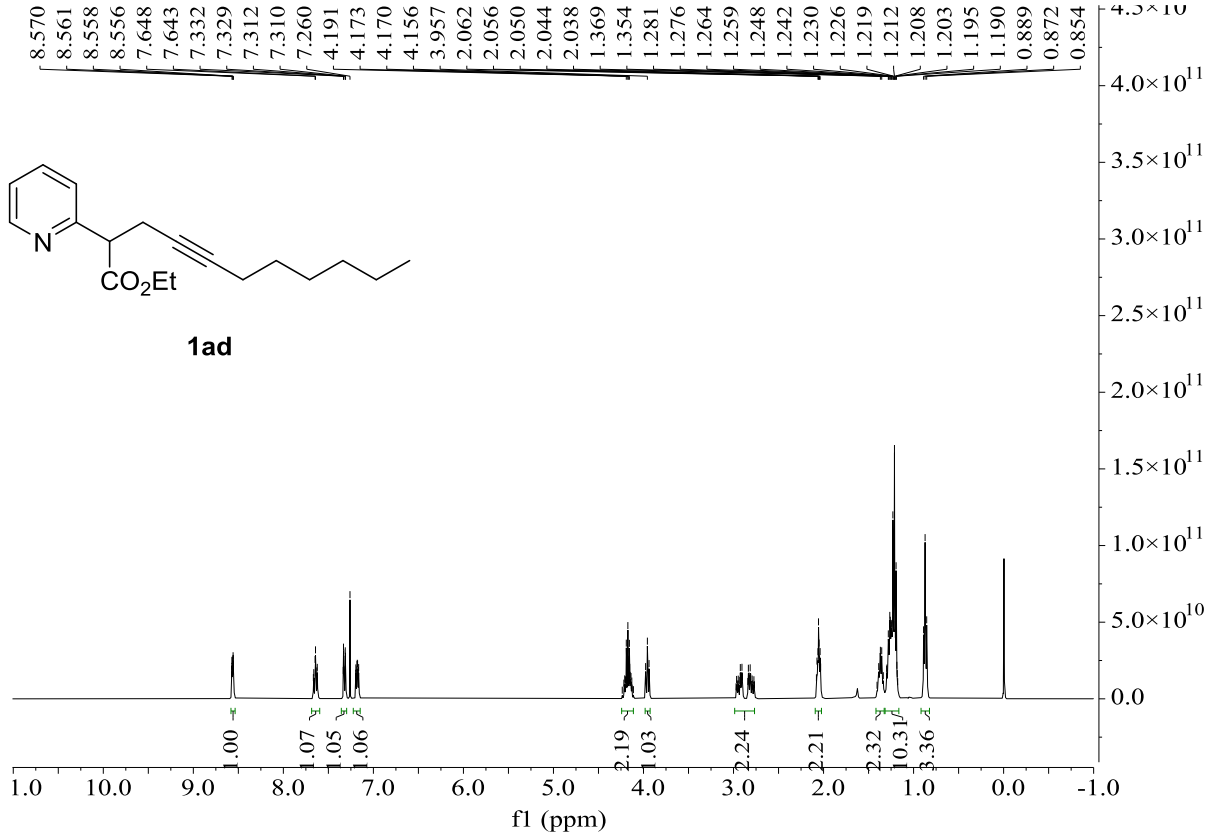
^{1}H NMR Spectrum of 1ab at 25 °C ($CDCl_3$, 400 MHz)



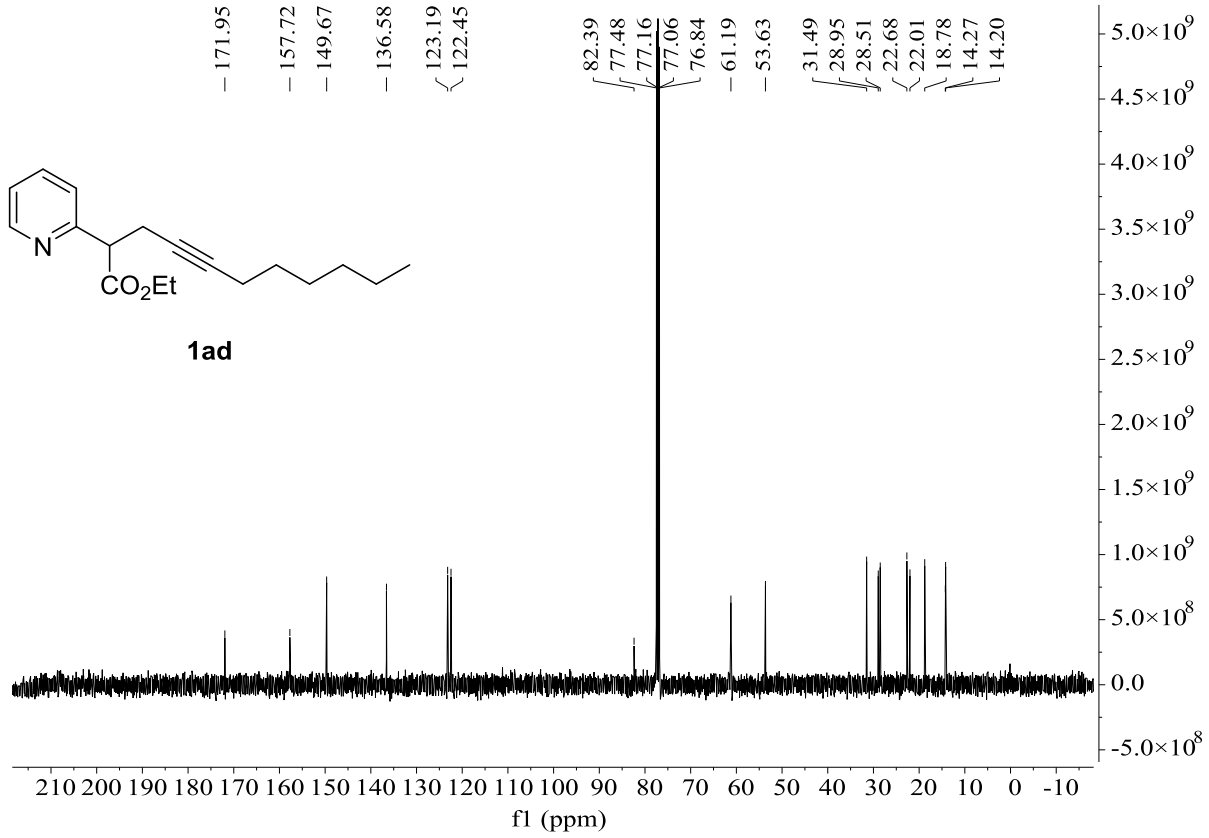
^{13}C NMR Spectrum of 1ab at 25 °C ($CDCl_3$, 100 MHz)



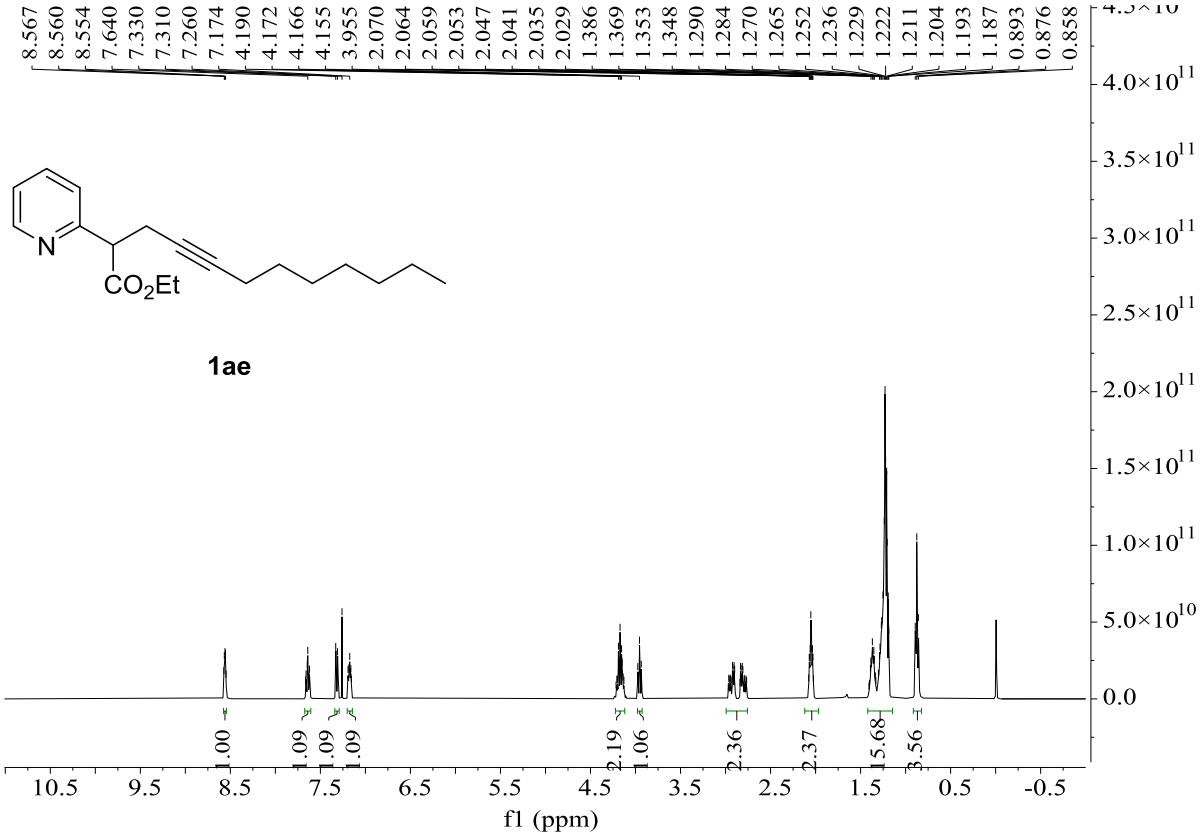
^{1}H NMR Spectrum of 1ad at 25 °C ($CDCl_3$, 400 MHz)



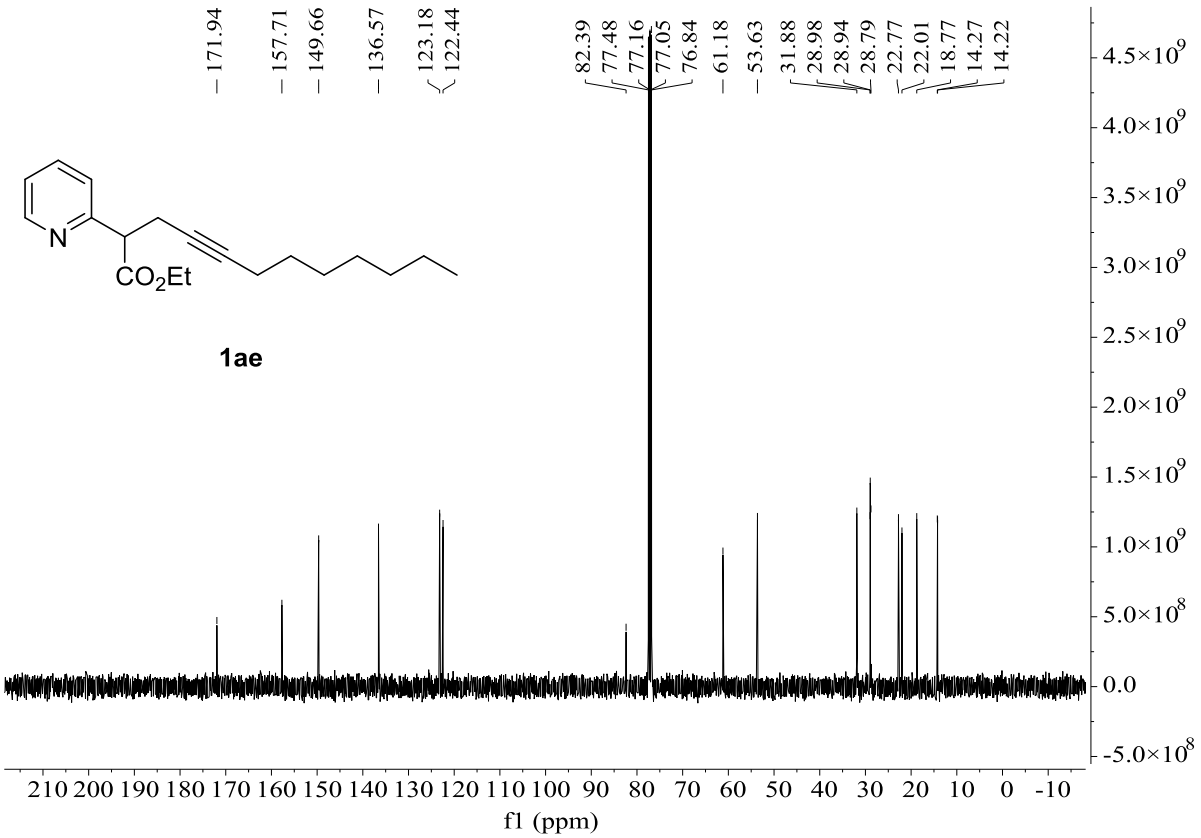
^{13}C NMR Spectrum of 1ad at 25 °C ($CDCl_3$, 100 MHz)



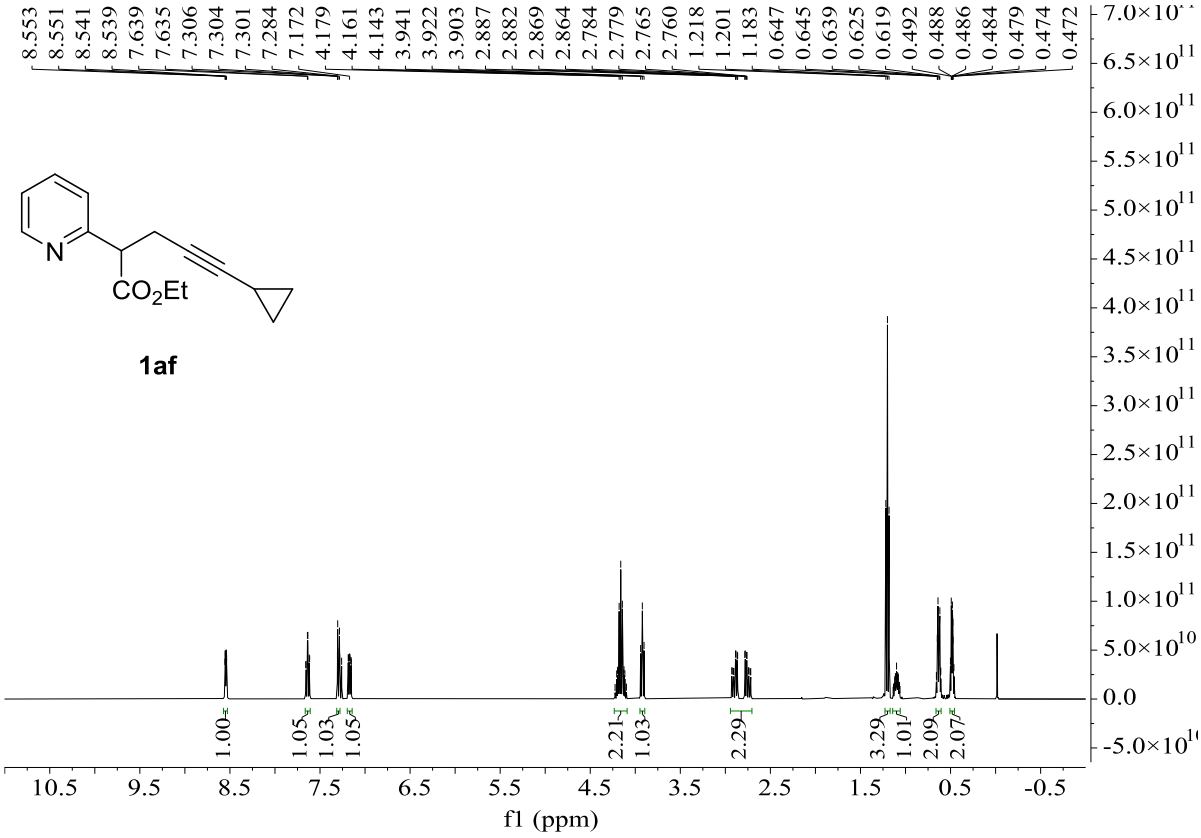
^{1}H NMR Spectrum of 1ae at 25 °C ($CDCl_3$, 400 MHz)



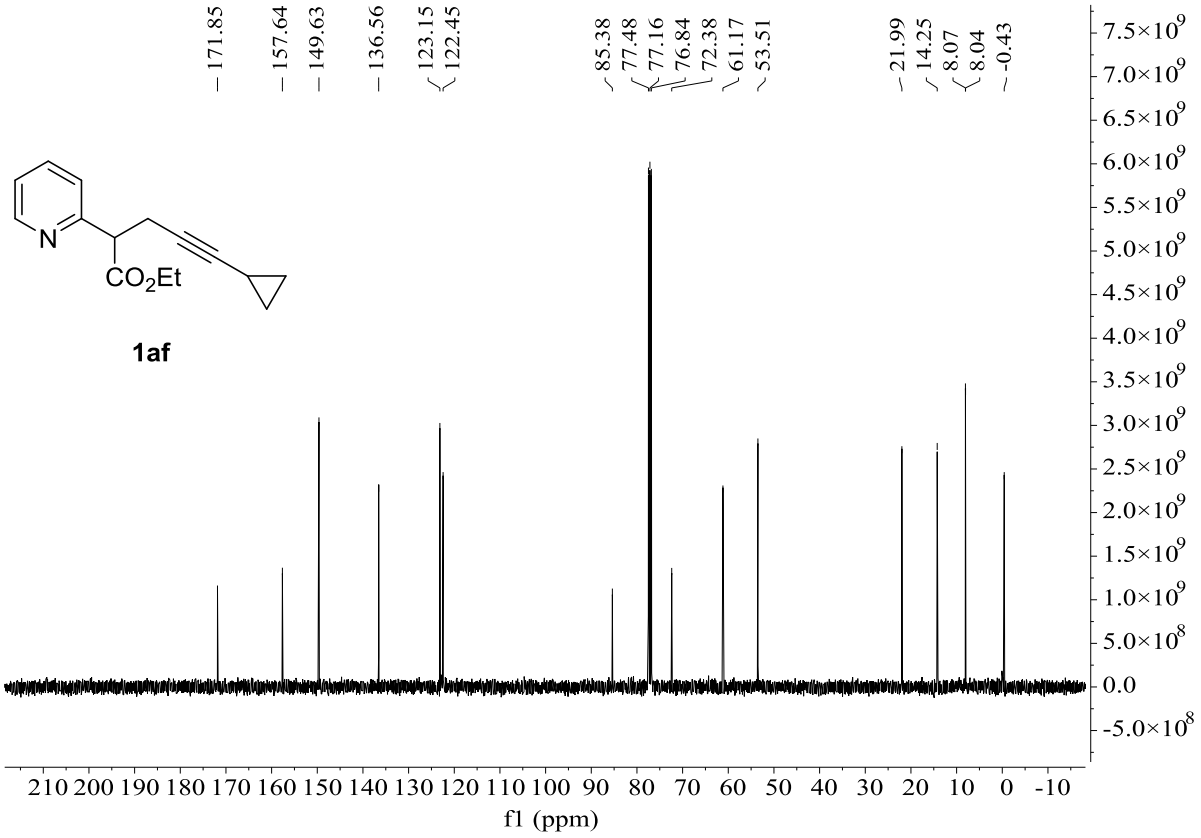
^{13}C NMR Spectrum of 1ae at 25 °C ($CDCl_3$, 100 MHz)



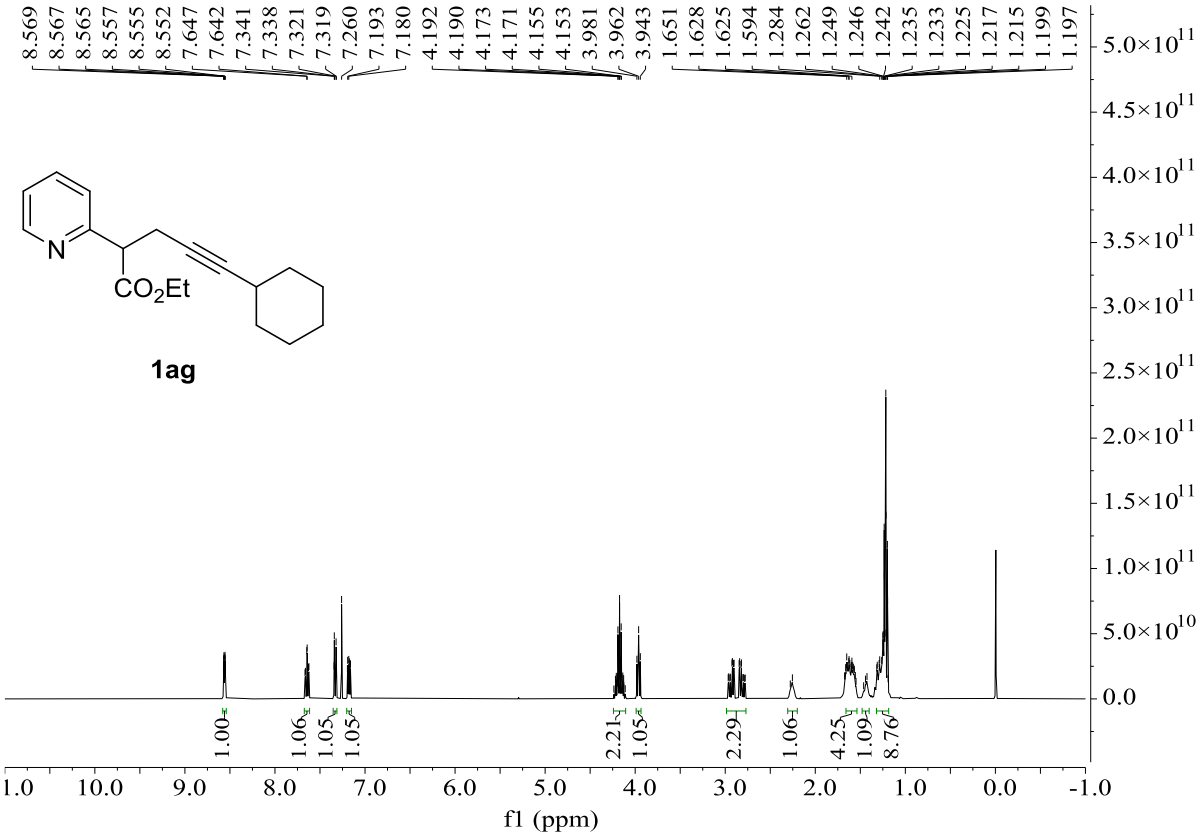
^{1}H NMR Spectrum of 1af at 25 °C ($CDCl_3$, 400 MHz)



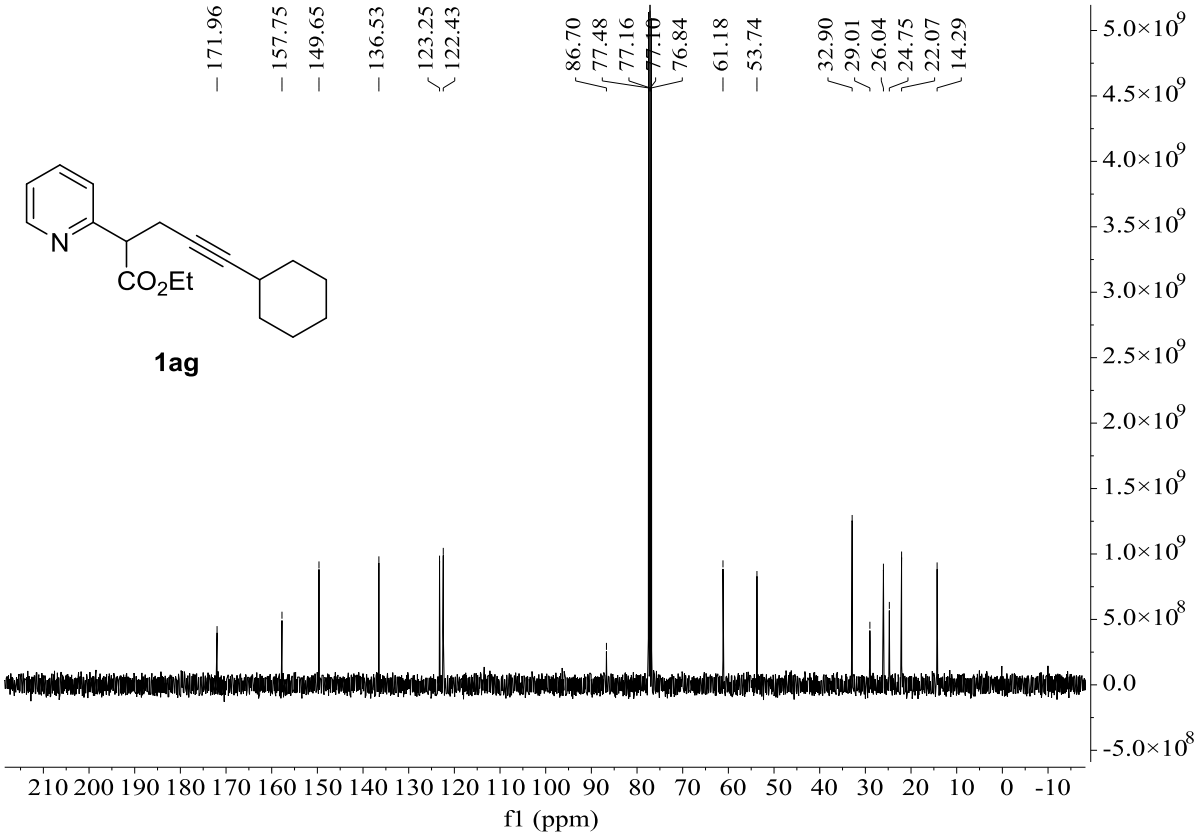
^{13}C NMR Spectrum of 1af at 25 °C ($CDCl_3$, 100 MHz)



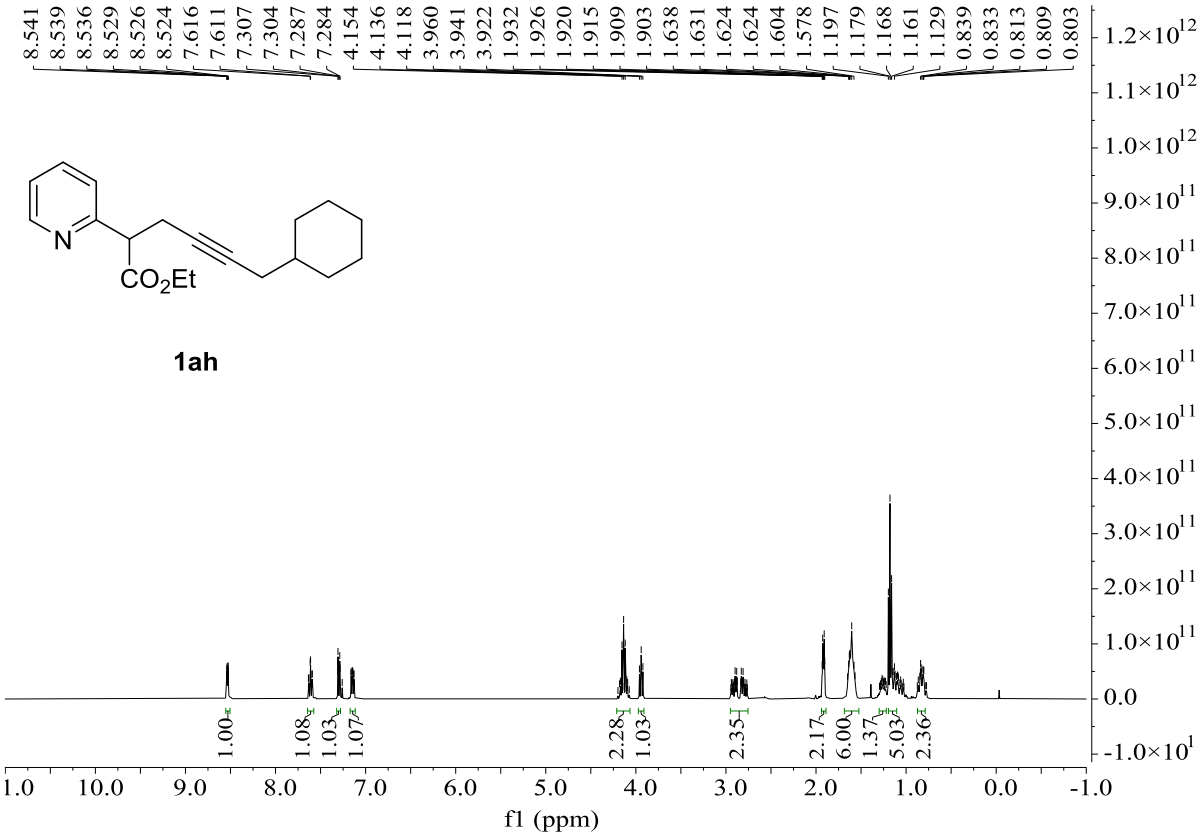
^{1}H NMR Spectrum of 1ag at 25 °C ($CDCl_3$, 400 MHz)



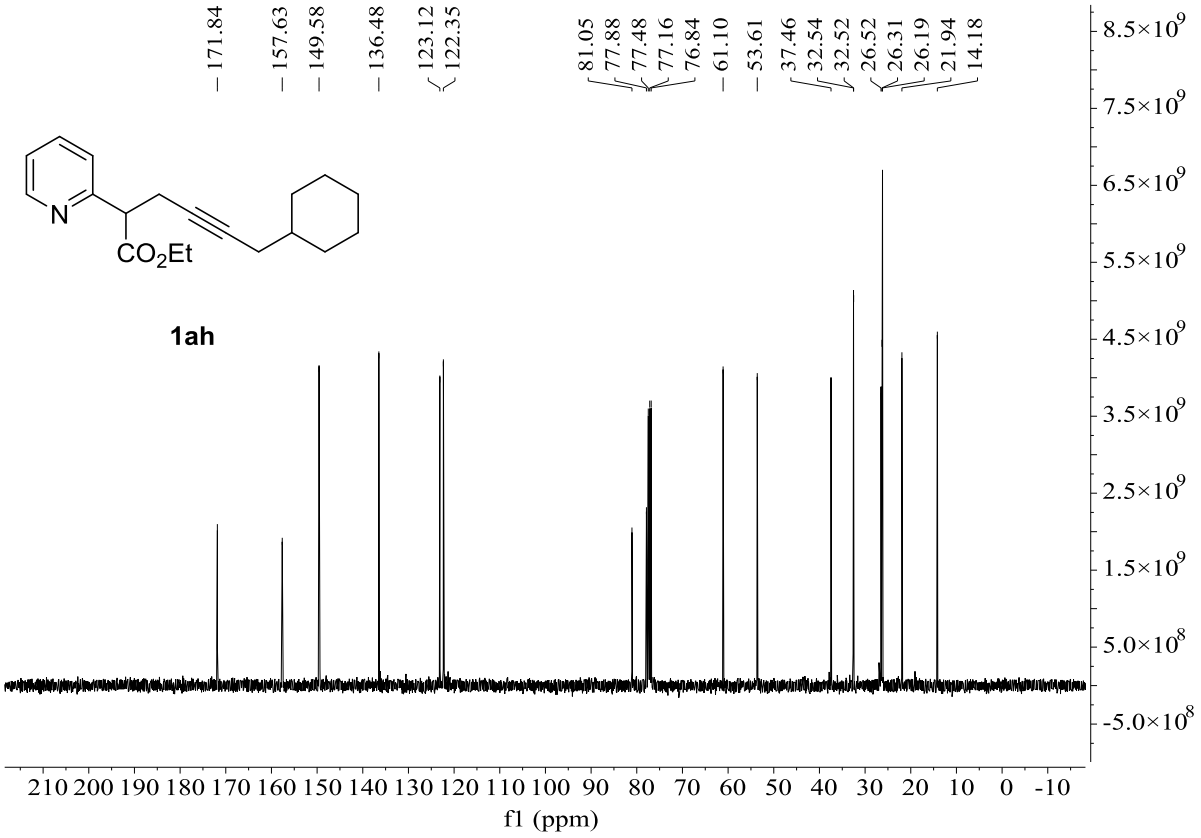
^{13}C NMR Spectrum of 1ag at 25 °C ($CDCl_3$, 100 MHz)



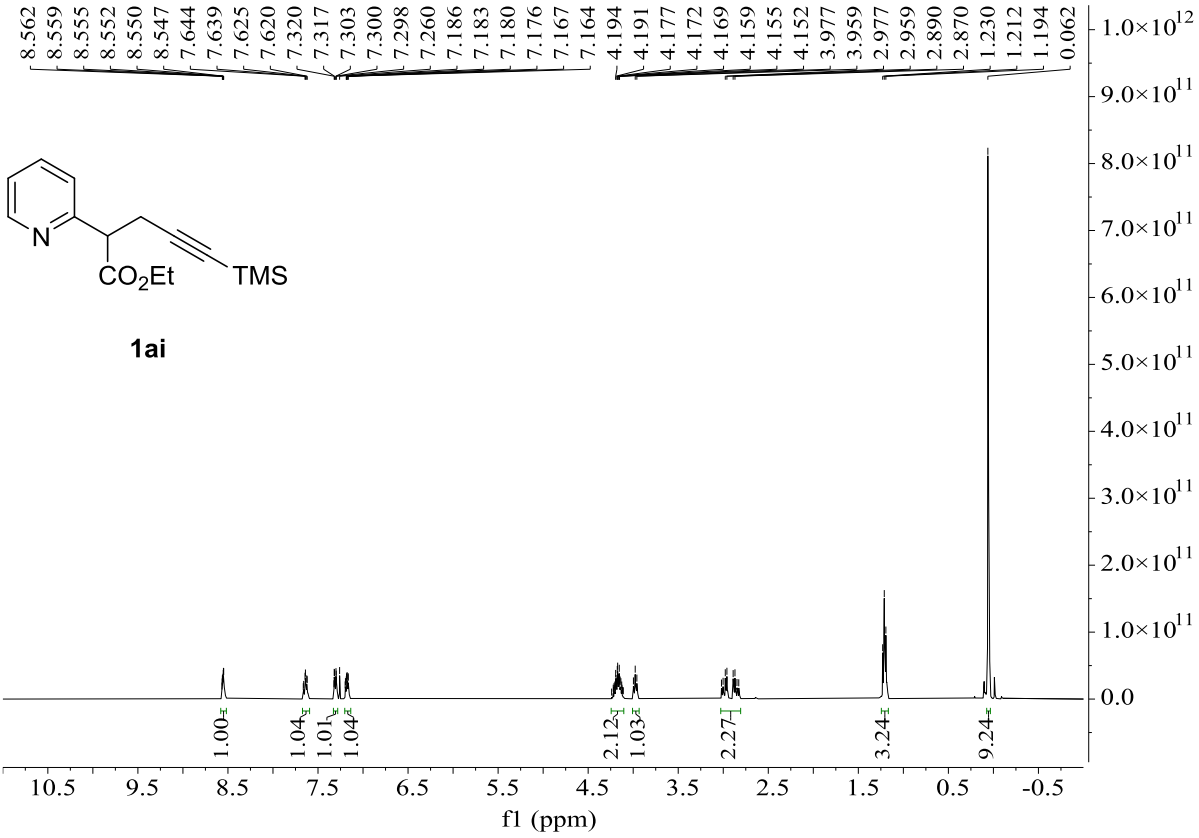
^{1}H NMR Spectrum of 1ah at 25 °C ($CDCl_3$, 400 MHz)



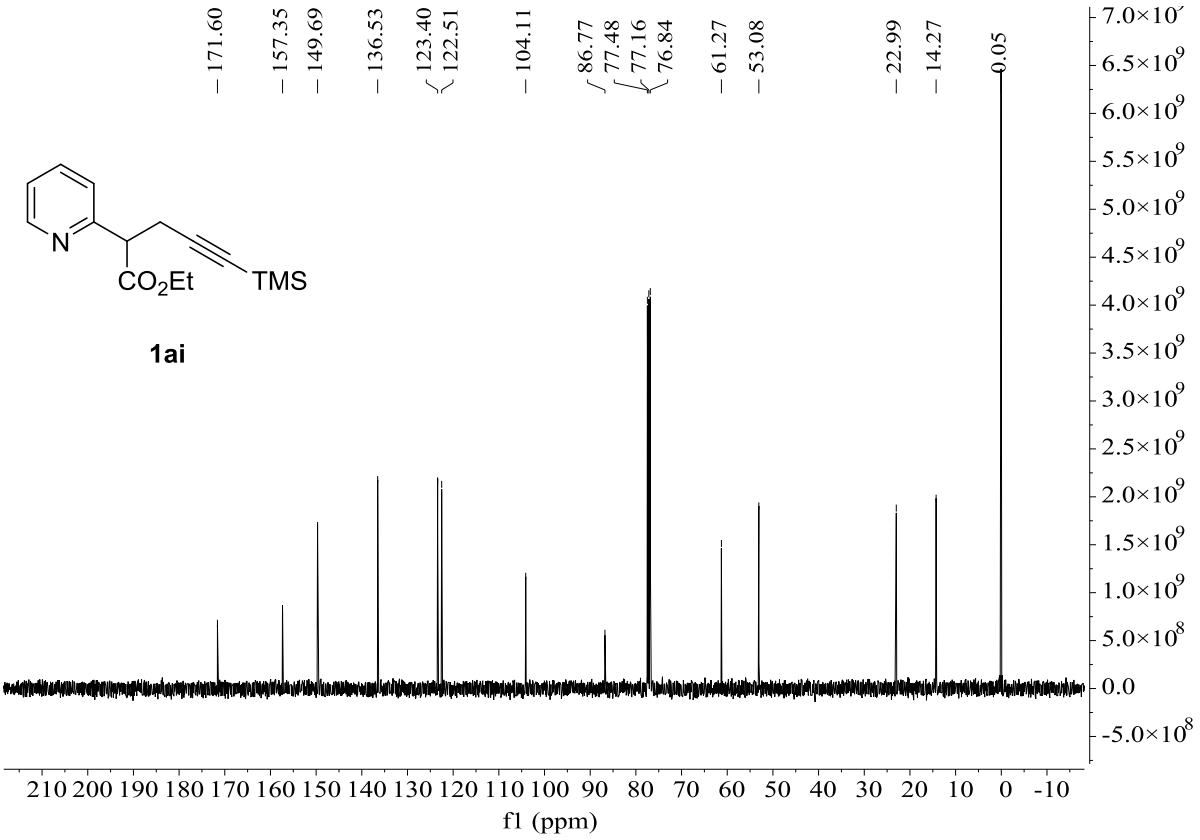
^{13}C NMR Spectrum of 1ah at 25 °C ($CDCl_3$, 400 MHz)



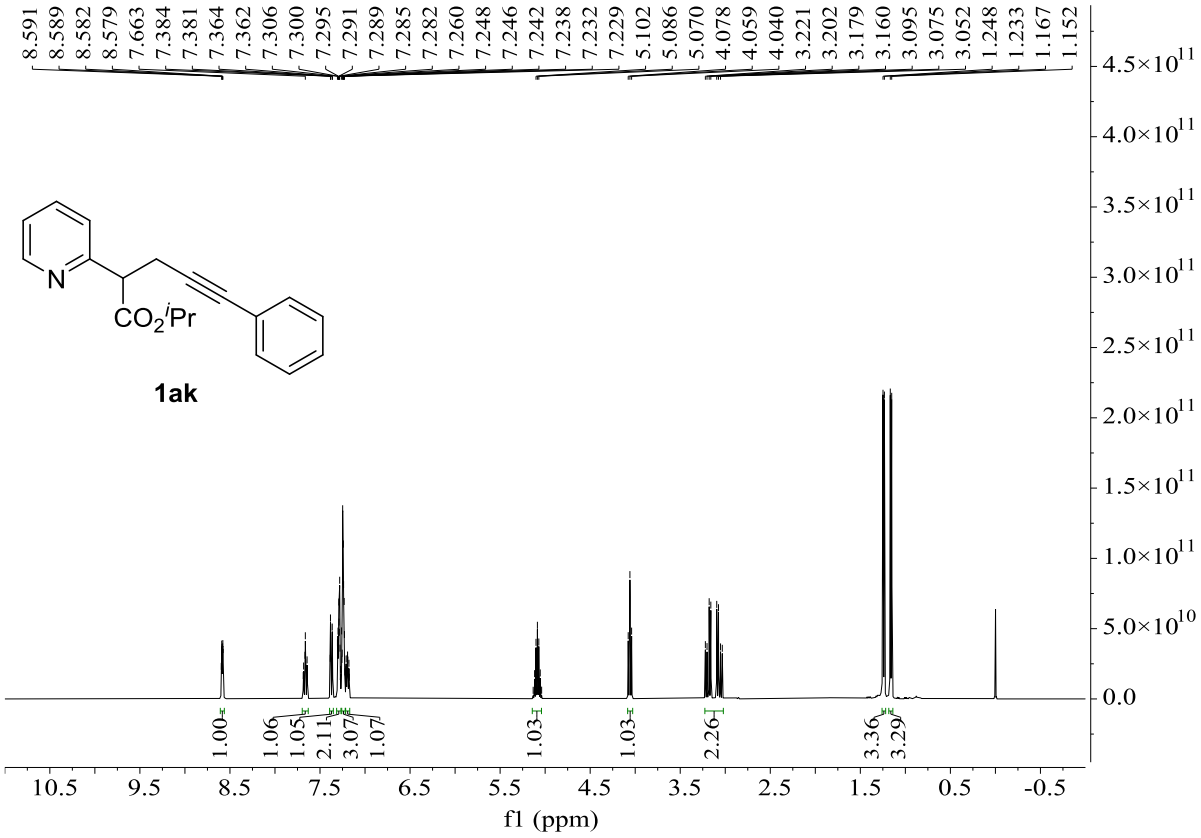
^{1}H NMR Spectrum of 1ai at 25 °C ($CDCl_3$, 400 MHz)



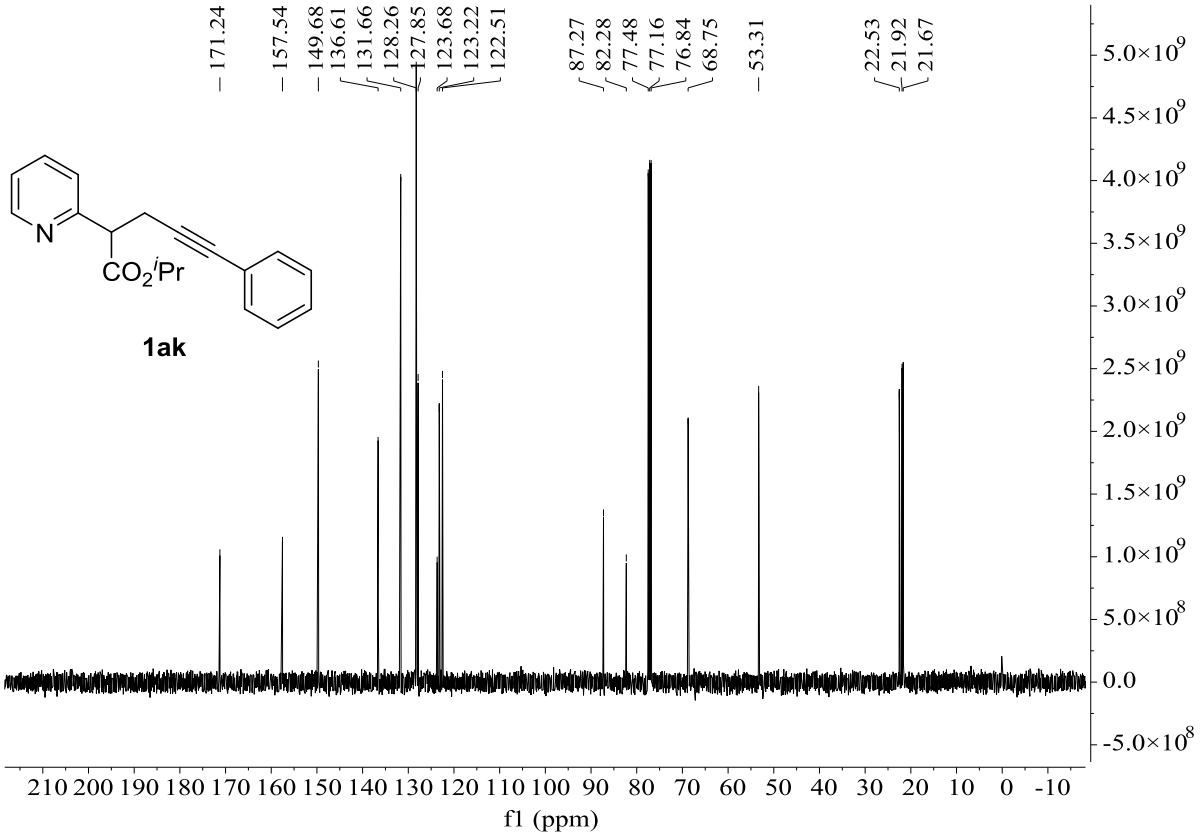
^{13}C NMR Spectrum of 1ai at 25 °C ($CDCl_3$, 100 MHz)



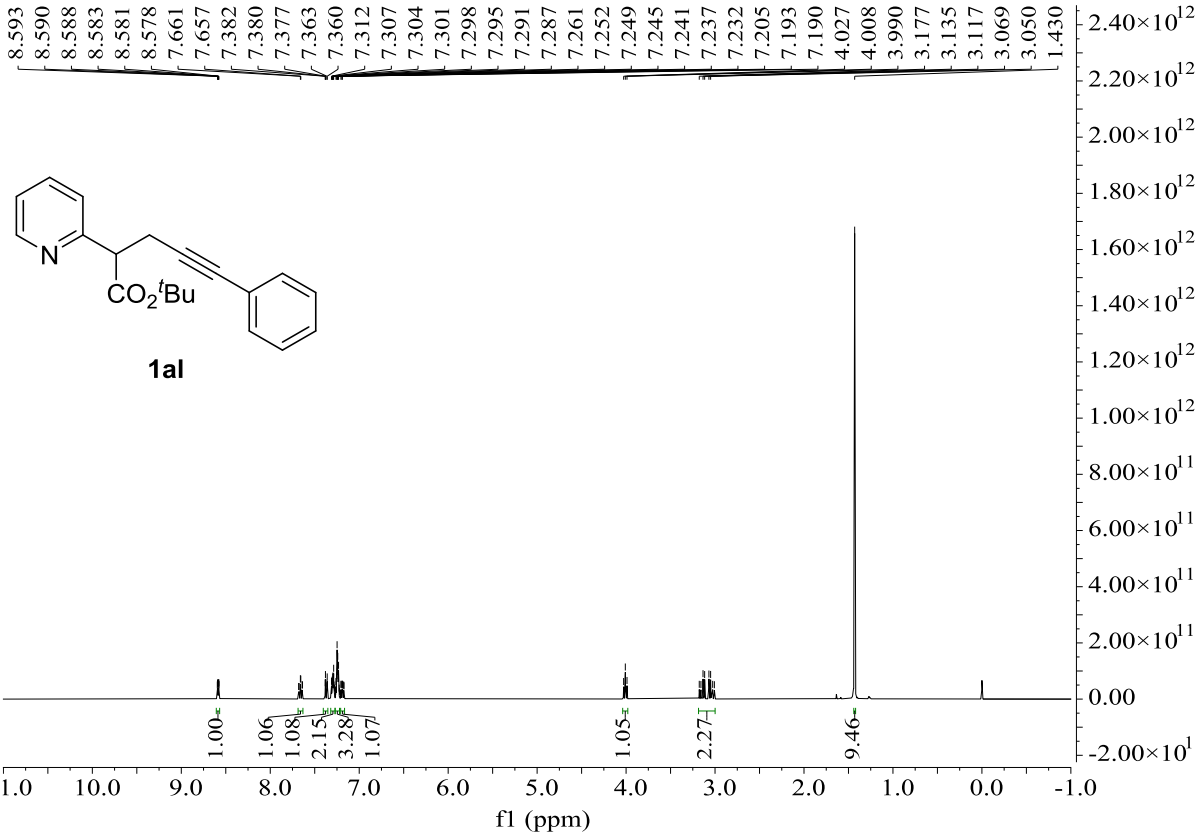
^{1}H NMR Spectrum of 1ak at 25 °C ($CDCl_3$, 400 MHz)



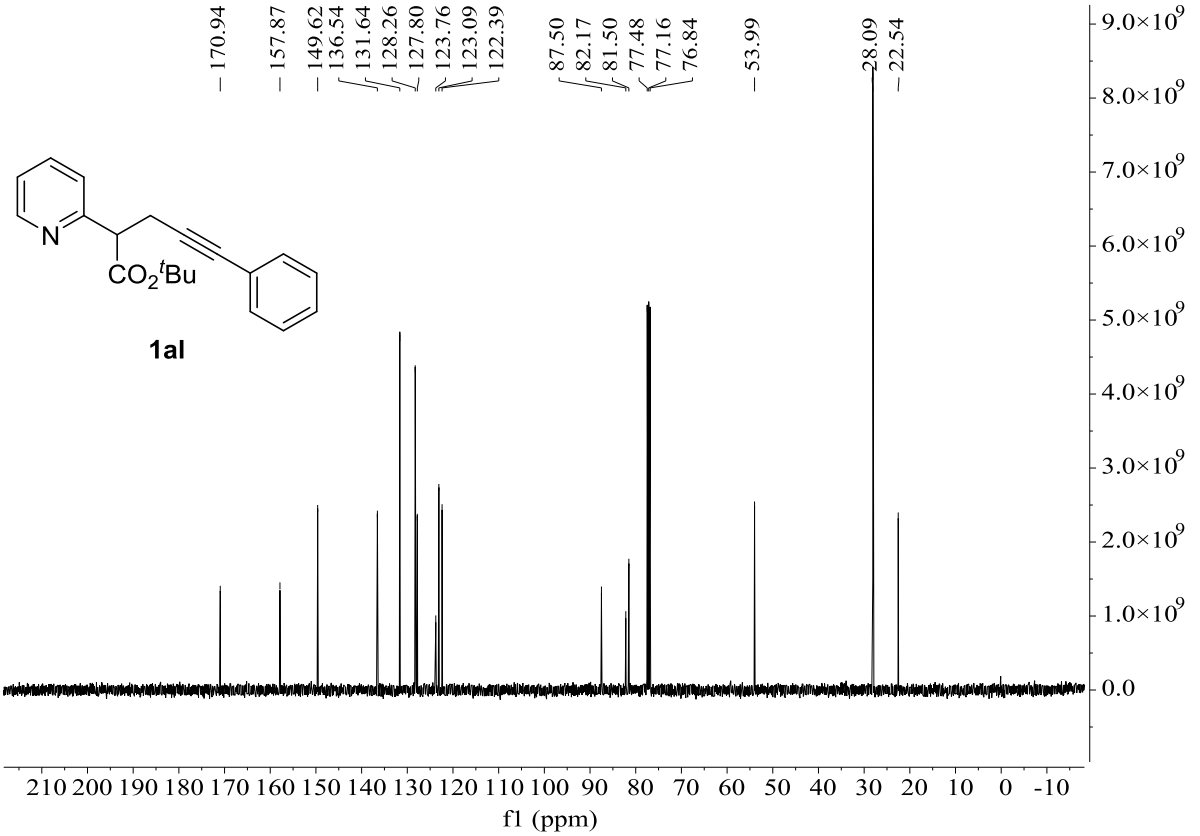
^{13}C NMR Spectrum of 1ak at 25 °C ($CDCl_3$, 100 MHz)



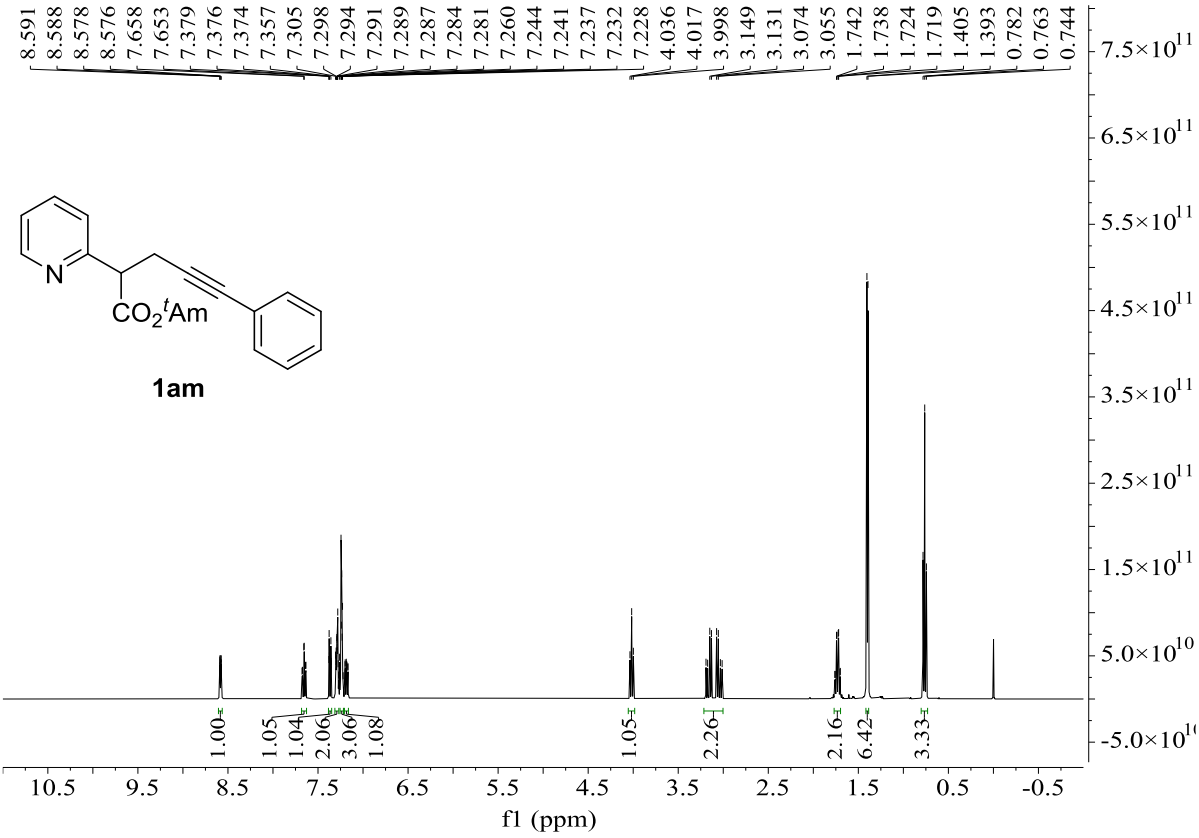
^{1}H NMR Spectrum of 1al at 25 °C ($CDCl_3$, 400 MHz)



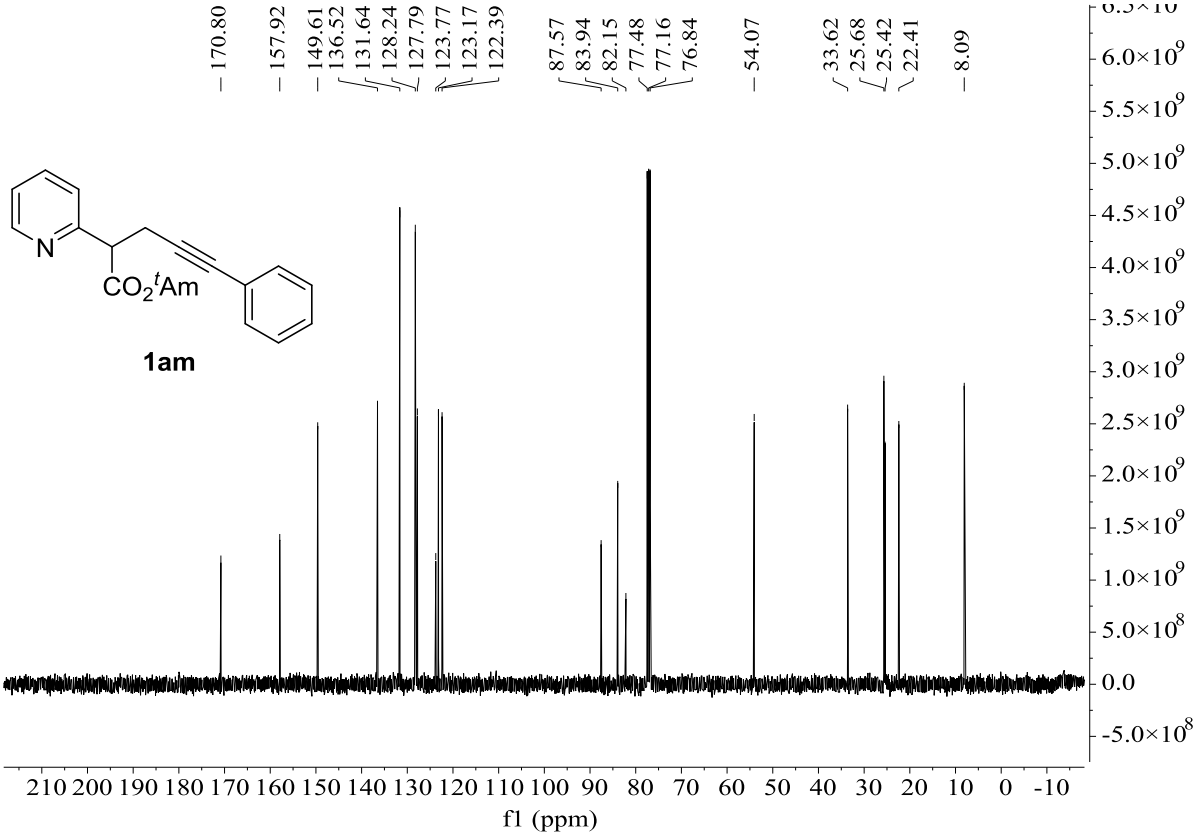
^{13}C NMR Spectrum of 1al at 25 °C ($CDCl_3$, 100 MHz)



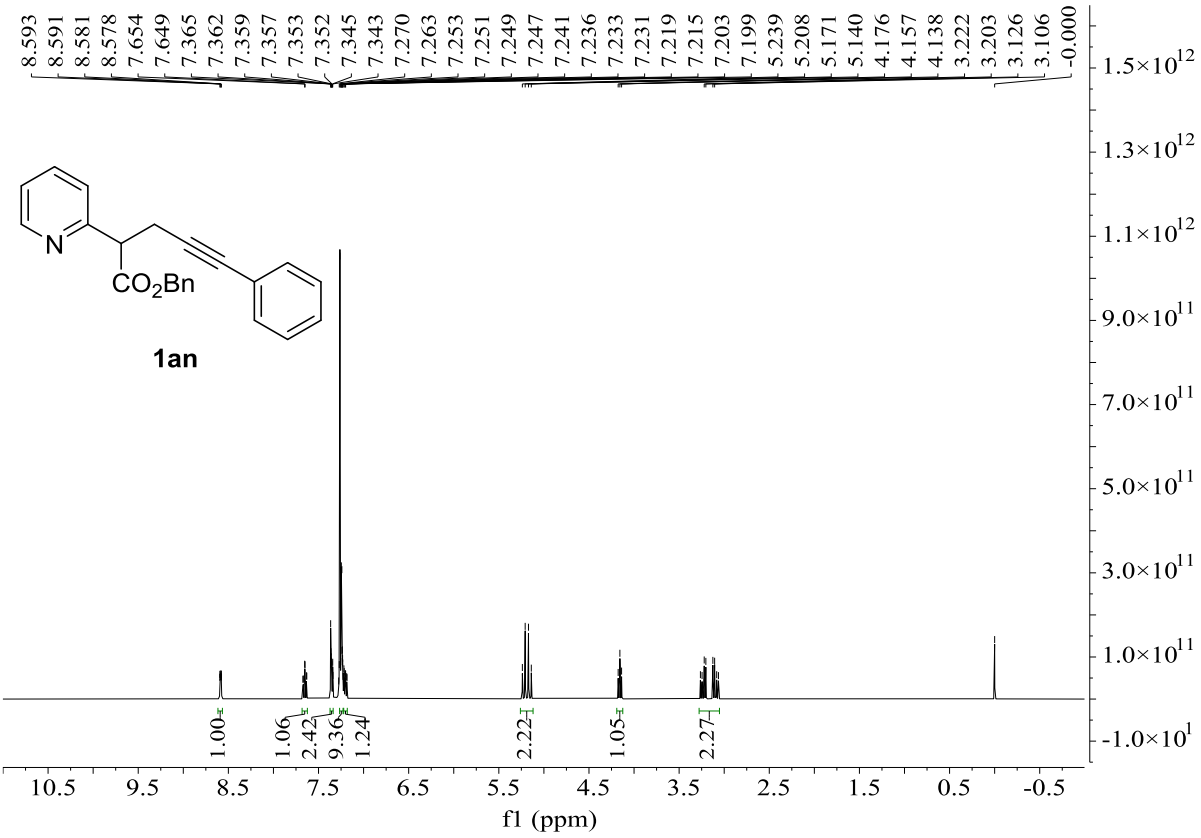
^{1}H NMR Spectrum of 1am at 25 °C ($CDCl_3$, 400 MHz)



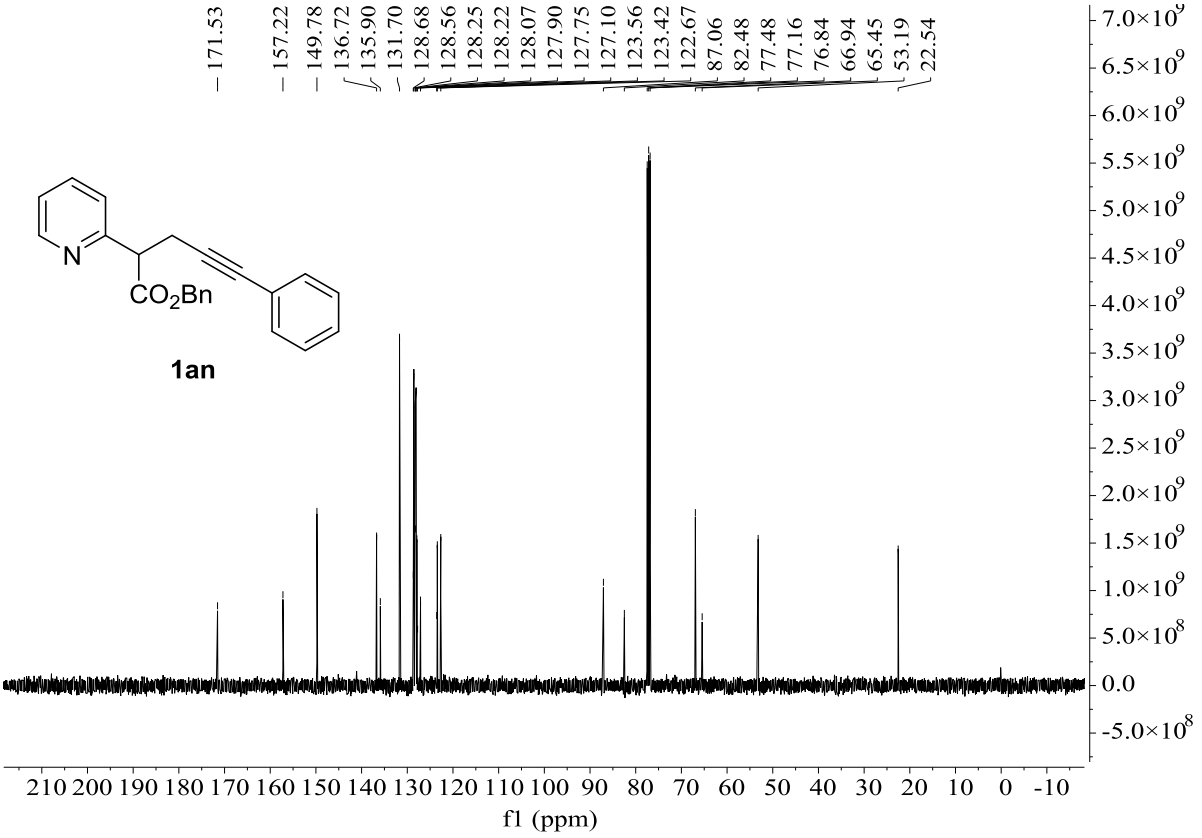
^{13}C NMR Spectrum of 1am at 25 °C ($CDCl_3$, 100 MHz)



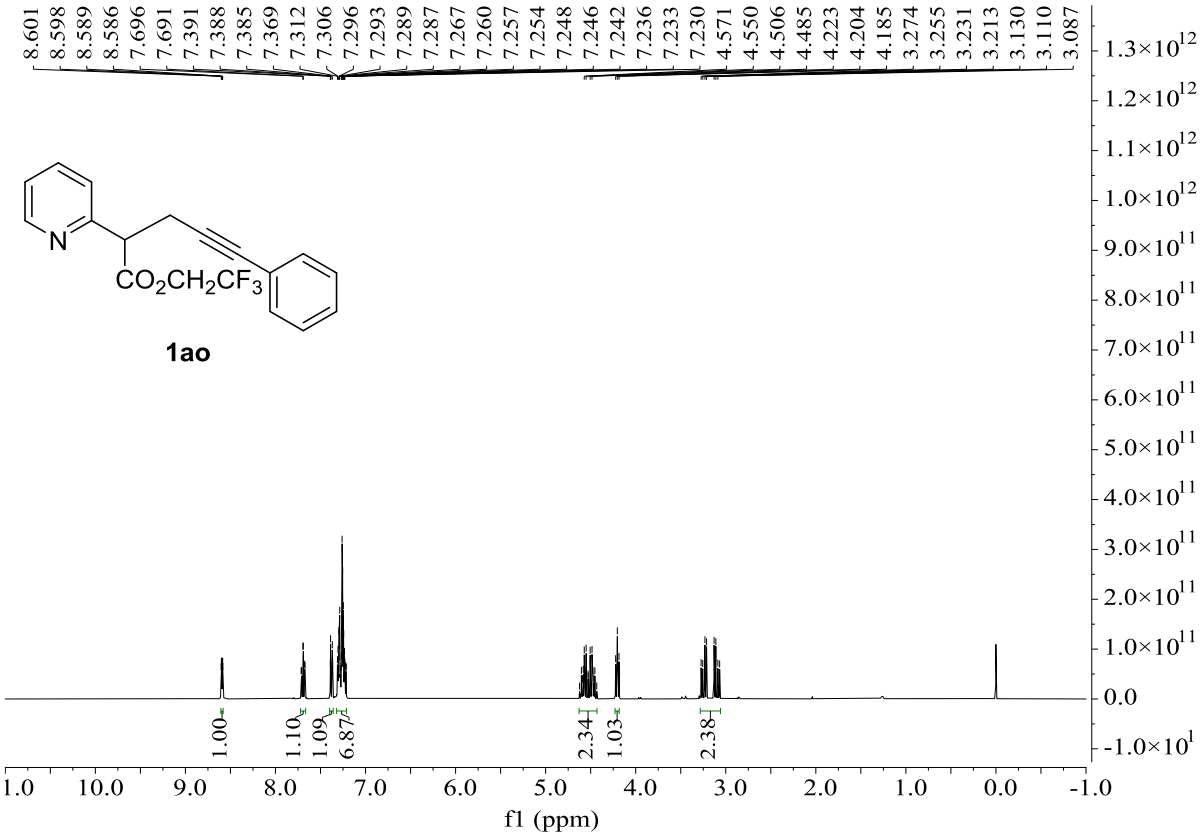
^{1}H NMR Spectrum of 1an at 25 °C ($CDCl_3$, 400 MHz)



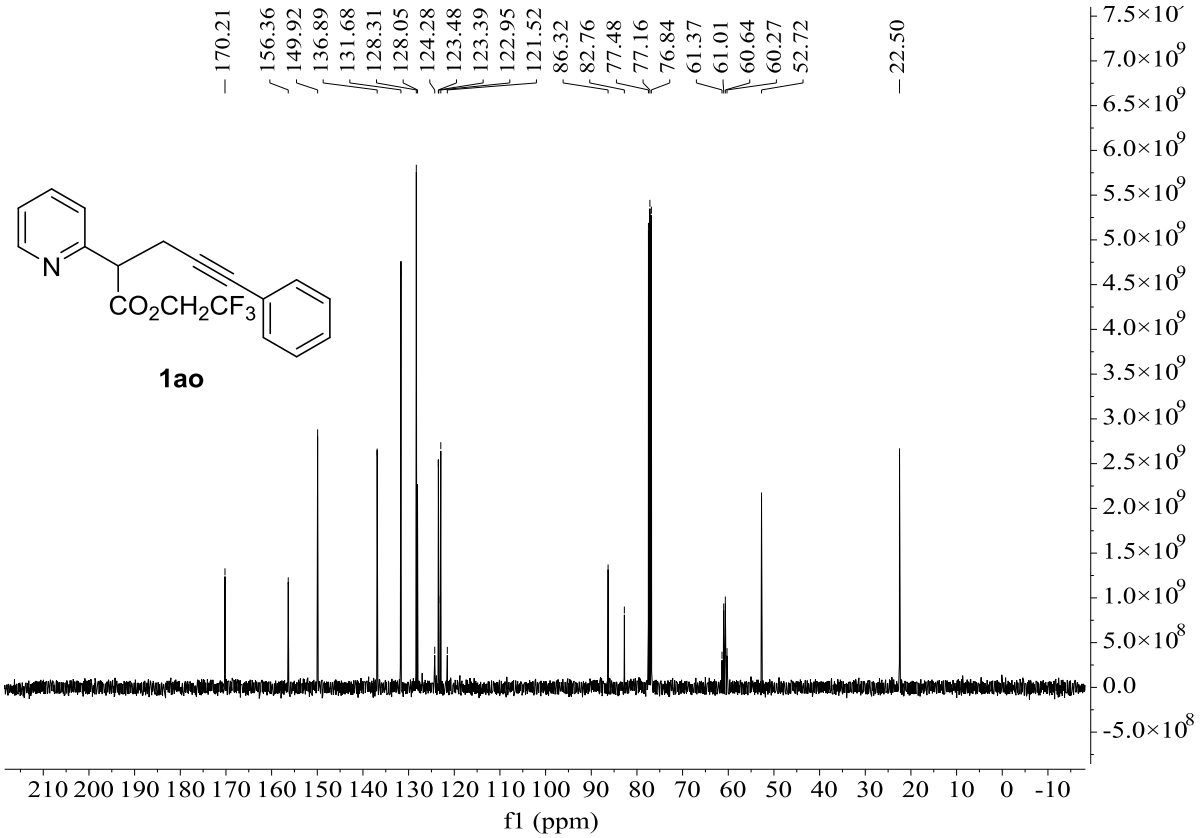
^{13}C NMR Spectrum of 1an at 25 °C ($CDCl_3$, 100 MHz)



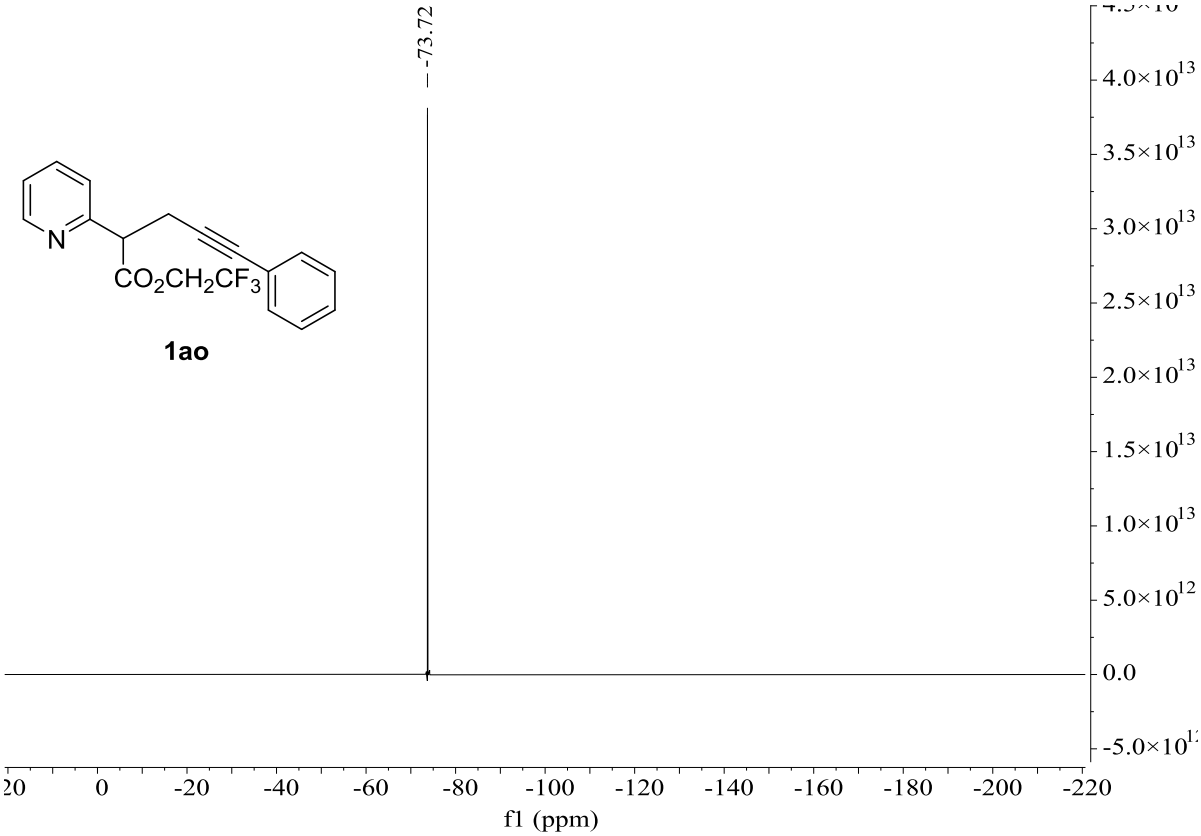
^{1}H NMR Spectrum of 1ao at 25 °C ($CDCl_3$, 400 MHz)



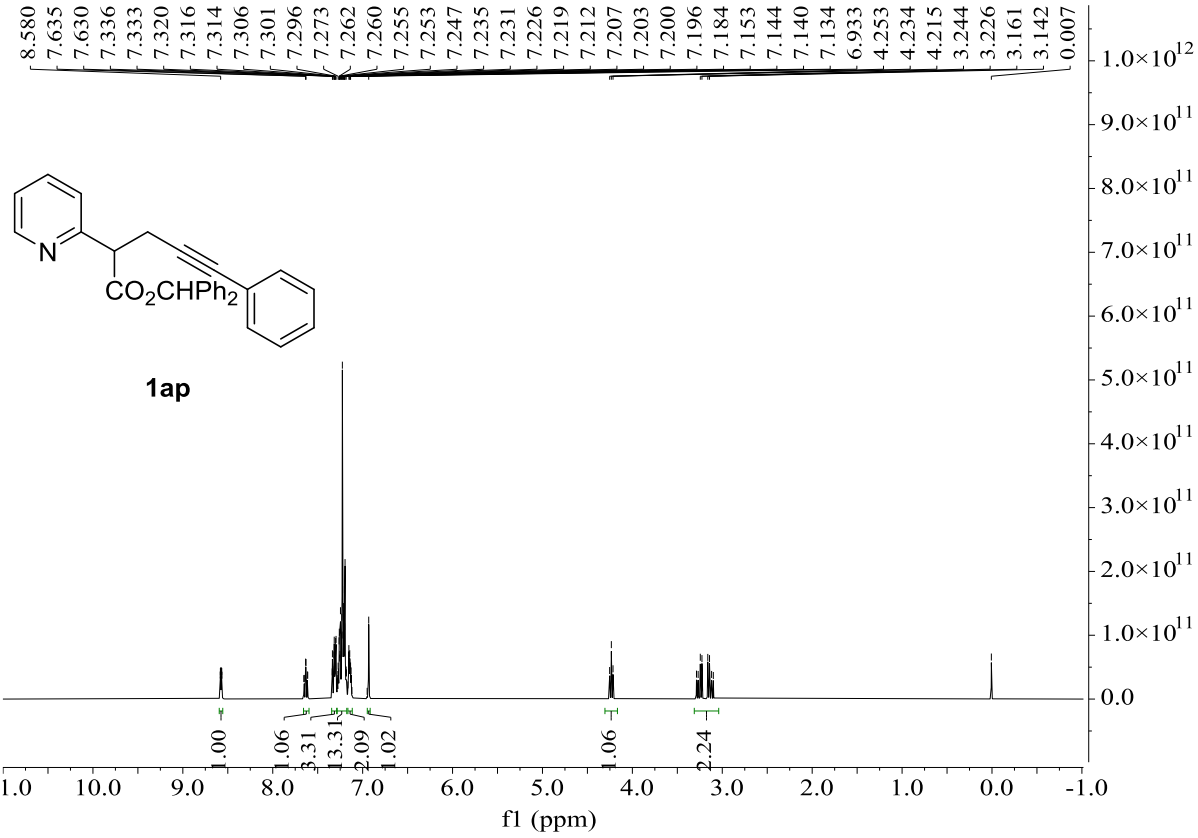
^{13}C NMR Spectrum of 1ao at 25 °C ($CDCl_3$, 100 MHz)



