Supporting Information

For

Radical aza-cyclization of α-imino-oxy acids for synthesis of alkene-containing *N*-heterocycles via dual cobaloxime and photoredox catalysis

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1. General remarks

^{1}H NMR spectra were recorded on 400 or 600 MHz (100 or 150 MHz for ^{13}C NMR) agilent NMR spectrometer with $CDCl_3$ as solvent and tetramethylsilane (TMS) as the internal standard. Chemical shifts were reported in parts per million (ppm, δ scale) downfield from TMS at 0.00 ppm and referenced to $CDCl_3$ at 7.26 ppm (for ^{1}H NMR) and 77.16 ppm (for ^{13}C NMR). HRMS was recorded on a GCT Premier™ (CI) or Agilent 6540 Q-TOF (ESI) Mass Spectrometer. Infrared (FT-IR) spectra were recorded on a Varian 1000FT-IR, ν_{max} in cm^{-1}. All commercially available reagents and solvents were used as received unless otherwise specified.

2. Photochemical reaction setup

Household blue LED strips (22 W) were coiled around the inside of a glassware with 15 cm diameter (Figure S1). The LED strips were wrapped in aluminum foil to maintain a specific reaction temperature. In this case, the reaction temperature is approximately 35 °C. Optimum yields were then observed.

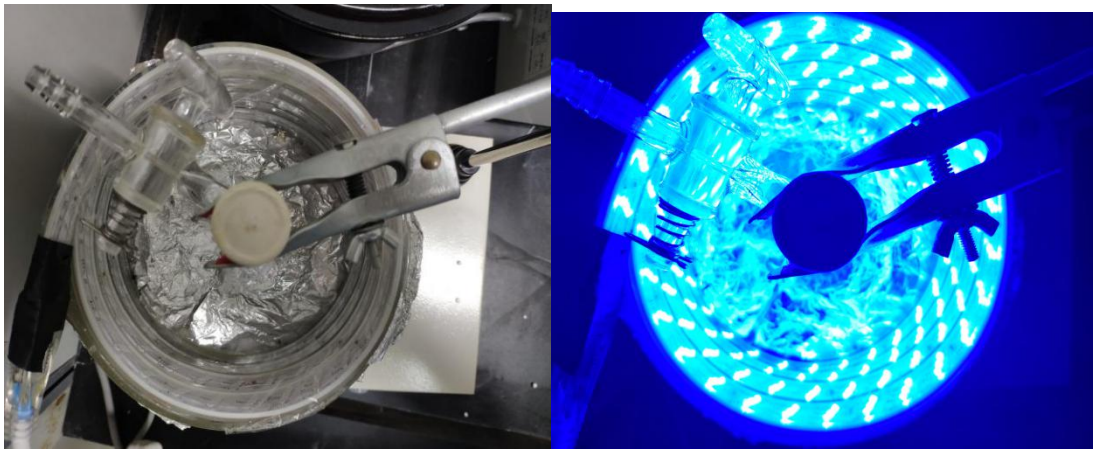
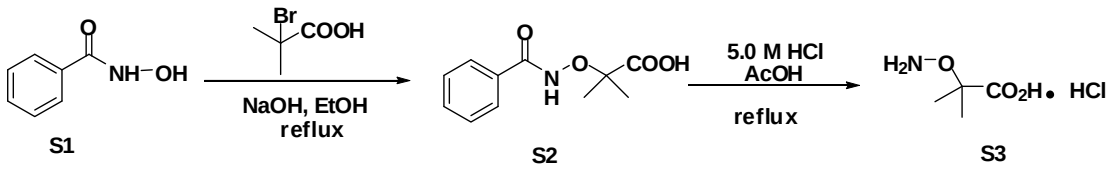


Figure S1. Reaction setup

3. General procedures for synthesis of substrates

3.1 Preparation of *α*-amino-oxy acid hydrochloride

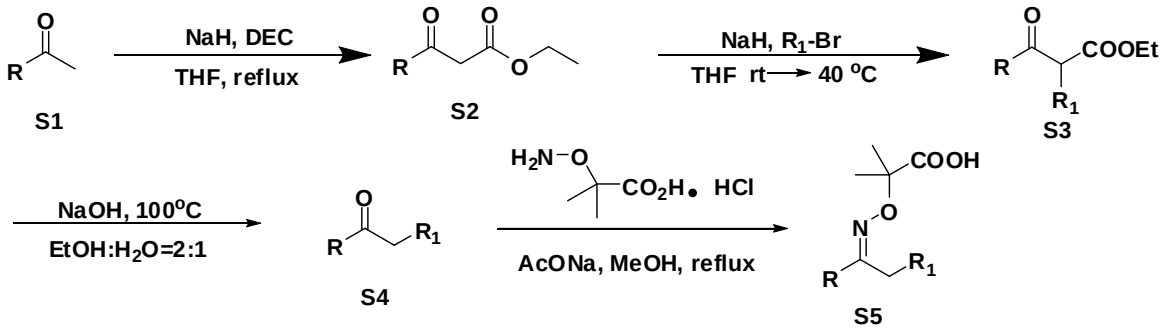


1) Preparation of 2-(benzamidooxy)acetic acid (**S2**):[1] A mixture of *N*-

hydroxybenzamide **S1** (13.7 g, 0.10 mol), 2-bromo-2-methylpropanoic acid (9.0 mL, 0.10 mol) and NaOH (8.0 g, 0.20 mol) in EtOH (150 mL) was refluxed overnight. After cooling to room temperature, the solvent was removed under reduced pressure and the residue was dissolved in H_2O (200 mL) and acidified to pH = 1.0. The aqueous solution was extracted with EtOAc (250 mL × 3) and the organic layers were combined, dried with $MgSO_4$ and concentrated under reduced pressure to give a white solid, which was further purified by recrystallization from cold EtOAc/pentane (1:2) to give 2-(benzamidooxy)propanoic acid (**S2**) as a faint yellow solid (18.2 g, 88%).

2) 2-(Aminooxy)propanoic acid hydrochloride (**S3**) was prepared according to a modified literature method:[1] 5.0 M HCl (100 mL) was added to a stirring solution of **S2** (10.5 g, 50.0 mmol) in AcOH (30 mL). The mixture was refluxed overnight. After cooling to room temperature, the precipitated solid (benzoic acid) was filtered off and discarded. The filtrate was concentrated under reduced pressure to give a solid, which was further recrystallized from cold Et_2O to give **S3** as a white solid (6.7 g, 94%).

3.2 General procedure for synthesis of oxime acids.



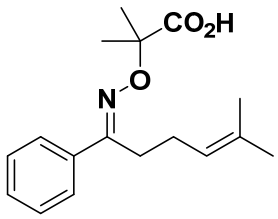
1) To a stirred solution of ketones (20 mmol) in Tetrahydrofuran (100 mL), Diethyl carbonate (40 mmol) and NaH (100 mmol, 60 %) were added. The reaction mixture was refluxed overnight. After cooling to room temperature, the reaction mixture was quenched by ice water, then extracted with EtOAc (100 mL × 3). The combined organic layer was dried over sodium sulfate and evaporated under reduced pressure. The residue was purified by flash column chromatography on silica gel to give **S2**.[2]

2) A solution of **S2** (1.0 equiv.) in THF (0.2 M) was treated with NaH (1.0 equiv., 60 % in mineral oil), stirred for 1 h and treated with the bromide substrates (1.1 equiv.). The mixture was warmed to 40 ºC and stirred overnight. The mixture was cooled to room temperature. The crude product was absorbed on silica and purified by column chromatography on silica gel eluting with petrol–ethyl acetate (50:1) to give

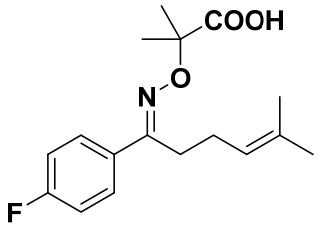
S3.[3]

3) A solution of **S3** (1.0 equiv.) in EtOH/H_2O (0.01 M, 2:1) was treated with NaOH (4.0 equiv.) and refluxed for 2 h. The mixture was cooled to room temperature and MeOH was removed *in vaco*. EtOAc was added and the layers were separated. The aqueous layer was then washed with EtOAc (× 3) and the combined organic fractions were dried ($MgSO_4$), filtered and evaporated. Purification is done by running column chromatography on silica gel, eluting with petrol/ethyl acetate (100:1) to obtain **S4**.[3]

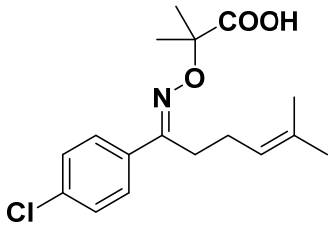
4) 2-(aminooxy)-2-methylpropanoic acid hydrochloride (1.2 equiv.) and NaOAc (2.4 equiv.) were added to a stirred solution of corresponding ketone (1.0 equiv.) in MeOH (0.2 M). The mixture was then stirred overnight at 50 ºC. Upon completion, the reaction was cooled to room temperature, quenched with H_2O (50 mL) and extracted with ethyl acetate. The combined organic extracts were then washed with H_2O (2 × 25 mL) and brine (25 mL), then dried over $MgSO_4$.The residue was purified by flash column chromatography on silica gel to give product **S5**.[3]



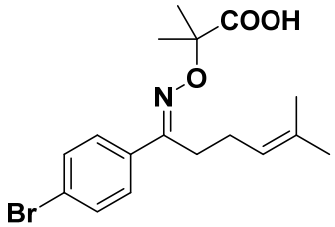
(*E*)-2-Methyl-2-(((5-methyl-1-phenylhex-4-en-1-ylidene)amino)oxy)propanoic acid (1aa): Prepared via general procedure A as a white solid (1.27 g, 88%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.86 (brs, 1H), 7.80 – 7.47 (m, 2H), 7.38 – 7.31 (m, 3H), 5.17 (t, *J* = 7.2 Hz, 1H), 2.90 – 2.65 (m, 2H), 2.31 – 2.19 (m, 2H), 1.66 (s, 3H), 1.61 (s, 6H), 1.56 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 177.3, 160.5, 135.0, 133.1, 129.6, 128.5, 126.5, 122.9, 81.6, 27.0, 25.6, 25.0, 24.3, 17.5. **FT-IR (thin film, KBr): ν (cm^{-1}):** 2921, 1711, 1449, 1304, 973, 791; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{17}H_{24}NO_3$ 290.1751, found 290.1754.



(*E*)-2-(((1-(4-Fluorophenyl)-5-methylhex-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1ab): Prepared via general procedure A as a white solid (1.03 g, 67%); m.p. 80-82 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.73 – 7.48 (m, 2H), 7.08 – 6.96 (m, 2H), 5.31 – 4.99 (m, 1H), 2.89 – 2.67 (m, 2H), 2.23 (q, *J* = 7.6 Hz, 2H), 1.65 (s, 3H), 1.59 (s, 6H), 1.54 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 176.6, 163.6 (d, *J* = 250.0 Hz), 159.6, 133.2, 131.0, 128.3 (d, *J* = 8.3 Hz), 122.7, 115.5 (d, *J* = 21.7 Hz), 81.7, 26.9, 25.6, 25.0, 24.3, 17.6**; FT-IR (thin film, KBr): ν (cm^{-1}):** 2988, 1711, 1607, 1513, 1302, 1177, 972, 835; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{17}H_{23}FNO_3$ 308.1662, found 308.1662.

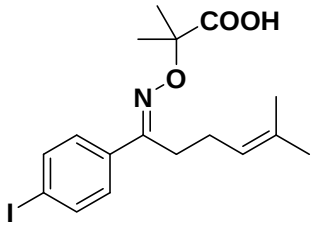


(*E*)-2-(((1-(4-Chlorophenyl)-5-methylhex-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1ac): Prepared via general procedure A as a white solid (1.3 g, 80%); m.p. 67-69 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.59 (brs, 1H), 7.55 (d, *J* = 8.6 Hz, 2H), 7.31 (d, *J* = 8.6 Hz, 2H), 5.16 (t, *J* = 7.3 Hz, 1H), 2.99 – 2.55 (m, 2H), 2.35 – 2.09 (m, 2H), 1.66 (s, 3H), 1.61 (s, 5H), 1.56 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 179.1, 158.5, 135.4, 133.8, 133.1, 128.6, 127.7, 122.9, 81.6, 26.9, 25.6, 25.0, 24.2, 17.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1711, 1493, 1453, 1298, 972, 830; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{17}H_{23}{}^{35}ClNO_3$ 324.1361, found 324.1358.

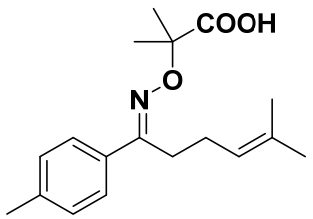


(*E*)-2-(((1-(4-Bromophenyl)-5-methylhex-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1ad): Prepared via general procedure A as a white solid (1.3 g, 70%); m.p. 77-79 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.52 – 7.42 (m, 4H), 5.15 (t, *J* = 7.3 Hz, 1H), 2.81 – 2.74 (m, 2H), 2.28 – 2.17 (m, 2H), 1.66 (s, 3H), 1.60 (s, 6H),

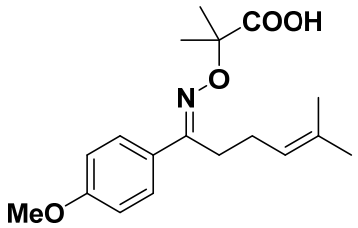
1.55 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 177.5, 159.1, 134.0, 133.2, 131.6, 128.0, 123.9, 122.7, 103.6, 81.7, 32.2 26.72, 25.6, 24.9, 24.2, 17.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2925, 1711, 1452, 1318, 1176, 972, 830; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{17}H_{23}{}^{79}BrNO_3$ 368.0856, found 368.0861.



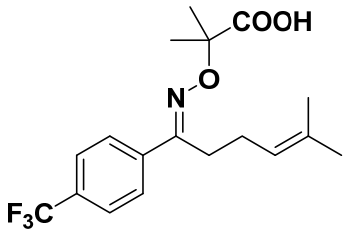
(*E*)-2-(((1-(4-Iodophenyl)-5-methylhex-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid(1ae): Prepared via general procedure A as a yellow solid (934 mg, 45%); m.p. 89-91 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.07 (brs, 1H), 7.68 (d, *J* = 8.5 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 5.15 (t, *J* = 7.4 Hz, 1H), 2.79 – 2.72 (m, 2H), 2.28 – 2.17 (m, 2H), 1.66 (s, 3H), 1.60 (s, 6H), 1.56 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 178.6, 158.9, 137.6, 134.8, 133.1, 128.1, 122.9, 95.7, 81.7, 26.7, 25.65, 25.0, 24.2, 17.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2925, 1711, 1541, 1452, 1318, 1096, 994, 830; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{17}H_{23}INO_3$ 416.0717, found 416.0719.



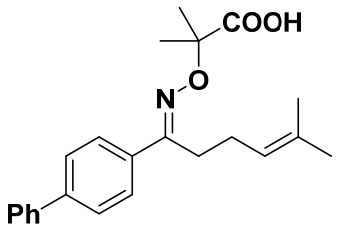
(*E*)-2-Methyl-2-(((5-methyl-1-(p-tolyl)hex-4-en-1-ylidene)amino)oxy)propanoic acid (1af): Prepared via general procedure A as a white solid (1.4 g, 90%); m.p. 67-68 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.51 (d, *J* = 8.2 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 5.17 (t, *J* = 7.3 Hz, 1H), 2.84 – 2.75 (m, 2H), 2.37 (s, 3H), 2.28 – 2.22 (m, 2H), 1.68 (s, 3H), 1.60 (s, 6H), 1.57 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 177.3, 160.5, 139.8, 133.0, 132.0, 129.2, 126.4, 122.9, 81.6, 27.0, 25.6, 25.1, 24.3, 21.3, 17.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2924, 1717, 1452, 1297, 1173, 970, 820; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{18}H_{26}NO_3$ 304.1907, found 304.1906.



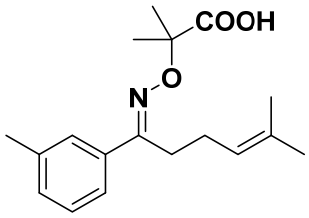
(*E*)-2-(((1-(4-Methoxyphenyl)-5-methylhex-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1ag): Prepared via general procedure A as a white solid (1.4 g, 90%); m.p. 97-98 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.57 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 5.17 (t, *J* = 7.3 Hz, 1H), 3.83 (s, 3H), 3.06 – 2.56 (m, 2H), 2.45 – 2.11 (m, 2H), 1.67 (s, 3H), 1.59 (s, 5H), 1.56 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 177.1, 160.9, 160.4, 133.1, 127.9, 127.2, 122.9, 114.0, 81.6, 55.4, 26.9, 25.7, 25.2, 24.4, 17.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2924, 1714, 1605, 1514, 1455, 1260, 969, 832; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{18}H_{26}NO_4$ 320.1856, found 320.1861.



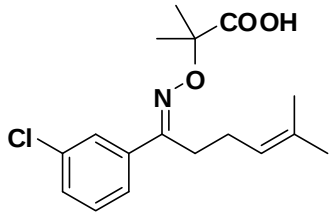
(*E*)-2-methyl-2-(((5-methyl-1-(4-(trifluoromethyl)phenyl)hex-4-en-1-ylidene)amino)oxy)propanoic acid (1ah): Prepared via general procedure A as a white solid (1.2 g, 70%); m.p. 92-93 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.72 (d, *J* = 8.2 Hz, 2H), 7.60 (d, *J* = 8.3 Hz, 2H), 5.19 – 5.11 (m, 1H), 2.93 – 2.67 (m, 2H), 2.25 (d, *J* = 7.7 Hz, 2H), 1.66 (s, 3H), 1.62 (s, 6H), 1.55 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 178.6, 158.4, 138.9, 133.2, 131.1 (q, *J* = 32.4 Hz), 126.7, 125.4 (q, *J* = 3.6 Hz), 122.8, 81.8 , 26.8 , 25.6 , 24.9 , 24.2, 17.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1717, 1615, 1520, 1455, 1260, 970, 832; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{18}H_{23}F_3NO_3$ 358.1625, found 358.1622.



(*E*)-2-(((1-([1,1'-Biphenyl]-4-yl)-5-methylhex-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1ai): Prepared via general procedure A as a white solid (1.5 g, 82 %); m.p. 88-90 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.70 (d, *J* = 8.4 Hz, 2H), 7.66 – 7.56 (m, 4H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.37 (t, *J* = 7.4 Hz, 1H), 5.19 (t, *J* = 7.3 Hz, 1H), 3.07 – 2.63 (m, 2H), 2.29 (q, *J* = 7.6 Hz, 2H), 1.69 (s, 3H), 1.63 (s, 6H), 1.58 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 179.0, 159.5, 142.2, 140.4, 134.2, 133.0, 128.9, 127.7, 127.2, 127.1, 126.9, 123.2, 81.6, 26.98, 25.7, 25.2, 24.3, 17.7; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2924, 1714, 1615, 1520, 1450, 1176, 975, 844; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{23}H_{28}NO_3$ 366.2064, found 366.2062.

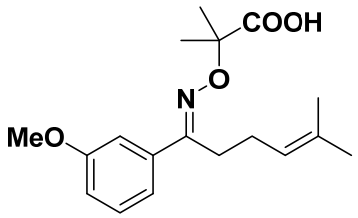


(*E*)-2-Methyl-2-(((5-methyl-1-(m-tolyl)hex-4-en-1-ylidene)amino)oxy)propanoic acid (1aj): Prepared via general procedure A as a white solid (1.3 g, 87%); m.p. 86-87 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.42 – 7.37 (m, 2H), 7.30 – 7.24 (m, 1H), 7.23 – 7.19 (m, 1H), 5.24 – 5.12 (m, 1H), 2.86 – 2.77 (m, 2H), 2.38 (s, 3H), 2.30 – 2.19 (m, 2H), 1.68 (d, *J* = 0.6 Hz, 3H), 1.61 (s, 6H), 1.57 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 176.7, 161.2, 138.3, 134.8, 133.2, 130.6, 128.5, 127.1, 123.7, 122.9, 81.7, 27.2, 25.7, 25.1, 24.4, 21.5, 17.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2917, 1717, 1615, 1520, 1305, 1174, 970, 827; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{18}H_{26}NO_3$ 304.1907, found 304.1911.

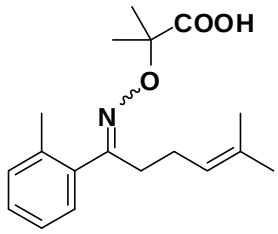


(*E*)-2-(((1-(3-Chlorophenyl)-5-methylhex-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1ak): Prepared via general procedure A as a white solid (1.3 g, 79%); m.p. 77-79 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.59 (t, *J* = 1.7 Hz, 1H), 7.49 (dt, *J* = 7.6, 1.3 Hz, 1H), 7.36 – 7.31 (m, 1H), 7.30 – 7.25 (m, 1H), 5.21 – 5.13 (m,

1H), 2.81 – 2.73 (m, 2H), 2.32 – 2.20 (m, 2H), 1.67 (s, 3H), 1.62 (s, 6H), 1.57 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 178.8, 158.4, 137.3, 134.5, 133.1, 129.7, 129.3, 126.6, 124.6, 122.9, 81.7, 26.9, 25.6, 24.9, 24.2, 17.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2917, 1717, 1615, 1520, 1305, 1174, 970, 827; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{17}H_{23}{}^{35}ClNO_3$ 324.1361, found 324.1358.

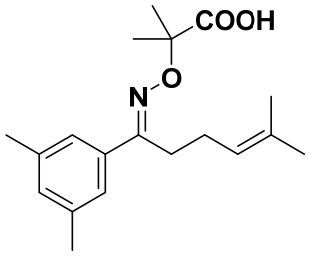


(*E*)-2-(((1-(3-Methoxyphenyl)-5-methylhex-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1al): Prepared via general procedure A as a white solid (1.4 g, 79%); m.p. 84-85 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 11.50 (s, 1H), 7.24 (t, *J* = 7.8 Hz, 1H), 7.19 – 7.14 (m, 2H), 6.88 (d, *J* = 7.6 Hz, 1H), 5.16 (t, *J* = 7.1 Hz, 1H), 3.78 (s, 3H), 2.82 – 2.71 (m, 2H), 2.29 – 2.16 (m, 2H), 1.65 (s, 3H), 1.59 (s, 6H), 1.56 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 177.0, 160.4, 159.6, 136.4, 133.1, 129.5, 122.9, 119.0, 115.1, 112.1, 81.7, 55.3, 27.1, 25.6, 25.1, 24.3, 17.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2924, 1715, 1615, 1457, 1377, 972, 835; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{18}H_{26}NO_4$ 320.1856, found 320.1854.

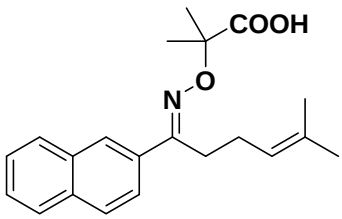


2-Methyl-2-(((5-methyl-1-(o-tolyl)hex-4-en-1-ylidene)amino)oxy)propanoic acid (1am): Prepared via general procedure A as a white solid (1.3 g, 87%); m.p. 62-64 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.28 – 7.24 (m, 1H), 7.24 – 7.16 (m, 3H), 5.12 – 5.04 (m, 1H), 2.82 – 2.69 (m, 1.4H)/2.51 (t, *J* = 7.5 Hz, 0.6H), 2.31 (s, 2.1H)/2.22 (s, 0.9H), 2.20 – 2.13 (m, 2H), 1.67 (s, 0.9H)/1.65 (s, 2.1H), 1.58 (s, 4.2H)/1.52 (s, 1.8H), 1.55 (s, 0.9H)/ 1.43 (s, 2.1H): **^{13}C NMR (150 MHz, $CDCl_3$)** δ 177.5/177.0, 162.6/160.9, 136.2/134.9, 135.6/134.4, 132.9/132.8, 130.8/129.8, 128.6, 128.35/128.31, 125.6/125.4, 123.0/122.8, 81.3, 36.3, 25.6, 24.3, 24.2/24.1, 20.2/19.6, 17.8/17.6; **FT-**

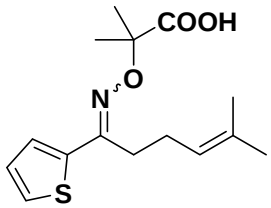
IR (thin film, KBr): ν (cm^{-1}): 2925, 1717, 1454, 1378, 1173, 968, 759; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{18}H_{26}NO_3$ 304.1907, found 304.1903.



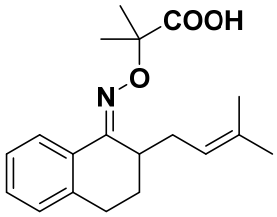
(*E*)-2-(((1-(3,5-Dimethylphenyl)-5-methylhex-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1an): Prepared via general procedure A as a white solid (1.2 g, 70%); m.p. 98-99 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.19 (s, 2H), 7.05 (s, 1H), 5.21 – 5.14 (m, 1H), 2.90 – 2.63 (m, 2H), 2.34 (s, 6H), 2.28 – 2.19 (m, 2H), 1.68 (s, 3H), 1.60 (s, 6H), 1.57 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 176.4, 161.5, 138.1, 134.7, 133.1, 131.5, 124.3, 122.9, 81.7, 27.3, 25.6, 25.1, 24.4, 21.3, 17.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2924, 1734, 1458, 1300, 1171, 967, 878; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{19}H_{28}NO_3$ 318.2064, found 318.2062.



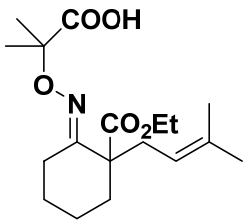
(*E*)-2-Methyl-2-(((5-methyl-1-(naphthalen-2-yl)hex-4-en-1-ylidene)amino)oxy)propanoic acid (1ao): Prepared via general procedure A as a white solid (1.5 g, 90%); m.p. 99-99.5 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 11.65 (s, 1H), 8.04 (s, 1H), 7.94 – 7.86 (m, 2H), 7.85 – 7.76 (m, 2H), 7.56 – 7.47 (m, 2H), 5.29 (t, *J* = 6.7 Hz, 1H), 2.96 (t, *J* = 7.8 Hz, 2H), 2.43 – 2.32 (m, 2H), 1.73 (s, 3H), 1.71 (s, 6H), 1.64 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 179.6, 159.4, 133.8, 133.1, 132.9, 132.8, 128.6, 128.2, 127.7, 126.7, 126.4, 126.2, 123.9, 123.3, 81.6, 26.8, 25.7, 25.3, 24.3, 17.7; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1704, 1450, 1300, 1174, 982, 831; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{21}H_{26}NO_3$ 340.1907, found 340.1902.



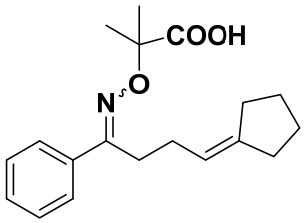
2-Methyl-2-(((5-methyl-1-(thiophen-2-yl)hex-4-en-1-ylidene)amino)oxy) propanoic acid (1ap): Prepared via general procedure A as a white solid (1.3 g, 92%); m.p. 66-67 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 11.69 (brs, 1H), 7.46 – 7.40 (m, 1H), 7.18 – 7.12 (m, 1H), 7.00 (t, *J* = 4.3 Hz, 0.5H)/6.91 (t, *J* = 4.1 Hz, 0.5H), 5.18 – 5.03 (m, 1H), 2.66 (t, *J* = 7.6 Hz, 2H), 2.32 – 2.18 (m, 2H) , 1.60 (s, 3H), 1.58 (s, 3H)/1.52 (s, 3H), 1.52 (s, 1.5H)/1.46 (s, 1.5H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 179.6/179.0, 155.2/150.6, 139.7/133.0, 132.7/131.7, 130.9/129.9, 127.3/127.0, 126.5/125.73, 123.1/123.1, 82.2/81.5, 33.9/27.8, 26.3/25.72, 25.67/25.4, 24.22/24.16, 17.7/17.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2928, 1713, 1559, 1247, 1097, 960, 897; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{15}H_{22}NO_3S$ 296.1315, found 296.1311.



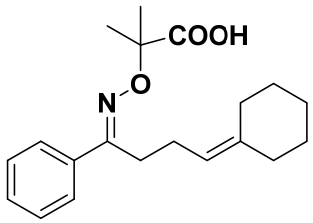
(*E*)-2-Methyl-2-(((2-(3-methylbut-2-en-1-yl)-3,4-dihydronaphthalen-1(2H)-ylidene)amino)oxy)propanoic acid (1aq): Prepared via general procedure A as a white solid (1.3 g, 86%); m.p. 106-107 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.91 (dd, *J* = 7.9, 0.9 Hz, 1H), 7.29 (dt, *J* = 7.2, 4.8 Hz, 1H), 7.16 (dd, *J* = 13.7, 7.5 Hz, 2H), 5.44 – 4.77 (m, 1H), 3.51 (td, *J* = 9.4, 4.9 Hz, 1H), 2.93 (qd, *J* = 12.7, 6.5 Hz, 1H), 2.76 – 2.61 (m, 1H), 2.46 – 2.27 (m, 1H), 2.10 (ddd, *J* = 27.7, 16.7, 8.4 Hz, 1H), 1.99 – 1.80 (m, 2H), 1.78 – 1.67 (m, 3H), 1.61 (s, 9H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 177.5, 159.3, 139.0, 133.6, 129.7, 129.3, 128.9, 126.3, 124.8, 121.7, 81.6, 32.9, 27.3, 25.8, 24.8, 24.5, 24.3, 24.2, 17.8; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1711, 1583, 1343, 1241, 950, 890; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{19}H_{26}NO_3$ 316.1907, found 316.1906.