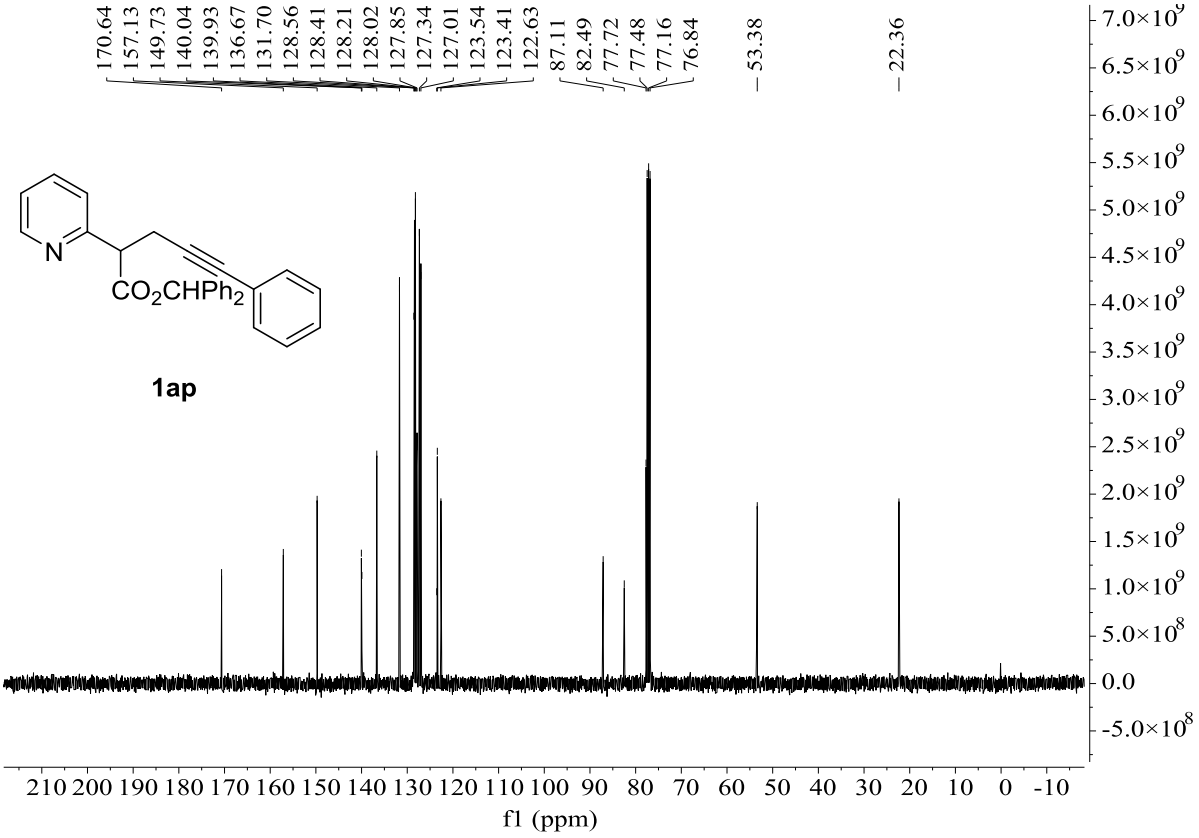
^{19}F NMR Spectrum of 1ao at 25 °C ($CDCl_3$, 376 MHz)



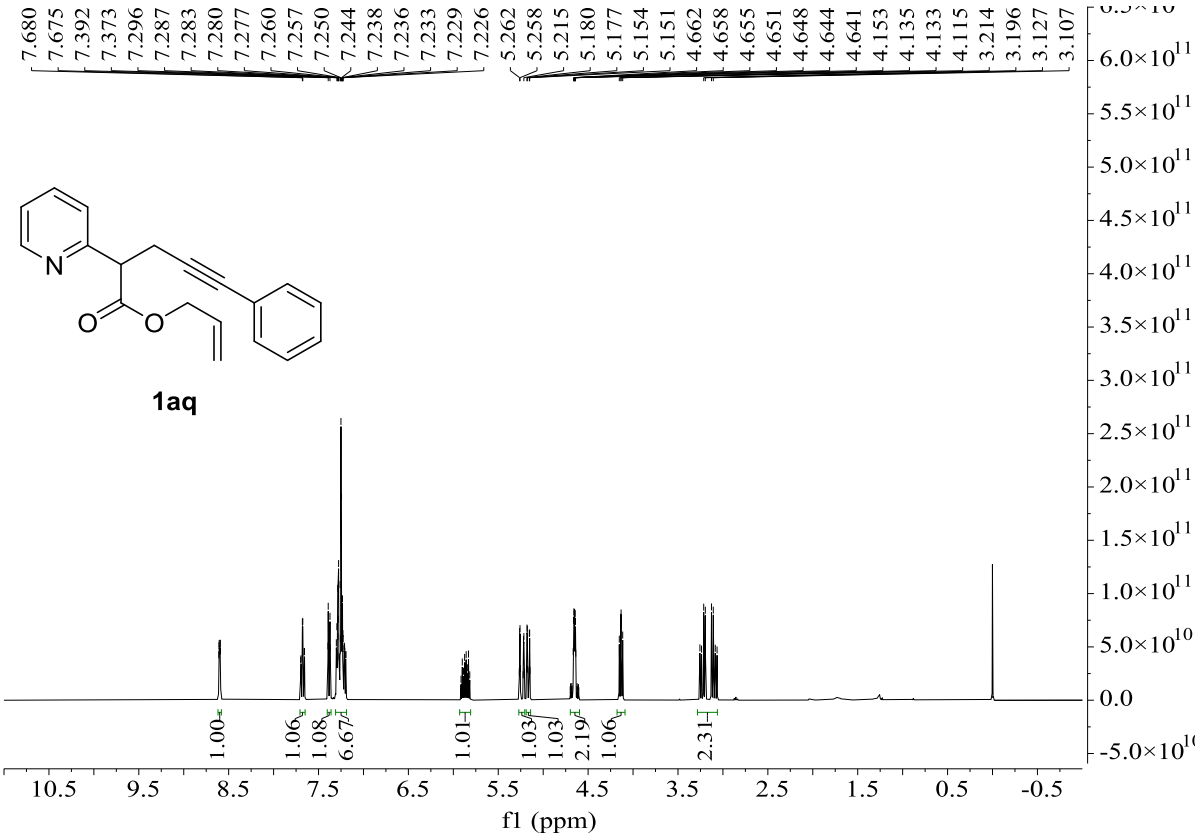
^{1}H NMR Spectrum of 1ap at 25 °C ($CDCl_3$, 400 MHz)



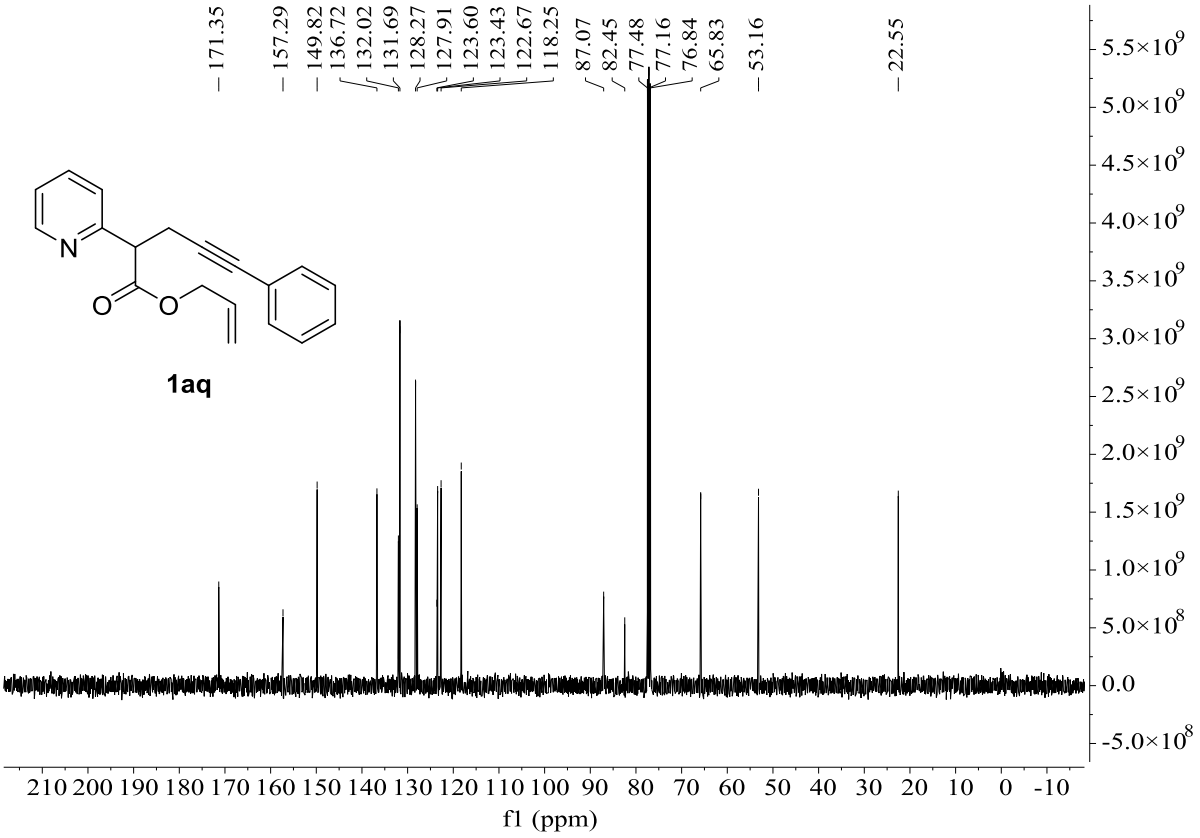
^{13}C NMR Spectrum of 1ap at 25 °C ($CDCl_3$, 100 MHz)



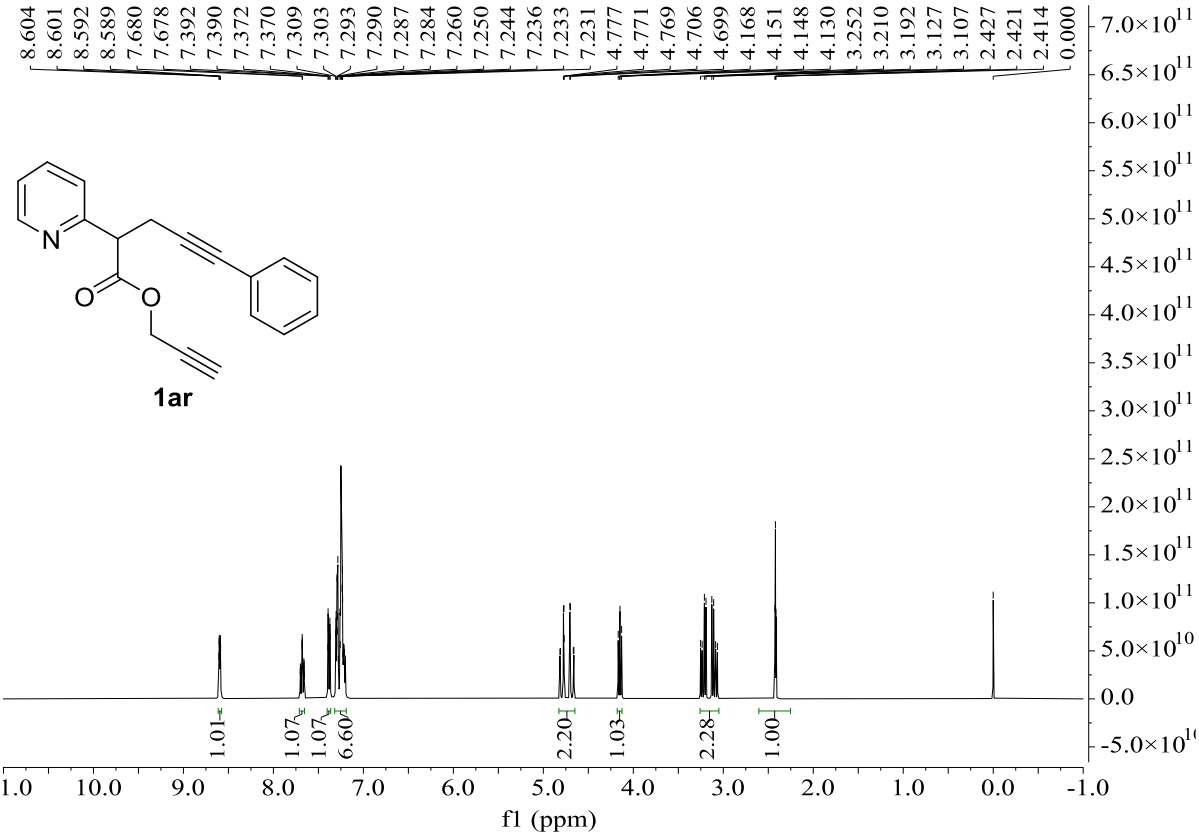
^{1}H NMR Spectrum of 1aq at 25 °C ($CDCl_3$, 400 MHz)



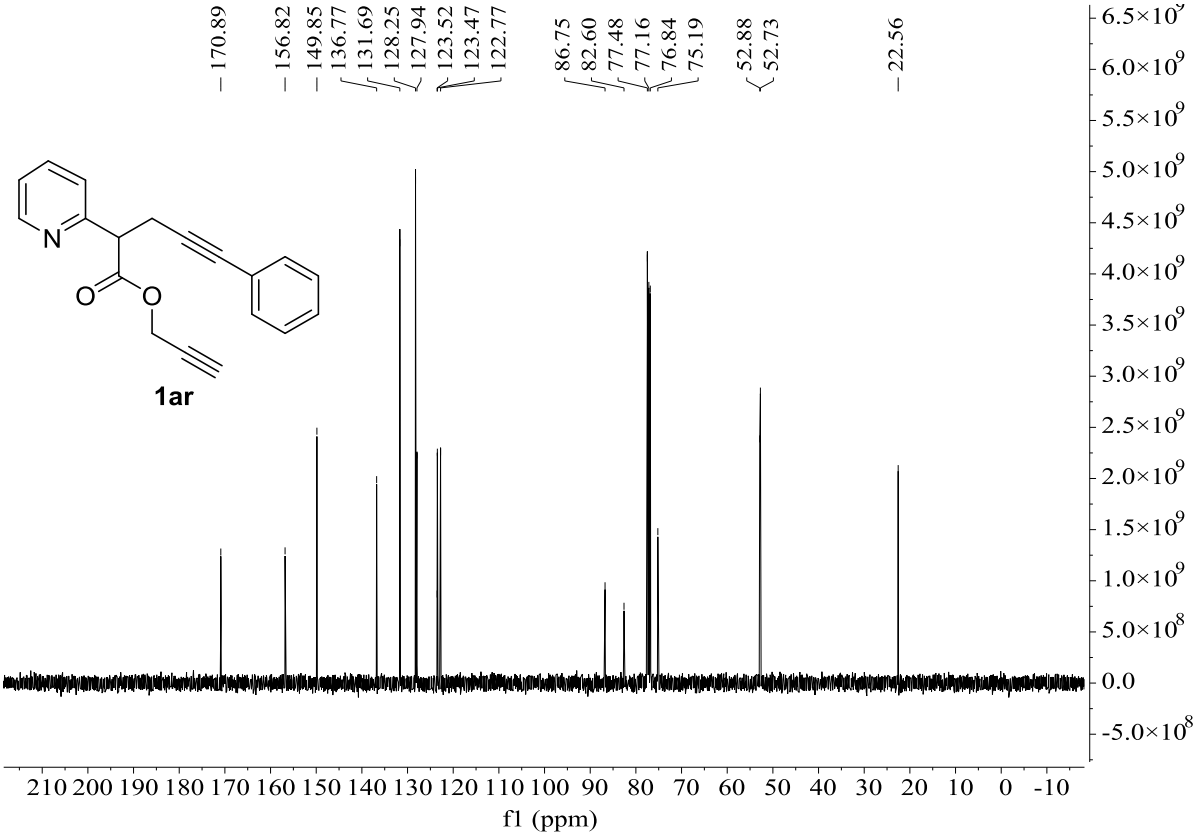
^{13}C NMR Spectrum of 1aq at 25 °C ($CDCl_3$, 100 MHz)



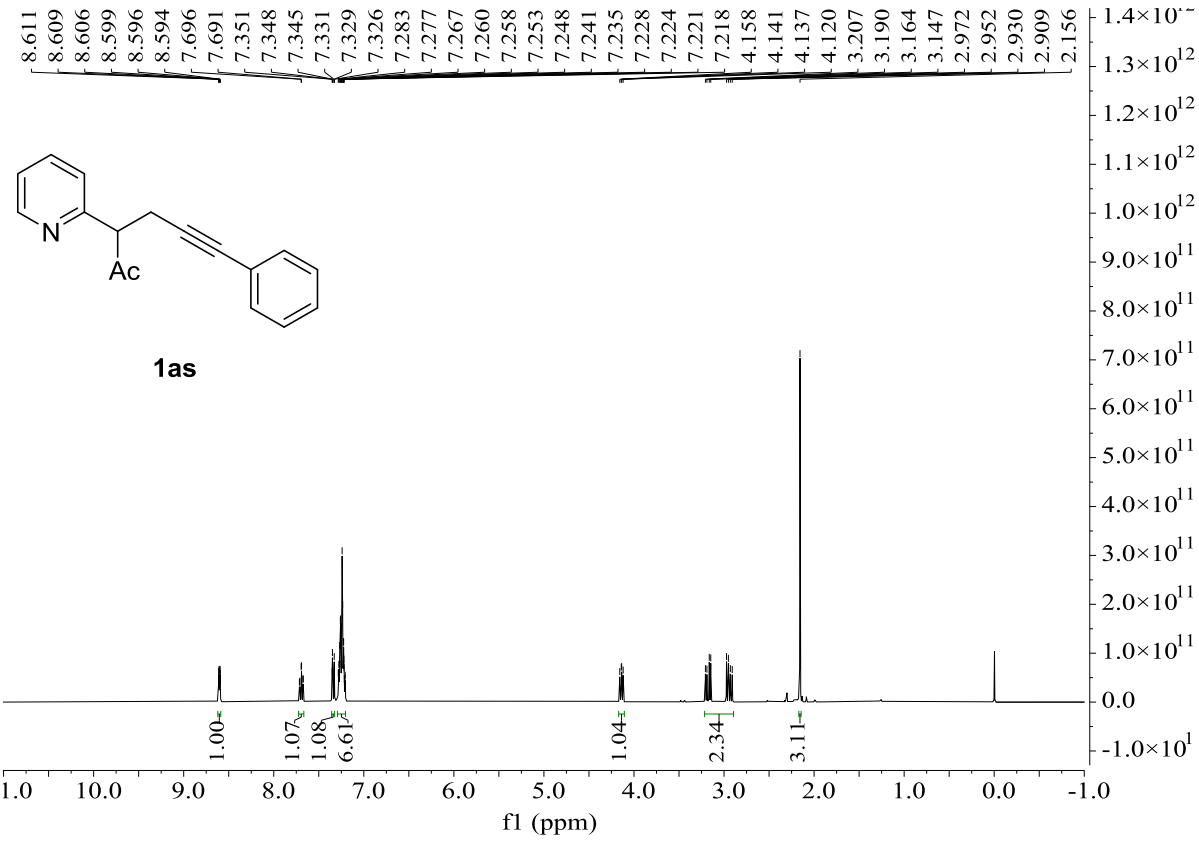
^{1}H NMR Spectrum of 1ar at 25 °C ($CDCl_3$, 400 MHz)



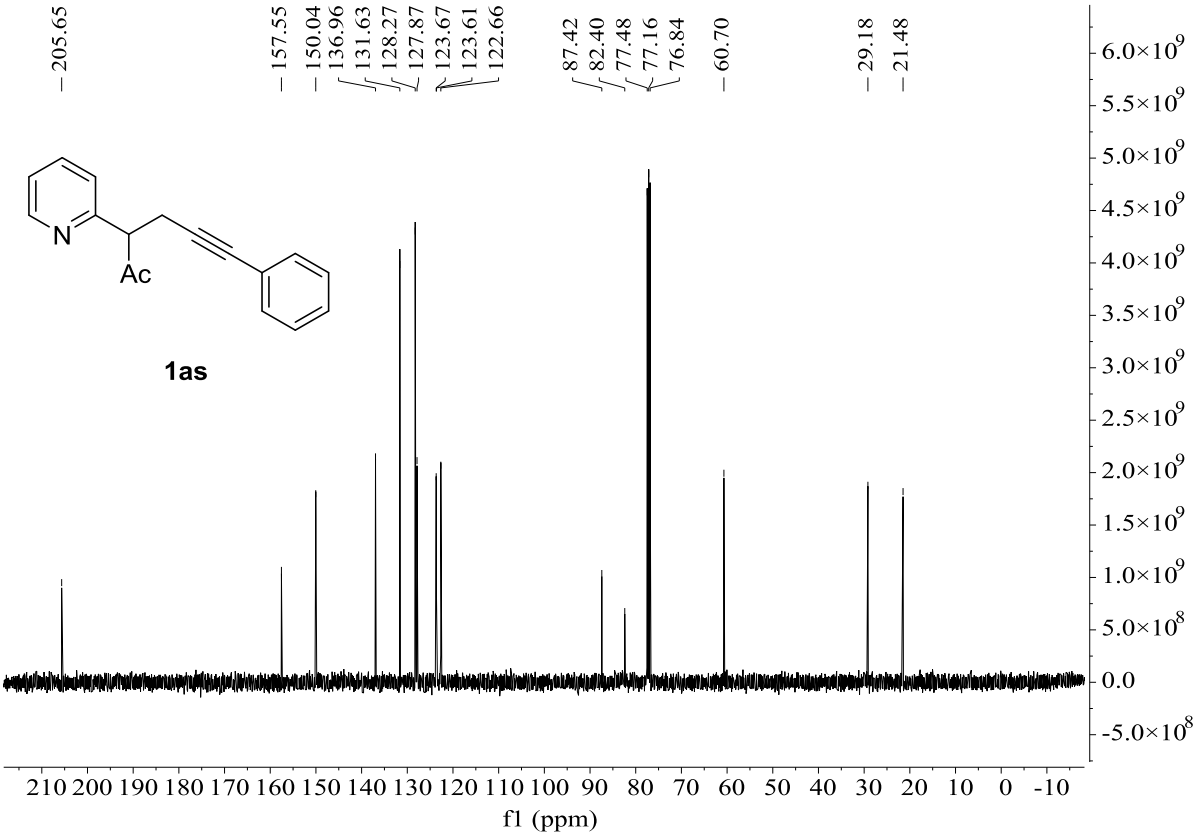
^{13}C NMR Spectrum of 1ar at 25 °C ($CDCl_3$, 100 MHz)



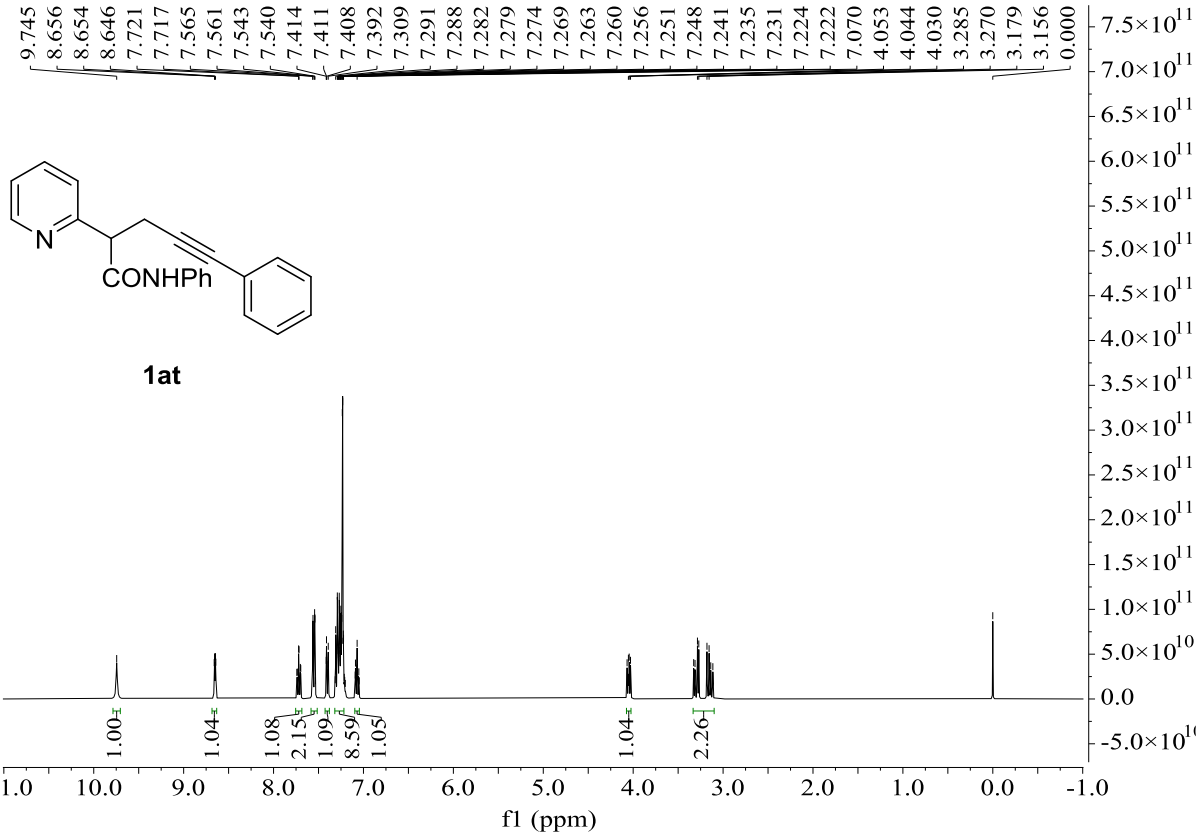
^{1}H NMR Spectrum of 1as at 25 °C ($CDCl_3$, 400 MHz)



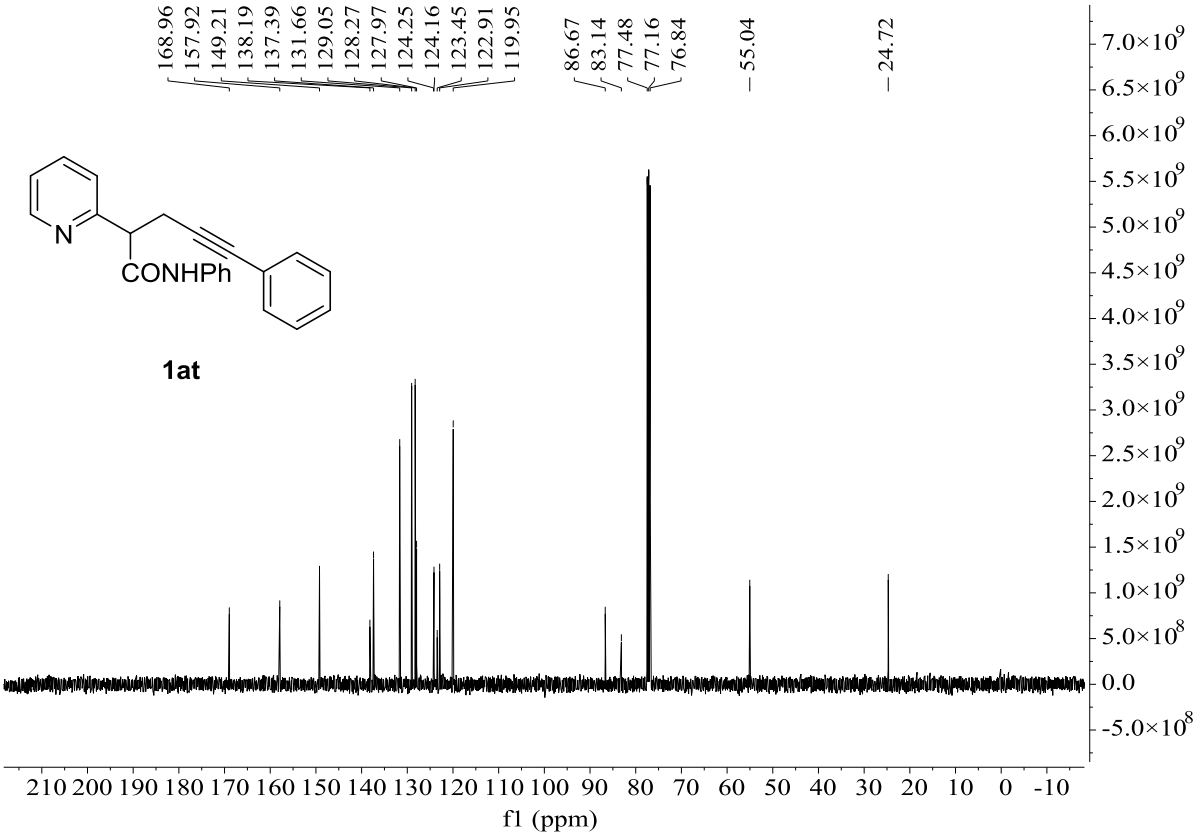
^{13}C NMR Spectrum of 1as at 25 °C ($CDCl_3$, 100 MHz)



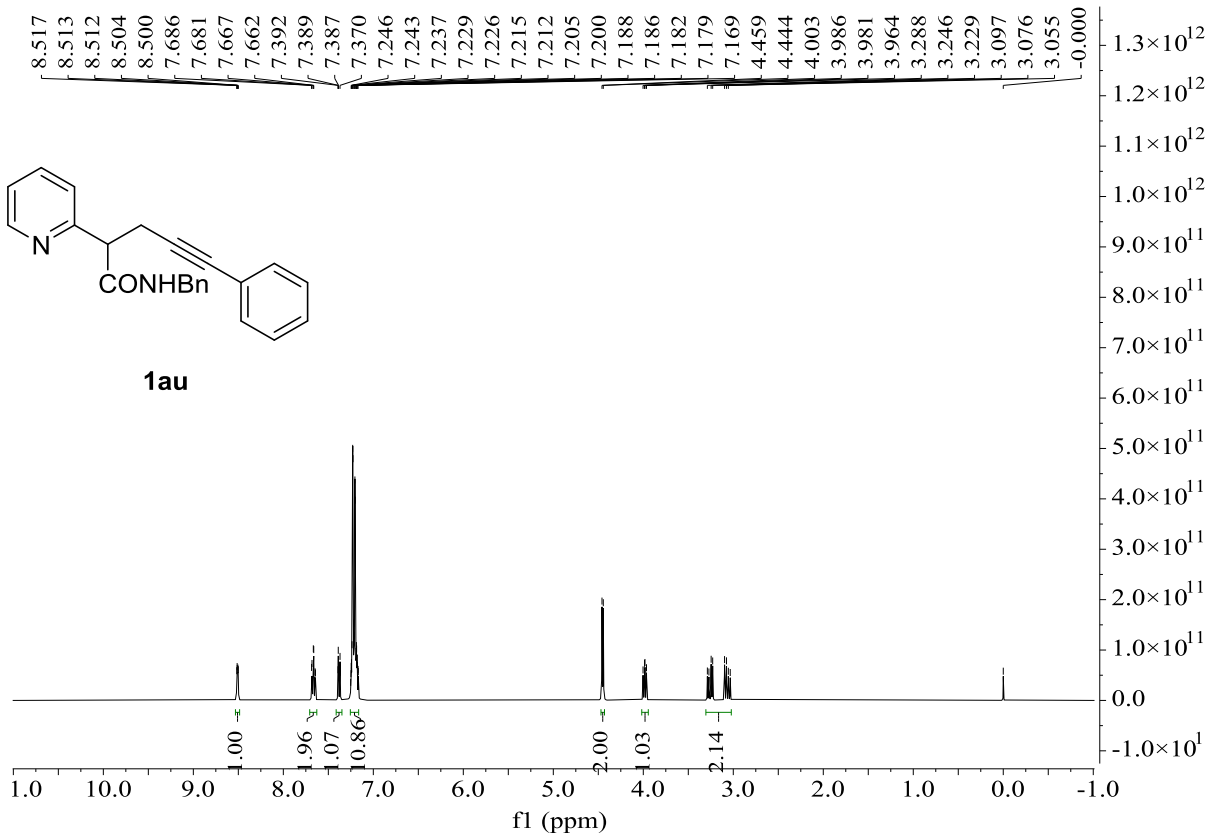
^{1}H NMR Spectrum of 1at at 25 °C ($CDCl_3$, 400 MHz)



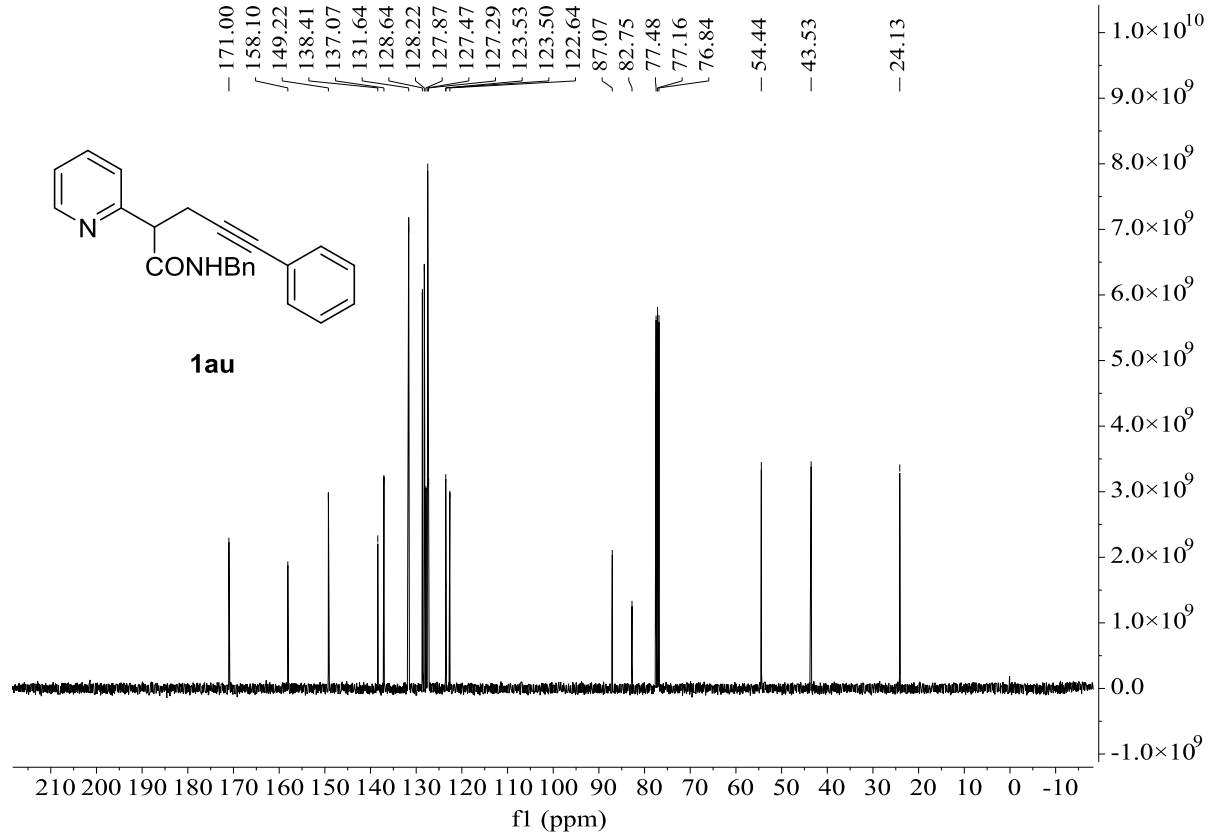
^{13}C NMR Spectrum of 1at at 25 °C ($CDCl_3$, 100 MHz)



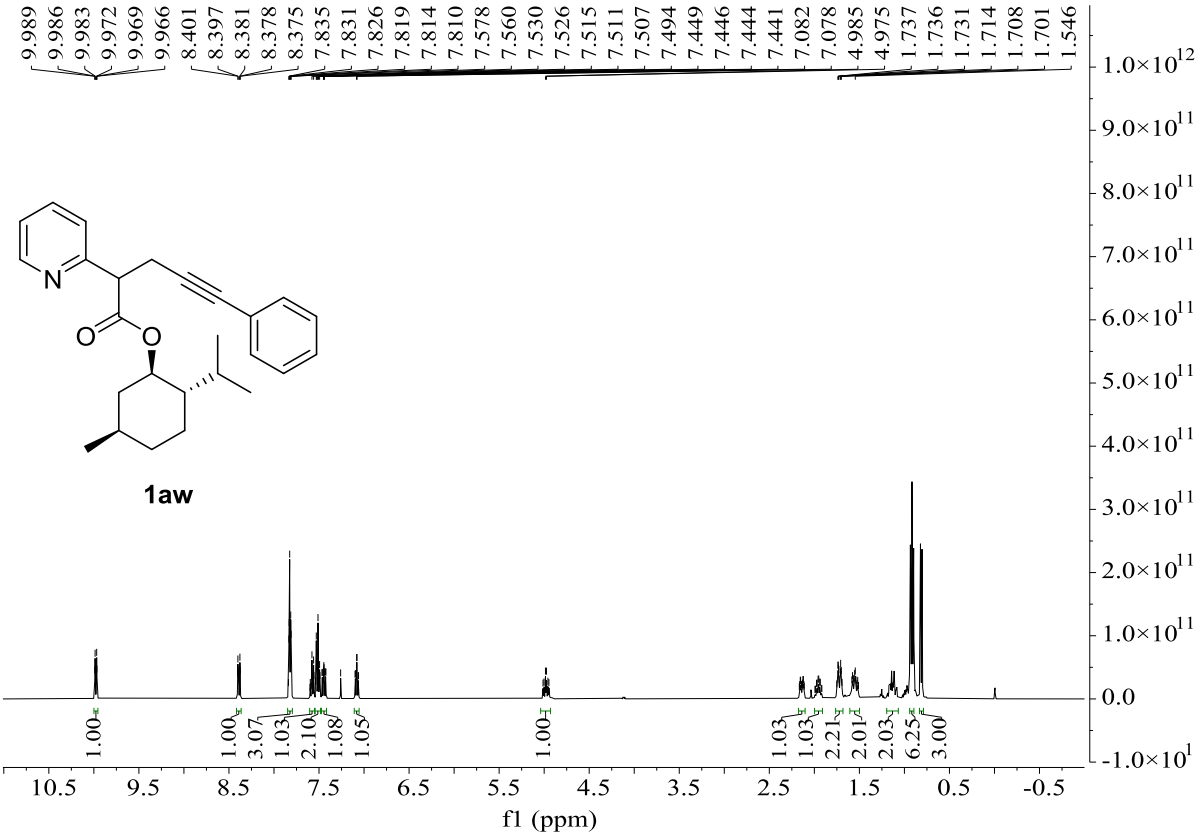
^{1}H NMR Spectrum of 1au at 25 °C ($CDCl_3$, 400 MHz)



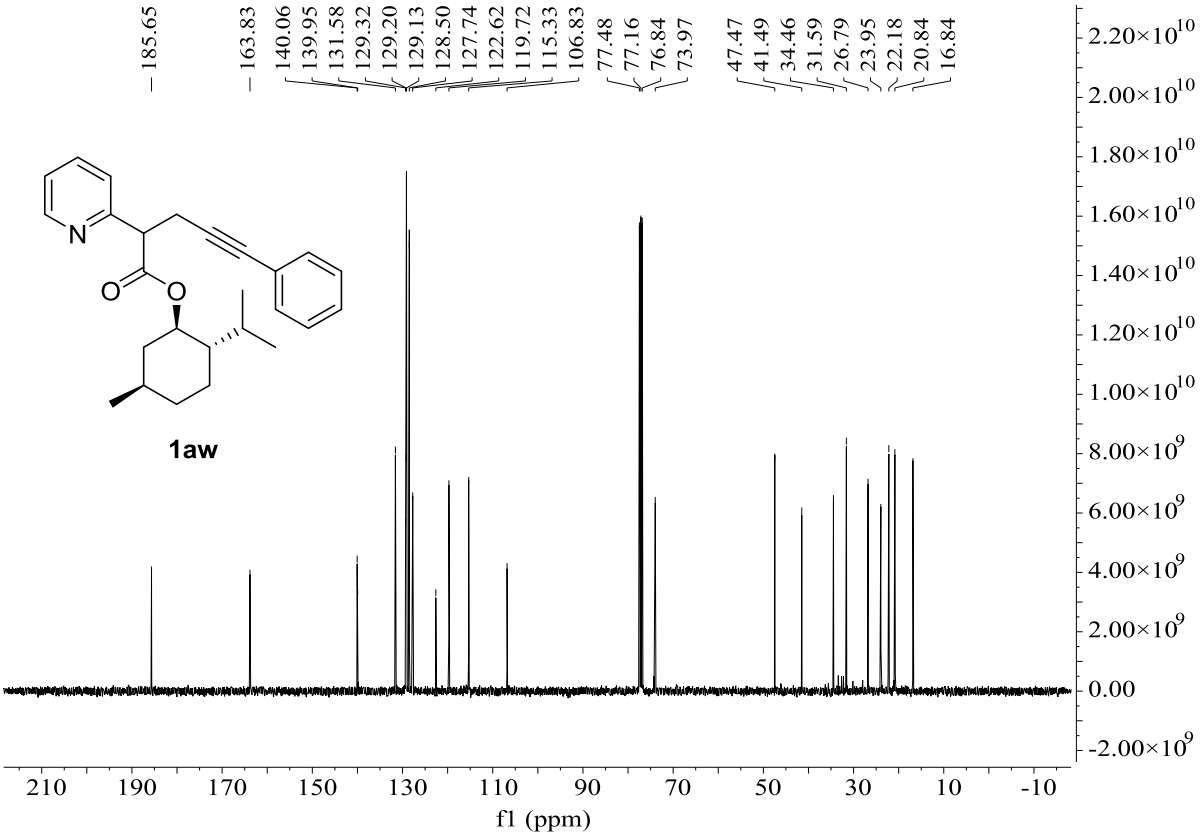
^{13}C NMR Spectrum of 1au at 25 °C ($CDCl_3$, 100 MHz)



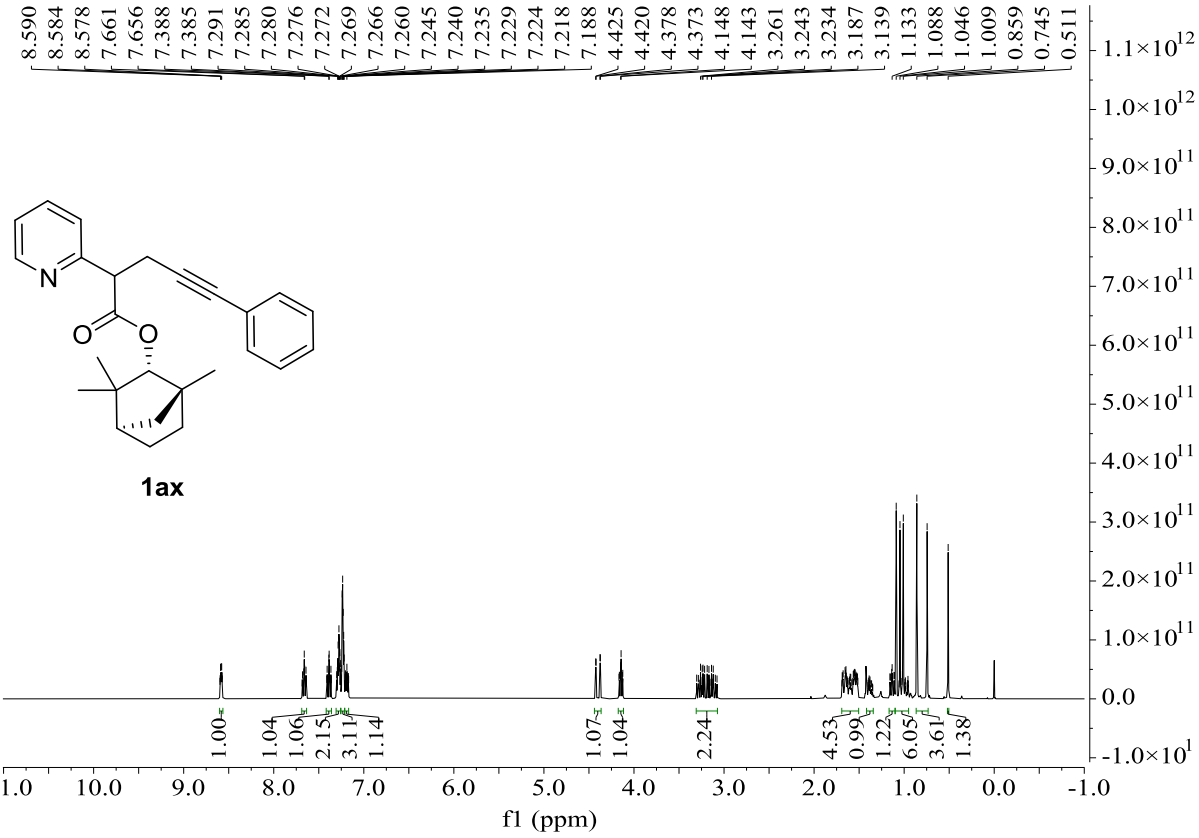
^{1}H NMR Spectrum of 1aw at 25 °C ($CDCl_3$, 400 MHz)



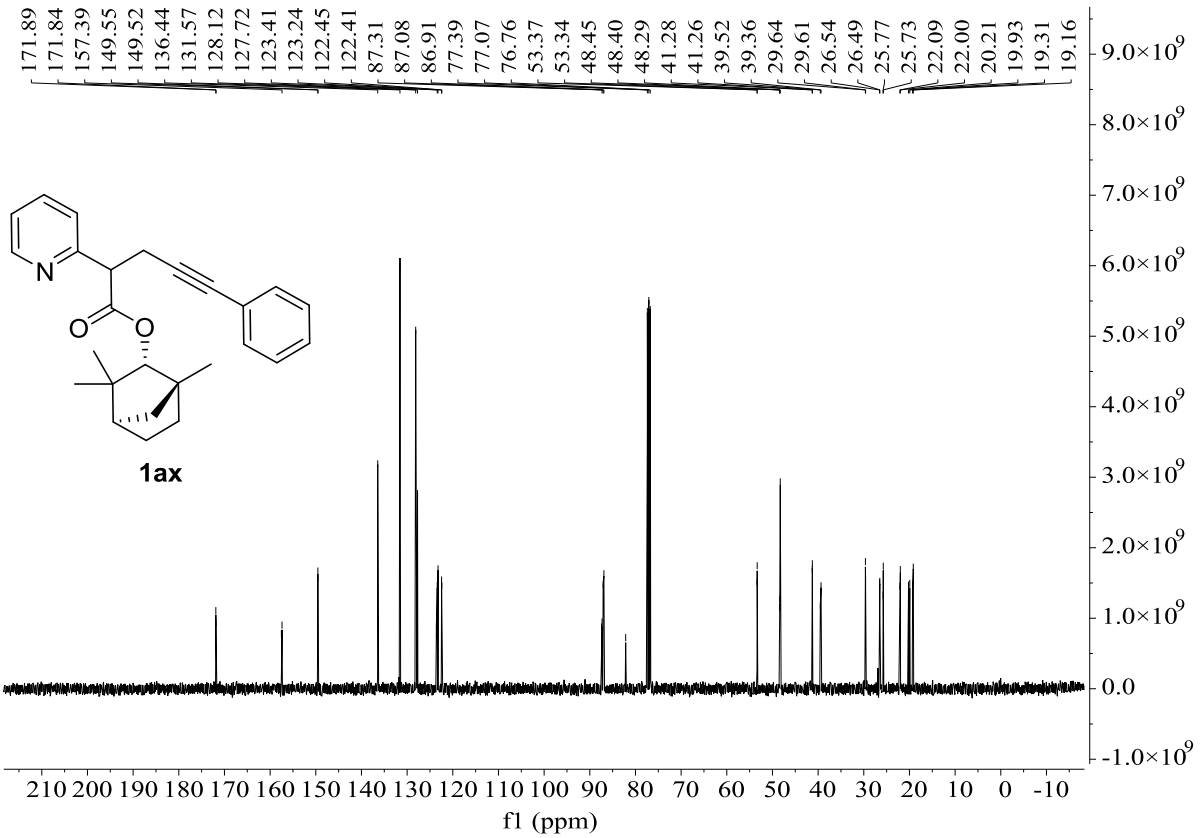
^{13}C NMR Spectrum of 1aw at 25 °C ($CDCl_3$, 100 MHz)



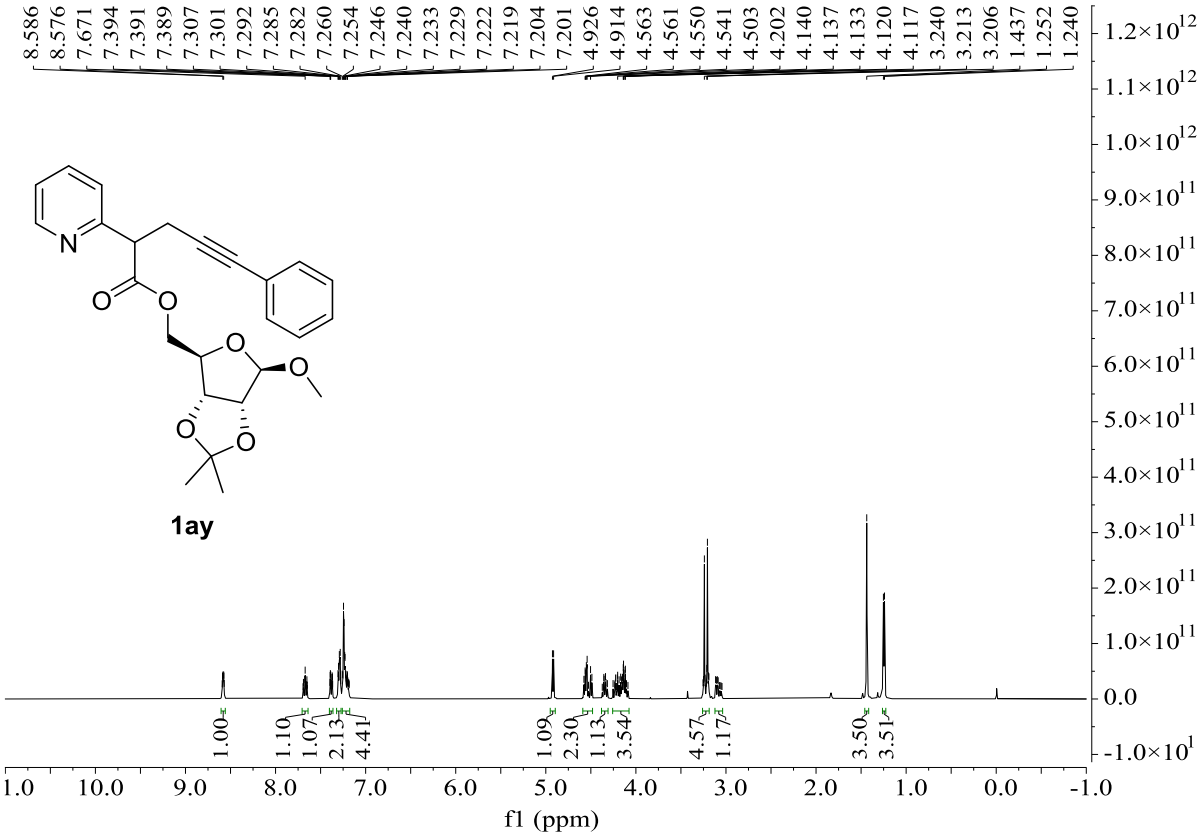
^{1}H NMR Spectrum of 1ax at 25 °C ($CDCl_3$, 400 MHz)



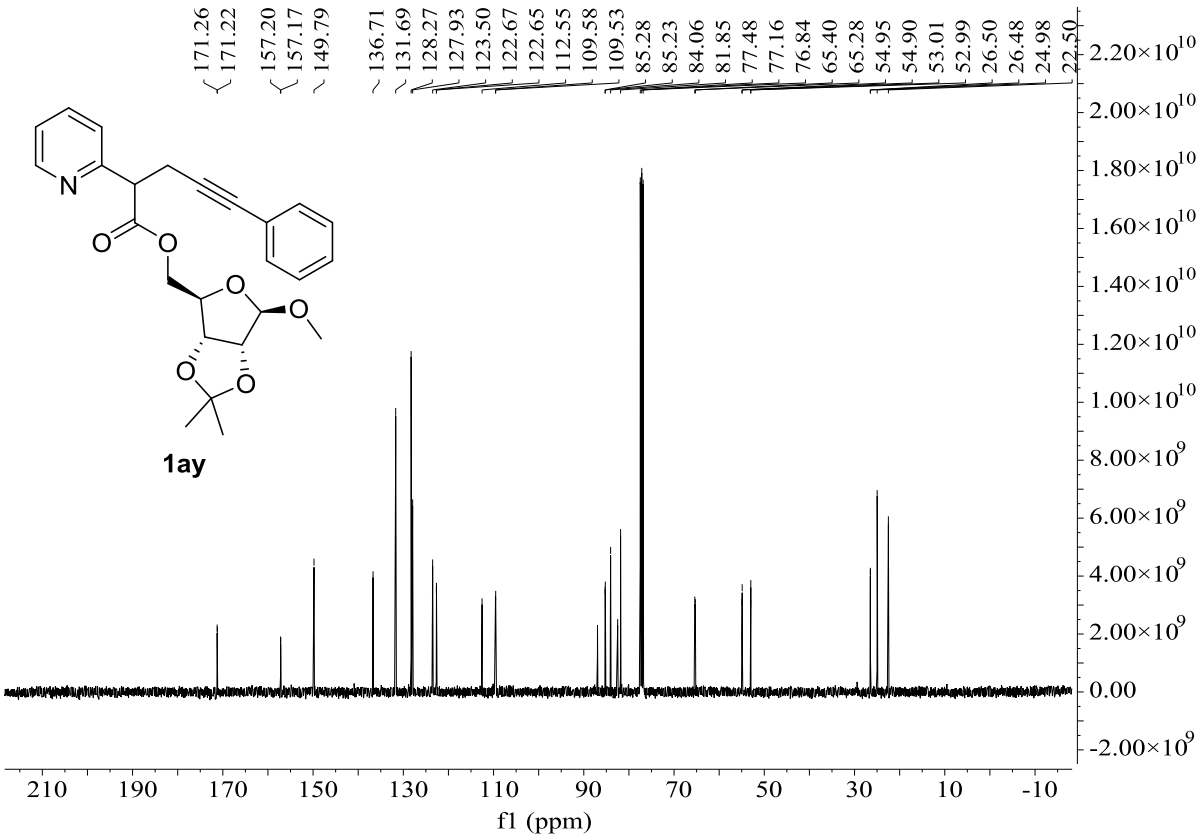
^{13}C NMR Spectrum of 1ax at 25 °C ($CDCl_3$, 100 MHz)



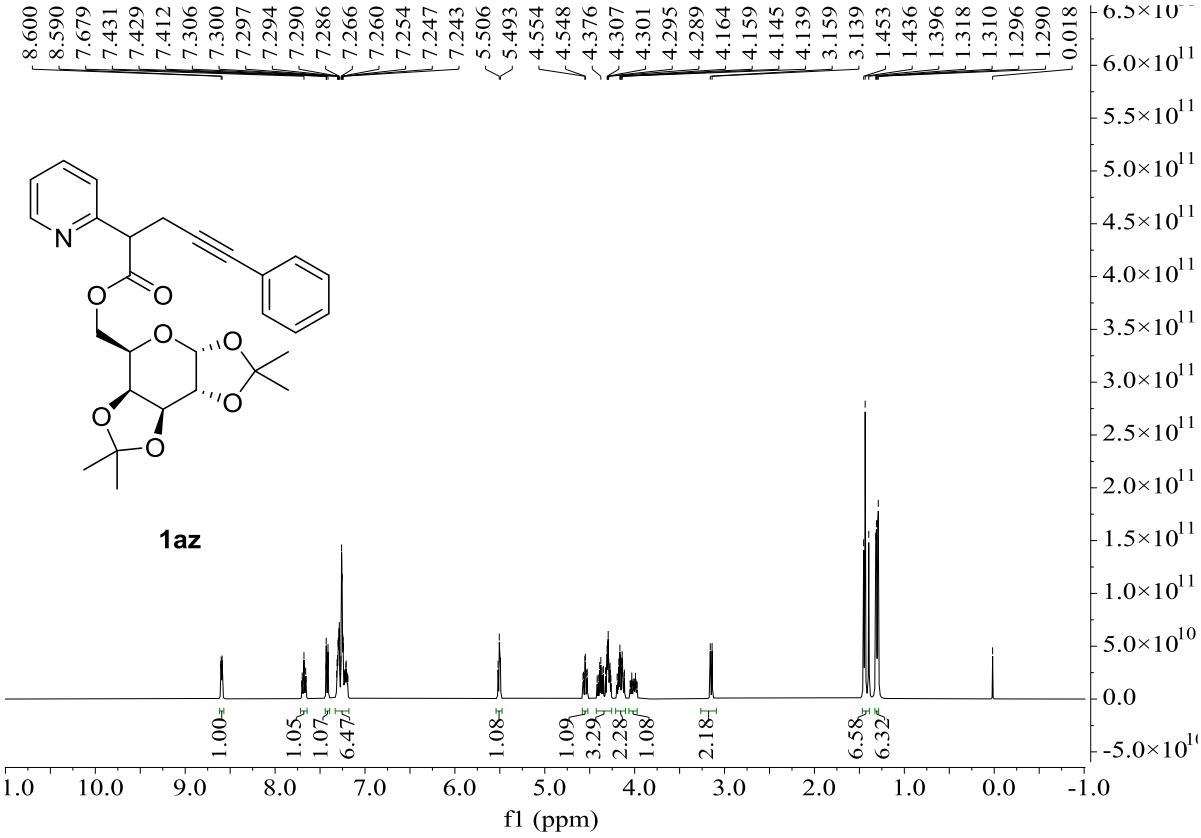
^{13}C NMR Spectrum of 1ay at 25 °C ($CDCl_3$, 100 MHz)



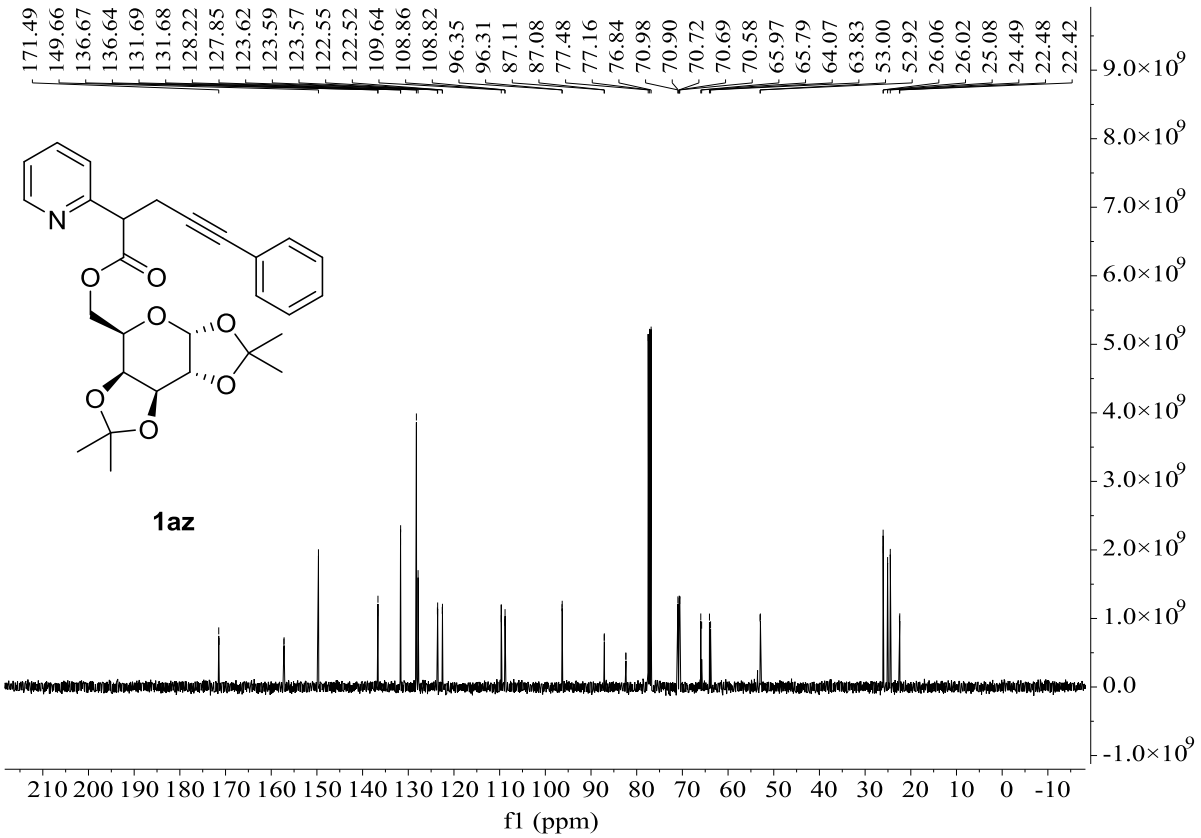
^{13}C NMR Spectrum of 1ay at 25 °C ($CDCl_3$, 100 MHz)



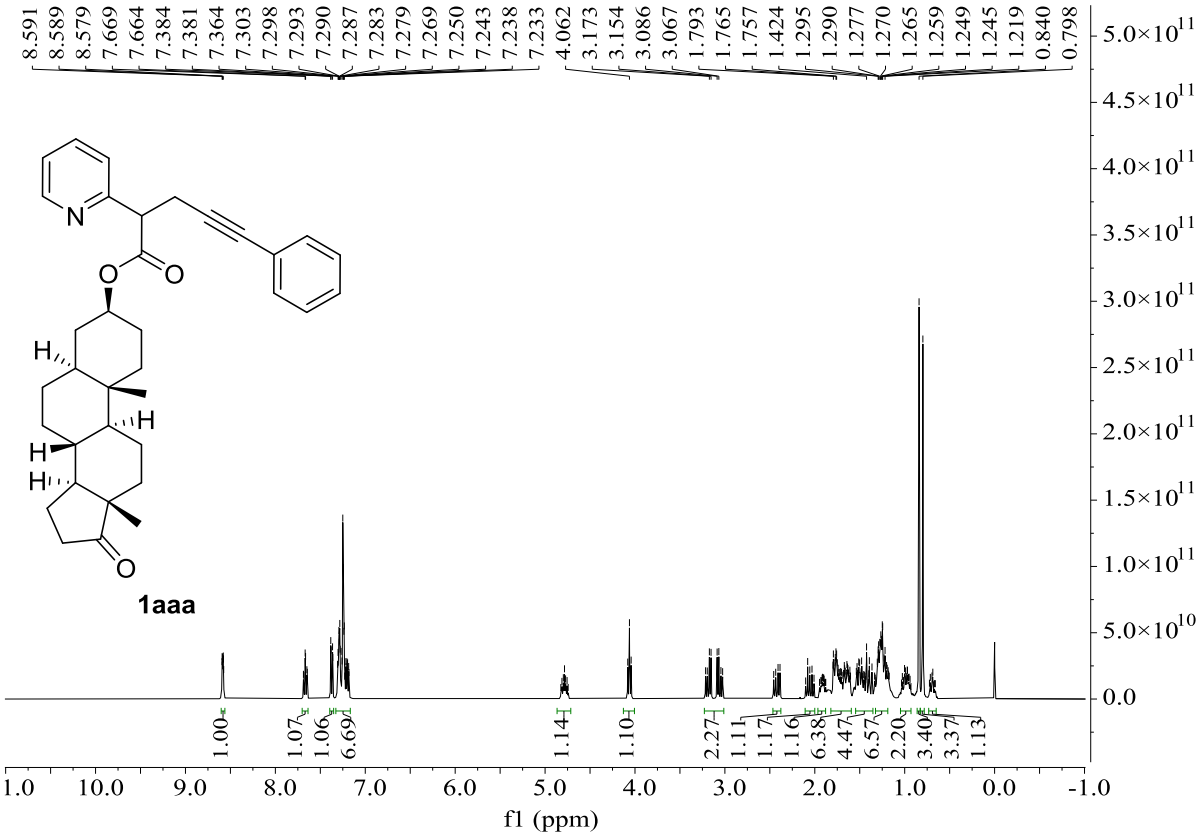
^{1}H NMR Spectrum of 1az at 25 °C ($CDCl_3$, 400 MHz)



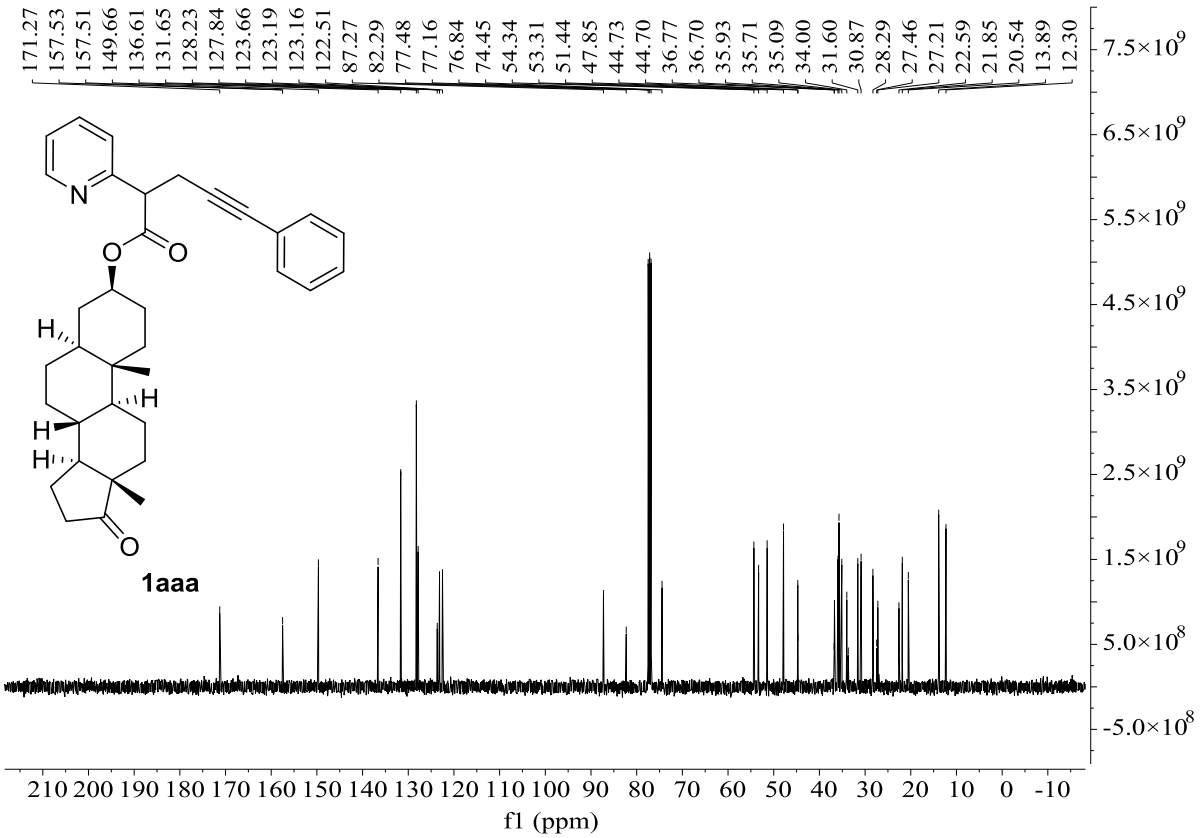
^{13}C NMR Spectrum of 1az at 25 °C ($CDCl_3$, 100 MHz)



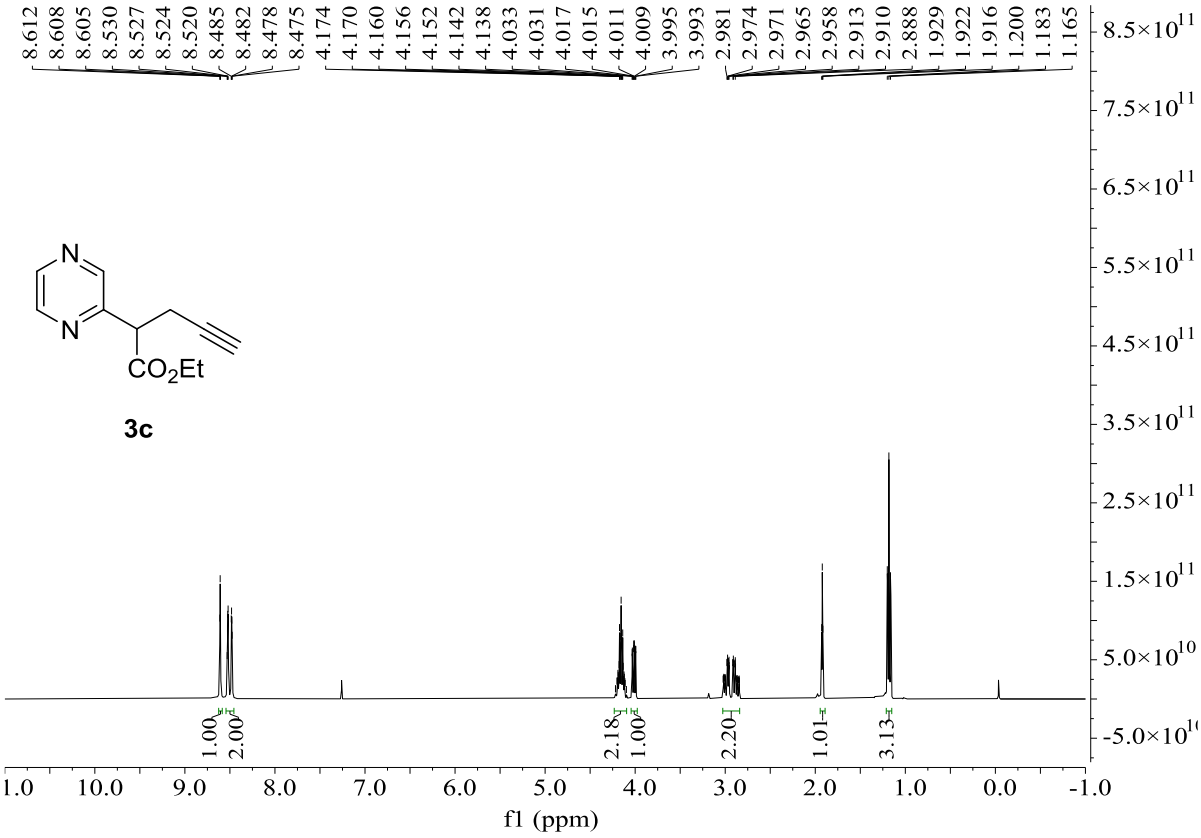
^{1}H NMR Spectrum of 1aaa at 25 °C ($CDCl_3$, 400 MHz)



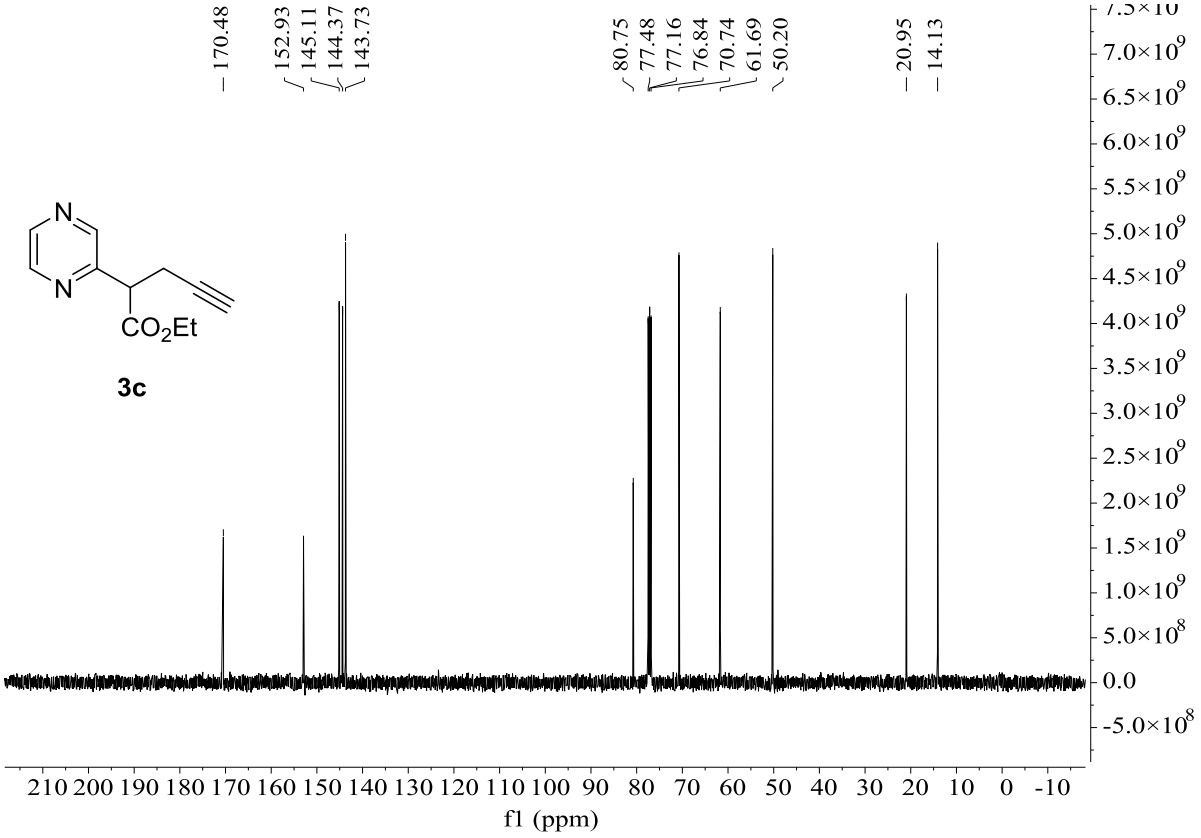
^{13}C NMR Spectrum of 1aaa at 25 °C ($CDCl_3$, 100 MHz)



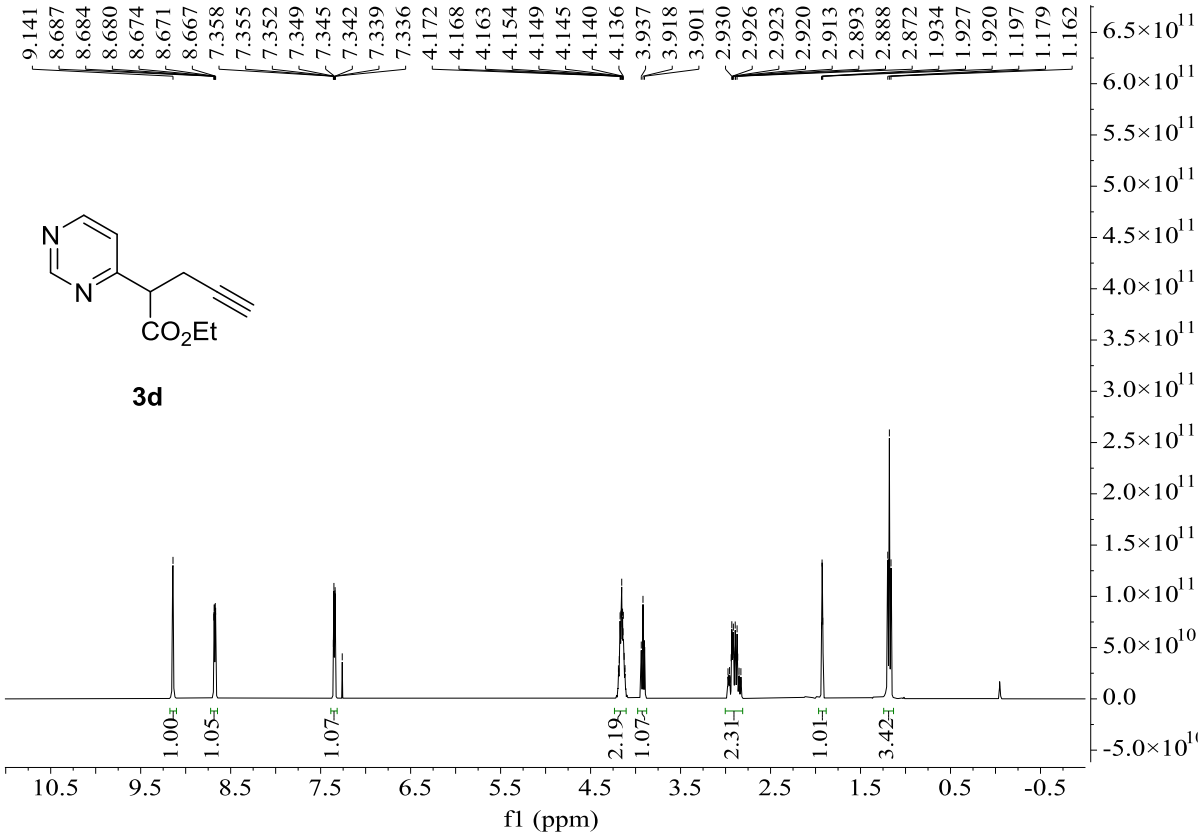
^{1}H NMR Spectrum of 3c at 25 °C ($CDCl_3$, 400 MHz)



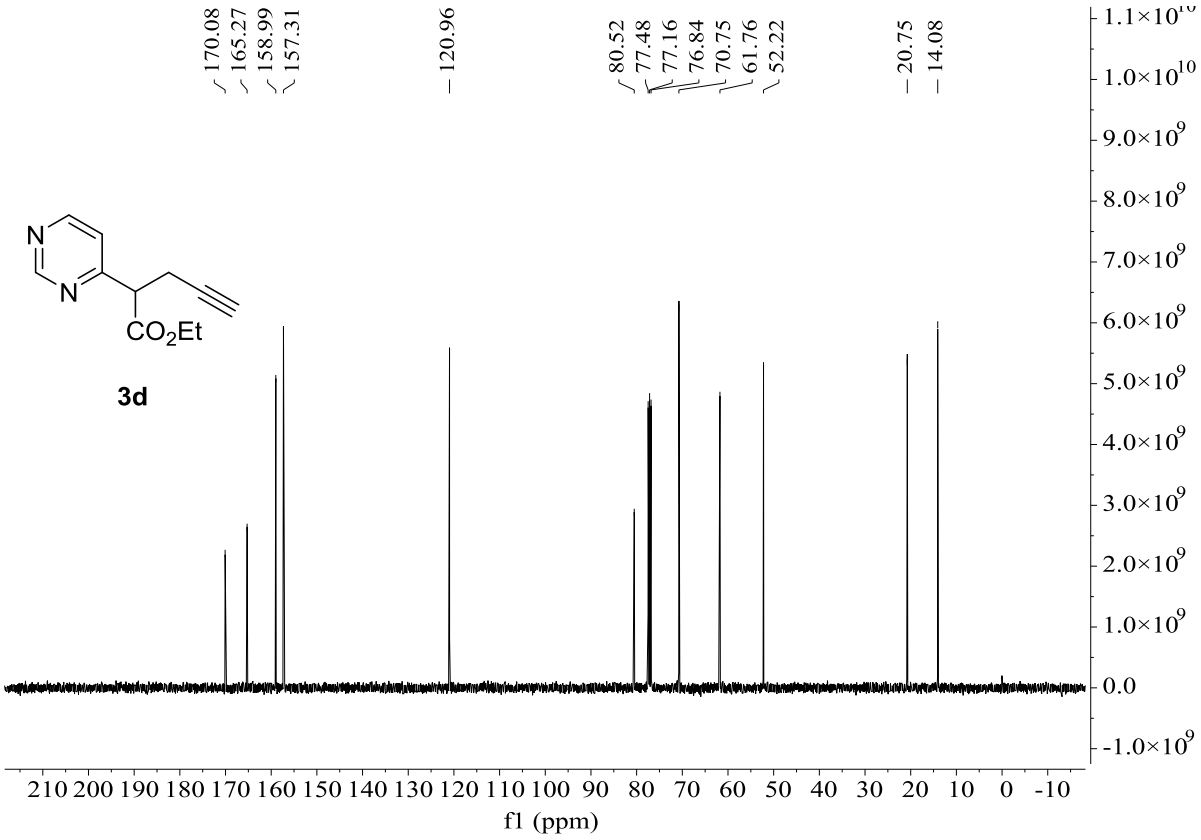
^{13}C NMR Spectrum of 3c at 25 °C ($CDCl_3$, 100 MHz)



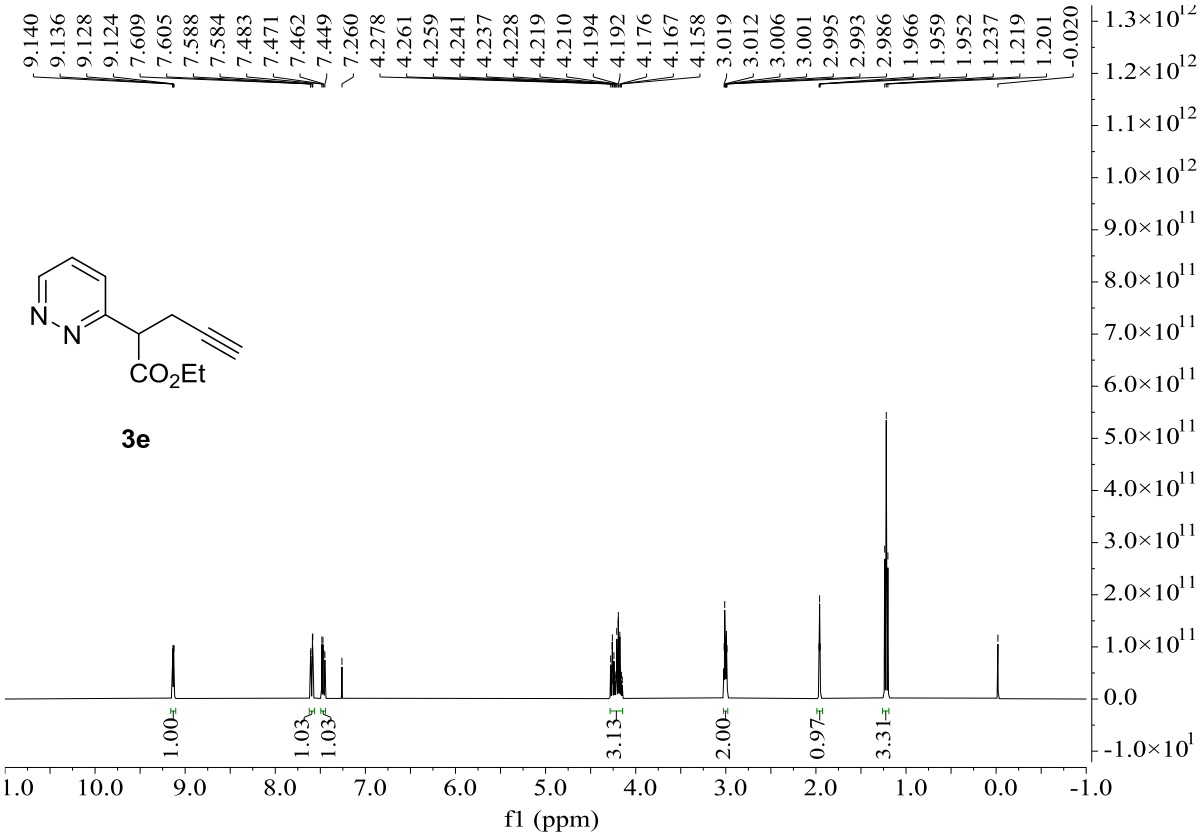
^{1}H NMR Spectrum of 3d at 25 °C ($CDCl_3$, 400 MHz)



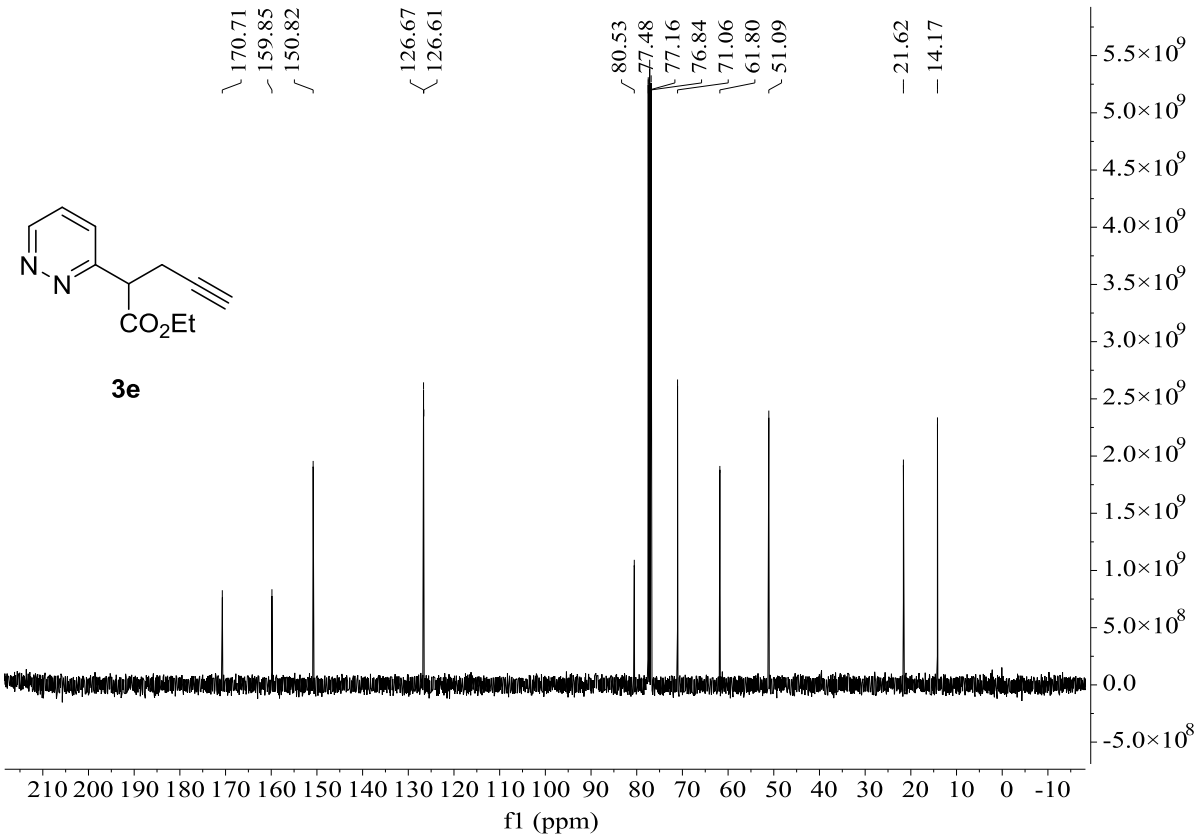
^{13}C NMR Spectrum of 3d at 25 °C ($CDCl_3$, 100 MHz)



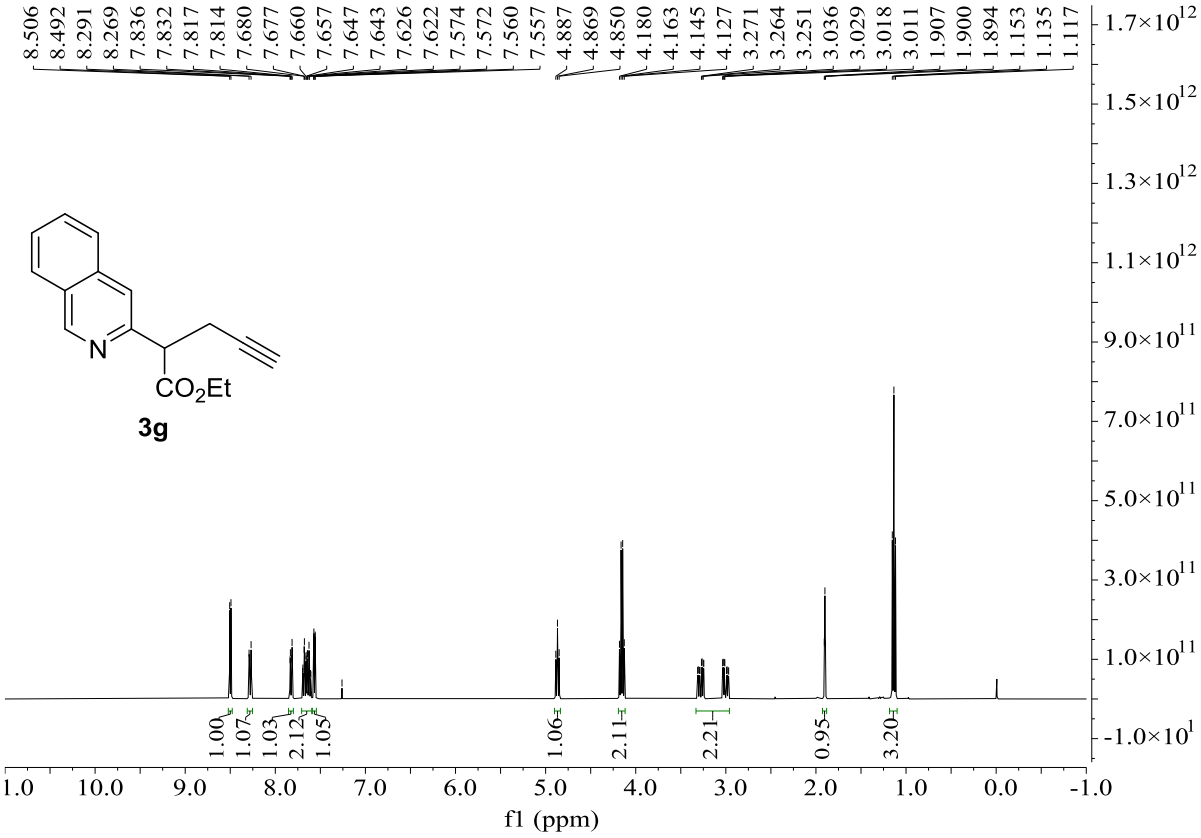
^{1}H NMR Spectrum of 3e at 25 °C ($CDCl_3$, 400 MHz)



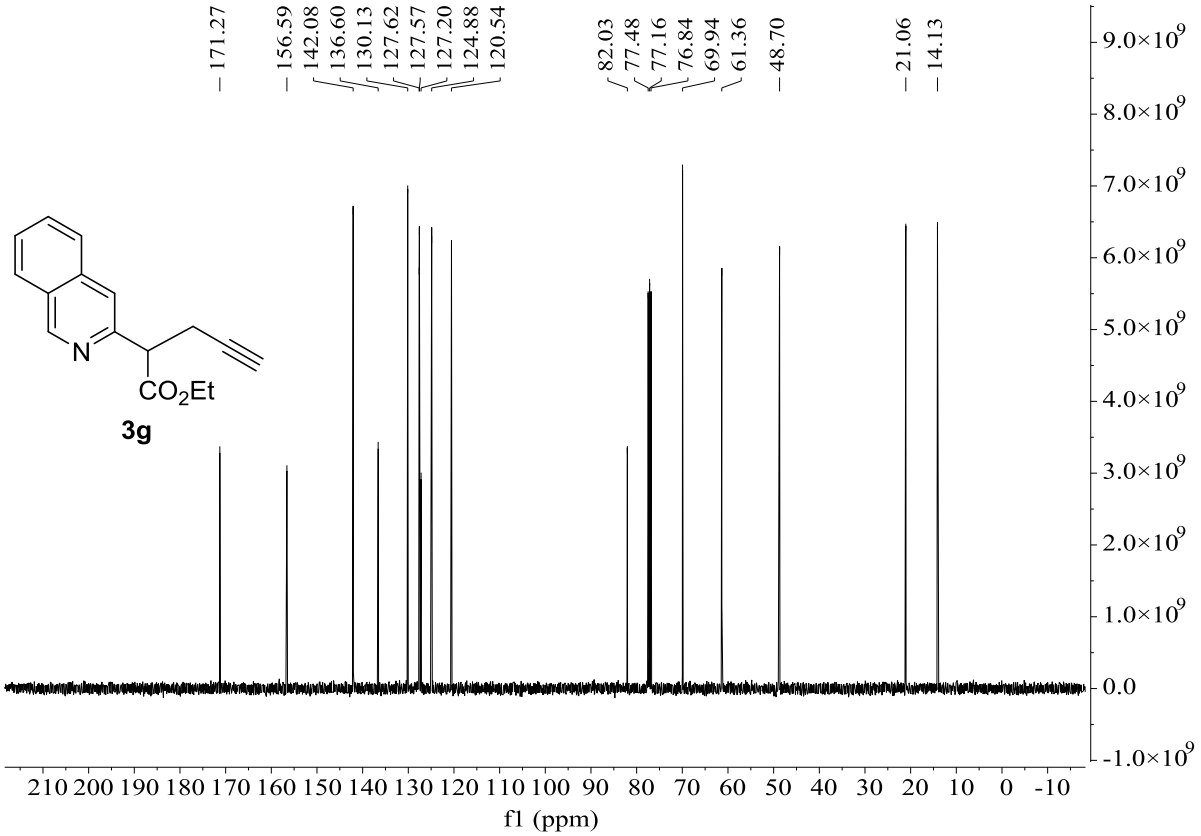
^{13}C NMR Spectrum of 3e at 25 °C ($CDCl_3$, 100 MHz)



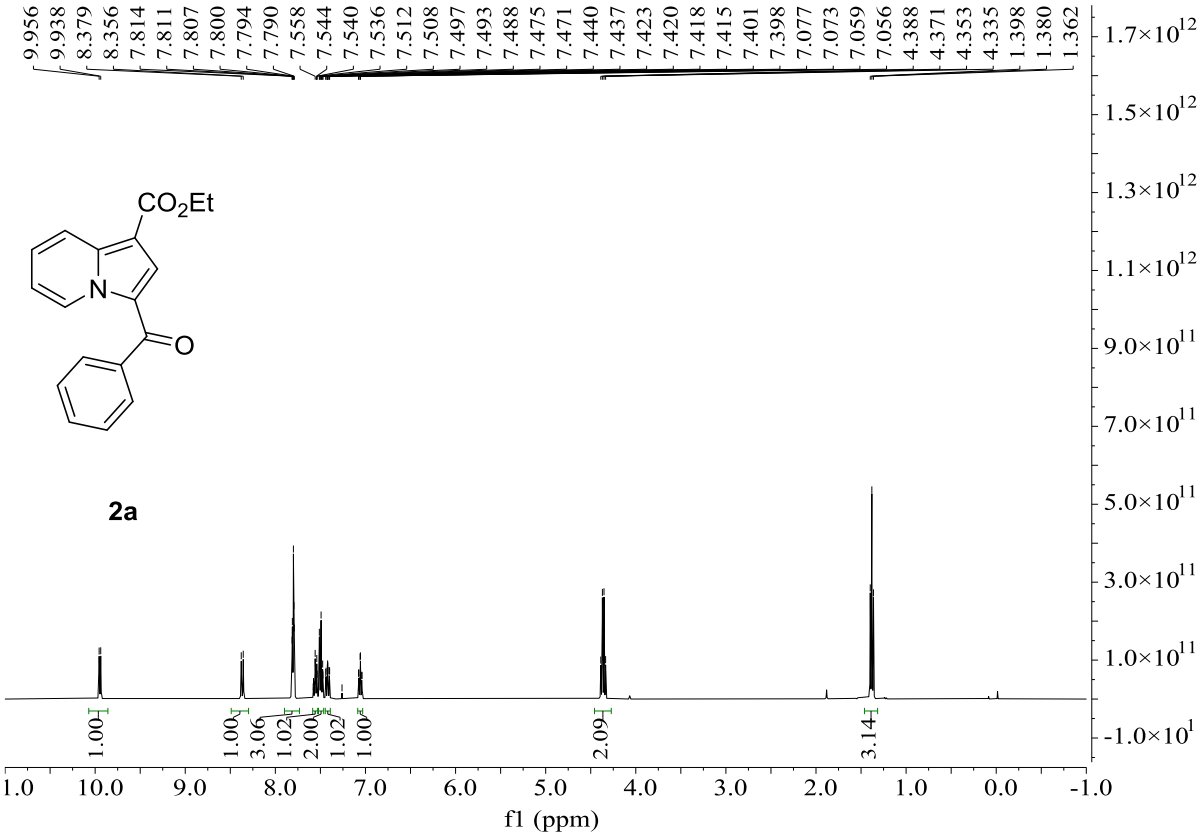
^{1}H NMR Spectrum of 3g at 25 °C ($CDCl_3$, 400 MHz)



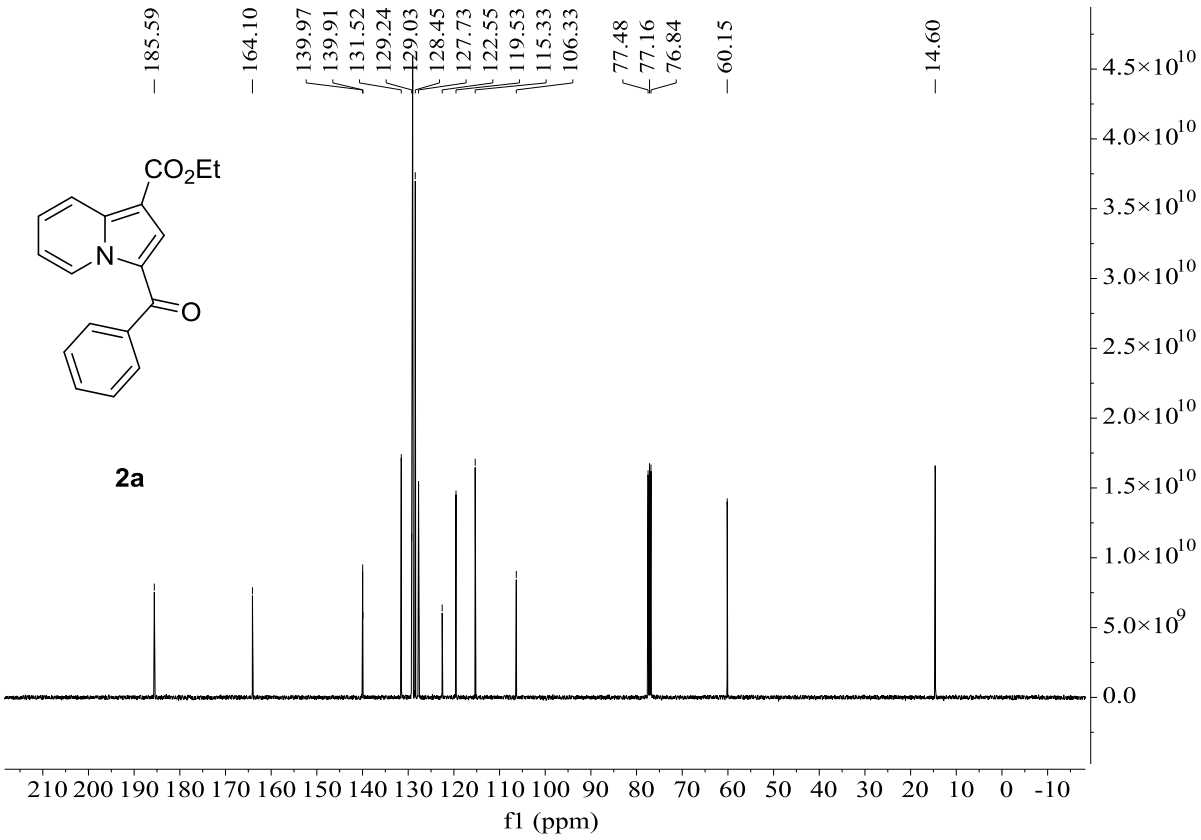
^{13}C NMR Spectrum of 3g at 25 °C ($CDCl_3$, 100 MHz)



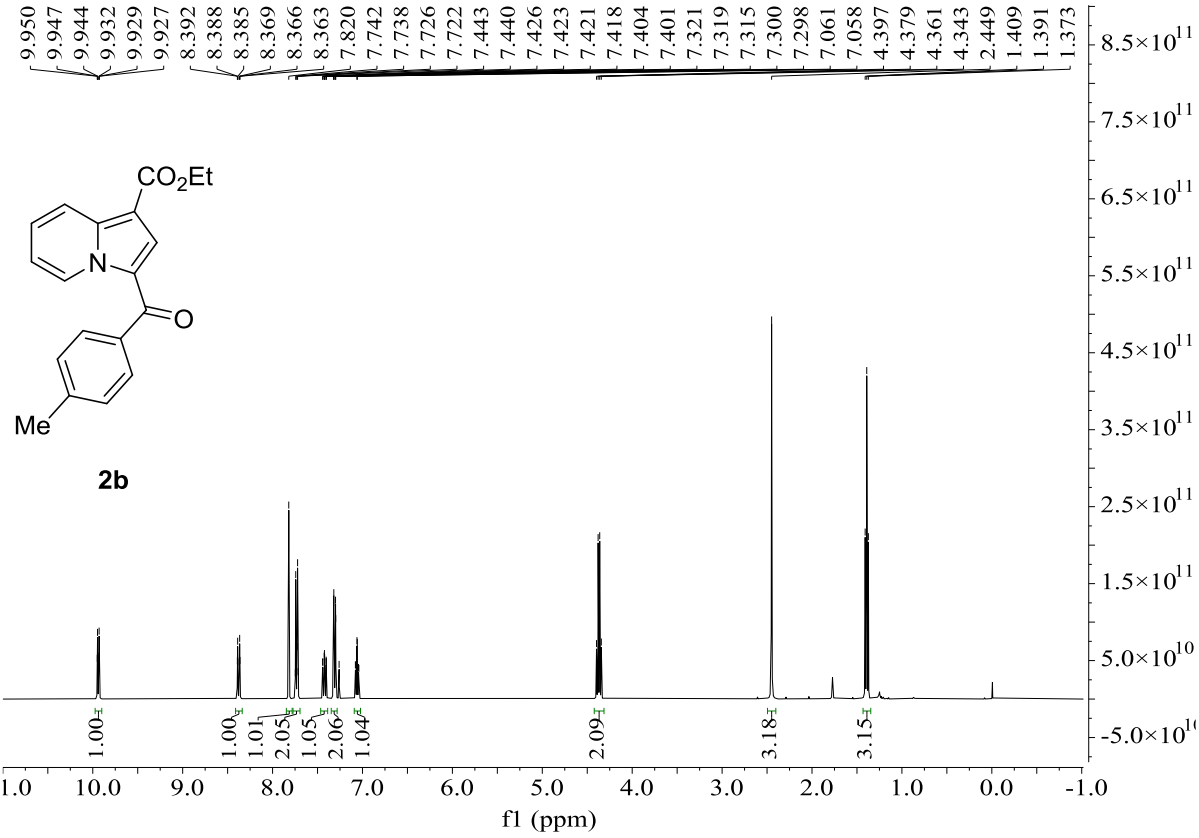
^{1}H NMR Spectrum of 2a at 25 °C ($CDCl_3$, 400 MHz)



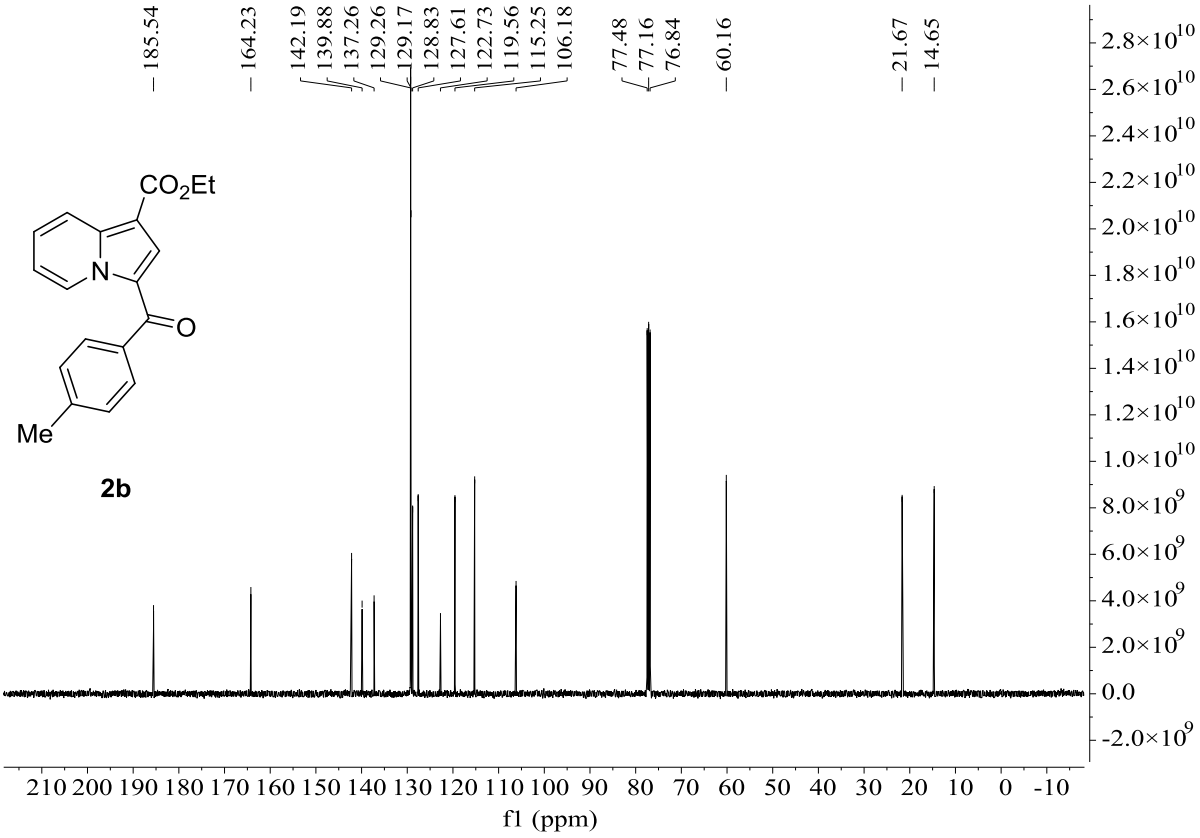
^{13}C NMR Spectrum of 2a at 25 °C ($CDCl_3$, 100 MHz)



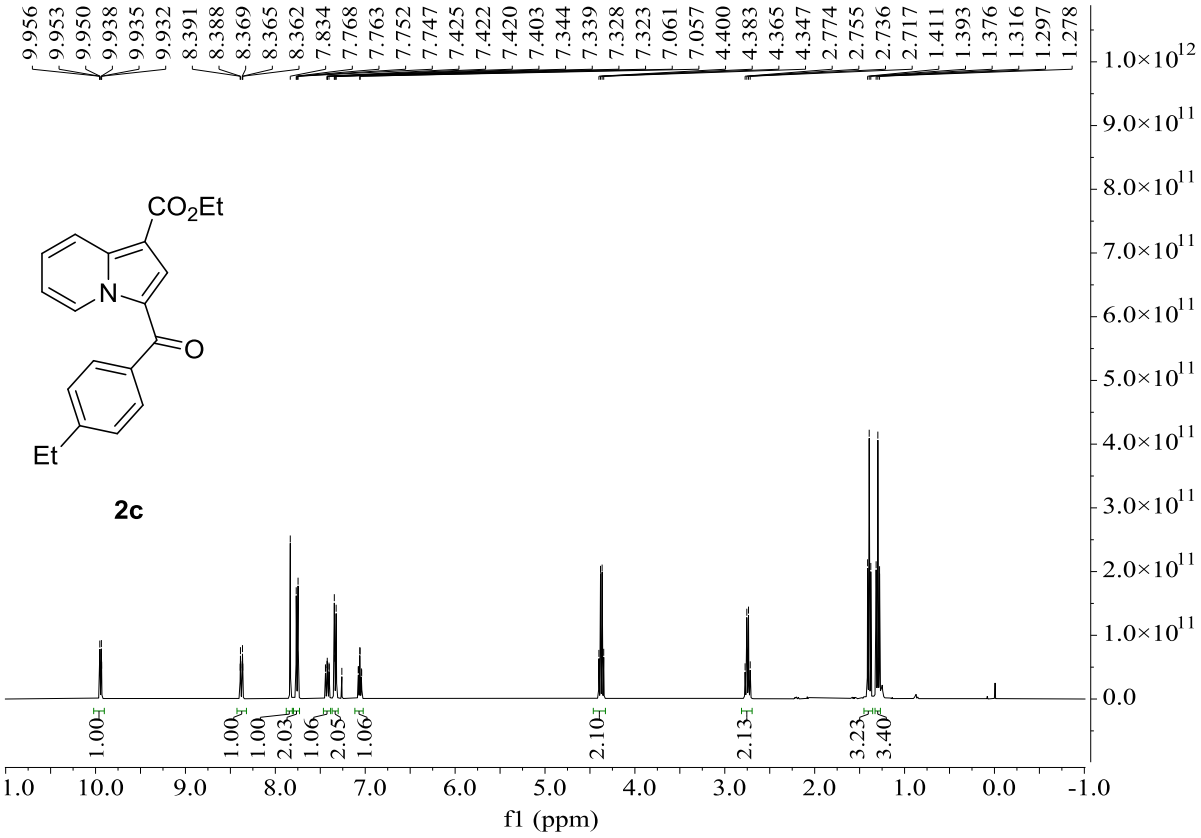
^{1}H NMR Spectrum of 2b at 25 °C ($CDCl_3$, 400 MHz)



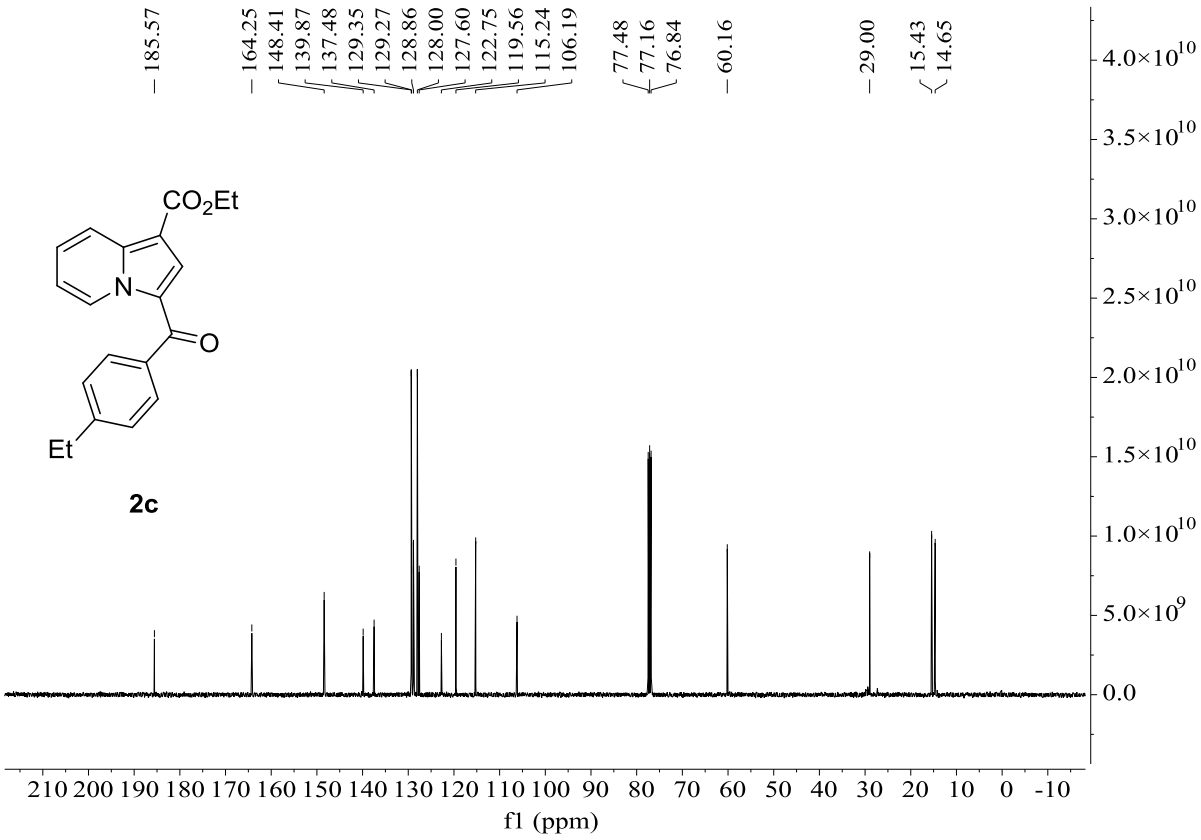
^{13}C NMR Spectrum of 2b at 25 °C ($CDCl_3$, 100 MHz)



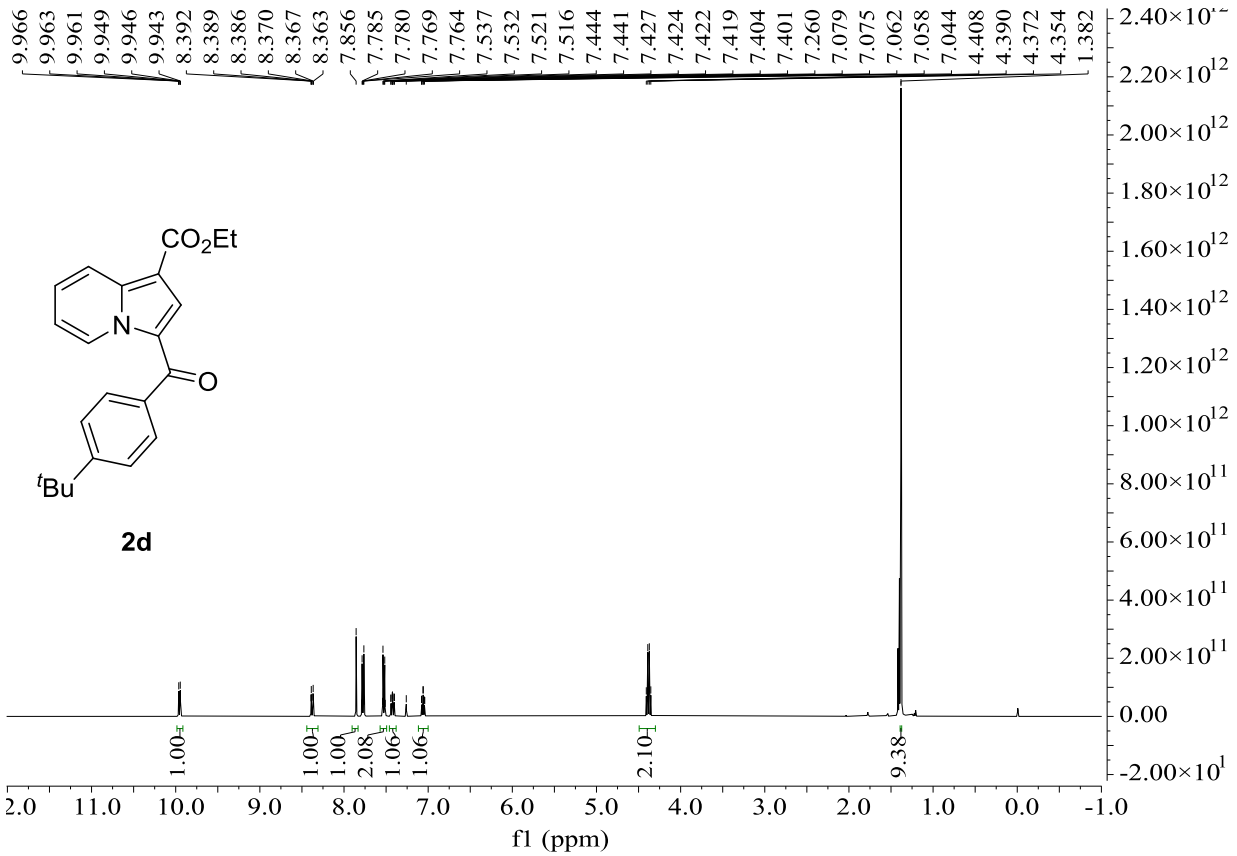
^{1}H NMR Spectrum of 2c at 25 °C ($CDCl_3$, 400 MHz)



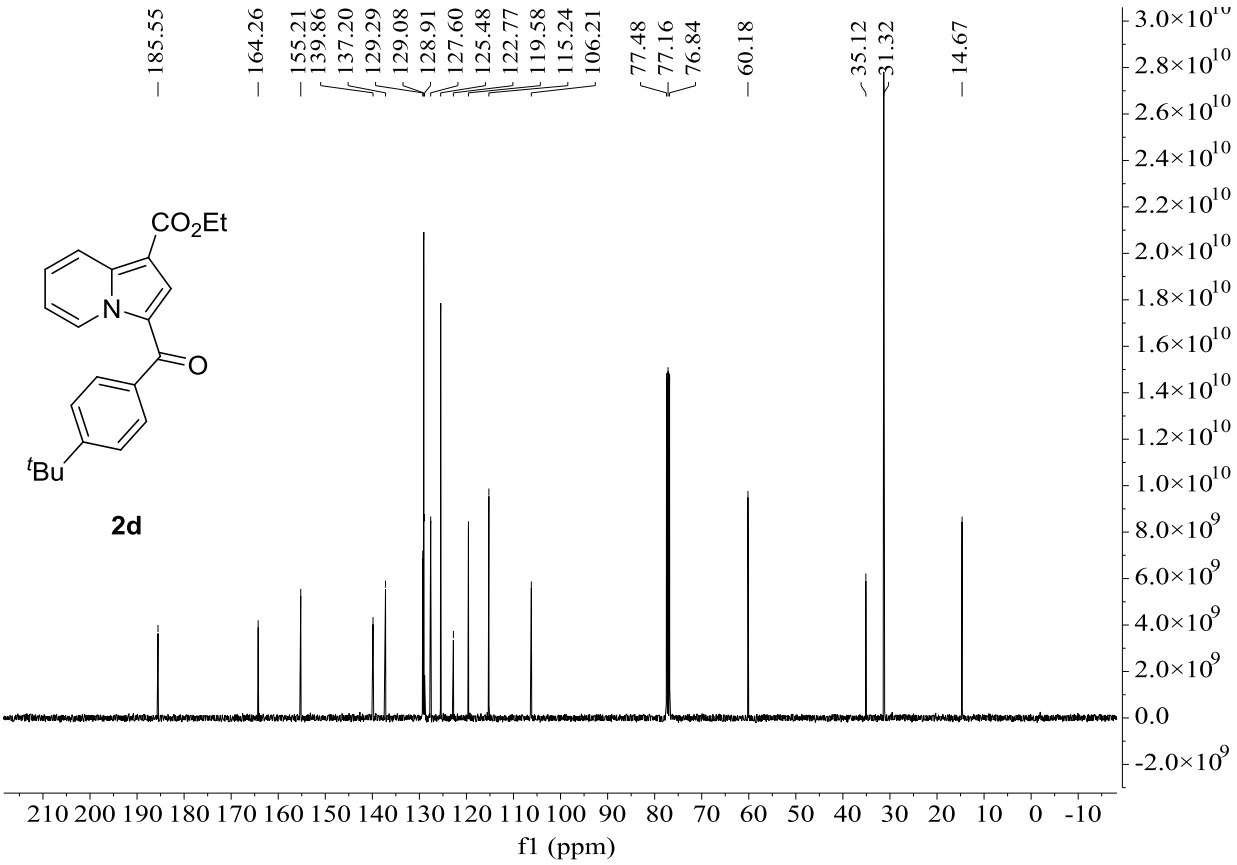
^{13}C NMR Spectrum of 2c at 25 °C ($CDCl_3$, 100 MHz)



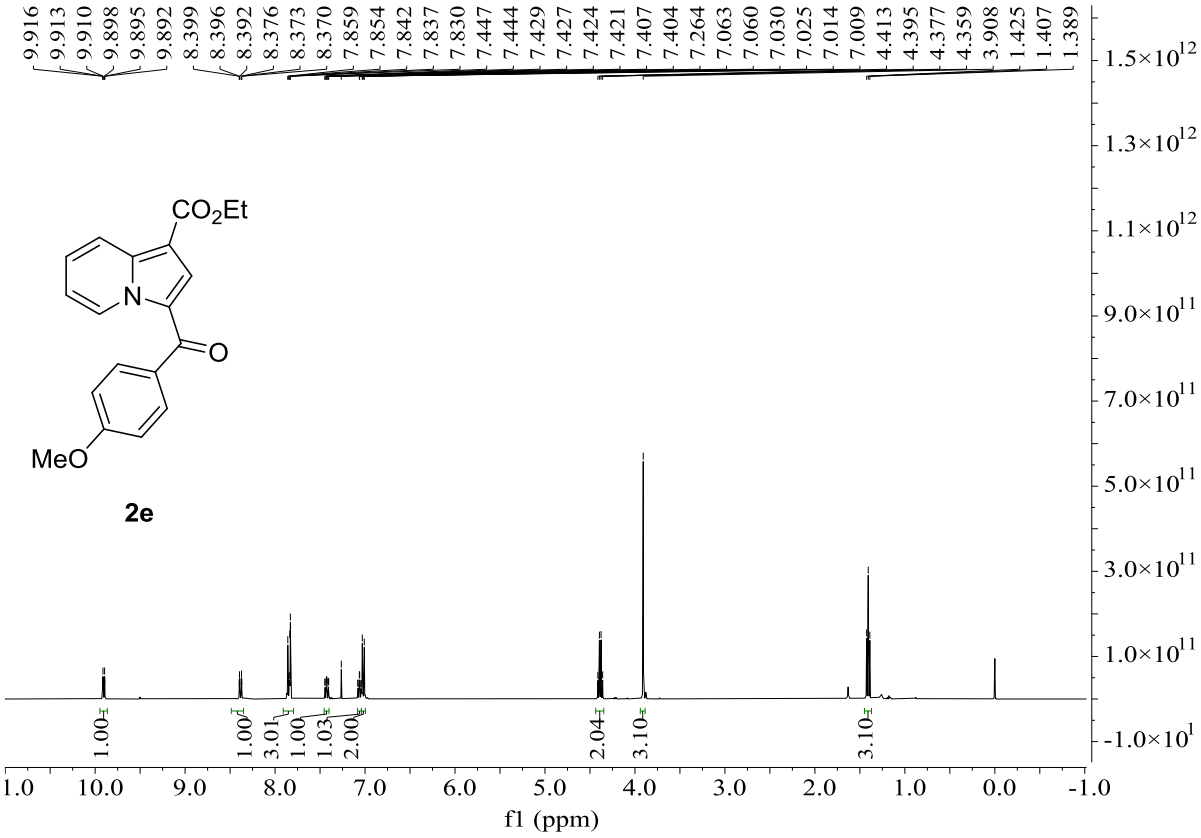
^{1}H NMR Spectrum of 2d at 25 °C ($CDCl_3$, 400 MHz)



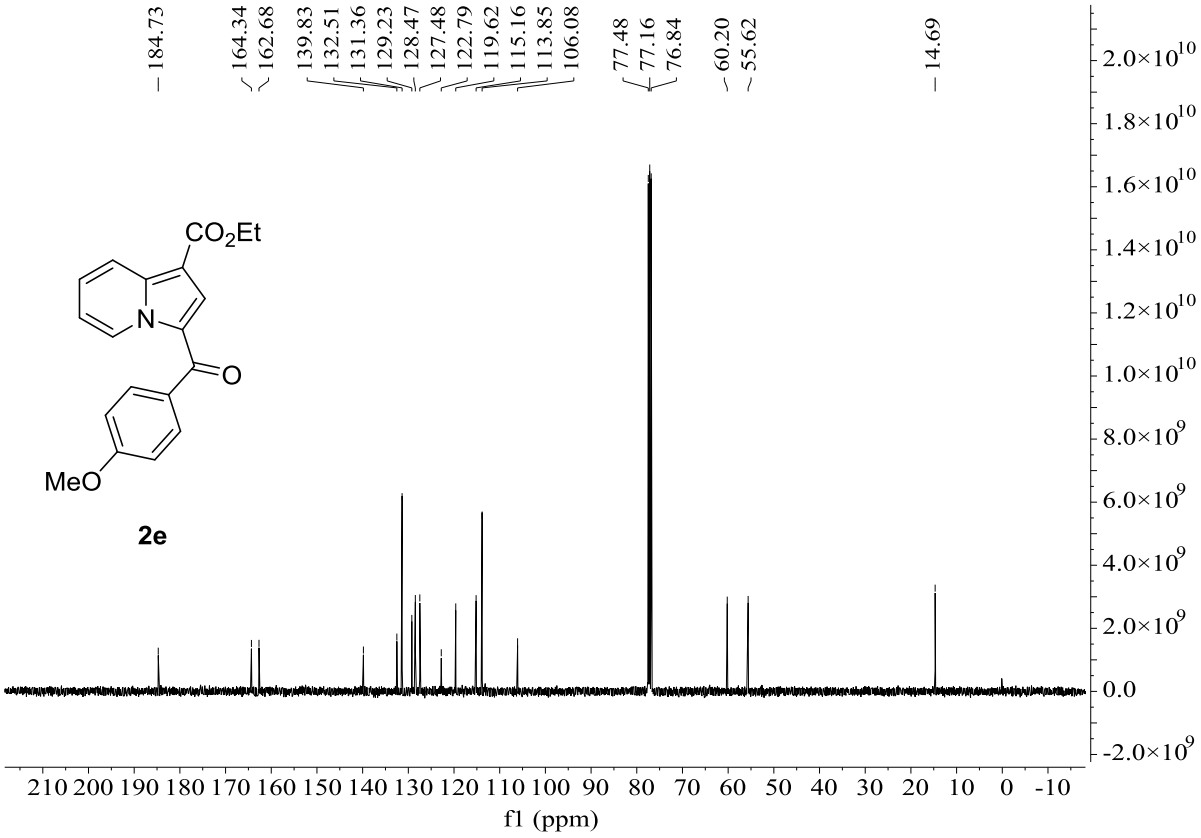
^{13}C NMR Spectrum of 2d at 25 °C ($CDCl_3$, 100 MHz)



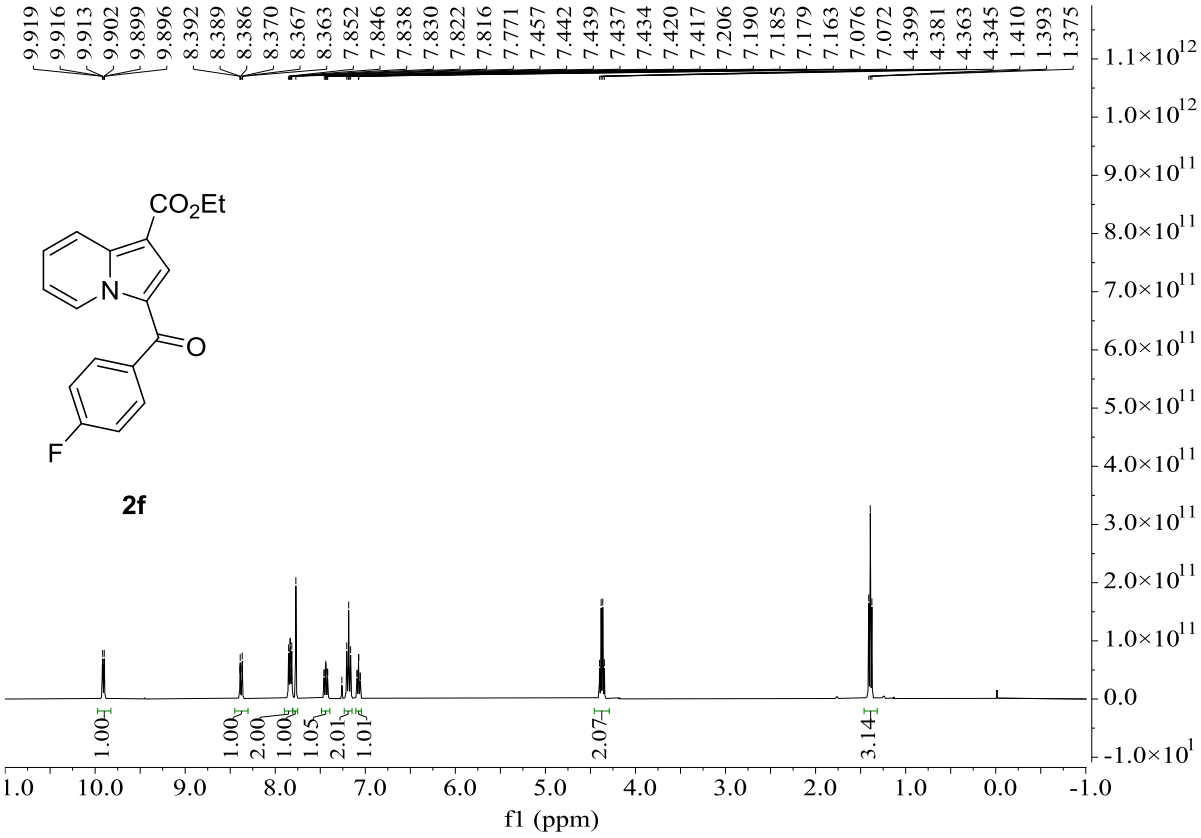
^{1}H NMR Spectrum of 2e at 25 °C ($CDCl_3$, 400 MHz)



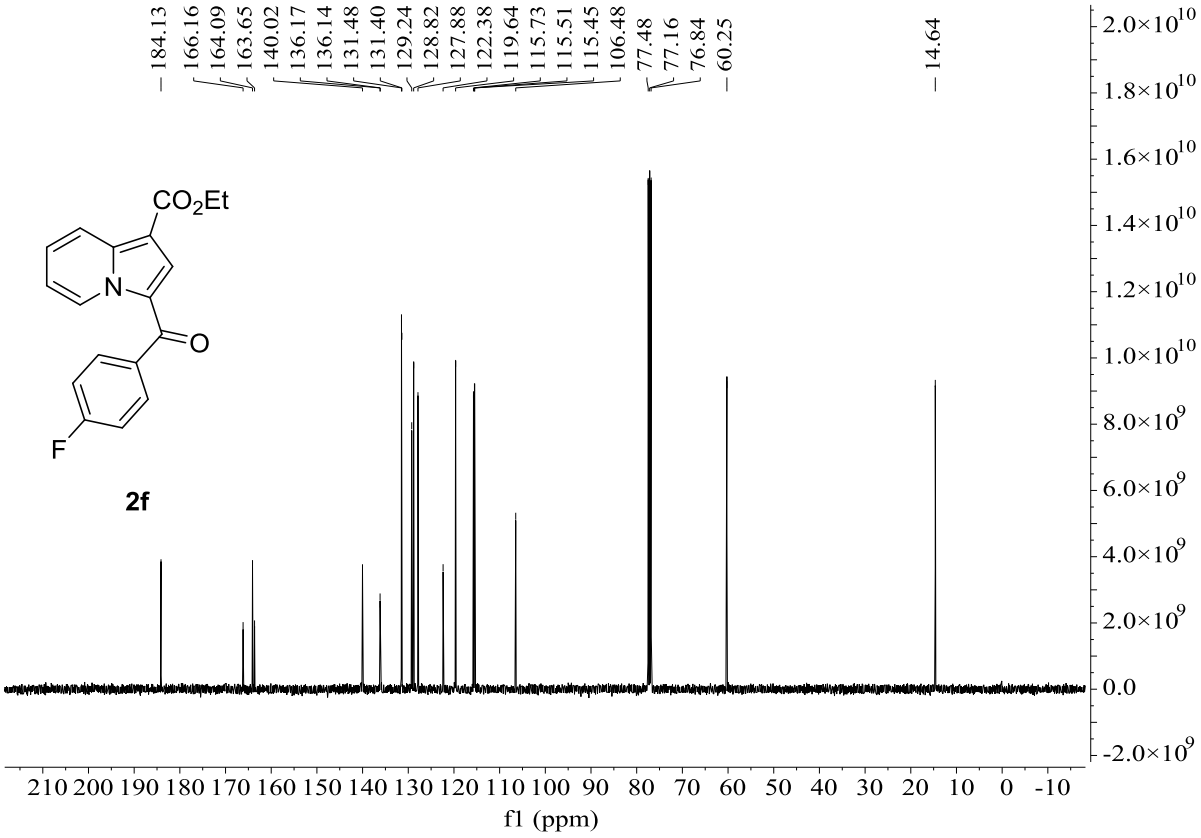
^{13}C NMR Spectrum of 2e at 25 °C ($CDCl_3$, 100 MHz)



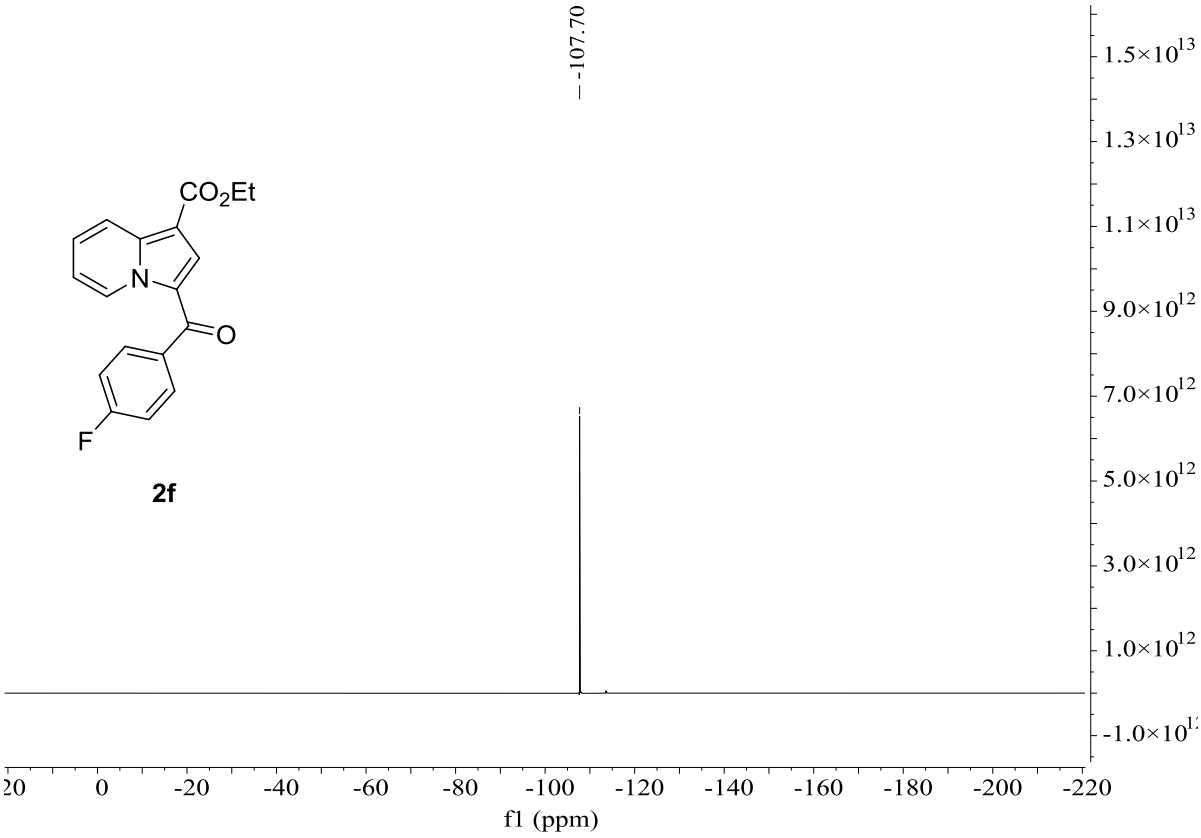
^{1}H NMR Spectrum of 2f at 25 °C ($CDCl_3$, 400 MHz)



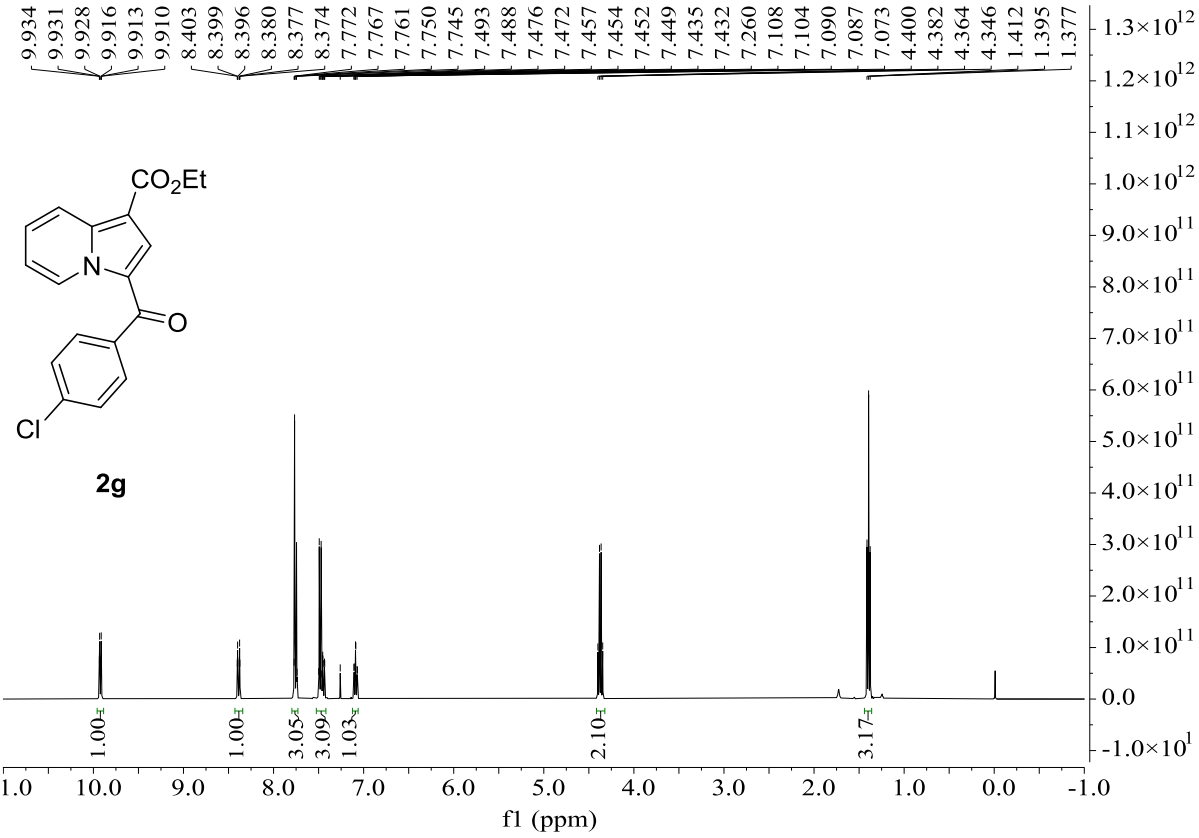
^{13}C NMR Spectrum of 2f at 25 °C ($CDCl_3$, 100 MHz)



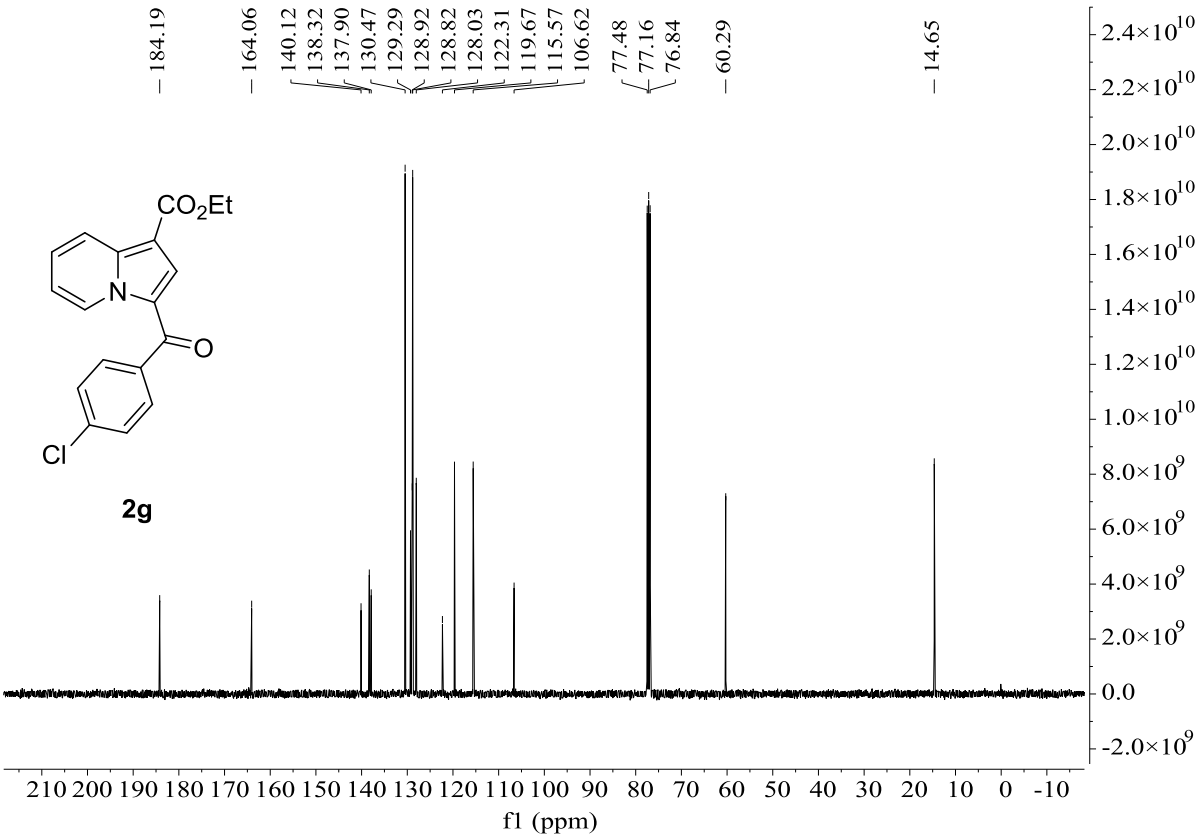
^{19}F NMR Spectrum of 2h at 25 ºC ($CDCl_3$, 376 MHz)



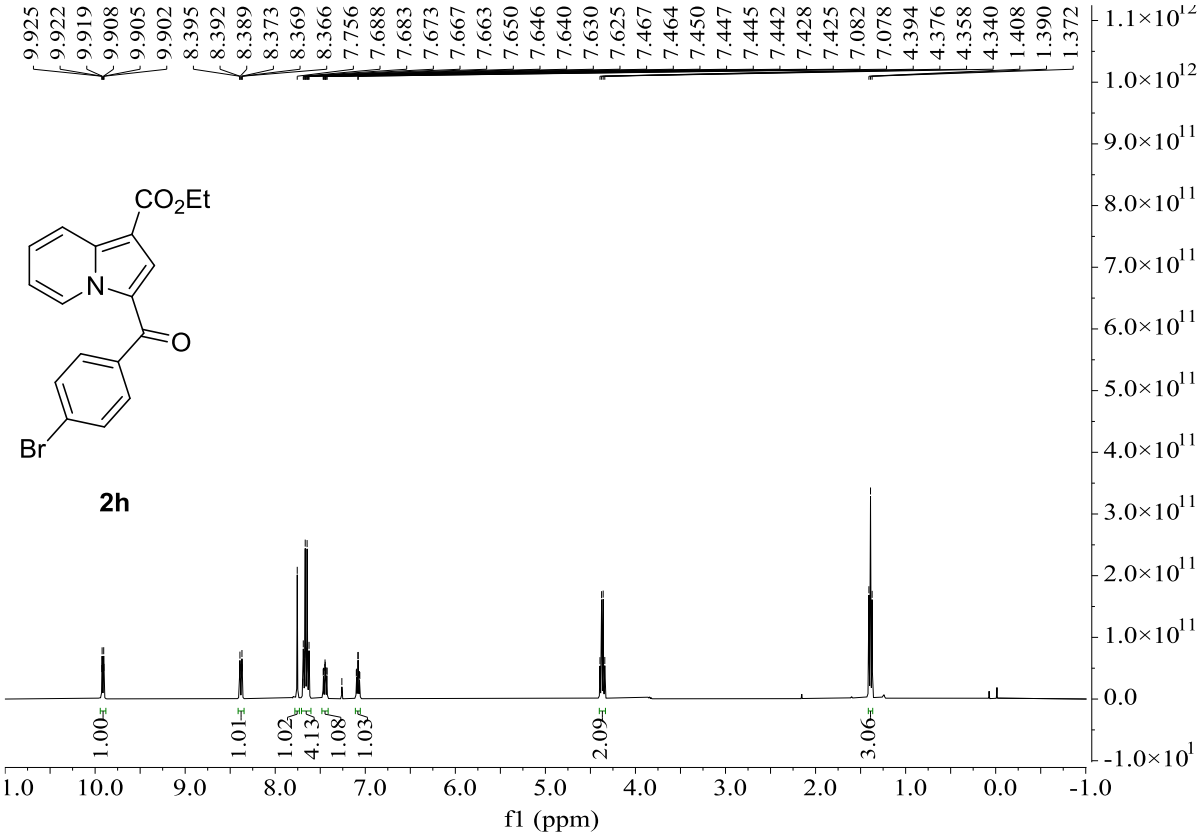
^{1}H NMR Spectrum of 2g at 25 °C ($CDCl_3$, 400 MHz)



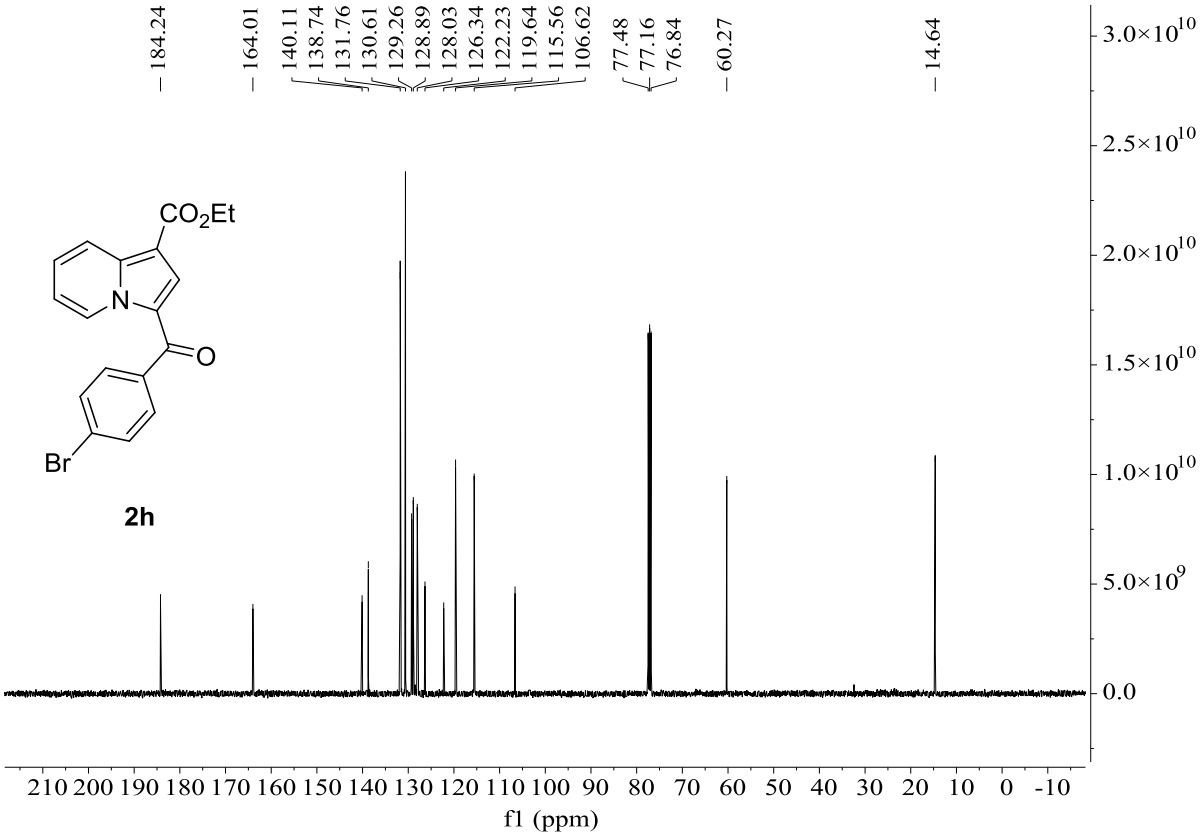
^{13}C NMR Spectrum of 2g at 25 °C ($CDCl_3$, 100 MHz)



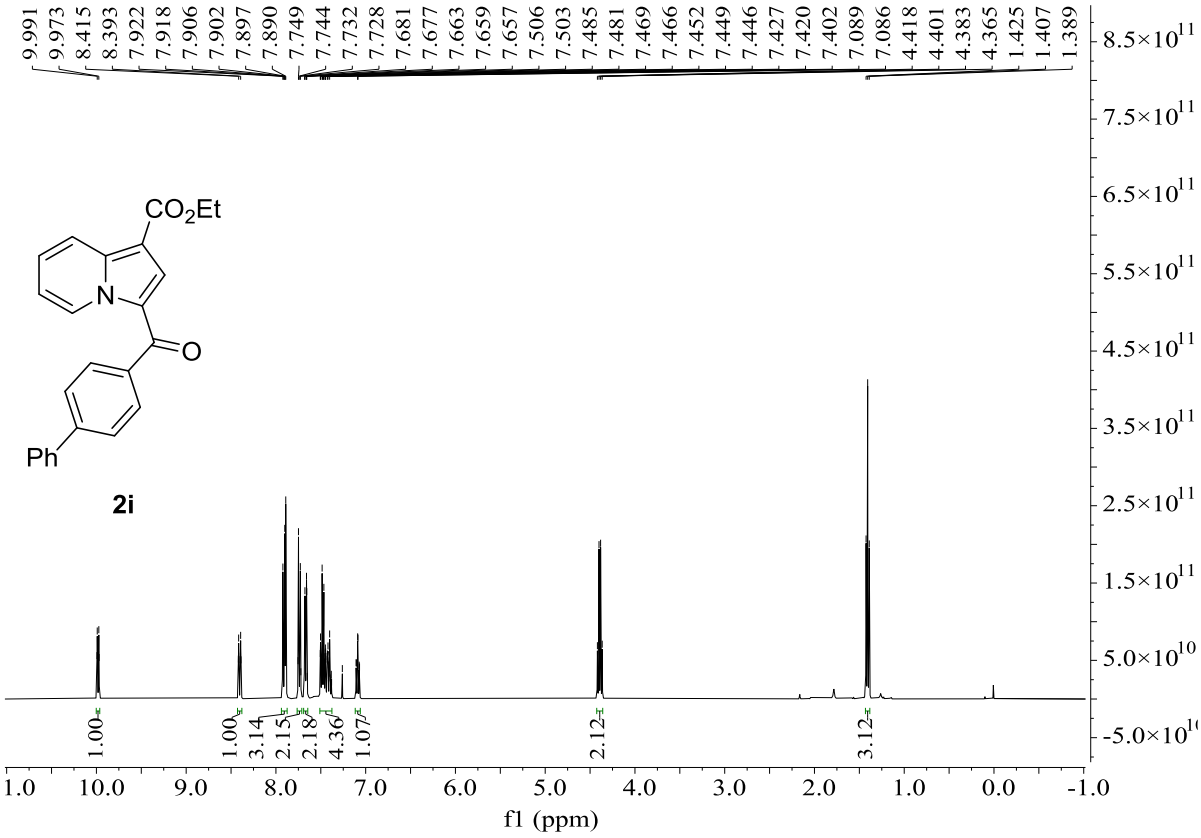
^{1}H NMR Spectrum of 2h at 25 °C ($CDCl_3$, 400 MHz)



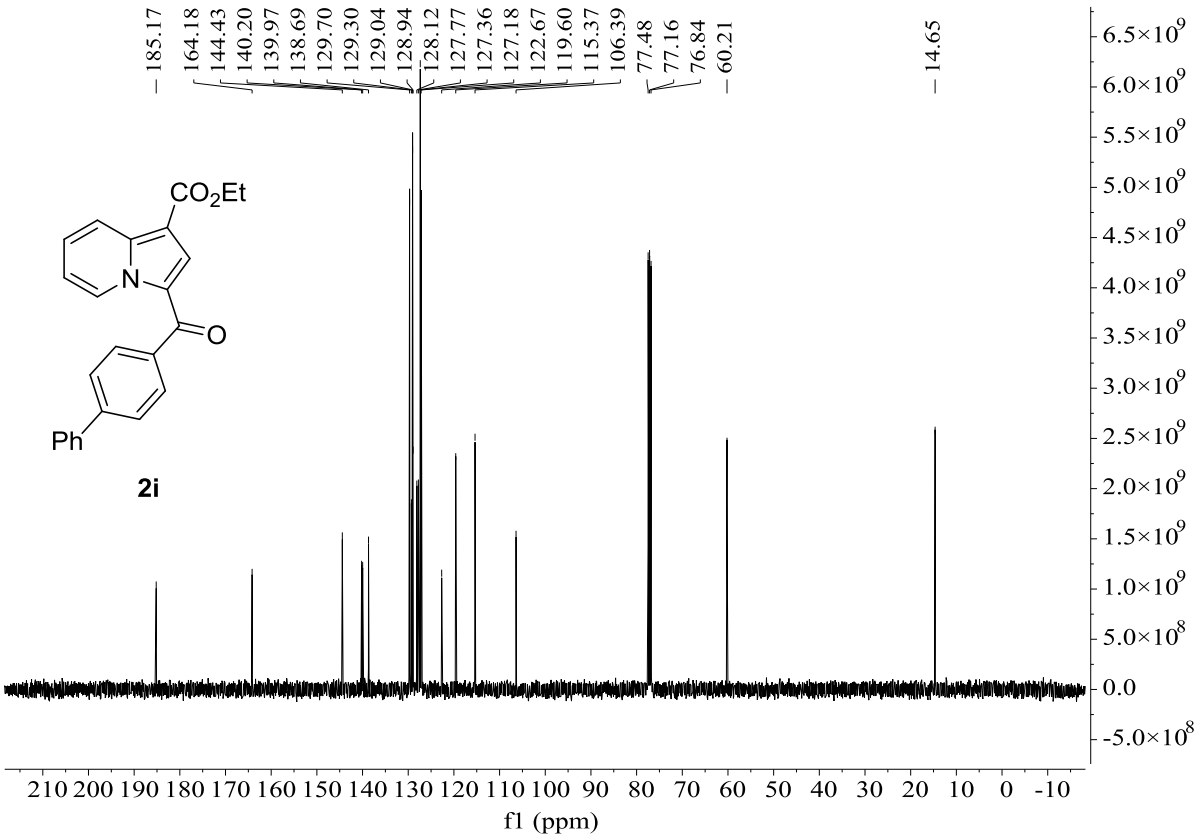
^{13}C NMR Spectrum of 2h at 25 °C ($CDCl_3$, 100 MHz)



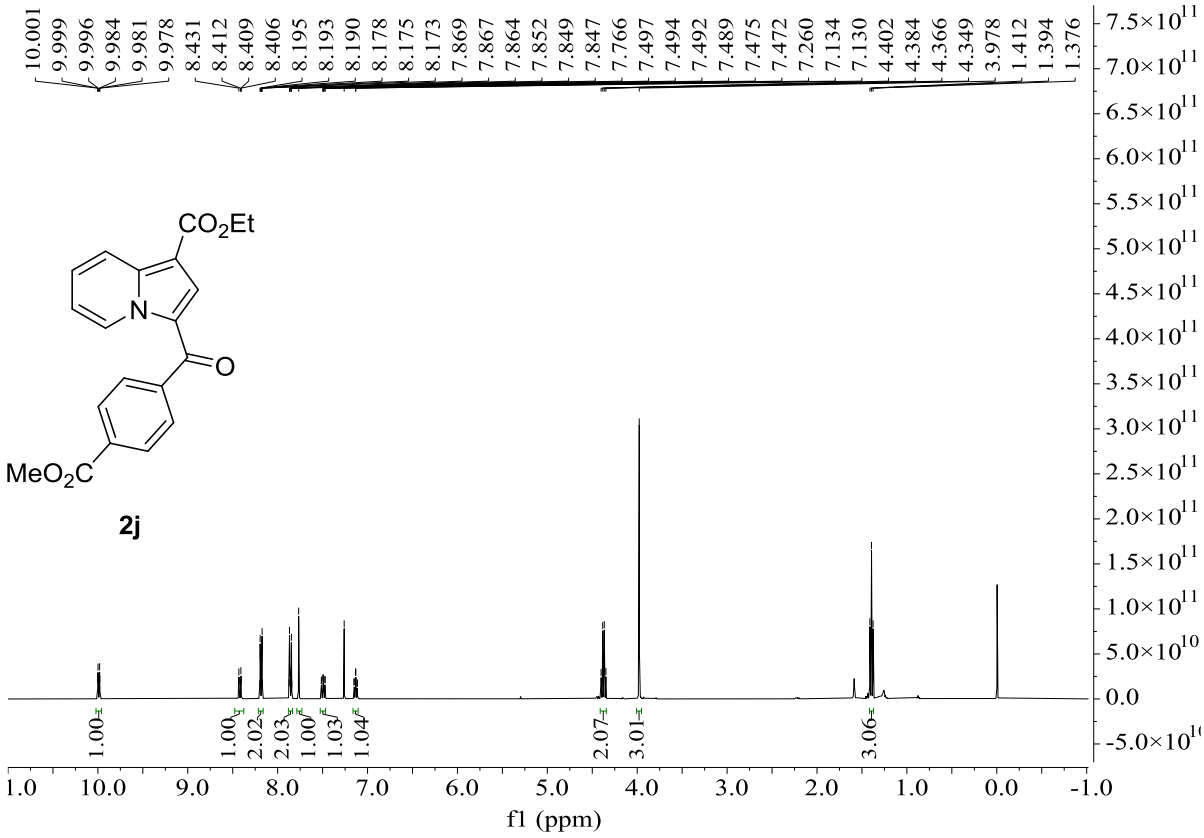
^{1}H NMR Spectrum of 2i at 25 °C ($CDCl_3$, 400 MHz)