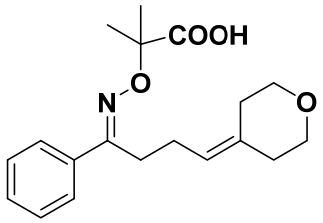
(*Z*)-2-(((2-(Ethoxycarbonyl)-2-(3-methylbut-2-en-1-yl)cyclohexylidene)amino)oxy)-2-methylpropanoic acid (1ar): Prepared via general procedure A as a white solid; m.p. 93-94 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.83 (s, 1H), 5.13 – 5.03 (m, 1H), 4.20 – 4.06 (m, 2H), 3.22 – 3.04 (m, 1H), 2.54 (dd, *J* = 14.6, 7.0 Hz, 1H), 2.44 – 2.26 (m, 2H), 2.00 – 1.84 (m, 1H), 1.81 – 1.71 (m, 1H), 1.70 – 1.61 (m, 1H), 1.65 (s, 3H), 1.56 (s, 3H), 1.51 (s, 6H), 1.49 – 1.33 (m, 3H), 1.26 – 1.18 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 177.7, 173.2, 162.11 134.1, 119.1, 81.2, 61.0, 54.2, 35.1, 34.0, 25.9, 25.4, 24.2, 24.12, 24.10, 22.5, 17.8, 14.1; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2928, 1713, 1559, 1247, 1097, 960, 897; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{18}H_{29}NNaO_5$ 362.1938, found 362.1940.



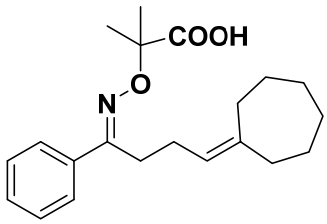
2-(((4-Cyclopentylidene-1-phenylbutylidene)amino)oxy)-2-methylpropanoic acid (1as): Prepared via general procedure A as a white solid (1.1 g, 78%); m.p. 86-87 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.56 (m, 2H), 7.42 – 7.32 (m, 3H), 5.41 – 5.22 (m, 1H), 2.87 – 2.81 (m, 1.3H)/2.81 – 2.74 (m, 0.7H), 2.32 – 2.07 (m, 6H), 1.90 – 1.53 (m, 4H), 1.61 (s, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 178.0/177.8, 160.5/160.4, 144.8/143.8, 135.2/129.6, 128.53/128.50, 126.51/126.48, 124.1, 118.4, 81.62/81.58, 34.9/33.6, 32.5/31.1, 28.5/26.8, 26.6/26.5, 26.33/26.26, 24.5/23.4, 24.3; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2948, 1711, 1541, 1396, 1241, 954, 837; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{19}H_{26}NO_3$ 316.1907, found 316.1912.



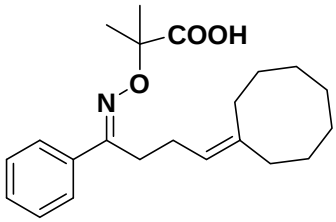
(*E*)-2-(((4-Cyclohexylidene-1-phenylbutylidene)amino)oxy)-2-methylpropanoic acid (1at): Prepared via general procedure A as a white solid; m.p. 93-95 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.26 (s, 1H), 7.66 – 7.60 (m, 2H), 7.40 – 7.32 (m, 3H), 5.14 (t, *J* = 7.5 Hz, 1H), 2.85 – 2.76 (m, 2H), 2.33 – 2.23 (m, 2H), 2.12 – 2.07 (m, 2H), 2.07 – 2.02 (m, 2H), 1.62 (s, 6H), 1.56 – 1.41 (m, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 178.7, 159.9, 141.1, 135.3, 129.4, 128.5, 126.5, 119.7, 81.5, 37.1, 28.6, 28.5, 27.8, 27.4, 26.9, 24.3, 24.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2882, 1713, 1584, 1391, 1206, 952, 864; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{20}H_{28}NO_3$ 330.2069, found 330.2061.



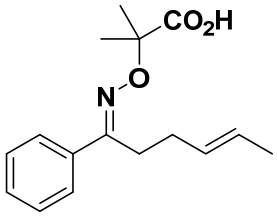
(*E*)-2-Methyl-2-(((1-phenyl-4-(tetrahydro-4H-pyran-4-ylidene)butylidene)amino) oxy)propanoic acid (1au): Prepared via general procedure A as a white solid; m.p. 81-83 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.27 (brs, 1H), 7.63 – 7.59 (m, 2H), 7.38 – 7.32 (m, 3H), 5.24 (t, *J* = 7.5 Hz, 1H), 3.65 – 3.53 (m, 4H), 2.86 – 2.78 (m, 2H), 2.30 (dd, *J* = 15.4, 7.6 Hz, 2H), 2.20 (t, *J* = 5.1 Hz, 2H), 2.15 (t, *J* = 5.2 Hz, 2H), 1.60 (s, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 178.4, 159.2, 135.5, 135.3, 129.4, 128.5, 126.5, 121.9, 81.5, 69.4, 68.7, 36.78, 29.5, 27.0, 24.3, 23.9; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2922, 1699, 1559, 1457, 967, 743; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{19}H_{25}NNaO_4$ 354.1676 found 354.1680.



(*E*)-2-(((4-Cycloheptylidene-1-phenylbutylidene)amino)oxy)-2-methylpropanoic acid (1av): Prepared via general procedure A as a white solid; m.p. 80-82 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 – 7.59 (m, 2H), 7.41 – 7.34 (m, 3H), 5.17 (t, *J* = 7.3 Hz, 1H), 2.88 – 2.77 (m, 2H), 2.29 – 2.21 (m, 2H), 2.20 – 2.13 (m, 4H), 1.61 (s, 6H), 1.56 – 1.43 (m, 8H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 160.8, 142.8, 135.0, 129.7, 128.6, 126.5, 126.2, 123.2, 81.7, 37.8, 29.8, 29.1, 29.0, 27.2, 27.0, 24.7, 24.4; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2922, 1699, 1559, 1457, 967, 743; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{21}H_{30}NO_3$ 344.2220, found 344.2225.

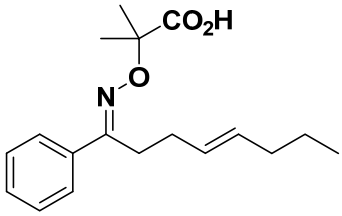


(*E*)-2-(((4-Cyclooctylidene-1-phenylbutylidene)amino)oxy)-2-methylpropanoic acid (1aw): Prepared via general procedure A as a white solid; m.p. 82-84 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.66 – 7.60 (m, 2H), 7.39 – 7.34 (m, 3H), 5.20 (t, *J* = 7.3 Hz, 1H), 2.89 – 2.77 (m, 2H), 2.28 (dd, *J* = 15.6, 7.6 Hz, 2H), 2.18 – 2.08 (m, 4H), 1.62 (s, 3H), 1.65 – 1.61 (m, 3H), 1.53 – 1.41 (m, 7H).; **^{13}C NMR (100 MHz, $CDCl_3$)** δ 178.0, 160.3, 142.4, 135.1, 129.6, 128.5, 126.5, 123.6, 81.6, 37.7, 29.0, 27.23, 27.18, 26.9, 26.3, 26.1, 24.9, 24.3; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2922, 1699, 1559, 1457, 967, 743; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{22}H_{32}NO_3$ 358.2377, found 358.2381.

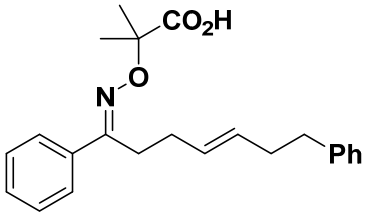


2-Methyl-2-((((*1E,4E*)-1-phenylhex-4-en-1-ylidene)amino)oxy)propanoic acid (1ba) Prepared via general procedure A as a white solid; m.p. 71-72 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.65 – 7.55 (m, 2H), 7.41 – 7.34 (m, 3H), 5.48 – 5.42 (m, 2H), 2.89 – 2.80 (m, 2H), 2.32 – 2.19 (m, 2H), 1.66 – 1.62 (m, 3H), 1.61 (s, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 177.6, 160.3, 135.0, 129.8, 129.6, 128.6, 126.5, 126.0, 81.7,

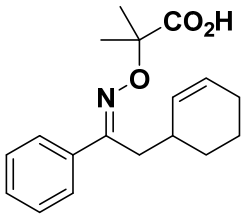
29.4, 26.93, 24.3, 17.9; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2928, 1706, 1450, 1177, 973, 768; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{16}H_{22}NO_3$ 276.1594, found 276.1596.



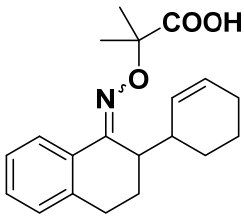
2-methyl-2-((((1E,4E)-1-phenyloct-4-en-1-ylidene)amino)oxy)propanoic acid (1bb): Prepared via general procedure A as a white solid; m.p. 62-63 ℃; **^{1}H NMR (600 MHz, CDCl$_3$)** δ ^{1}H NMR (600 MHz, cdcl$_3$) δ 7.60 (dd, *J* = 7.8, 1.8 Hz, 2H), 7.40 – 7.35 (m, 3H), 5.44 – 5.41 (m, 2H), 2.89 – 2.82 (m, 2H), 2.30 – 2.23 (m, 2H), 1.97 – 1.91 (m, 2H), 1.61 (s, 6H), 1.37 – 1.31 (m, 2H), 0.87 (t, *J* = 7.4 Hz, 2H); **^{13}C NMR (150 MHz, CDCl$_3$)** δ 177.2, 160.4, 135.0, 131.5, 129.6, 128.6, 128.5, 126.5, 81.7, 34.5, 29.4, 27.0, 24.3, 22.5, 13.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2930, 1720, 1530, 1170, 971, 767; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{18}H_{26}NO_3$ 304.1907, found 304.1907.



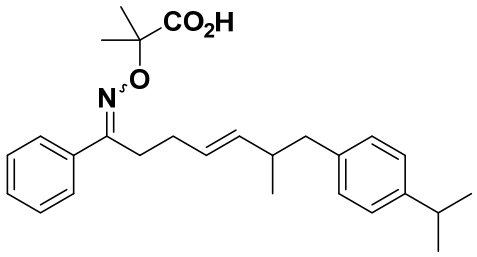
2-((((1E,4E)-1,7-diphenylhept-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1bc): Prepared via general procedure A as a white solid; m.p. 73-74 ℃; **^{1}H NMR (600 MHz, CDCl$_3$)** δ 7.65 – 7.60 (m, 2H), 7.42 – 7.33 (m, 3H), 7.30 (t, *J* = 7.5 Hz, 2H), 7.23 – 7.16 (m, 3H), 5.53 – 5.48 (m, 2H), 2.86 (t, *J* = 7.7 Hz, 2H), 2.68 – 2.64 (m, 2H), 2.34 – 2.26 (m, 4H), 1.64 (s, 6H); **^{13}C NMR (150 MHz, CDCl$_3$)** δ 178.5, 159.7, 142.0, 135.3, 130.5, 129.5, 129.4, 128.5, 128.4, 128.3, 126.5, 125.8, 81.5, 35.9, 34.4, 29.4, 26.8, 24.3; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1715, 1534, 1172, 969, 765; **HRMS** (ESI) [M + Na]$^+$ Calculated for $C_{23}H_{27}NNaO_3$ 388.1883, found 388.1884.



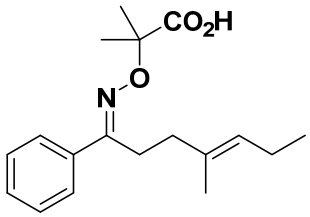
(*E*)-2-(((2-(Cyclohex-2-en-1-yl)-1-phenylethylidene)amino)oxy)-2-methylpropanoic acid (1bd): Prepared via general procedure A as a white solid (670 mg, 65%); m.p. 62-63 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.63 – 7.58 (m, 2H), 7.40 – 7.33 (m, 3H), 5.71 – 5.66 (m, 1H), 5.56 – 5.50 (m, 1H), 2.88 (dd, *J* = 13.1, 8.9 Hz, 1H), 2.77 (dd, *J* = 13.1, 6.7 Hz, 1H), 2.47 – 2.40 (m, 1H), 1.99 – 1.93 (m, 2H), 1.74 – 1.64 (m, 2H), 1.60 (s, 3H), 1.59 (s, 3H), 1.50 – 1.43 (m, 1H), 1.35 – 1.28 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 177.2, 159.6, 135.2, 130.5, 129.6, 128.5, 127.9, 126.6, 81.7, 33.2, 32.5, 28.6, 25.1, 24.3, 20.9; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1711, 1452, 1299, 1172, 927, 766; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{18}H_{24}NO_3$ 302.1751, found 302.1746.



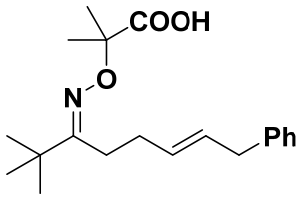
2-(((2-(Cyclohex-2-en-1-yl)-3,4-dihydronaphthalen-1(*2H*)-ylidene)amino)oxy)-2-methylpropanoic acid (1be): Prepared via general procedure A as a white solid; m.p. 119-121 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.84 – 7.73 (m, 1H), 7.36 – 7.28 (m, 1H), 7.23 – 7.12 (m, 2H), 5.81 – 5.76 (m, 0.6H)/5.45 – 5.35 (m, 0.4H), 5.74 – 5.65 (m, 1H), 3.53 – 3.38 (m, 1H), 3.05 – 2.87 (m, 1H), 2.86 – 2.67 (m, 1H), 2.38 – 1.38 (m, 15H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 176.6/176.2, 160.2/159.5, 138.8/138.7, 129.88/129.87, 129.8/129.6, 129.7/129.4, 129.0/128.8, 127.9/127.8, 126.39/126.36, 125.3/125.2, 81.9/81.7, 36.8/36.5, 33.4/33.2, 26.6, 25.4/25.0, 24.8/24.7, 24.5/24.2, 24.39/24.36, 23.7/23.6, 21.1/20.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1711, 1541, 1324, 952, 882; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{20}H_{26}NO_3$ 328.1907, found 328.1905.



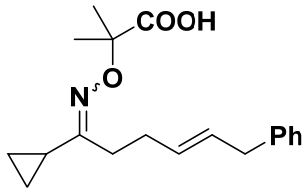
2-((((*1E,4E*)-7-(4-Isopropylphenyl)-6-methyl-1-phenylhept-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1bf): Prepared via general procedure A as a white solid; m.p. 112-114℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.63 – 7.54 (m, 2H), 7.41 – 7.33 (m, 3H), 7.17 – 7.10 (m, 2H), 7.08 – 7.01 (m, 2H), 5.47 – 5.24 (m, 2H), 2.94 – 2.66 (m, 3H), 2.65 – 2.56 (m, 1H), 2.53 – 2.31 (m, 2H), 2.32 – 2.12 (m, 2H), 1.63 (s, 5H)/1.61 (s, 1H), 1.25 (d, *J* = 6.9 Hz, 5H)/1.21 (d, *J* = 6.9 Hz, 1H), 0.94 (d, *J* = 6.6 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 178.5, 159.8/159.6, 146.3/146.2, 138.1/138.0, 136.8/136.7, 135.3/135.1, 129.4, 129.1, 128.5/128.0, 127.0/126.9, 126.5/126.4, 126.0, 81.5, 43.3/43.2, 38.19, 33.9/33.7, 29.4, 27.0/26.8, 24.4/24.3, 24.1, 20.9/19.7; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2960, 1715, 1452, 1172, 969, 766; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{27}H_{35}NNaO_3$ 444.2509, found 444.2514.



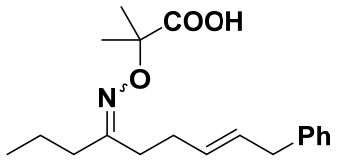
2-Methyl-2-((((*1E,4E*)-4-methyl-1-phenylhept-4-en-1-ylidene)amino)oxy) propanoic acid (1bg): Prepared via general procedure A as a white solid (1.2 g, 83%); m.p. 69-70 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 11.20 (s, 1H), 7.59 (dd, *J* = 6.6, 3.1 Hz, 2H), 7.40 – 7.29 (m, 3H), 5.11 (t, *J* = 6.9 Hz, 1H), 2.93 – 2.82 (m, 2H), 2.27 – 2.17 (m, 2H), 1.96 (p, *J* = 7.4 Hz, 2H), 1.64 (s, 3H), 1.60 (s, 6H), 0.91 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 179.2, 159.9, 135.5, 133.3, 129.4, 128.5, 127.5, 126.5, 81.4, 36.2, 25.9, 24.2, 21.2, 15.7, 14.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2963, 1706, 1450, 1171, 970, 764; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{18}H_{26}NO_3$ 304.1907, found 304.1912.



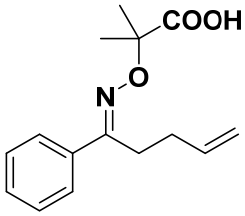
2-((((*3E,6E*)-2,2-Dimethyl-8-phenyloct-6-en-3-ylidene)amino)oxy)-2-methylpropanoic acid (1bh): Prepared via general procedure A as a white solid; m.p. 59-61 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.31 (t, *J* = 7.6 Hz, 2H), 7.25 – 7.17 (m, 3H), 5.69 – 5.62 (m, 1H), 5.55 (dt, *J* = 13.6, 6.5 Hz, 1H), 3.37 (d, *J* = 6.5 Hz, 2H), 2.42 – 2.35 (m, 2H), 2.33 – 2.25 (m, 2H), 1.53 (s, 6H), 1.15 (s, 9H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 177.5, 169.0, 140.6, 130.8, 129.6, 128.5, 128.4, 126.0, 81.0, 38.9, 37.9, 29.4, 27.6, 26.5, 24.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2924, 1625, 1450, 1337, 970; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{20}H_{29}NNaO_3$ 354.2040, found 354.2035.



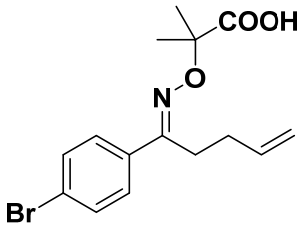
2-(((*4E*-1-Cyclopropyl-6-phenylhex-4-en-1-ylidene)amino)oxy)-2-methyl propanoic acid (1bi): Prepared via general procedure A as a white solid; m.p. 46-47 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.34 – 7.27 (m, 2H), 7.24 – 7.15 (m, 3H), 5.71 – 5.58 (m, 1H), 5.56 – 5.43 (m, 1H), 3.34 (t, *J* = 6.9 Hz, 2H), 2.41 – 2.20 (m, 4H), 1.96 – 1.88 (m, 0.6H)/ 1.55 – 1.48 (m, 0.4H), 1.53 (s, 2H)/1.49 (s, 4H), 0.93 – 0.69 (m, 4H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 177.5/177.3, 164.4/163.3, 140.6/140.5, 130.2, 130.0/129.9, 128.5, 128.4/128.3, 126.0/125.9, 81.0/80.9, 38.93/38.91, 29.2/29.0, 28.7/28.0, 24.3/24.2, 14.3/9.4, 6.2, 5.7; **FT-IR (thin film, KBr): ν (cm^{-1}):**2880, 1710, 1461, 1172, 950; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{19}H_{26}NO_3$ 316.1913, found 316.1913.



2-Methyl-2-(((*7E*-9-phenylnon-7-en-4-ylidene)amino)oxy)propanoic acid (1bj): Prepared via general procedure A as a white solid; m.p. 48-49 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.33 – 7.27 (m, 2H), 7.24 – 7.12 (m, 3H), 5.67 – 5.60 (m, 1H), 5.53 – 5.46 (m, 1H), 3.34 (t, *J* = 6.5 Hz, 2H), 2.42 (t, *J* = 7.7 Hz, 1H)/2.19 (t, *J* = 7.5 Hz, 1H), 2.34 – 2.30 (m, 2H), 2.29 – 2.25 (m, 2H), 1.59 – 1.52 (m, 2H), 1.51 (s, 3H)/1.50 (s, 3H), 0.96 (t, *J* = 7.4 Hz, 1.5H)/0.92 (t, *J* = 7.4 Hz, 1.5H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 177.8/177.7, 163.24/163.18, 140.6/140.5, 130.2/130.1, 130.1/130.0, 128.5/128.4, 128.37/128.34, 126.0/125.9, 80.8, 39.0/38.9, 36.4/34.1, 30.5/28.8, 28.6/28.4, 24.22/24.21, 19.2/19.1, 14.2/13.6; **FT-IR (thin film, KBr): ν (cm^{-1}) :** 2887, 1703, 1630, 1450, 970; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{19}H_{28}NO_3$ 318.2069, found 318.2063.

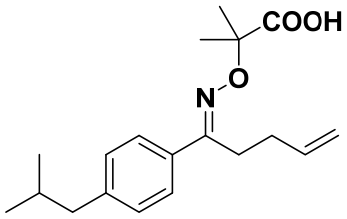


(*E*)-2-Methyl-2-(((1-phenylpent-4-en-1-ylidene)amino)oxy)propanoic acid (1ca) : Prepared via general procedure A as a white solid; m.p. 58-59 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 11.62 (brs, 1H), 7.71 – 7.55 (m, 2H), 7.44 – 7.33 (m, 3H), 5.98 – 5.80 (m, 1H), 5.08 (d, *J* = 17.1 Hz, 1H), 5.02 (d, *J* = 10.1 Hz, 1H), 2.91 (t, *J* = 7.8 Hz, 2H), 2.45 – 2.30 (m, 2H), 1.65 (s, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 179.9, 158.9, 137.6, 135.4, 129.4, 128.5, 126.5, 115.2, 81.4, 30.5, 26.2, 24.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2963, 1706, 1450, 1171, 970, 764; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{15}H_{19}NNaO_3$ 284.1257, found 284.1253.

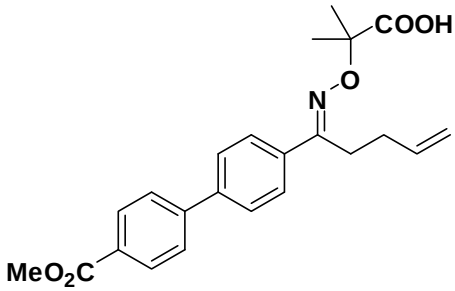


(*E*)-2-(((1-(4-Bromophenyl)pent-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1cb); Prepared via general procedure A as a white solid; m.p. 75-76 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.49 – 7.44 (m, 4H), 5.90 – 5.77 (m, 1H), 5.07 – 4.94 (m, 2H), 2.91 – 2.77 (m, 2H), 2.36 – 2.24 (m, 2H), 1.60 (s, 6H); **^{13}C NMR (150 MHz,**

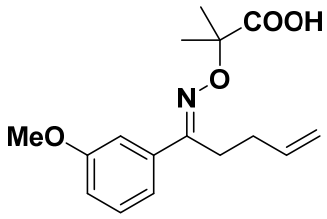
$CDCl_3$) δ 178.0, 158.4, 137.1, 134.0, 131.7, 128.0, 123.8, 115.4, 81.7, 30.3, 25.9, 24.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2925, 1711, 1452, 1318, 1176, 972, 830; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{15}H_{19}{}^{79}BrNO_3$ 340.0543,found 340.0545.



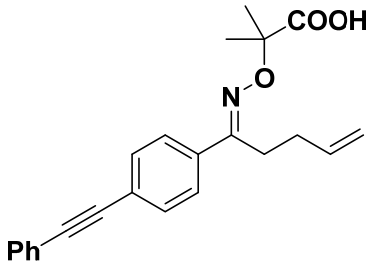
(*E*)-2-(((1-(4-Isobutylphenyl)pent-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1cc): Prepared via general procedure A as a white solid; m.p. 68-69 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.53 (d, *J* = 7.6 Hz, 2H), 7.15 (d, *J* = 7.9 Hz, 2H), 5.86 (ddt, *J* = 16.8, 10.4, 6.6 Hz, 1H), 5.05 (d, *J* = 17.1 Hz, 1H), 4.99 (d, *J* = 10.2 Hz, 1H), 2.95 – 2.79 (m, 1H), 2.49 (d, *J* = 7.2 Hz, 2H), 2.37 – 2.29 (m, 2H), 1.91 – 1.83 (m, 1H), 1.61 (s, 6H), 0.91 (d, *J* = 6.4 Hz, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 178.2, 159.7, 143.5, 137.4, 132.4, 129.3, 126.2, 115.2, 81.5, 45.2, 30.5, 30.1, 26.2, 24.3, 22.3; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2924, 1717, 1452, 1297, 1173, 970, 820; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{19}H_{27}NNaO_3$ 340.1883, found 340.1878.



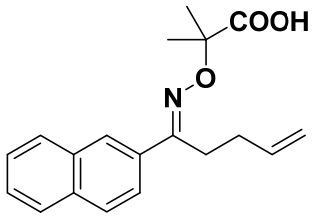
(*E*)-2-(((1-(4'-acetoxy-[1,1'-biphenyl]-4-yl)pent-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1cd): Prepared via general procedure A as a white solid; m.p. 129-130 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.10 (d, *J* = 8.4 Hz, 2H), 7.71 (d, *J* = 8.4 Hz, 2H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.60 (d, *J* = 8.4 Hz, 2H), 5.87 (ddt, *J* = 16.8, 10.1, 6.6 Hz, 1H), 5.07 (dd, *J* = 17.1, 1.5 Hz, 1H), 5.00 (d, *J* = 10.2 Hz, 1H), 3.94 (s, 3H), 3.08 – 2.83 (m, 2H), 2.36 (q, *J* = 7.3 Hz, 2H), 1.63 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 178.6, 166.9, 158.7, 144.7, 140.9, 137.3, 135.0, 130.1, 127.3, 127.0, 126.9, 126.2, 115.3, 81.7, 52.2, 30.5, 26.1, 24.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2931, 1717, 1605, 1278, 1179, 920; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{23}H_{25}NNaO_5$ 418.1625, found 418.1621.



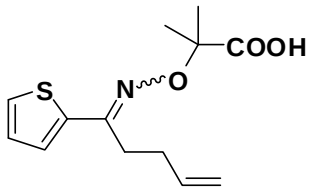
(*E*)-2-(((1-(3-Methoxyphenyl)pent-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1ce): Prepared via general procedure A as a white solid; m.p. 129-130 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.26 (t, *J* = 8.1 Hz, 1H), 7.19 – 7.14 (m, 2H), 6.94 – 6.88 (m, 1H), 5.84 (ddt, *J* = 16.8, 10.2, 6.6 Hz, 1H), 5.04 (dd, *J* = 17.1, 1.5 Hz, 1H), 4.98 (dd, *J* = 10.3, 1.0 Hz, 1H), 3.79 (s, 3H), 2.89 – 2.81 (m, 2H), 2.36 – 2.27 (m, 2H), 1.60 (s, 6H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 179.4, 159.6, 158.8, 137.5, 136.7, 129.2, 119.0, 115.1, 114.8, 112.1, 81.5, 55.2, 30.5, 26.3, 24.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2924, 1715, 1615, 1457, 1377, 972, 835; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{16}H_{21}NNaO_4$ 314.1363, found 314.1361.



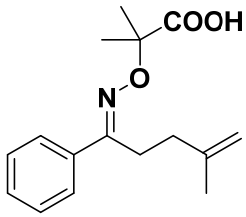
(*E*)-2-Methyl-2-(((1-(4-(phenylethynyl)phenyl)pent-4-en-1-ylidene)amino)oxy) propanoic acid (1cf): Prepared via general procedure A as a white solid; m.p. 117-119 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.61 (d, *J* = 8.4 Hz, 2H), 7.57 – 7.50 (m, 4H), 7.40 – 7.32 (m, 3H), 5.86 (ddt, *J* = 16.8, 10.2, 6.6 Hz, 1H), 5.06 (dd, *J* = 17.0, 1.3 Hz, 1H), 5.00 (d, *J* = 10.2 Hz, 1H), 2.98 – 2.76 (m, 2H), 2.34 (dd, *J* = 14.8, 7.3 Hz, 2H), 1.63 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 178.6, 158.6, 137.3, 134.8, 131.7, 131.6, 128.4, 128.3, 126.3, 124.4, 123.0, 115.3, 90.9, 89.0, 81.67, 3.43, 25.9, 24.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2958, 1789, 1540, 1389, 952; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{23}H_{23}NNaO_3$ 384.1570, found 384.1567.