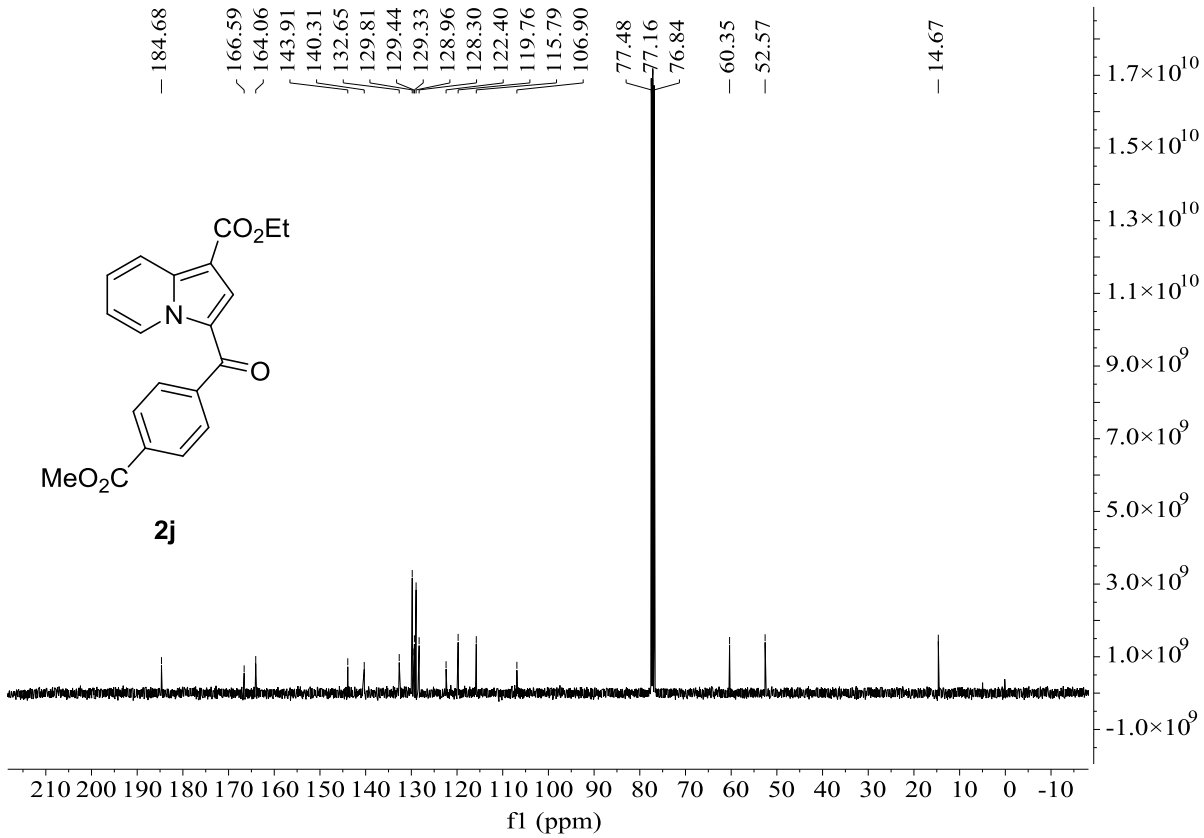
^{1}H NMR Spectrum of 2i at 25 °C ($CDCl_3$, 400 MHz)



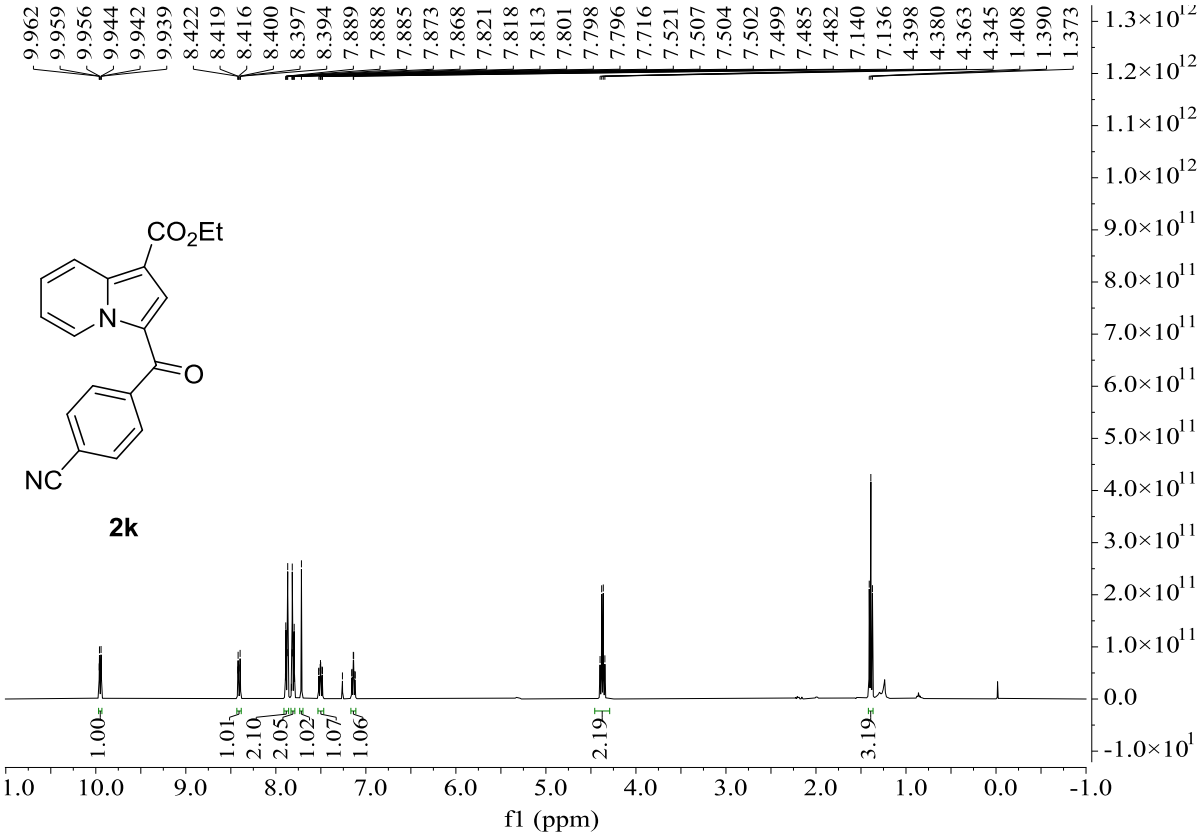
^{1}H NMR Spectrum of 2j at 25 °C ($CDCl_3$, 400 MHz)



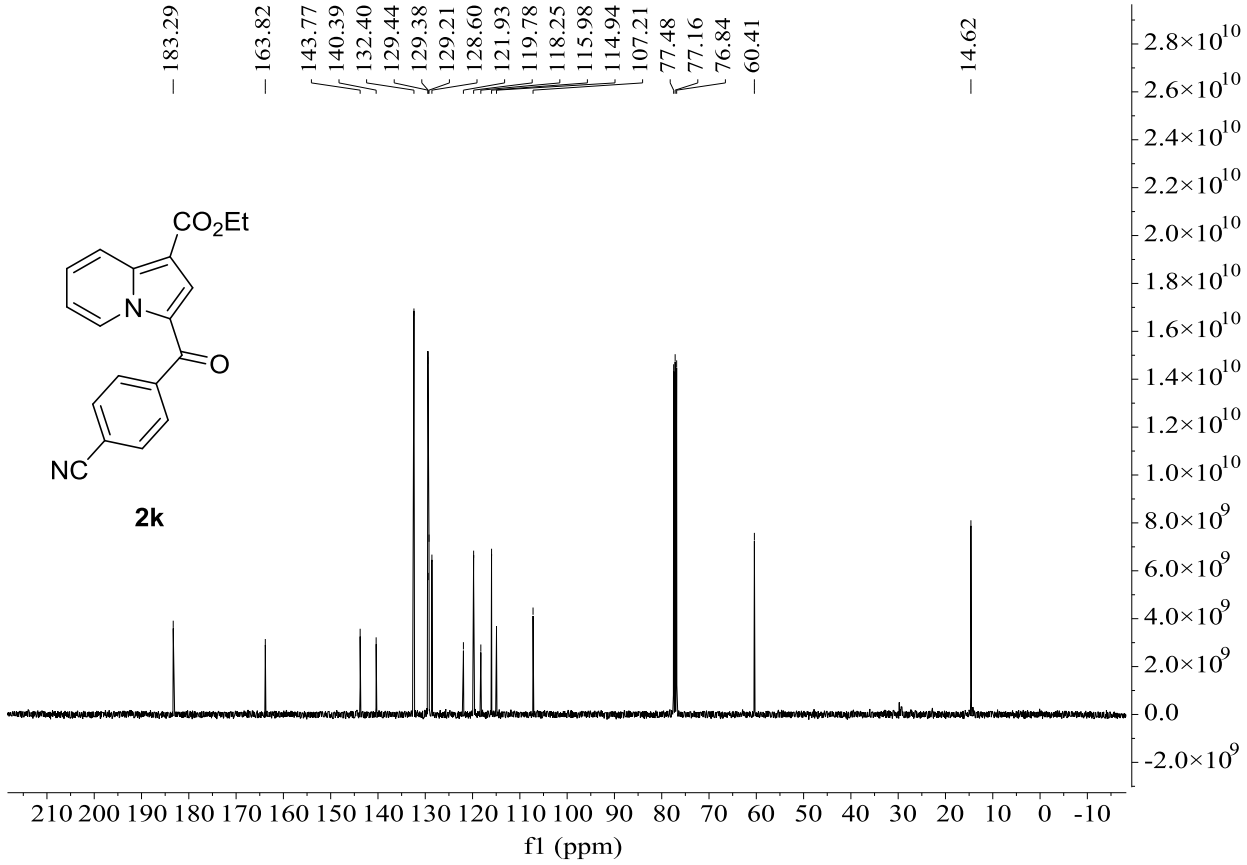
^{13}C NMR Spectrum of 2j at 25 °C ($CDCl_3$, 100 MHz)



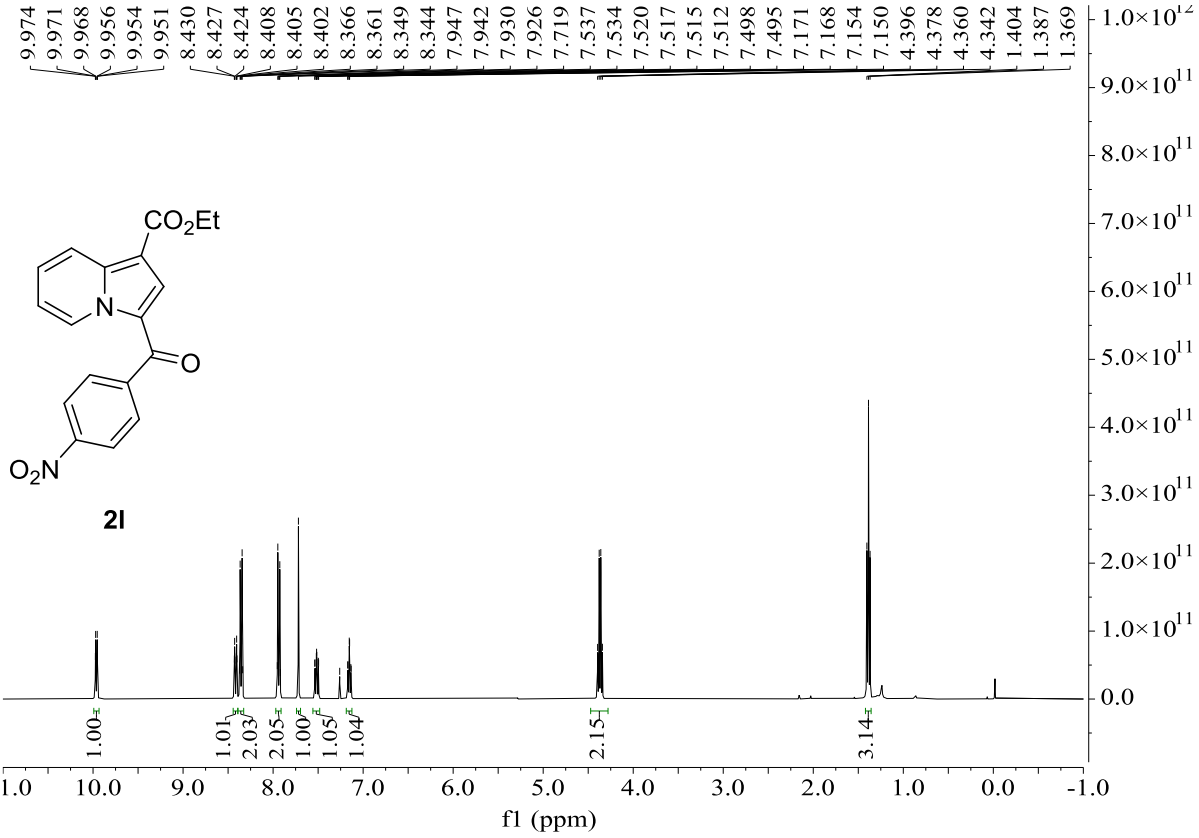
^{1}H NMR Spectrum of 2k at 25 °C ($CDCl_3$, 400 MHz)



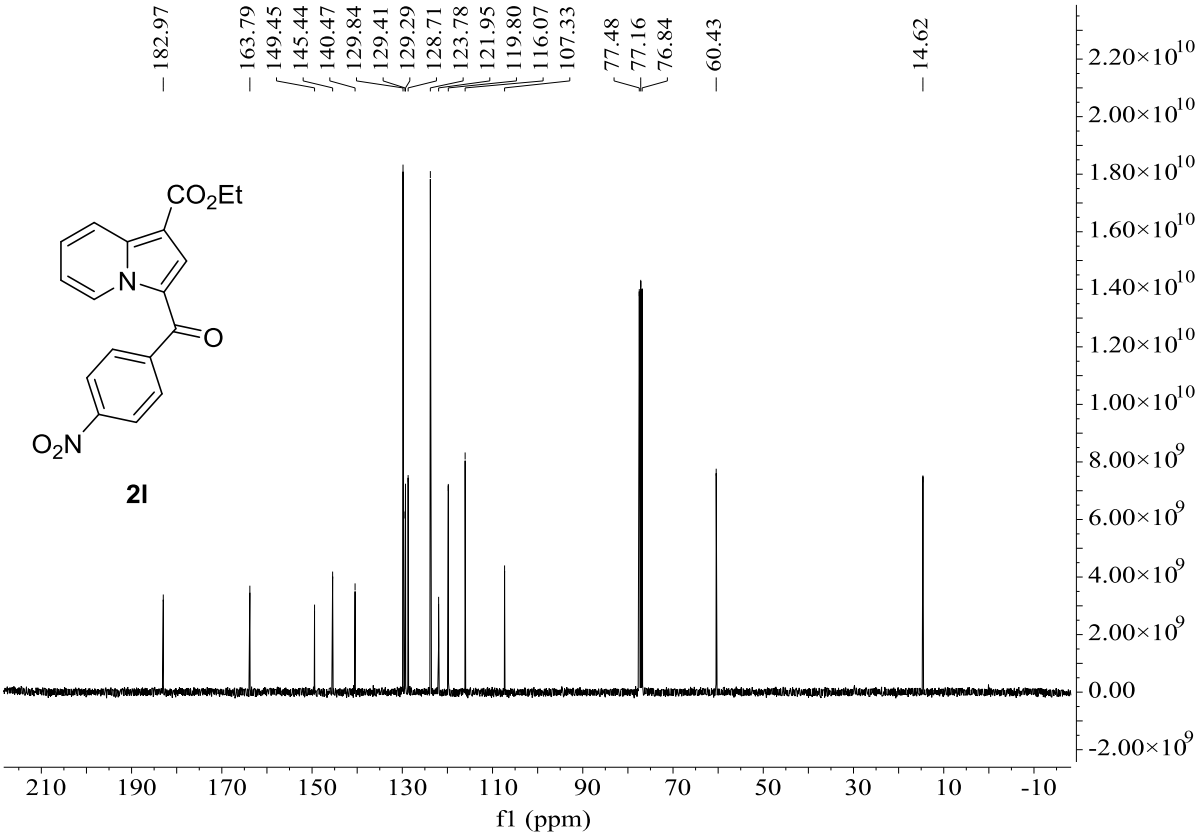
^{13}C NMR Spectrum of 2k at 25 °C ($CDCl_3$, 100 MHz)



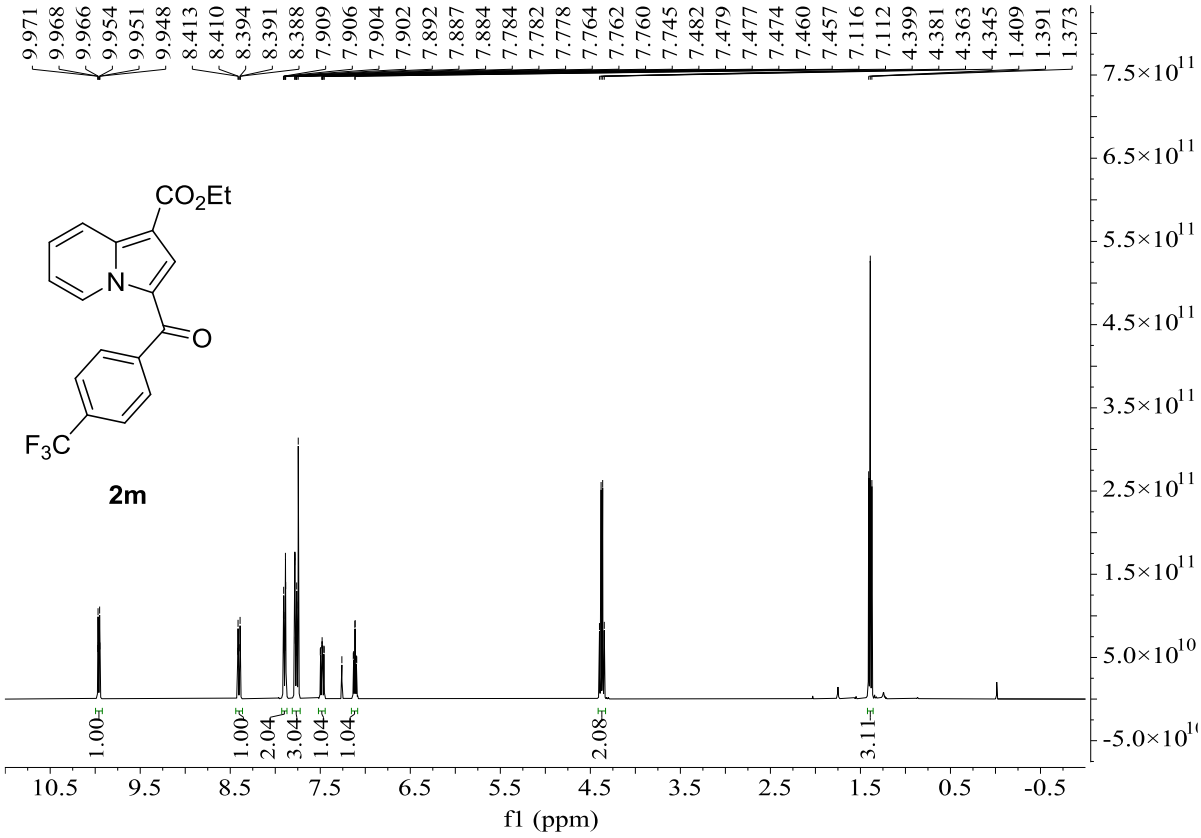
^{1}H NMR Spectrum of 2l at 25 °C ($CDCl_3$, 400 MHz)



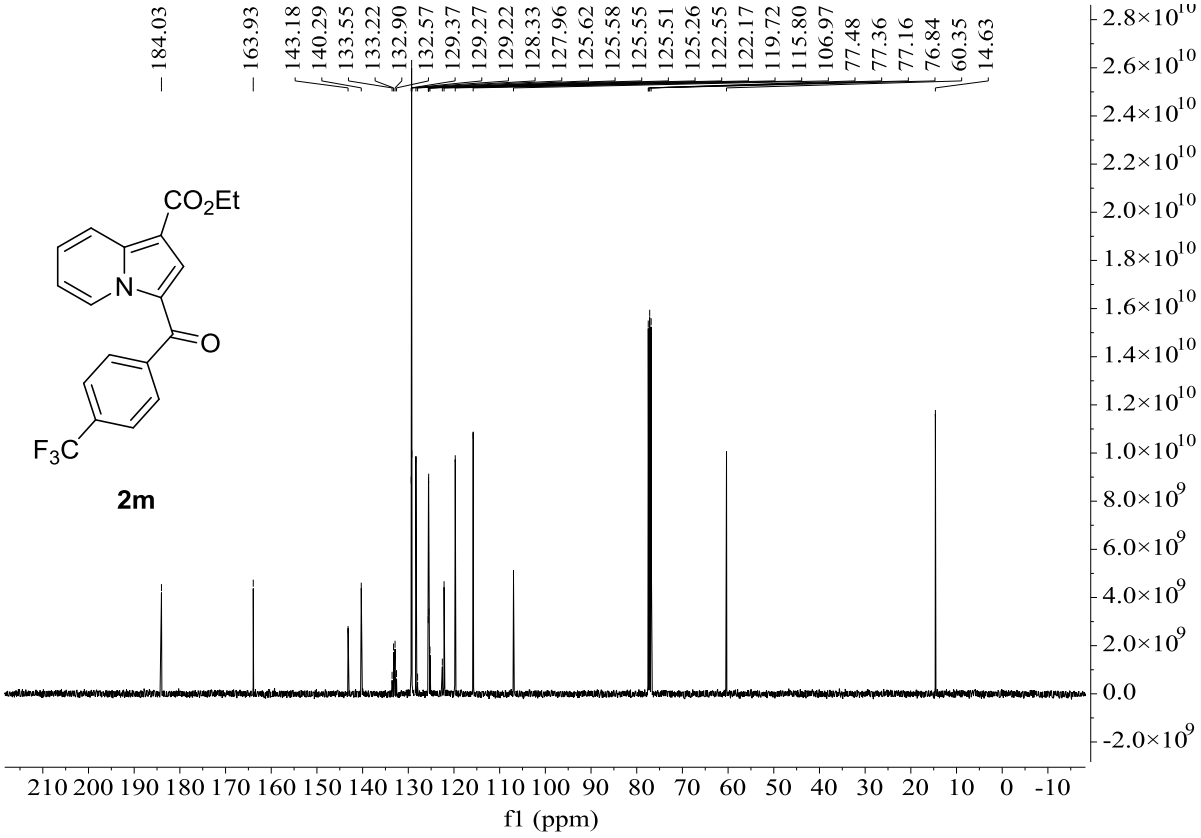
^{13}C NMR Spectrum of 2l at 25 °C ($CDCl_3$, 100 MHz)



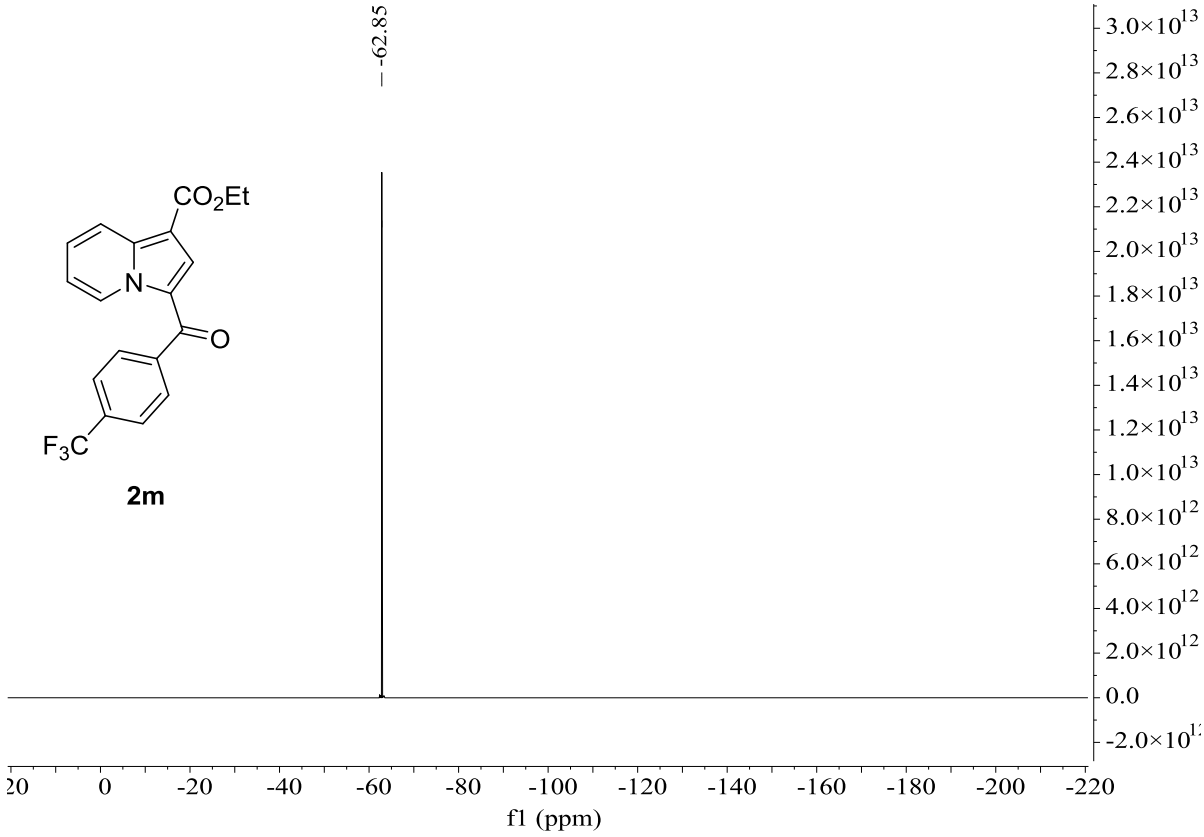
^{1}H NMR Spectrum of 2m at 25 °C ($CDCl_3$, 400 MHz)



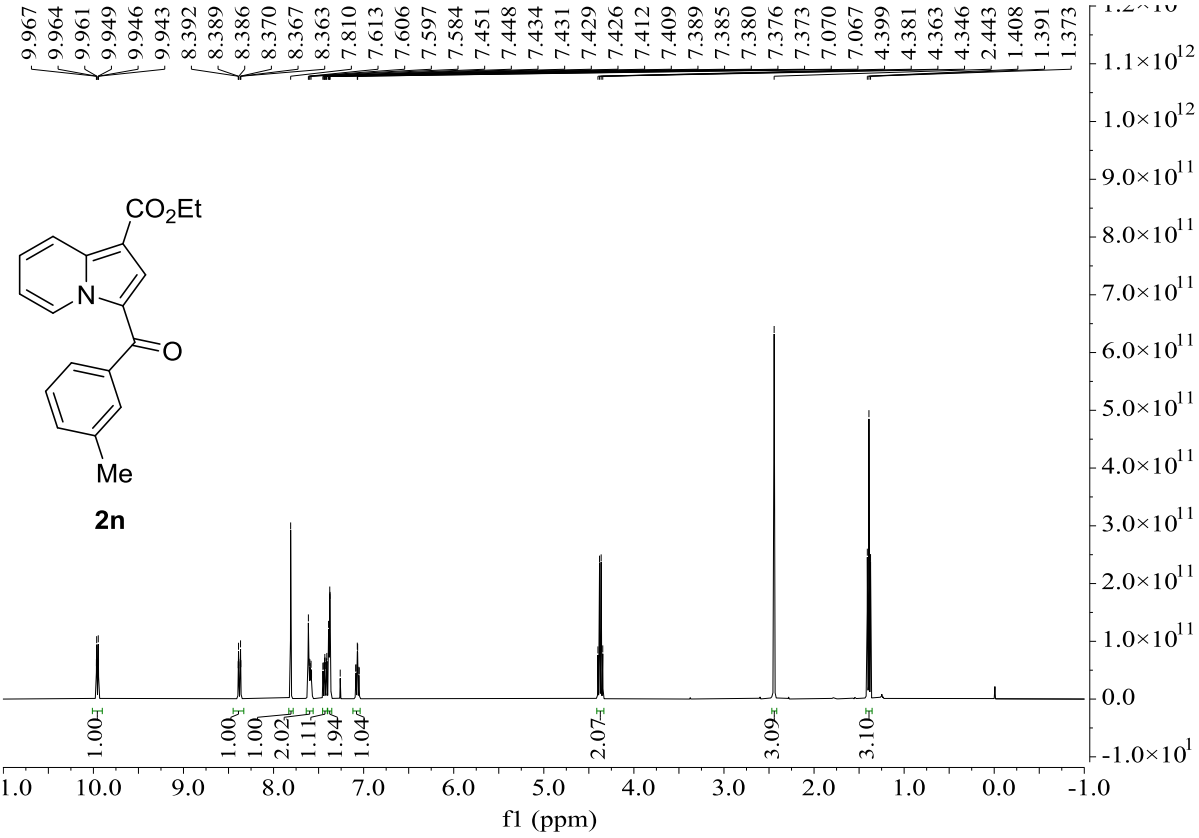
^{13}C NMR Spectrum of 2m at 25 °C ($CDCl_3$, 100 MHz)



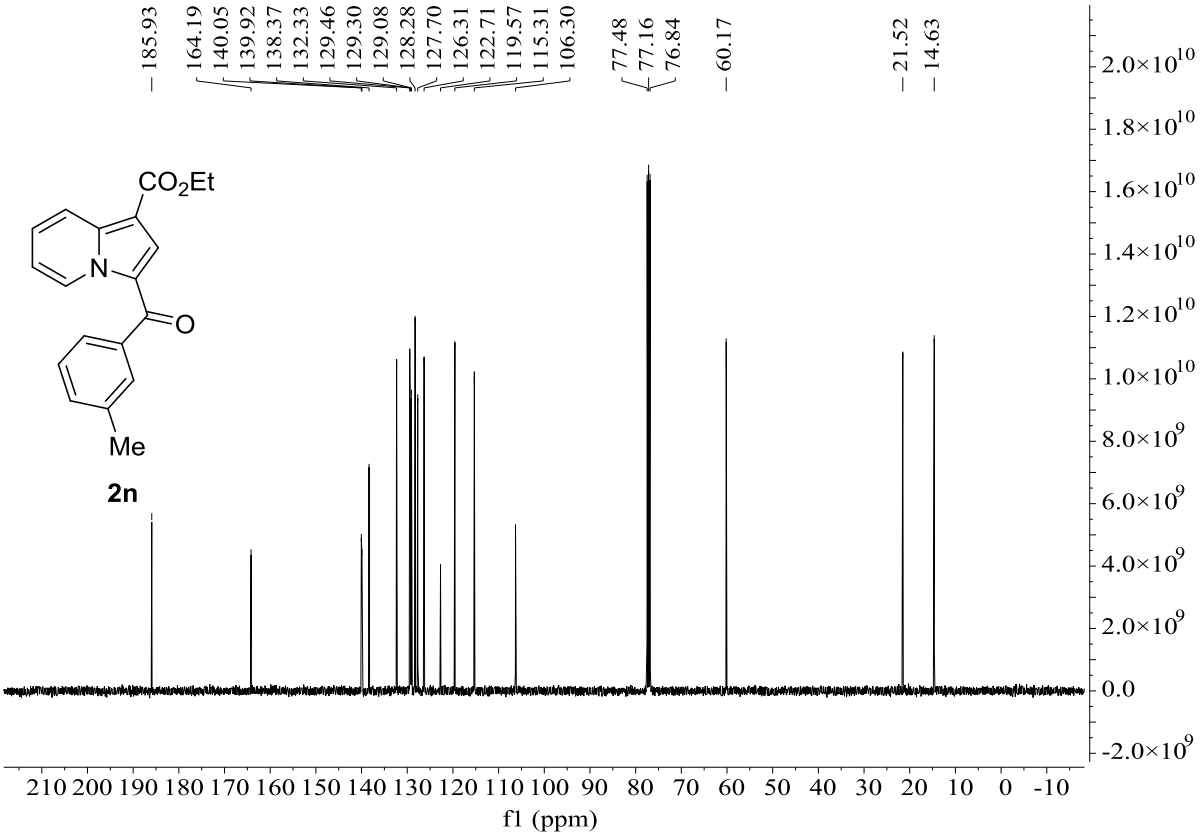
^{19}F NMR Spectrum of 2m at 25 °C ($CDCl_3$, 376 MHz)



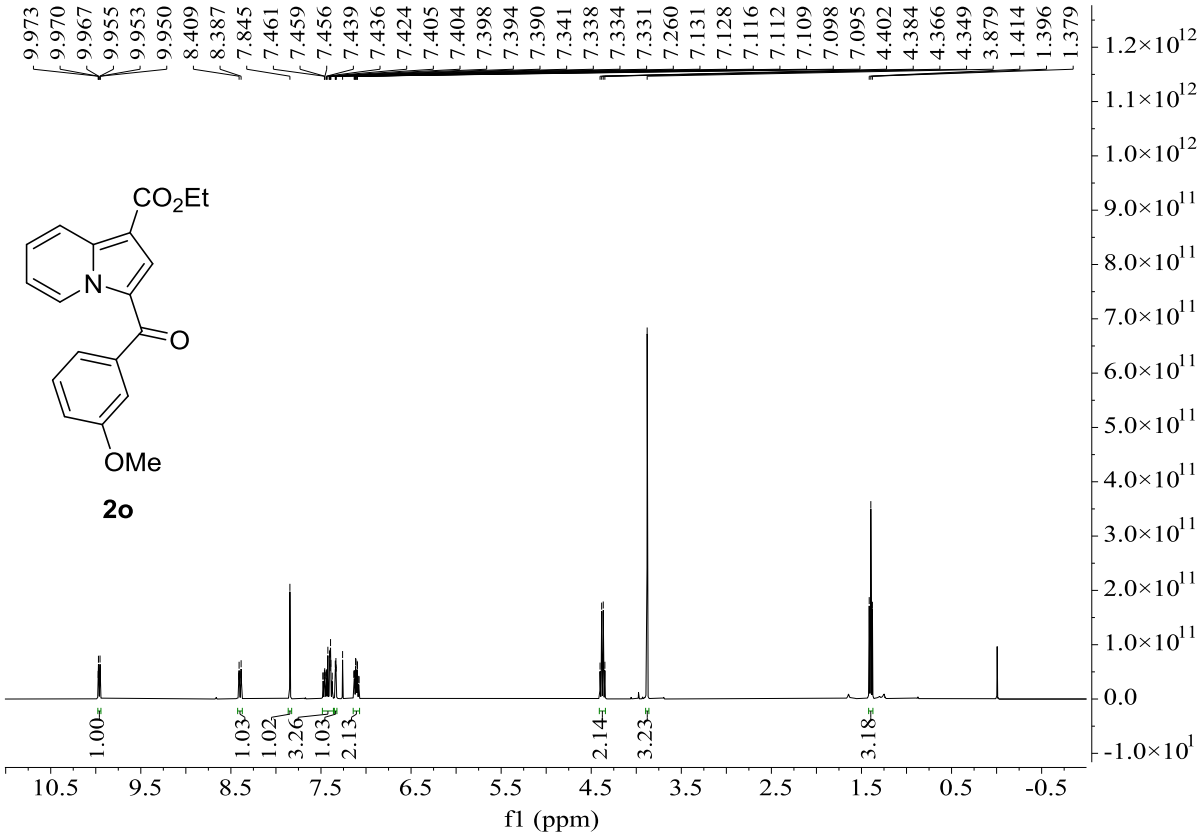
^{1}H NMR Spectrum of 2n at 25 °C ($CDCl_3$, 400 MHz)



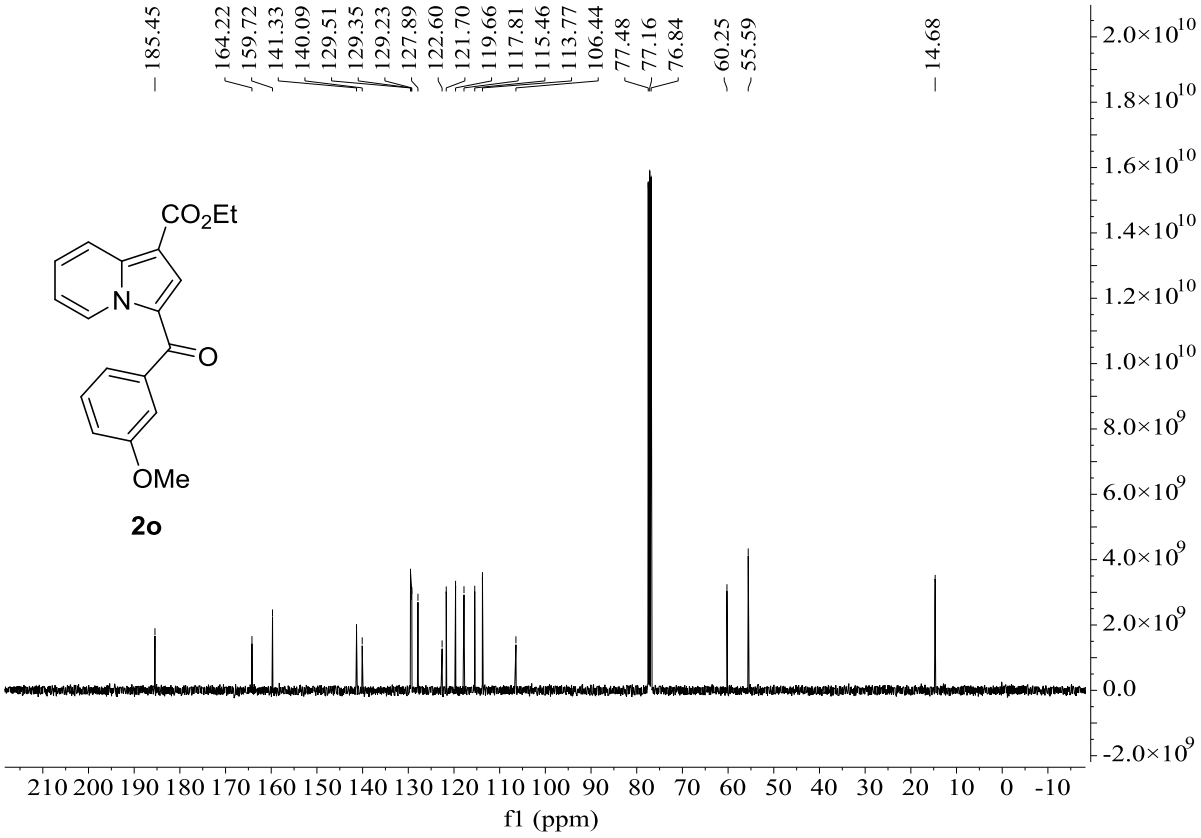
^{13}C NMR Spectrum of 2n at 25 °C ($CDCl_3$, 100 MHz)



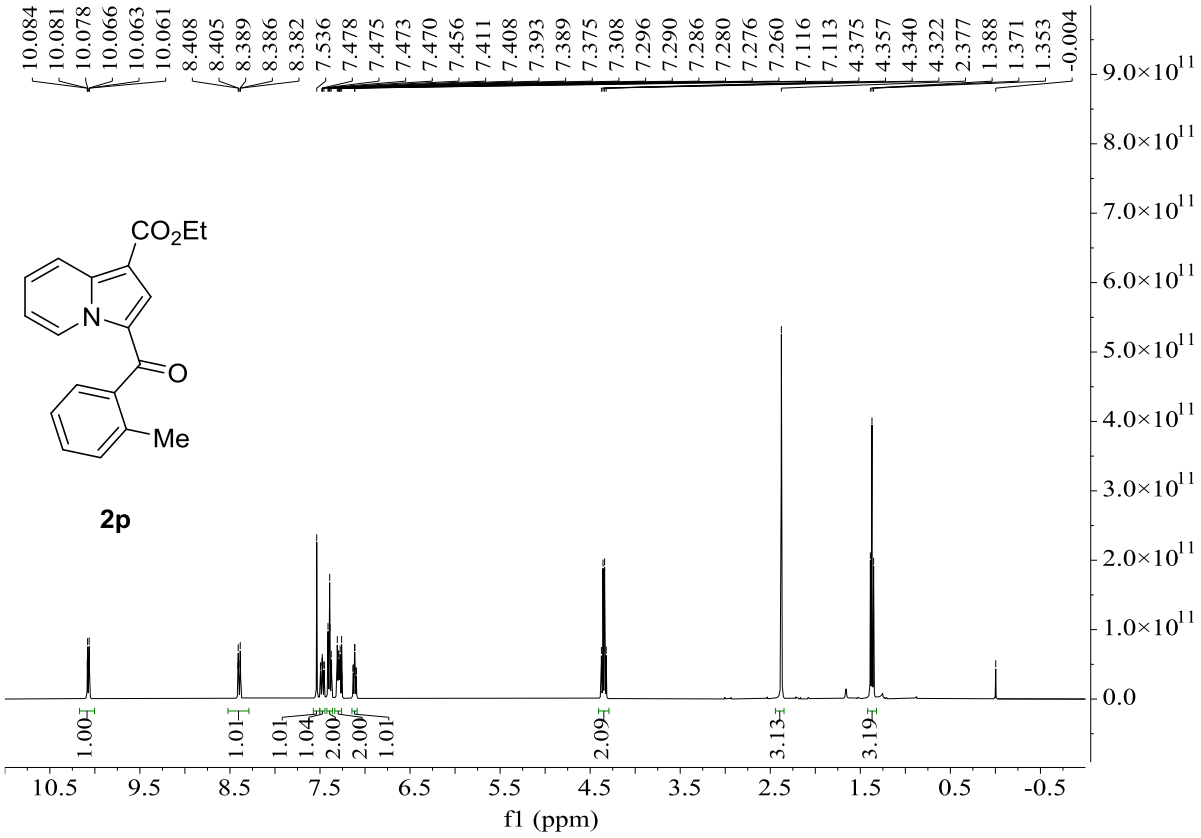
^{1}H NMR Spectrum of 2o at 25 °C ($CDCl_3$, 400 MHz)



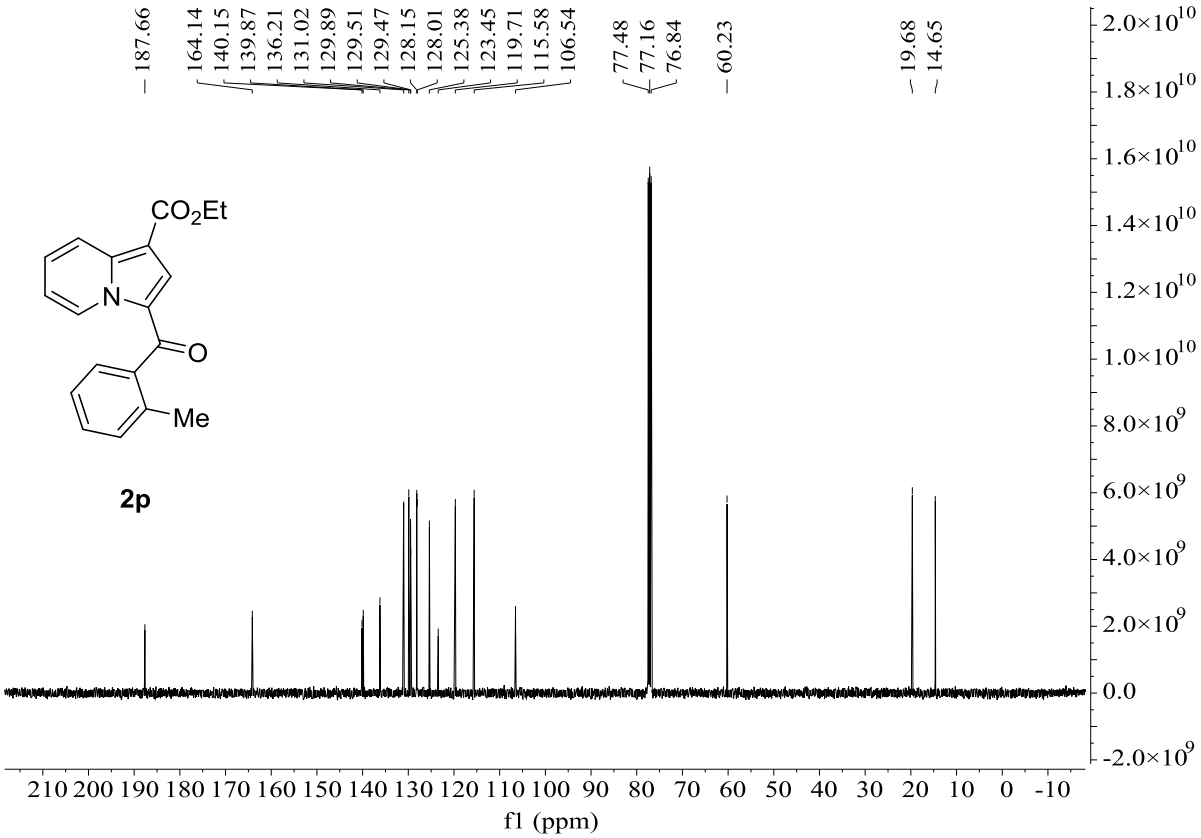
^{13}C NMR Spectrum of 2o at 25 °C ($CDCl_3$, 100 MHz)



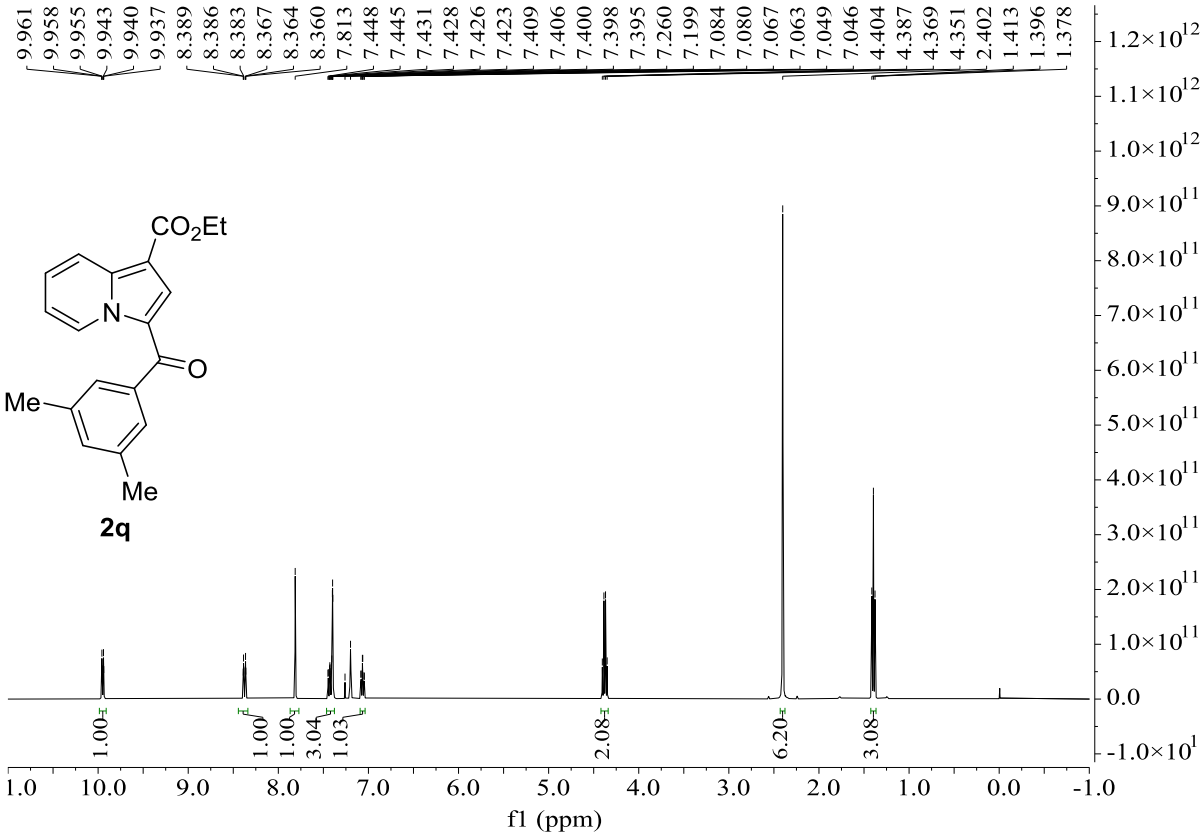
^{1}H NMR Spectrum of 2p at 25 °C ($CDCl_3$, 400 MHz)



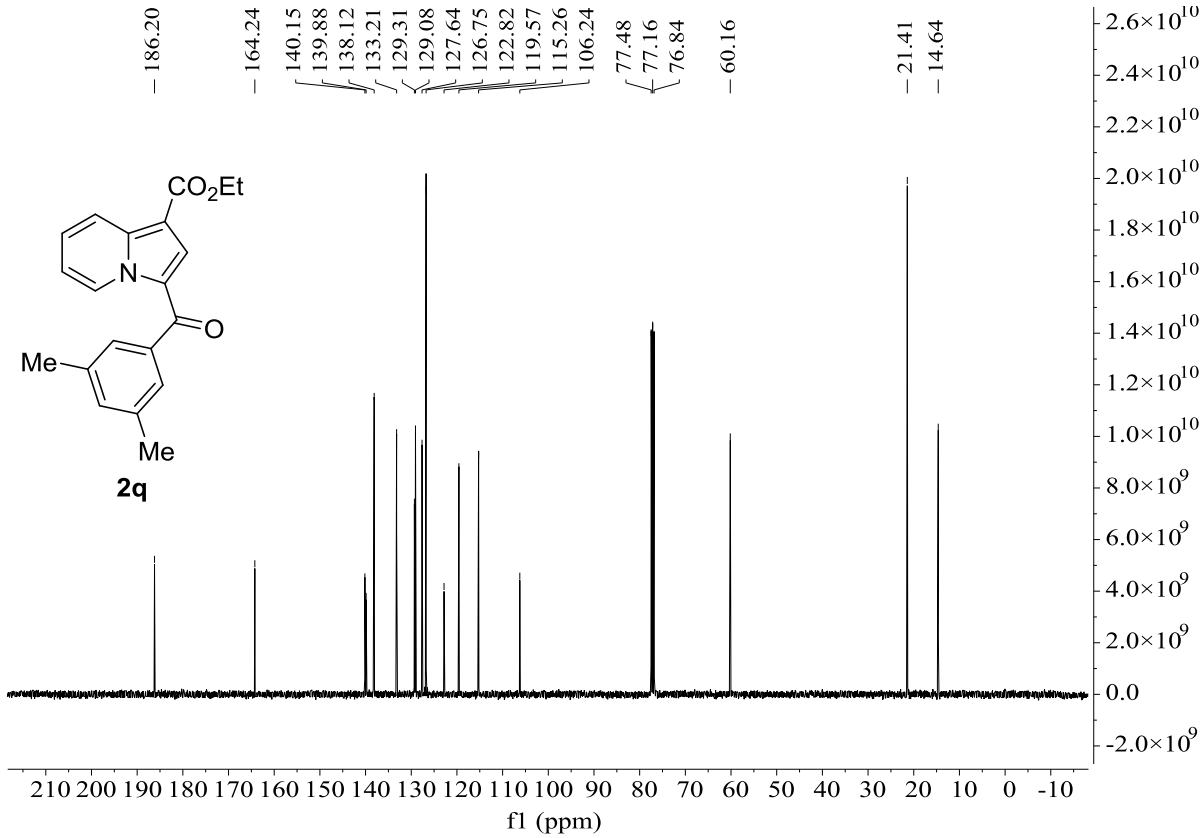
^{13}C NMR Spectrum of 2p at 25 °C ($CDCl_3$, 100 MHz)



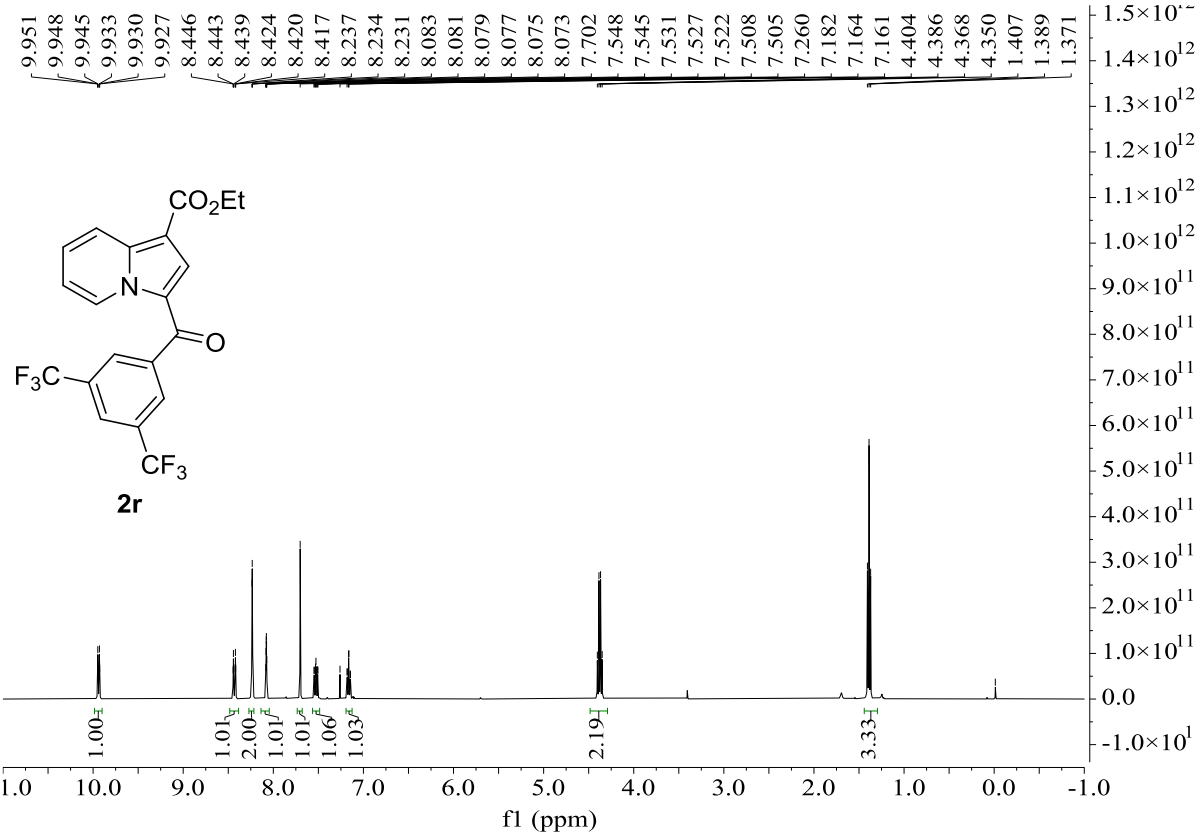
^{1}H NMR Spectrum of 2q at 25 °C ($CDCl_3$, 400 MHz)



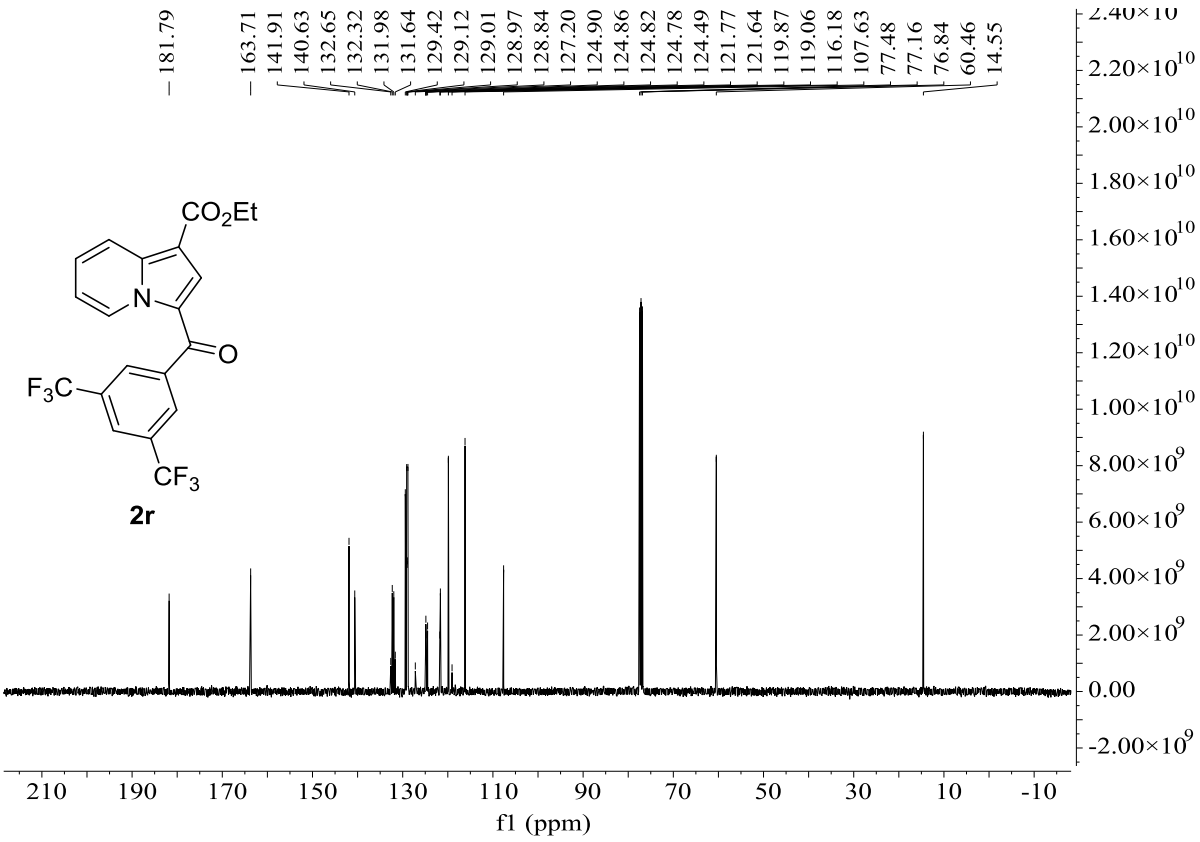
^{13}C NMR Spectrum of 2q at 25 °C ($CDCl_3$, 100 MHz)



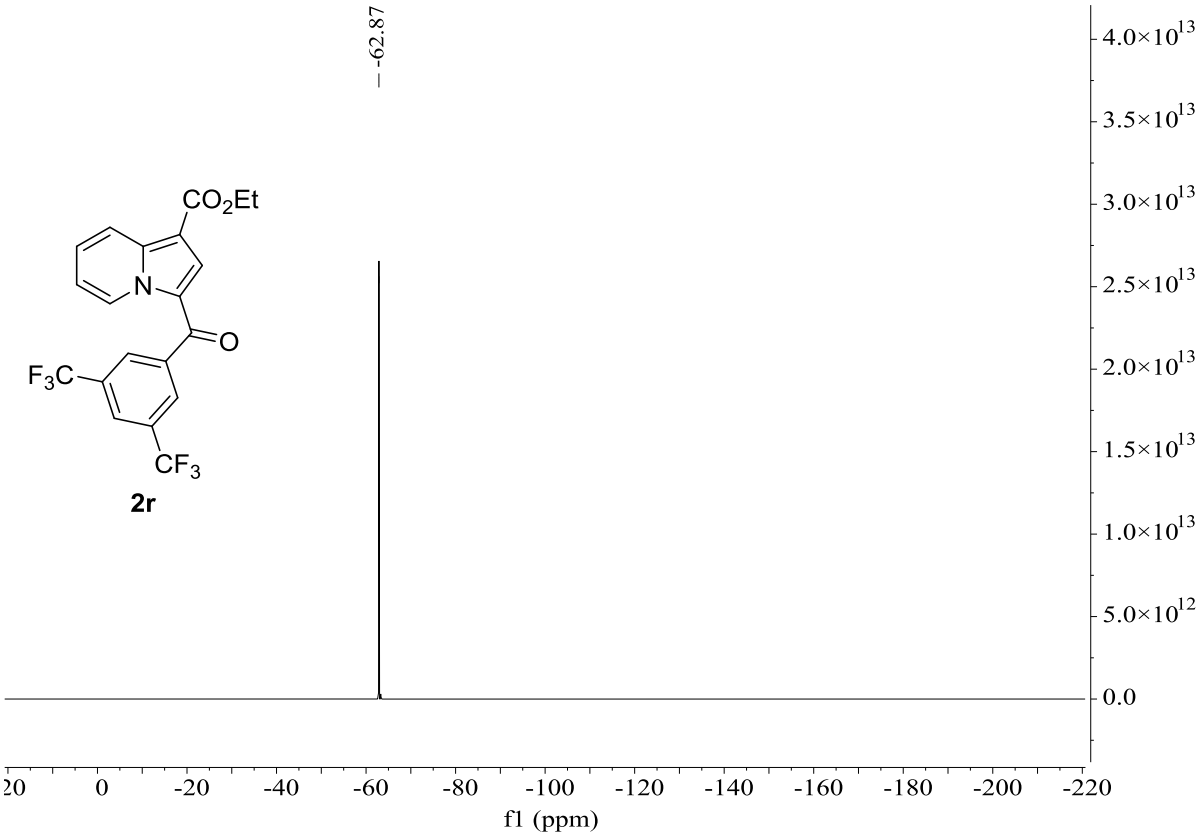
^{1}H NMR Spectrum of 2r at 25 °C ($CDCl_3$, 400 MHz)



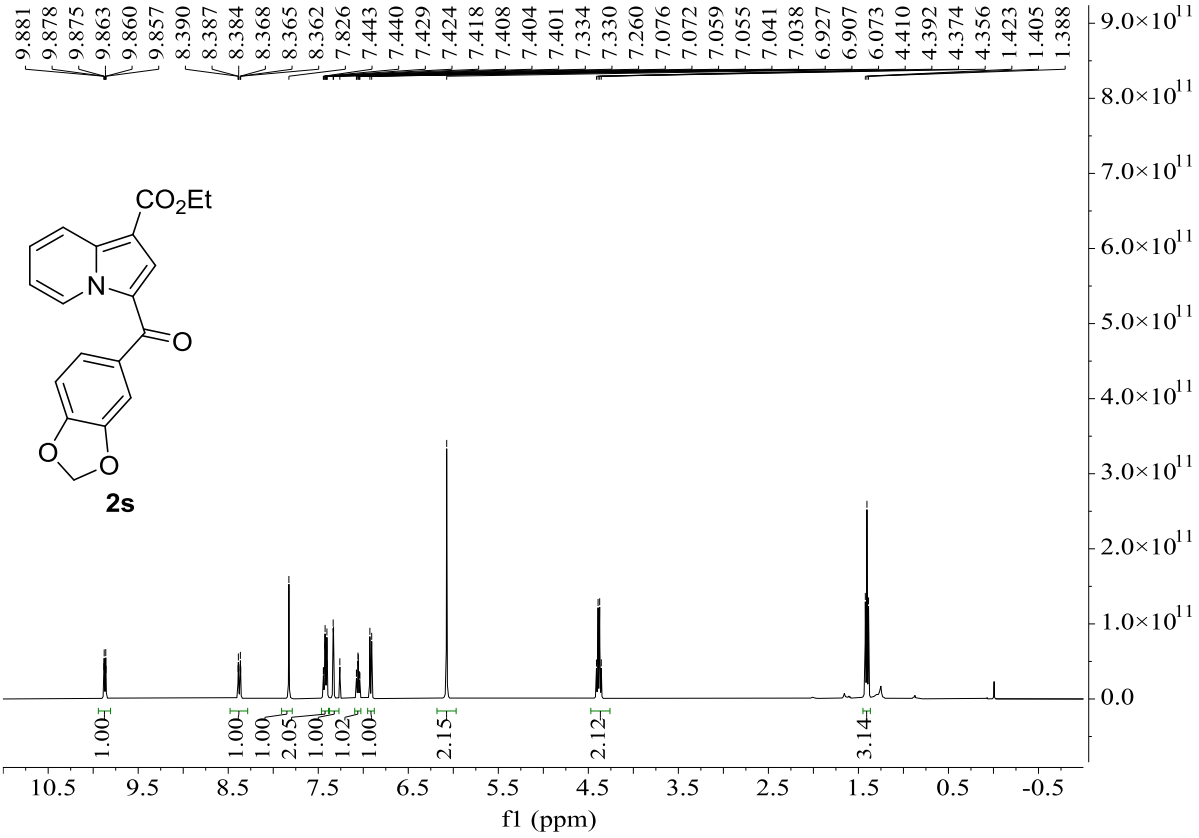
^{13}C NMR Spectrum of 2r at 25 °C ($CDCl_3$, 100 MHz)



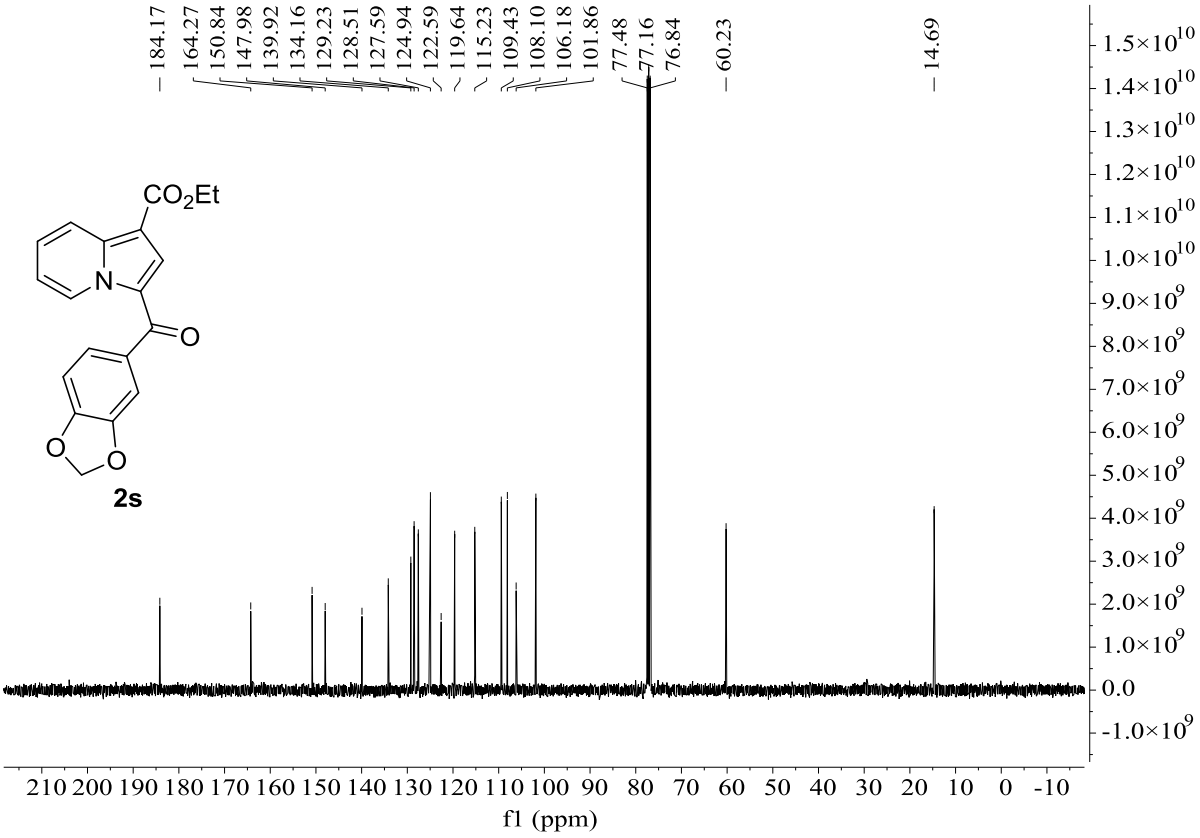
^{19}F NMR Spectrum of 2r at 25 °C ($CDCl_3$, 376 MHz)



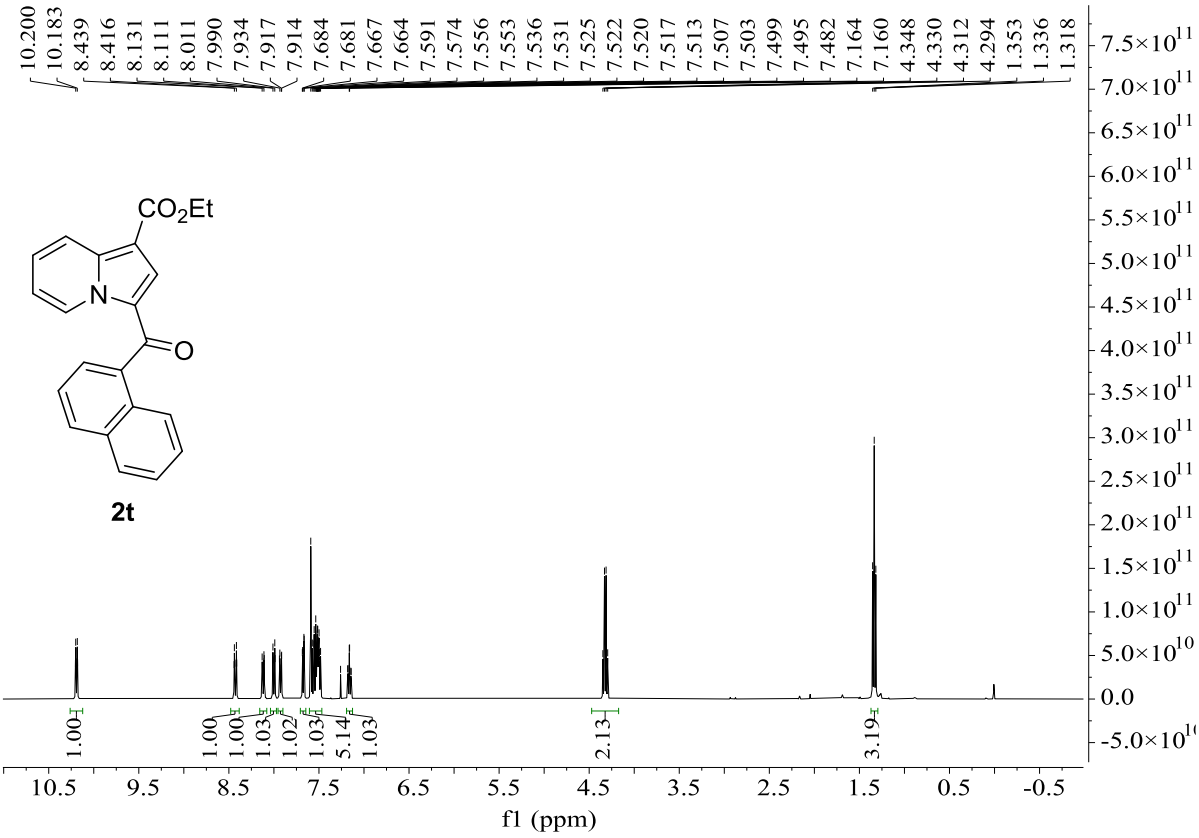
^{1}H NMR Spectrum of 2s at 25 °C ($CDCl_3$, 400 MHz)



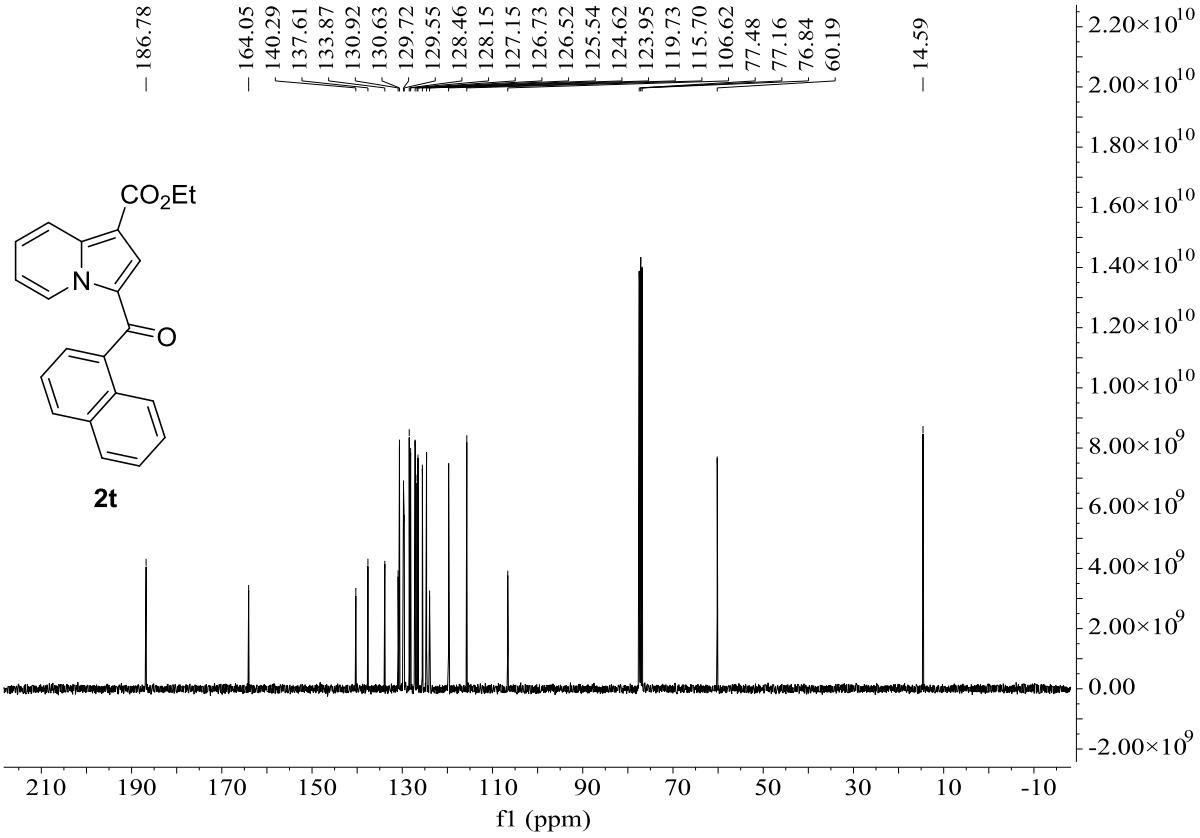
^{13}C NMR Spectrum of 2s at 25 °C ($CDCl_3$, 100 MHz)



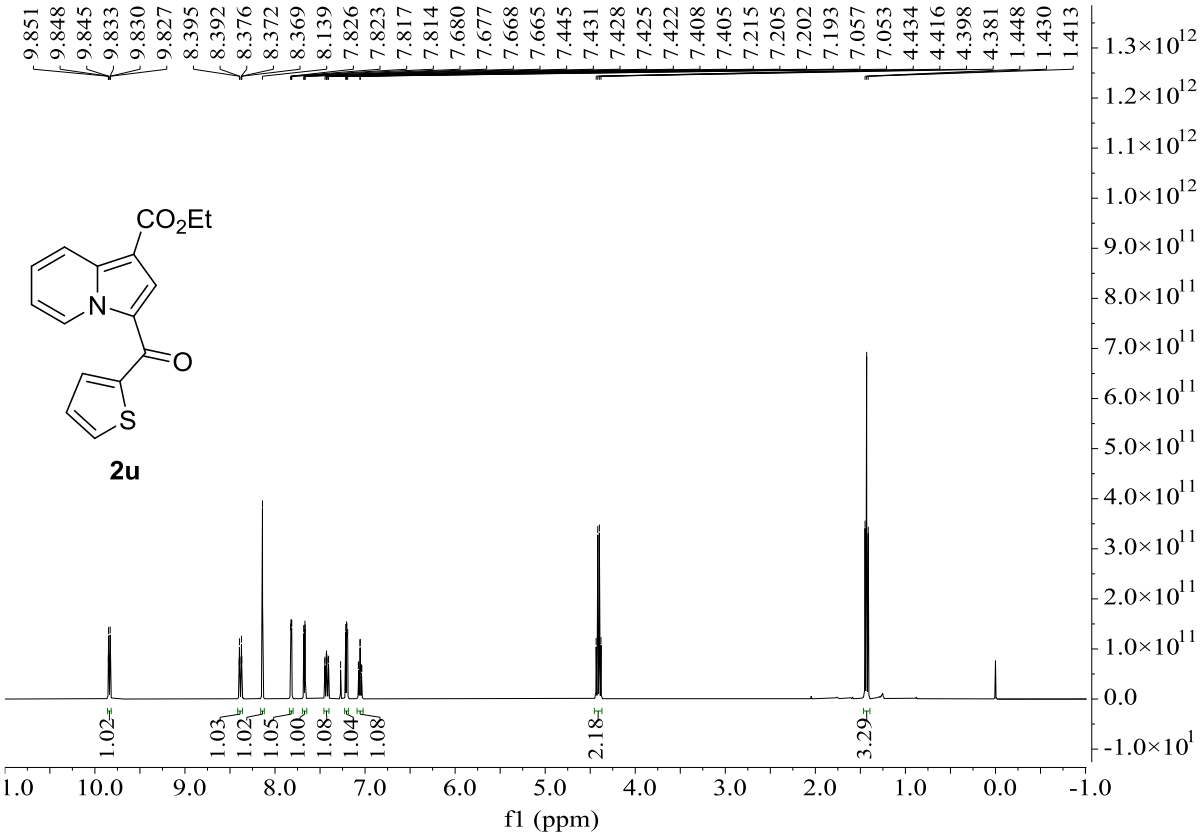
^{1}H NMR Spectrum of 2t at 25 °C ($CDCl_3$, 400 MHz)



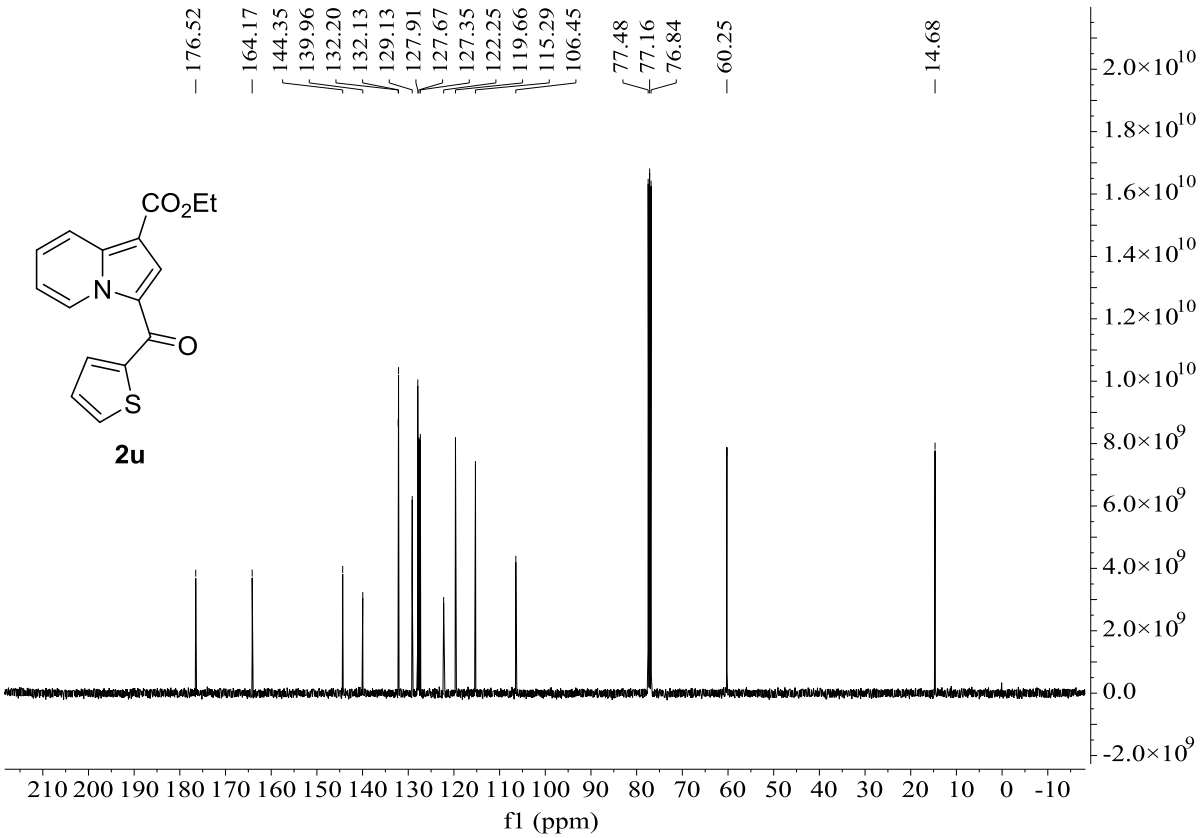
^{13}C NMR Spectrum of 2t at 25 °C ($CDCl_3$, 100 MHz)



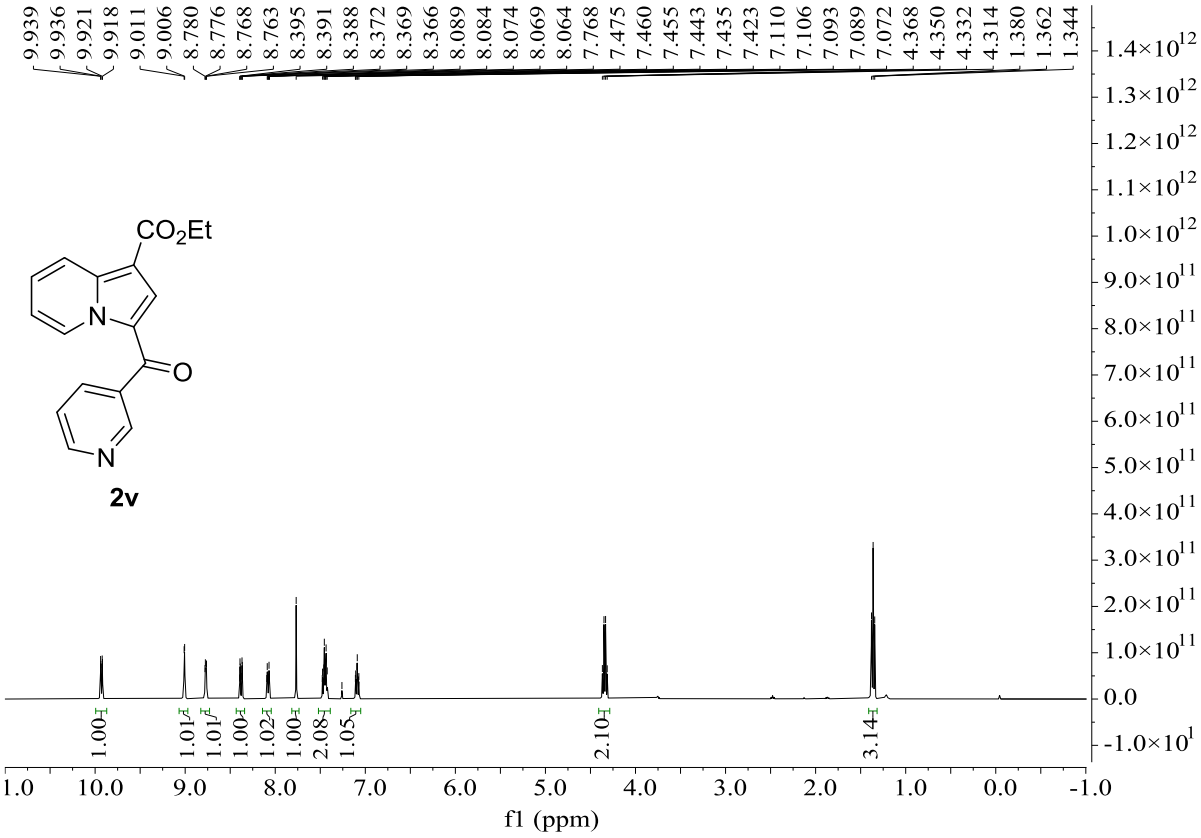
^{1}H NMR Spectrum of 2u at 25 °C ($CDCl_3$, 400 MHz)



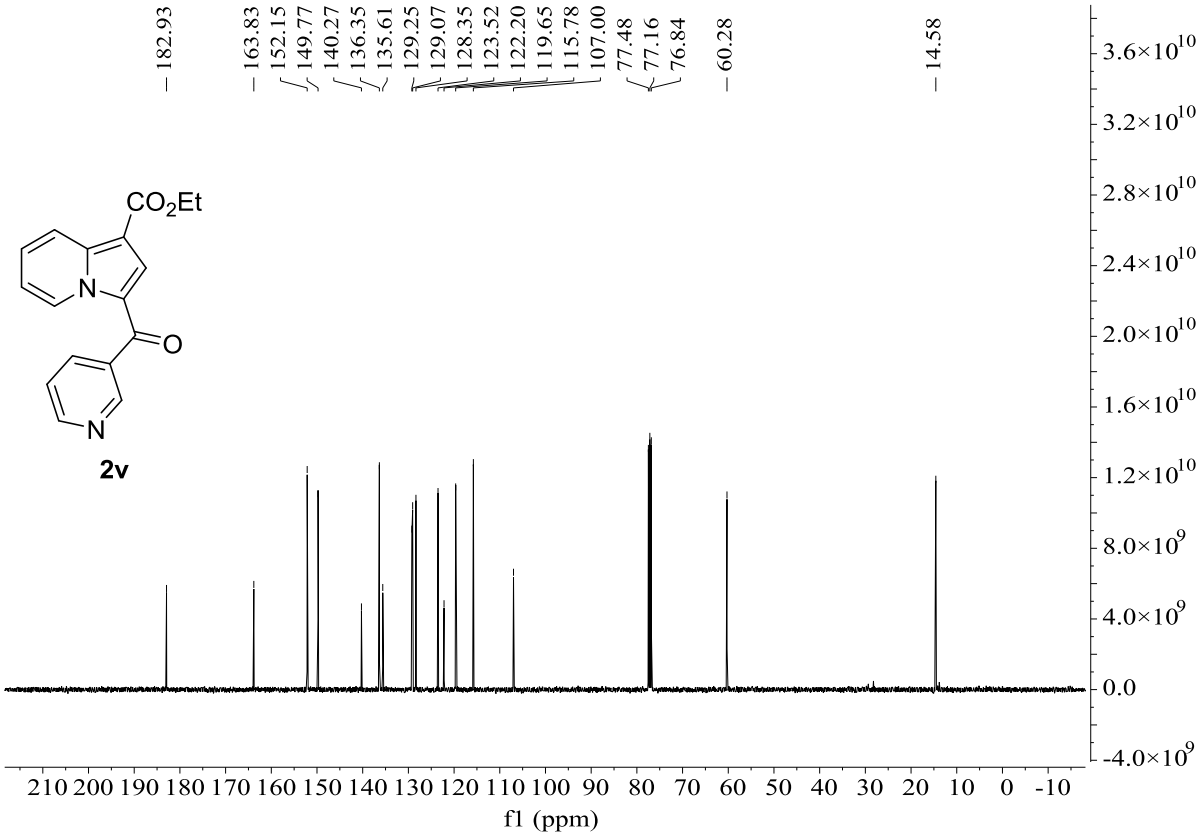
^{13}C NMR Spectrum of 2u at 25 °C ($CDCl_3$, 100 MHz)



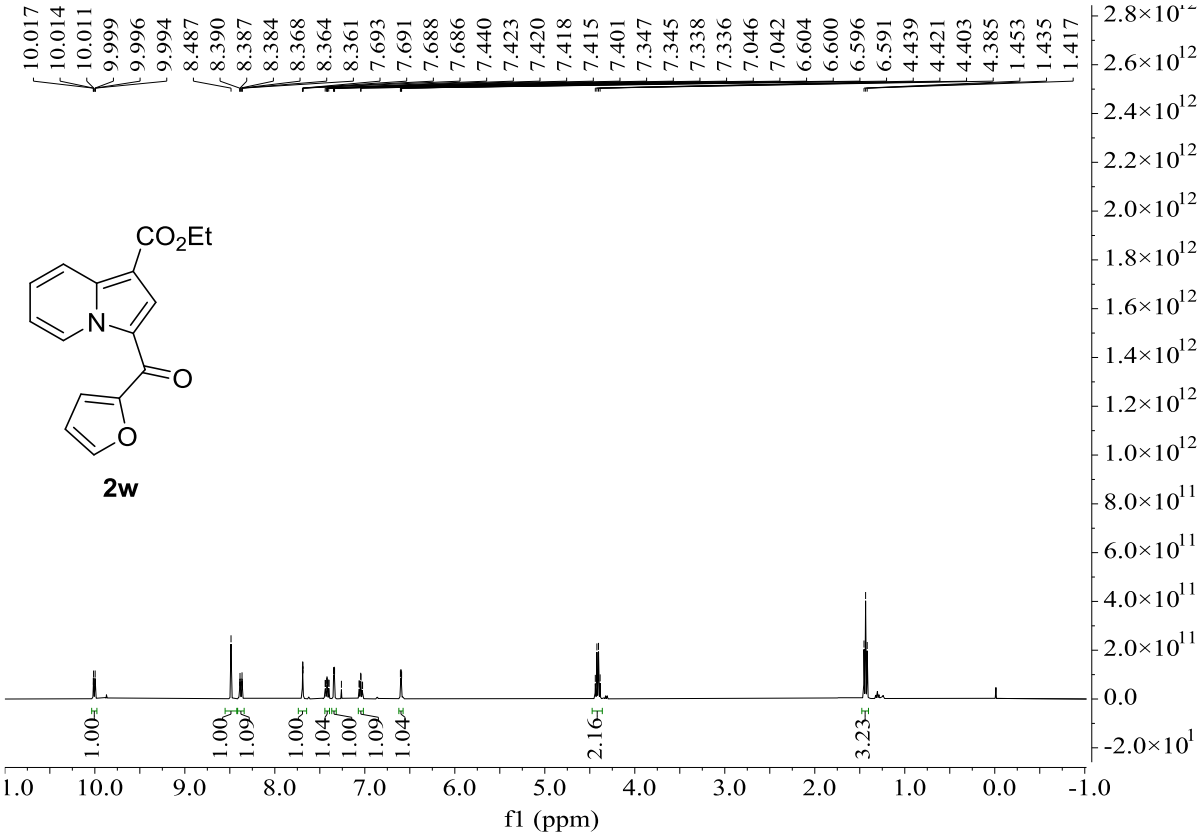
^{1}H NMR Spectrum of 2v at 25 °C ($CDCl_3$, 400 MHz)



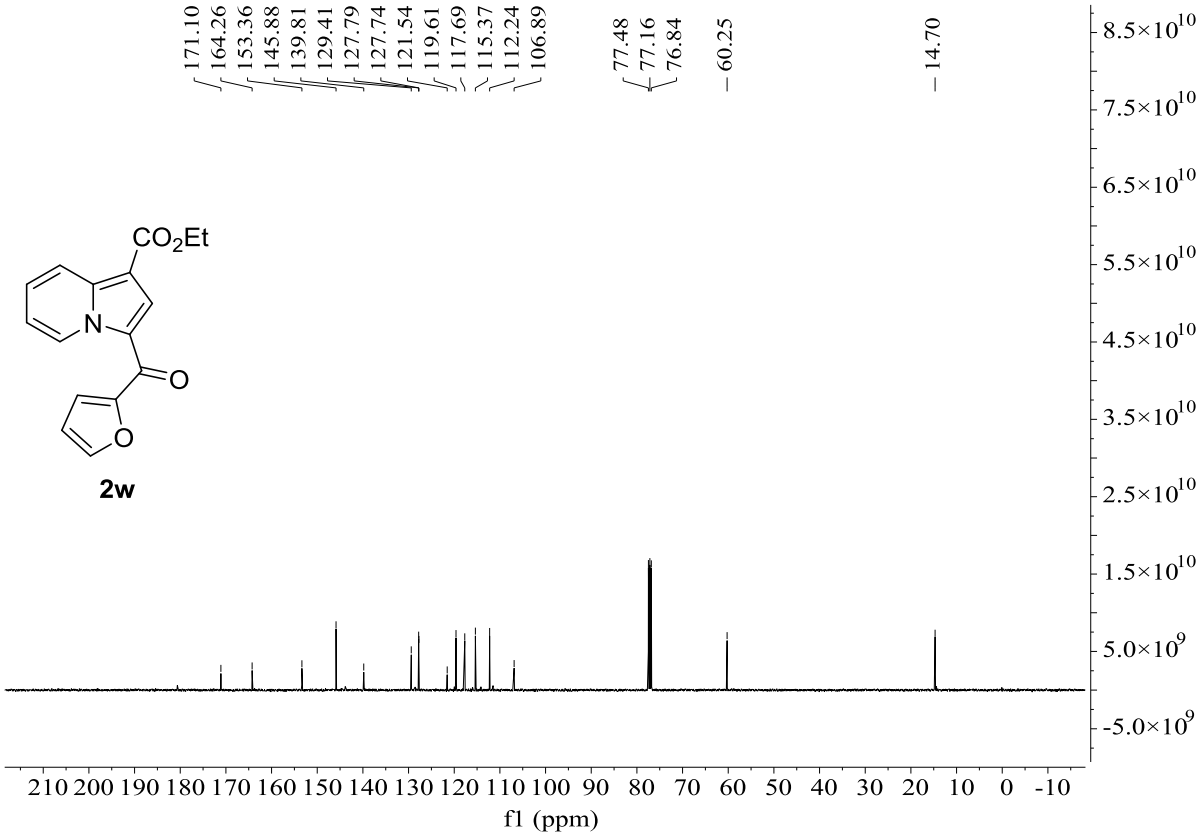
^{13}C NMR Spectrum of 2v at 25 °C ($CDCl_3$, 100 MHz)



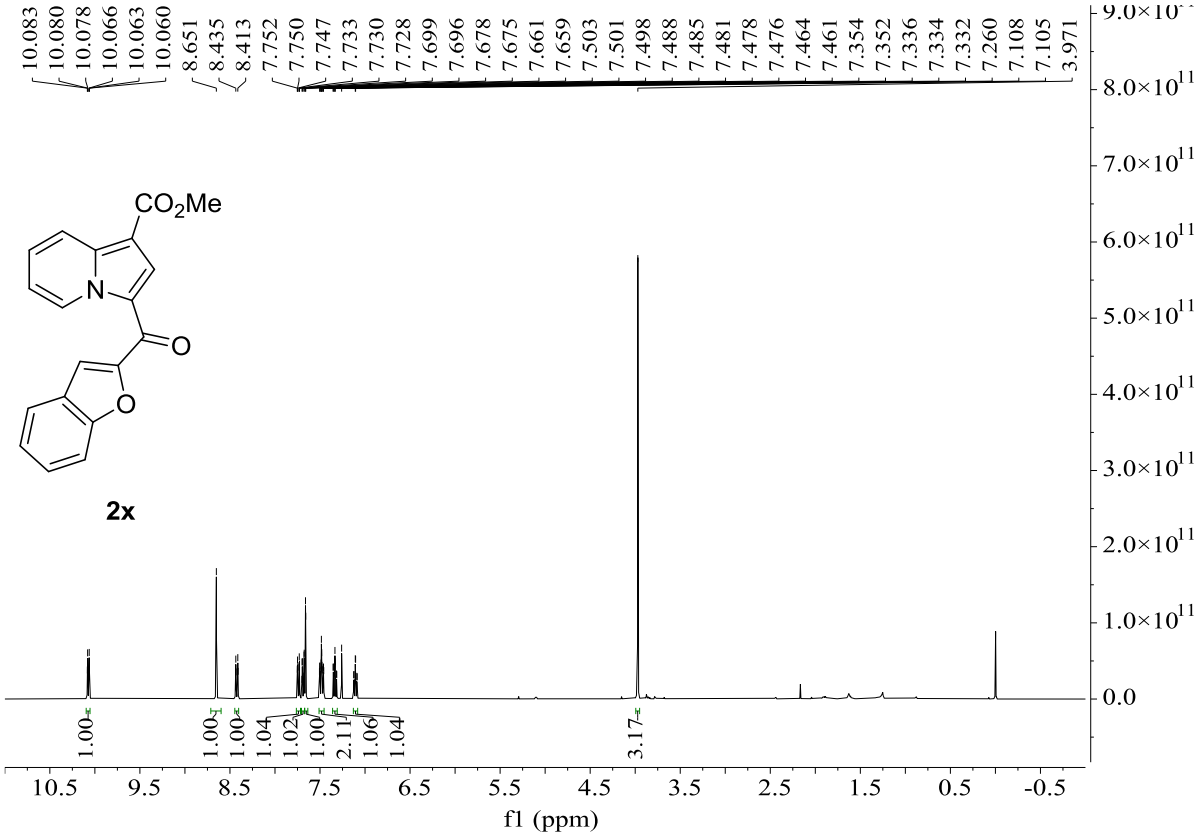
^{1}H NMR Spectrum of 2w at 25 °C ($CDCl_3$, 400 MHz)



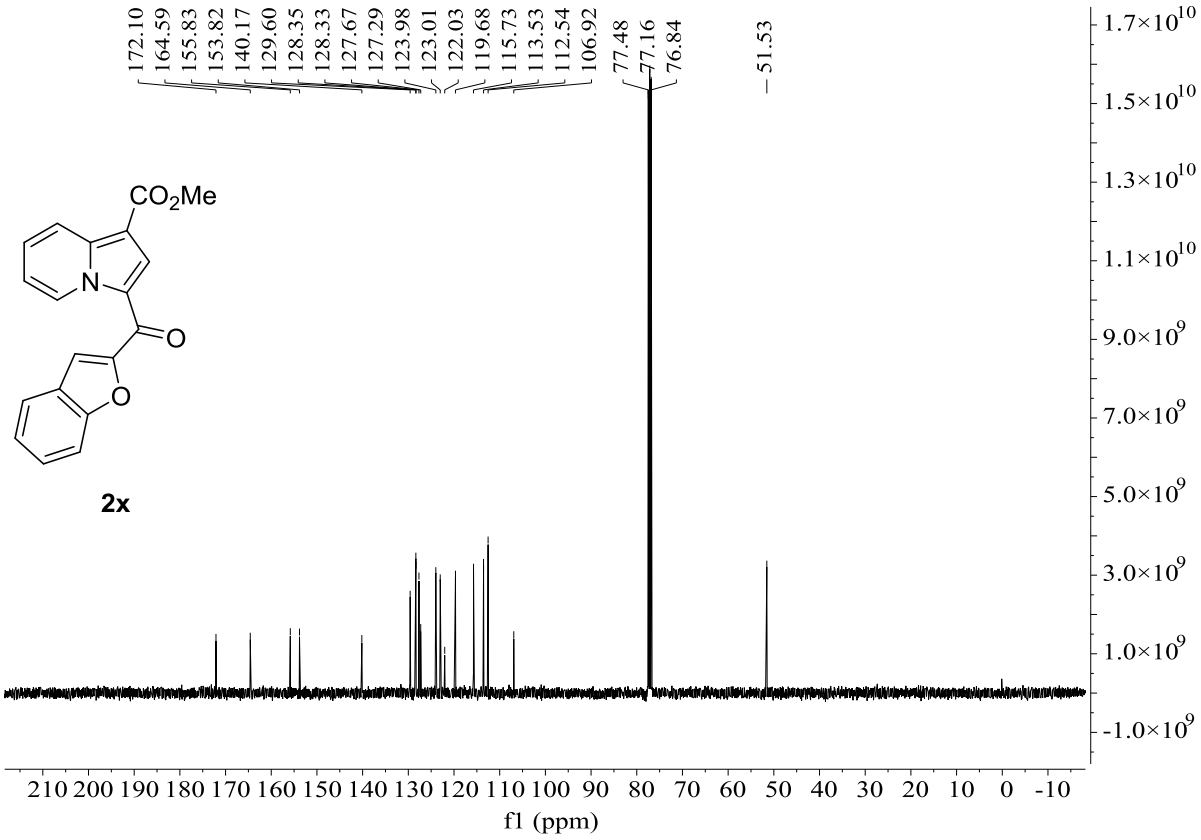
^{13}C NMR Spectrum of 2w at 25 °C ($CDCl_3$, 100 MHz)



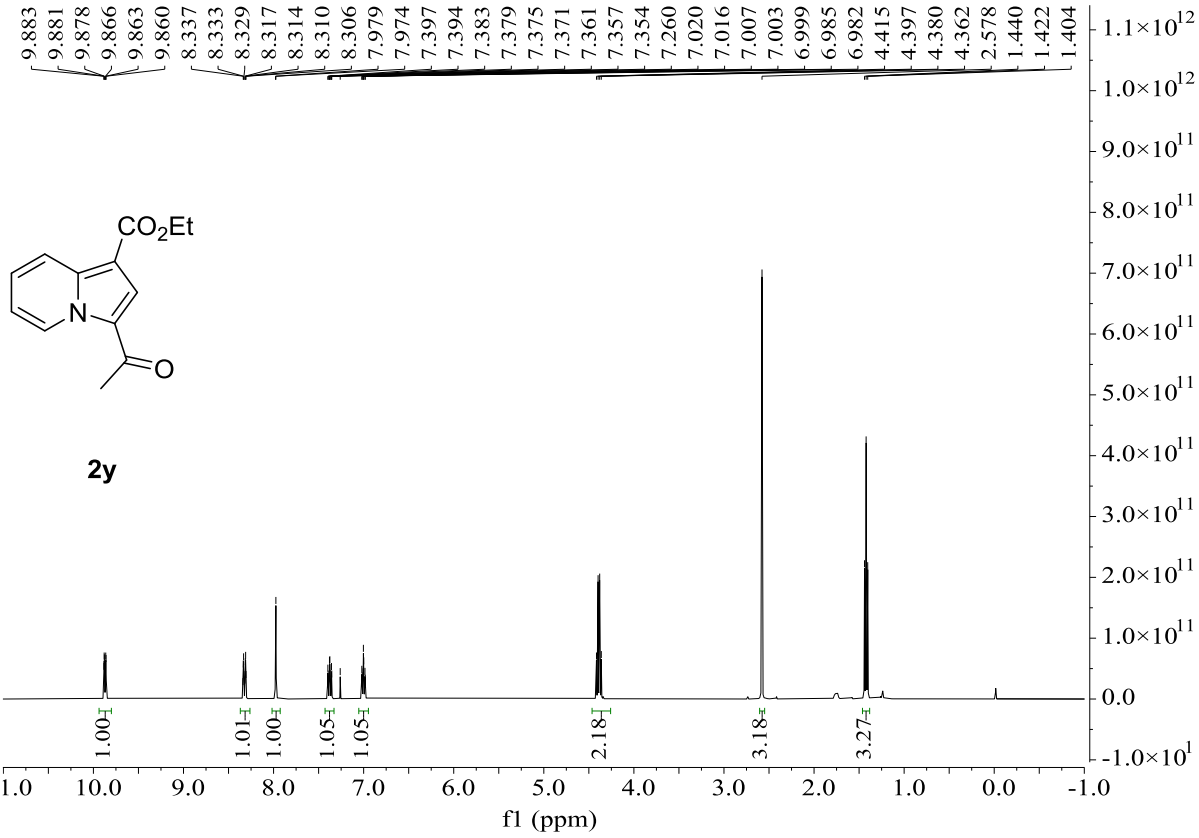
^{1}H NMR Spectrum of 2x at 25 °C ($CDCl_3$, 400 MHz)



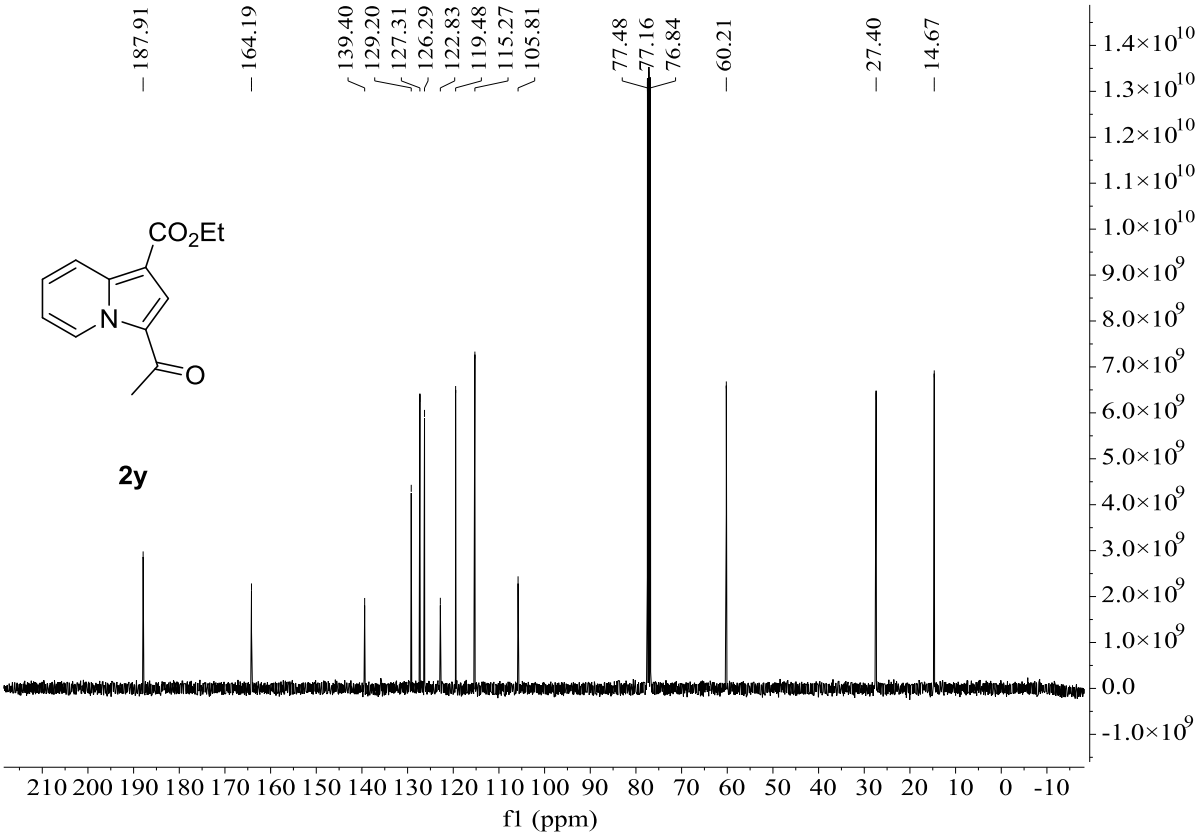
^{13}C NMR Spectrum of 2x at 25 °C ($CDCl_3$, 100 MHz)



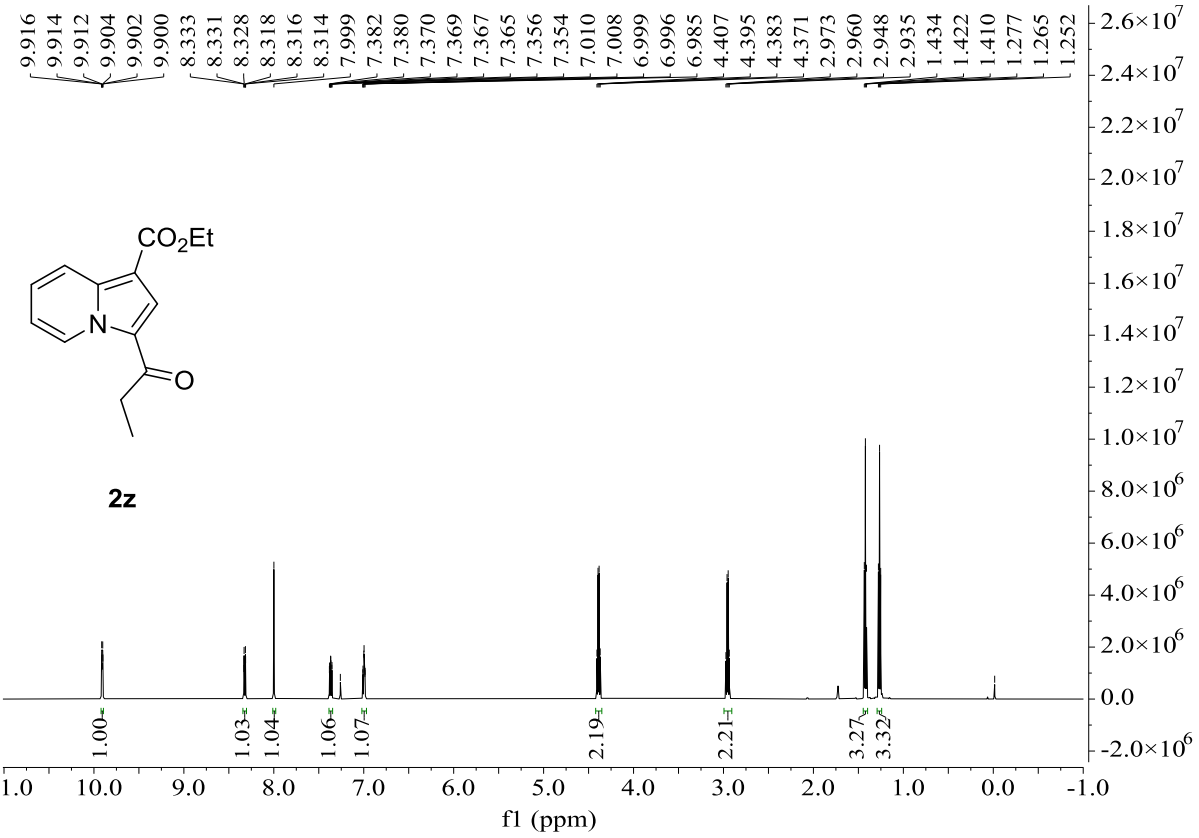
^{1}H NMR Spectrum of 2y at 25 °C ($CDCl_3$, 400 MHz)



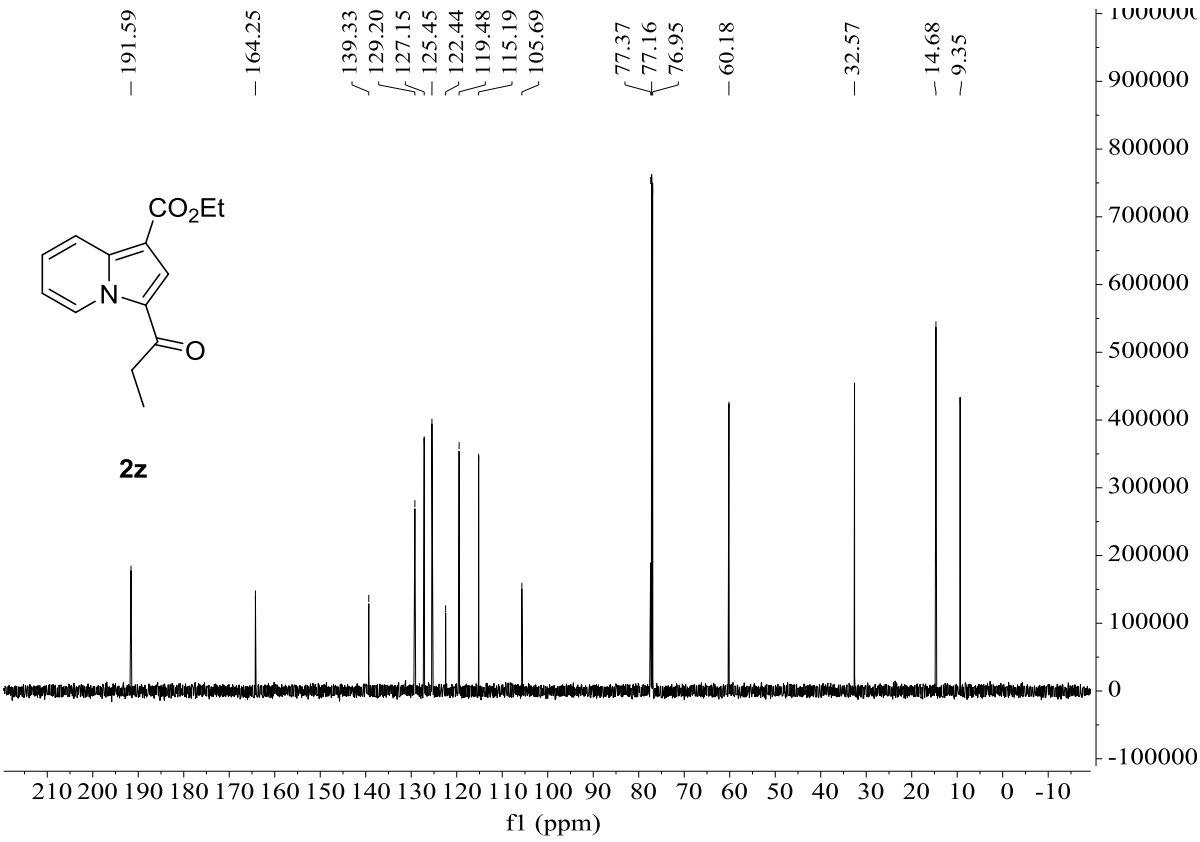
^{13}C NMR Spectrum of 2y at 25 °C ($CDCl_3$, 100 MHz)



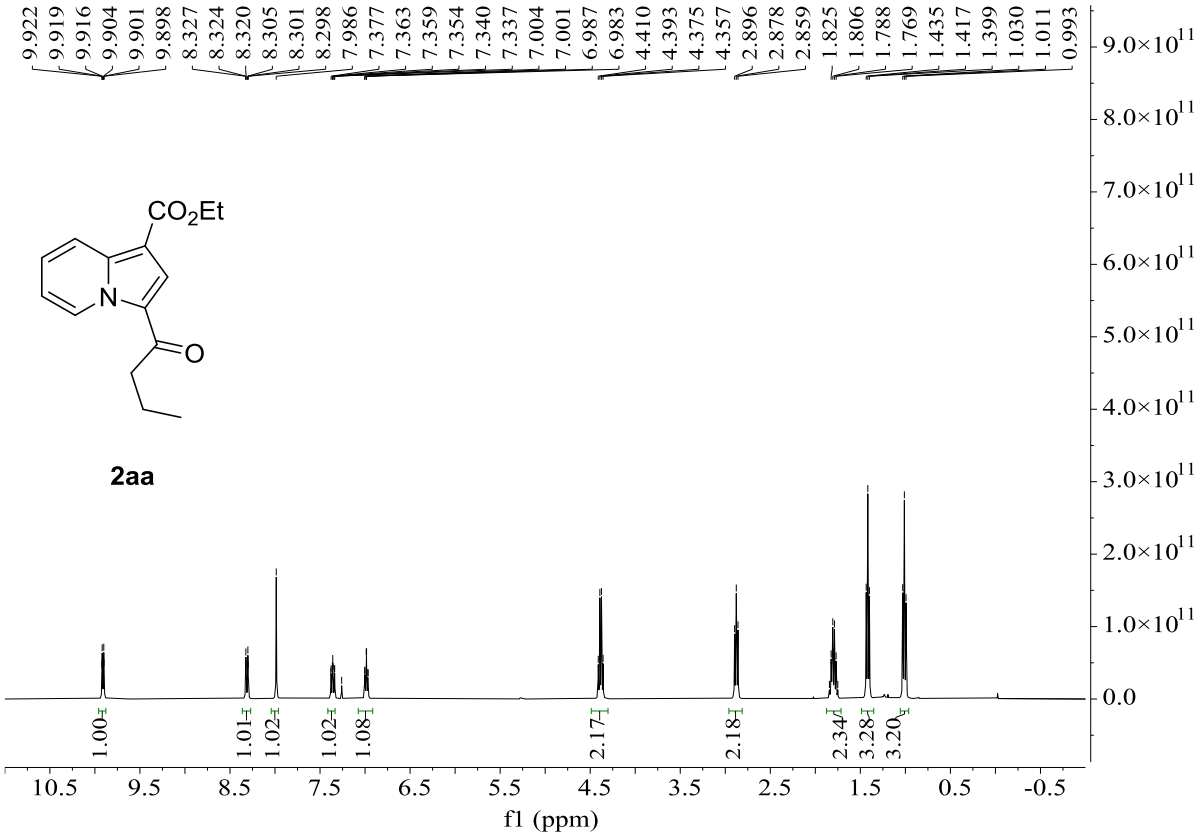
^{1}H NMR Spectrum of 2z at 25 °C ($CDCl_3$, 600 MHz)



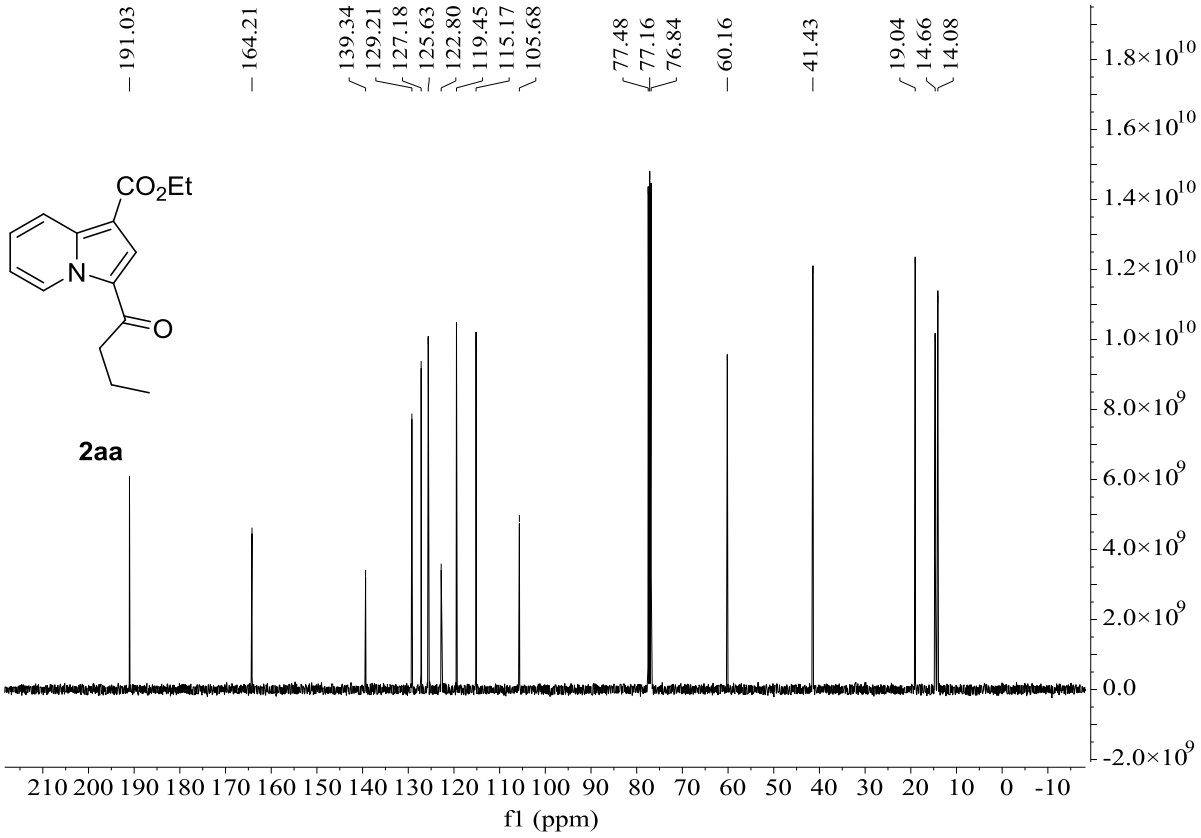
^{13}C NMR Spectrum of 2z at 25 °C ($CDCl_3$, 150 MHz)



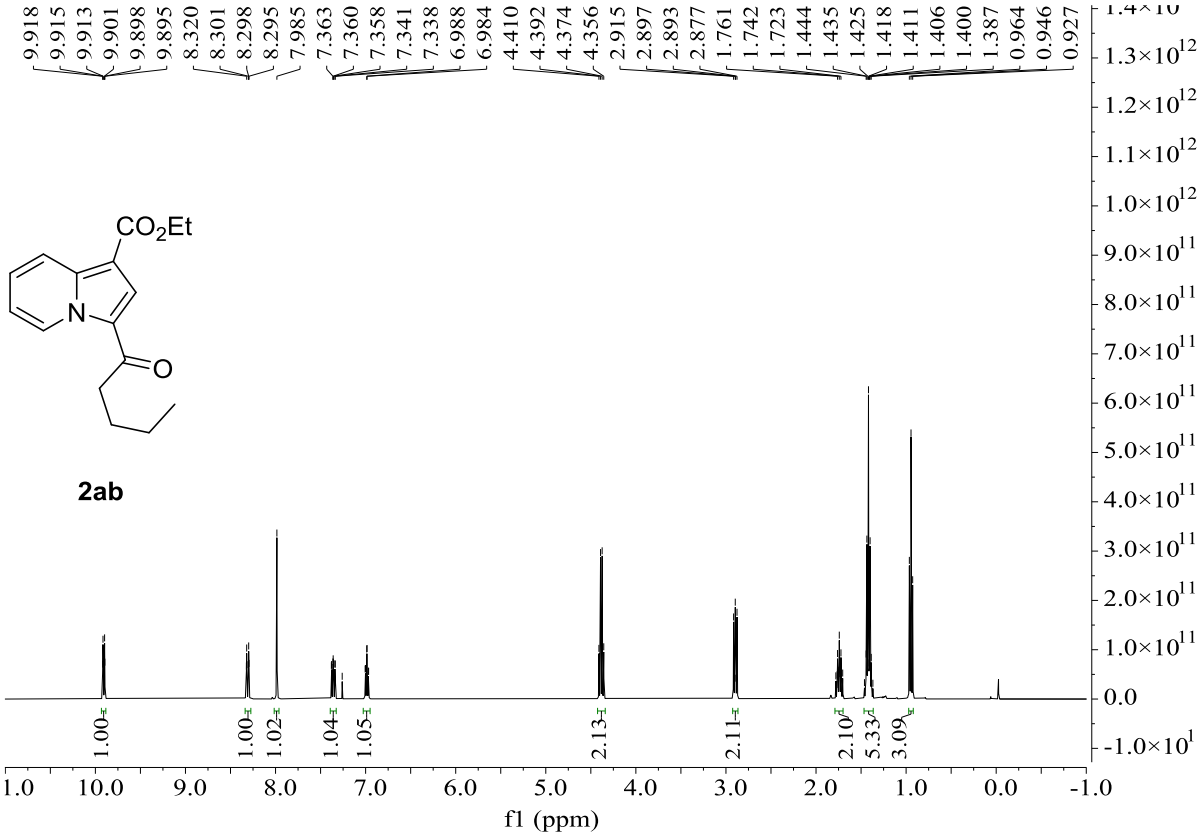
^{1}H NMR Spectrum of 2aa at 25 °C ($CDCl_3$, 400 MHz)



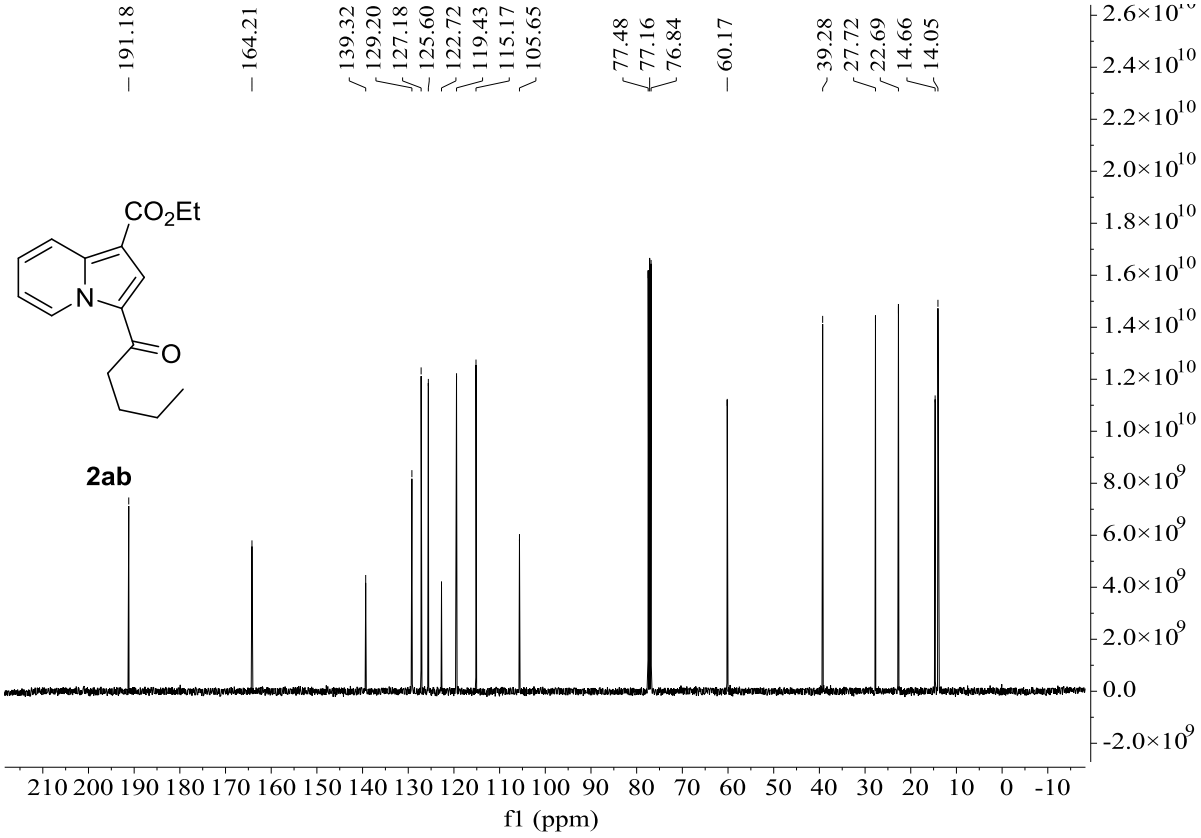
^{13}C NMR Spectrum of 2aa at 25 °C ($CDCl_3$, 100 MHz)



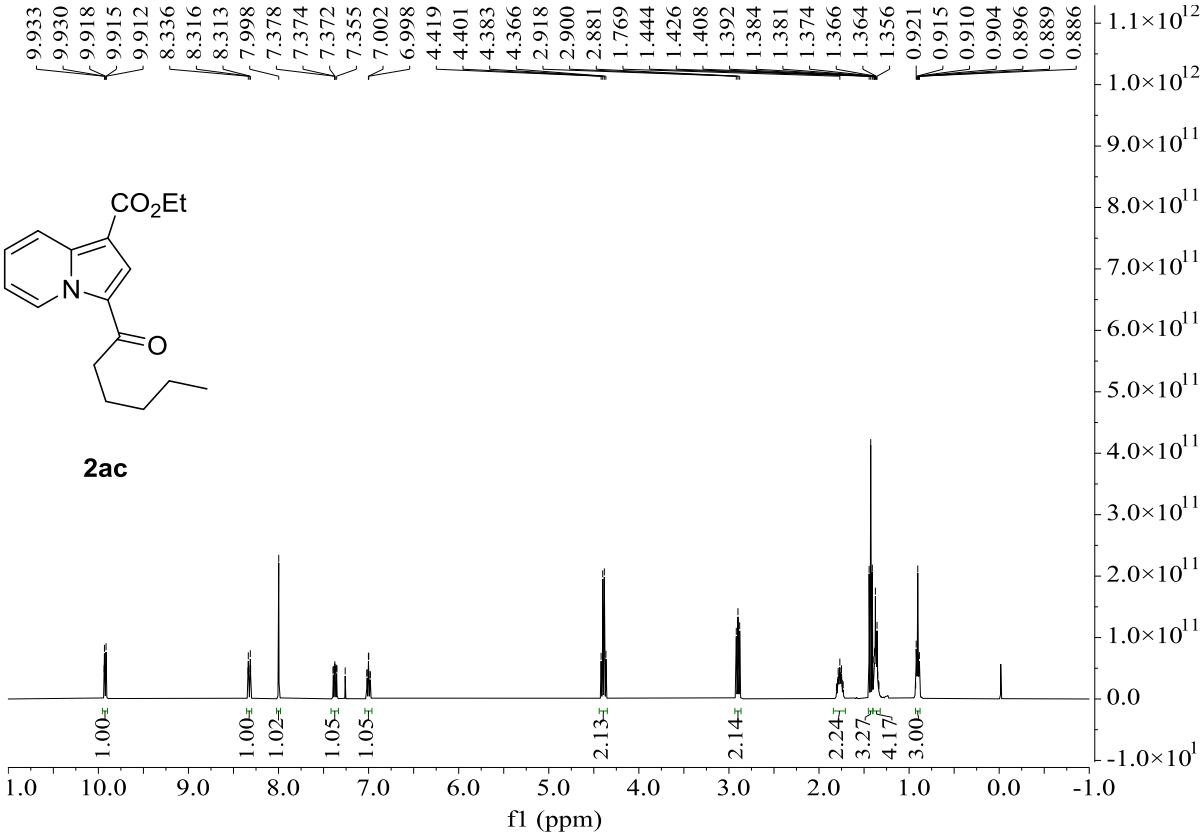
^{1}H NMR Spectrum of 2ab at 25 °C ($CDCl_3$, 400 MHz)



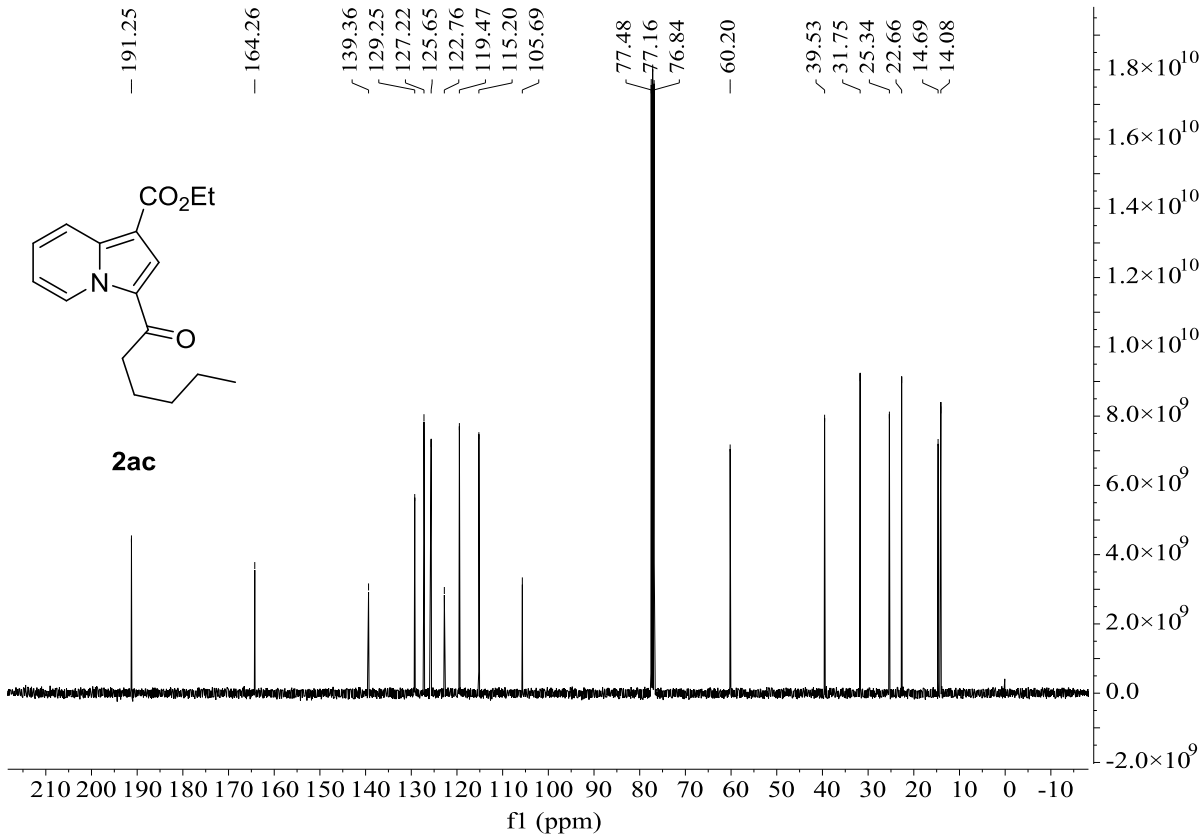
^{13}C NMR Spectrum of 2ab at 25 °C ($CDCl_3$, 100 MHz)



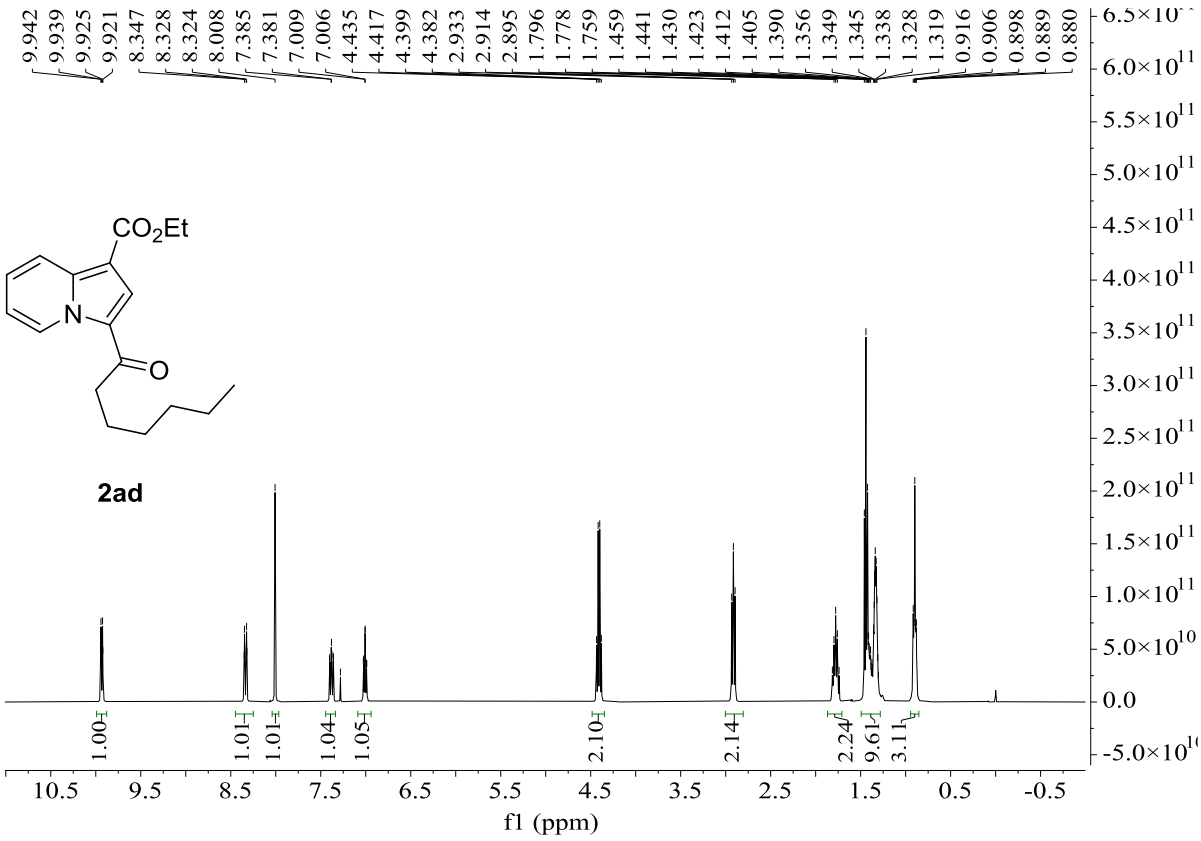
^{1}H NMR Spectrum of 2ac at 25 °C ($CDCl_3$, 400 MHz)



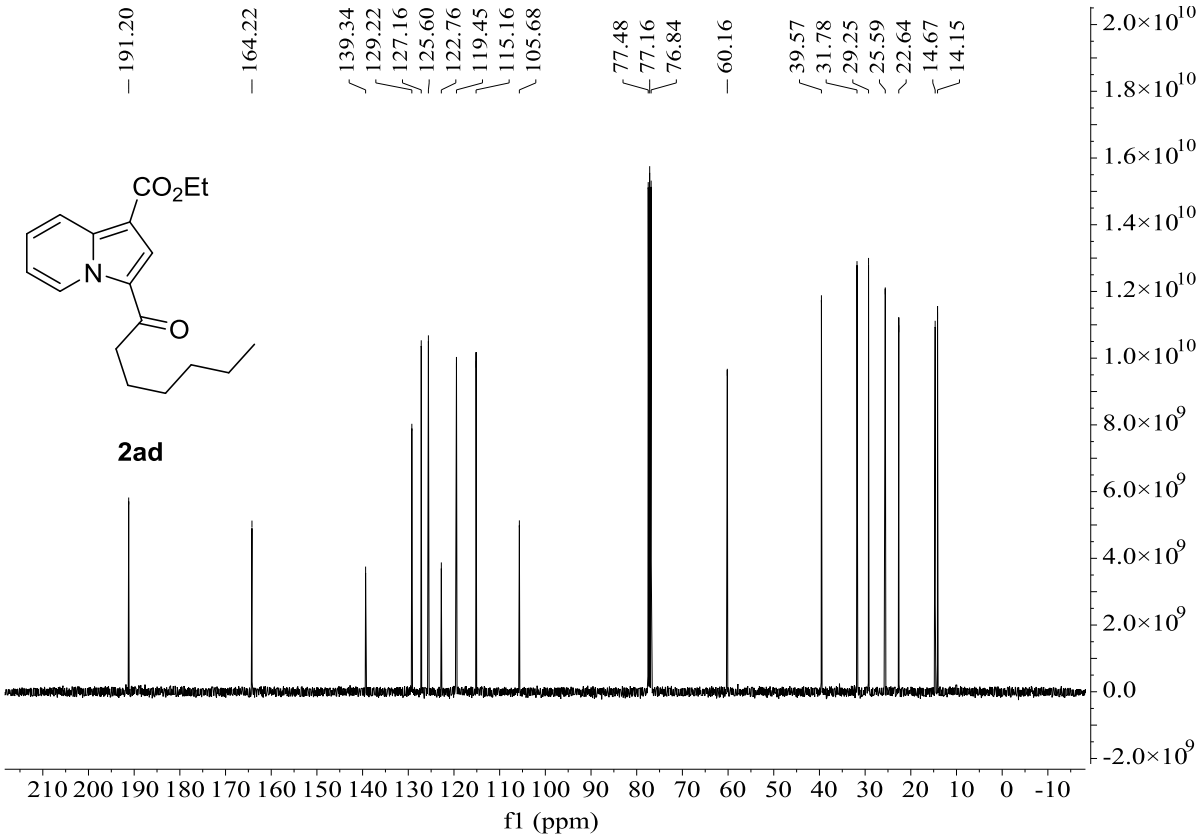
^{13}C NMR Spectrum of 2ac at 25 °C ($CDCl_3$, 100 MHz)



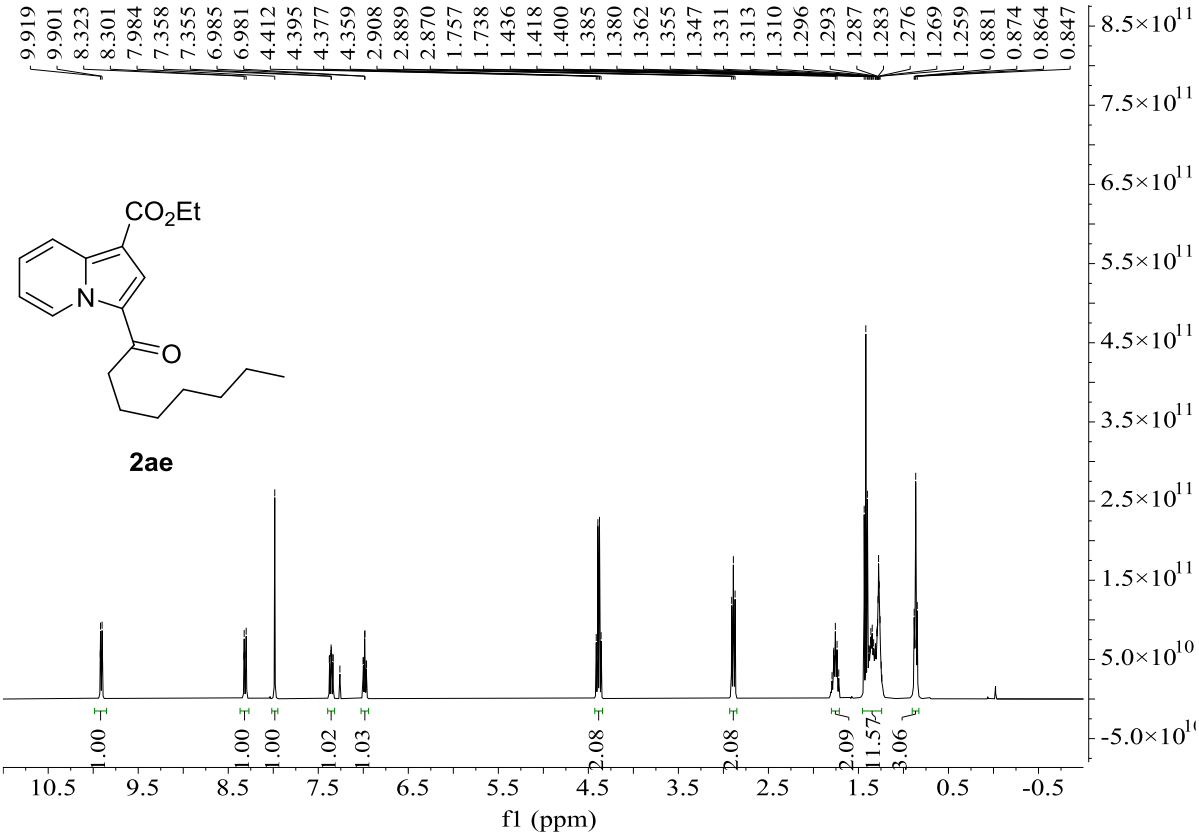
^{1}H NMR Spectrum of 2ad at 25 °C ($CDCl_3$, 400 MHz)



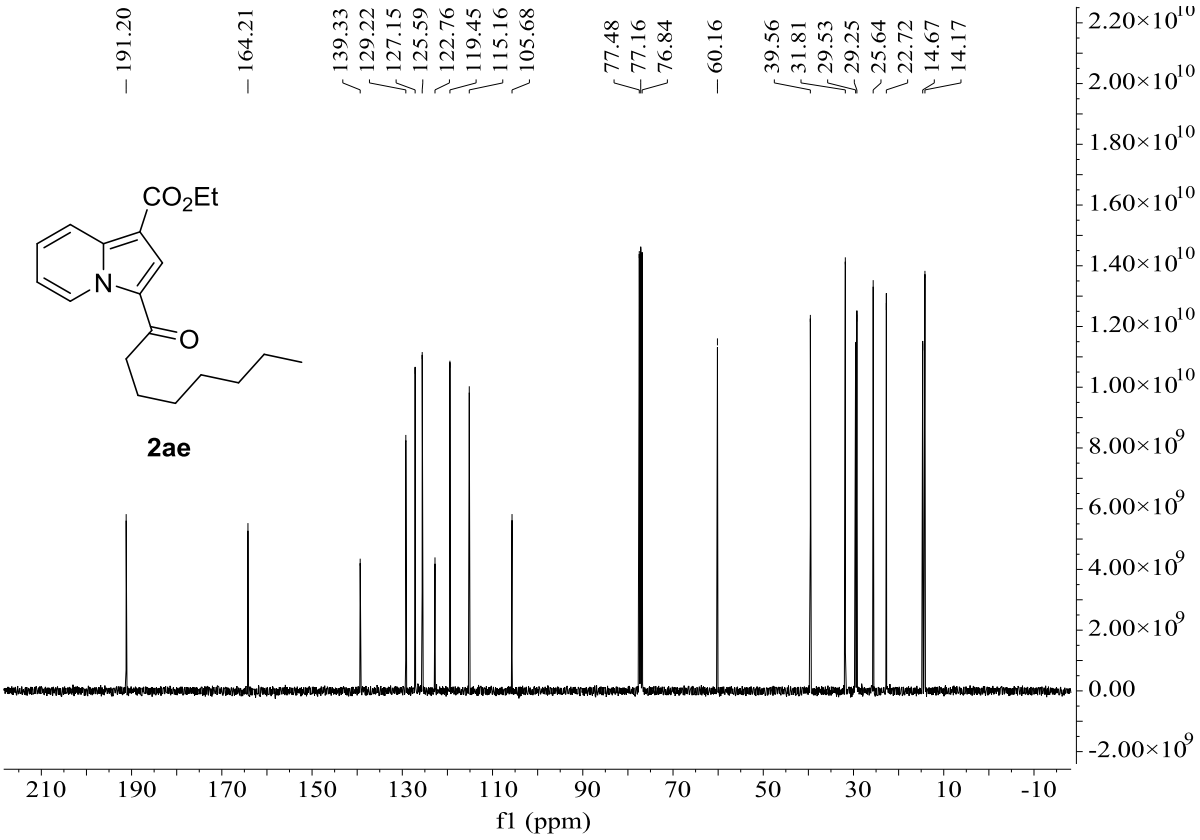
^{13}C NMR Spectrum of 2ad at 25 °C ($CDCl_3$, 100 MHz)



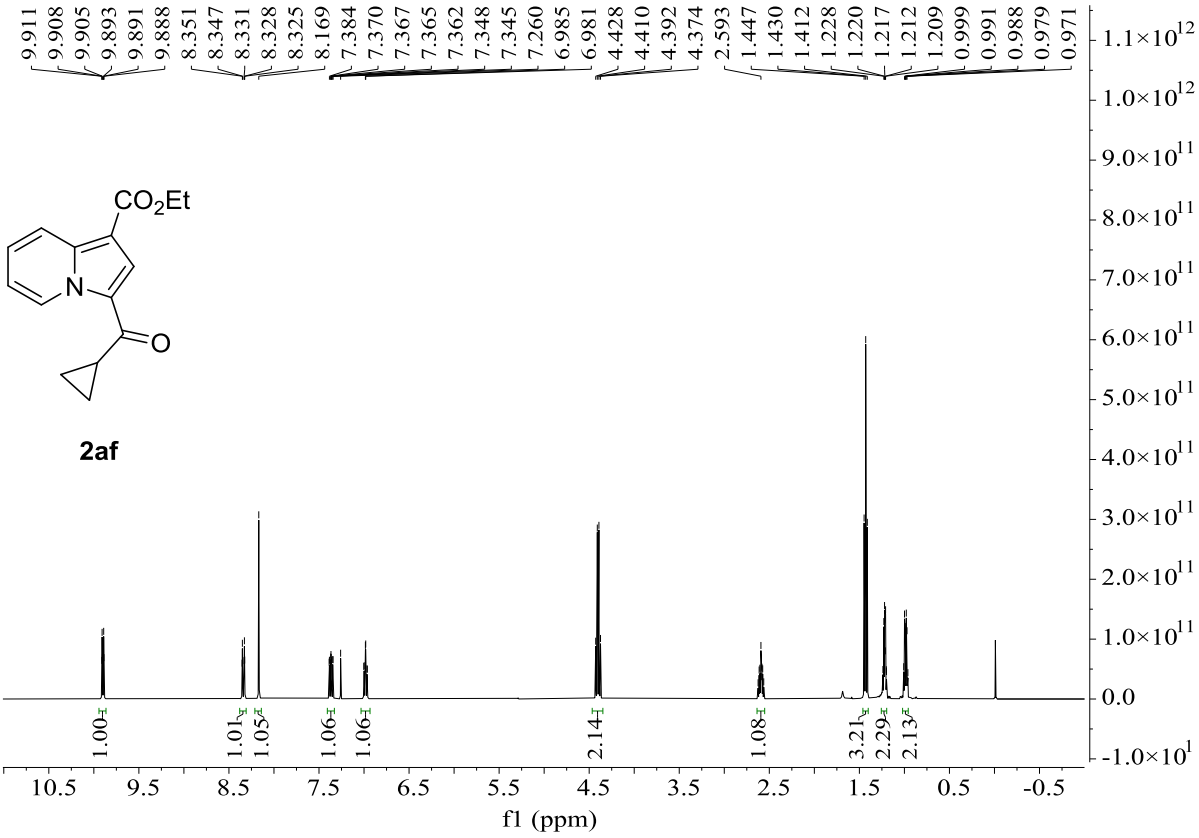
^{1}H NMR Spectrum of 2ae at 25 °C ($CDCl_3$, 400 MHz)



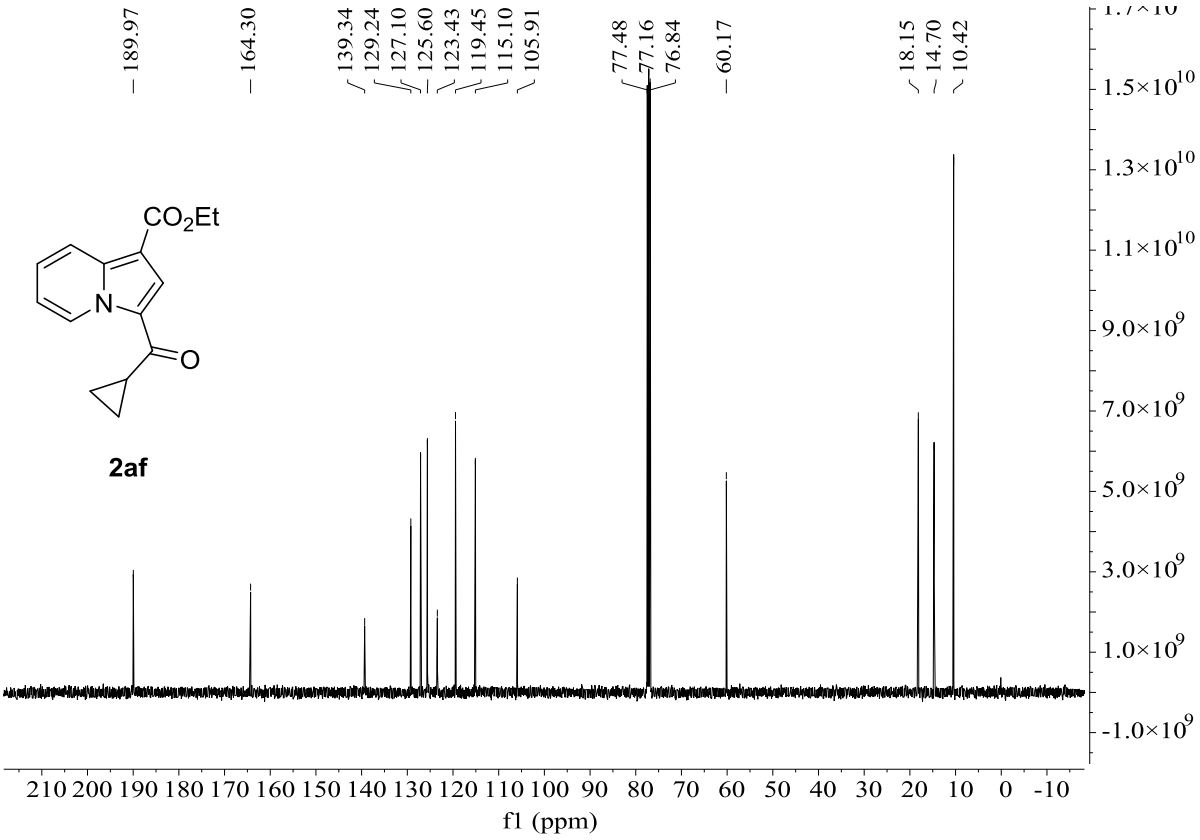
^{13}C NMR Spectrum of 2ae at 25 °C ($CDCl_3$, 100 MHz)



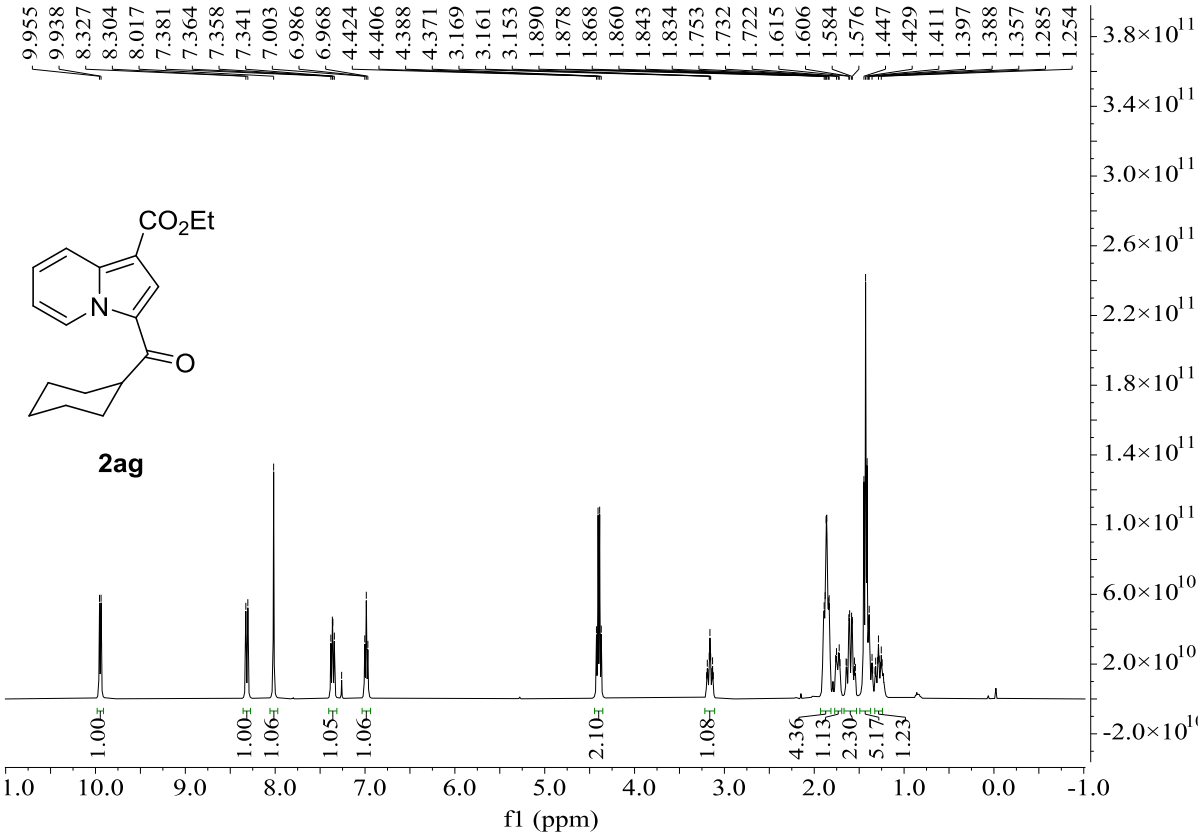
^{1}H NMR Spectrum of 2af at 25 °C ($CDCl_3$, 400 MHz)



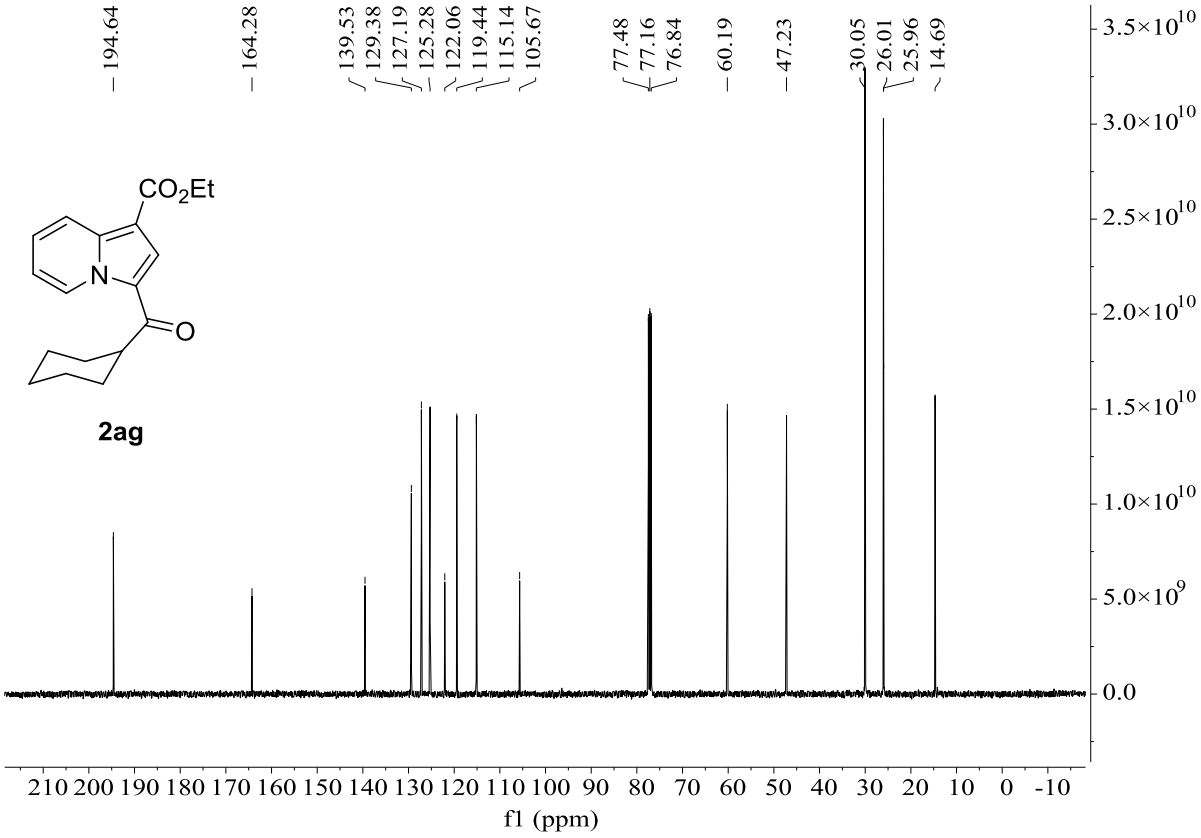
^{13}C NMR Spectrum of 2af at 25 °C ($CDCl_3$, 100 MHz)