(*E*)-2-Methyl-2-(((1-(naphthalen-2-yl)pent-4-en-1-ylidene)amino)oxy)propanoic acid (1cg): Prepared via general procedure A as a white solid; m.p. 96-98 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.53 (s, 1H), 8.02 (s, 1H), 7.92 – 7.85 (m, 2H), 7.85 – 7.75 (m, 2H), 7.55 – 7.47 (m, 2H), 5.95 (ddt, *J* = 16.8, 10.2, 6.6 Hz, 1H), 5.13 (dd, *J* = 17.1, 1.4 Hz, 1H), 5.05 (d, *J* = 10.2 Hz, 1H), 3.12 – 2.89 (m, 2H), 2.45 (dd, *J* = 14.9, 7.2 Hz, 2H), 1.71 (s, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 179.6, 159.0, 137.6, 133.8, 133.1, 132.8, 128.6, 128.2, 127.7, 126.8, 126.4, 126.2, 123.9, 115.3, 81.7, 30.7, 26.0, 24.3; **IR:** 2926, 1704, 1450, 1300, 1174, 982, 831; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{19}H_{21}NNaO_3$ 334.1414, found 334.1409.

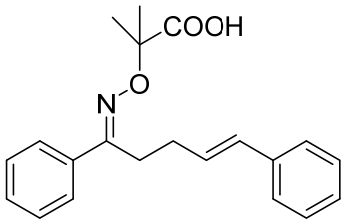


2-Methyl-2-(((1-(thiophen-2-yl)pent-4-en-1-ylidene)amino)oxy)propanoic acid (1ch): Prepared via general procedure A as a white solid; m.p. 65-66 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.90 (s, 1H), 7.53 – 7.44 (m, 0.6H)/7.25 – 7.18 (m, 1.4H), 7.07 (dd, *J* = 5.1, 3.9 Hz, 0.3H)/6.98 (dd, *J* = 5.0, 3.7 Hz, 0.7H), 5.95 – 5.78 (m, 1H), 5.10 – 4.95 (m, 2H), 2.86 – 2.76 (m, 2H), 2.48 – 2.31 (m, 2H), 1.66 (s, 1.8H)/1.59 (s, 4.2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 179.3/178.7, 155.0/150.3, 139.5/137.5, 137.4/131.7, 131.1/130.0, 127.5/127.1, 126.7/125.8, 115.5/115.4, 82.4/81.7, 33.2/31.6, 30.8/27.1, 24.3/24.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2928, 1713, 1559, 1247, 1097, 960, 897; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{13}H_{17}NNaO_3S$ 290.0821, found 290.0819.



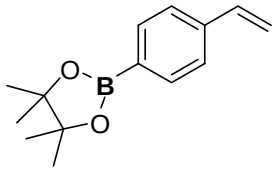
(*E*)-2-Methyl-2-(((4-methyl-1-phenylpent-4-en-1-ylidene)amino)oxy)propanoic acid (1ci): Prepared via general procedure A as a white solid; m.p. 99-100 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.60 (dd, *J* = 7.7, 1.7 Hz, 2H), 7.42 – 7.34 (m, 3H), 4.75 (s, 1H), 4.70 (s, 1H), 3.05 – 2.82 (m, 2H), 2.38 – 2.16 (m, 2H), 1.78 (s, 3H), 1.61 (s, 6H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 177.4, 160.2, 144.7, 134.9, 129.6, 128.6, 126.4,

110.7, 81.7, 34.3, 25.4, 24.7, 22.3; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1706, 1447, 1299, 1178, 960; **HRMS** (ESI) [M + Na]$^+$ Calculated for $C_{16}H_{21}NNaO_3$ 298.1414, found 298.1409.

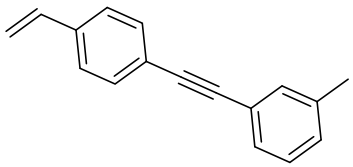


2-((((*1E,4E*)-1,5-Diphenylpent-4-en-1-ylidene)amino)oxy)-2-methylpropanoic acid (1cj): Prepared via general procedure A as a white solid; m.p. 89-91 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.50 (dd, *J* = 6.6, 2.9 Hz, 2H), 7.28 – 7.20 (m, 7H), 7.19 – 7.15 (m, 1H), 6.29 (d, *J* = 15.8 Hz, 1H), 6.11 (dt, *J* = 15.7, 6.9 Hz, 1H), 2.88 (t, *J* = 7.7 Hz, 2H), 2.34 (q, *J* = 7.2 Hz, 2H), 1.37 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 180.8, 158.6, 137.6, 135.9, 130.2, 129.7, 128.9, 128.4, 128.3, 126.9, 126.6, 126.0, 82.6, 29.7, 26.6, 24.8; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2960, 1714, 1454, 1165, 965; **HRMS** (CI) [M + H]$^+$ Calculated for $C_{21}H_{24}NO_3$ 338.1756, found 338.1754.

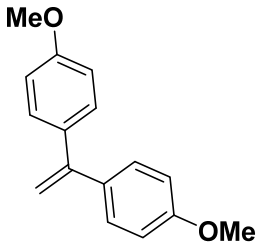
3.3 Procedures for the preparation of alkenes (4a-4g).



4,4,5,5-Tetramethyl-2-(4-vinylphenyl)-1,3,2-dioxaborolane (4a)[4]: 4-vinylphenyl) boronic acid (10 mmol) was added to a mixture of 2,3-dimethylbutane-2,3-diol (20 mmol) and anhydrous $MgSO_4$ (10 mmol) in Et_2O (0.2 M) at room temperature and then stirred for 12h. Next, the reaction mixture was filtered, then concentrated on a rotary evaporator. The residue was purified by flash column chromatography on silica gel (petroleum/ethyl acetate = 20:1) to give the product as a white solid (1.98 g, 86 %); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.47 (d, *J* = 8.0 Hz, 2H), 6.78 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.86 (dd, *J* = 17.6, 0.8 Hz, 1H), 5.34 (dd, *J* = 10.9, 0.8 Hz, 1H), 1.39 (s, 12H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 140.3, 136.9, 135.1, 125.6, 114.9, 83.7, 24.9; **^{11}B NMR (128 MHz, $CDCl_3$)** δ 30.90 (s).

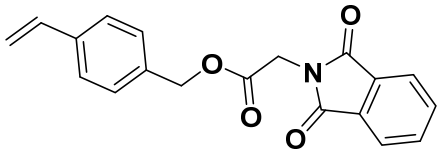


1-Methyl-3-((4-vinylphenyl)ethynyl)benzene (4b): $PdCl_2$•PPh_3 (0.25 mmol, 0.05 equiv.), 1-ethynyl-3-methylbenzene (6 mmol,1.2 equiv), 1-bromo-4-vinylbenzene (5 mmol, 1.0 equiv.), and NEt_3(20 mmol ,4 equiv.) were added to anhydrous DMF (15 ml,0.33 M) and stirred at room temperature for 5 minutes. CuI (0.25 mmol,0.05 equiv.) was added to the mixture and then heated to 80 °C for 24h. Cool the mixture to room temperature, quenched with H_2O (10 mL) and extracted with Et_2O (3 × 25 mL). The combined organic layers were washed with H_2O (2 × 25 mL) and brine (25 mL), then dried over $MgSO_4$ and concentrated. The residue was purified by flash column chromatography on silica gel (petroleum ether) and yellow solid was obtained (60%, 655 mg). m.p. 53-54. ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.41 (d, *J* = 8.3 Hz, 2H), 7.29 (dd, *J* = 15.7, 8.1 Hz, 4H), 7.16 (dd, *J* = 8.6, 6.5 Hz, 1H), 7.07 (d, *J* = 7.6 Hz, 1H), 6.63 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.70 (d, *J* = 17.6 Hz, 1H), 5.22 (d, *J* = 10.9 Hz, 1H), 2.28 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 138.1, 137.4, 136.3, 132.2, 131.8, 129.2, 128.7, 128.3, 126.2, 123.1, 122.7, 114.7, 90.3, 89.1, 21.3; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1711, 1611, 1561, 870**; HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{17}H_{14}Na$ 241.0988, found 241.0978.

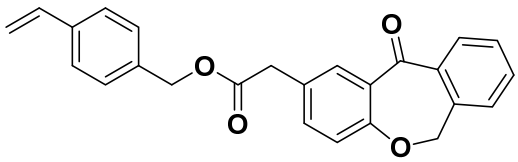


4,4'-(Ethene-1,1-diyl)bis(methoxybenzene) (4c): Ph_3PMeBr (4.29 g, 12.0 mmol) was added to an oven-dried transparent Schlenk tube (100 mL) equipped with a stirring bar. The tube was evacuated and filled with argon. Anhydrous tetrahydrofuran (40.0 mL) was added to the tube under argon, then *n*-BuLi (2.5 M, 4.8 mL, 12.0 mmol) was added dropwise to the solution at 0 °C. The mixture was stirred at 0 °C for 15 min, then a THF solution of bis(4-methoxyphenyl)methanone (10 mmol) was added at 0 °C. The reaction mixture was stirred at r.t. for 24 h. The resulting solution was

quenched with aqueous solution of NH_4Cl and the mixture was extracted with ethyl acetate (3 × 40 mL). The combined organic phases were dried over $MgSO_4$. The residue was purified by column chromatography on silica gel to obtain white solid. (48%,1.15 g); **^{1}H NMR (600 MHz, $CDCl_3$)** δ ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.4 Hz, 4H), 6.86 (d, *J* = 8.5 Hz, 4H), 5.29 (s, 2H), 3.83 (s, 6H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 159.3, 149.0, 134.3, 129.4, 113.5, 111.6, 55.3.

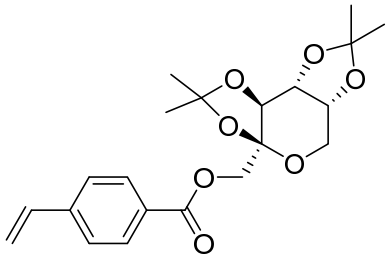


4-(3-(5-Phenyl-3,4-dihydro-2H-pyrrol-2-yl)prop-1-en-1-yl) benzyl 2-(1,3-dioxoisoindolin-2-yl)acetate (4d): In a dry RB flask, 2-(1,3-dioxoisoindolin-2-yl) acetic acid (1.25 g, 5 mmol) was weigh out and DMF (25 mL) was added. K_2CO_3 (1.04 g, 6.5 mmol) and KI (1.25 g, 6.5 mmol) was then added and stirred. To this stirring suspension 4-vinylbenzyl chloride (839 mg, 5.5 mmol) was added and stirred for 12h at room temperature. After the reaction is completed, EtOAc (50 mL) and water (30 mL) were added to the mixture and extracted and washed three times with water. The organic layer was collected and dried. The residue was purified by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 20: 1). White solid. (75%, 1.94 g) ; m.p. 105-106 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.94 – 7.80 (m, 2H), 7.80 – 7.65 (m, 2H), 7.39 (d, *J* = 8.1 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 6.70 (dd, *J* = 17.6, 10.9 Hz, 1H), 6.70 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.75 (d, *J* = 17.6 Hz, 1H), 5.26 (d, *J* = 10.9 Hz, 1H), 5.18 (s, 2H), 4.48 (s, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 167.4, 167.2, 137.9, 136.3, 134.5, 134.3, 132.0, 128.6, 126.5, 123.6, 114.5, 67.3, 39.0; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2923,1849,1544, 1344,905; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{19}H_{15}NNaO_4$ 344.0893, found 344.0891.

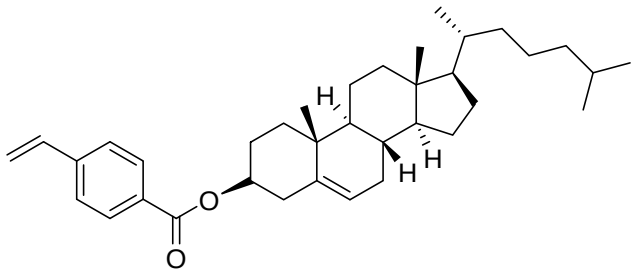


4-Vinylbenzyl 2-(11-oxo-6,11-dihydrodibenzo[b,e]oxepin-2-yl)acetate (4e): Isoxepac (20 mmol) was added to a solution of (4-vinylphenyl)methanol (20 mmol) and DMAP (20 mmol) in CH_2Cl_2 (0.2 M) at room temperature. DIC (22 mmol) was then added and stirred overnight. The residue was purified by flash column

chromatography on silica gel (petroleum ether: ethyl acetate = 5: 1). White solid. (82%, 6.3 g); m.p. 86-87 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.13 (d, *J* = 2.2 Hz, 1H), 7.89 (d, *J* = 7.6 Hz, 1H), 7.55 (dd, *J* = 7.4, 1.2 Hz, 1H), 7.51 – 7.45 (m, 1H), 7.40 (ddd, *J* = 18.7, 9.5, 5.1 Hz, 4H), 7.29 (d, *J* = 8.1 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 1H), 6.71 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.75 (d, *J* = 17.6 Hz, 1H), 5.26 (d, *J* = 10.9 Hz, 1H), 5.19 (s, 2H), 5.13 (s, 2H), 3.69 (s, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 190.8, 171.2, 160.5, 140.5, 137.7, 136.4, 135.6, 135.2, 132.8, 132.5, 129.5, 129.3, 128.5, 127.8, 127.7, 126.4, 125.2, 121.1, 114.4, 73.7, 66.5, 40.2; **FT-IR (thin film, KBr):** 2919, 1828, 1644, 1482, 1299, 912; **HRMS** (ESI) [M + Na]$^+$ Calculated for $C_{25}H_{20}NaO_4$ 407.1254, found 407.1253.



((3aS,5aR,8aR,8bS)-2,2,7,7-tetramethyltetrahydro-3aH-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-3a-yl)methyl 4-vinylbenzoate (4f): 4-vinylbenzoic acid (20 mmol) was added to a mixture of diacetonefructose (20 mmol) and DMAP (20 mmol) in CH_2Cl_2 (0.2 M) at room temperature. DIC (22 mmol) was then added and stirred overnight. The residue was purified by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 10: 1). White solid. (89%, 6.9 g); m.p. 111.-112 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.01 (d, *J* = 8.4 Hz, 2H), 7.44 (d, *J* = 8.3 Hz, 2H), 6.73 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.84 (d, *J* = 17.6 Hz, 1H), 5.37 (d, *J* = 11.0 Hz, 1H), 4.67 (d, *J* = 11.8 Hz, 1H), 4.63 (dd, *J* = 7.9, 2.6 Hz, 1H), 4.46 (d, *J* = 2.6 Hz, 1H), 4.31 (d, *J* = 11.8 Hz, 1H), 4.24 (dd, *J* = 7.9, 1.1 Hz, 1H), 3.94 (dd, *J* = 13.0, 1.8 Hz, 1H), 3.78 (d, *J* = 12.9 Hz, 1H), 1.53 (s, 3H), 1.45 (s, 3H), 1.35 (s, 3H), 1.33 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 165.6, 142.1, 135.9, 130.0, 129.0, 126.1, 116.6, 109.1, 108.8, 101.7, 265, 25.9, 25.5, 24.0; **FT-IR (thin film, KBr): ν (cm^{-1}):**2924, 1721, 1605, 1461, 1376, 1070, 860; **HRMS** (ESI) [M + Na]$^+$ Calculated for $C_{21}H_{26}NaO_7$ 413.1571, found 413.1576.



(*3R,8S,9S,10R,13R,14S,17R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 4-vinylbenzoate (4g): 4-vinylbenzoic acid (20 mmol), was added to a mixture of cholesterol (20 mmol), DMAP (20 mmol) and DCC (20 mmol) in CH_2Cl_2 (0.2 M) at room temperature and then stirred overnight. Next, the reaction mixture was filtered, concentrated on a rotary evaporator and the residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1) to give a white solid. (88%, 5.66 g). m.p. 172-173 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.99 (d, *J* = 8.3 Hz, 2H), 7.45 (d, *J* = 8.3 Hz, 2H), 6.75 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.86 (d, *J* = 17.6 Hz, 1H), 5.42 (d, *J* = 4.0 Hz, 1H), 5.37 (d, *J* = 10.9 Hz, 1H), 4.99 – 4.76 (m, 1H), 2.46 (d, *J* = 7.7 Hz, 2H), 2.09 – 1.95 (m, 3H), 1.94 – 1.66 (m, 3H), 1.65 – 1.43 (m, 8H), 1.35 (ddd, *J* = 19.9, 15.5, 8.0 Hz, 4H), 1.26 – 1.12 (m, 5H), 1.07 (s, 3H), 0.99 (dd, *J* = 10.5, 5.8 Hz, 3H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.87 (dd, *J* = 6.6, 1.6 Hz, 6H), 0.69 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 165.7, 141.7, 139.6, 136.1, 130.0, 129.8, 126.0, 122.7, 116.3, 74.5, 56.7, 56.1, 50.0, 42.3, 39.7, 39.5, 38.2, 37.0, 36.6, 36.2, 35.8, 31.9, 31.9, 28.2, 28.0, 27.9, 24.3, 23.8, 22.8, 22.5, 21.0, 19.4, 18.7, 11.8; **FT-IR (thin film, KBr): ν (cm^{-1}):**2879, 1710, 1615, 1468, 1274, 1120, 856; **HRMS** (ESI) $[M + Na]^+$ Calculated for $C_{36}H_{52}NaO_2$ 539.3860, found 539.3857.

Reference

(1) D. Mchale, J. Green, P. Mamalis, *J. Chem. Soc.* **1960**, 225.

(2) Q. Wang, W. Huang, H. Yuan, Q. Cai, L. Chen, H. Lv, X. Zhang, *J. Am. Chem. Soc*. **2014**, *136*, 16120.

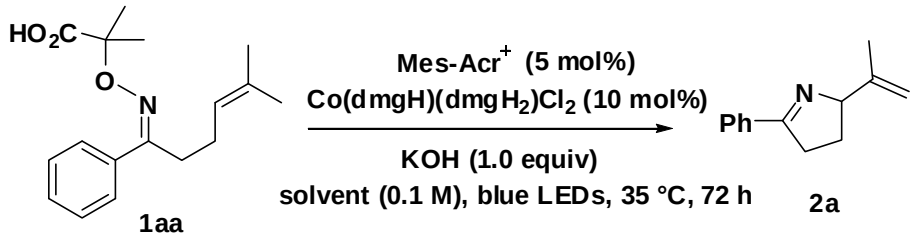
(3) J. Davies, N. S. Sheikh, D. Leonori, *Angew. Chem., Int. Ed.* **2017**, *56*, 13361.

(4) Y. Qin, V. Sukul, D. Pagakos, C. Cui, F. Jäkle, *Macromolecules*. **2005**, *38*, 8987.

4. Optimization of the reaction conditions

4.1 Optimization of aza-Heck cyclization

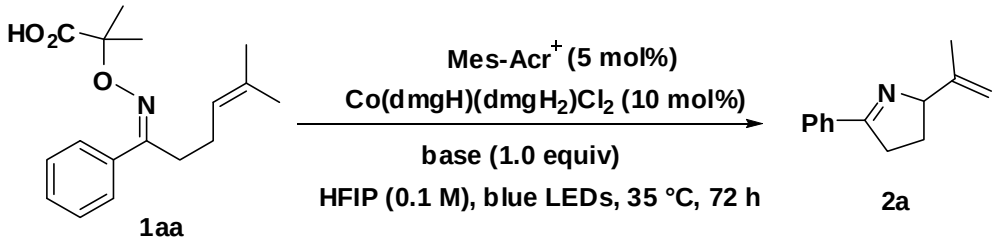
Table S1. Screening of solvents



Entry	Solvent	Base (1.0 equiv.)	Yield (%)[a]
1	**HFIP**	**KOH**	**80**
2	CF_3CH_2OH	KOH	54
3	toluene	KOH	15
4	DCM	KOH	21
5	DMSO	KOH	27
6	DMF、DCE、*tert*-Butanol、1,4-Dioxane、or THF	KOH	<10
7	CH_3NO_2	KOH	36
8	CH_3CN	KOH	31

[a]Isolated yield.

Table S2. Evaluation of bases

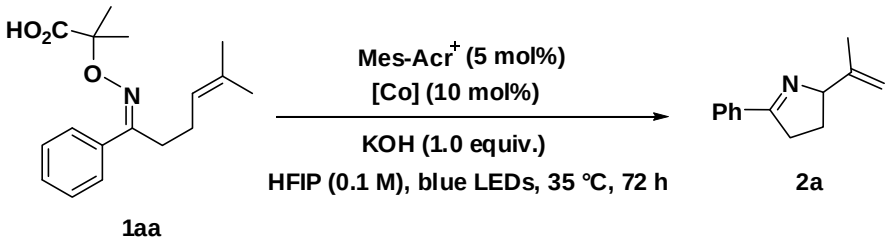


Entry	Solvent	Base (1.0 equiv.)	Yield (%)[a]
1	**HFIP**	**KOH**	**80**
2	HFIP	Cs_2CO_3	67

3	HFIP	K_2CO_3	63
4	HFIP	Na_2CO_3	32
5	HFIP	$NaHCO_3$	68
6	HFIP	K_3PO_4	48
7	HFIP	Na_3PO_4	57
8	HFIP	Li_3PO_4	19
9	HFIP	K_2HPO_4	63
10	HFIP	KH_2PO_4	62
11	HFIP	Na_2HPO_4	58
12	HFIP	NaOH	74
13	HFIP	*t*-BuOK	69
14	HFIP	*t*-BuONa	56
15	HFIP	DIPEA	67
16	HFIP	TMG	77
17	HFIP	Et_3N	68
18	HFIP	DMAP	62
19	HFIP	2,6-Lutidine	66
20	HFIP	KOH (2.0 equiv.)	78
21	HFIP	KOH (0.5 equiv.)	68

[a]Isolated yield.

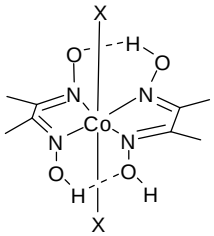
Table S3. Evaluation of cobalt catalysts



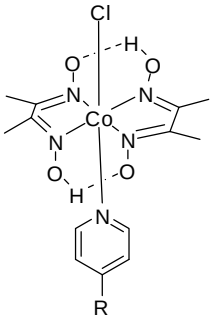
Entry	[Co]	Yield (%)[a]
1	**$Co(dmgH)(dmgH_2)Cl_2$**	**80**
2	$Co(dmgH)_2PyCl$	67
3	$Co(dmgH)(dmgH_2)(PF_6)_2$	63
4	$Co(dmgBF_2)_2 \bullet 2H_2O$	32

5	$Co(dmgH)_2(4\text{-}OMe\text{-}Py)Cl$	68
6	$Co(dmgH)_2(4\text{-}CF_3\text{-}Py)Cl$	48
7	$Co(dmgH)_2(4\text{-}CN\text{-}Py)Cl$	57
8	$Co(dmgH)_2(DMAP)Cl$	19
9	$Co(dmgH)_2(3,5\text{-di-}Me\text{-}Py)Cl$	63
10	$CoCl_2$	62
11	$CoBr_2$	58

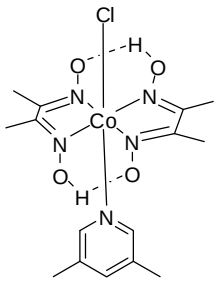
[a]Isolated yield.



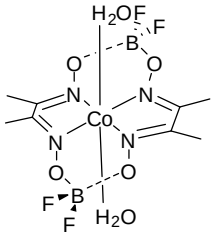
X = Cl, $Co(dmgH)(dmgH_2)Cl_2$
X = PF_6, $Co(dmgH)(dmgH_2)(PF_6)_2$



R = H, $Co(dmgH)_2PyCl$
R = OMe, $Co(dmgH)_2(4\text{-}OMe\text{-}Py)Cl$
R = CF_3, $Co(dmgH)_2(4\text{-}CF_3\text{-}Py)Cl$
R = CN, $Co(dmgH)_2(4\text{-}CN\text{-}Py)Cl$
R = NMe_2, $Co(dmgH)_2(DMAP)Cl$

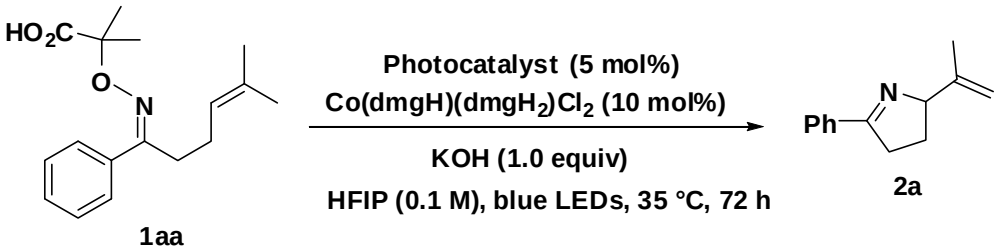


$Co(dmgH)_2(3,5\text{-di-}Me\text{-}Py)Cl$



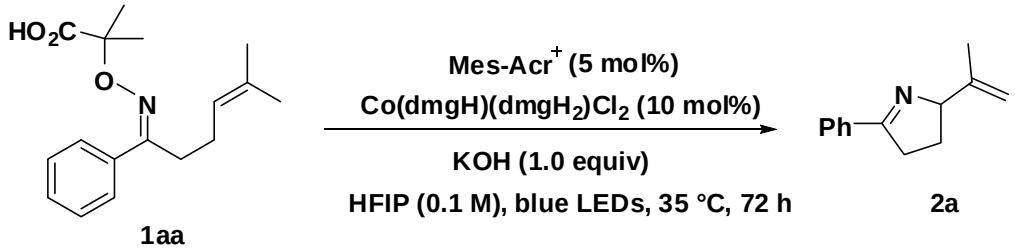
$Co(dmgBF_2)_2 \bullet 2H_2O$

Table S4. Evaluation of photocatalysts



Entry	Photocatalyst	Yield (%)
1	**Mes-Acr$^+$**	**80**
2	$[Ir(ppy)_2(dtbbpy)]PF_6$	0
3	$Ir[dF(CF_3)ppy]_2(dtbpy)]PF_6$	30
4	$Ru(bpy)_3(PF_6)_2$	No reaction (NR)
5	$Ru(bpy)_3Cl_2 \bullet 6H_2O$	NR
6	Eosin Y	NR

Table S5. Control experiments

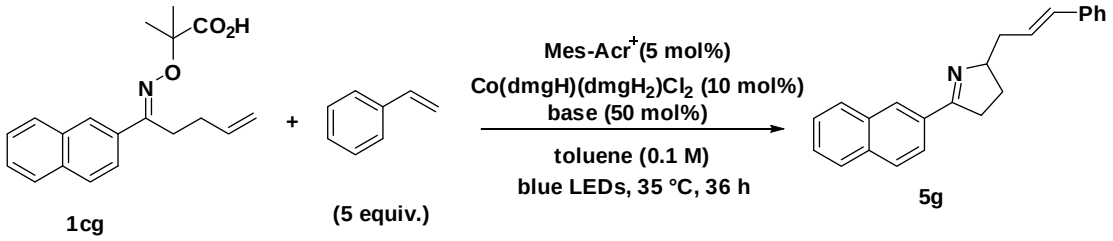


Entry	Photocatalyst	Cobalt catalyst	Base	yield(%)[a]
1	**Mes-Acr$^+$**	**Co(dmgH)(dmgH$_2$)Cl$_2$**	**KOH**	**80**
2	–	Co(dmgH)(dmgH$_2$)Cl$_2$	KOH	0
3	Mes-Acr$^+$	–	KOH	0
4	Mes-Acr$^+$	Co(dmgH)(dmgH$_2$)Cl$_2$	–	30
5[b]	Mes-Acr$^+$	Co(dmgH)(dmgH$_2$)Cl$_2$	KOH	0
6[c]	Mes-Acr$^+$	Co(dmgH)(dmgH$_2$)Cl$_2$	KOH	78
7[d]	Mes-Acr$^+$	Co(dmgH)(dmgH$_2$)Cl$_2$	KOH	70

[a]Isolated yield. [b]In the dark. [c]25 °C. [d]55 °C.

4.2 Optimization of cyclization/Heck-type coupling of α-imino-oxy acids with alkenes

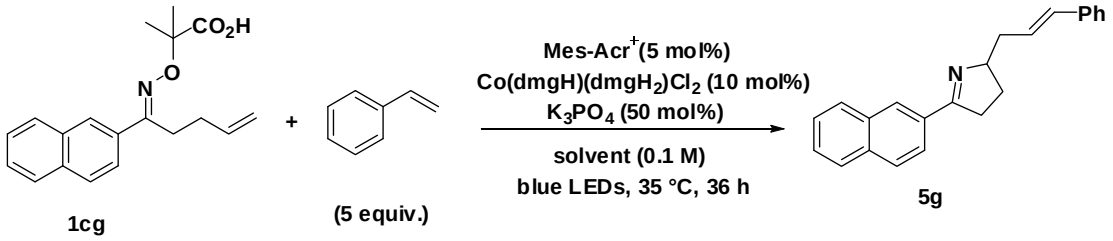
Table S6. Evaluation of bases



Entry	Solvent	Base (50 mol%)	Yield (%)[a]	*E/Z* [b]
1	**toluene**	**K$_3$PO$_4$**	**5**	**>20:1**
2	toluene	K$_3$CO$_3$ or KOH	Trace	–
3	toluene	KF, CsF or Cs$_2$CO$_3$	N.D.	–
4	toluene	Et$_3$N or TMG	N.D.	–

[a]Yields were determined by crude ^{1}H NMR spectra using dibromomethane as an internal standard.
[b]*E*/*Z* Ratios were determined by ^{1}H NMR. TMG = 1,1,3,3-tetramethylguanidine.