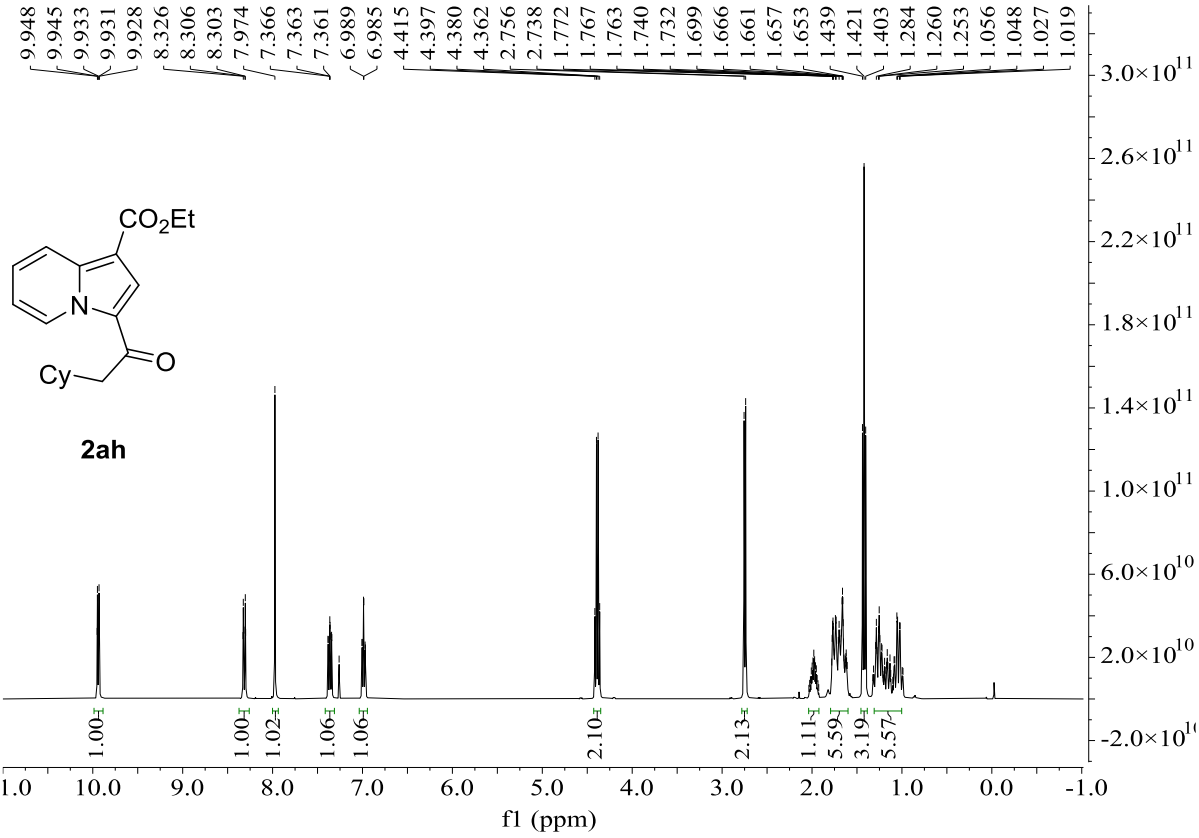
^{1}H NMR Spectrum of 2ag at 25 °C ($CDCl_3$, 400 MHz)



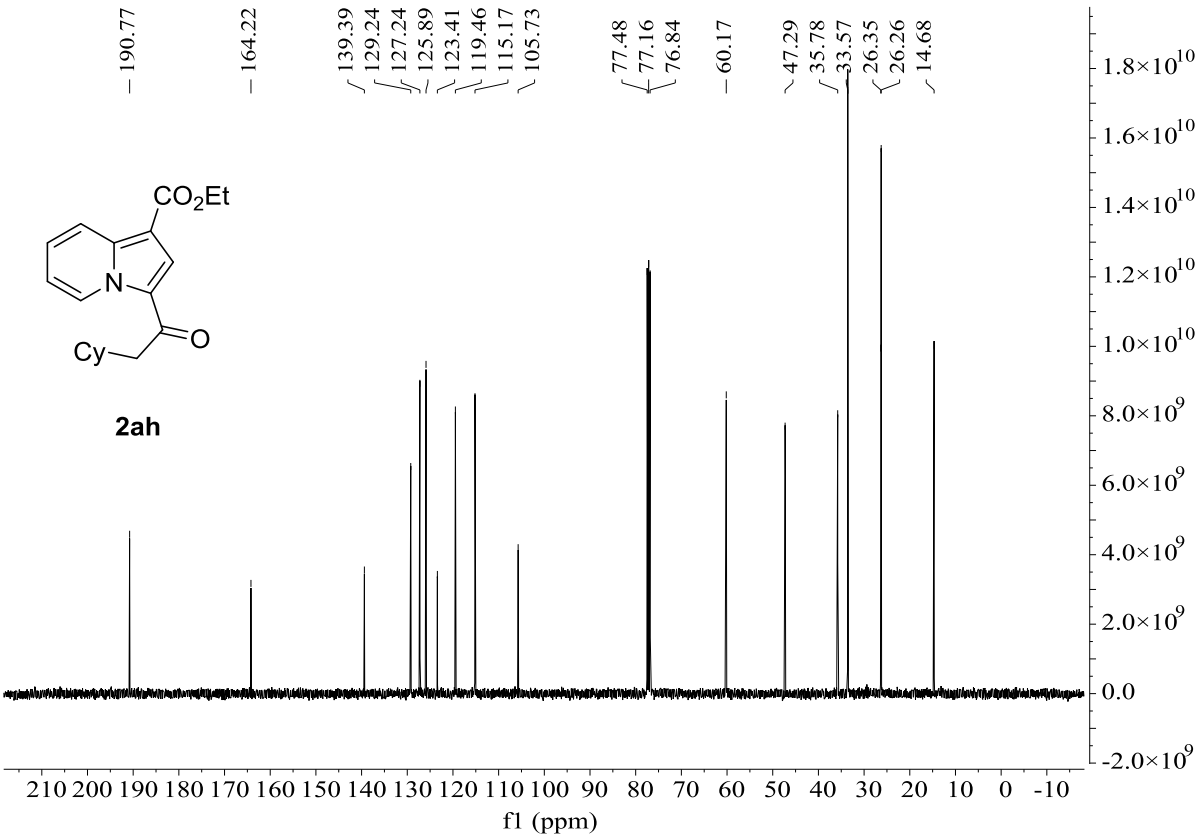
^{13}C NMR Spectrum of 2ag at 25 °C ($CDCl_3$, 100 MHz)



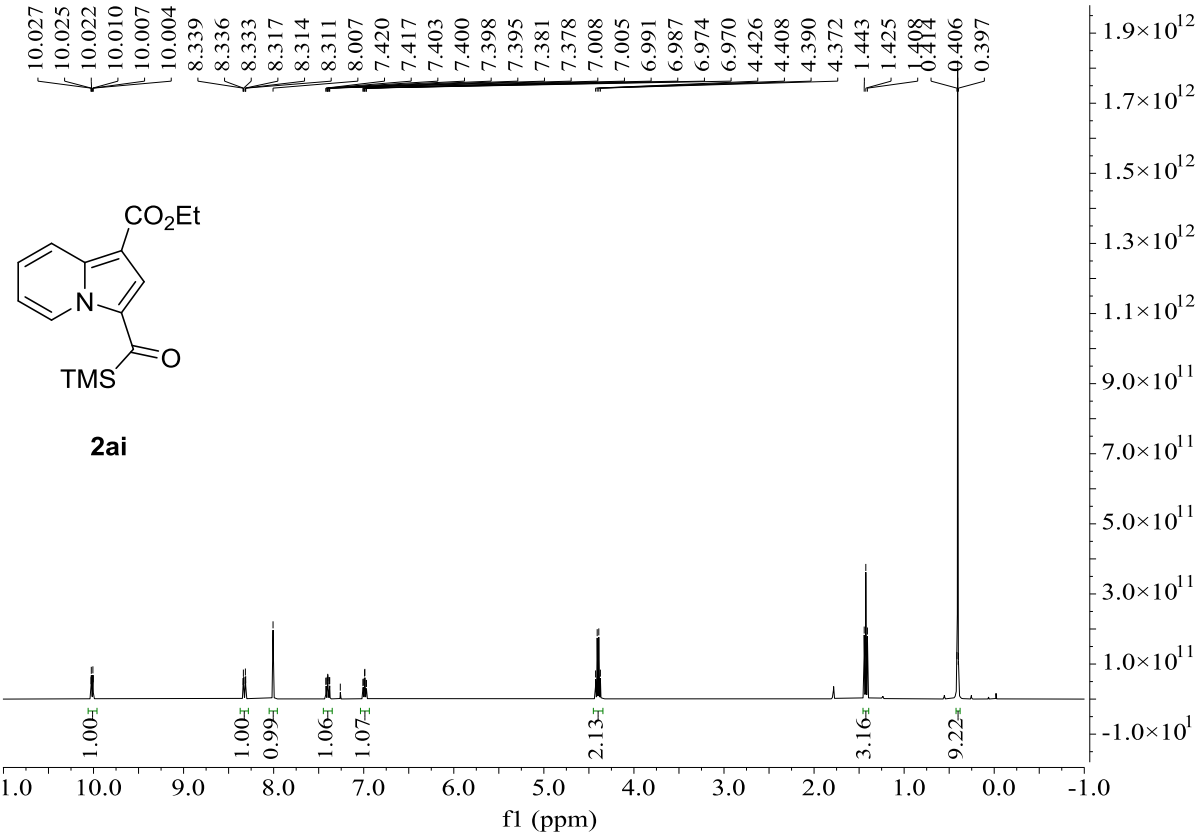
^{1}H NMR Spectrum of 2ah at 25 °C ($CDCl_3$, 400 MHz)



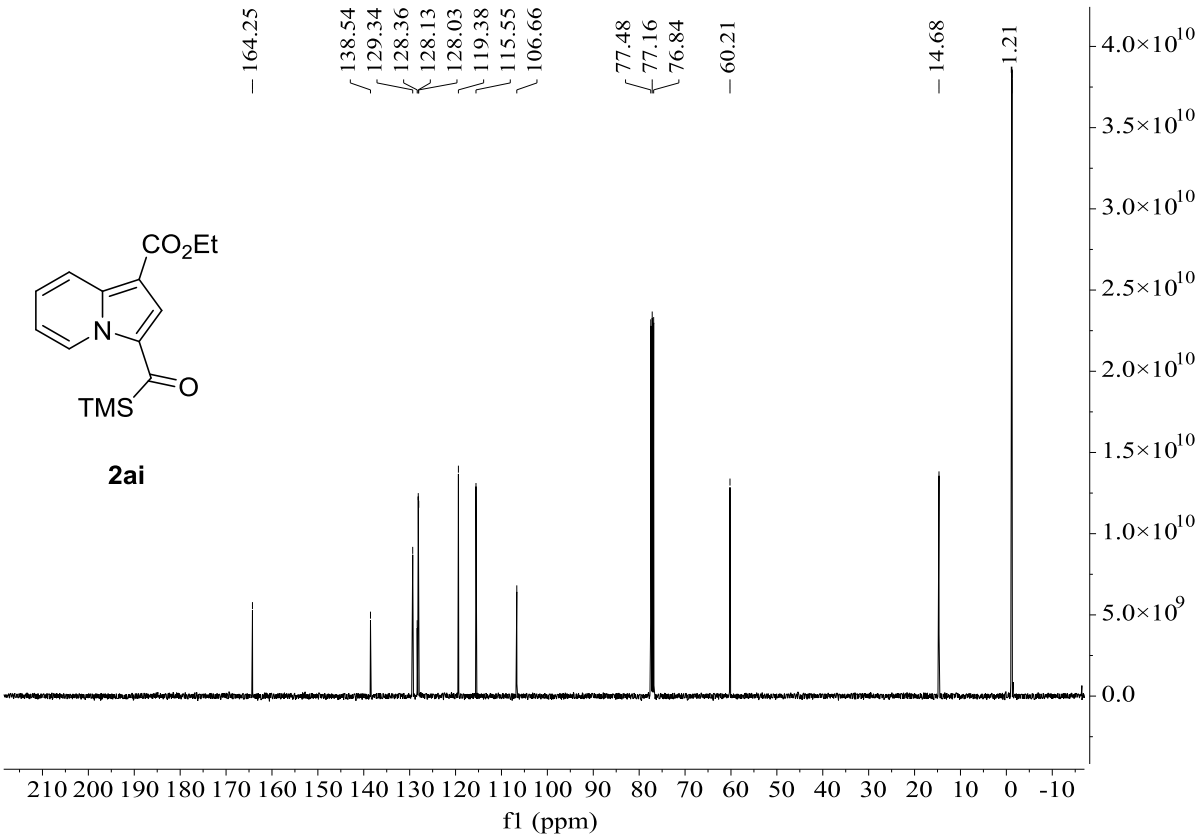
^{13}C NMR Spectrum of 2ah at 25 °C ($CDCl_3$, 100 MHz)



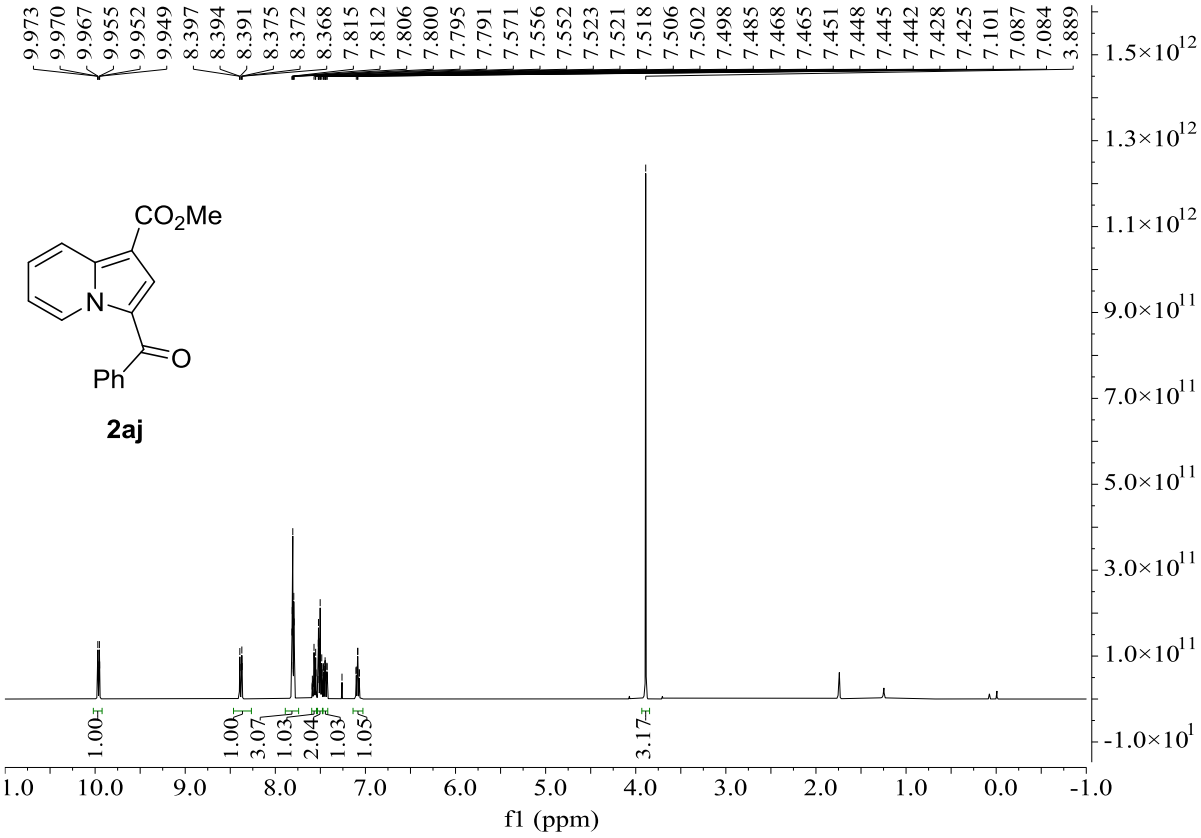
^{1}H NMR Spectrum of 2ai at 25 °C ($CDCl_3$, 400 MHz)



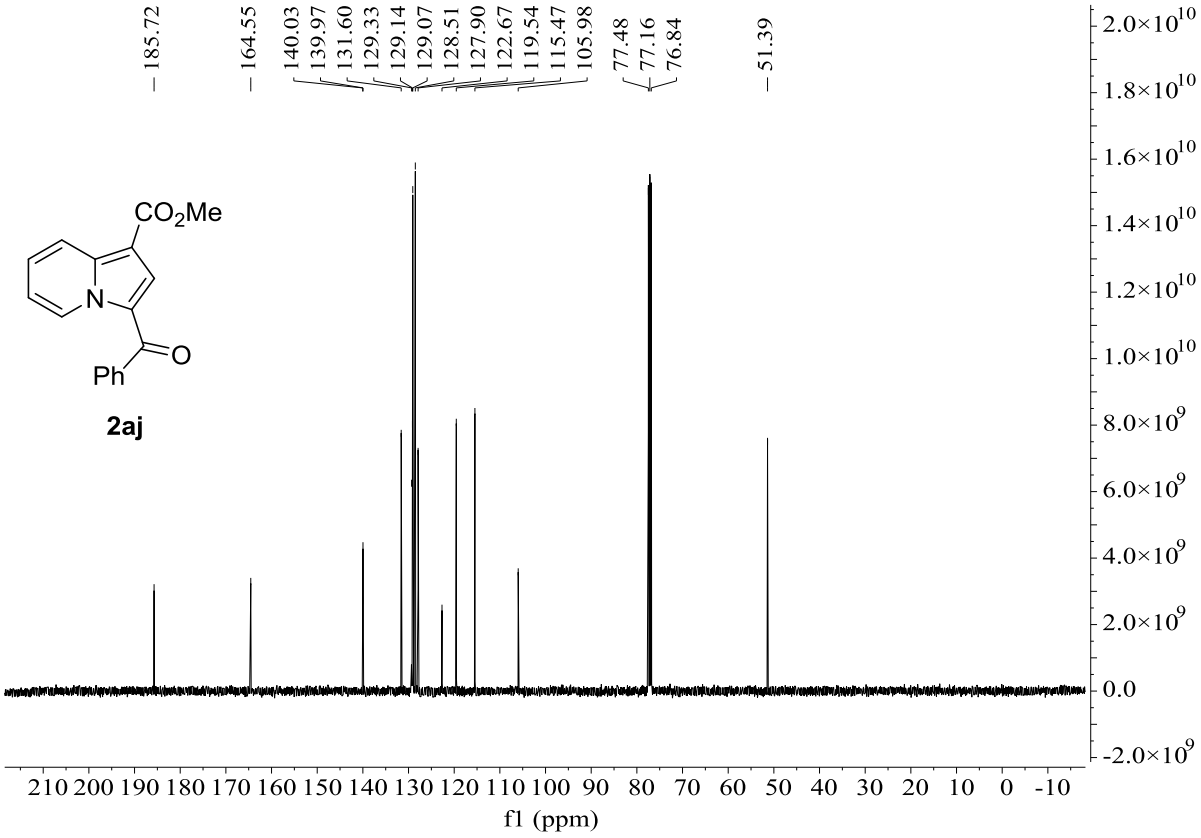
^{13}C NMR Spectrum of 2ai at 25 °C ($CDCl_3$, 100 MHz)



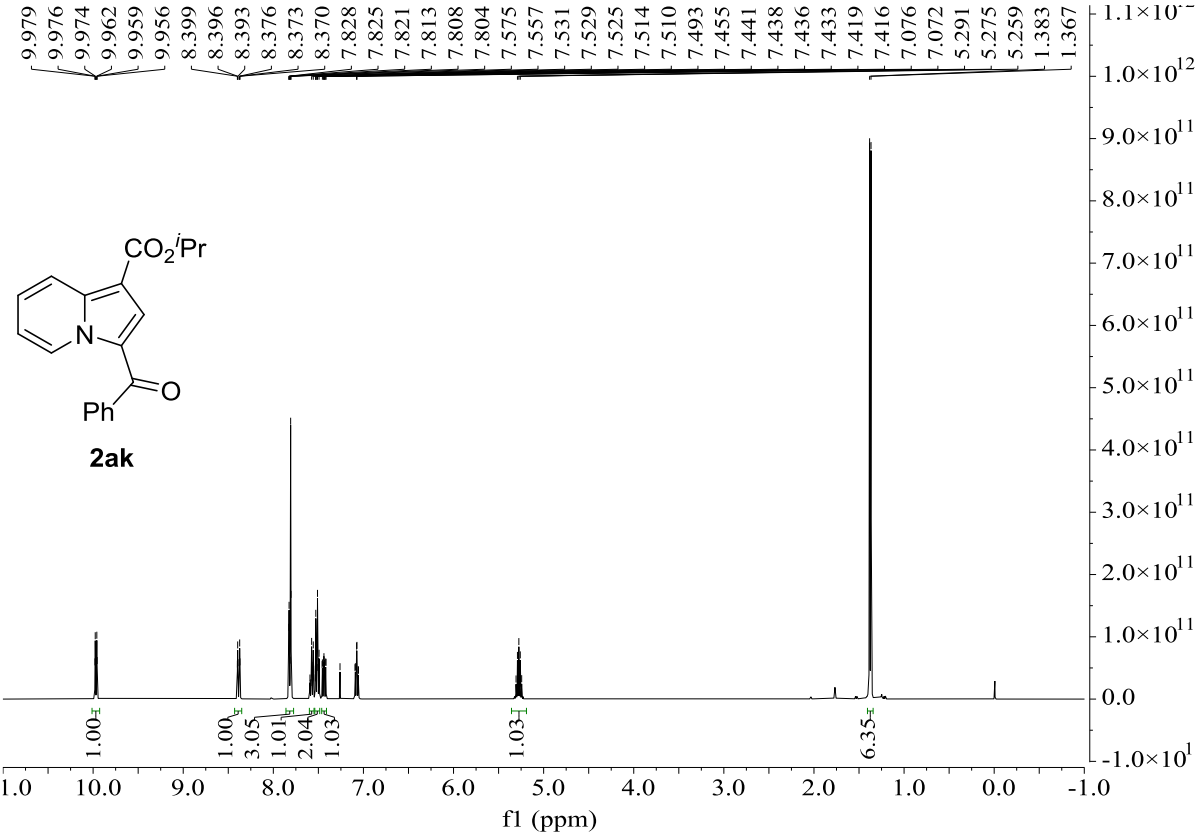
^{1}H NMR Spectrum of 2aj at 25 °C ($CDCl_3$, 400 MHz)



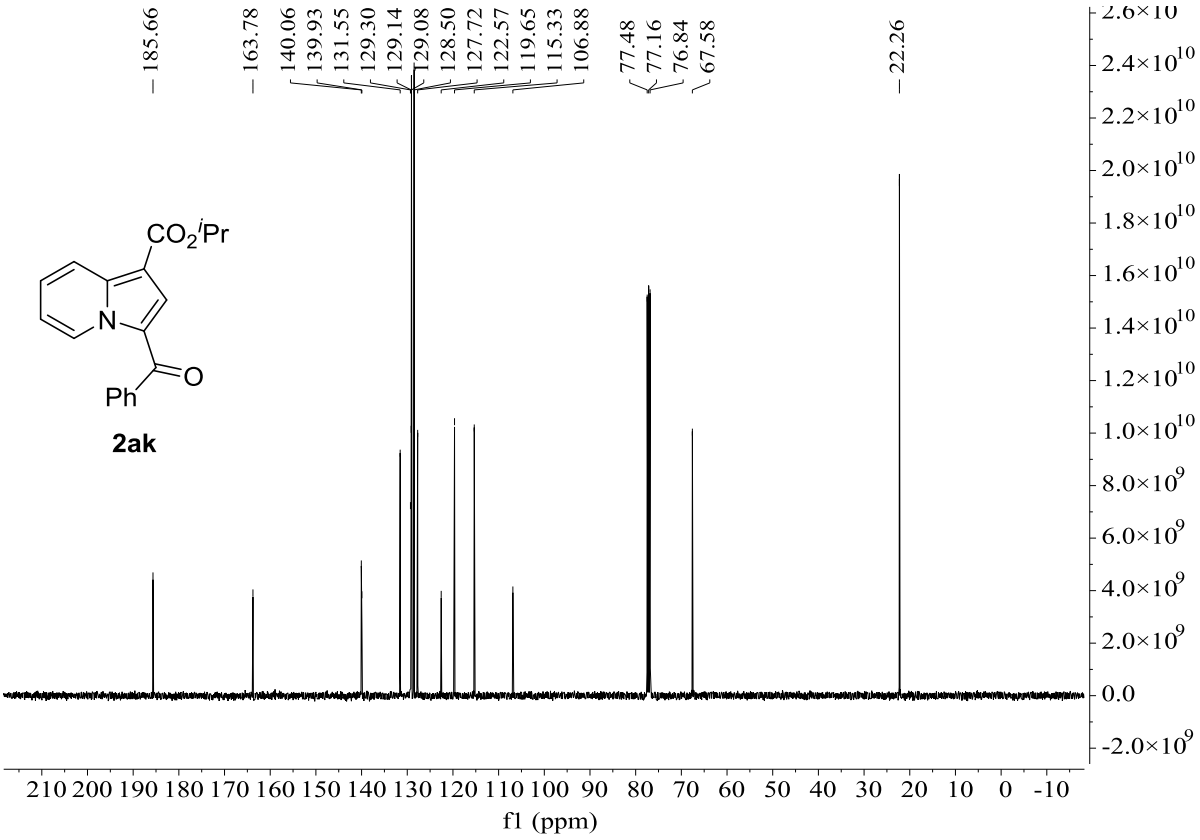
^{13}C NMR Spectrum of 2aj at 25 °C ($CDCl_3$, 100 MHz)



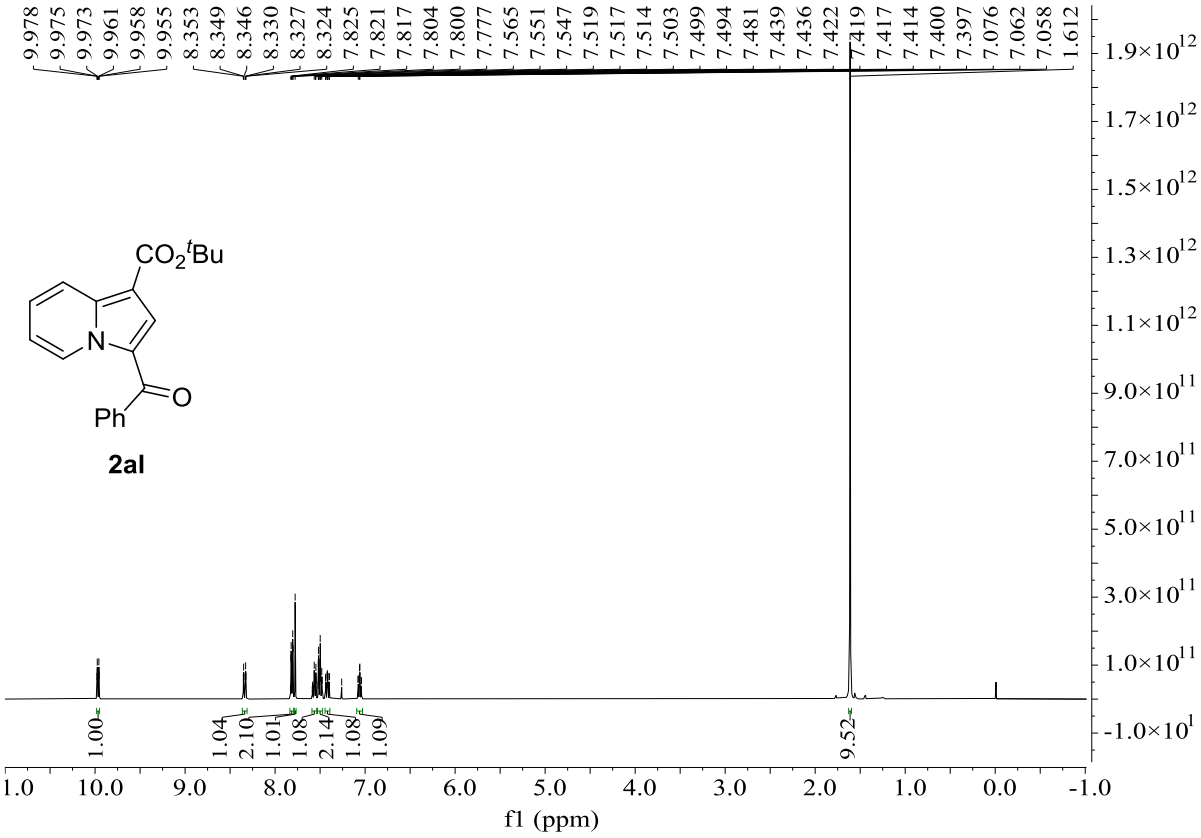
^{1}H NMR Spectrum of 2ak at 25 °C ($CDCl_3$, 400 MHz)



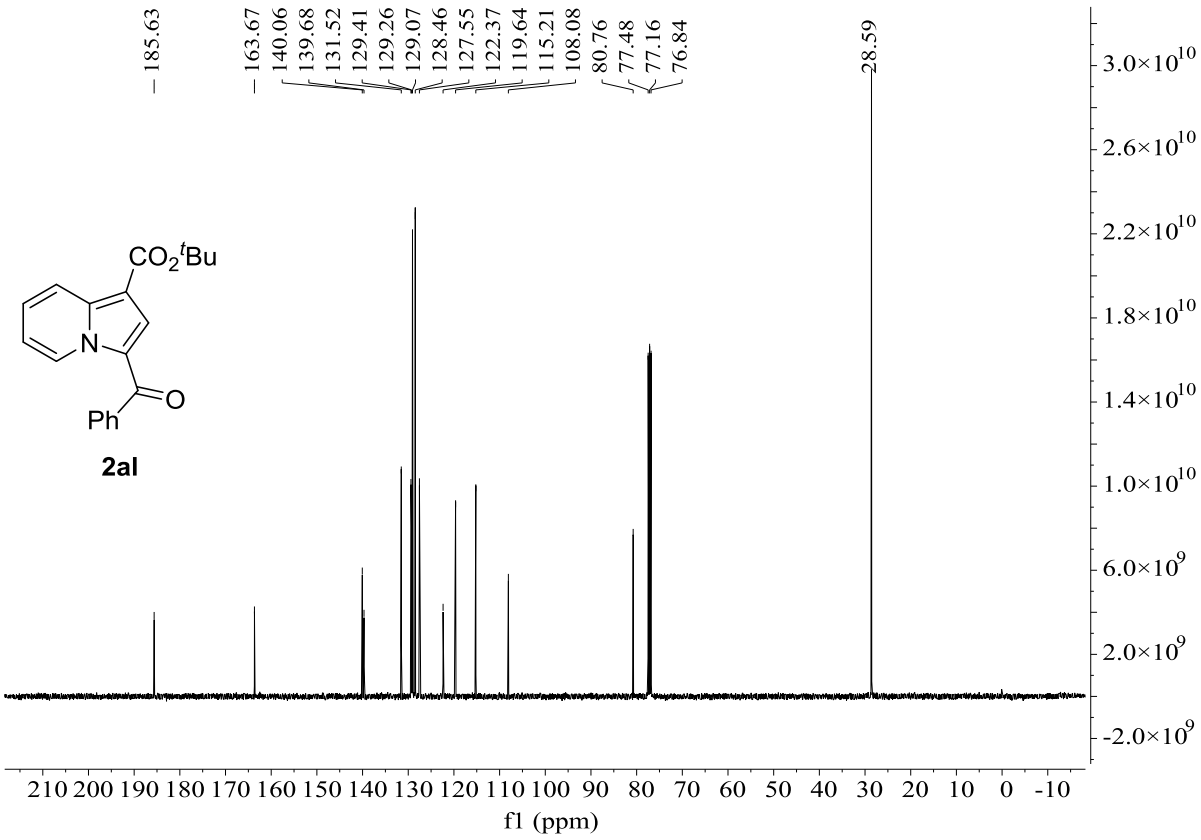
^{13}C NMR Spectrum of 2ak at 25 °C ($CDCl_3$, 100 MHz)



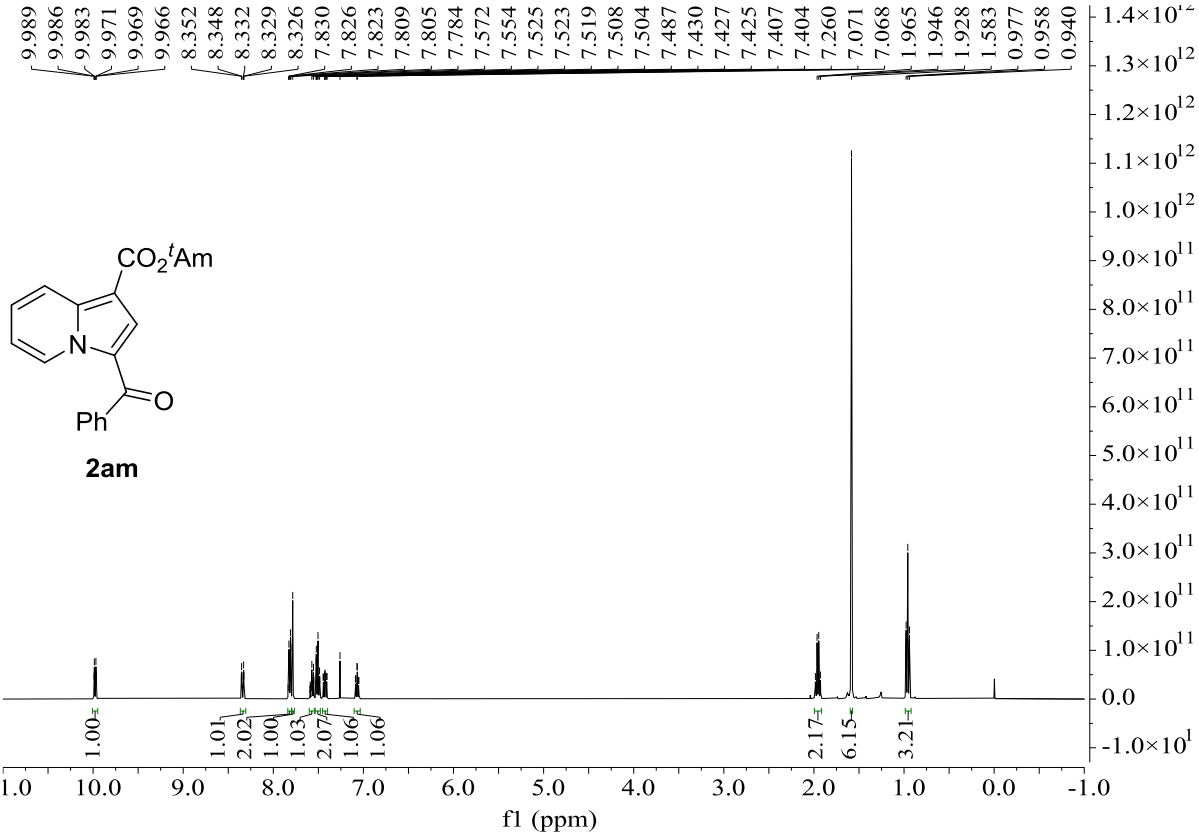
^{1}H NMR Spectrum of 2al at 25 °C ($CDCl_3$, 400 MHz)



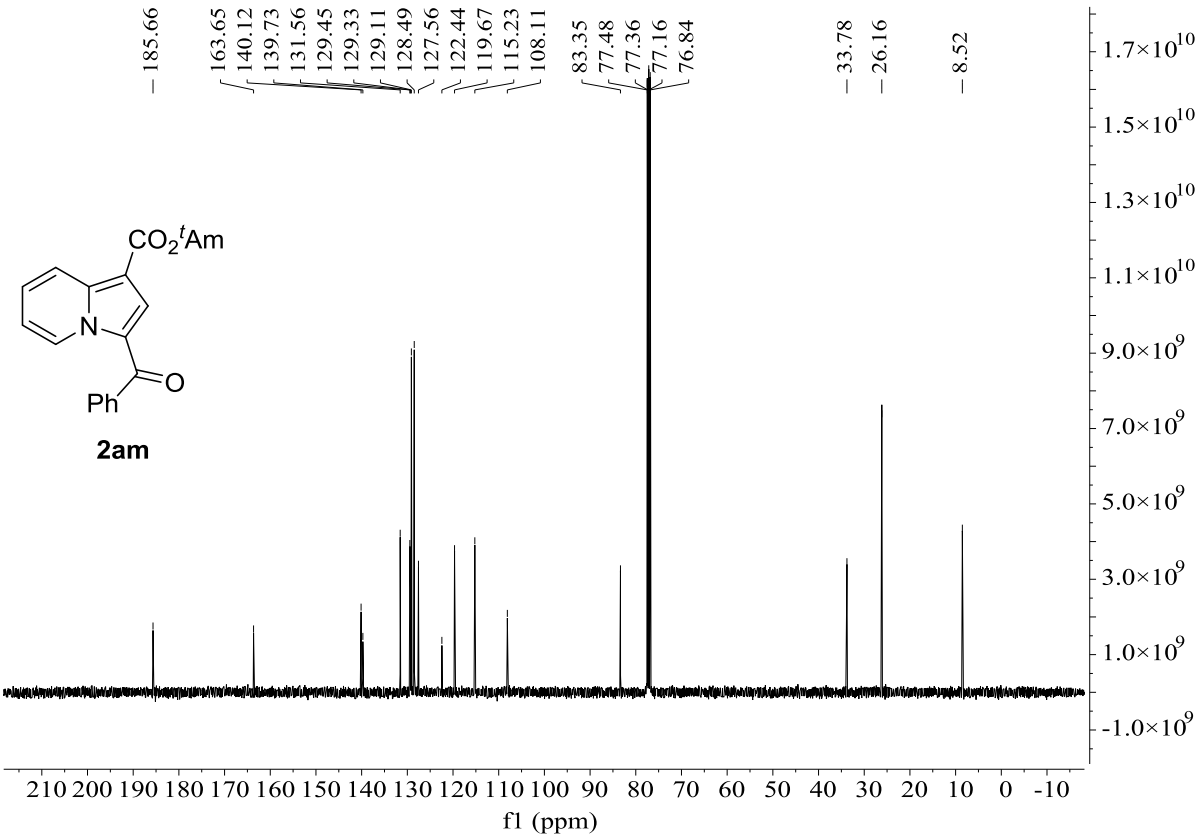
^{13}C NMR Spectrum of 2al at 25 °C ($CDCl_3$, 100 MHz)



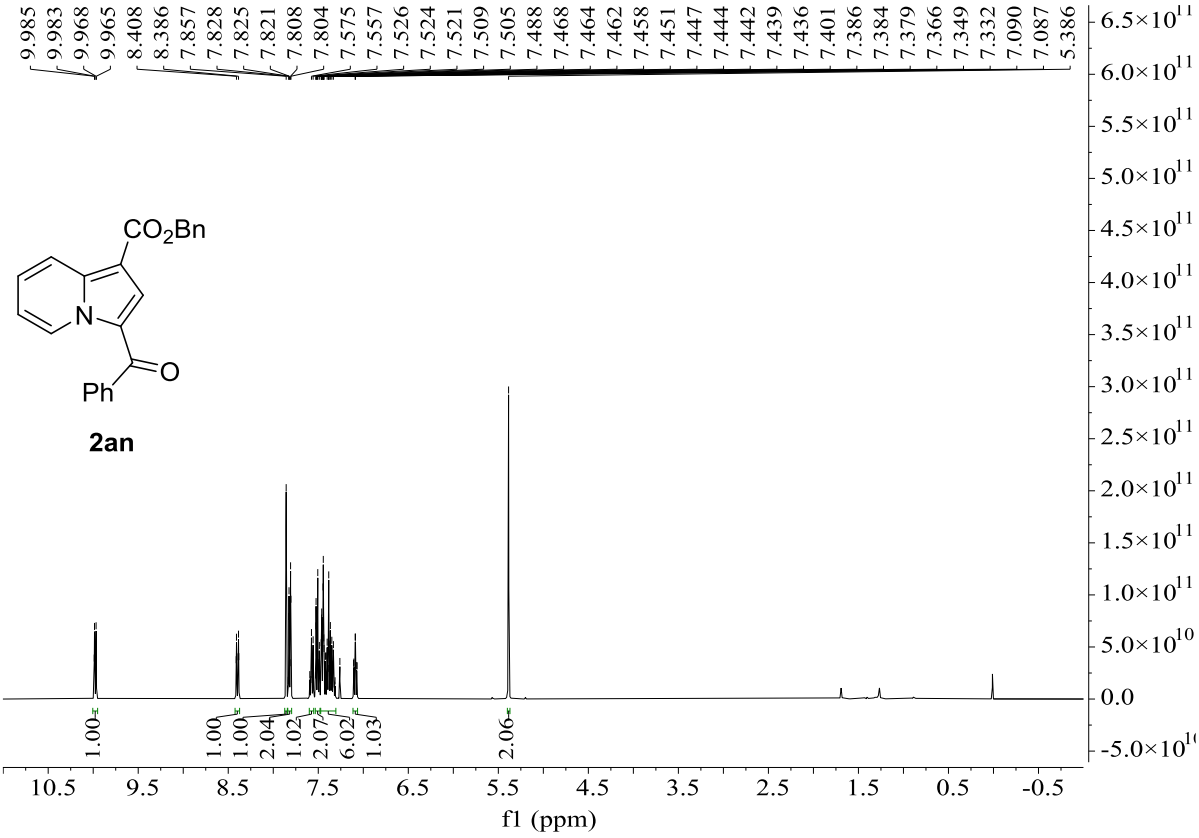
^{1}H NMR Spectrum of 2am at 25 °C ($CDCl_3$, 400 MHz)



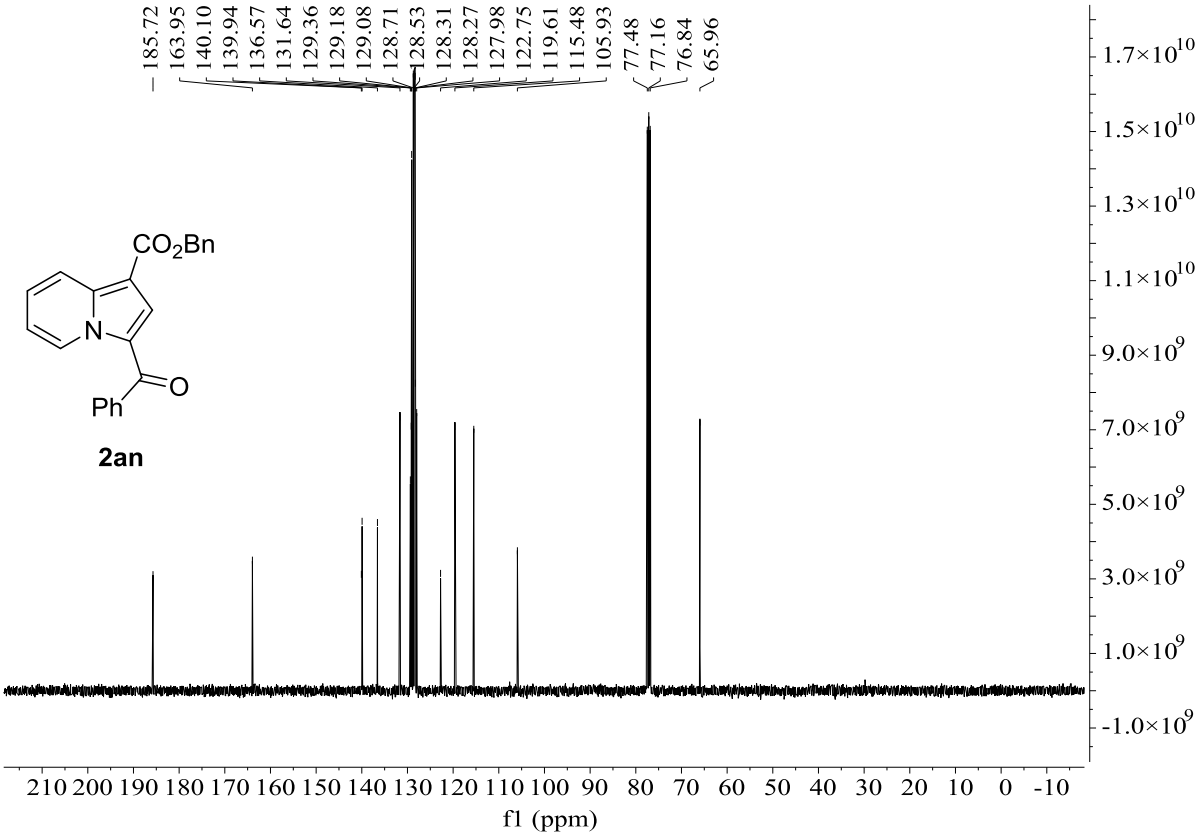
^{13}C NMR Spectrum of 2am at 25 °C ($CDCl_3$, 100 MHz)



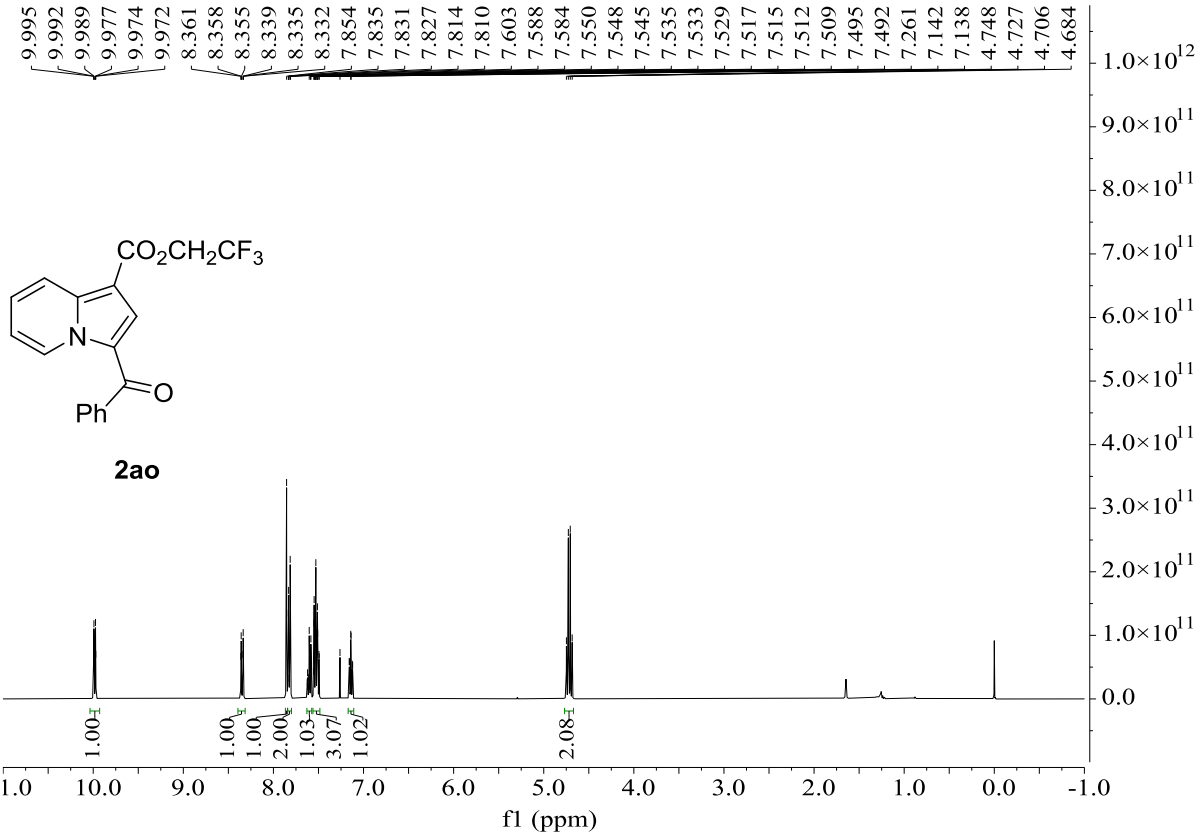
^{1}H NMR Spectrum of 2an at 25 °C ($CDCl_3$, 400 MHz)



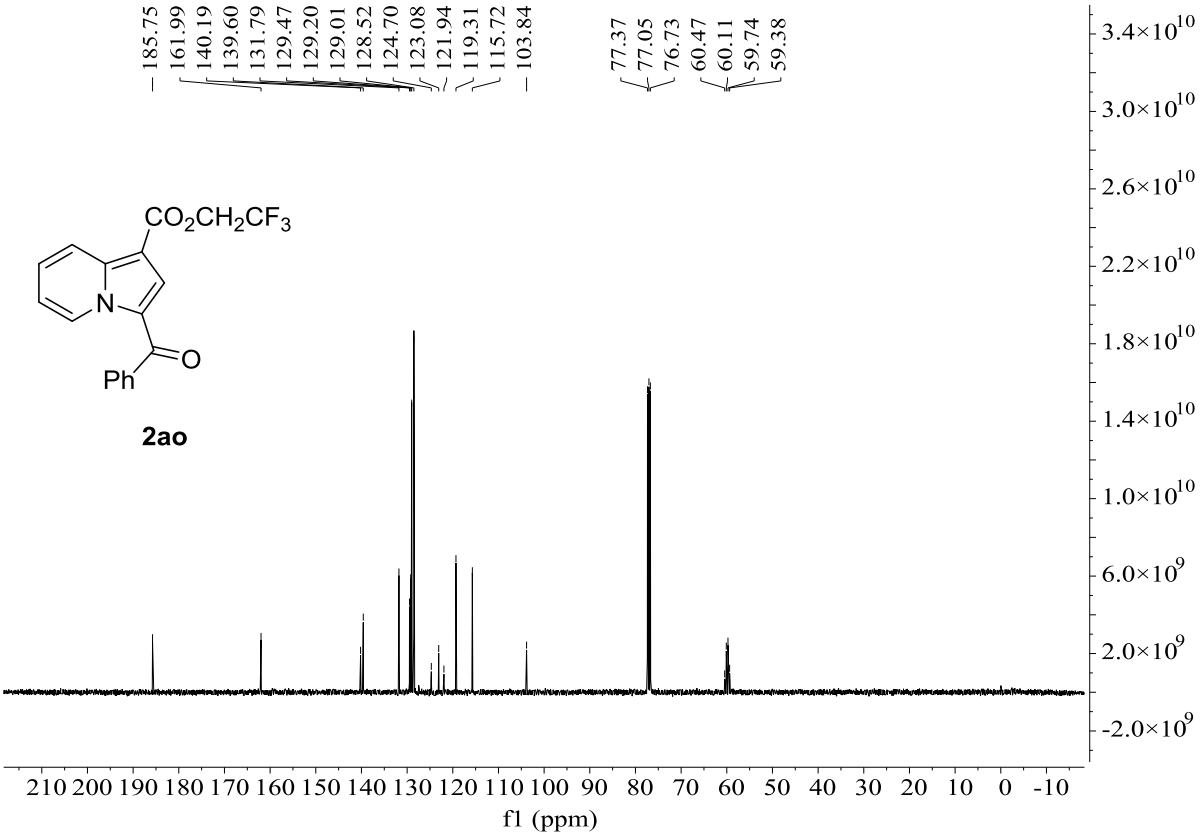
^{13}C NMR Spectrum of 2an at 25 °C ($CDCl_3$, 100 MHz)



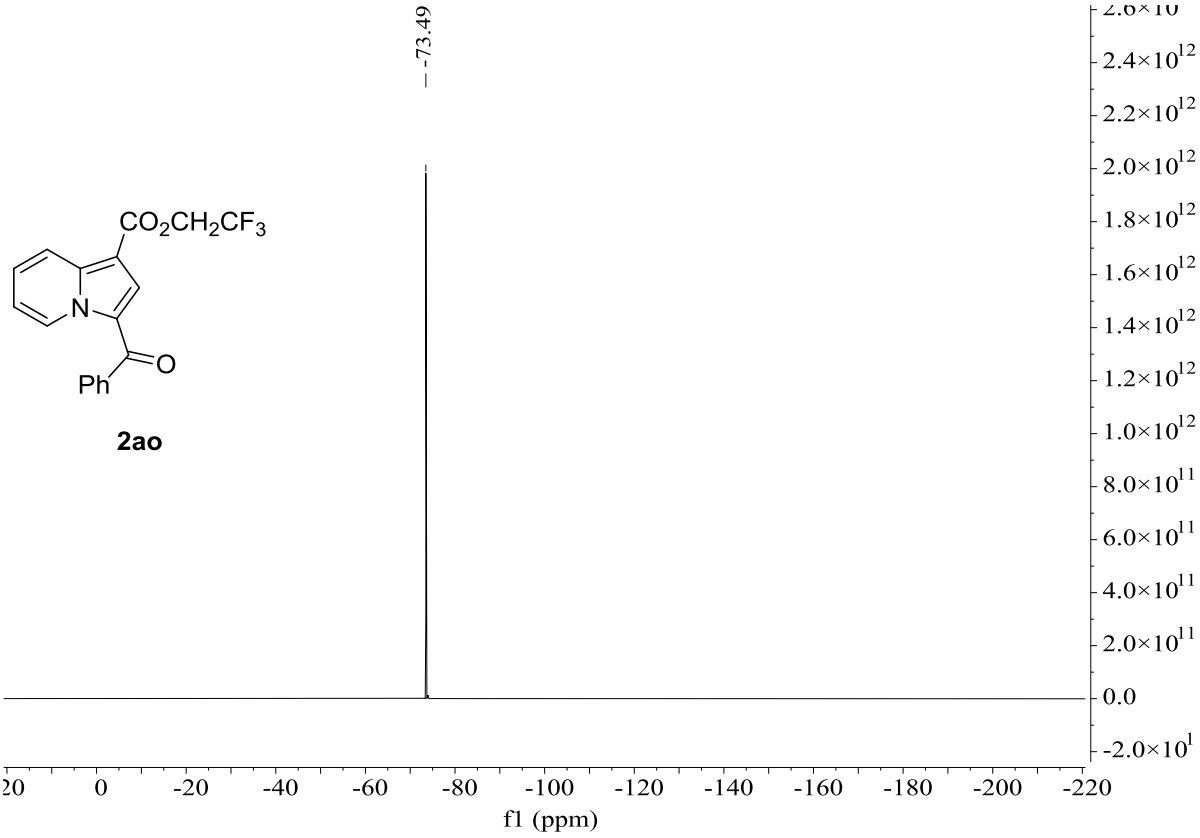
^{1}H NMR Spectrum of 2ao at 25 °C ($CDCl_3$, 400 MHz)



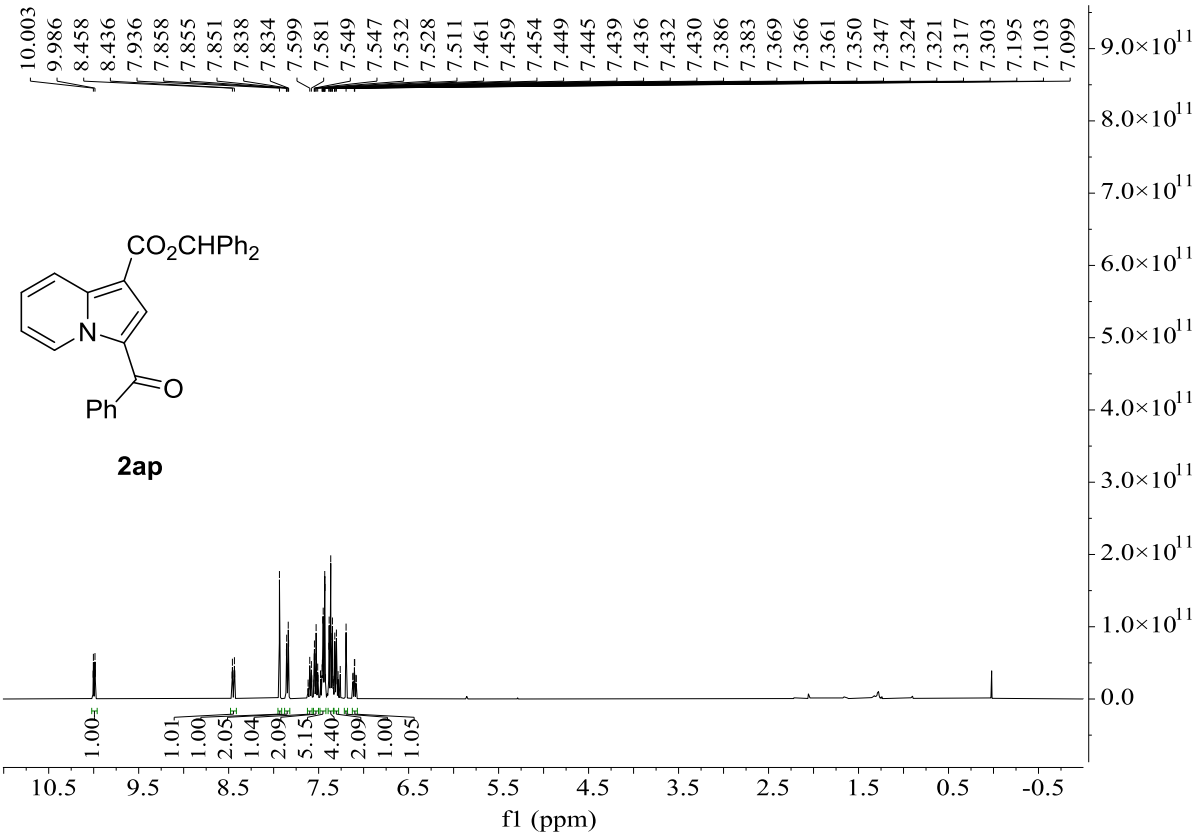
^{13}C NMR Spectrum of 2ao at 25 °C ($CDCl_3$, 100 MHz)



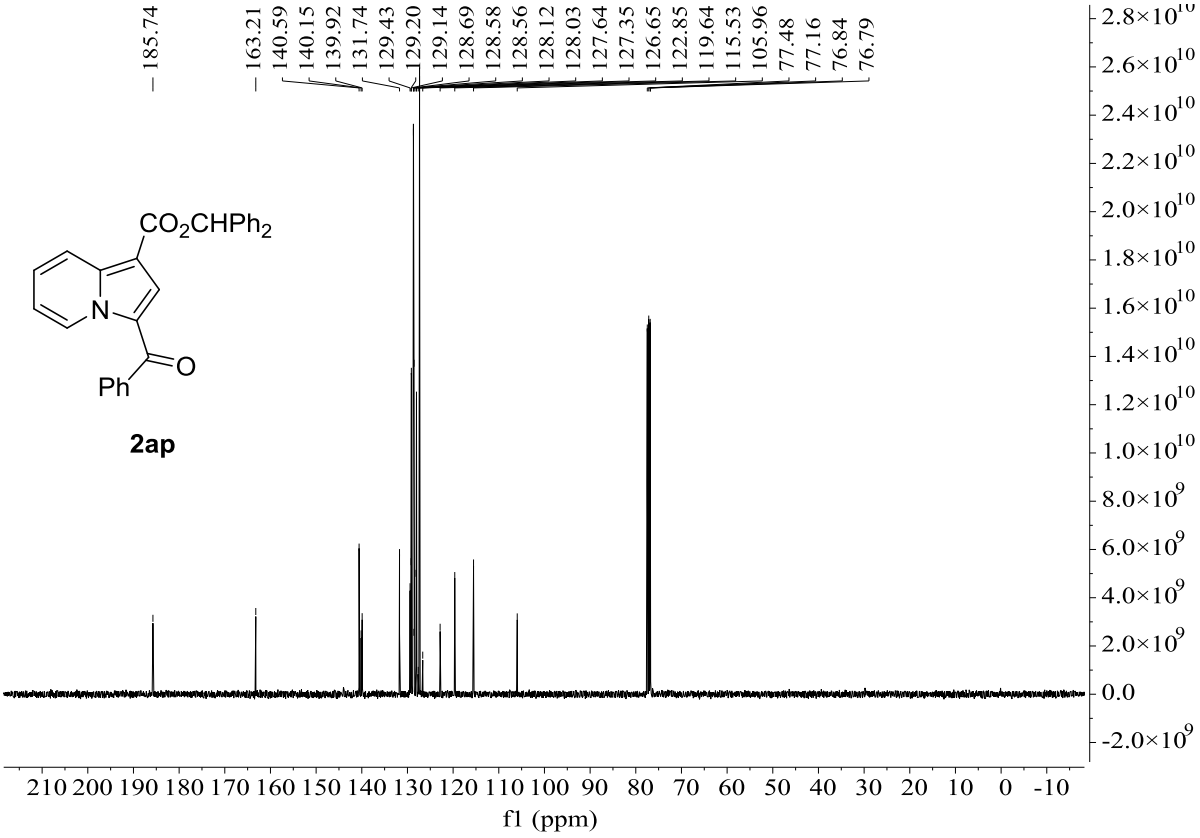
^{19}F NMR Spectrum of 2ao at 25 °C ($CDCl_3$, 376 MHz)



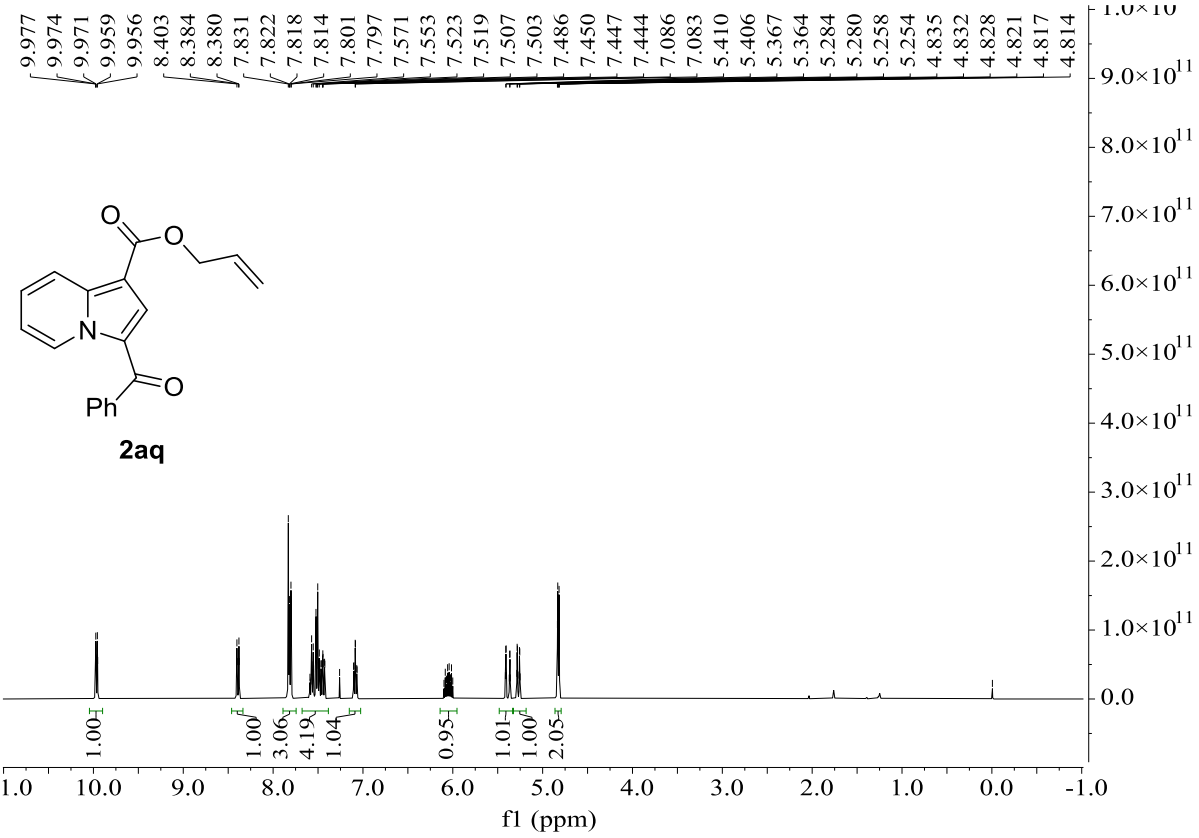
^{1}H NMR Spectrum of 2ap at 25 °C ($CDCl_3$, 400 MHz)



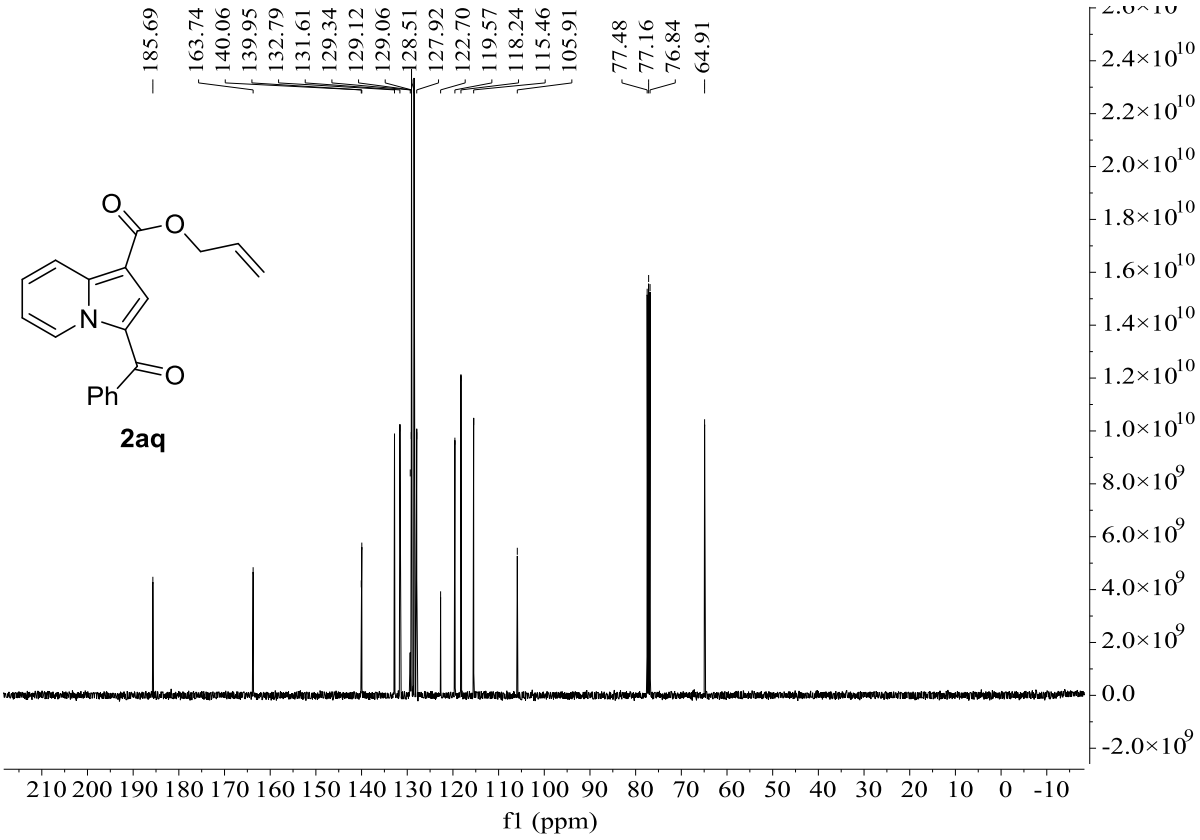
^{13}C NMR Spectrum of 2ap at 25 °C ($CDCl_3$, 100 MHz)



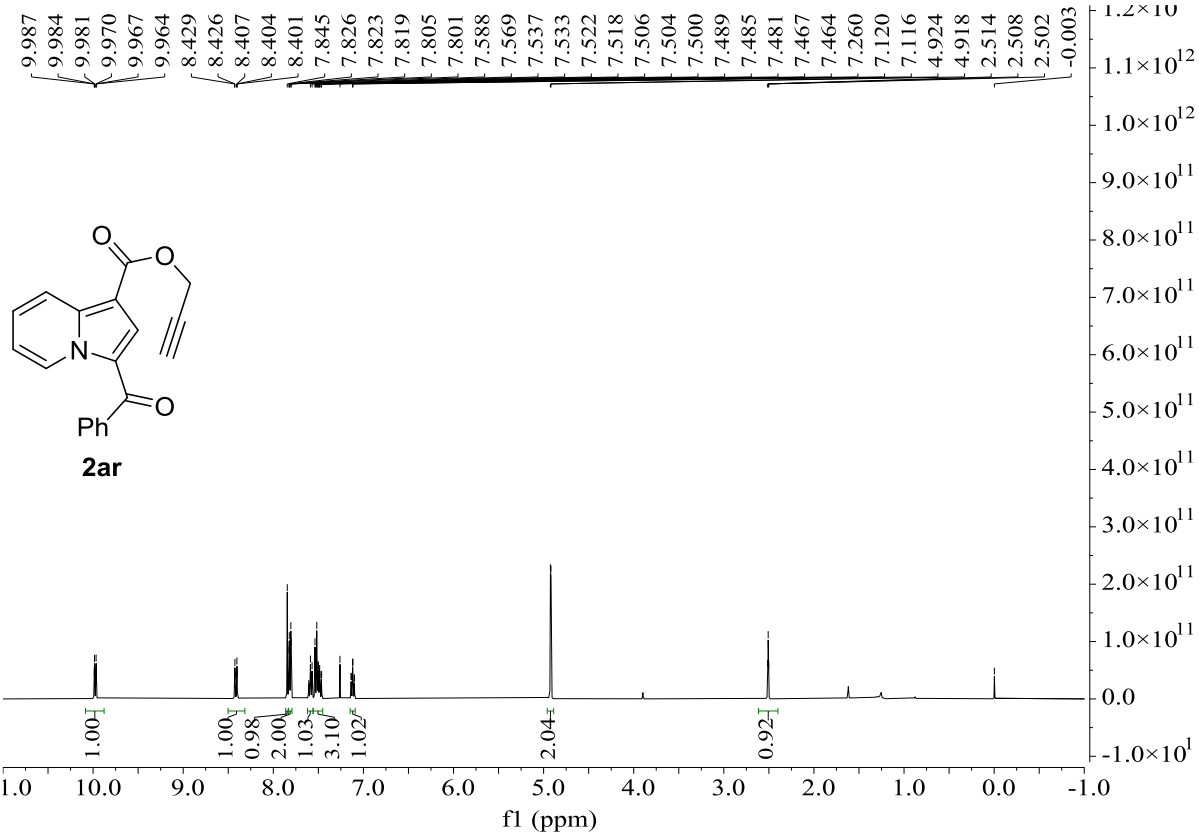
^{1}H NMR Spectrum of 2aq at 25 °C ($CDCl_3$, 400 MHz)



^{13}C NMR Spectrum of 2aq at 25 °C ($CDCl_3$, 100 MHz)

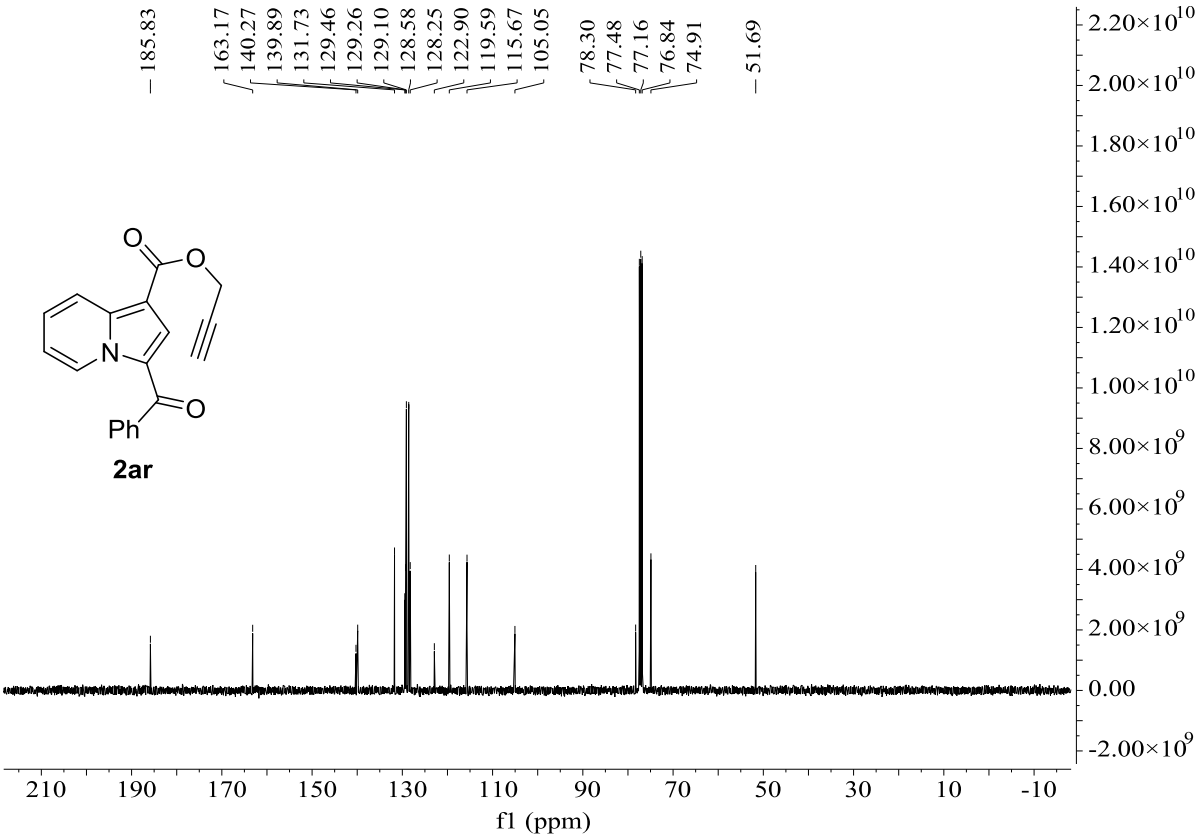


^{1}H NMR Spectrum of 2ar at 25 °C ($CDCl_3$, 400 MHz)



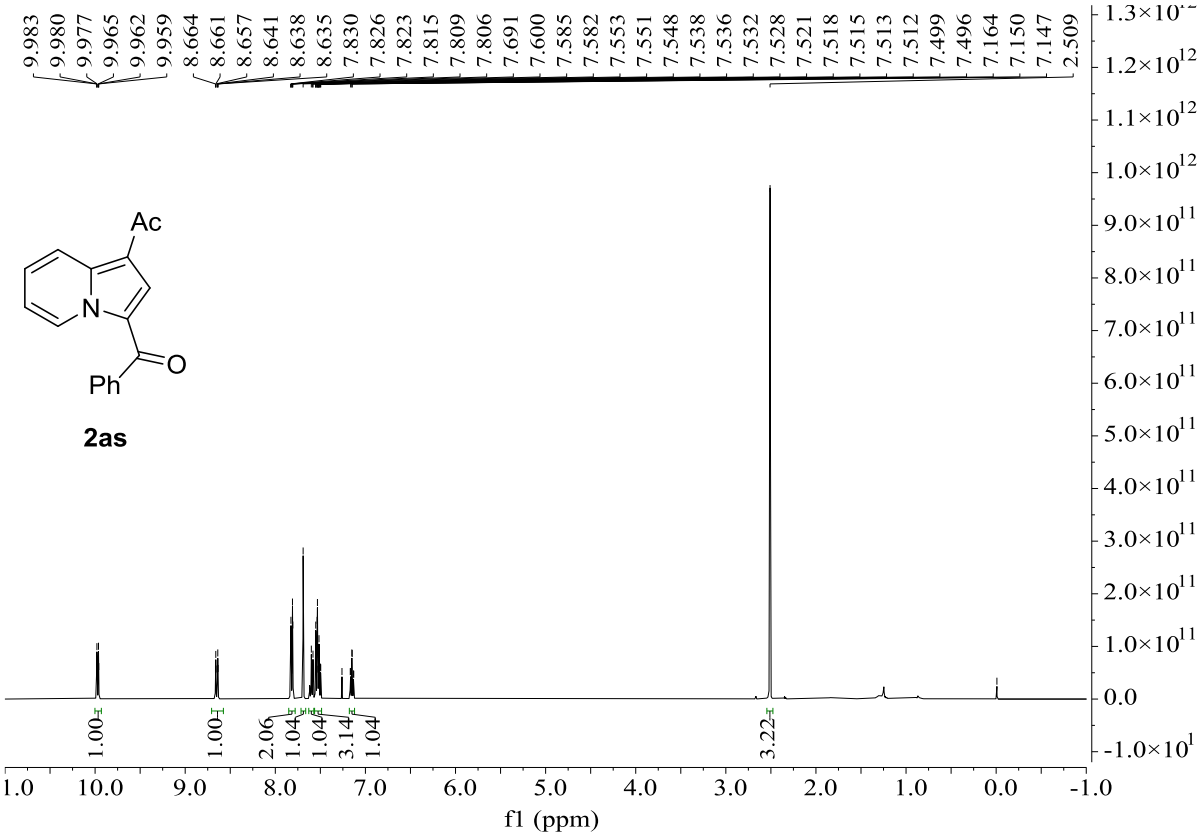
2ar

^{13}C NMR Spectrum of 2ar at 25 °C ($CDCl_3$, 100 MHz)

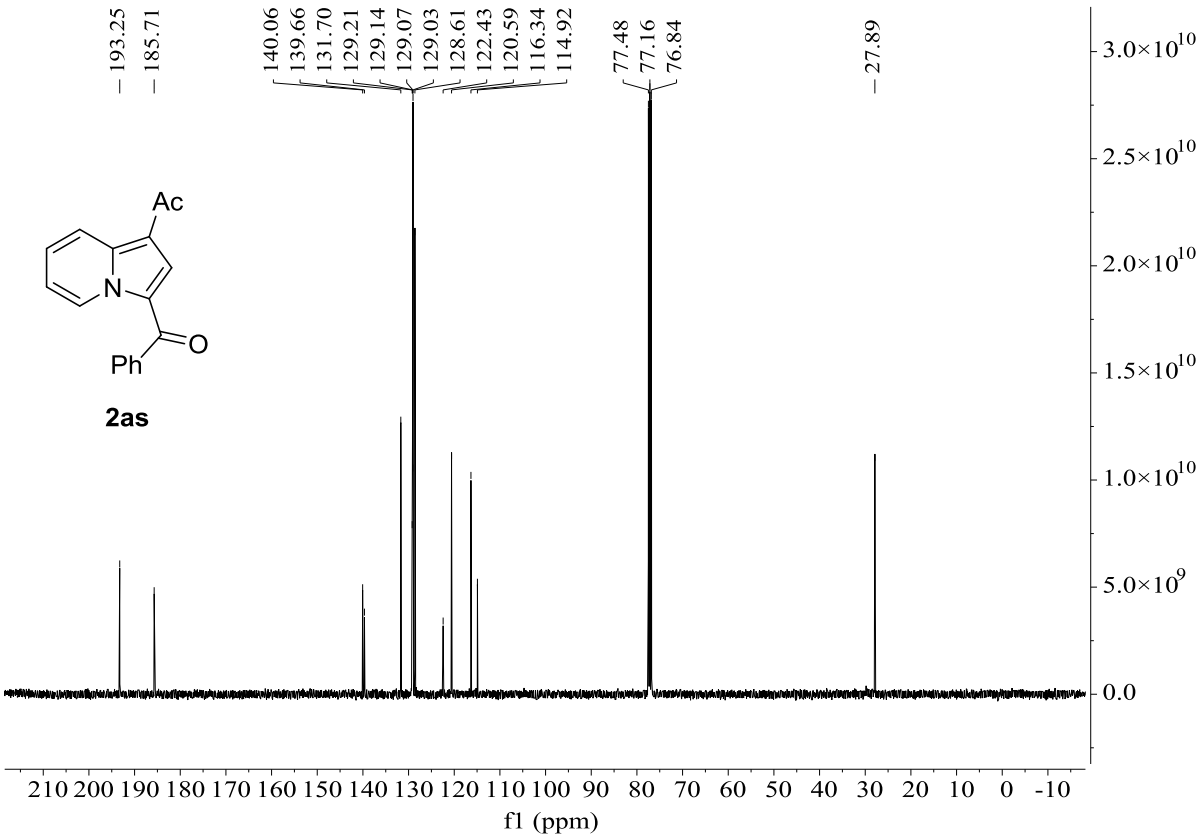


2ar

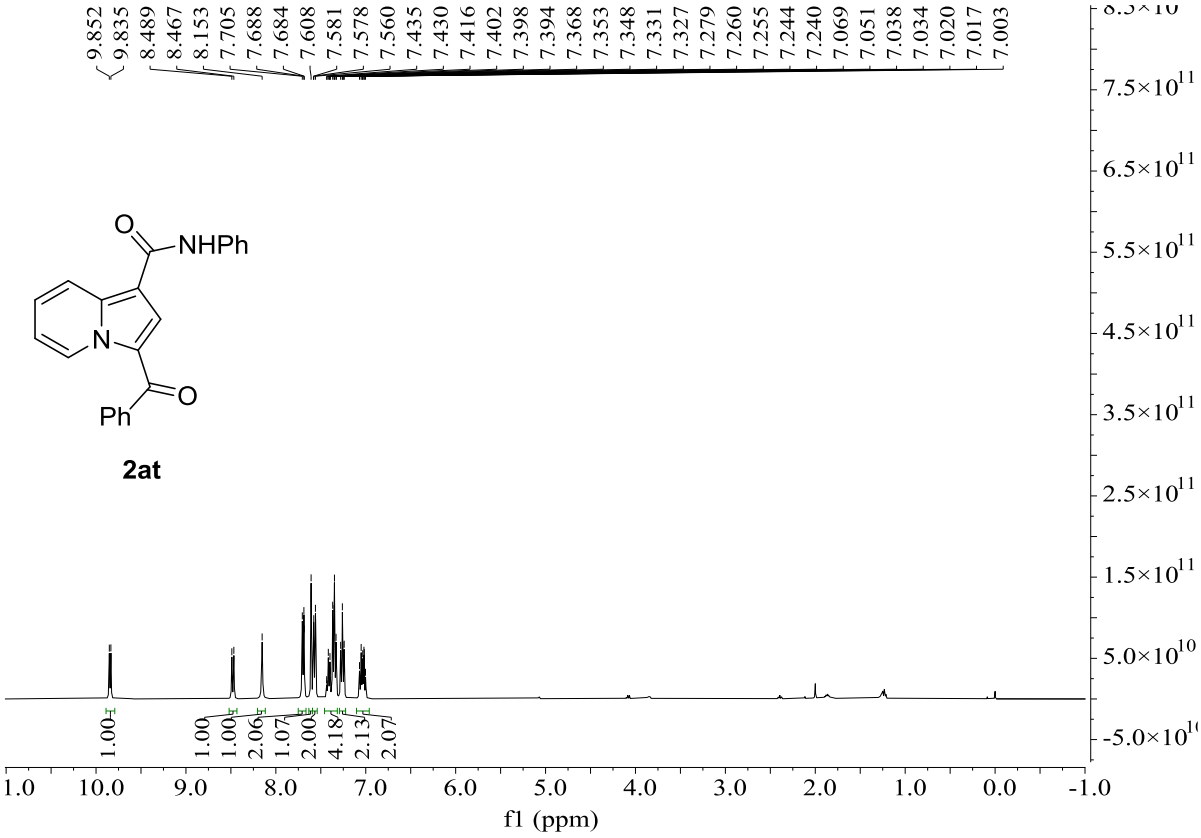
^{1}H NMR Spectrum of 2as at 25 °C ($CDCl_3$, 400 MHz)



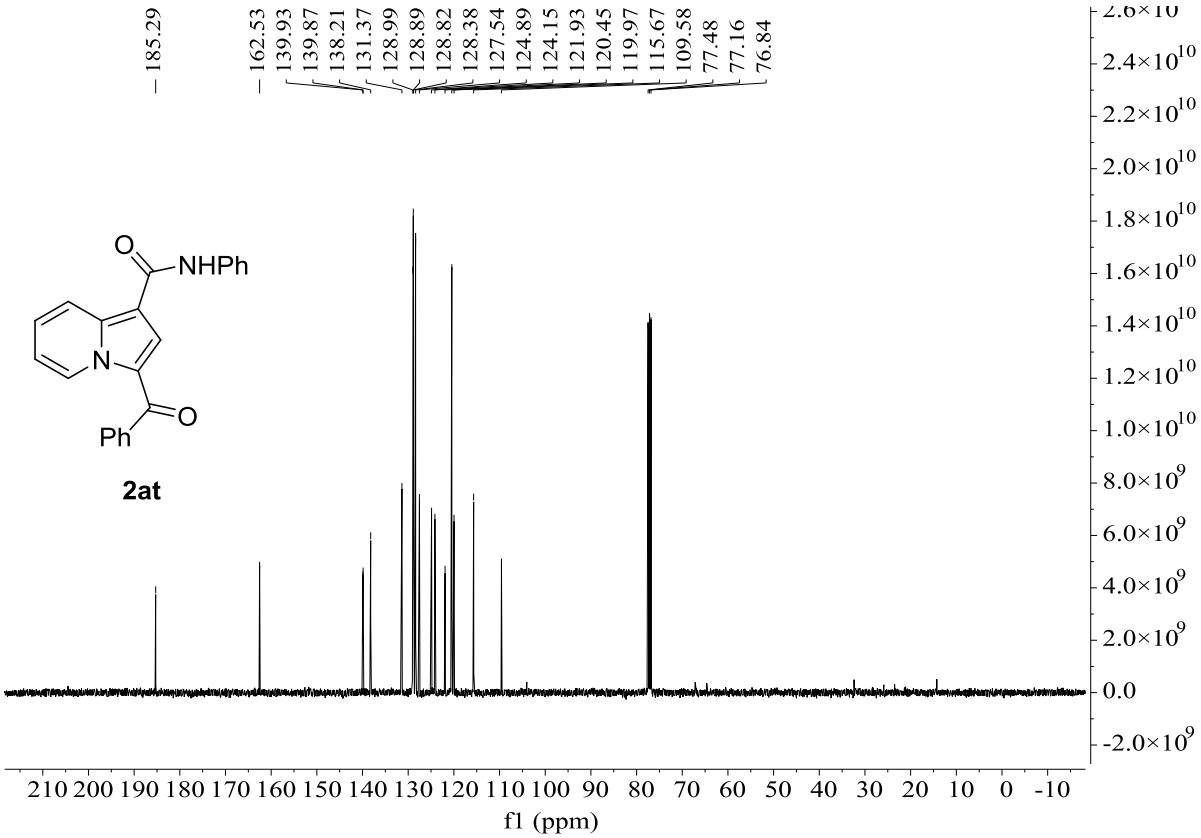
^{13}C NMR Spectrum of 2as at 25 °C ($CDCl_3$, 100 MHz)



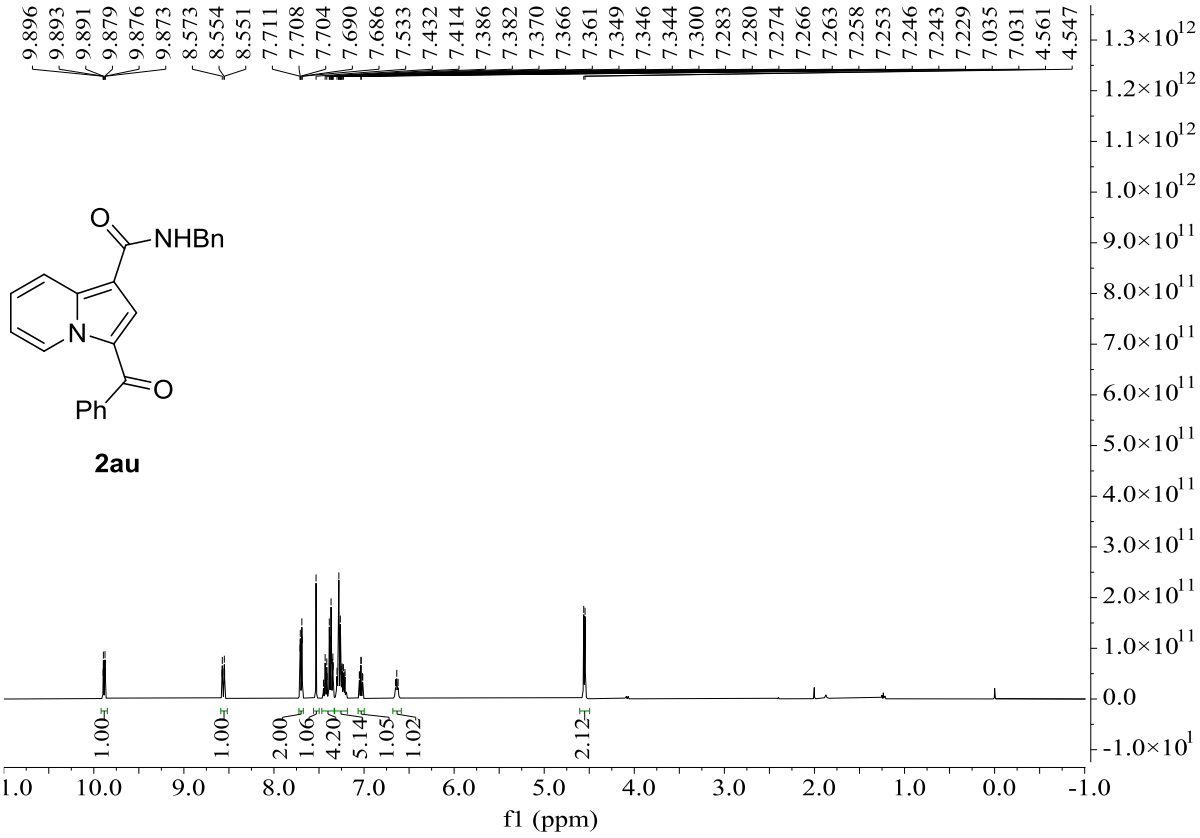
^{1}H NMR Spectrum of 2at at 25 °C ($CDCl_3$, 400 MHz)



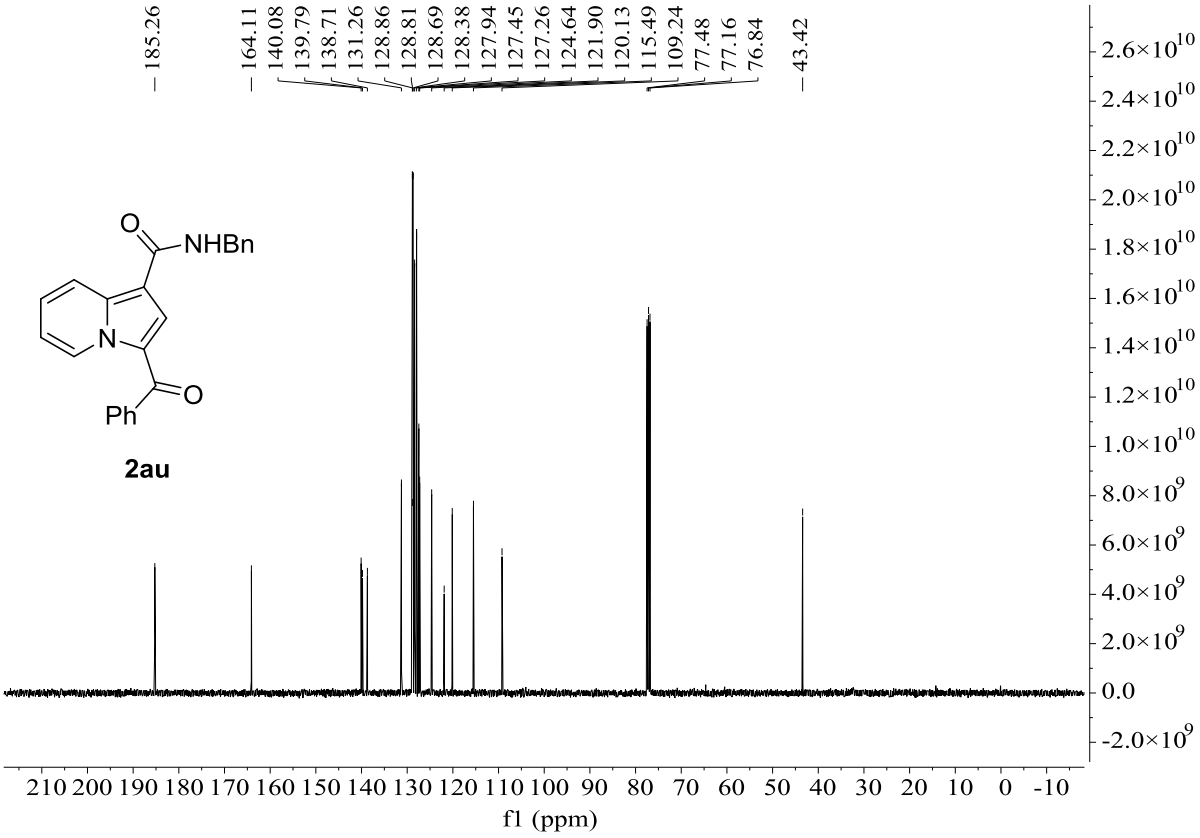
^{13}C NMR Spectrum of 2at at 25 °C ($CDCl_3$, 100 MHz)



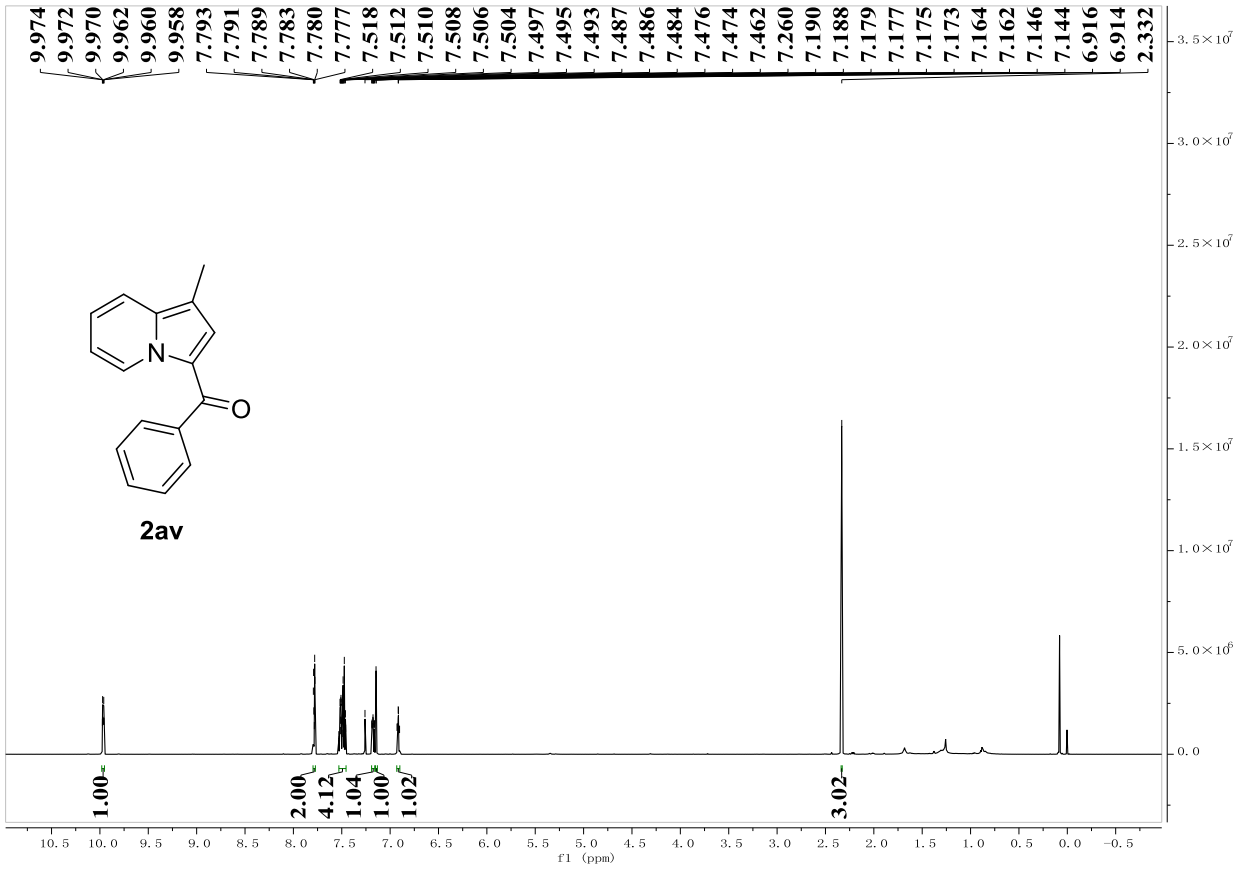
^{1}H NMR Spectrum of 2au at 25 °C ($CDCl_3$, 400 MHz)



^{13}C NMR Spectrum of 2au at 25 °C ($CDCl_3$, 100 MHz)

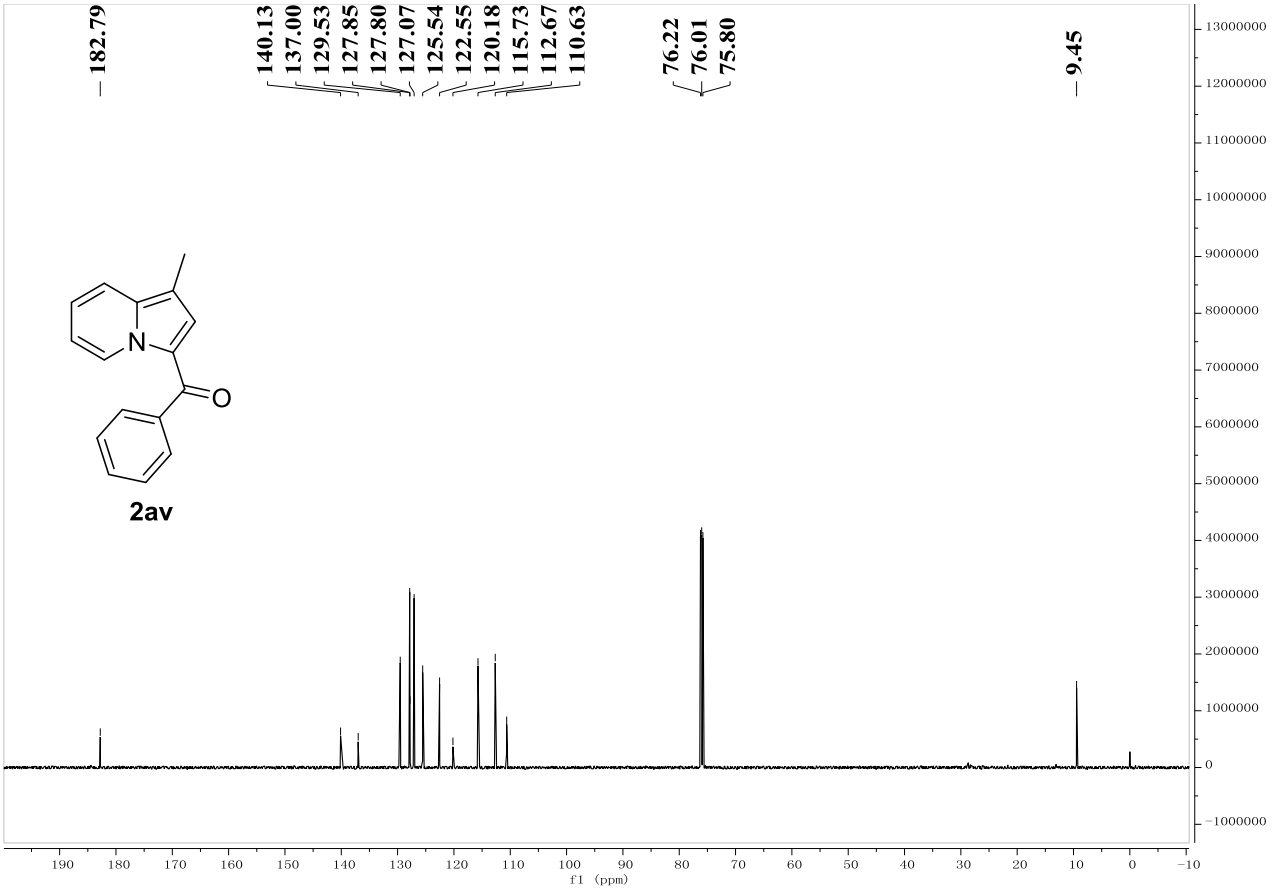


^{1}H NMR Spectrum of 2av at 25 °C ($CDCl_3$, 600 MHz)



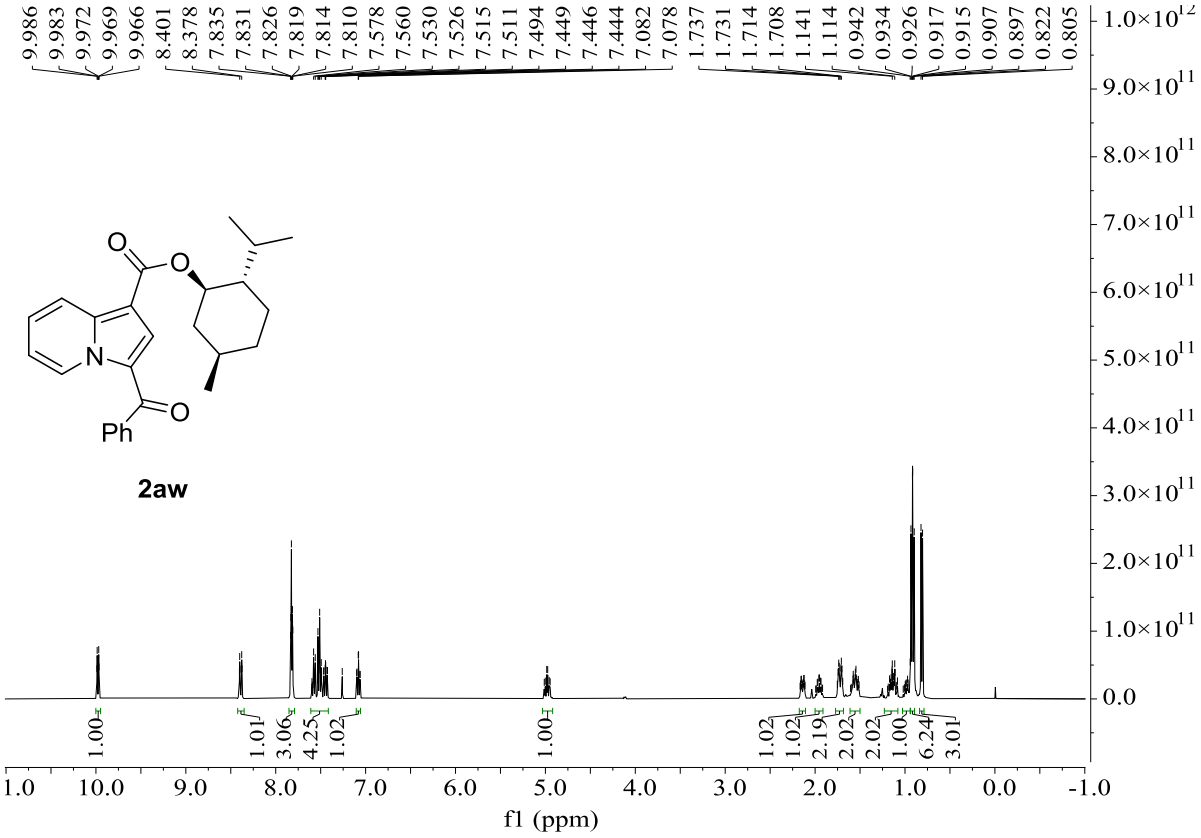
2av

^{13}C NMR Spectrum of 2av at 25 °C ($CDCl_3$, 150 MHz)

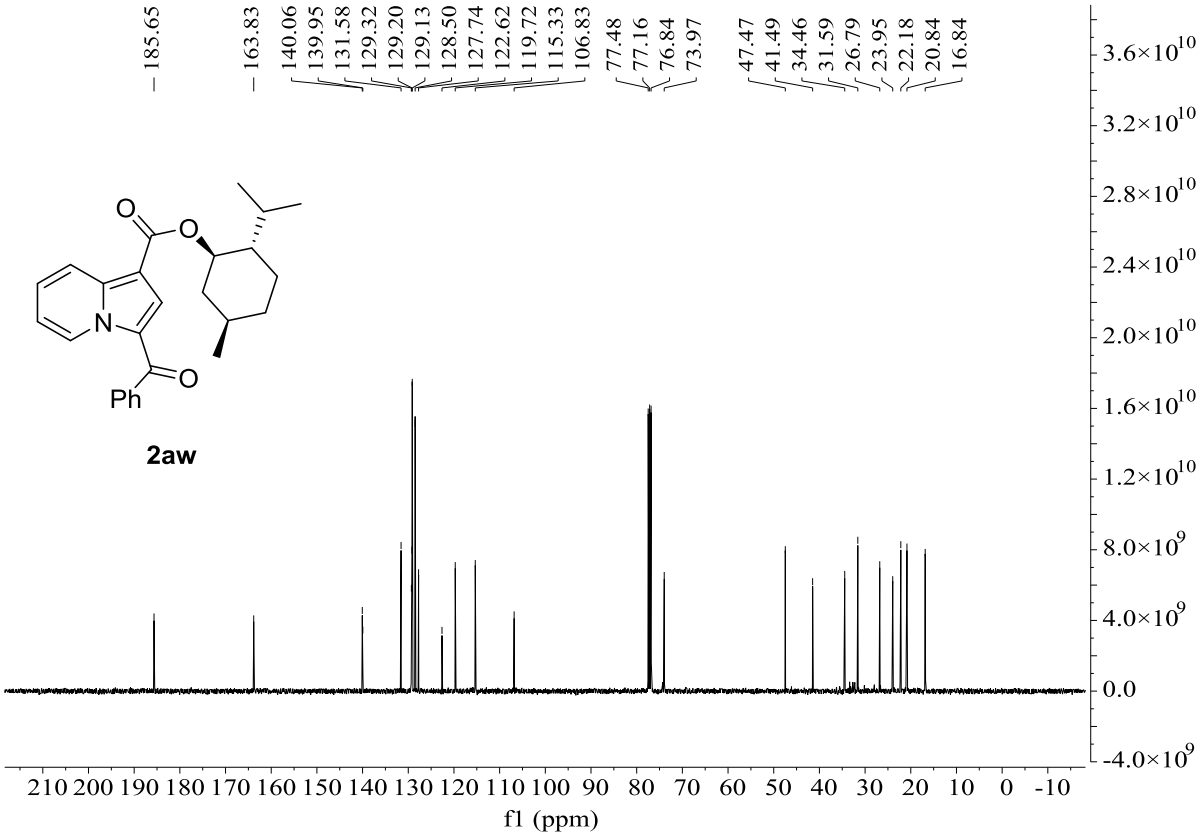


2av

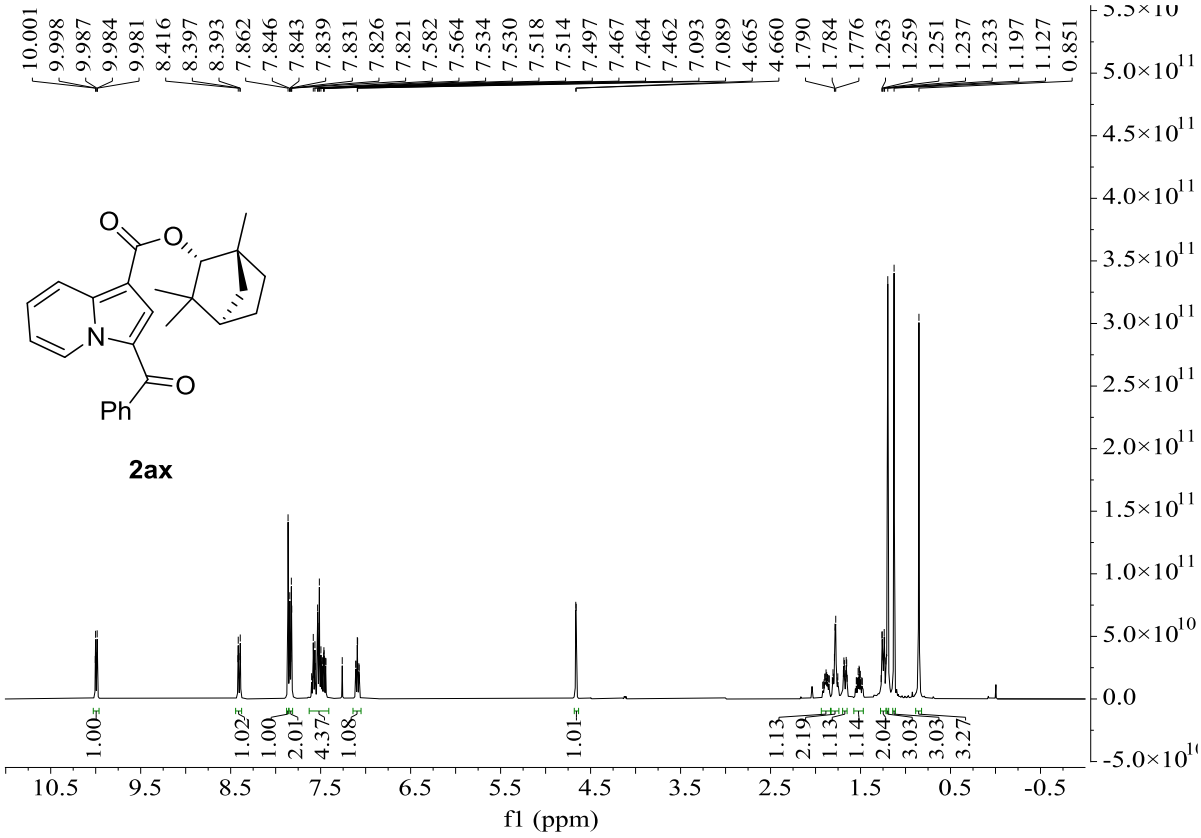
^{1}H NMR Spectrum of 2aw at 25 °C ($CDCl_3$, 400 MHz)



^{13}C NMR Spectrum of 2aw at 25 °C ($CDCl_3$, 100 MHz)

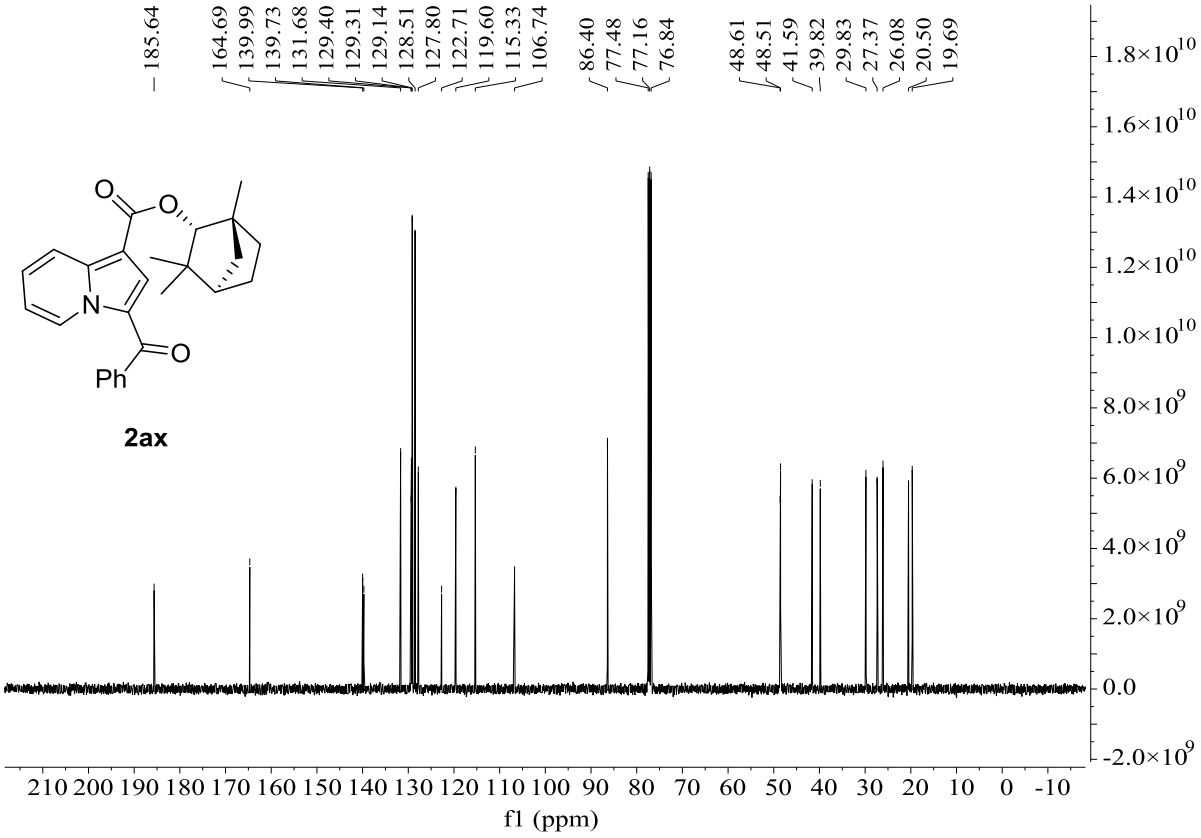


^{1}H NMR Spectrum of 2ax at 25 °C ($CDCl_3$, 400 MHz)



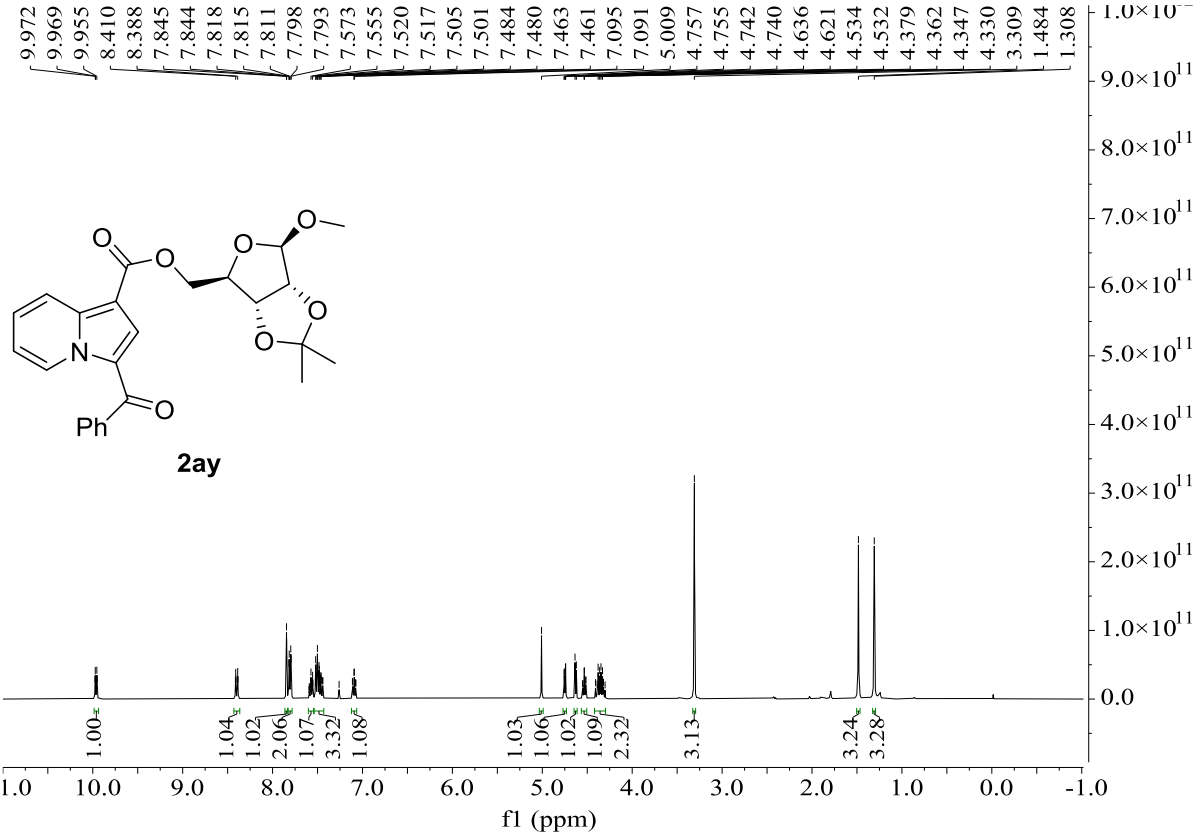
2ax

^{13}C NMR Spectrum of 2ax at 25 °C ($CDCl_3$, 100 MHz)

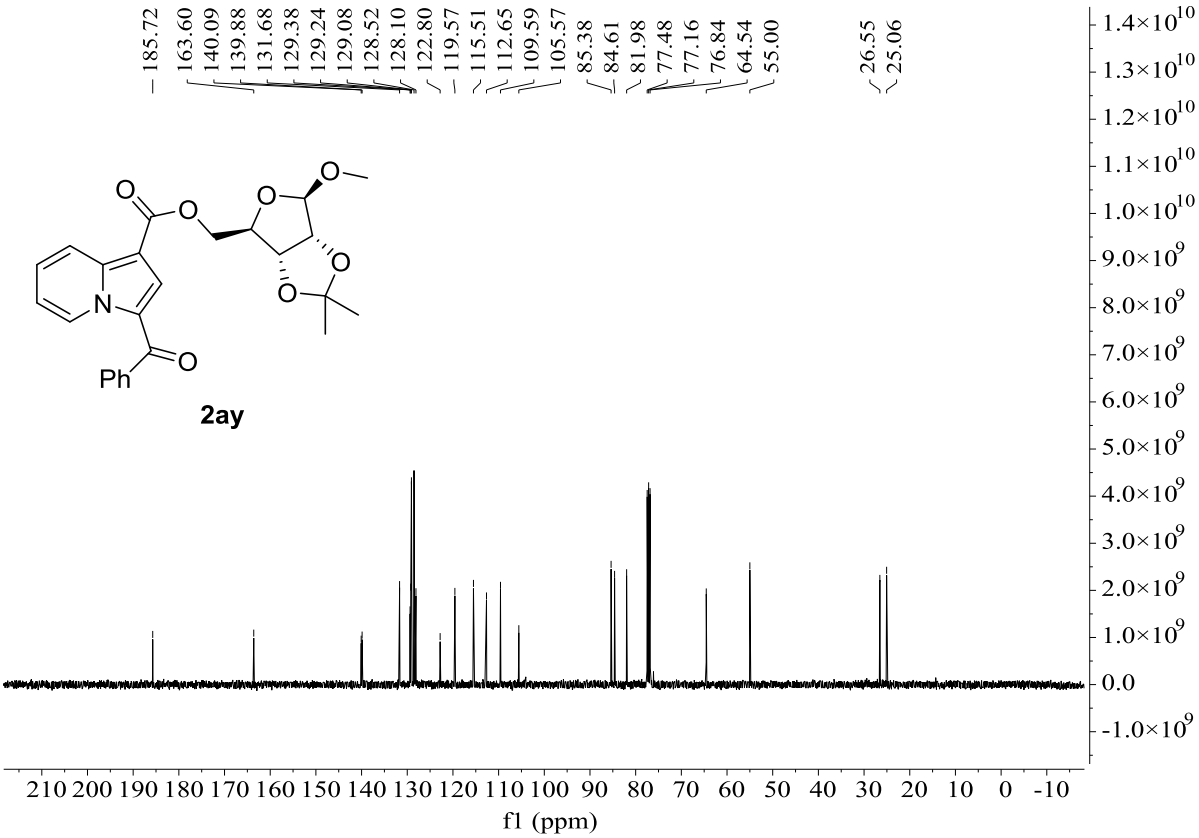


2ax

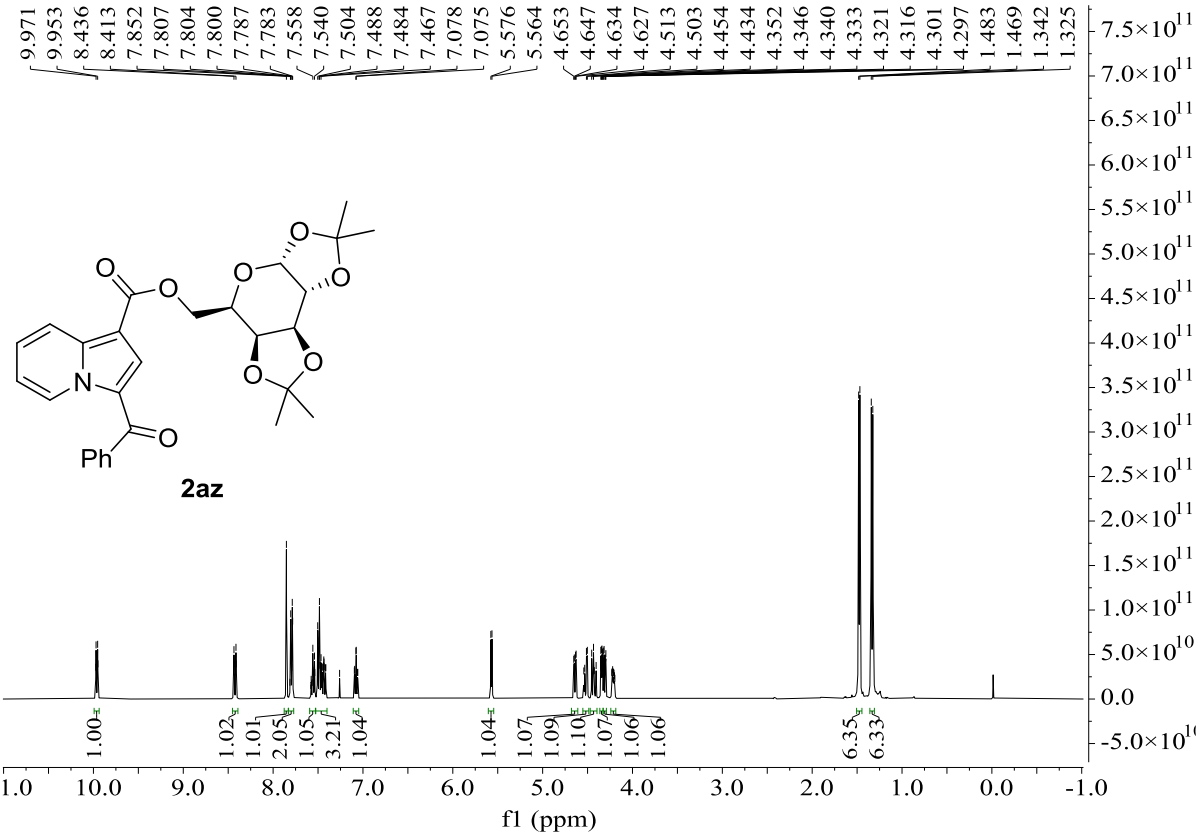
^{1}H NMR Spectrum of 2ay at 25 °C ($CDCl_3$, 400 MHz)



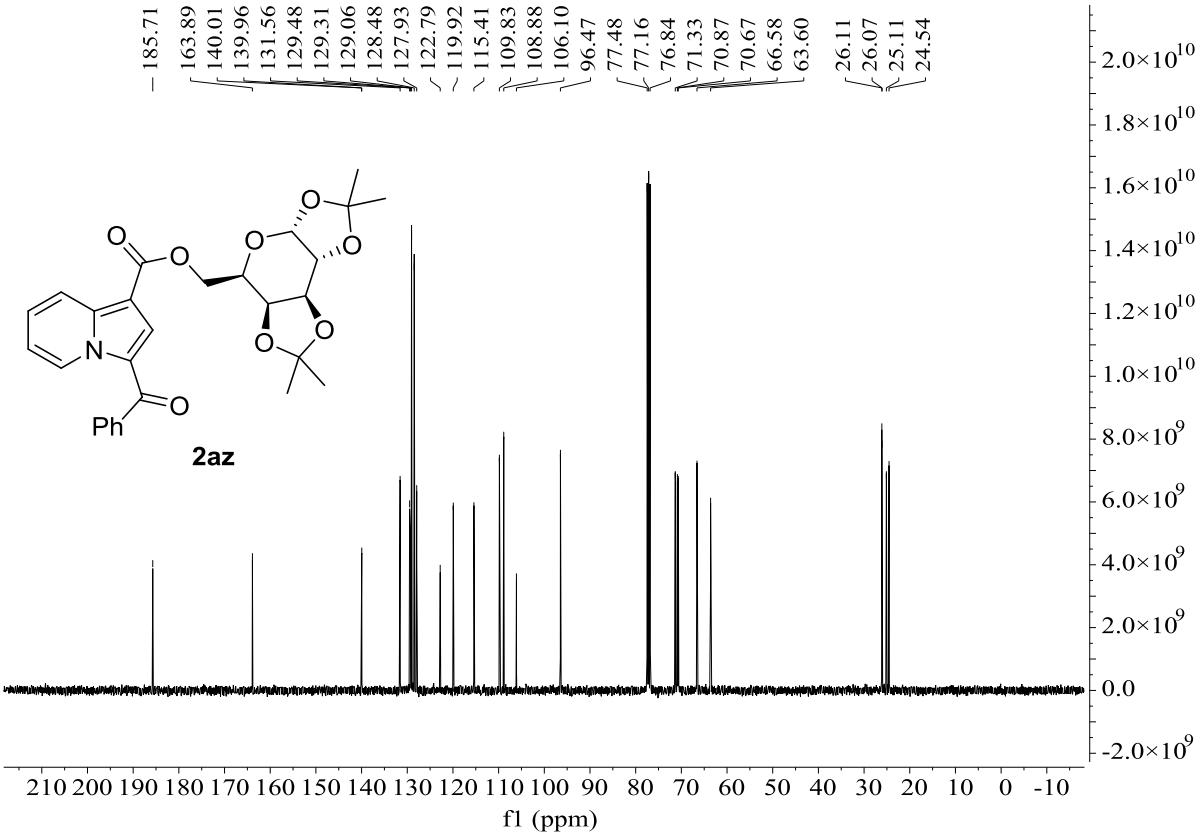
^{13}C NMR Spectrum of 2ay at 25 °C ($CDCl_3$, 100 MHz)



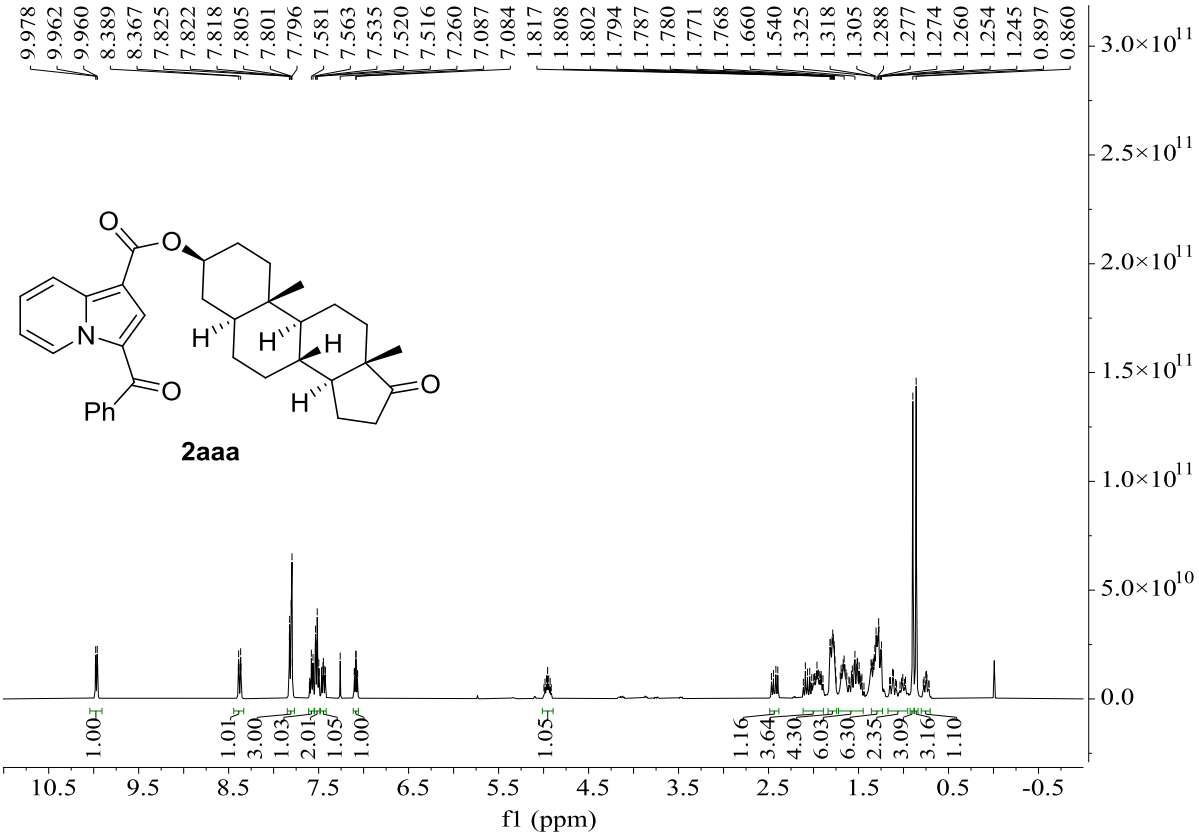
^{1}H NMR Spectrum of 2az at 25 °C ($CDCl_3$, 400 MHz)



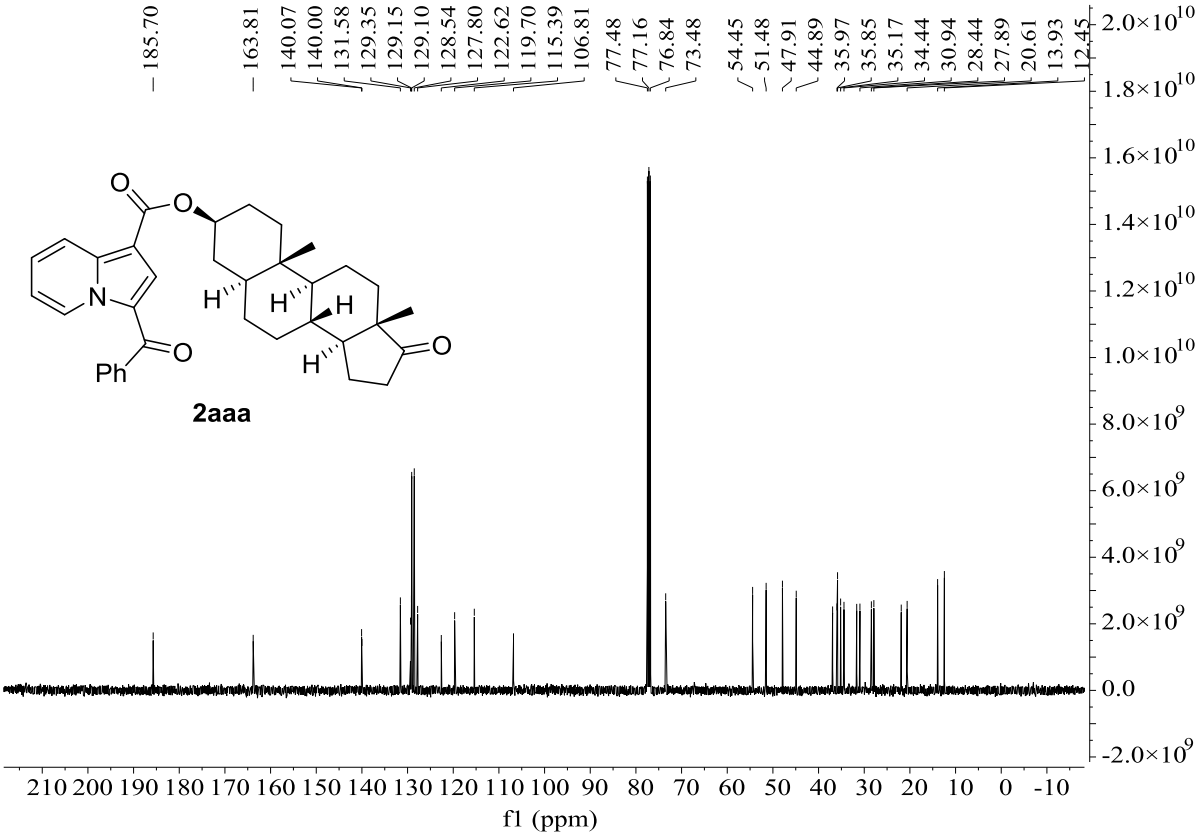
^{13}C NMR Spectrum of 2az at 25 °C ($CDCl_3$, 100 MHz)



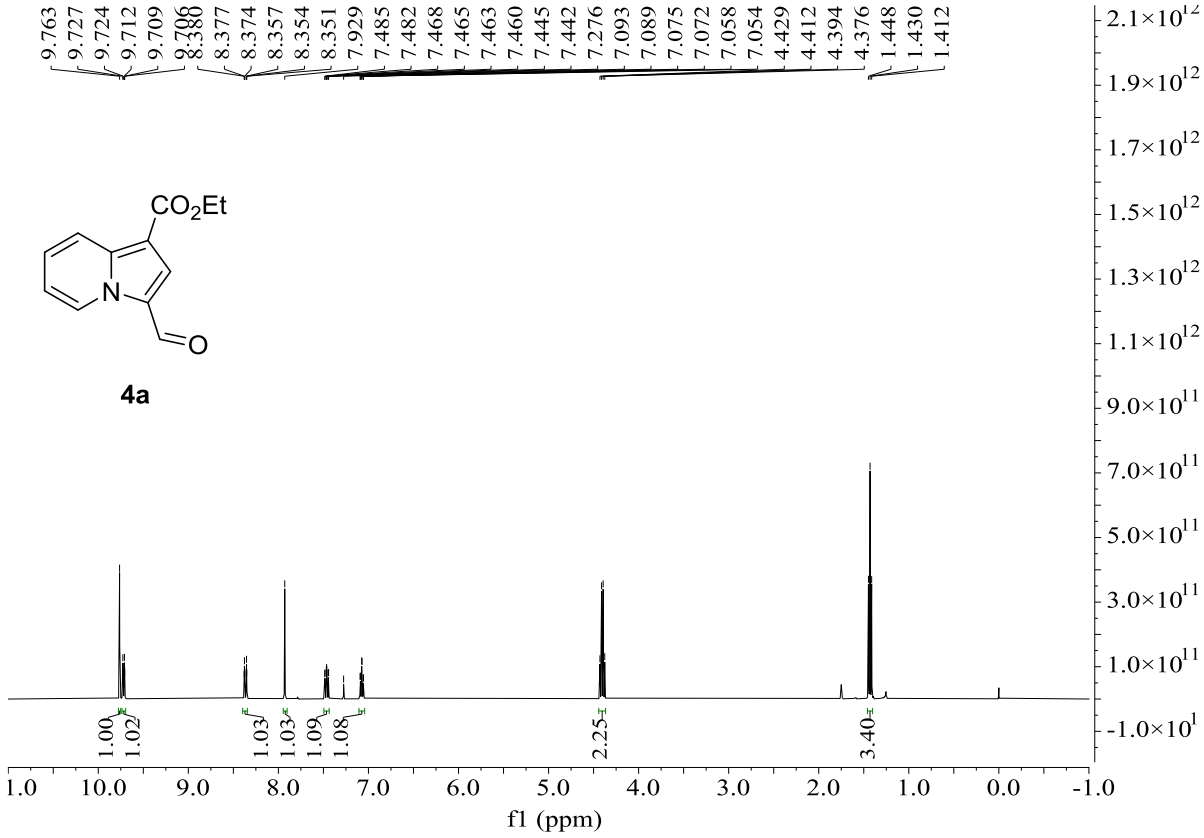
^{1}H NMR Spectrum of 2aaa at 25 °C ($CDCl_3$, 400 MHz)



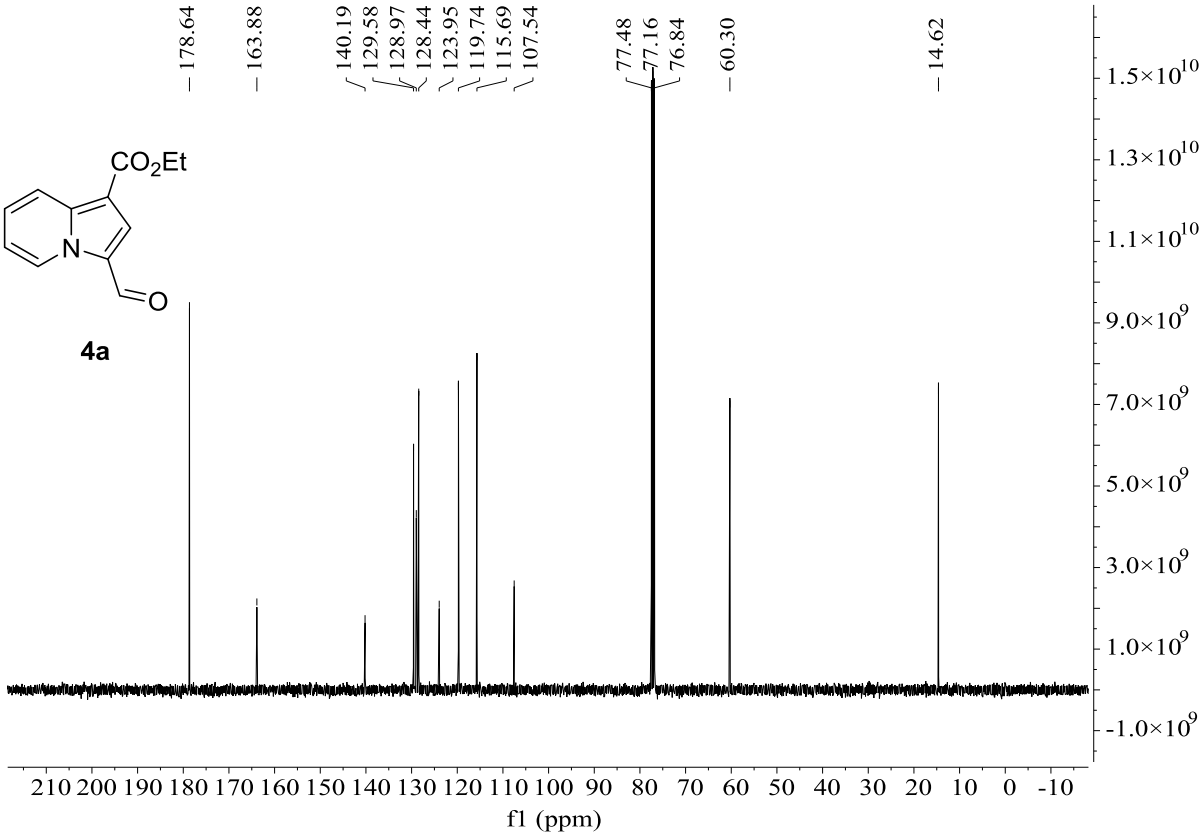
^{13}C NMR Spectrum of 2aaa at 25 °C ($CDCl_3$, 100 MHz)



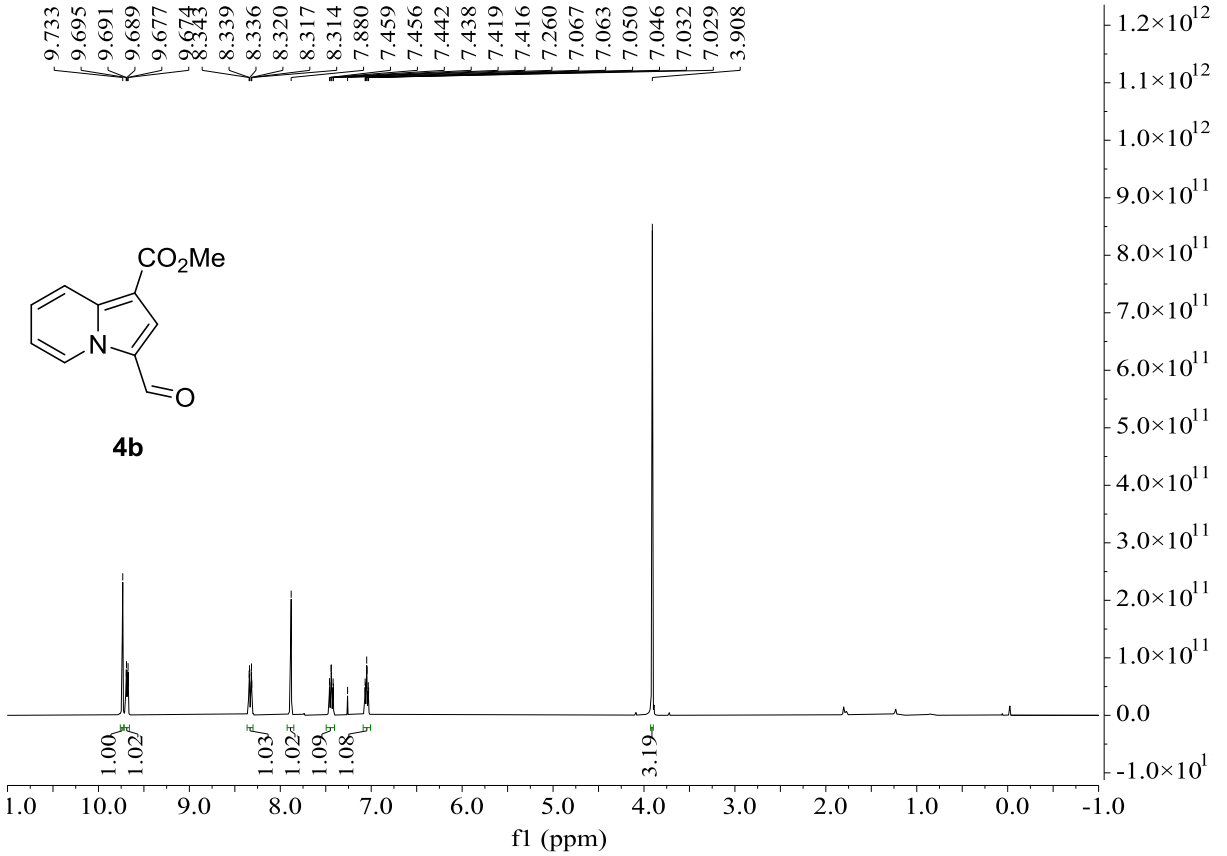
^{1}H NMR Spectrum of 4a at 25 °C ($CDCl_3$, 400 MHz)



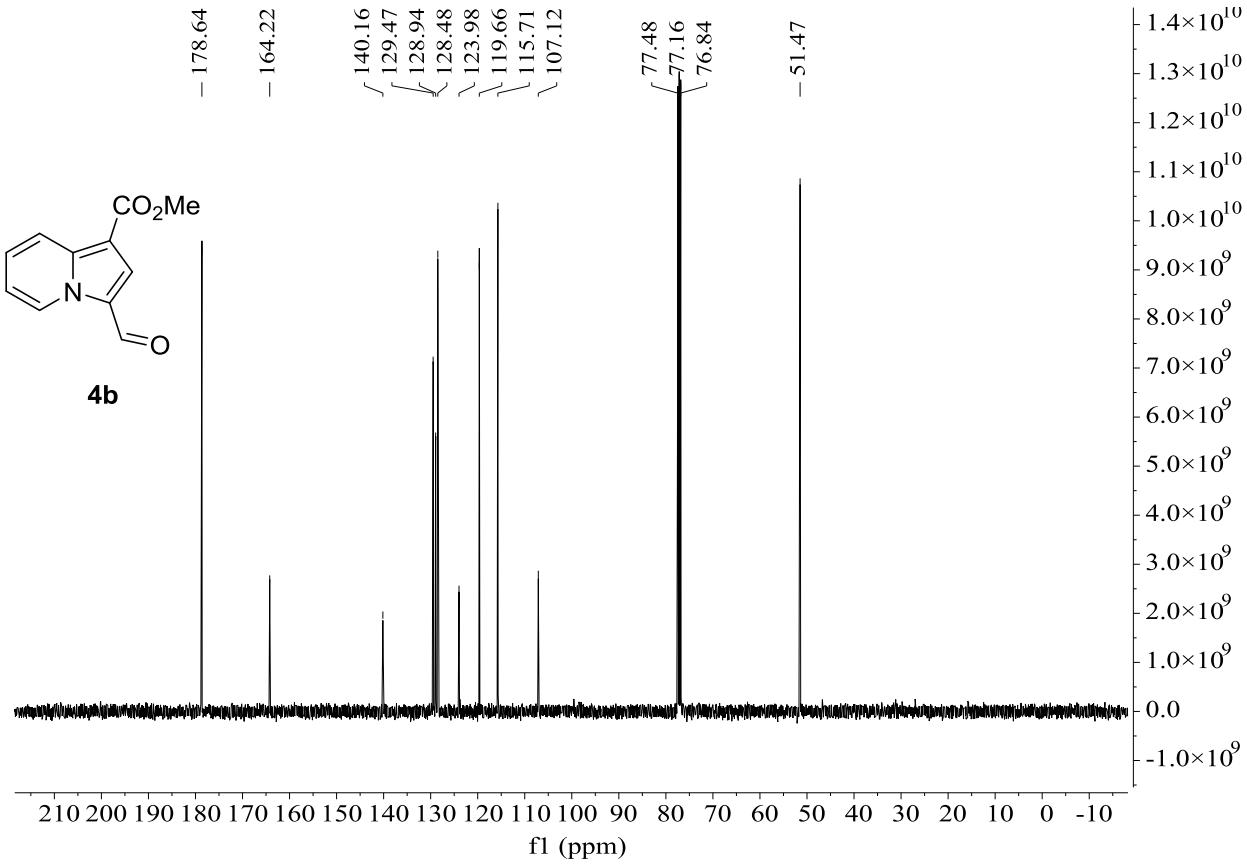
^{13}C NMR Spectrum of 4a at 25 °C ($CDCl_3$, 100 MHz)



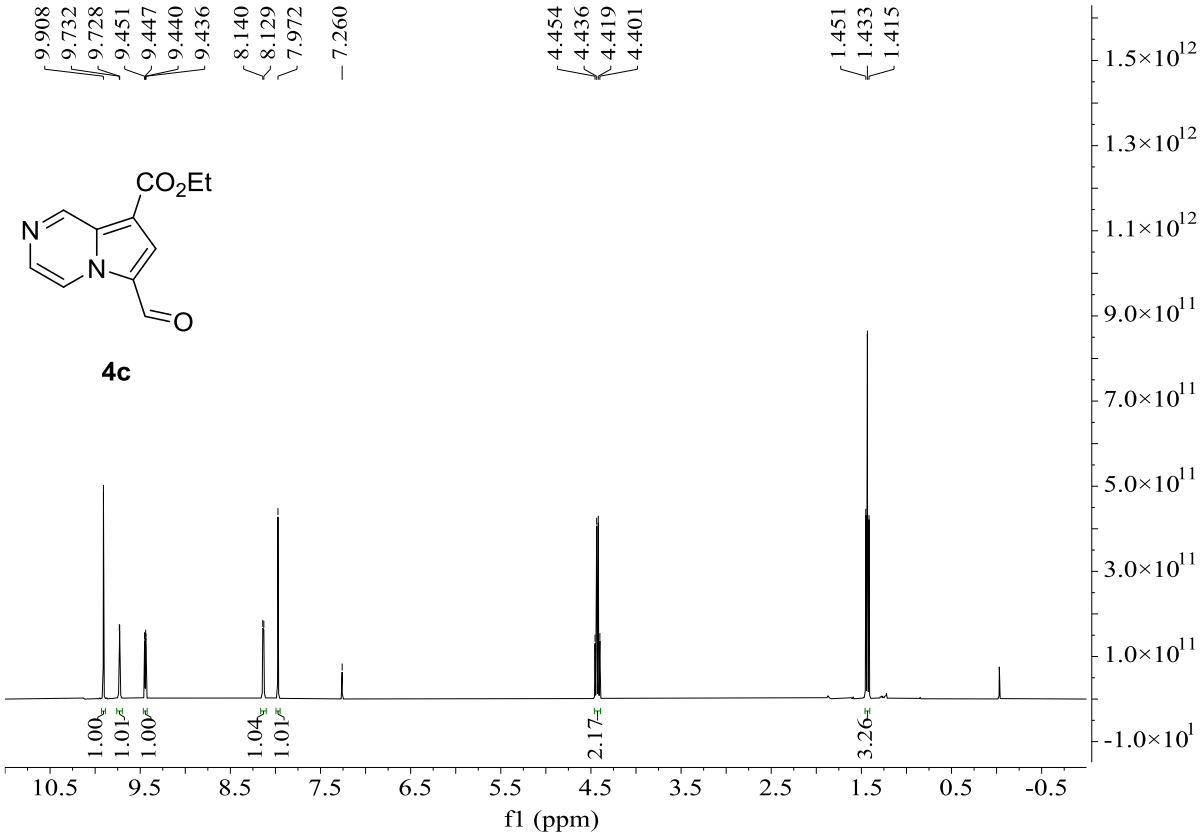
^{1}H NMR Spectrum of 4b at 25 °C ($CDCl_3$, 400 MHz)



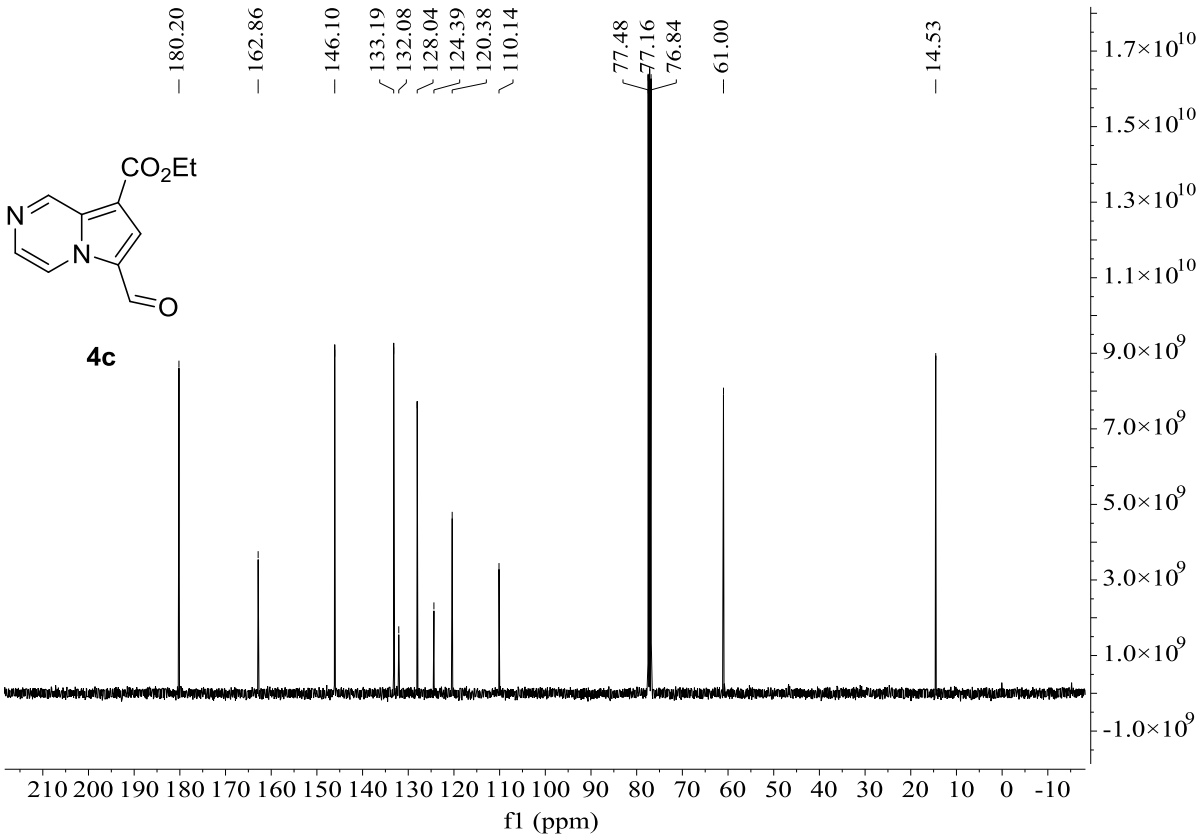
^{13}C NMR Spectrum of 4b at 25 °C ($CDCl_3$, 100 MHz)



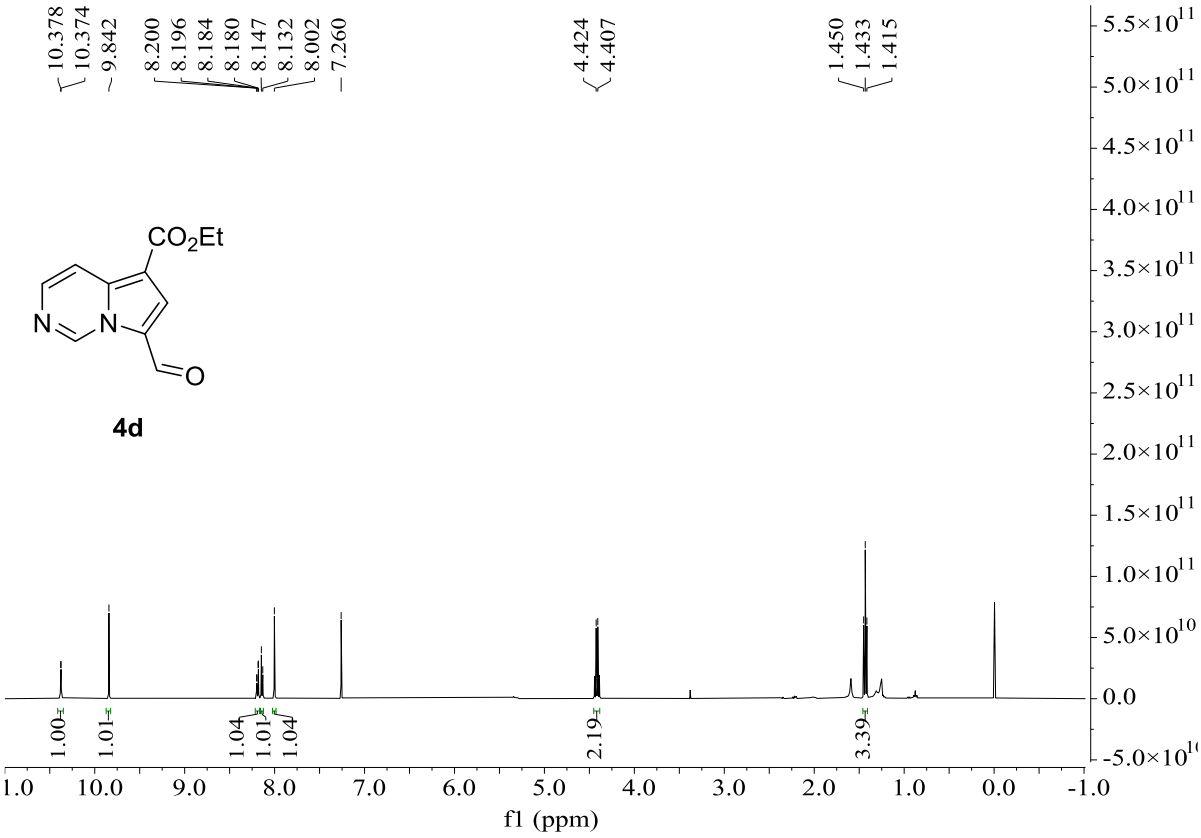
^{1}H NMR Spectrum of 4c at 25 °C ($CDCl_3$, 400 MHz)