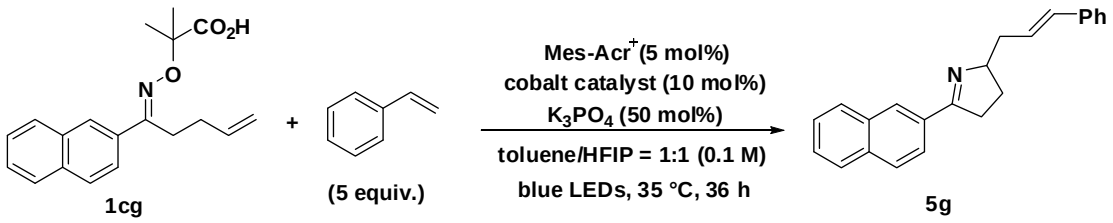
Table S7. Screening of solvents.



Entry	Base (50 mol%)	Solvent	Yield (%)[a]	*E*/*Z* [b]
1	K_3PO_4	DCE or DCM	8	>20:1
2	K_3PO_4	DMSO or DMF	trace	_
3	K_3PO_4	$PhCF_3$, PhCN, or PhH	N.D.	_
4	K_3PO_4	EA or EtOH	trace	_
5	K_3PO_4	1,4-dioxane	trace	_
6	K_3PO_4	CH_3CN	trace	_
7	K_3PO_4	THF	trace	_
8	K_3PO_4	CH_3NO_2	N.D.	_
9	K_3PO_4	HFIP	N.D.	_
10	K_3PO_4	DME	N.D.	_
11	**K_3PO_4**	**toluene/HFIP = 1:1**	**20**	**>20:1**
12	K_3PO_4	toluene/TFE = 1:1	13	>20:1
13	K_3PO_4	$PhCF_3$/HFIP = 1:1	15	>20:1
14	K_3PO_4	DME/HFIP = 1:1	trace	_
15	K_3PO_4	THF/HFIP = 1:1	trace	_
16	K_3PO_4	toluene/HFIP = 1:3	<5	_
17	K_3PO_4	toluene/HFIP = 3:1	15	>20:1
18	K_3PO_4	toluene/DCM = 1:1	17	>20:1

[a]Yields were determined by crude ^{1}H NMR spectra using dibromomethane as an internal standard.
[b]*E*/*Z* ratios were determined by ^{1}H NMR.

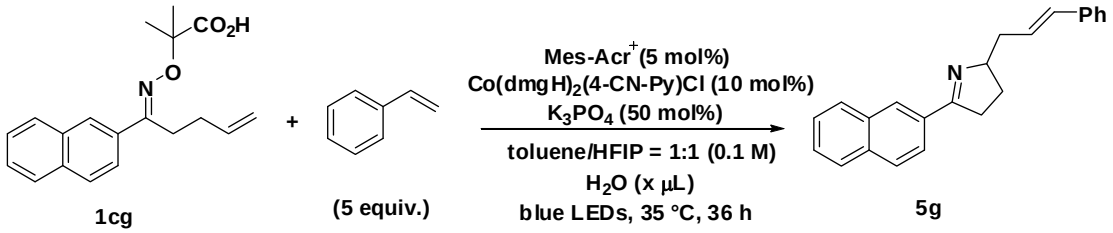
Table S8. Evaluation of cobalt catalysts



Entry	Solvent	[Co]	Yield (%)[a]	*E/Z* [b]
1	toluene/HFIP=1:1	$Co(dmgH)_2PyCl$	22	>20:1
2	toluene/HFIP=1:1	$Co(dmgH)_2$(3,5-di-Me-Py)Cl	23	10:1
3	**toluene/HFIP=1:1**	**$Co(dmgH)_2$(4-CN-Py)Cl**	**30**	>20:1
4	toluene/HFIP=1:1	$Co(dmgH)_2$(4-OMe-Py)Cl	18	>20:1
5	toluene/HFIP=1:1	$Co(dmgH)_2$(4-CF_3-Py)Cl	27	8:1

[a]Yields were determined by crude ^{1}H NMR spectra using dibromomethane as an internal standard.
[b]*E*/*Z* ratios were determined by ^{1}H NMR.

Table S9. Evaluation of the amount of water

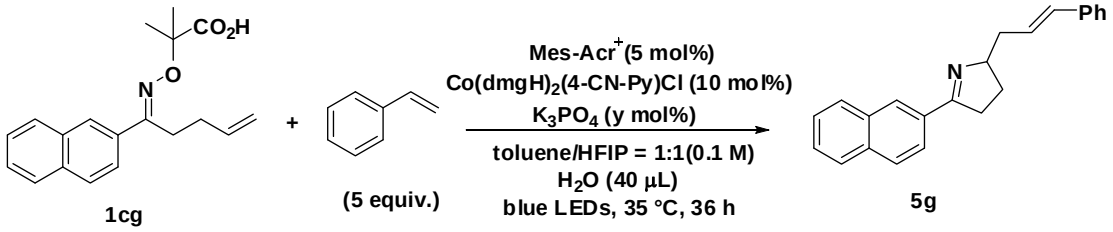


Entry	Solvent	H_2O (μL)	Yield (%)[a]	*E/Z* [b]
1	toluene/HFIP = 1:1	0	30	>20:1
2	toluene/HFIP = 1:1	2	36	>20:1
3	toluene/HFIP = 1:1	5	37	>20:1
4	toluene/HFIP = 1:1	10	42	>20:1
5	toluene/HFIP = 1:1	20	44	>20:1
6	**toluene/HFIP = 1:1**	**40**	**48**	**>20:1**
7	toluene/HFIP = 1:1	60	32	>20:1
8	toluene/HFIP = 1:1	100	29	>20:1

9	toluene/HFIP = 1:1	150	28	>20:1
10	toluene/HFIP = 1:1	200	15	>20:1

[a]Yields were determined by crude ^{1}H NMR spectra using dibromomethane as an internal standard. [b]*E*/*Z* ratios were determined by ^{1}H NMR.

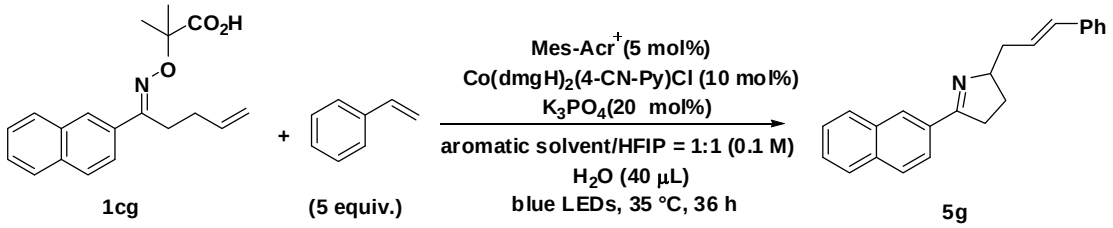
Table S10. Evaluation of the equivalent of K_3PO_4



Entry	H_2O	K_3PO_4 (mol%)	Yield (%)[a]	*E*/*Z*[b]
1	40	100	22	>20:1
2	40	50	48	>20:1
3	**40**	**20**	**52**	**>20:1**
4	40	10	23	>20:1

[a]Isolated yield. [b]*E*/*Z* ratio determined by ^{1}H NMR.

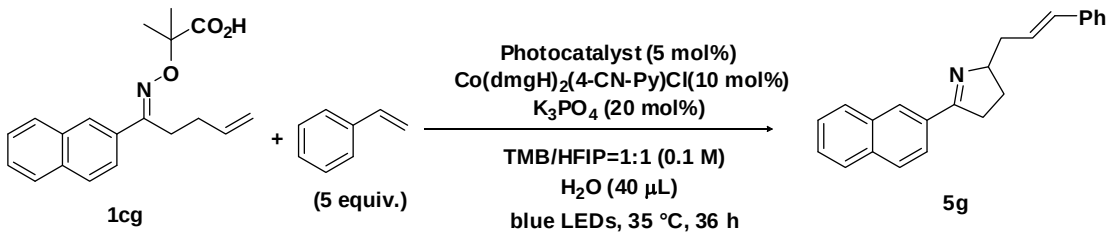
Table S11. Evaluation of aromatic solvents



Entry	Solvent	Yield (%)[a]	*E*/*Z*[b]
1	Toluene/HFIP = 1:1	52	>20:1
2	Pxylene/HFIP = 1:1	58	>20:1
3	**TMB/HFIP = 1:1**	**65**	**>20:1**
4	PhCl/HFIP = 1:1	<10	–
5	$PhCF_3$/HFIP = 1:1	25	>20:1

[a]Isolated yield. [b]*E*/*Z* ratio determined by ^{1}H NMR.

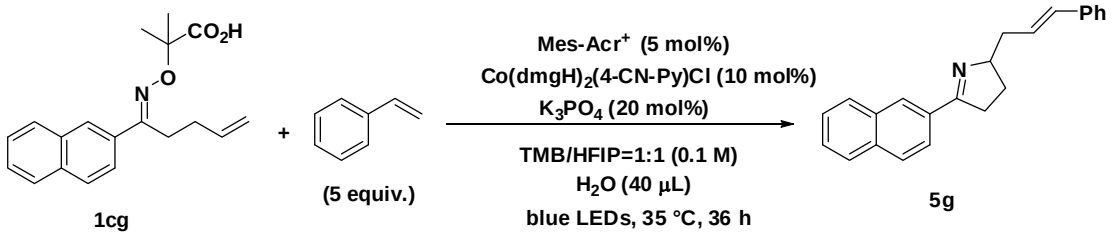
Table S12. Evaluation of photocatalysts.



Entry	Photocatalyst	yield(%)[a]	E/Z[b]
1	**Mes-Acr$^+$**	**65**	**>20:1**
2	$[Ir(ppy)_2(dtbbpy)]PF_6$	28	>20:1
3	$Ir[dF(CF_3)ppy]_2(dtbpy)]PF_6$	18	>20:1
4	$Ru(bpy)_3(PF_6)_2$	0	–
5	$Ru(bpy)_3Cl_2•6H_2O$	0	–
6	Eosin Y	0	–

[a]Isolated yield. [b]*E*/*Z* ratio determined by ^{1}H NMR.

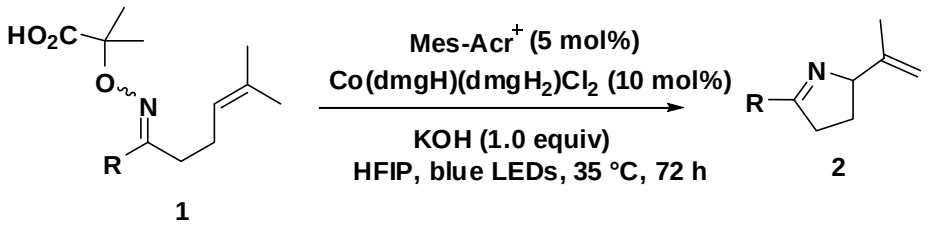
Table S13. Control experiments



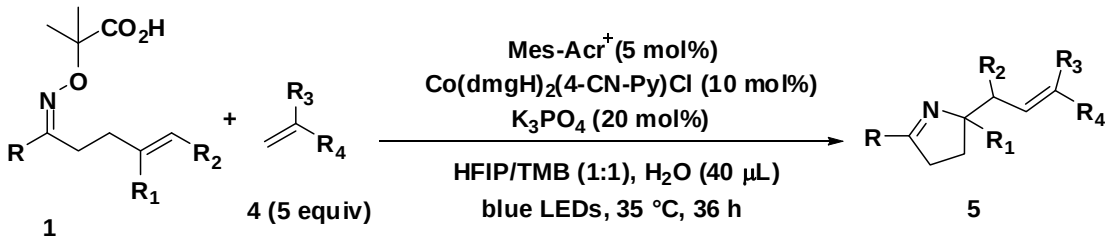
Entry	Photocatalyst	Cobalt catalyst	Base	Yield (%)[a]	*E/Z*[b]
1	**Mes-Acr+**	**$Co(dmgH)_2(4\text{-}CN\text{-}Py)Cl$**	**K_3PO_4**	**65**	**>20:1**
2	–	$Co(dmgH)_2(4\text{-}CN\text{-}Py)Cl$	K_3PO_4	0	–
3	Mes-Acr+	–	K_3PO_4	0	–
4	Mes-Acr+	$Co(dmgH)_2(4\text{-}CN\text{-}Py)Cl$	–	15	>20:1
5[c]	Mes-Acr+	$Co(dmgH)_2(4\text{-}CN\text{-}Py)Cl$	K_3PO_4	57	>20:1
6[d]	Mes-Acr+	$Co(dmgH)_2(4\text{-}CN\text{-}Py)Cl$	K_3PO_4	44	>20:1

[a]Isolated yield. [b]*E*/*Z* ratio determined by ^{1}H NMR. [c]25 °C. [d]55 °C.

5. General procedures for aza-Heck cyclization

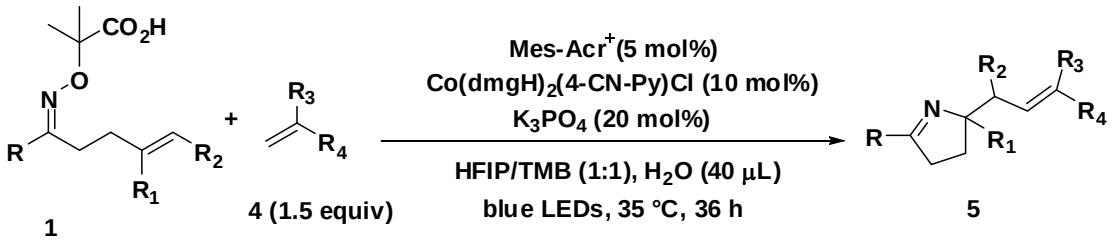


General procedure A: To a 10 mL oven-dried round-bottom Schlenk bottle equipped with a magnetic stir bar, the corresponding oxime acid (0.2 mmol, 1.0 equiv.), Mes-$Acr^{+}ClO_4^{-}$ (4.2 mg, 0.01 mmol, 5 mol%), $Co(dmgH)(dmgH_2)Cl_2$ (7.2 mg, 0.02 mmol, 10 mol%), and potassium hydroxide (11.2 mg, 0.2 mmol, 1.0 equiv.) was added. Dry hexafluoroisopropanol (2 mL) were then added under argon atmosphere. The resulting mixture was sealed and then subjected to freeze-pump-thaw for three times. After that, the reaction was placed under a 22W blue LED and irradiated for 72 hours. The temperature was maintained at 35 ℃ when the LED light was on. After the reaction was finished (monitored by TLC), the solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel.



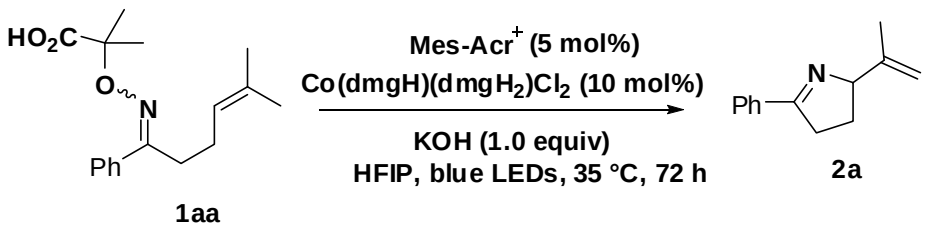
General procedure B: To a 10 mL oven-dried round-bottom Schlenk bottle equipped with a magnetic stir bar, the corresponding oxime acid (0.2 mmol, 1.0 equiv.), Mes-$Acr^{+}ClO_4^{-}$ (4.2 mg, 0.01 mmol, 5 mol%), $Co(dmgH)_2(4\text{-}CN\text{-}Py)Cl$ (8.8 mg, 0.02 mmol, 10 mol%) and potassium phosphate (8.4 mg, 0.04 mmol, 20 mol%) were added. The corresponding alkene (1 mmol, 5 equiv.), H_2O (40 μL) and dry mesitylene/hexafluoroisopropanol = 1:1 (2 mL) were then added under argon atmosphere. The resulting mixture was sealed and then subjected to freeze-pump-thaw for three times. After that, the reaction was placed under a 22W blue LED and irradiated for 36 hours. The temperature was maintained at 35 ℃ when the LED light

was on. After the reaction was finished (monitored by TLC), the solvent was then removed under reduced pressure and the residue was purified by flash column chromatography on silica gel.



General procedure C: To a 10 mL oven-dried round-bottom Schlenk bottle equipped with a magnetic stir bar, the corresponding oxime acid (0.2 mmol, 1.0 equiv.), Mes-$Acr^+ClO_4^-$ (4.2 mg, 0.01 mmol, 5 mol%), $Co(dmgH)_2(4\text{-}CN\text{-}Py)Cl$ (8.8 mg, 0.02 mmol, 10 mol%), alkene (0.3 mmol, 1.5 equiv., if solid) and potassium phosphate (8.4 mg, 0.04 mmol, 20 mol%) were added. The corresponding alkene (0.3 mmol, 1.5 equiv., if liquid), H_2O (40 μL) and dry mesitylene/hexafluoroisopropanol = 1:1 (2 mL) were then added under argon atmosphere. The resulting mixture was sealed and then subjected to freeze-pump-thaw for three times. After that, the reaction was placed under a 22W blue LED and irradiated for 36 hours. The temperature was maintained at 35 ℃ when the LED light was on. After the reaction was finished (monitored by TLC), the solvent was then removed under reduced pressure and the residue was purified by flash column chromatography on silica gel.

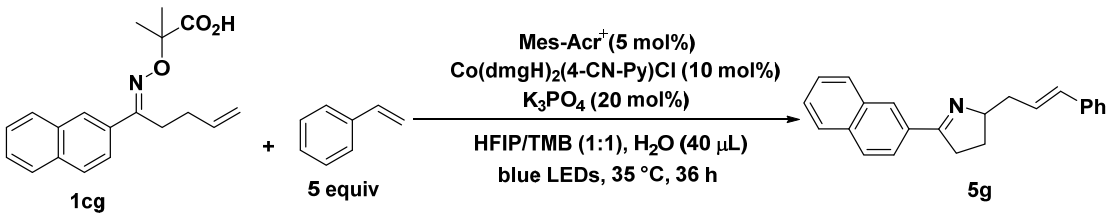
Gram-scale synthesis of compound 2a



To a 100 mL oven-dried round-bottom Schlenk bottle equipped with a magnetic stir bar, the corresponding oxime acid (4 mmol, 1.16 g, 1.0 equiv.), Mes-$Acr^+ClO_4^-$ (0.2 mmol, 84 mg, 5 mol%), $Co(dmgH)(dmgH_2)Cl_2$ (0.4 mmol, 144 mg, 10 mol%), and potassium hydroxide (224 mg, 4 mmol, 1.0 equiv.) was added. Dry hexafluoroisopropanol (40 mL) were then added under argon atmosphere. The

resulting mixture was sealed and then subjected to freeze-pump-thaw for three times. After that, the reaction was placed under a 22W blue LED and irradiated for 72 hours. The temperature was maintained at 35 ℃ when the LED light was on. After the reaction was finished (monitored by TLC), the solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give the title compound in 75% yield (555 mg);

Gram-scale synthesis of compound 5g



To a 100 mL oven-dried round-bottom Schlenk bottle equipped with a magnetic stir bar, the corresponding oxime acid (4 mmol, 1.24 g, 1.0 equiv), Mes-Acr$^+$ClO$_4^-$ (0.2 mmol, 84 mg, 5 mol%), Co(dmgH)$_2$(4-CN-Py)Cl (0.4 mmol, 176 mg, 10 mol%) and potassium phosphate (0.8 mmol, 168 mg, 20 mol%) were added. Styrene (20 mmol, 2.3 mL, 5 equiv.), H_2O (0.8 mL) and dry mesitylene/hexafluoroisopropanol = 1:1 (40 mL) were then added under argon atmosphere. The resulting mixture was sealed and then subjected to freeze-pump-thaw for three times. After that, the reaction was placed under a 22W blue LED and irradiated for 36 hours. The temperature was maintained at 35 ℃ when the LED light was on. After the reaction was finished (monitored by TLC), the solvent was then removed under reduced pressure and the residue was purified by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give the title compound in 62% yield (773 mg);

6. Mechanistic Investigation

6.1. Stern-Volmer fluorescence quenching experiments

In a typical experiment, a solution of 9-mesityl-10-methylacridinium perchlorate (Mes-Acr$^+$) in PhMe/HFIP = 1:1 (5×10^{-5} M) was added the appropriate amount of

quencher in a quartz cuvette. Then the emission of samples was collected. The emission intensity at 510 nm was collected with excited wavelength of 450 nm.

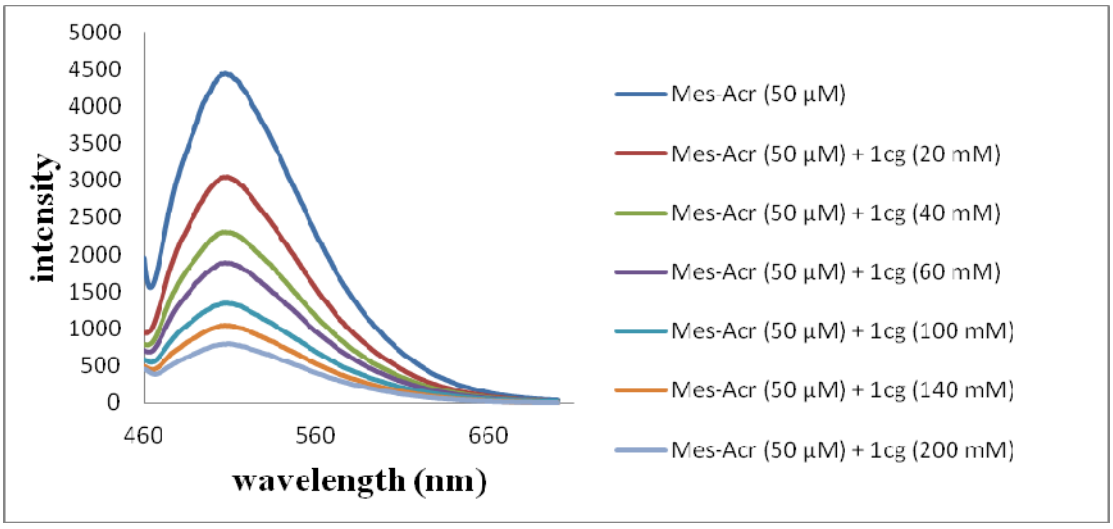


Figure S2. Emission quenching by oxime acid **1cg**

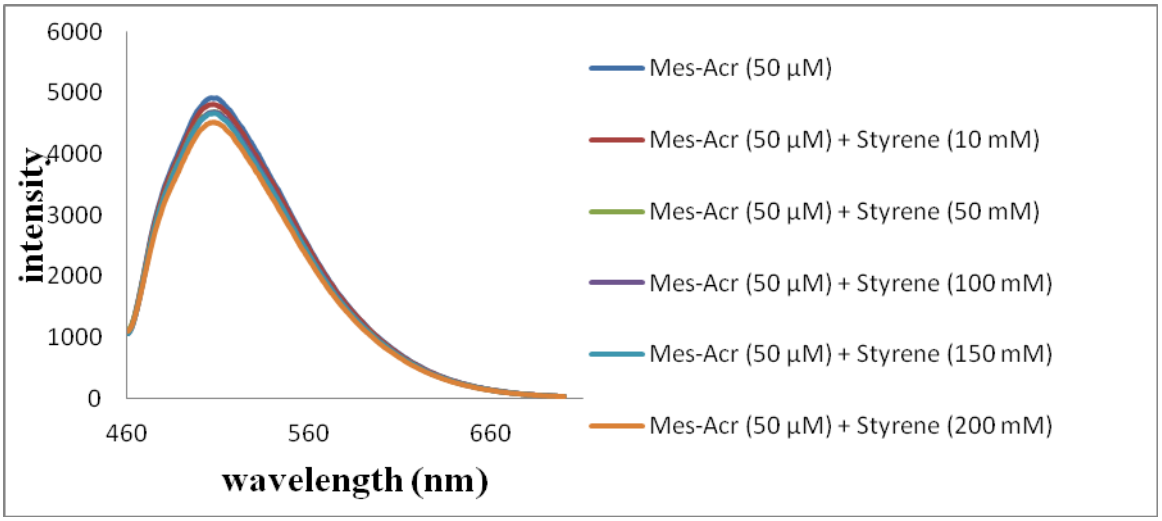


Figure S3. Emission quenching by styrene

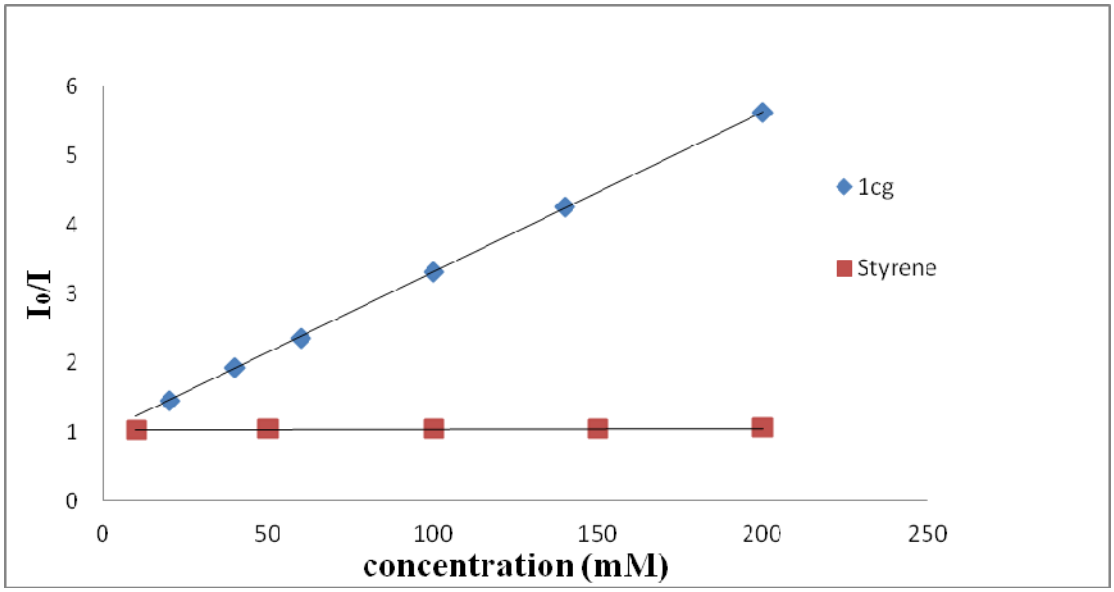
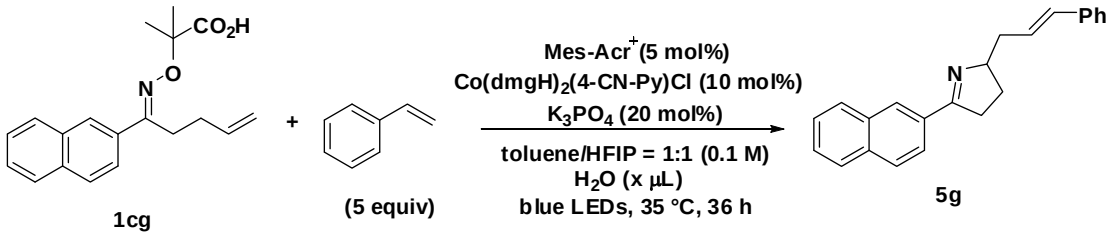


Figure S4. Combined Stern−Volmer emission quenching data.

6.2. Study of the effect of water amount on yield



To a 10 mL oven-dried round-bottom Schlenk bottle equipped with a magnetic stir bar, (*E*)-2-methyl-2-(((1-(naphthalen-2-yl)pent-4-en-1-ylidene)amino)oxy)propanoic acid (**1cg**, 62 mg, 0.2 mmol, 1.0 equiv.), Mes-Acr$^+$ClO$_4^-$ (4.2 mg, 0.01 mmol, 5 mol%), Co(dmgH)$_2$(4-CN-Py)Cl (8.8 mg, 0.02 mmol, 10 mol%), and potassium phosphate (8.4 mg, 0.04 mmol, 20 mol%) were added. Styrene (114 μL, 1 mmol, 5 equiv.), H_2O (0-200 μL), dry toluene/hexafluoroisopropanol = 1:1 (2 mL) were then added under argon atmosphere. The resulting mixture was sealed and then subjected to freeze-pump-thaw for three times. After that, the reaction was placed under a 22W blue LED and irradiated for 36 hours. The temperature was maintained at 35℃ when the LED light was on. Yields were determined by crude ^{1}H NMR spectra using dibromomethane as an internal standard.