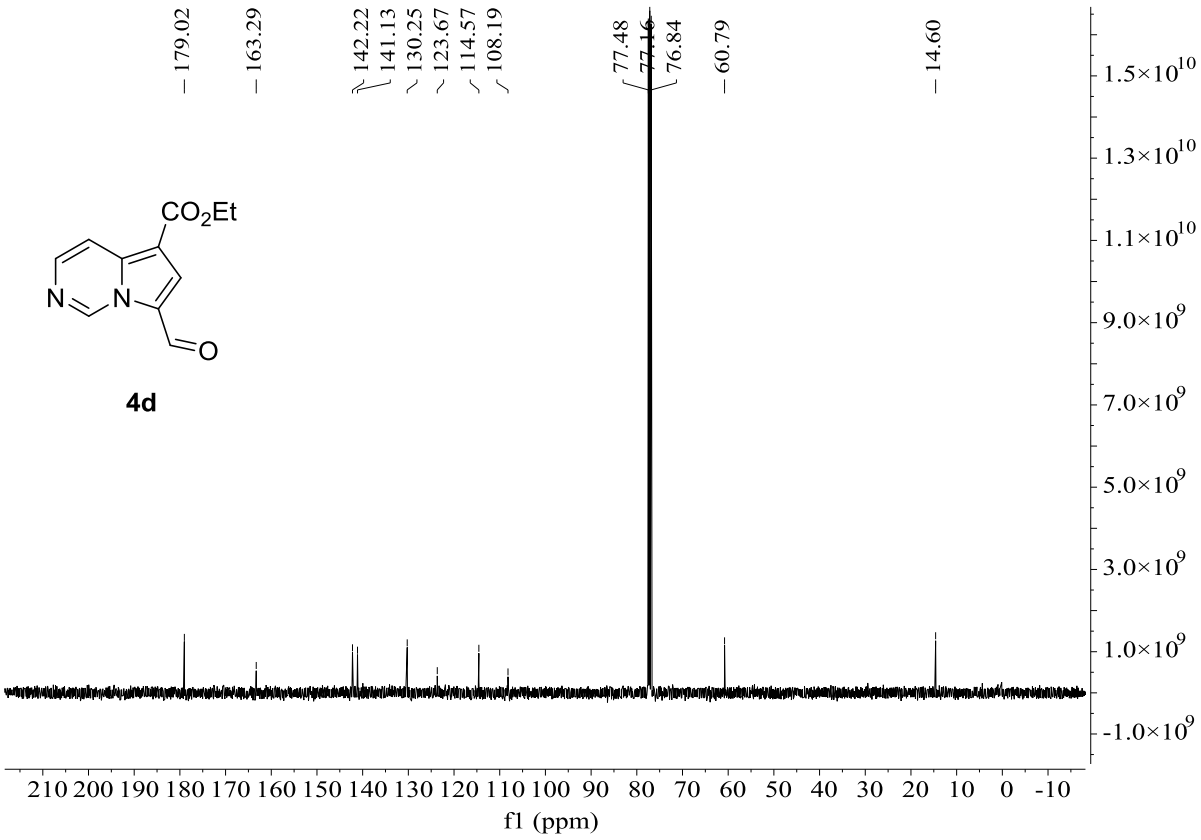
^{13}C NMR Spectrum of 4c at 25 °C ($CDCl_3$, 100 MHz)



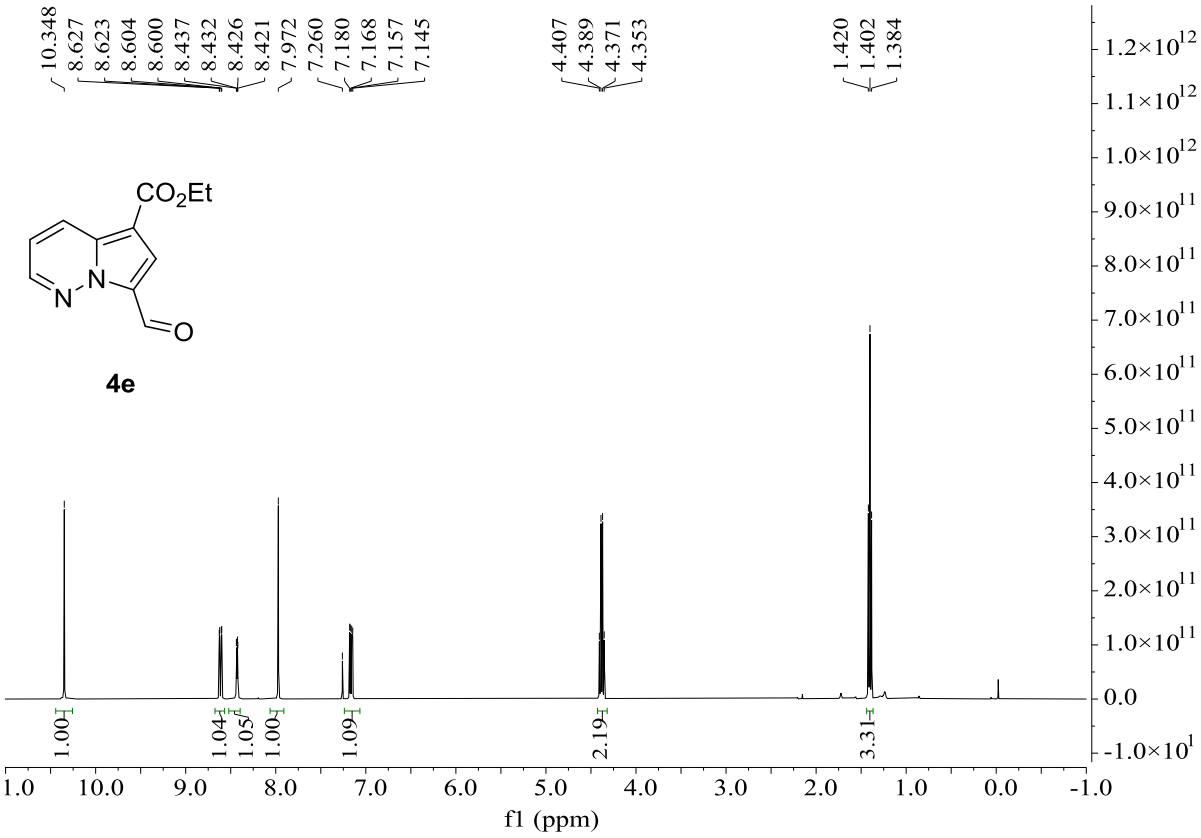
^{1}H NMR Spectrum of 4d at 25 °C ($CDCl_3$, 400 MHz)



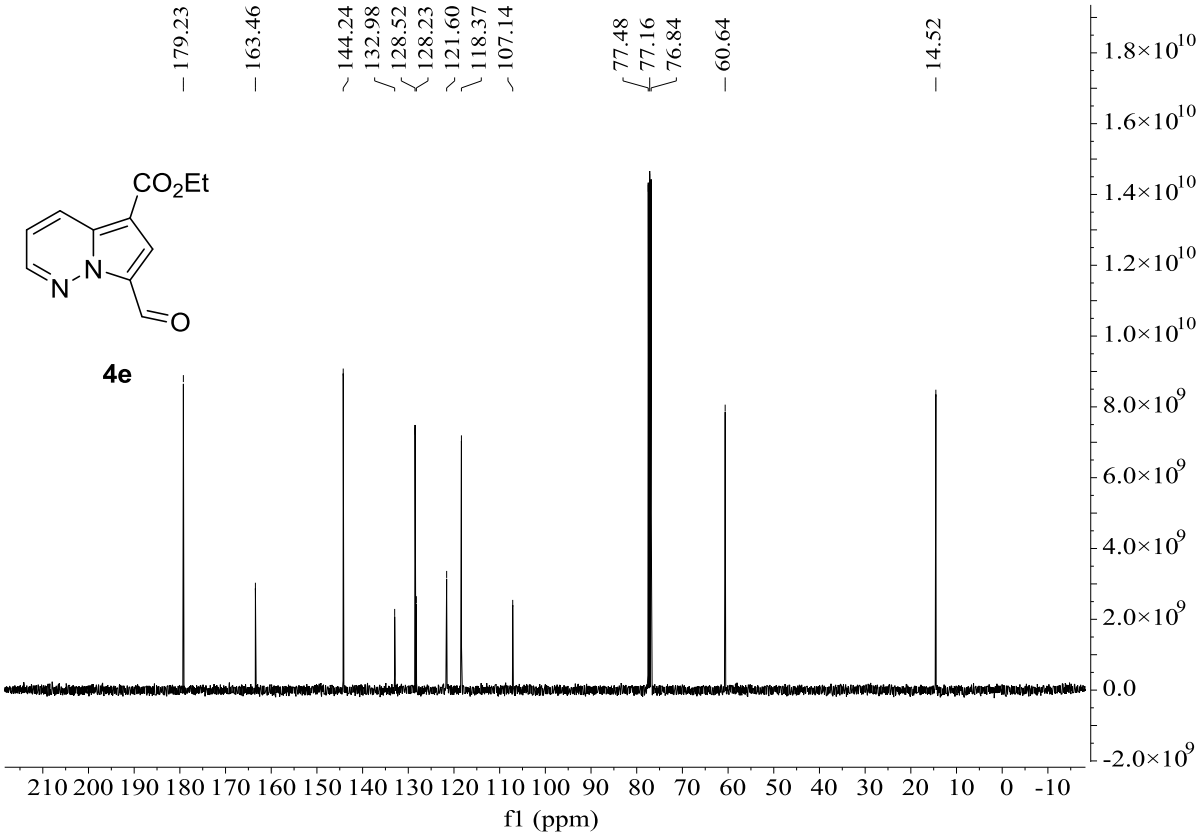
^{13}C NMR Spectrum of 4d at 25 °C ($CDCl_3$, 100 MHz)



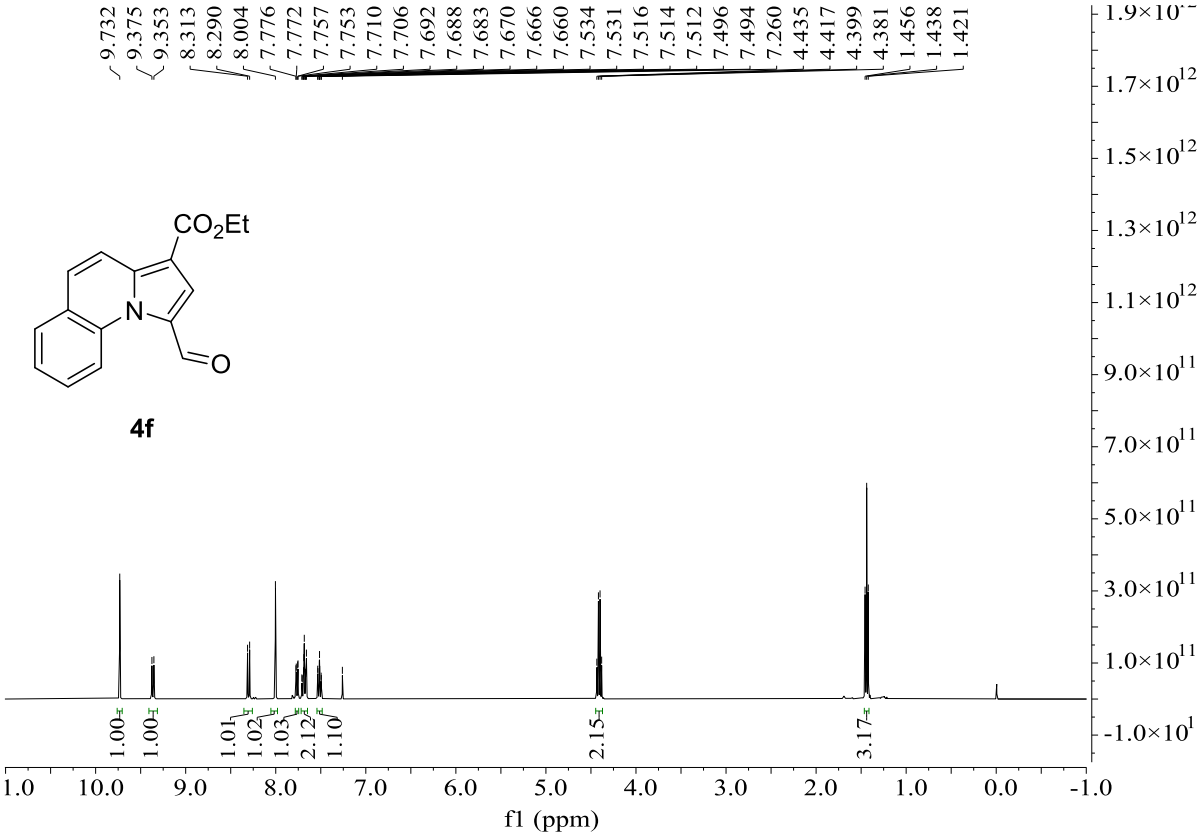
^{1}H NMR Spectrum of 4e at 25 °C ($CDCl_3$, 400 MHz)



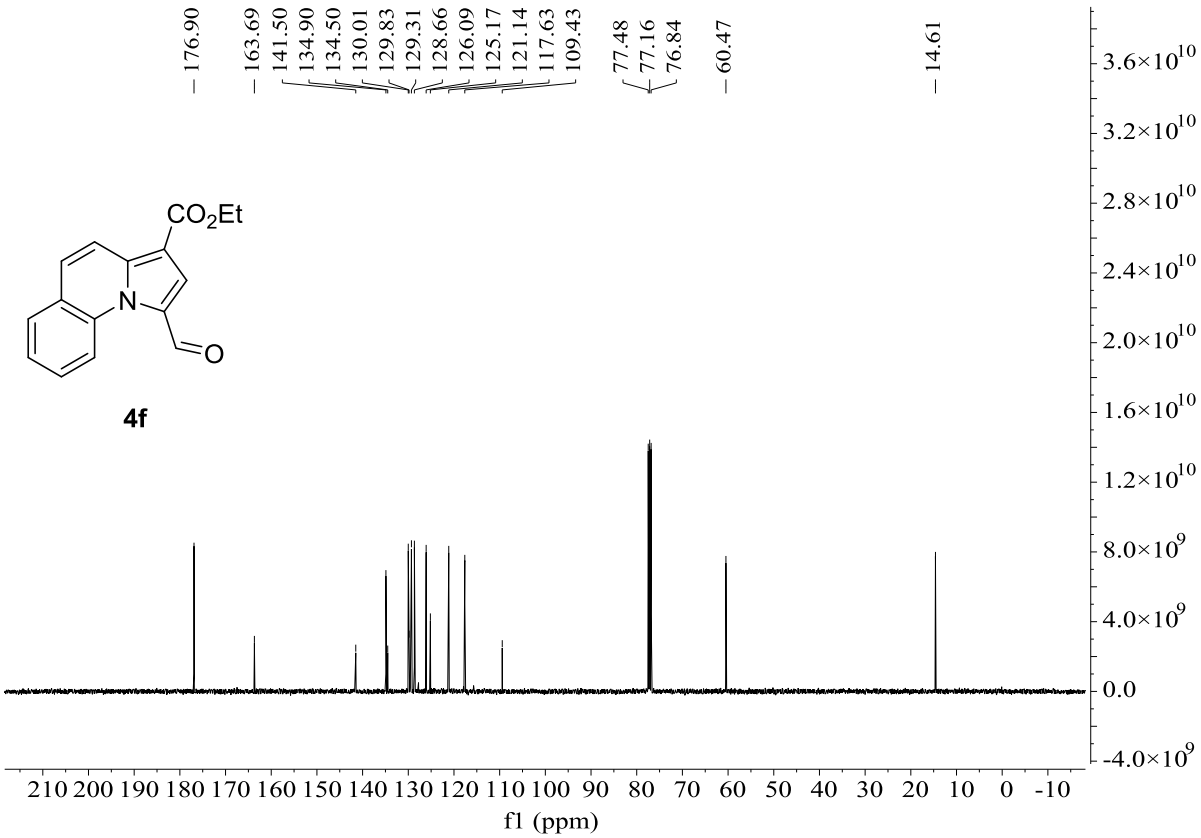
^{13}C NMR Spectrum of 4e at 25 °C ($CDCl_3$, 100 MHz)



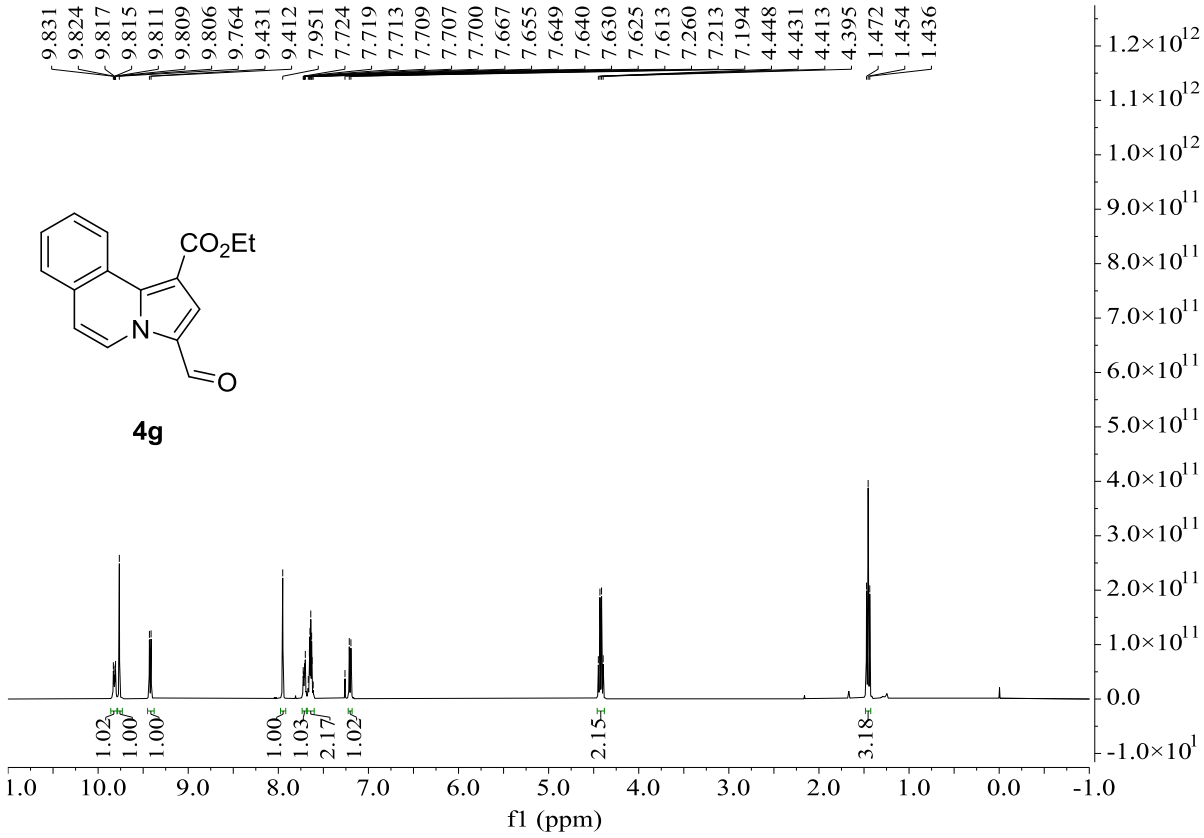
^{1}H NMR Spectrum of 4f at 25 °C ($CDCl_3$, 400 MHz)



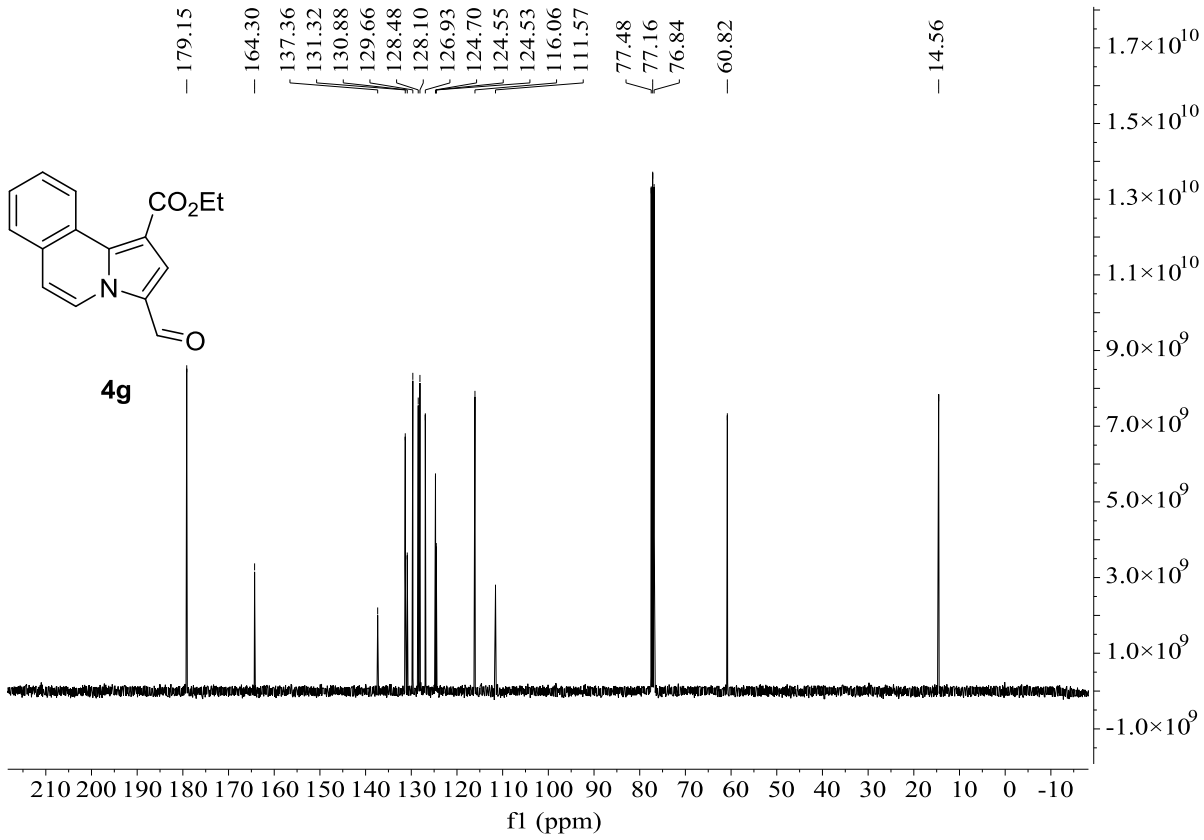
^{13}C NMR Spectrum of 4f at 25 °C ($CDCl_3$, 100 MHz)



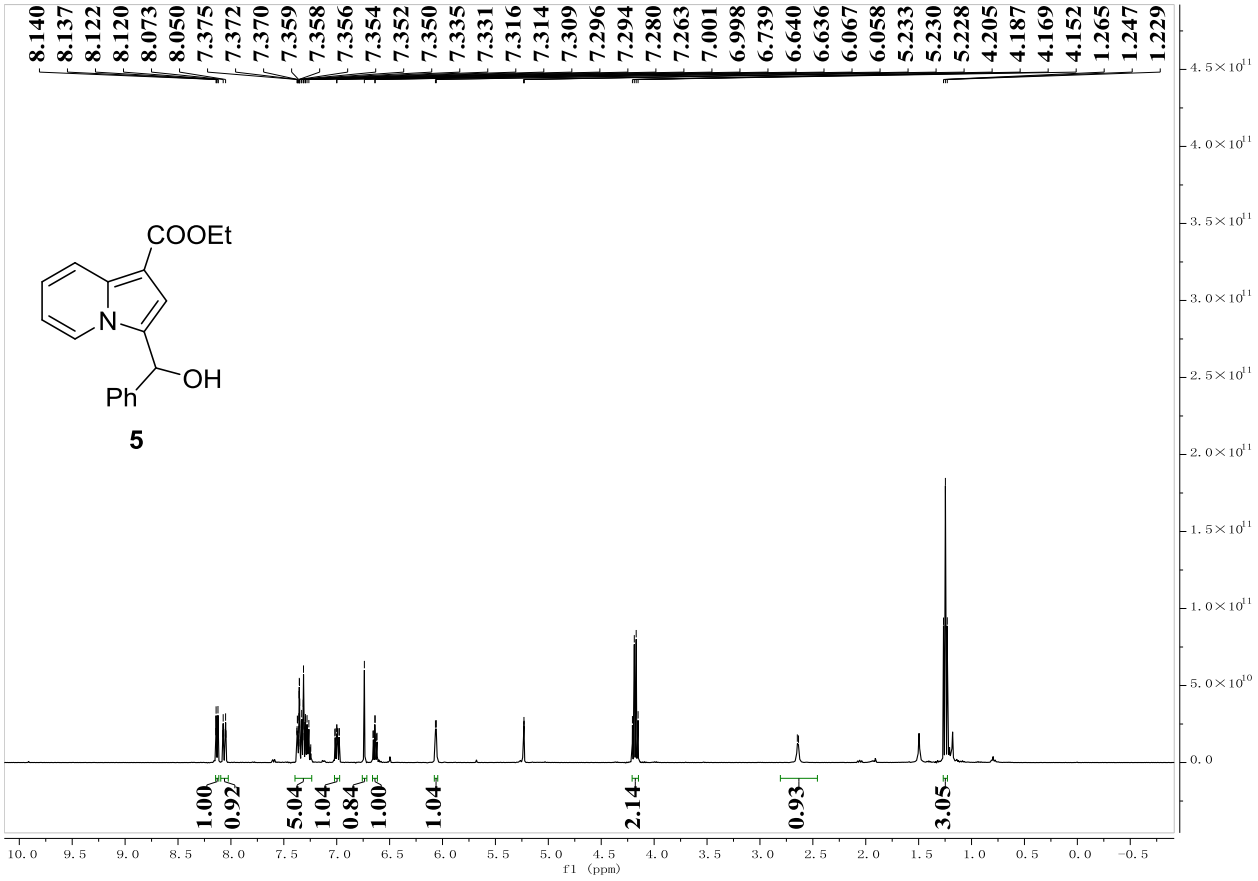
^{1}H NMR Spectrum of 4g at 25 °C ($CDCl_3$, 400 MHz)



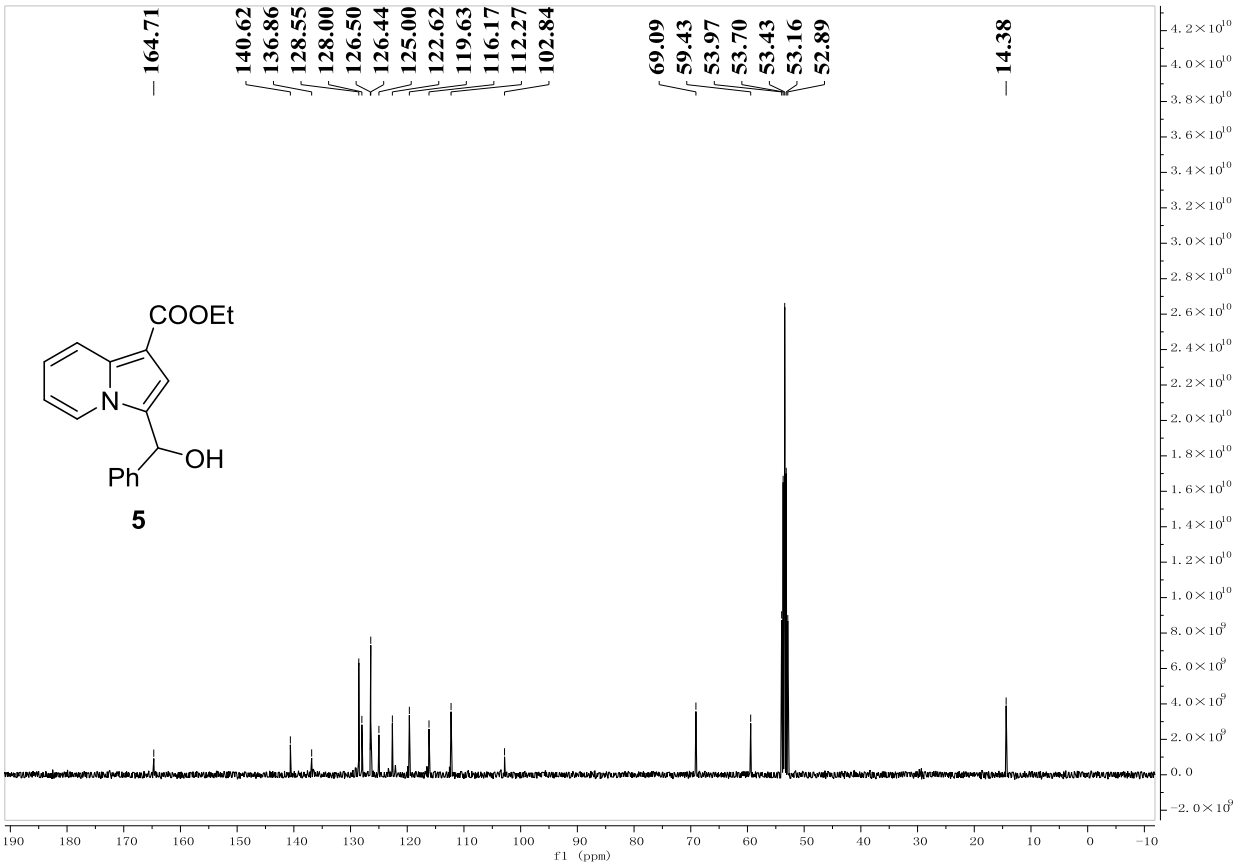
^{13}C NMR Spectrum of 4g at 25 °C ($CDCl_3$, 100 MHz)



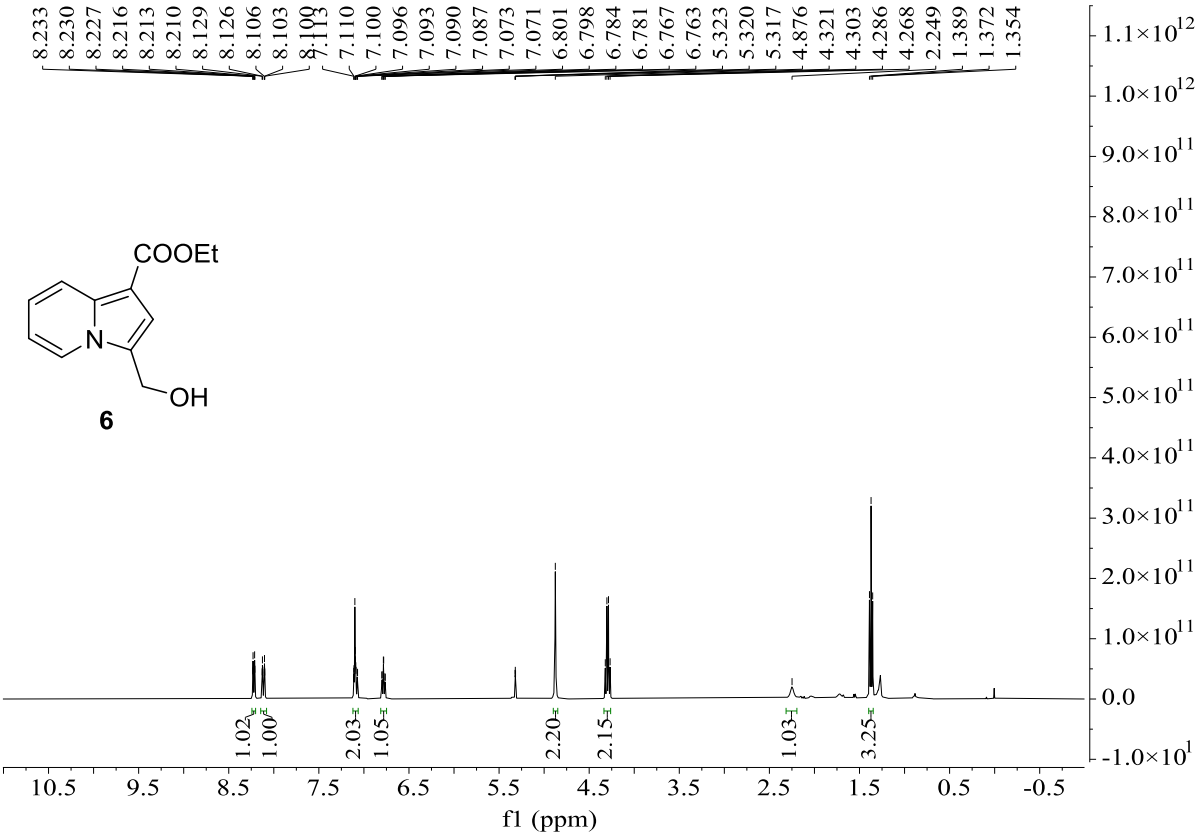
^{1}H NMR Spectrum of 5 at 25 °C (CD_2Cl_2, 400 MHz)



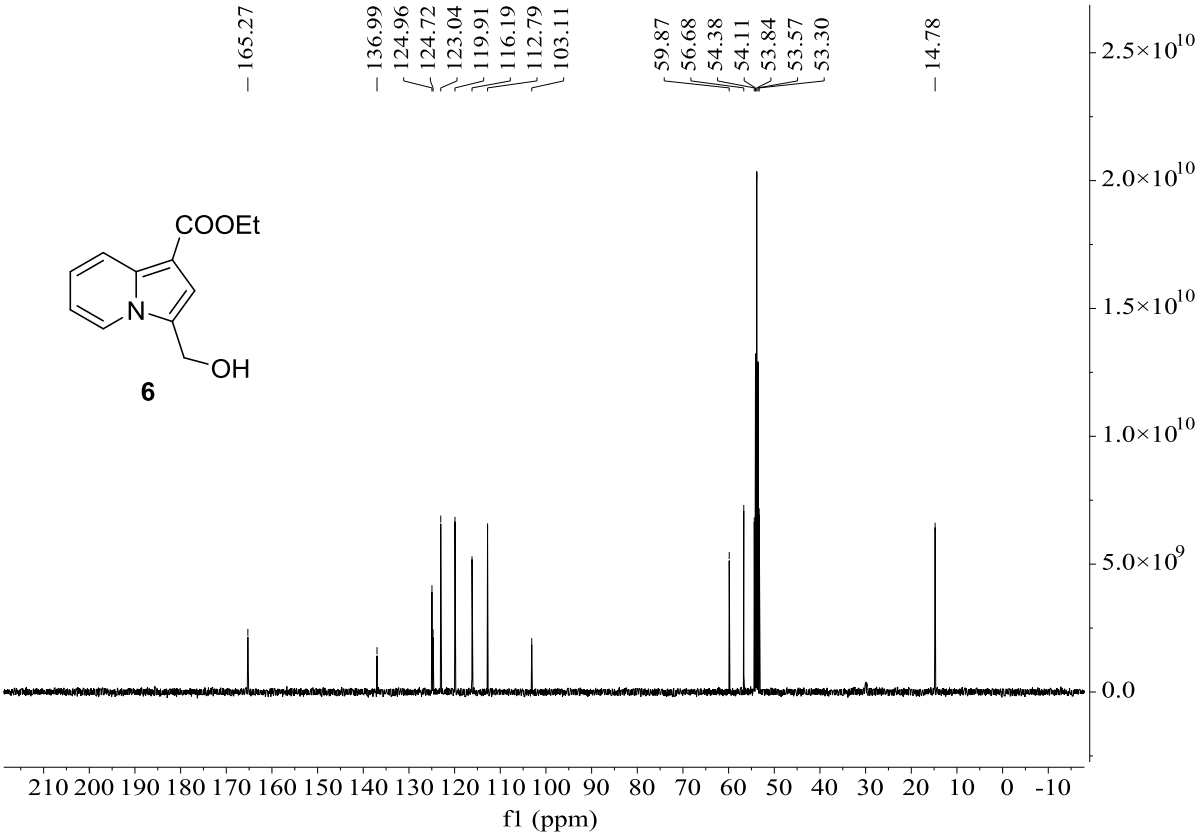
^{13}C NMR Spectrum of 5 at 25 °C (CD_2Cl_2, 100 MHz)



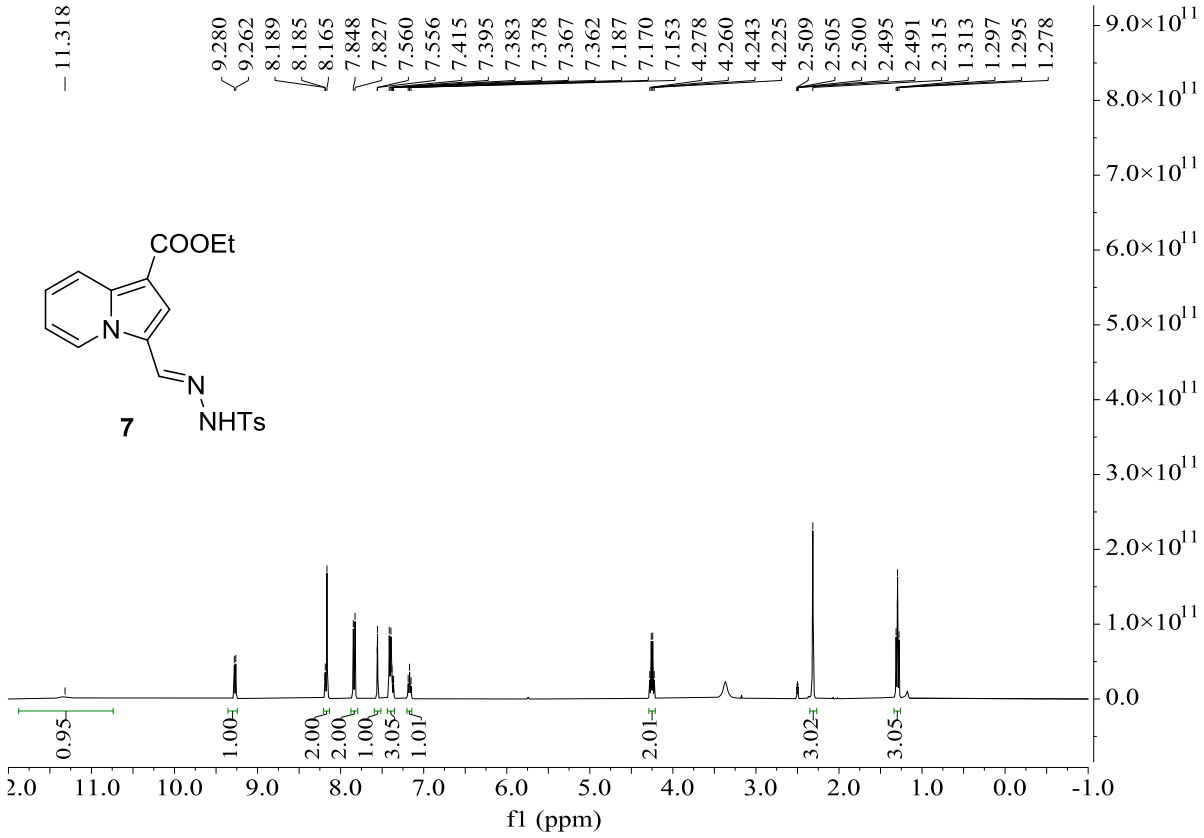
^{1}H NMR Spectrum of 6 at 25 °C (CD_2Cl_2, 400 MHz)



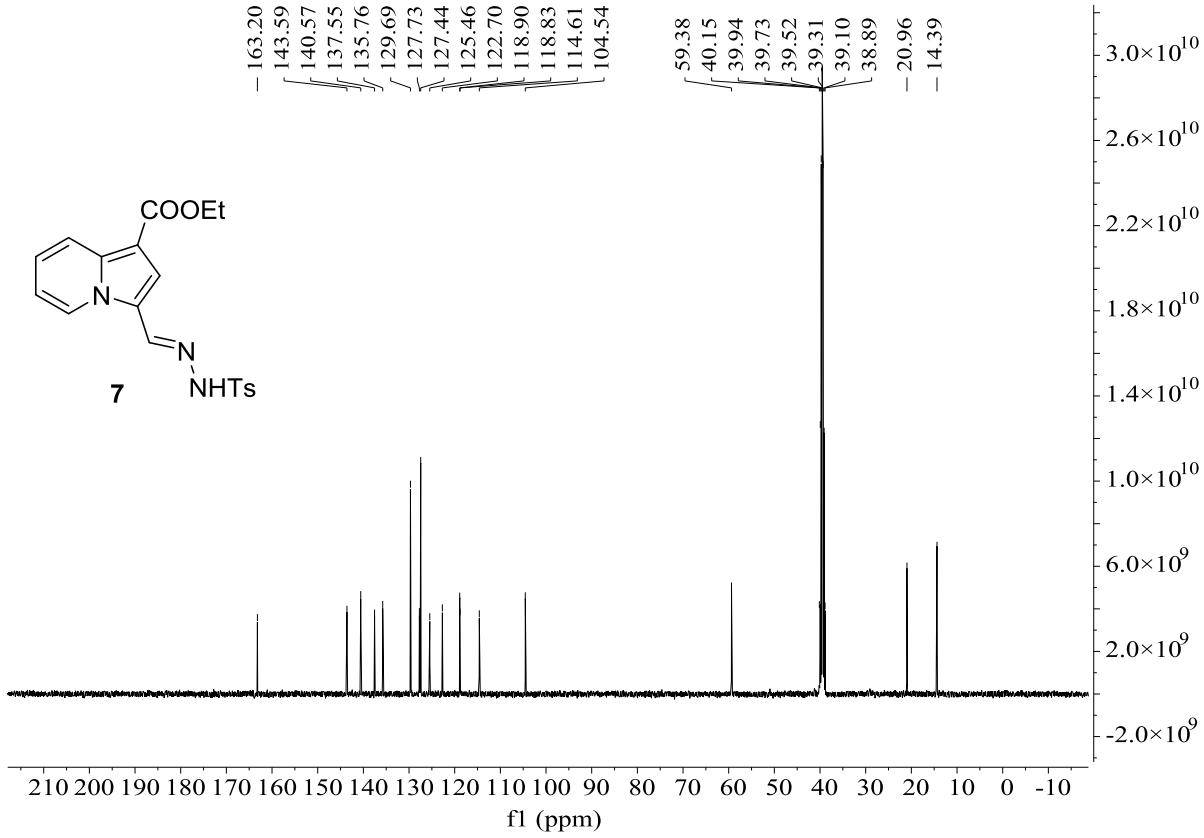
^{13}C NMR Spectrum of 6 at 25 °C (CD_2Cl_2, 100 MHz)



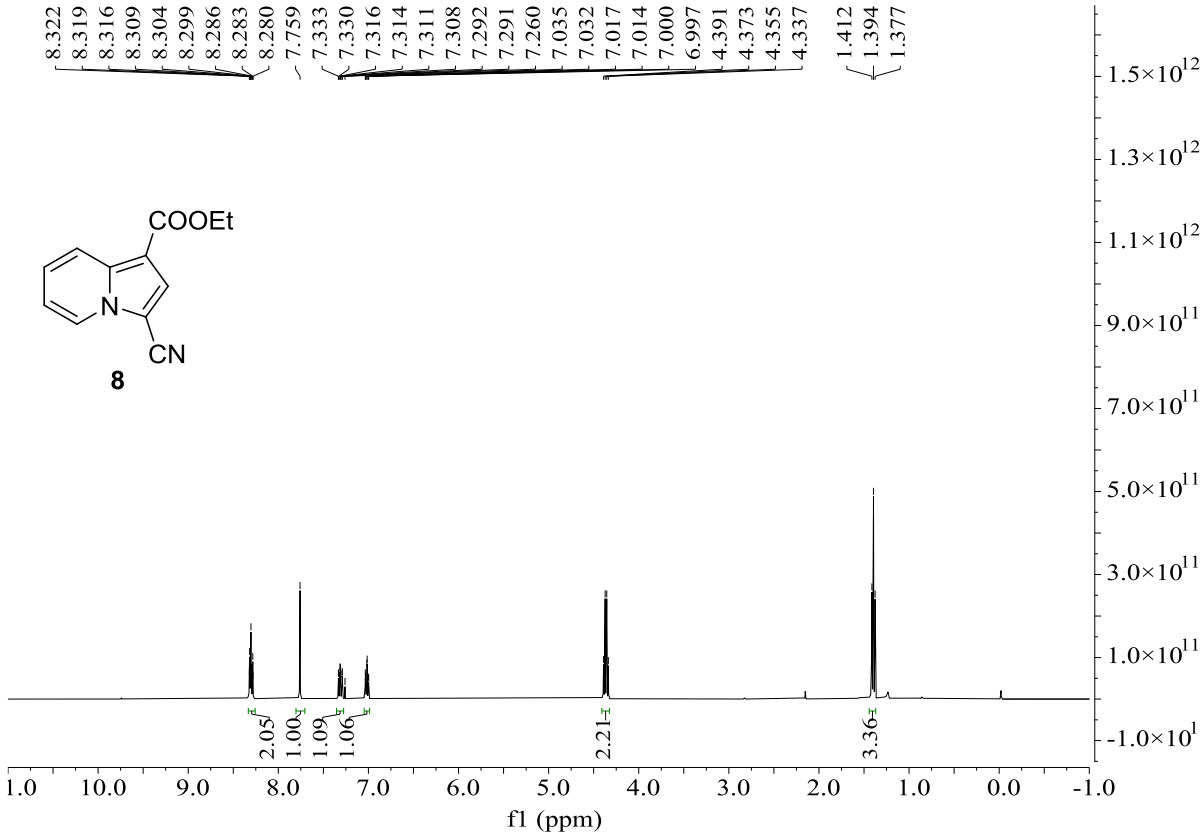
^{1}H NMR Spectrum of 7 at 25 °C (DMSO-d_6, 400 MHz)



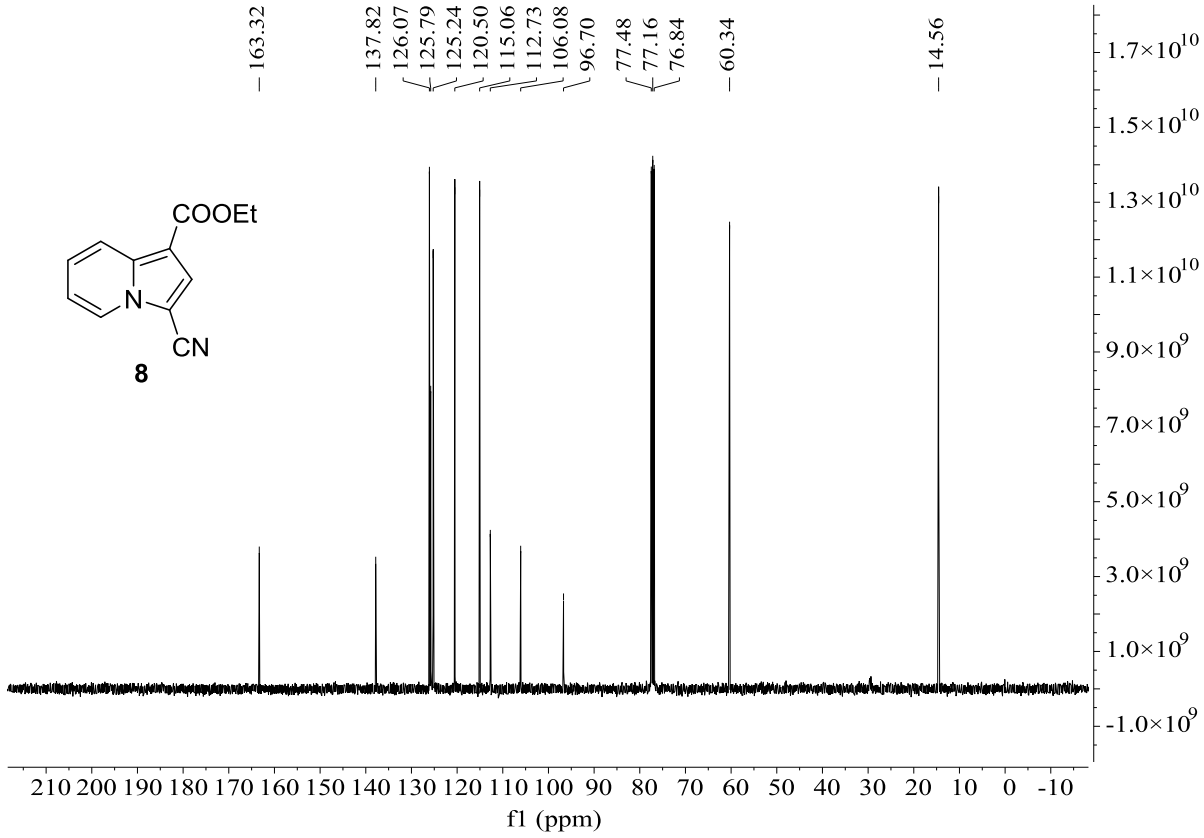
^{13}C NMR Spectrum of 7 at 25 °C (DMSO-d_6, 100 MHz)



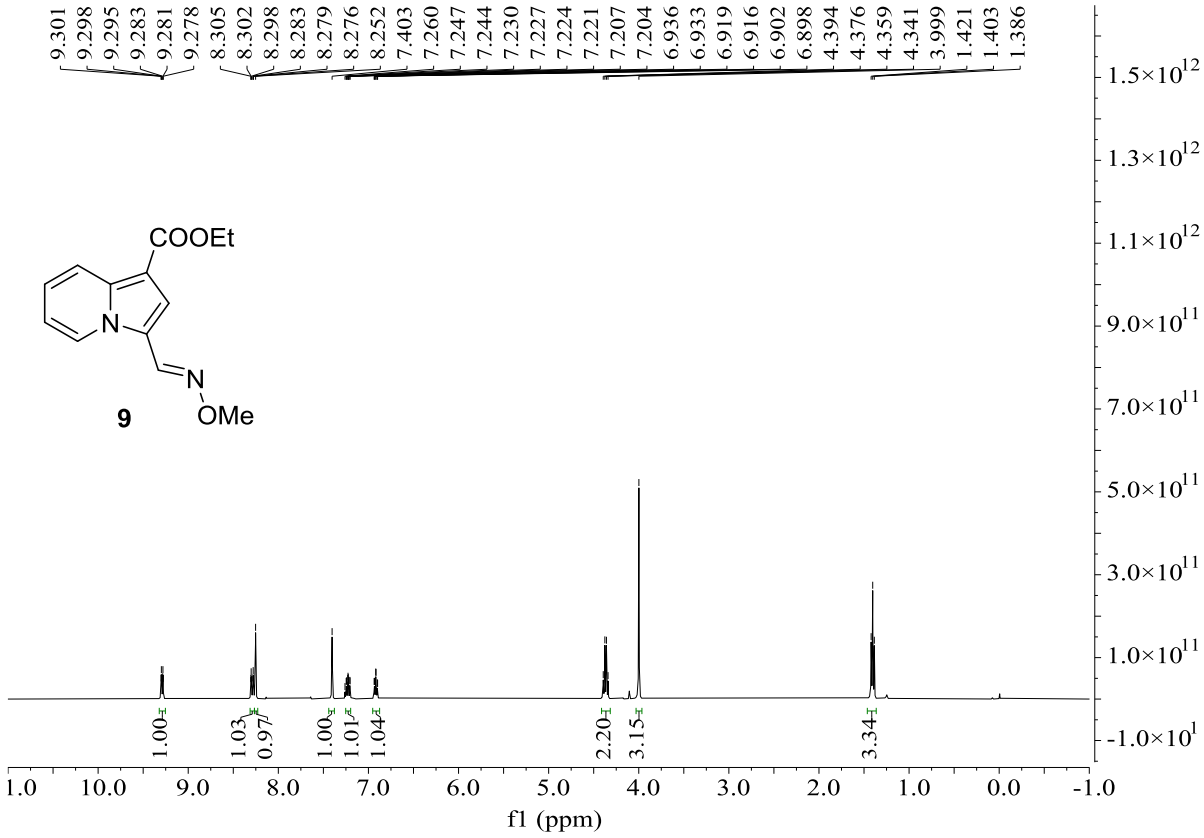
^{1}H NMR Spectrum of 8 at 25 °C ($CDCl_3$, 400 MHz)



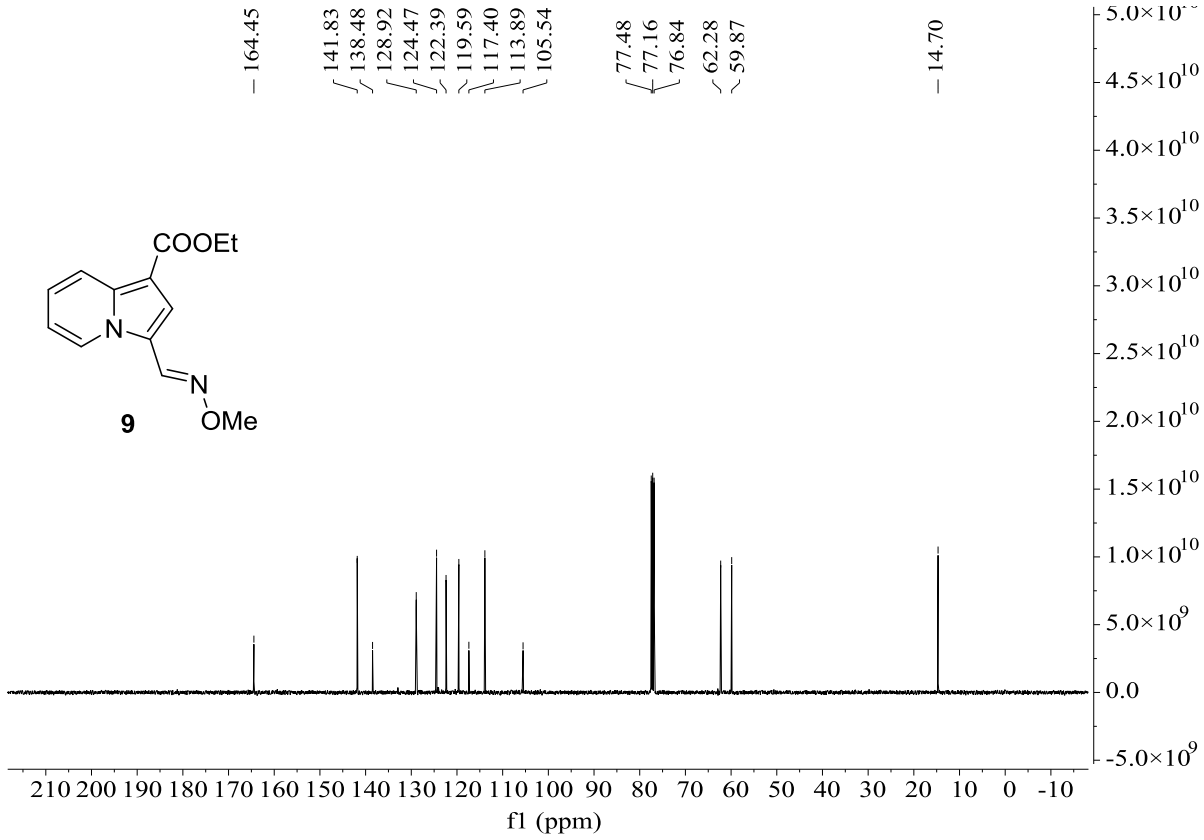
^{13}C NMR Spectrum of 8 at 25 °C ($CDCl_3$, 100 MHz)



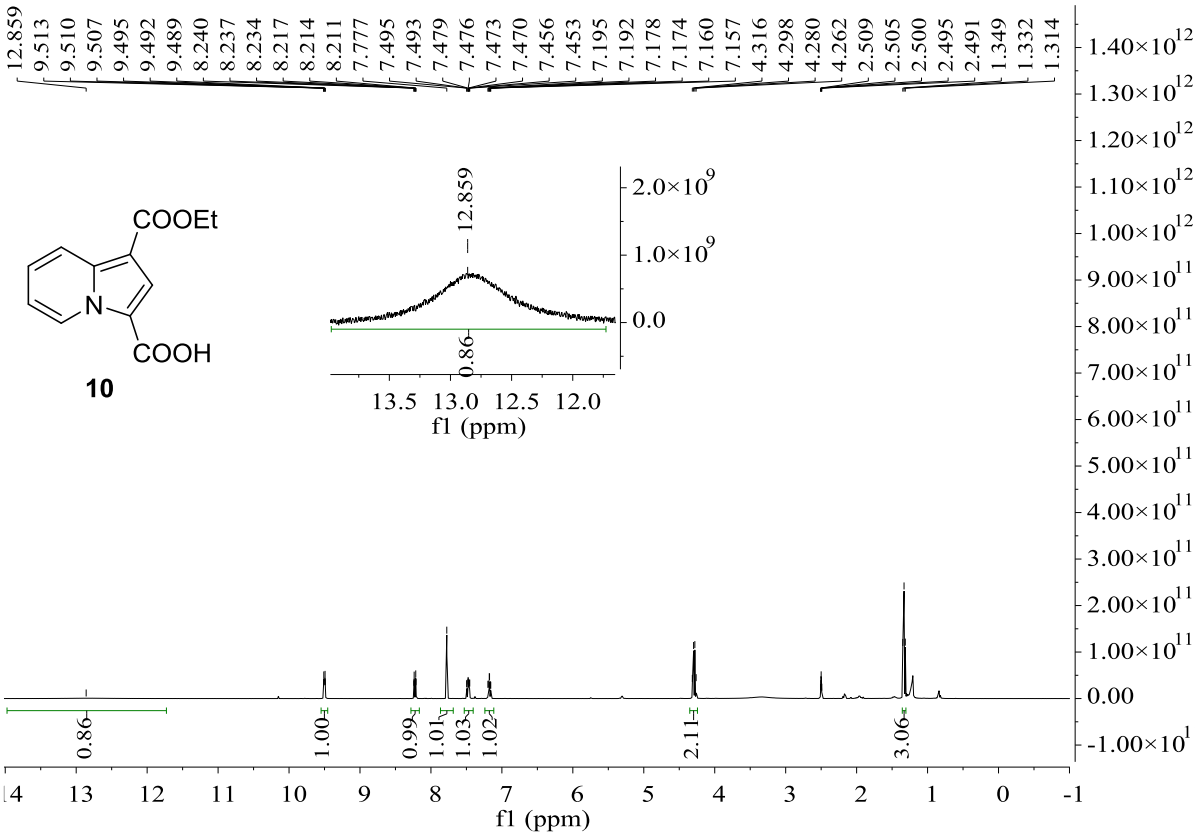
^{1}H NMR Spectrum of 9 at 25 °C ($CDCl_3$, 400 MHz)



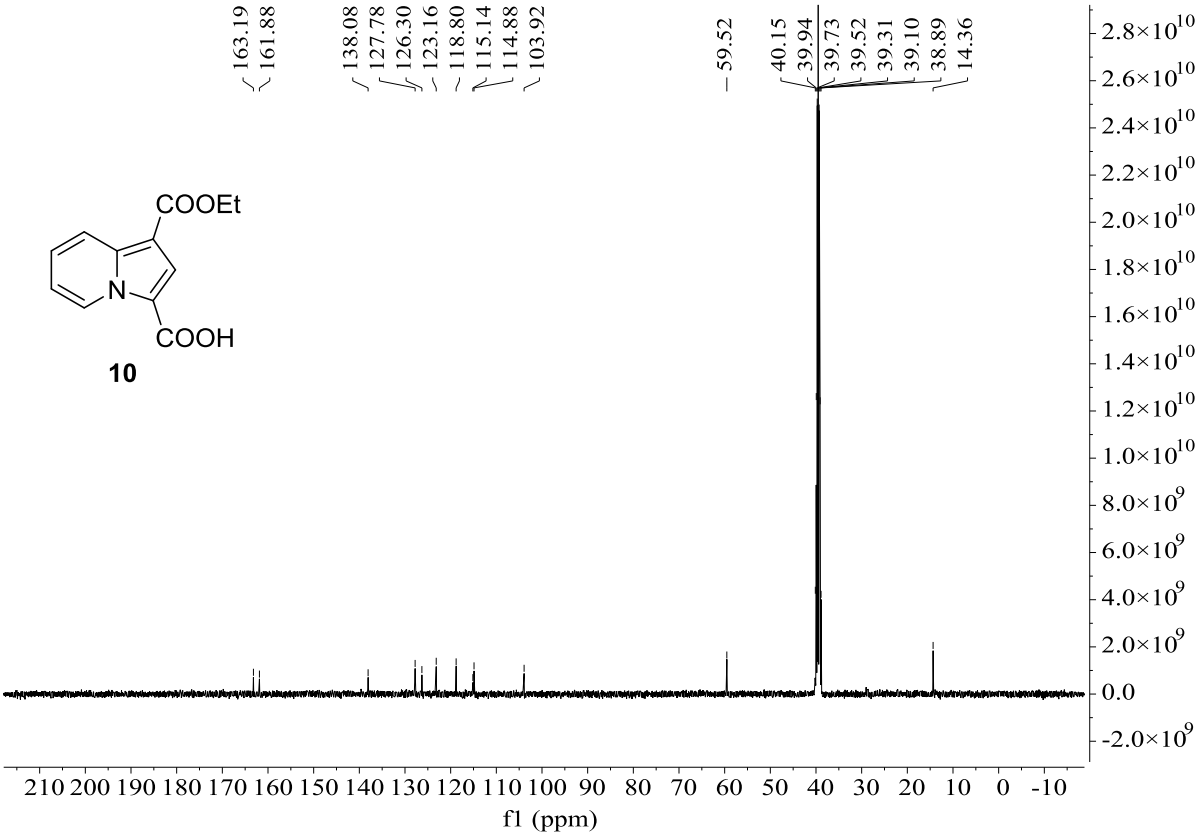
^{13}C NMR Spectrum of 9 at 25 °C ($CDCl_3$, 100 MHz)



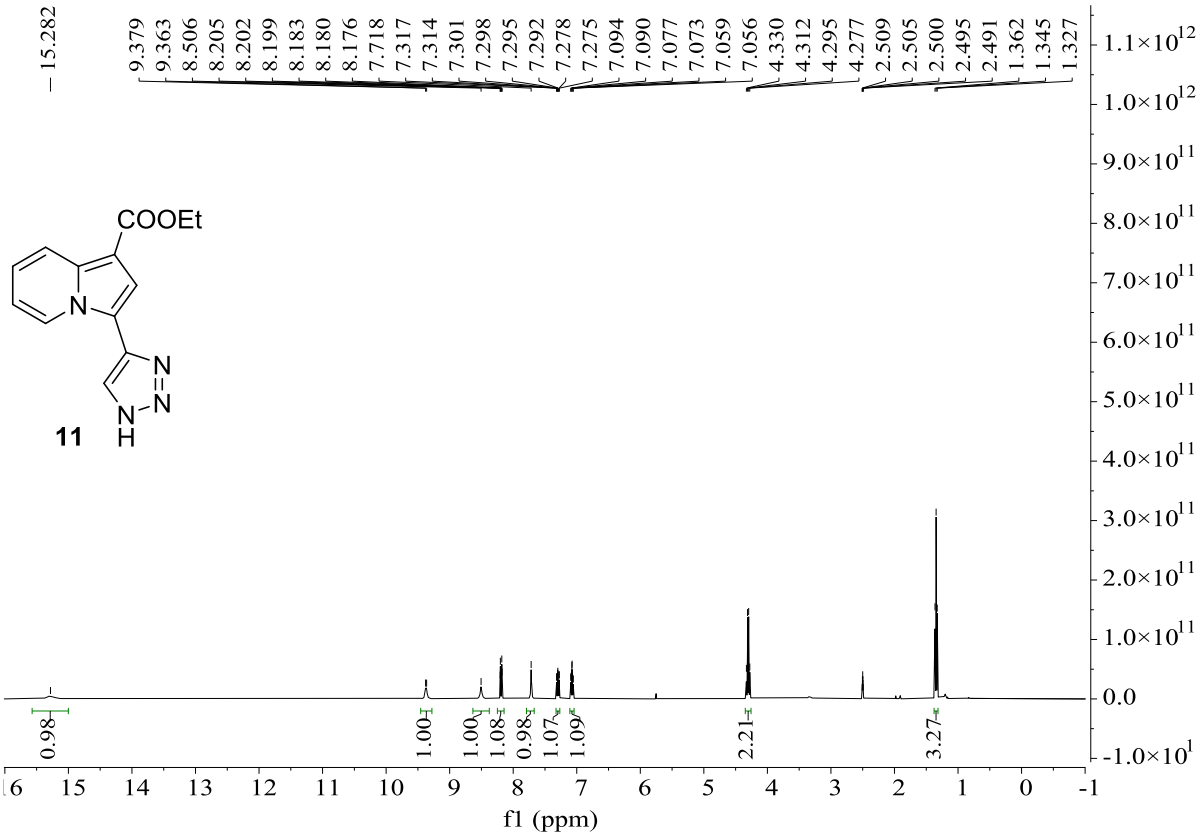
^{1}H NMR Spectrum of 10 at 25 °C (DMSO-d_6, 400 MHz)



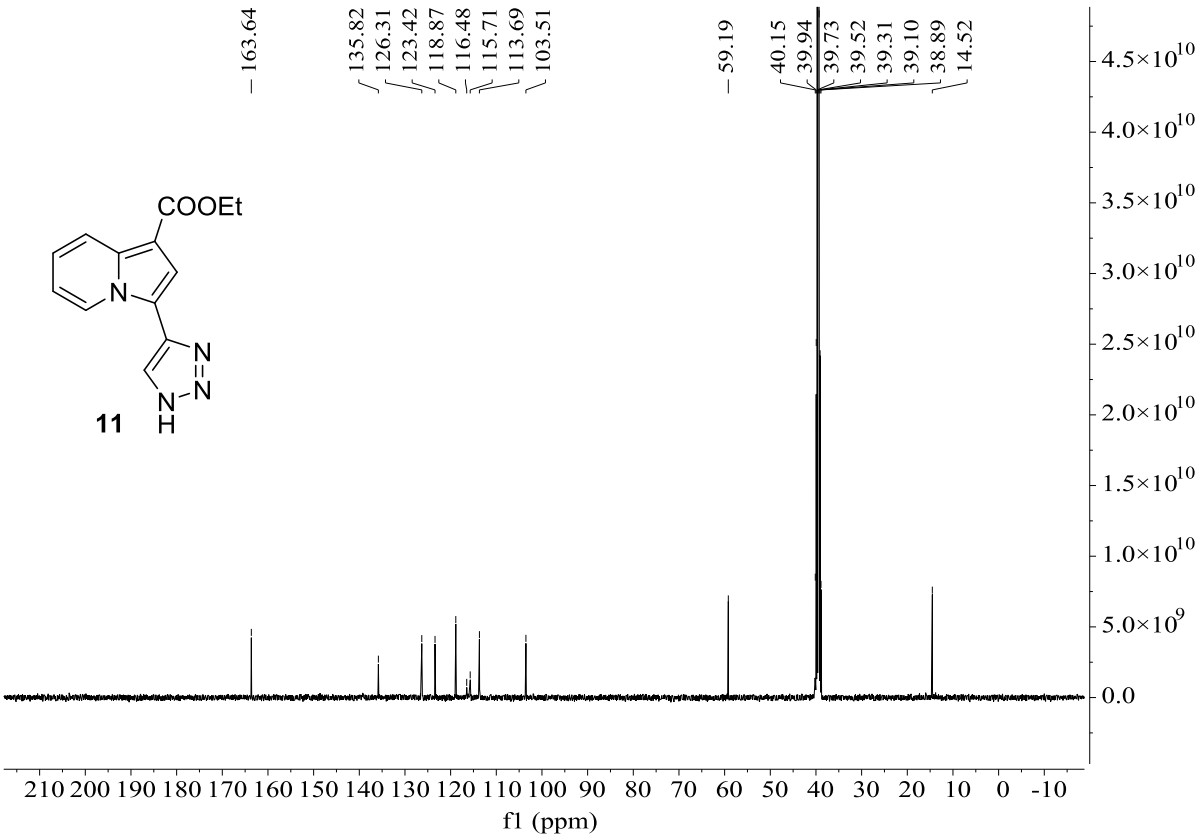
^{13}C NMR Spectrum of 10 at 25 °C (DMSO-d_6, 100 MHz)



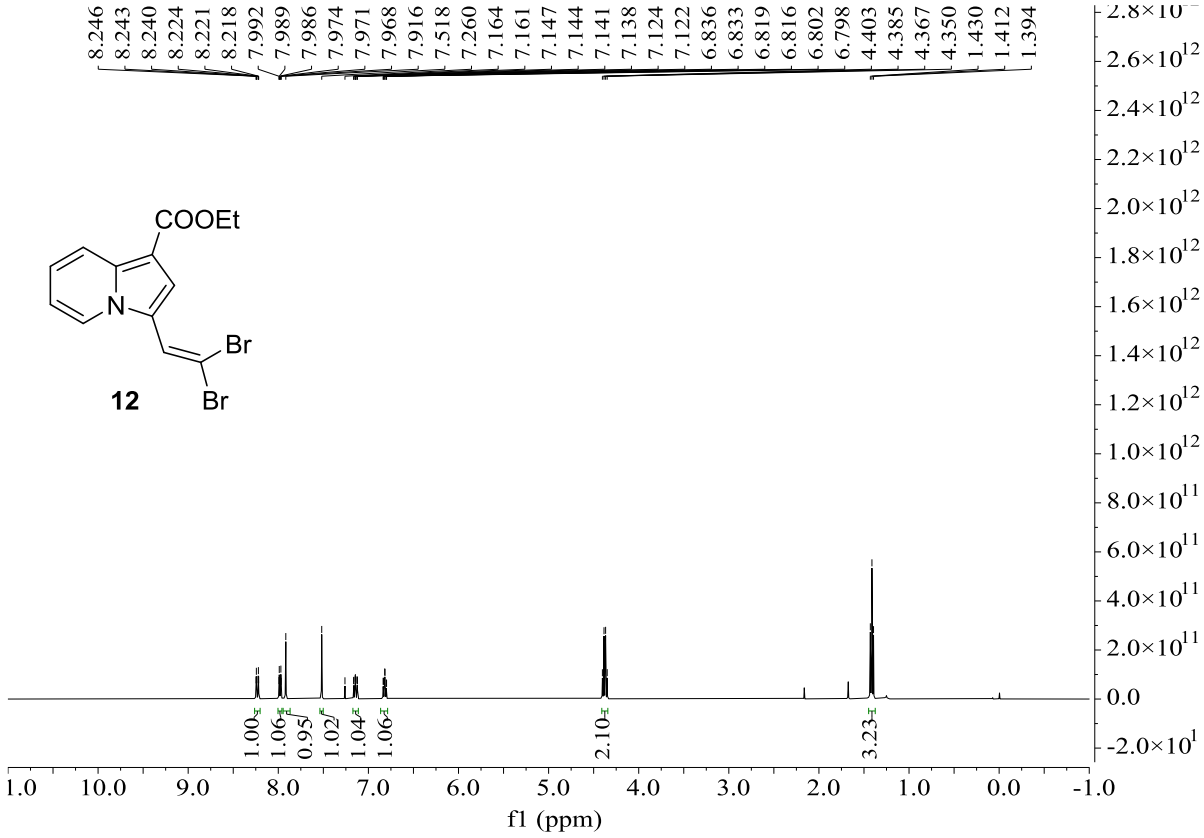
^{1}H NMR Spectrum of 11 at 25 °C (DMSO-d_6, 400 MHz)



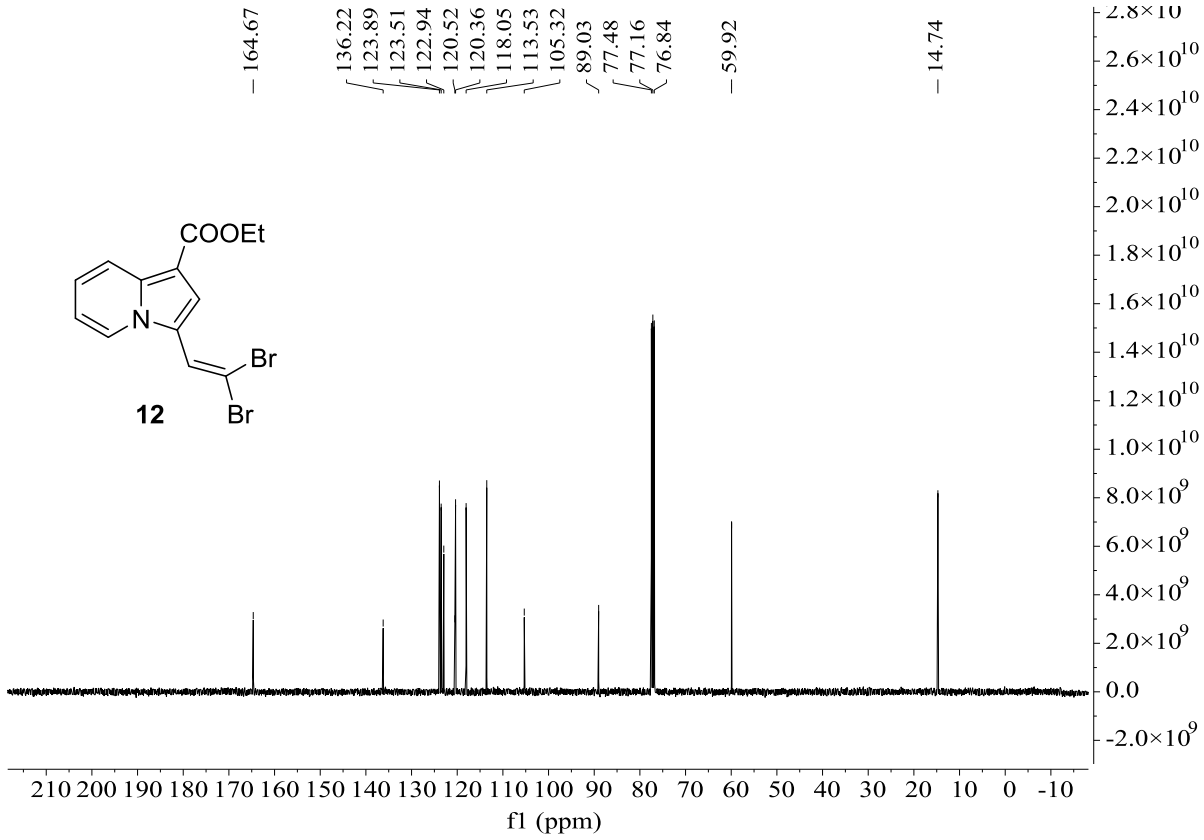
^{13}C NMR Spectrum of 11 at 25 °C (DMSO-d_6, 100 MHz)



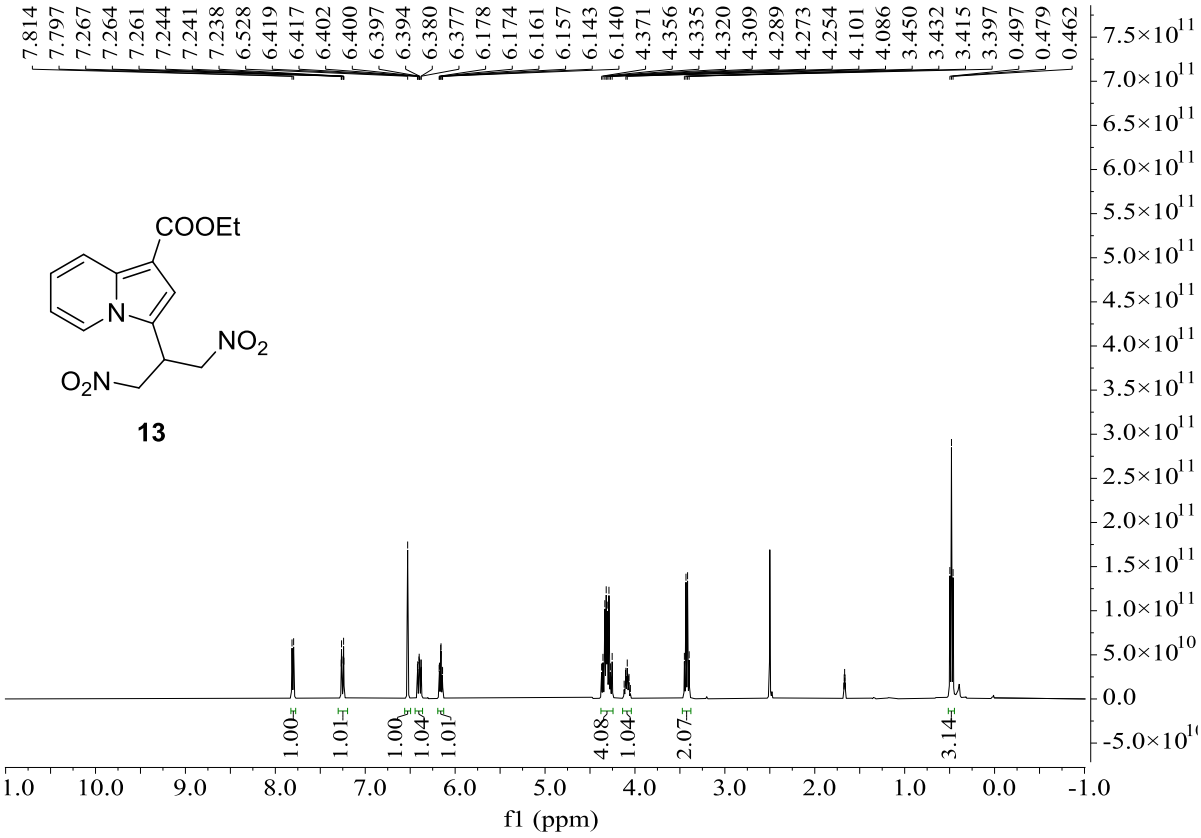
^{1}H NMR Spectrum of 12 at 25 °C ($CDCl_3$, 400 MHz)



^{13}C NMR Spectrum of 12 at 25 °C ($CDCl_3$, 100 MHz)



^{1}H NMR Spectrum of 13 at 25 °C (DMSO-d_6, 400 MHz)



^{13}C NMR Spectrum of 13 at 25 °C (DMSO-d_6, 100 MHz)