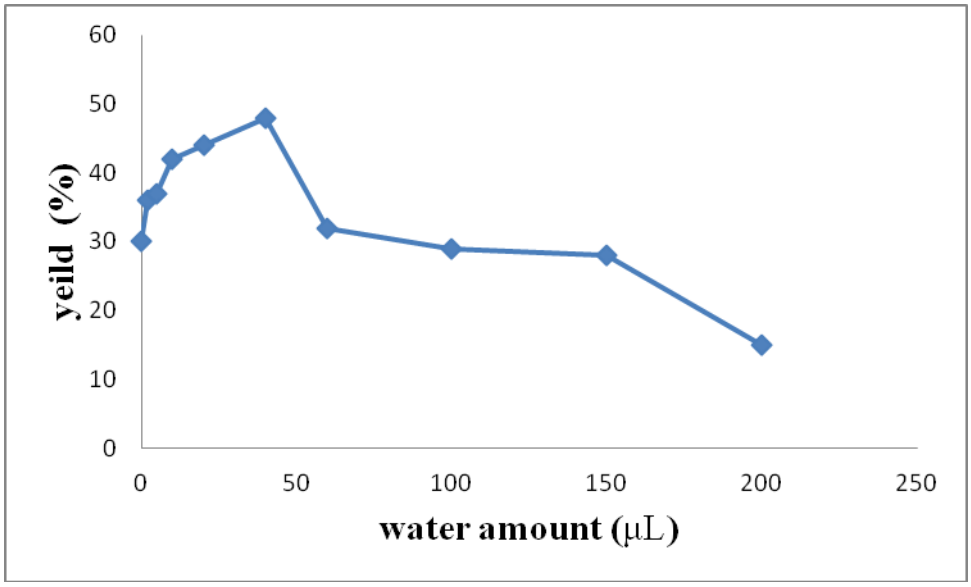


Figure S5. The effect of water amount

6.3 Determination of gas produced in reaction with GC

Qualitative detection of hydrogen was obtained by Agilent 7890b gas chromatography (GC). Following general procedure B, (*E*)-2-methyl-2-(((1-(naphthalen-2-yl)pent-4-en-1-ylidene)amino)oxy)propanoic acid and styrene were added in a sealed tube. After the reaction finished, the sealed tube was cooled in liquid nitrogen. Gas produced in the sealed tube was injected into gas chromatography with syringe. By comparing with the retention time of standard gas, it is determined that the produced gas is hydrogen.

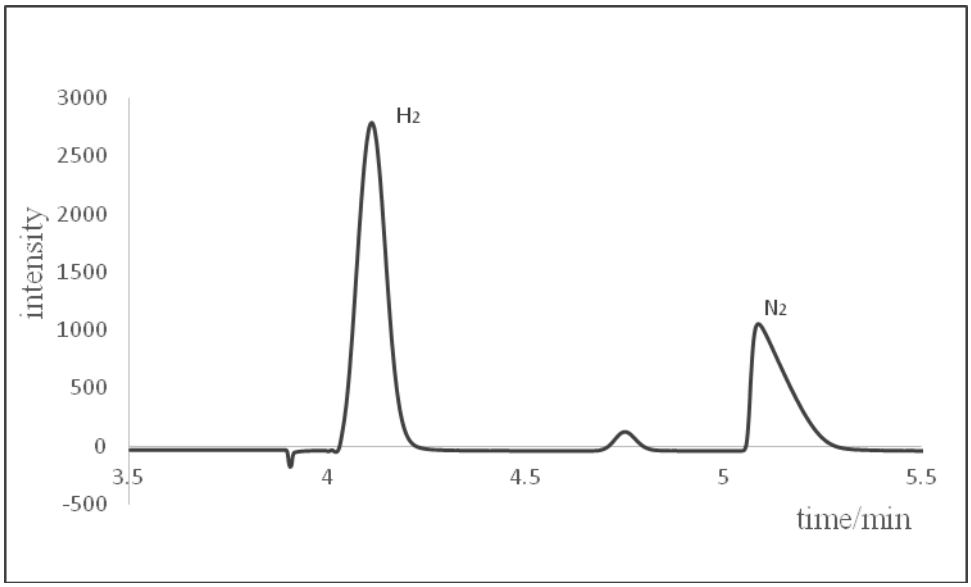
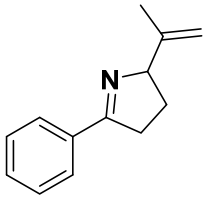
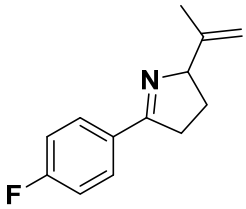


Figure S6. Hydrogen gas extrusion

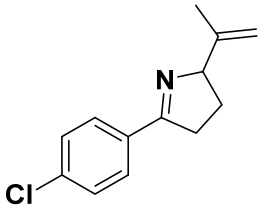
7. Characterization of the products



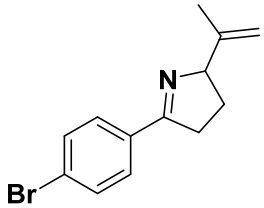
5-Phenyl-2-(prop-1-en-2-yl)-3,4-dihydro-*2H*-pyrrole (2a): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 80% yield (30 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.98 – 7.84 (m, 2H), 7.50 – 7.37 (m, 3H), 4.95 (s, 1H), 4.84 (s, 1H), 4.73 (t, *J* = 7.4 Hz, 1H), 3.04 (dddd, *J* = 15.1, 9.9, 5.1, 2.0 Hz, 1H), 3.00 – 2.89 (m, 1H), 2.35 – 2.21 (m, 1H), 1.85 – 1.78 (m, 1H), 1.77 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 173.2, 146.7, 134.4, 130.5, 128.4, 127.8, 110.3, 77.5, 35.2, 28.2, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1717, 1541, 1335, 1066, 894, 766; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{13}H_{16}N$ 186.1283, found 186.1289.



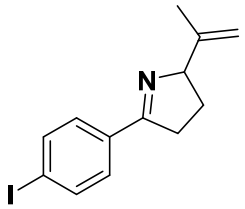
5-(4-Fluorophenyl)-2-(prop-1-en-2-yl)-3,4-dihydro-*2H*-pyrrole (2b): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 55% yield (22 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.04 – 7.76 (m, 2H), 7.19 – 6.93 (m, 2H), 4.97 – 4.90 (m, 1H), 4.86 – 4.78 (m, 1H), 4.70 (t, *J* = 7.5 Hz, 1H), 3.01 (dddd, *J* = 17.1, 9.9, 5.2, 2.1 Hz, 1H), 2.91 (dddd, *J* = 16.9, 9.4, 7.4, 1.8 Hz, 1H), 2.36 – 2.13 (m, 1H), 1.88 – 1.77 (m, 1H), 1.76 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 171.9, 164.2 (d, *J* = 250.7 Hz), 146.7, 130.8, 129.9 (d, *J* = 8.6 Hz), 115.4 (d, *J* = 21.8 Hz), 110.3, 77.6, 35.2, 28.3, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2933, 1713, 1602, 1558, 1227, 896; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{13}H_{15}FN$ 204.1189, found 204.1182.



5-(4-Chlorophenyl)-2-(prop-1-en-2-yl)-3,4-dihydro-*2H*-pyrrole (2c): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 69% yield (30 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.92 – 7.76 (m, 2H), 7.49 – 7.32 (m, 2H), 5.00 – 4.88 (m, 1H), 4.87 – 4.80 (m, 1H), 4.71 (t, *J* = 7.5 Hz, 1H), 3.01 (dddd, *J* = 17.1, 9.9, 5.2, 2.2 Hz, 1H), 2.90 (dddd, *J* = 16.9, 9.4, 7.4, 1.9 Hz, 1H), 2.38 – 2.17 (m, 1H), 1.88 – 1.77 (m, 1H), 1.76 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 172.0, 146.7, 136.5, 133.0, 129.1, 128.6, 110.4, 77.7, 35.2, 28.3, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2936, 1614 , 1558, 1448, 893; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{13}H_{15}{}^{35}ClN$ 220.0893, found 220.0888.

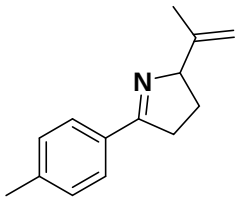


5-(4-Bromophenyl)-2-(prop-1-en-2-yl)-3,4-dihydro-*2H*-pyrrole (2d): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 66% yield (35 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 8.5 Hz, 2H), 7.53 (d, *J* = 8.5 Hz, 2H), 4.92 (s, 1H), 4.83 (s, 1H), 4.69 (t, *J* = 7.5 Hz, 1H), 3.00 (dddd, *J* = 15.1, 9.8, 5.1, 2.0 Hz, 1H), 2.94 – 2.82 (m, 1H), 2.43 – 2.16 (m, 1H), 1.87 – 1.77 (m, 1H), 1.76 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.1, 146.6, 133.3, 131.6, 129.3, 124.9, 110.4, 77.7, 35.1, 28.3, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2940, 1615 , 1497, 1334, 894; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{13}H_{15}{}^{79}BrN$ 264.0388, found 264.0380.

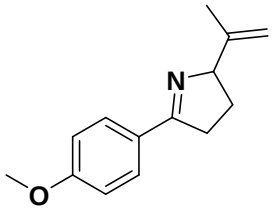


5-(4-Iodophenyl)-2-(prop-1-en-2-yl)-3,4-dihydro-*2H*-pyrrole (2e): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a light yellow solid; 53% yield (33 mg); m.p. 78-79 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.60 (d, *J* = 8.3 Hz, 2H), 4.92 (s, 1H), 4.83 (s, 1H), 4.69 (t, *J* = 7.4 Hz, 1H), 3.03 – 2.95 (m, 1H), 2.88 (dt, *J* = 16.9, 8.5 Hz, 1H), 2.31 – 2.23 (m, 1H), 1.84 – 1.77 (m, 1H), 1.75 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ

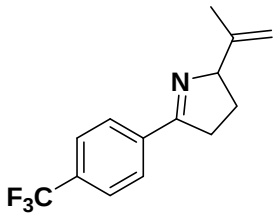
172.3, 146.6, 137.6, 134.0, 129.4, 110.4, 97.2, 77.7, 35.1, 28.3, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2922, 1699 ,1489, 1286, 890; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{13}H_{15}IN$ 312.0244, found 312.0241.



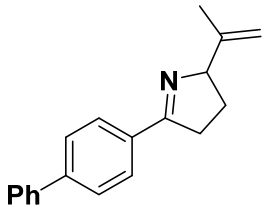
2-(Prop-1-en-2-yl)-5-(*p*-tolyl)-3,4-dihydro-*2H*-pyrrole (2f): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 84% yield (34 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.79 (d, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 4.95 – 4.92 (m, 1H), 4.84 – 4.81 (m, 1H), 4.77 – 4.64 (m, 1H), 3.01 (dddd, *J* = 17.1, 9.9, 5.3, 2.1 Hz, 1H), 2.97 – 2.86 (m, 1H), 2.39 (s, 3H), 2.33 – 2.14 (m, 1H), 1.86 – 1.78 (m, 1H), 1.76 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 173.0, 147.0, 140.7, 131.8, 129.1, 127.8, 110.2, 77.5, 35.1, 28.2, 21.4, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2922, 1699 , 1489, 1286, 890; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{14}H_{18}N$ 200.1434, found 200.1431.



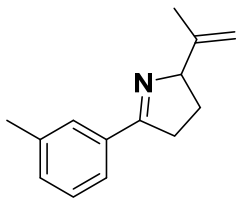
5-(4-Methoxyphenyl)-2-(prop-1-en-2-yl)-3,4-dihydro-*2H*-pyrrole (2g): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 74% yield (32 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.84 (d, *J* = 8.6 Hz, 2H), 6.92 (d, *J* = 8.6 Hz, 2H), 4.93 (d, *J* = 0.7 Hz, 1H), 4.82 (s, 1H), 4.69 (t, *J* = 7.4 Hz, 1H), 3.84 (s, 3H), 3.07 – 2.95 (m, 1H), 2.95 – 2.86 (m, 1H), 2.34 – 2.19 (m, 1H), 1.83 – 1.76 (m, 1H), 1.75 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 172.6, 161.6, 146.9, 129.5, 127.2, 113.8, 110.3, 77.3, 55.4, 35.1, 28.3, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2924, 1614 ,1335, 1066, 894; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{14}H_{18}NO$ 216.1383, found 216.1378.



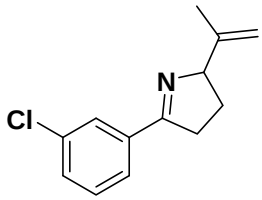
2-(Prop-1-en-2-yl)-5-(4-(trifluoromethyl)phenyl)-3,4-dihydro-*2H*-pyrrole (2h): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 55% yield (28 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.99 (d, *J* = 8.2 Hz, 2H), 7.67 (d, *J* = 8.3 Hz, 2H), 4.94 (d, *J* = 0.7 Hz, 1H), 4.85 (s, 1H), 4.75 (t, *J* = 7.5 Hz, 1H), 3.08 – 3.02 (m, 1H), 2.99 – 2.88 (m, 1H), 2.39 – 2.23 (m, 1H), 1.90 – 1.79 (m, 1H), 1.77 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 171.9, 146.4, 137.6, 132.3 (q, *J* = 32.4 Hz), 128.0, 125.3 (q, *J* = 3.7 Hz), 123.9 (q, *J* = 272.3 Hz), 110.5, 77.8, 35.2, 28.2, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2924, 1614 , 1335, 1066, 894; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{14}H_{15}F_3N$ 254.1157, found 254.1151.



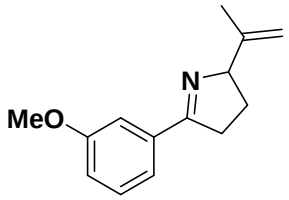
5-([1,1'-Biphenyl]-4-yl)-2-(prop-1-en-2-yl)-3,4-dihydro-*2H*-pyrrole (2i): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 64% yield (33mg), **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.65 (t, *J* = 7.9 Hz, 4H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.38 (t, *J* = 7.3 Hz, 1H), 4.98 (s, 1H), 4.86 (s, 1H), 4.76 (t, *J* = 7.4 Hz, 1H), 3.15 – 3.01 (m, 1H), 2.97 (dddd, *J* = 16.9, 9.3, 7.3, 1.7 Hz, 1H), 2.40 – 2.21 (m, 1H), 1.91 – 1.81 (m, 1H), 1.79 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.7, 146.8, 143.1, 140.4, 133.4, 128.8, 128.3, 127.7, 127.1, 127.0, 110.3, 77.7, 35.2, 28.3, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1616 ,1435, 1078, 893; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{19}H_{20}N$ 262.1590, found 262.1592.



2-(Prop-1-en-2-yl)-5-(m-tolyl)-3,4-dihydro-*2H*-pyrrole (2j): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 75% yield (30 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.77 (s, 1H), 7.64 (d, *J* = 7.5 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.25 (d, *J* = 7.5 Hz, 1H), 5.07 – 4.89 (m, 1H), 4.89 – 4.79 (m, 1H), 4.72 (t, *J* = 7.4 Hz, 1H), 3.03 (dddd, *J* = 17.2, 9.9, 5.3, 2.1 Hz, 1H), 2.92 (dddd, *J* = 16.9, 9.3, 7.3, 1.8 Hz, 1H), 2.39 (s, 3H), 2.34 – 2.19 (m, 1H), 1.88 – 1.78 (m, 1H), 1.77 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 173.2, 146.8, 138.1, 134.4, 131.2, 128.2, 125.0, 110.3, 77.5, 35.2, 28.2, 21.3, 19.4; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2969, 1698, 1457, 1078, 786; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{14}H_{18}N$ 200.1434, found 200.1430.

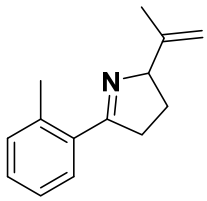


5-(3-Chlorophenyl)-2-(prop-1-en-2-yl)-3,4-dihydro-*2H*-pyrrole (2k): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 58% yield (26 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.90 (t, *J* = 1.7 Hz, 1H), 7.74 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.43 – 7.38 (m, 1H), 7.34 (t, *J* = 7.8 Hz, 1H), 4.94 (s, 1H), 4.85 (s, 1H), 4.72 (t, *J* = 7.5 Hz, 1H), 3.01 (dddd, *J* = 17.1, 9.9, 5.1, 2.2 Hz, 1H), 2.90 (dddd, *J* = 17.0, 9.4, 7.4, 1.9 Hz, 1H), 2.35 – 2.23 (m, 1H), 1.87 – 1.79 (m, 1H), 1.77 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 171.9, 146.5, 136.2, 134.5, 130.4, 129.6, 127.8, 125.9, 110.4, 77.7, 35.2, 28.2, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2963, 1684 , 1507, 1077, 897; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{13}H_{15}{}^{35}ClN$ 220.0888, found 220.0889.

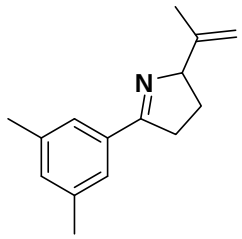


5-(3-Methoxyphenyl)-2-(prop-1-en-2-yl)-3,4-dihydro-*2H*-pyrrole (2l): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 74% yield (32 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.51 (dd, *J* = 2.3, 1.5 Hz, 1H), 7.40 (dd, *J* = 6.5, 1.1 Hz, 1H), 7.32 (t, *J* = 7.9 Hz, 1H), 6.99 (ddd, *J* = 8.1, 2.6, 0.8 Hz, 1H), 4.96 – 4.92 (m, 1H), 4.85 – 4.82 (m, 1H), 4.72 (t, *J* = 7.3 Hz,

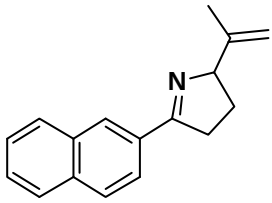
1H), 3.85 (s, 3H), 3.03 (dddd, *J* = 17.2, 9.8, 5.3, 2.1 Hz, 1H), 2.97 – 2.87 (m, 1H), 2.34 – 2.23 (m, 1H), 1.86 – 1.78 (m, 1H), 1.77 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 173.2, 159.8, 146.9, 136.0, 129.5, 120.7, 117.1, 112.2, 110.4, 77.7, 55.6, 35.4, 28.4, 19.7; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1580, 1457, 1046, 895; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{14}H_{18}ON$ 216.1388, found 216.1385.



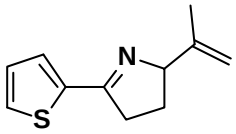
2-(Prop-1-en-2-yl)-5-(o-tolyl)-3,4-dihydro-*2H*-pyrrole (2m): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 52% yield (21 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.47 – 7.41 (m, 1H), 7.31 – 7.18 (m, 3H), 5.02 – 4.94 (m, 1H), 4.87 – 4.81 (m, 1H), 4.71 (t, *J* = 7.7 Hz, 1H), 3.01 – 2.90 (m, 2H), 2.55 (s, 3H), 2.31 – 2.20 (m, 1H), 1.80 (s, 3H), 1.78 – 1.68 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 175.0, 147.0, 137.3, 135.0, 131.3, 129.0, 128.8, 125.5, 110.0, 78.2, 38.6, 28.5, 21.7, 19.8; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2933, 1613, 1455, 1034, 894; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{14}H_{18}N$ 200.1439, found 200.1431.



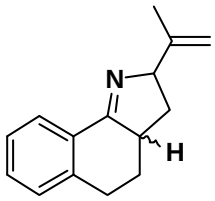
5-(3,5-Dimethylphenyl)-2-(prop-1-en-2-yl)-3,4-dihydro-*2H*-pyrrole (2n): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 87% yield (37 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.51 (s, 2H), 7.08 (s, 1H), 4.96 – 4.92 (m, 1H), 4.85 – 4.81 (m, 1H), 4.70 (t, *J* = 7.4 Hz, 1H), 3.02 (dddd, *J* = 17.2, 9.9, 5.3, 2.1 Hz, 1H), 2.90 (dddd, *J* = 16.9, 9.6, 7.2, 1.8 Hz, 1H), 2.35 (s, 6H), 2.30 – 2.19 (m, 1H), 1.81 – 1.77 (m, 1H), 1.76 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 173.4, 146.9, 137.9, 134.4, 132.1, 125.6, 110.3, 77.6, 35.3, 28.2, 21.2, 19.4; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2920, 1615, 1419, 1055, 893; **HRMS** (CI) $[M+H]^+$ Calculated for $C_{15}H_{20}N$ 214.1596, found 214.1594.



5-(Naphthalen-2-yl)-2-(prop-1-en-2-yl)-3,4-dihydro-*2H*-pyrrole (2o): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 93% yield (44 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.21 (s, 1H), 8.17 (dd, *J* = 8.6, 1.4 Hz, 1H), 7.93 – 7.88 (m, 1H), 7.86 (t, *J* = 8.5 Hz, 2H), 7.56 – 7.48 (m, 2H), 5.01 (d, *J* = 0.8 Hz, 1H), 4.88 (s, 1H), 4.78 (t, *J* = 7.4 Hz, 1H), 3.22 – 3.10 (m, 1H), 3.11 – 3.00 (m, 1H), 2.39 – 2.27 (m, 1H), 1.93 – 1.82 (m, 1H), 1.81 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 173.1, 146.8, 134.4, 133.0, 131.9, 128.7, 128.3, 128.1, 127.7, 127.1, 126.4, 124.7, 110.4, 77.7, 35.2, 28.3, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2966, 1611, 1458, 1125, 896; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{17}H_{18}N$ 236.1439, found 236.1433.

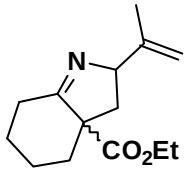


2-(Prop-1-en-2-yl)-5-(thiophen-2-yl)-3,4-dihydro-*2H*-pyrrole (**2p**): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 95% yield (36 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.42 (d, *J* = 5.0 Hz, 1H), 7.35 (d, *J* = 3.5 Hz, 1H), 7.11 – 7.02 (m, 1H), 4.92 (d, *J* = 0.8 Hz, 1H), 4.82 (s, 1H), 4.69 (t, *J* = 7.3 Hz, 1H), 3.07 – 2.97 (m, 1H), 2.97 – 2.86 (m, 1H), 2.31 – 2.22 (m, 1H), 1.87 – 1.78 (m, 1H), 1.75 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 167.6, 146.6, 139.5, 129.2, 127.4, 110.5, 77.4, 35.8, 28.6, 19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2966, 1611, 1458, 1125, 896; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{11}H_{14}NS$ 192.0841, found 192.0837.

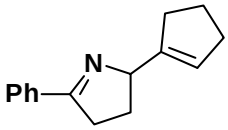


2-(Prop-1-en-2-yl)-3,3a,4,5-tetrahydro-*2H*-benzo[g]indole (2q): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 93% yield (39 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.18 – 8.12

(m, 1H), 7.36 – 7.29 (m, 1H), 7.26 – 7.21 (m, 1H), 7.21 – 7.15 (m, 1H), 5.11 – 5.03 (m, 0.6H)/4.76 – 4.74 (m, 0.4H), 4.87 (s, 0.6H)/4.74 – 4.72 (m, 0.4H), 4.81 (d, *J* = 9.3 Hz, 0.4H)/4.45 – 4.35 (m, 0.6H), 3.10 – 2.78 (m, 3H), 2.43 (ddd, *J* = 12.2, 7.7, 6.6 Hz, 0.6H)/2.16 (ddd, *J* = 12.6, 8.0, 1.3 Hz, 0.4H), 2.28 – 2.20 (m, 1H), 1.86 – 1.79 (m, 0.4H)/1.38 (ddd, *J* = 18.8, 11.3, 9.1 Hz, 0.6H), 1.78 (s, 1.8H)/1.75 (s, 1.2H), 1.73 – 1.55 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 174.3/173.5, 147.1/145.7, 141.1/141.0, 130.8/130.7, 130.2/130.1, 128.82/128.79, 126.5/126.4, 126.10/126.08, 110.6/110.2, 75.9/75.4, 47.8/45.9, 36.8/35.1, 30.1/29.6, 29.98/29.96, 20.3/19.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2929, 1618, 1507, 1473, 895; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{15}H_{18}N$ 224.1434, found 224.1430.

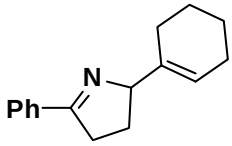


Ethyl 2-(prop-1-en-2-yl)-2,3,4,5,6,7-hexahydro-*3aH*-indole-3a-carboxylate (2r): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 40% yield (19 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 4.99 (d, *J* = 0.7 Hz, 0.25H)/4.96 (d, *J* = 0.7 Hz, 0.75H), 4.85 (s, 0.25H)/4.83 (s, 0.75H), 4.58 – 4.52 (m, 0.25H)/4.46 (td, *J* = 8.4, 3.5 Hz, 0.75H), 4.22 – 4.09 (m, 2H), 2.78 – 2.54 (m, 2H), 2.50 – 2.42 (m, 1H), 2.32 – 2.21 (m, 1H), 2.02 – 1.93 (m, 1H), 1.78 (s, 0.75H)/1.74 (s, 2.25H), 1.73 – 1.38 (m, 4H), 1.31 – 1.20 (m, 4H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 175.7/172.6, 173.2/169.9, 146.2/146.0, 110.7/110.6, 74.9, 61.1, 42.5, 37.9, 30.9, 26.7, 24.6, 23.3, 19.4, 14.2/14.1; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2922, 1610, 1486, 1071, 861; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{14}H_{22}NO_2$ 236.1645, found 236.1632.

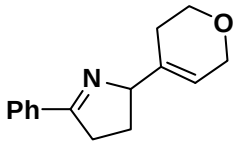


2-(Cyclopent-1-en-1-yl)-5-phenyl-3,4-dihydro-*2H*-pyrrole (2s): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 57% yield (24 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.88 (dd, *J* = 7.5, 1.3 Hz, 2H), 7.46 – 7.37 (m, 3H), 5.58 (s, 1H), 4.89 (t, *J* = 7.0 Hz, 1H), 3.08 – 2.97 (m, 1H), 2.98 – 2.88 (m, 1H), 2.42 – 2.27 (m, 4H), 2.27 – 2.18 (m, 1H), 1.94 – 1.89 (m, 2H), 1.89 – 1.81 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 173.0, 145.8,

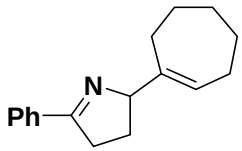
134.5, 130.5, 128.4, 127.8, 124.8, 72.9, 35.2, 32.3, 28.0, 23.3; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2947, 1614, 1342, 1054, 765; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{15}H_{18}N$ 212.1434, found 212.1432.



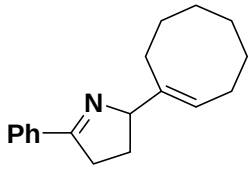
2-(Cyclohex-1-en-1-yl)-5-phenyl-3,4-dihydro-*2H*-pyrrole (2t): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 75% yield (34 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.08 – 7.72 (m, 2H), 7.48 – 7.29 (m, 3H), 5.70 – 5.62 (m, 1H), 4.65 (t, *J* = 7.3 Hz, 1H), 3.02 (dddd, *J* = 17.4, 9.9, 5.4, 2.2 Hz, 1H), 2.91 (dddd, *J* = 16.9, 9.6, 7.1, 1.7 Hz, 1H), 2.22 (dddd, *J* = 12.9, 9.8, 8.4, 5.4 Hz, 1H), 2.11 – 2.00 (m, 2H), 2.00 – 1.90 (m, 2H), 1.88 – 1.74 (m, 1H), 1.71 – 1.53 (m, 4H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 172.8, 139.2, 134.6, 130.4, 128.4, 127.8, 121.8, 78.2, 35.3, 28.2, 25.14, 25.10, 22.8, 22.6; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2886, 1586, 1559, 1483, 1209, 946, 714; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{16}H_{20}N$ 226.1590, found 226.1587.



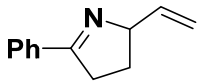
2-(3,6-Dihydro-*2H*-pyran-4-yl)-5-phenyl-3,4-dihydro-*2H*-pyrrole (2u): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 57% yield (26 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.00 – 7.74 (m, 2H), 7.59 – 7.32 (m, 3H), 5.69 – 5.65 (m, 1H), 4.69 (t, *J* = 7.3 Hz, 1H), 4.24 – 4.10 (m, 2H), 4.00 – 3.68 (m, 2H), 3.10 – 2.97 (m, 1H), 2.94 (dddd, *J* = 16.9, 9.3, 7.3, 1.7 Hz, 1H), 2.35 – 2.20 (m, 1H), 2.20 – 2.00 (m, 2H), 1.85 – 1.77 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 173.5, 137.2, 134.4, 130.6, 128.4, 127.8, 120.3, 77.0, 65.4, 64.2, 35.24, 28.0, 25.7; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1616, 1340, 1126, 847; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{15}H_{18}NO$ 228.1388, found 228.1392.



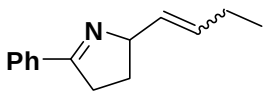
2-(Cyclohept-1-en-1-yl)-5-phenyl-3,4-dihydro-*2H*-pyrrole (2v): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 83% yield (40 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.00 – 7.71 (m, 2H), 7.54 – 7.33 (m, 3H), 5.81 (t, *J* = 6.5 Hz, 1H), 4.68 (t, *J* = 7.4 Hz, 1H), 3.08 – 2.96 (m, 1H), 2.96 – 2.84 (m, 1H), 2.29 – 2.17 (m, 2H), 2.17 – 2.10 (m, 2H), 2.10 – 2.02 (m, 2H), 1.84 – 1.67 (m, 3H), 1.56 – 1.47 (m, 4H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.9, 145.3, 134.6, 130.3, 128.3, 127.7, 126.8, 79.6, 35.1, 32.7, 29.7, 28.3, 28.1, 27.2, 27.0; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2920, 1613, 1446, 1250, 845; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{17}H_{22}N$ 240.1747, found 240.1744.



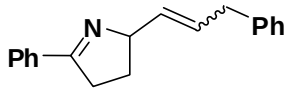
(*E*)-2-(cyclooct-1-en-1-yl)-5-phenyl-3,4-dihydro-*2H*-pyrrole (2w): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 70% yield (35 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.88 (dd, *J* = 7.4, 1.4 Hz, 2H), 7.51 – 7.30 (m, 3H), 5.59 (t, *J* = 8.2 Hz, 1H), 4.73 (t, *J* = 7.3 Hz, 1H), 3.06 – 2.95 (m, 1H), 2.95 – 2.86 (m, 1H), 2.31 – 2.21 (m, 1H), 2.21 – 2.12 (m, 4H), 1.85 – 1.75 (m, 1H), 1.62 – 1.49 (m, 8H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 172.8, 141.9, 134.7, 130.3, 128.4, 127.8, 124.6, 78.4, 35.1, 29.7, 29.5, 28.8, 27.0, 26.5, 26.4, 26.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1615, 1559, 1483, 1209, 946; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{18}H_{24}N$ 254.1903, found 254.1908.



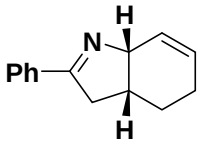
5-Phenyl-2-vinyl-3,4-dihydro-*2H*-pyrrole (3a): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 56% yield (19 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.99 – 7.79 (m, 2H), 7.56 – 7.32 (m, 3H), 6.00 (ddd, *J* = 17.1, 10.3, 6.7 Hz, 1H), 5.27 (dt, *J* = 17.2, 1.5 Hz, 1H), 5.17 – 5.06 (m, 1H), 4.82 – 4.64 (m, 1H), 3.06 (dddd, *J* = 16.8, 9.7, 5.1, 2.0 Hz, 1H), 2.92 (dddd, *J* = 16.9, 9.4, 7.5, 1.8 Hz, 1H), 2.41 – 2.15 (m, 1H), 1.88 – 1.64 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 173.2, 140.1, 134.4, 130.5, 128.4, 127.7, 114.6, 74.1, 35.0, 29.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2916, 1616, 1507, 919; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{12}H_{14}N$ 172.1126, found 172.1122.



2-(But-1-en-1-yl)-5-phenyl-3,4-dihydro-*2H*-pyrrole (3b): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 70% yield (28 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.15 – 7.75 (m, 2H), 7.51 – 7.33 (m, 3H), 5.84 – 5.65 (m, 0.8H)/5.44 – 5.33 (m, 0.2H), 5.61 – 5.47 (m, 1H), 5.00 (q, *J* = 7.8 Hz, 0.2H)/4.68 (q, *J* = 7.2 Hz, 0.8H), 3.16 – 2.99 (m, 1H), 2.97 – 2.79 (m, 1H), 2.38 – 2.18 (m, 1.4H), 2.13 – 2.04 (m, 1.6H), 1.82 – 1.66 (m, 1H), 1.05 (d, *J* = 7.5 Hz, 0.75H)/1.01 (t, *J* = 7.4 Hz, 2.25H); **^{13}C NMR (100 MHz, $CDCl_3$)** 172.3, 134.6, 132.9/132.7, 131.4/130.8, 130.4, 128.4, 127.8, 74.6/69.8, 35.3/35.1, 30.5/29.8, 25.4/21.1, 14.4/13.5; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2963, 1612, 1412, 1339, 968; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{14}H_{18}N$ 200.1439, found 200.1438.

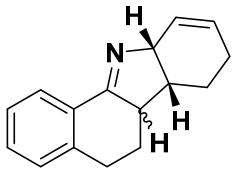


5-Phenyl-2-(3-phenylprop-1-en-1-yl)-3,4-dihydro-*2H*-pyrrole (3c): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 51% yield (27 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.94 – 7.78 (m, 2H), 7.50 – 7.35 (m, 3H), 7.34 – 7.24 (m, 3H), 7.24 – 7.15 (m, 2H), 5.93 – 5.80 (m, 0.75H)/5.78 – 5.69 (m, 0.25H), 5.70 – 5.62 (m, 0.75H)/5.61 – 5.54 (m, 0.25H), 5.23 – 5.06 (m, 0.25H)/4.73 (q, *J* = 7.1 Hz, 0.75H), 3.72 – 3.55 (m, 0.5H)/3.47 – 3.34 (m, 1.5H), 3.19 – 2.97 (m, 1H), 2.97 – 2.82 (m, 1H), 2.46 – 2.13 (m, 1H), 1.89 – 1.66 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 173.4/173.1, 140.6/140.4, 134.4/133.4, 133.0/130.5, 129.7/129.2, 128.6, 128.5/128.37, 128.35, 127.8, 126.0, 74.2/69.7, 38.8/35.4, 35.1/34.1, 30.4/29.7; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2963, 1612, 1412, 1339, 968; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{19}H_{20}N$ 262.1596, found 262.1594.

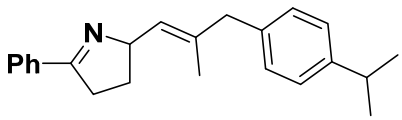


***cis*-2-Phenyl-3a,4,5,7a-tetrahydro-*3H*-indole (3d)**: Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 56% yield (22 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.01 – 7.74 (m, 2H), 7.46 – 7.38 (m, 3 H), 6.29 – 6.22 (m, 1H), 6.03 – 5.96 (m, 1H), 4.46 – 4.40 (m, 1H), 3.14 (ddd, *J* = 16.6, 8.2, 2.3 Hz, 1H), 2.87 – 2.77 (m, 1H), 2.66 – 2.53 (m, 1H), 2.06 –

1.94 (m, 2H), 1.68 (ddd, J = 13.3, 9.0, 4.5 Hz, 1H), 1.32 – 1.27 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 171.6, 134.6, 130.5, 130.0, 128.4, 127.7, 127.3, 69.9, 42.0, 35.2, 25.4, 23.2; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2926, 1612, 1450, 1345, 1264, 765; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{14}H_{16}N$ 198.1277, found 198.1274.

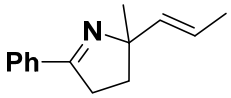


6,6a,6b,7,8,10a-Hexahydro-*5H*-benzo[*a*]carbazole (3e): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 49% yield (22 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.18 (d, J = 7.5 Hz, 0.5H)/ 8.12 (d, J = 7.4 Hz, 0.5H), 7.38 – 7.31 (m, 1H), 7.25 – 7.18 (m, 2H), 6.38 – 6.29 (m, 1H)/6.06 – 5.97 (m, 0.5H), 5.86 – 5.69 (m, 1H), 4.80 – 4.74 (m, 0.5H)/4.25 – 4.17 (m, 0.5H), 3.16 – 3.03 (m, 0.5H), 3.02 – 2.89 (m, 2H), 2.90 – 2.79 (m, 0.5H), 2.58 – 2.45 (m, 0.5H), 2.39 – 2.31 (m, 0.5H), 2.31 – 2.22 (m, 0.5H), 2.17 – 1.73 (m, 4H), 1.62 – 1.55 (m, 0.5H), 1.53 – 1.48 (m, 0.5H), 1.11 (ddd, J = 25.5, 12.5, 4.8 Hz, 0.5H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 173.7/171.0, 141.2/141.1, 130.7/130.3, 130.6/130.26, 130.5/130.23, 128.8/128.7, 127.6/127.4, 127.0/126.4, 125.9/125.7, 68.9/68.7, 51.0/46.9, 42.4/39.7, 30.2/30.0, 28.1/23.9, 22.9/21.34, 20.30/20.27; **FT-IR (thin film, KBr): ν (cm^{-1}):** 2925, 1612, 1468, 1352, 772; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{16}H_{18}N$ 224.1434, found 224.1431.

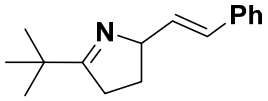


(*E*)-2-(3-(4-Isopropylphenyl)-2-methylprop-1-en-1-yl)-5-phenyl-3,4-dihydro-*2H*-pyrrole (3f): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 55% yield (35 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.89 (dd, J = 7.6, 1.9 Hz, 2H), 7.59 – 7.35 (m, 3H), 7.24 – 7.04 (m, 4H), 5.36 (d, J = 9.2 Hz, 1H), 5.15 – 4.99 (m, 1H), 3.70 (d, J = 14.6 Hz, 1H), 3.40 (d, J = 14.6 Hz, 1H), 3.12 (dddd, J = 16.2, 9.8, 4.2, 1.8 Hz, 1H), 2.96 – 2.83 (m, 2H), 2.47 – 2.27 (m, 1H), 1.81 – 1.73 (m, 1H), 1.70 (d, J = 1.1 Hz, 3H), 1.26 (d, J = 6.9 Hz, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 173.0, 146.5, 137.1, 135.9, 134.6, 130.4, 129.1, 128.6, 128.4, 127.8, 126.4, 70.7, 38.0, 35.4, 33.7, 30.7, 24.1, 23.5; **FT-IR (thin film,**

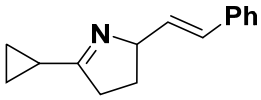
KBr): ν (cm^{-1}): 2962, 1612, 1454, 1334, 780; **HRMS** (CI) [M + H]$^+$ Calculated for $C_{23}H_{28}N$ 318.2222, found 318.2223.



(*E*)-2-methyl-5-phenyl-2-(prop-1-en-1-yl)-3,4-dihydro-*2H*-pyrrole (3g): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 63% yield (25 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.91 – 7.76 (m, 2H), 7.48 – 7.30 (m, 3H), 5.75 – 5.63 (m, 1H), 5.55 (dq, *J* = 15.3, 6.4 Hz, 1H), 3.13 – 2.91 (m, 2H), 2.04 (ddd, *J* = 12.6, 9.1, 5.9 Hz, 1H), 1.86 (ddd, *J* = 12.5, 9.2, 6.8 Hz, 1H), 1.68 (dd, *J* = 6.4, 0.7 Hz, 3H), 1.41 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 170.8, 137.4, 134.7, 130.3, 128.3, 127.7, 122.1, 76.1, 35.9, 34.9, 27.2, 17.9; **FT-IR (thin film, KBr):** 2934, 1612, 1454, 1341, 721; **HRMS** (CI) [M + H]$^+$ Calculated for $C_{14}H_{18}N$ 200.1439, found 200.1438.

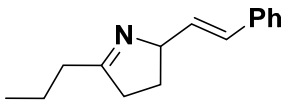


(*E*)-5-(*tert*-Butyl)-2-styryl-3,4-dihydro-*2H*-pyrrole (3h): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 52% yield (24 mg); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.40 – 7.28 (m, 4H), 7.26 – 7.19 (m, 1H), 6.54 (d, *J* = 11.5 Hz, 1H), 5.57 (dd, *J* = 11.5, 9.6 Hz, 1H), 4.97 – 4.85 (m, 1H), 2.71 (dddd, *J* = 16.9, 9.7, 4.5, 1.6 Hz, 1H), 2.63 – 2.46 (m, 1H), 2.30 – 2.10 (m, 1H), 1.63 (dddd, *J* = 12.7, 9.7, 8.2, 7.2 Hz, 2H), 1.18 (s, 9H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 185.3, 137.2, 134.8, 129.4, 128.9, 128.1, 126.8, 69.4, 35.8, 33.8, 30.9, 28.2; **FT-IR (thin film, KBr):** 2924, 1627, 1454, 1190, 771; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{16}H_{22}N$ 228.1747, found 228.1746.

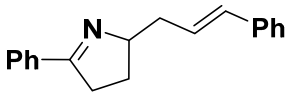


(*E*)-5-Cyclopropyl-2-styryl-3,4-dihydro-*2H*-pyrrole (3i): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 47% yield (20 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.37 (d, *J* = 7.6 Hz, 2H), 7.28 (t, *J* = 7.6 Hz, 2H), 7.20 (t, *J* = 7.3 Hz, 1H), 6.53 (d, *J* = 15.9 Hz, 1H), 6.24 (dd, *J* = 15.9, 6.9 Hz, 1H), 4.63 (dd, *J* = 13.6, 6.7 Hz, 1H), 2.51 – 2.41 (m, 1H), 2.37 (dt, *J* = 16.9, 8.4 Hz, 1H), 2.23 – 2.12 (m, 1H), 1.89 – 1.81 (m, 1H), 1.71 – 1.63 (m,

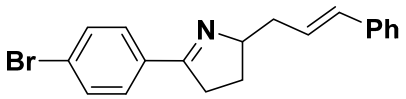
1H), 0.93 – 0.85 (m, 4H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 179.3, 136.3, 131.0, 128.5, 127.4, 126.1, 125.3, 72.3, 33.8, 28.4, 13.2, 6.55, 6.47; **FT-IR (thin film, KBr):** 2989, 1664, 1470, 1224, 715; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{15}H_{18}N$ 212.1439, found 212.1440.



(*E*)-5-Propyl-2-styryl-3,4-dihydro-*2H*-pyrrole (3j): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 45% yield (19 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.40 – 7.35 (m, 1H), 7.29 (t, *J* = 7.7 Hz, 2H), 7.20 (t, *J* = 7.3 Hz, 2H), 6.55 (d, *J* = 15.9 Hz, 1H), 6.26 (dd, *J* = 15.9, 6.8 Hz, 1H), 4.70 – 4.64 (m, 1H), 2.66 – 2.56 (m, 1H), 2.54 – 2.46 (m, 1H), 2.38 (t, *J* = 7.6 Hz, 2H), 2.25 – 2.17 (m, 1H), 1.79 – 1.60 (m, 3H), 0.98 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 179.2, 137.3, 132.0, 129.5, 128.4, 127.2, 126.3, 73.5, 37.3, 35.9, 29.6, 19.9, 14.0; **FT-IR (thin film, KBr):** 3009, 1647, 1454, 1223, 684; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{15}H_{20}N$ 214.1596, found 214.1597.

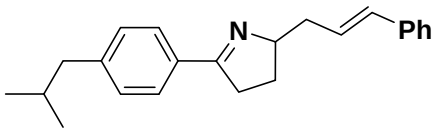


2-Cinnamyl-5-phenyl-3,4-dihydro-*2H*-pyrrole (5a): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 4:1) to give a brown solid; 61% yield (32 mg); m.p. 87 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.87 (d, *J* = 6.4 Hz, 2H), 7.49 – 7.40 (m, 3H), 7.37 (d, *J* = 7.5 Hz, 2H), 7.30 (t, *J* = 7.4 Hz, 2H), 7.21 (t, *J* = 7.0 Hz, 1H), 6.50 (d, *J* = 15.8 Hz, 1H), 6.39 – 6.21 (m, 1H), 4.44 – 4.31 (m, 1H), 3.12 – 2.98 (m, 1H), 2.91 (dt, *J* = 17.0, 8.5 Hz, 1H), 2.87 – 2.74 (m, 1H), 2.56 – 2.42 (m, 1H), 2.27 – 2.15 (m, 1H), 1.82 – 1.67 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 172.7, 137.7, 134.6, 132.0, 130.5, 128.50, 128.48, 127.8, 127.5, 127.0, 126.1, 72.9, 39.9, 35.1, 27.8; **FT-IR (thin film, KBr):** 2924, 1625, 1454, 1337, 800; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{19}H_{20}N$ 262.1590, found 262.1587.

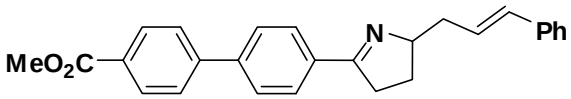


5-(4-Bromophenyl)-2-cinnamyl-3,4-dihydro-2H-pyrrole (5b): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 4:1) to give a

brown solid; 44% yield (30 mg); m.p. 112 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.72 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.36 (d, *J* = 7.7 Hz, 2H), 7.30 (t, *J* = 7.6 Hz, 2H), 7.21 (t, *J* = 7.3 Hz, 1H), 6.49 (d, *J* = 15.8 Hz, 1H), 6.40 – 6.17 (m, 1H), 4.36 (p, *J* = 7.4 Hz, 1H), 3.02 – 2.91 (m, 1H), 2.92 – 2.82 (m, 1H), 2.82 – 2.71 (m, 1H), 2.53 – 2.42 (m, 1H), 2.25 – 2.16 (m, 1H), 1.81 – 1.69 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 171.5, 137.6, 133.5, 132.1, 131.6, 129.2, 128.5, 127.2, 127.0, 126.1, 124.9, 73.0, 39.8, 35.0, 27.9; **FT-IR (thin film, KBr):** 2921, 1613, 1493, 1328, 814; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{19}H_{19}N^{79}Br$ 340.0695, found 340.0699.

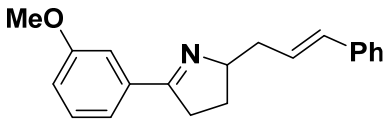


2-Cinnamyl-5-(4-isobutylphenyl)-3,4-dihydro-*2H*-pyrrole (5c): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 4:1) to give a brown solid; 49% yield (31 mg); m.p. 105 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.78 (d, *J* = 8.1 Hz, 2H), 7.37 (d, *J* = 7.6 Hz, 2H), 7.30 (t, *J* = 7.6 Hz, 2H), 7.22 – 7.18 (m, 3H), 6.50 (d, *J* = 15.8 Hz, 1H), 6.34 – 6.27 (m, 1H), 4.41 – 4.33 (m, 1H), 3.06 – 2.97 (m, 1H), 2.94 – 2.86 (m, 1H), 2.85 – 2.75 (m, 1H), 2.52 (d, *J* = 7.2 Hz, 2H), 2.49 – 2.42 (m, 1H), 2.22 – 2.16 (m, 1H), 1.90 (dp, *J* = 13.5, 6.7 Hz, 1H), 1.79 – 1.69 (m, 1H), 0.92 (d, *J* = 6.6 Hz, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.6, 144.4, 137.7, 132.1, 131.9, 129.2, 128.5, 127.6, 127.0, 126.1, 72.7, 45.3, 39.9, 35.0, 30.2, 27.8, 22.3. **FT-IR (thin film, KBr):** 2952, 1612, 1493, 1331, 856; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{23}H_{28}N$ 318.2216, found 318.2218.

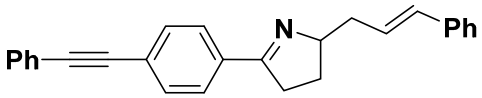


4'-(2-Cinnamyl-3,4-dihydro-*2H*-pyrrol-5-yl)-[1,1'-biphenyl]-4-yl acetate (5d): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 4:1) to give a white solid; 40% yield (32 mg); m.p. 171-172 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.12 (d, *J* = 8.3 Hz, 2H), 7.95 (d, *J* = 8.2 Hz, 2H), 7.71 – 7.66 (m, 4H), 7.36 (d, *J* = 7.5 Hz, 2H), 7.29 (t, *J* = 7.6 Hz, 2H), 7.20 (t, *J* = 7.3 Hz, 1H), 6.50 (d, *J* = 15.8 Hz, 1H), 6.34 – 6.26 (m, 1H), 4.46 – 4.37 (m, 1H), 3.95 (s, 3H), 3.10 – 3.01 (m, 1H), 2.94 (dt, *J* = 16.9, 8.3 Hz, 1H), 2.84 – 2.76 (m, 1H), 2.54 – 2.45 (m, 1H), 2.27 – 2.19 (m, 1H), 1.82 – 1.73 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.1,

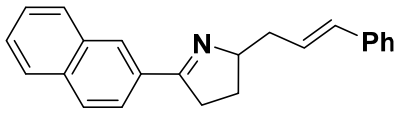
166.9, 144.7, 141.8, 137.6, 134.2, 132.0, 130.1, 129.3, 128.5, 128.3, 127.3, 127.2, 127.0, 126.0, 73.0, 52.1, 39.8, 35.1, 27.8; **FT-IR (thin film, KBr):** 2926, 1703, 1602, 1298, 1176, 838; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{27}H_{26}NO_2$ 396.1958, found 396.1955.



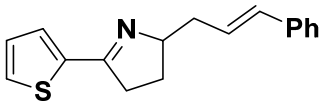
2-Cinnamyl-5-(3-methoxyphenyl)-3,4-dihydro-*2H*-pyrrole (5e): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a white solid; 67% yield (39 mg); m.p. 116 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.48 (s, 1H), 7.40 – 7.35 (m, 3H), 7.34 – 7.28 (m, 3H), 7.21 (t, *J* = 7.3 Hz, 1H), 7.03 – 6.96 (m, 1H), 6.50 (d, *J* = 15.8 Hz, 1H), 6.39 – 6.22 (m, 1H), 4.49 – 4.21 (m, 1H), 3.86 (s, 3H), 3.06 – 2.95 (m, 1H), 2.90 (dt, *J* = 17.0, 8.6 Hz, 1H), 2.80 (dt, *J* = 12.5, 6.1 Hz, 1H), 2.52 – 2.40 (m, 1H), 2.23 – 2.13 (m, 1H), 1.79 – 1.72 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.6, 159.6, 137.6, 135.9, 132.0, 129.4, 128.5, 127.4, 127.0, 126.1, 120.5, 116.9, 112.2, 72.8, 55.4, 39.8, 35.2, 27.8; **FT-IR (thin film, KBr):** 2928, 1602, 1579, 1430, 1039, 784; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{20}H_{22}NO$ 292.1696, found 292.1700.



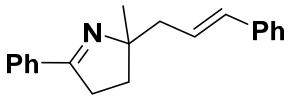
2-Cinnamyl-5-(4-(phenylethynyl)phenyl)-3,4-dihydro-*2H*-pyrrole (5f): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a yellowish solid; 37% yield (27 mg); m.p. 112 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.84 (d, *J* = 8.1 Hz, 2H), 7.66 – 7.49 (m, 4H), 7.36 (d, *J* = 8.7 Hz, 5H), 7.30 (t, *J* = 7.6 Hz, 2H), 7.21 (t, *J* = 7.2 Hz, 1H), 6.50 (d, *J* = 15.8 Hz, 1H), 6.36 – 6.21 (m, 1H), 4.49 – 4.33 (m, 1H), 3.09 – 2.95 (m, 1H), 2.90 (dt, *J* = 16.9, 8.5 Hz, 1H), 2.83 – 2.76 (m, 1H), 2.48 (dt, *J* = 14.9, 7.6 Hz, 1H), 2.28 – 2.17 (m, 1H), 1.85 – 1.72 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.0, 137.8, 134.1, 132.0, 131.63, 131.59, 128.5, 128.4, 127.7, 127.3, 127.0, 126.1, 125.3, 123.0, 91.2, 89.1, 73.0, 39.8, 35.0, 27.9; **FT-IR (thin film, KBr):** 2921, 1602, 1514, 1338, 1194, 850; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{27}H_{24}N$ 362.1903, found 362.1907.



2-Cinnamyl-5-(naphthalen-2-yl)-3,4-dihydro-*2H*-pyrrole (5g): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a white solid; 65% yield (40 mg); m.p. 104 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.19 (s, 1H), 8.12 (d, *J* = 8.6 Hz, 1H), 7.98 – 7.79 (m, 3H), 7.57 – 7.47 (m, 2H), 7.37 (d, *J* = 7.5 Hz, 2H), 7.30 (t, *J* = 7.4 Hz, 2H), 7.20 (t, *J* = 7.1 Hz, 1H), 6.52 (d, *J* = 15.9 Hz, 1H), 6.41 – 6.12 (m, 1H), 4.58 – 4.33 (m, 1H), 3.22 – 3.11 (m, 1H), 3.04 (dt, *J* = 16.9, 8.5 Hz, 1H), 2.89 – 2.78 (m, 1H), 2.61 – 2.46 (m, 1H), 2.34 – 2.19 (m, 1H), 1.88 – 1.78 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.6, 137.7, 134.4, 133.0, 132.0, 128.7, 128.5, 128.3, 128.1, 127.8, 127.4, 127.1, 127.0, 126.4, 126.1, 124.7, 73.0, 39.9, 35.1, 27.9; **FT-IR (thin film, KBr):** 2924, 1625, 1454, 1337, 800; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{23}H_{22}N$ 312.1747, found 312.1744.

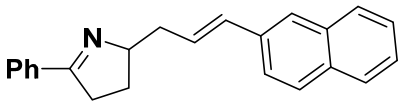


2-Cinnamyl-5-(thiophen-2-yl)-3,4-dihydro-*2H*-pyrrole (5h): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a colorless oil; 74% yield (40 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.42 (d, *J* = 5.0 Hz, 1H), 7.35 (d, *J* = 7.4 Hz, 2H), 7.33 (d, *J* = 3.5 Hz, 1H), 7.29 (t, *J* = 7.6 Hz, 2H), 7.20 (t, *J* = 7.3 Hz, 1H), 7.07 (dd, *J* = 4.9, 3.8 Hz, 1H), 6.48 (d, *J* = 15.8 Hz, 1H), 6.39 – 6.12 (m, 1H), 4.69 – 4.19 (m, 1H), 3.05 – 2.94 (m, 1H), 2.94 – 2.83 (m, 1H), 2.84 – 2.74 (m, 1H), 2.51 – 2.41 (m, 1H), 2.25 – 2.13 (m, 1H), 1.82 – 1.71 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 167.0, 139.5, 137.6, 132.0, 129.2, 129.0, 128.5, 127.4, 127.3, 127.0, 126.1, 72.7, 39.7, 35.7, 28.0; **FT-IR (thin film, KBr):** 2926, 1602, 1454, 1227, 790; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{17}H_{18}NS$ 268.1154, found 268.1156.

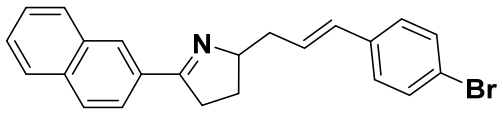


2-Cinnamyl-2-methyl-5-phenyl-3,4-dihydro-*2H*-pyrrole (5i): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a

white solid; 42% yield (23 mg); m.p. 102 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.85 (dd, *J* = 7.8, 1.6 Hz, 2H), 7.47 – 7.38 (m, 3H), 7.38 – 7.31 (m, 2H), 7.28 (t, *J* = 7.7 Hz, 2H), 7.20 (t, *J* = 7.3 Hz, 1H), 6.47 (d, *J* = 15.8 Hz, 1H), 6.31 – 6.16 (m, 1H), 3.03 (ddd, *J* = 16.0, 9.7, 6.2 Hz, 1H), 2.99 – 2.89 (m, 1H), 2.67 – 2.46 (m, 2H), 2.04 (ddd, *J* = 12.7, 9.8, 6.2 Hz, 1H), 1.78 (ddd, *J* = 12.7, 9.8, 6.0 Hz, 1H), 1.38 (s, 3H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 170.5, 137.7, 134.8, 132.7, 130.3, 128.44, 128.36, 127.7, 127.0, 126.9, 126.1, 76.3, 45.5, 35.4, 33.4, 27.5; **FT-IR (thin film, KBr):** 2924, 1604, 1456, 1335, 779; **HRMS** (ESI) $[M + H]^+$Calculated for $C_{20}H_{22}N$ 276.1747, found 276.1748.

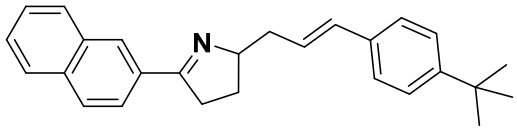


(*E*)-2-(3-(Naphthalen-2-yl)allyl)-5-phenyl-3,4-dihydro-*2H*-pyrrole (5j): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a brown solid; 56% yield (35 mg); m.p. 110 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.89 (d, *J* = 5.9 Hz, 2H), 7.83 – 7.74 (m, 3H), 7.69 (s, 1H), 7.59 (d, *J* = 8.5 Hz, 1H), 7.50 – 7.38 (m, 5H), 6.66 (d, *J* = 15.8 Hz, 1H), 6.59 – 6.24 (m, 1H), 4.63 – 4.27 (m, 1H), 3.14 – 2.99 (m, 1H), 2.95 (dd, *J* = 17.0, 8.8 Hz, 1H), 2.91 – 2.81 (m, 1H), 2.64 – 2.48 (m, 1H), 2.34 – 2.16 (m, 1H), 1.90 – 1.73 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.8, 135.1, 134.5, 133.7, 132.7, 132.1, 130.5, 128.4, 128.0, 127.9, 127.83, 127.76, 127.6, 126.1, 125.6, 125.5, 123.6, 72.9, 40.0, 35.1, 27.8; **FT-IR (thin film, KBr):** 2926, 1623, 1454, 1344, 780; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{23}H_{22}N$ 312.1747, found 312.1742.

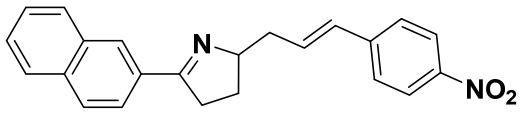


(*E*)-2-(3-(4-bromophenyl)allyl)-5-(naphthalen-2-yl)-3,4-dihydro-*2H*-pyrrole (5k): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a yellow solid; 53% yield (41 mg); m.p. 112-114 ℃; **^{1}H NMR (600 MHz, $CDCl_3$) δ** 8.16 (s, 1H), 8.12 – 8.08 (m, 1H), 7.89 (d, *J* = 7.4 Hz, 1H), 7.85 (t, *J* = 7.3 Hz, 2H), 7.53 – 7.48 (m, 2H), 7.43 – 7.36 (m, 2H), 7.21 (d, *J* = 8.3 Hz, 2H), 6.44 (d, *J* = 15.8 Hz, 1H), 6.35 – 6.28 (m, 1H), 4.47 – 4.37 (m, 1H), 3.17 – 3.11 (m, 1H), 3.07 – 2.98 (m, 1H), 2.82 – 2.76 (m, 1H), 2.54 – 2.46 (m, 1H), 2.28 – 2.21 (m,

1H), 1.80 – 1.72 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.7, 136.6, 134.4, 133.0, 132.0, 131.5, 130.8, 128.7, 128.4, 128.3, 128.1, 127.7, 127.6, 127.0, 126.3, 124.6, 120.6, 72.9, 39.9, 35.1, 28.0; **FT-IR (thin film, KBr):** 2921, 1613, 1486, 1124, 747; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{23}H_{21}{}^{79}BrN$ 390.0852, found 390.0850.

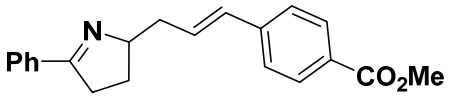


(*E*)-2-(3-(4-(*tert*-Butyl)phenyl)allyl)-5-(naphthalen-2-yl)-3,4-dihydro-*2H*-pyrrole (5l)：Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a yellowish solid; 48% yield (34 mg); m.p. 123-124 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.18 (s, 1H), 8.12 (d, *J* = 8.6 Hz, 1H), 7.90 (d, *J* = 7.6 Hz, 1H), 7.87 (t, *J* = 7.5 Hz, 2H), 7.55 – 7.49 (m, 2H), 7.35 – 7.29 (m, 4H), 6.50 (d, *J* = 15.8 Hz, 1H), 6.31 – 6.24 (m, 1H), 4.48 – 4.39 (m, 1H), 3.14 (ddd, *J* = 15.0, 9.9, 4.9 Hz, 1H), 3.07 – 2.99 (m, 1H), 2.87 – 2.79 (m, 1H), 2.55 – 2.47 (m, 1H), 2.28 – 2.19 (m, 1H), 1.84 – 1.76 (m, 1H), 1.31 (s, 9H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.5, 150.0, 134.9, 134.4, 133.0, 132.1, 131.7, 128.7, 128.2, 128.1, 127.7, 127.0, 126.6, 126.3, 125.8, 125.4, 124.6, 73.1, 39.9, 35.1, 34.5, 31.3, 27.8; **FT-IR (thin film, KBr):** 2959, 1609, 1458, 1359, 747; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{27}H_{30}N$ 368.2373, found 368.2375.

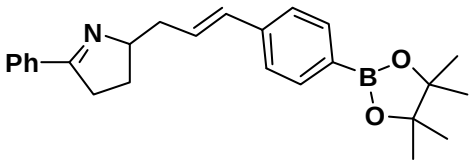


(*E*)-5-(Naphthalen-2-yl)-2-(3-(4-nitrophenyl)allyl)-3,4-dihydro-*2H*-pyrrole (5m): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a white solid; 46% yield (33 mg); m.p. 109-110 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.18 (s, 1H), 8.15 (d, *J* = 8.7 Hz, 2H), 8.11 (dd, *J* = 8.6, 1.4 Hz, 1H), 7.90 (d, *J* = 7.4 Hz, 1H), 7.87 (t, *J* = 7.6 Hz, 2H), 7.56 – 7.50 (m, 2H), 7.48 (d, *J* = 8.7 Hz, 2H), 6.62 – 6.55 (m, 2H), 4.51 – 4.40 (m, 1H), 3.21 – 3.13 (m, 1H), 3.12 – 3.01 (m, 1H), 2.87 – 2.78 (m, 1H), 2.66 – 2.56 (m, 1H), 2.33 – 2.26 (m, 1H), 1.82 – 1.72 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.9, 146.5, 144.1, 134.4, 133.1, 132.9, 131.9, 130.2, 128.7, 128.3, 128.2, 127.8, 127.2, 126.5, 126.4, 124.5, 123.9, 72.6, 40.2,

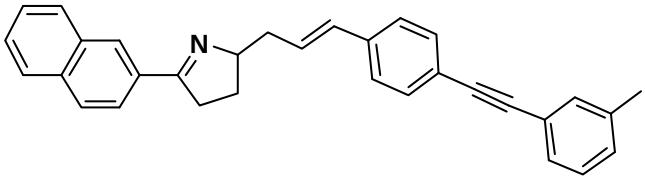
35.1, 28.2; **FT-IR (thin film, KBr):** 2881, 1598, 1511, 1349, 715; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{23}H_{21}N_2O_2$ 357.1598, found 357.1596.



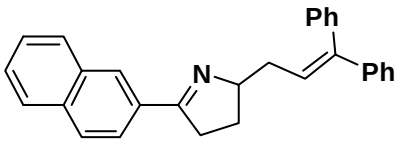
Methyl (*E*)-4-(3-(5-phenyl-3,4-dihydro-*2H*-pyrrol-2-yl)prop-1-en-1-yl)benzoate (5n): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a white solid; 64% yield (41 mg); m.p. 107-109 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.85 (dd, *J* = 7.8, 1.3 Hz, 2H), 7.50 – 7.34 (m, 5H), 6.53 (d, *J* = 15.9 Hz, 1H), 6.48 – 6.38 (m, 1H), 4.48 – 4.27 (m, 1H), 3.90 (s, 3H), 3.02 (dddd, *J* = 12.0, 7.1, 6.2, 3.7 Hz, 1H), 2.96 – 2.85 (m, 1H), 2.83 – 2.72 (m, 1H), 2.50 (dt, *J* = 14.6, 7.4 Hz, 1H), 2.21 (dddd, *J* = 12.8, 9.9, 7.9, 5.0 Hz, 1H), 1.77 – 1.66 (m, 1H); **^{3}C NMR (150 MHz, $CDCl_3$)** δ 172.8, 167.0, 142.1, 134.4, 131.1, 130.6, 130.5, 129.9, 128.43, 128.41, 127.7, 125.9, 72.7, 52.0, 40.0, 35.1, 27.9; **FT-IR (thin film, KBr):** 2881, 1598, 1511, 1349, 715; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{21}H_{22}NO_2$ 320.1645, found 320.1646.



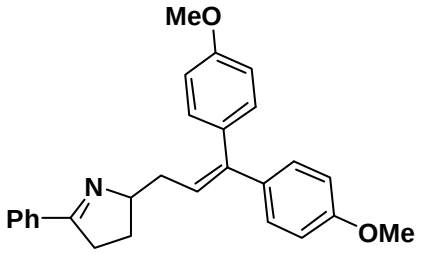
(*E*)-5-Phenyl-2-(3-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)allyl)-3,4-dihydro-*2H*-pyrrole (5o): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 4:1) to give a white solid; 45% yield (35 mg); m.p. 114 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.87 – 7.84 (m, 2H), 7.74 (d, *J* = 7.7 Hz, 2H), 7.46 – 7.39 (m, 3H), 7.36 (d, *J* = 7.8 Hz, 2H), 6.50 (d, *J* = 15.8 Hz, 1H), 6.40 – 6.32 (m, 1H), 4.37 (p, *J* = 7.1 Hz, 1H), 3.06 – 2.97 (m, 1H), 2.90 (dt, *J* = 16.9, 8.4 Hz, 1H), 2.83 – 2.76 (m, 1H), 2.50 – 2.43 (m, 1H), 2.25 – 2.14 (m, 1H), 1.78 – 1.70 (m, 1H), 1.34 (s, 12H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.6, 140.4, 134.9, 134.5, 131.9, 130.8, 130.4, 128.7, 128.4, 127.7, 125.4, 83.7, 72.8, 39.9, 35.1, 27.8, 24.8; **^{11}B NMR (128 MHz, $CDCl_3$)** δ 31.59 (s); **FT-IR (thin film, KBr):** 2924, 1726, 1606, 1454, 1352, 1140, 830; **HRMS** (ESI) [M + H]$^+$ Calculated for $C_{25}H_{31}BNO_2$ 388.2442, found 388.2446.



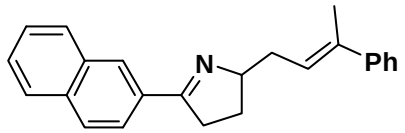
(*E*)-5-(Naphthalen-2-yl)-2-(3-(4-(m-tolylethynyl)phenyl)allyl)-3,4-dihydro-*2H*-pyrrole (5p): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a yellowish solid; 45% yield (38 mg); m.p. 123-124 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.18 (s, 1H), 8.12 (dd, *J* = 8.6, 1.5 Hz, 1H), 7.92 – 7.89 (m, 1H), 7.87 (t, *J* = 8.1 Hz, 2H), 7.56 – 7.49 (m, 2H), 7.46 (d, *J* = 8.2 Hz, 2H), 7.37 – 7.31 (m, 4H), 7.23 (t, *J* = 7.6 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 6.51 (d, *J* = 15.9 Hz, 1H), 6.41 – 6.35 (m, 1H), 4.47 – 4.40 (m, 1H), 3.16 (dddd, *J* = 14.9, 9.9, 4.9, 2.0 Hz, 1H), 3.09 – 3.01 (m, 1H), 2.87 – 2.80 (m, 1H), 2.57 – 2.50 (m, 1H), 2.35 (s, 3H), 2.27 (dddd, *J* = 12.7, 9.9, 7.8, 5.0 Hz, 1H), 1.82 – 1.75 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.6, 138.0, 137.5, 134.4, 133.0, 132.1, 132.0, 131.7, 131.4, 129.1, 128.7, 128.63, 128.61, 128.2, 128.1, 127.7, 127.0, 126.4, 126.0, 124.6, 123.1, 121.8, 90.0, 89.2, 72.9, 40.0, 35.1, 27.9, 21.2; **FT-IR (thin film, KBr):** 2921, 1598, 1521, 1345, 850; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{32}H_{28}N$ 426.2216, found 426.2219.



2-(3,3-Diphenylallyl)-5-(naphthalen-2-yl)-3,4-dihydro-*2H*-pyrrole (5q): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a white solid; 52% yield (40 mg); m.p. 108 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.17 (s, 1H), 8.11 (d, *J* = 8.6 Hz, 1H), 7.90 (d, *J* = 7.4 Hz, 1H), 7.86 (dd, *J* = 8.1, 4.0 Hz, 2H), 7.56 – 7.48 (m, 2H), 7.43 (d, *J* = 8.3 Hz, 1H), 7.37 (t, *J* = 7.4 Hz, 2H), 7.34 – 7.29 (m, 2H), 7.28 – 7.21 (m, 5H), 6.27 (t, *J* = 7.4 Hz, 1H), 4.45 – 4.35 (m, 1H), 3.15 – 3.08 (m, 1H), 3.02 (dt, *J* = 16.7, 8.6 Hz, 1H), 2.81 – 2.69 (m, 1H), 2.51 – 2.41 (m, 1H), 2.30 – 2.21 (m, 1H), 1.79 – 1.66 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.5, 143.2, 142.7, 140.1, 134.4, 133.0, 132.0, 130.0, 128.7, 128.2, 128.13, 128.09, 128.0, 127.7, 127.3, 127.0, 126.9, 126.5, 126.3, 125.8, 124.6, 73.3, 36.5, 35.1, 28.2; **FT-IR (thin film, KBr):** 2921, 1613, 1598, 1503, 1349, 752; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{29}H_{26}N$ 388.2060, found 388.2061.

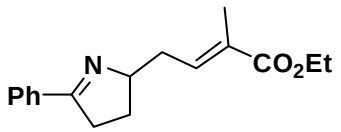


2-(3,3-Bis(4-methoxyphenyl)allyl)-5-phenyl-3,4-dihydro-*2H*-pyrrole (5r): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a white solid; 48% yield (38 mg); m.p. 113 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.84 (dd, *J* = 7.8, 1.5 Hz, 2H), 7.44 – 7.38 (m, 3H), 7.18 – 7.14 (m, 2H), 7.14 – 7.11 (m, 2H), 6.91 – 6.86 (m, 2H), 6.81 – 6.77 (m, 2H), 6.07 (t, *J* = 7.4 Hz, 1H), 4.32 (p, *J* = 7.5 Hz, 1H), 3.82 (s, 3H), 3.79 (s, 3H), 2.97 (dddd, *J* = 15.0, 9.9, 5.0, 2.1 Hz, 1H), 2.92 – 2.84 (m, 1H), 2.69 (ddd, *J* = 14.3, 7.1, 5.8 Hz, 1H), 2.42 – 2.35 (m, 1H), 2.18 (dddd, *J* = 12.8, 9.8, 7.8, 5.0 Hz, 1H), 1.70 – 1.58 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.3, 158.6, 158.4, 142.1, 135.8, 134.5, 132.6, 131.1, 130.3, 128.4, 128.3, 127.7, 124.5, 113.5, 113.4, 73.3, 55.2, 55.2, 36.5, 35.0, 28.1; **FT-IR (thin film, KBr):** 2930, 1612, 1454, 1178, 780; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{27}H_{28}NO_2$ 398.2115, found 398.2113.

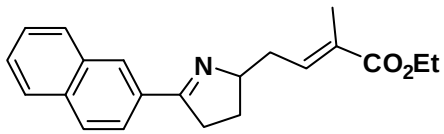


(*E*)-5-(Naphthalen-2-yl)-2-(3-phenylbut-2-en-1-yl)-3,4-dihydro-*2H*-pyrrole (5s): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a brown solid; 40% yield (26 mg); m.p. 98 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.18 (s, 1H), 8.12 (dd, *J* = 8.6, 1.4 Hz, 1H), 7.94 – 7.89 (m, 1H), 7.86 (t, *J* = 7.3 Hz, 2H), 7.58 – 7.48 (m, 2H), 7.42 – 7.36 (m, 2H), 7.31 (t, *J* = 7.7 Hz, 2H), 7.22 (t, *J* = 7.3 Hz, 1H), 5.99 – 5.77 (m, 1H), 4.44 (p, *J* = 7.5 Hz, 1H), 3.17 (dddd, *J* = 14.9, 9.8, 4.9, 2.0 Hz, 1H), 3.10 – 2.99 (m, 1H), 2.91 – 2.82 (m, 1H), 2.58 – 2.43 (m, 1H), 2.28 (dddd, *J* = 12.7, 9.9, 7.8, 5.0 Hz, 1H), 2.11 (s, 3H), 1.83 – 1.77 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.5, 143.9, 136.6, 134.4, 132.9, 132.1, 128.7, 128.2, 128.12, 128.08, 127.7, 127.0, 126.6, 126.3, 125.7, 124.9, 124.6, 73.2, 35.6, 35.1, 28.1,

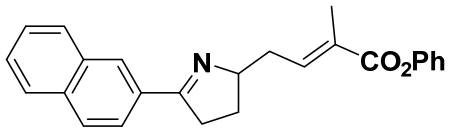
16.2; **FT-IR (thin film, KBr):** 2928, 1613, 1489, 1352, 744; **HRMS**(ESI) $[M + H]^+$ Calculated for $C_{24}H_{24}N$ 326.1903, found 326.1905.



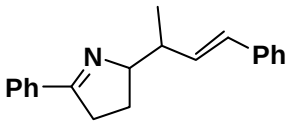
Ethyl (*E*)-2-methyl-4-(5-phenyl-3,4-dihydro-*2H*-pyrrol-2-yl)but-2-enoate (5t): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 10:1 to 4:1) to give a colorless oil; 50% yield (27 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.84 (d, *J* = 7.5 Hz, 2H), 7.51 – 7.34 (m, 3H), 6.88 (t, *J* = 7.4 Hz, 1H), 4.40 – 4.30 (m, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 3.08 – 2.99 (m, 1H), 2.96 – 2.87 (m, 1H), 2.80 – 2.70 (m, 1H), 2.42 (dt, *J* = 15.1, 7.7 Hz, 1H), 2.28 – 2.19 (m, 1H), 1.88 (s, 3H), 1.72 – 1.55 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.8, 168.1, 138.7, 134.4, 130.5, 129.4, 128.4, 127.7, 72.2, 60.5, 35.6, 35.1, 28.3, 14.3, 12.7; **FT-IR (thin film, KBr):** 2930, 1705, 1616, 1451, 1268, 760; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{17}H_{22}NO_2$ 272.1645, found 272.1651.



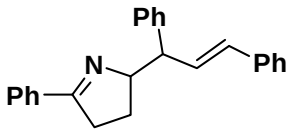
Ethyl (*E*)-2-methyl-4-(5-(naphthalen-2-yl)-3,4-dihydro-*2H*-pyrrol-2-yl)but-2-enoate (5u): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 10:1 to 4:1) to give a colorless oil; 63% yield (40 mg); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.17 (s, 1H), 8.09 (dd, *J* = 8.6, 1.2 Hz, 1H), 7.93 – 7.88 (m, 1H), 7.88 – 7.84 (m, 2H), 7.56 – 7.48 (m, 2H), 6.92 (t, *J* = 7.4 Hz, 1H), 4.45 – 4.33 (m, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 3.18 (dddd, *J* = 14.7, 9.8, 4.7, 1.9 Hz, 1H), 3.10 – 2.95 (m, 1H), 2.86 – 2.71 (m, 1H), 2.54 – 2.38 (m, 1H), 2.36 – 2.24 (m, 1H), 1.90 (s, 3H), 1.75 – 1.61 (m, 1H), 1.29 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.8, 168.1, 138.7, 134.4, 132.9, 131.9, 129.5, 128.7, 128.3, 128.1, 127.7, 127.1, 126.4, 124.5, 72.4, 60.5, 35.7, 35.1, 28.4, 14.3, 12.7; **FT-IR (thin film, KBr):** 2928, 1715, 1620, 1268, 747; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{21}H_{24}NO_2$ 322.1802, found 322.1800.



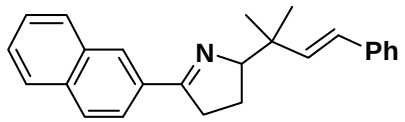
Phenyl (*E*)-2-methyl-4-(5-(naphthalen-2-yl)-3,4-dihydro-*2H*-pyrrol-2-yl)but-2-enoate (5v): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a white solid; 45% yield (34 mg): m.p. 113-115 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.18 (s, 1H), 8.10 (dd, *J* = 8.6, 1.5 Hz, 1H), 7.95 – 7.89 (m, 1H), 7.88 – 7.82 (m, 2H), 7.57 – 7.48 (m, 2H), 7.38 (t, *J* = 7.9 Hz, 2H), 7.25 – 7.17 (m, 2H), 7.15 – 7.08 (m, 2H), 4.46 (p, *J* = 7.4 Hz, 1H), 3.21 (dddd, *J* = 16.7, 9.9, 4.7, 2.0 Hz, 1H), 3.12 – 3.01 (m, 1H), 2.92 – 2.78 (m, 1H), 2.57 (dt, *J* = 15.0, 7.6 Hz, 1H), 2.34 (dddd, *J* = 12.6, 9.8, 7.8, 4.7 Hz, 1H), 2.03 (s, 3H), 1.74 (ddt, *J* = 12.8, 9.9, 7.3 Hz, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 173.0, 166.5, 151.1, 141.2, 134.4, 132.9, 131.84, 129.3, 128.9, 128.7, 128.3, 128.2, 127.7, 127.1, 126.4, 125.5, 124.5, 121.7, 72.3, 36.0, 35.2, 28.5, 12.8; **FT-IR (thin film, KBr):** 2928, 1736, 1655, 1489, 1278, 747; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{25}H_{24}NO_2$ 370.1802, found 370.1803.



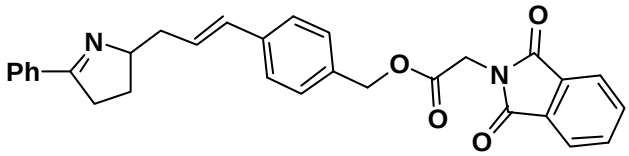
(*E*)-5-Phenyl-2-(4-phenylbut-3-en-2-yl)-3,4-dihydro-*2H*-pyrrole (5w): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a white solid; 43% yield (24 mg); m.p. 98-99 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.84 (m, 2H), 7.45 – 7.39 (m, 3H), 7.39 – 7.32 (m, 2H), 7.32 – 7.27 (m, 2H), 7.23 – 7.16 (m, 1H), 6.52 – 6.40 (m, 1H), 6.28 (dd, *J* = 16.0, 7.6 Hz, 0.6H)/6.21 (dd, *J* = 16.0, 7.4 Hz, 0.4H), 4.40 – 4.31 (m, 0.6H)/4.26 – 4.17 (m, 0.4H), 2.97 – 2.86 (m, 2H), 2.86 – 2.78 (m, 0.6H)/2.76 – 2.66 (m, 0.4H), 2.16 – 2.04 (m, 1H), 1.85 – 1.69 (m, 1H), 1.32 (d, *J* = 6.8 Hz, 1.2H)/1.18 (d, *J* = 6.9 Hz, 1.8H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.7/172.6, 137.80/137.78, 134.70/134.68, 133.6/132.9, 130.3, 129.8/129.6, 128.44, 128.36, 127.70/127.68, 126.9, 126.1/126.0, 77.9/77.6, 42.5/42.1, 35.3/35.2, 25.7/25.1, 17.7/16.0; **FT-IR (thin film, KBr):** 2922, 1611, 1512, 1352, 745; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{20}H_{22}N$ 276.1752, found 276.1748.



(*E*)-2-(1,3-Diphenylallyl)-5-phenyl-3,4-dihydro-*2H*-pyrrole (5x): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a yellow solid; 26% yield (18 mg); m.p. 118 ℃; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.91 – 7.82 (m, 2H), 7.48 – 7.36 (m, 7H), 7.35 – 7.27 (m, 4H), 7.25 – 7.16 (m, 2H), 6.72 (dd, *J* = 16.0, 7.7 Hz, 1H), 6.44 (d, *J* = 15.9 Hz, 1H), 4.75 – 4.65 (m, 1H), 3.66 (t, *J* = 7.2 Hz, 1H), 3.66 (t, *J* = 7.2 Hz, 1H), 2.88 – 2.69 (m, 2H), 2.14 – 2.00 (m, 1H), 1.78 – 1.69 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 173.1, 142.3, 137.7, 134.7, 131.7, 131.2, 130.4, 128.8, 128.44, 128.39, 128.3, 127.8, 127.1, 126.5, 126.3, 77.1, 54.9, 34.8, 27.2; **FT-IR (thin film, KBr):** 2924, 1615, 1545, 1478, 755; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{25}H_{24}N$ 338.1909, found 338.1910.

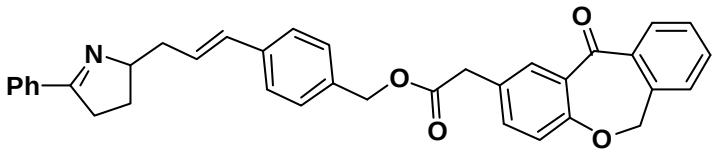


(*E*)-2-(2-Methyl-4-phenylbut-3-en-2-yl)-5-(naphthalen-2-yl)-3,4-dihydro-*2H*-pyrrole (5y): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a yellow solid; 12% yield (8 mg); m.p. 113-114 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.20 – 8.12 (m, 2H), 7.92 – 7.82 (m, 3H), 7.54 – 7.46 (m, 2H), 7.37 – 7.31 (m, 2H), 7.30 – 7.23 (m, 2H), 7.20 – 7.13 (m, 1H), 6.40 (d, *J* = 16.3 Hz, 1H), 6.30 (d, *J* = 16.3 Hz, 1H), 4.23 – 4.15 (m, 1H), 3.06 – 2.93 (m, 2H), 2.13 – 2.02 (m, 1H), 1.79 (ddt, *J* = 13.0, 9.4, 8.2 Hz, 1H), 1.31 (s, 3H), 1.28 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.6, 138.0, 137.8, 134.4, 133.0, 132.4, 128.7, 128.5, 128.1, 128.0, 127.8, 127.3, 126.9, 126.8, 126.3, 126.1, 124.8, 82.3, 41.0, 35.4, 25.3, 24.6, 24.4; **FT-IR (thin film, KBr):** 2959, 1608, 1489, 1134, 754; **HRMS** (CI) $[M + H]^+$ Calculated for $C_{25}H_{26}N$ 340.2065, found 340.2064.

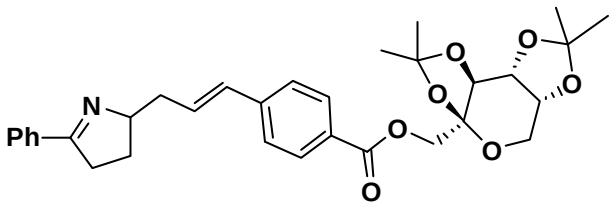


(*E*)-4-(3-(5-phenyl-3,4-dihydro-*2H*-pyrrol-2-yl)prop-1-en-1-yl)benzyl 2-(1,3-dioxoisoindolin-2-yl)acetate (5z): Purification by flash column chromatography on

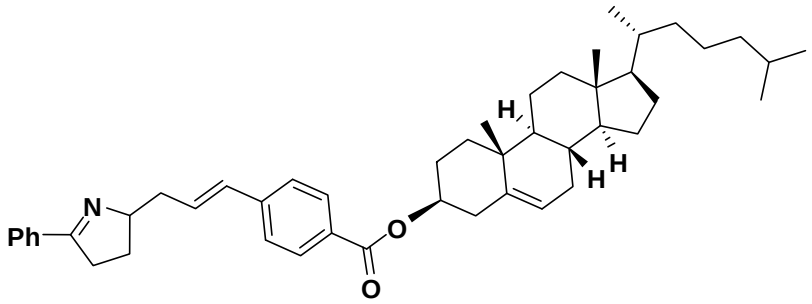
silica gel (petroleum ether/EtOAc = 20:1 to 8:1) to give a brown solid; 54% yield (52 mg); m.p. 171-172 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.91 – 7.83 (m, 4H), 7.76 – 7.72 (m, 2H), 7.45 – 7.39 (m, 3H), 7.35 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 6.4 Hz, 2H), 6.48 (d, *J* = 15.8 Hz, 1H), 6.35 – 6.27 (m, 1H), 5.16 (s, 2H), 4.48 (s, 2H), 4.37 (p, *J* = 7.4 Hz, 1H), 3.02 (dddd, *J* = 15.0, 9.9, 5.0, 2.1 Hz, 1H), 2.95 – 2.87 (m, 1H), 2.82 – 2.75 (m, 1H), 2.48 (dt, *J* = 14.9, 7.5 Hz, 1H), 2.20 (dddd, *J* = 12.9, 9.9, 7.9, 5.1 Hz, 1H), 1.77 – 1.68 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.6, 167.4, 167.2, 138.0, 134.5, 134.2, 133.5, 131.9, 131.4, 130.4, 128.6, 128.4, 128.3, 127.7, 126.3, 123.6, 72.8, 67.4, 39.9, 38.9, 35.1, 27.8; **FT-IR (thin film, KBr):** 2916, 1602, 1467, 1368, 780; **HRMS** (ESI) $[M+H]^+$ Calculated for $C_{30}H_{27}N_2O_4$ 479.1965, found 479.1960.



(*E*)-4-(3-(5-phenyl-3,4-dihydro-*2H*-pyrrol-2-yl)prop-1-en-1-yl)benzyl 2-(9-oxo-9H-xanthen-2-yl)acetate (5ba): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 8:1 to 2:1) to give a white solid; 44% yield (48 mg); m.p. 156-157 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.13 (d, *J* = 1.9 Hz, 1H), 7.89 (d, *J* = 7.6 Hz, 1H), 7.87 – 7.84 (m, 2H), 7.58 – 7.53 (m, 1H), 7.47 (t, *J* = 7.6 Hz, 1H), 7.45 – 7.39 (m, 4H), 7.38 – 7.32 (m, 3H), 7.26 (d, *J* = 7.8 Hz, 2H), 7.02 (d, *J* = 8.4 Hz, 1H), 6.48 (d, *J* = 15.8 Hz, 1H), 6.35 – 6.26 (m, 1H), 5.18 (s, 2H), 5.11 (s, 2H), 4.41 – 4.33 (m, 1H), 3.68 (s, 2H), 3.05 – 2.98 (m, 1H), 2.95 – 2.87 (m, 1H), 2.81 – 2.74 (m, 1H), 2.51 – 2.44 (m, 1H), 2.23 – 2.16 (m, 1H), 1.77 – 1.69 (m, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 190.8, 172.6, 171.2, 160.5, 140.4, 137.8, 136.3, 135.5, 134.5, 134.3, 132.7, 132.5, 131.4, 130.4, 129.5, 129.2, 128.5, 128.4, 128.1, 127.8, 127.70, 127.67, 126.2, 125.1, 121.0, 73.6, 72.8, 66.6, 40.2, 39.9, 35.1, 27.8; **FT-IR (thin film, KBr):** 2988, 1735, 1651, 1608, 1478, 1299, 765; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{36}H_{32}NO_4$ 542.2326, found 542.2324.



((*3aS,5aR,8aR,8bS*)-2,2,7,7-tetramethyltetrahydro-*3aH*-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-3a-yl)methyl 4-((*E*)-3-(5-phenyl-3,4-dihydro-2H-pyrrol-2-yl)prop-1-en-1-yl)benzoate (5bb): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 8:1 to 2:1) to give a white solid; 77% yield (84 mg); m.p. 181-182 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.99 (d, *J* = 8.2 Hz, 2H), 7.87 – 7.82 (m, 2H), 7.45 – 7.37 (m, 5H), 6.52 (d, *J* = 15.9 Hz, 1H), 6.48 – 6.42 (m, 1H), 4.68 (d, *J* = 11.8 Hz, 1H), 4.64 (dd, *J* = 7.9, 2.5 Hz, 1H), 4.47 (d, *J* = 2.6 Hz, 1H), 4.40 – 4.34 (m, 1H), 4.31 (d, *J* = 11.8 Hz, 1H), 4.25 (d, *J* = 8.0 Hz, 1H), 3.95 (dd, *J* = 13.0, 1.4 Hz, 1H), 3.80 (d, *J* = 13.0 Hz, 1H), 3.06 – 2.98 (m, 1H), 2.91 (dt, *J* = 16.2, 8.2 Hz, 1H), 2.81 – 2.74 (m, 1H), 2.50 (dt, *J* = 14.3, 7.3 Hz, 1H), 2.26 – 2.18 (m, 1H), 1.76 – 1.68 (m, 1H), 1.54 (s, 3H), 1.47 (s, 3H), 1.37 (s, 3H), 1.34 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.7, 165.8, 142.3, 134.5, 131.1, 130.8, 130.5, 130.1, 128.4, 128.1, 127.7, 125.9, 109.2, 108.8, 101.7, 72.7, 70.8, 70.5, 70.1, 65.1, 61.3, 40.0, 35.1, 28.0, 26.5, 25.9, 25.5, 24.0; **FT-IR (thin film, KBr):** 2924, 1721, 1605, 1461, 1376, 1070, 760; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{32}H_{38}NO_7$ 548.2643, found 548.2644.

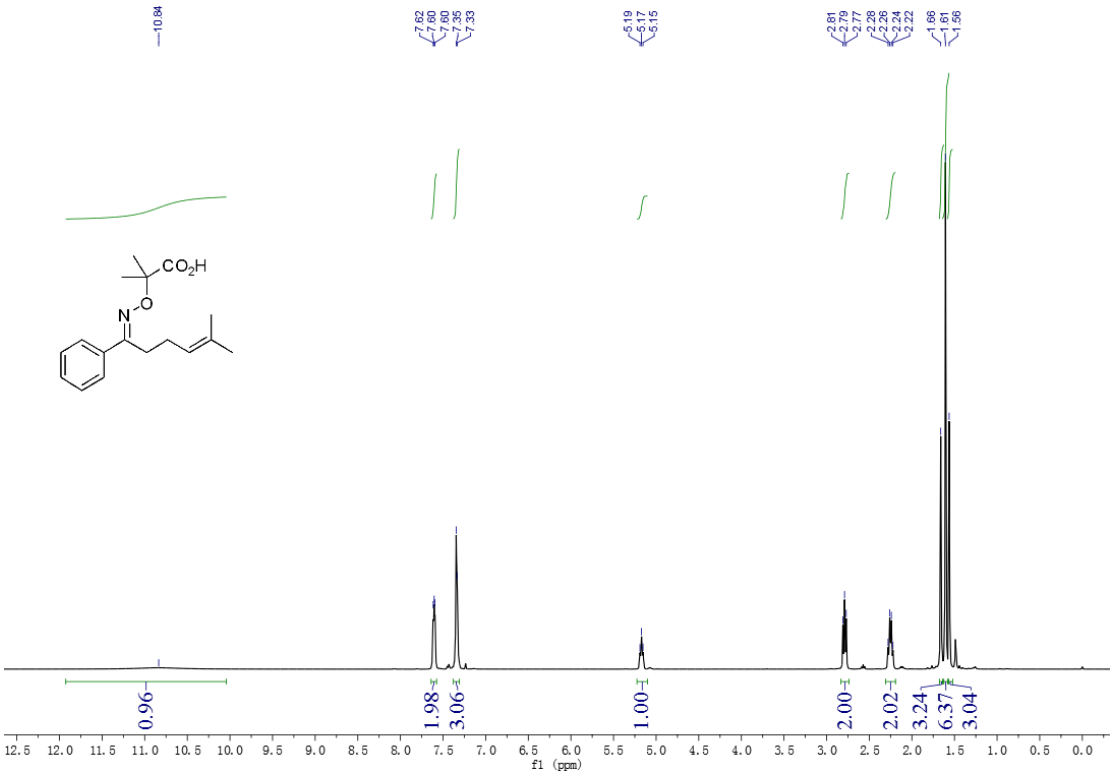


(*2S,8S,9S,10R,13R,14S,17R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-*1H*-cyclopenta[a]phenanthren-2-yl 4-((*E*)-3-(5-phenyl-3,4-dihydro-*2H*-pyrrol-2-yl)prop-1-en-1-yl)benzoate (5bc): Purification by flash column chromatography on silica gel (petroleum ether/EtOAc = 4:1 to 2:1) to give a brown solid; 36% yield (48 mg); m.p. 196-197 ℃; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.87 – 7.83 (m, 2H), 7.45 – 7.37 (m, 5H), 6.53 (d, *J* = 15.9 Hz, 1H), 6.47 – 6.40 (m, 1H), 5.42 (d, *J* = 4.1 Hz, 1H), 4.88 – 4.81 (m, 1H), 4.41 – 4.36 (m, 1H), 3.06 – 3.00 (m, 1H), 2.92 (dt, *J* = 18.2, 8.6 Hz, 1H), 2.81 – 2.76 (m, 1H), 2.50 (dt, *J* = 14.7, 7.5 Hz, 1H), 2.46 (d, *J* = 7.8 Hz, 2H), 2.25 – 2.18 (m, 1H), 2.04 – 1.97 (m, 3H), 1.93 – 1.89 (m, 1H), 1.86 – 1.81 (m, 1H), 1.77 – 1.70 (m, 2H), 1.62 – 1.55 (m, 2H), 1.55 – 1.51

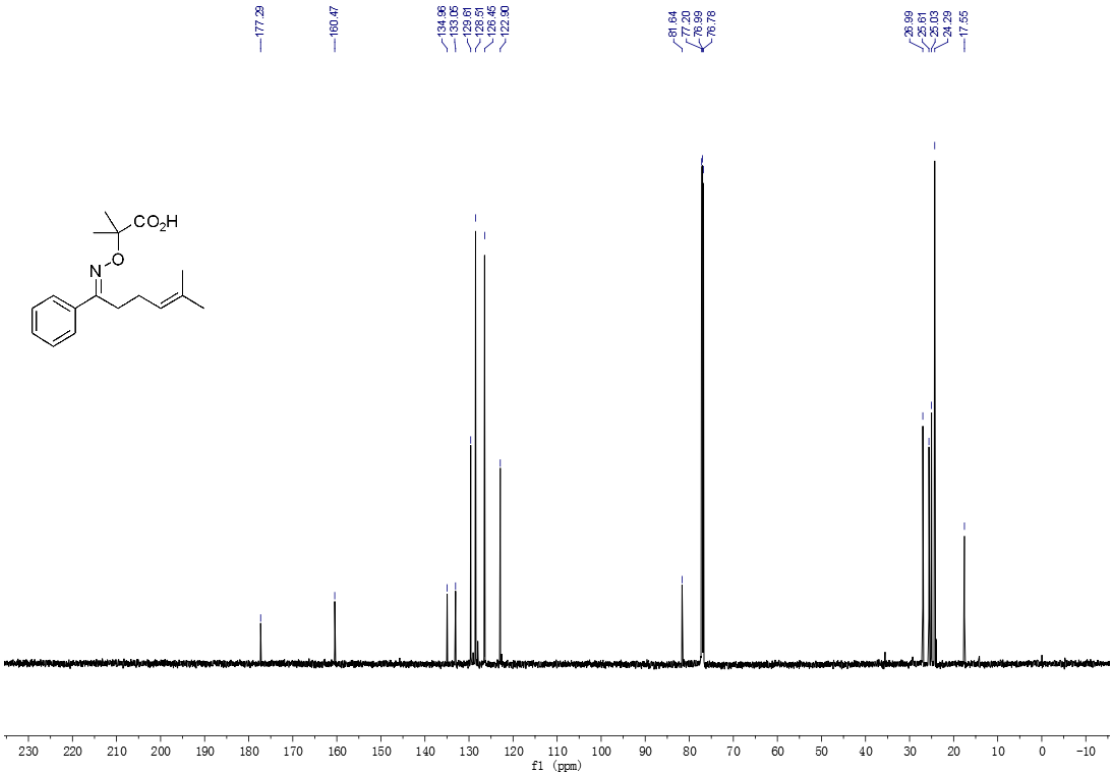
(m, 2H), 1.49 – 1.43 (m, 2H), 1.41 – 1.30 (m, 4H), 1.28 – 1.23 (m, 2H), 1.19 – 1.10 (m, 5H), 1.07 (s, 3H), 1.04 – 0.97 (m, 3H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.88 (d, *J* = 2.7 Hz, 3H), 0.86 (d, *J* = 2.6 Hz, 3H), 0.69 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 172.7, 165.8, 141.9, 139.7, 134.5, 131.2, 130.43, 130.38, 129.8, 129.2, 128.4, 127.7, 125.8, 122.7, 74.4, 72.7, 56.7, 56.1, 50.0, 42.3, 40.0, 39.7, 39.5, 38.2, 37.0, 36.6, 36.2, 35.8, 35.1, 31.92, 31.87, 28.2, 27.99, 27.97, 27.9, 24.3, 23.8, 22.8, 22.5, 21.0, 19.4, 18.7, 11.9; **FT-IR (thin film, KBr):** 2879, 1710, 1615, 1468, 1274, 1120, 856; **HRMS** (ESI) $[M + H]^+$ Calculated for $C_{47}H_{64}NO_2$ 674.4932, found 674.4933.

8. NMR spectra for the substrates and products

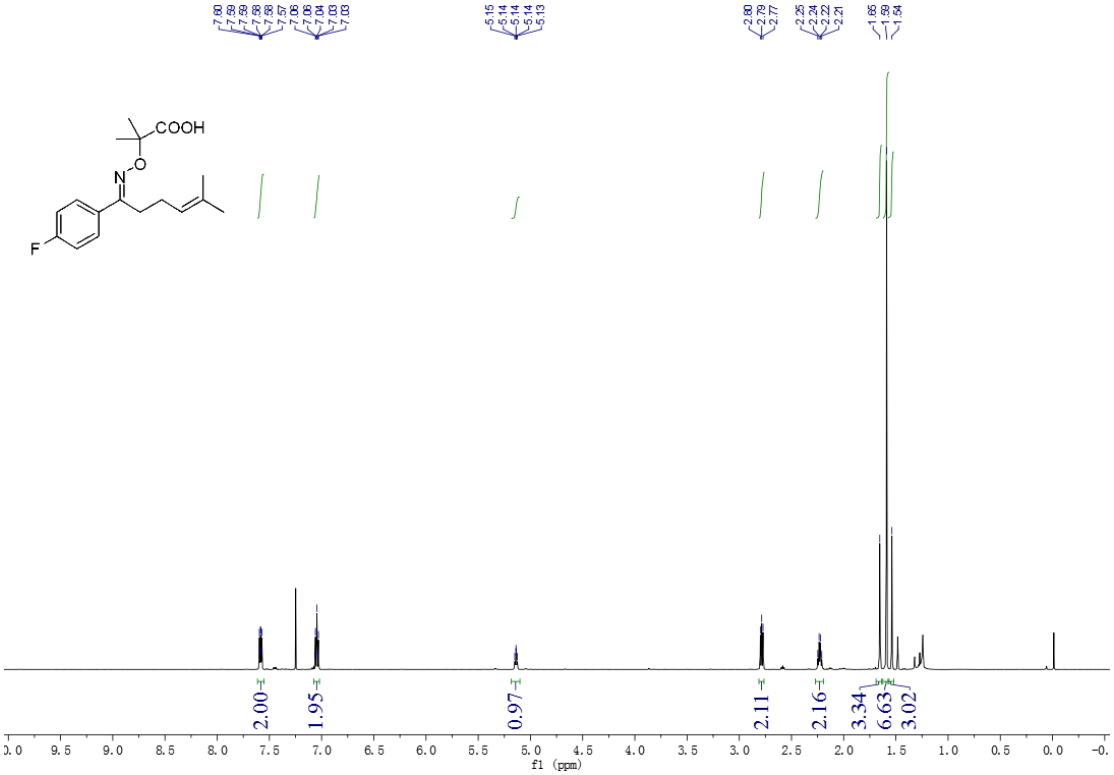
^{1}H NMR of **1aa**



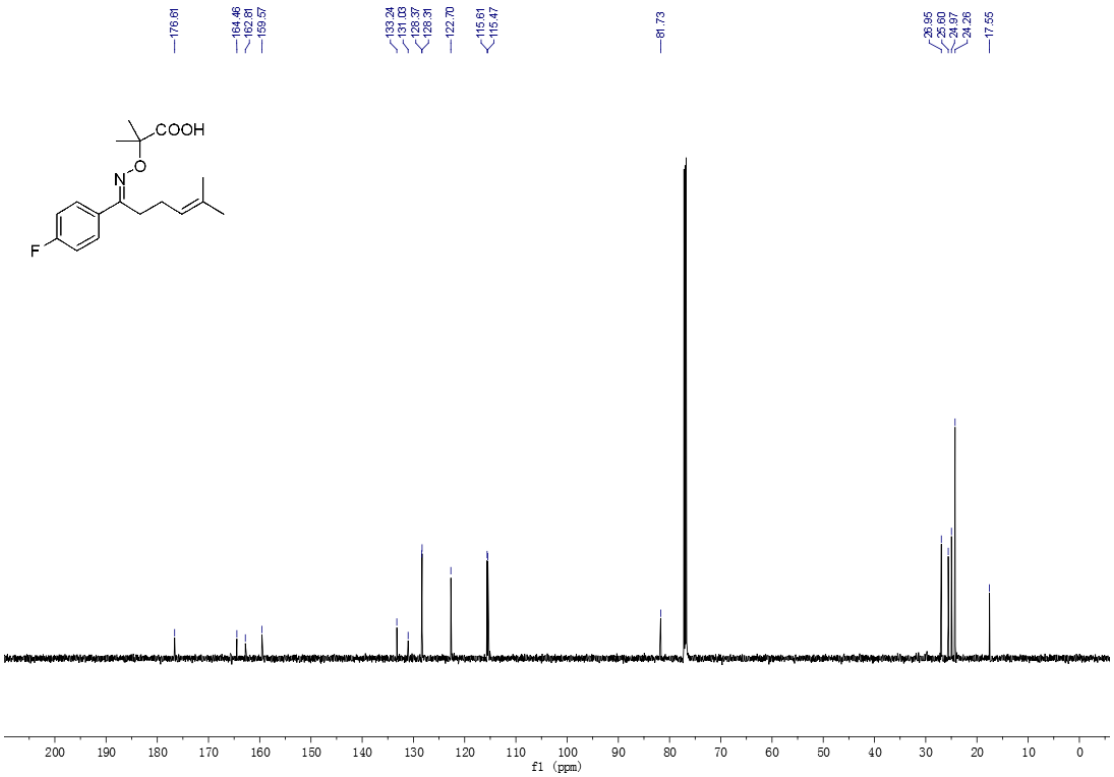
^{13}C NMR of **1aa**



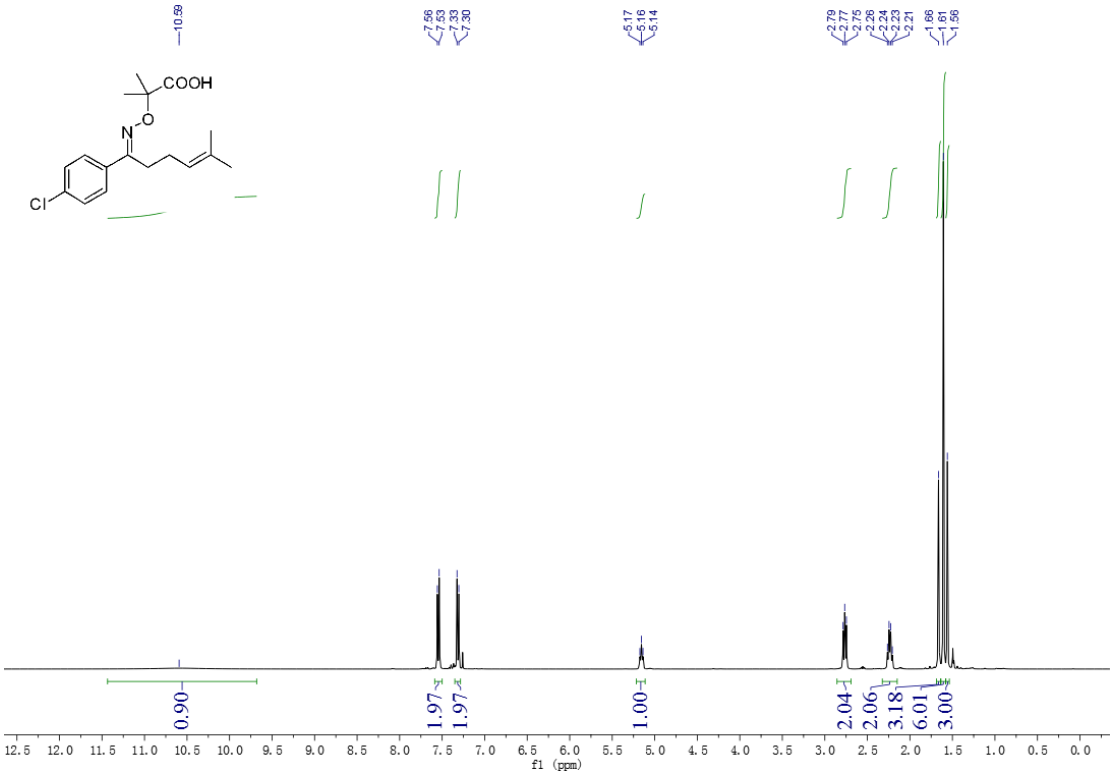
^{1}H NMR of **1ab**



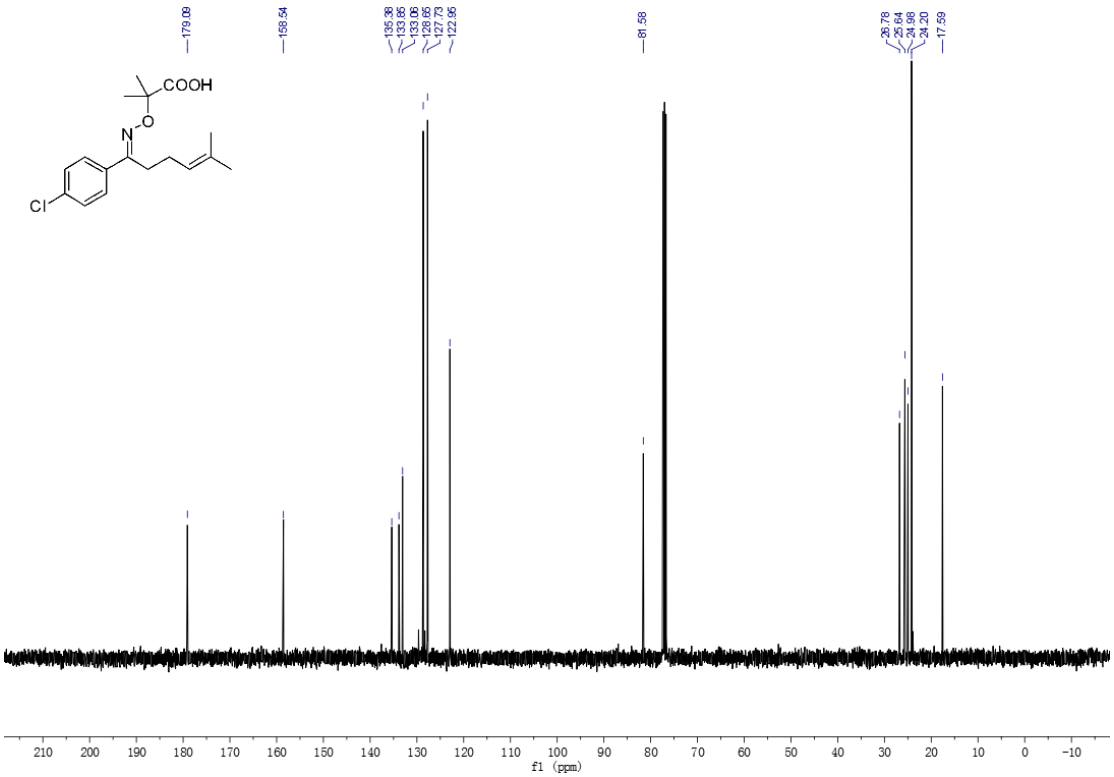
^{13}C NMR of **1ab**



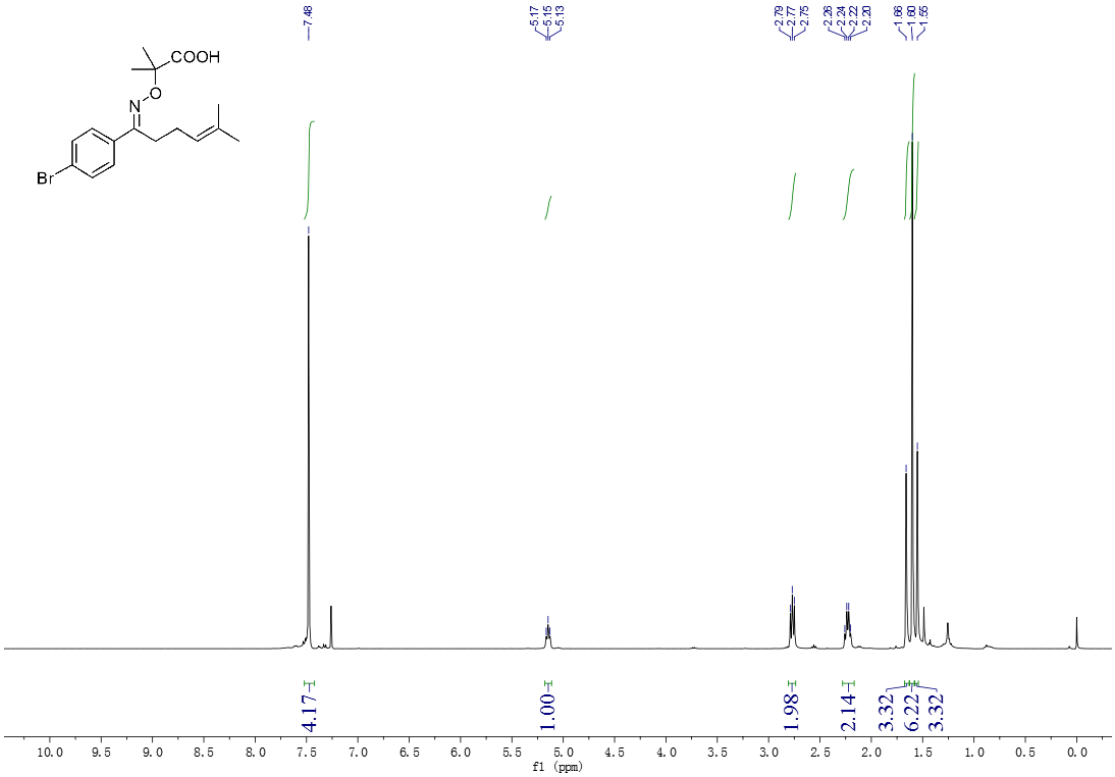
^{1}H NMR of **1ac**



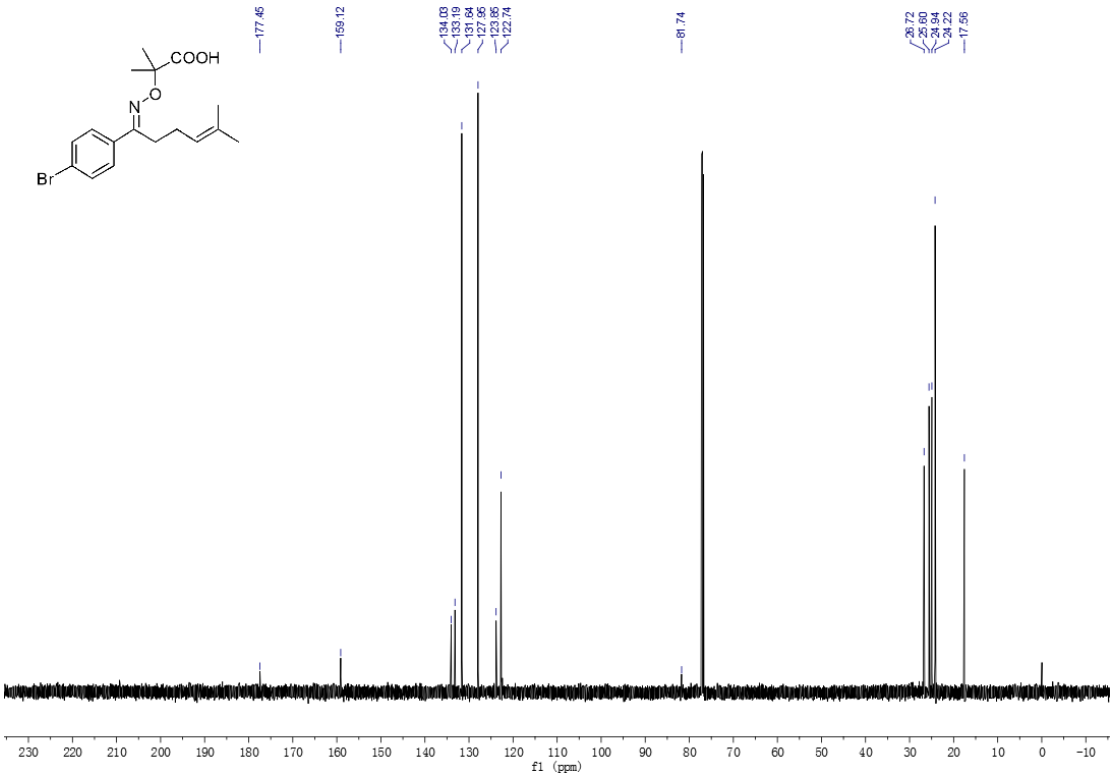
^{13}C NMR of **1ac**



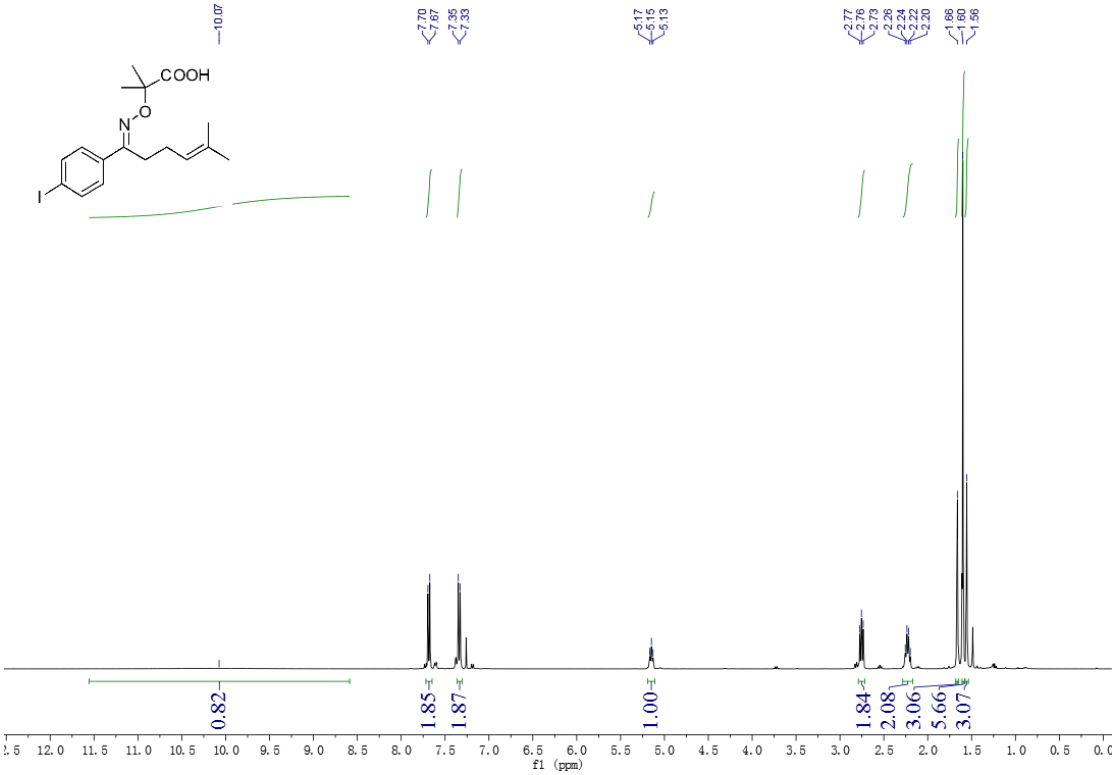
^{1}H NMR of **1ad**



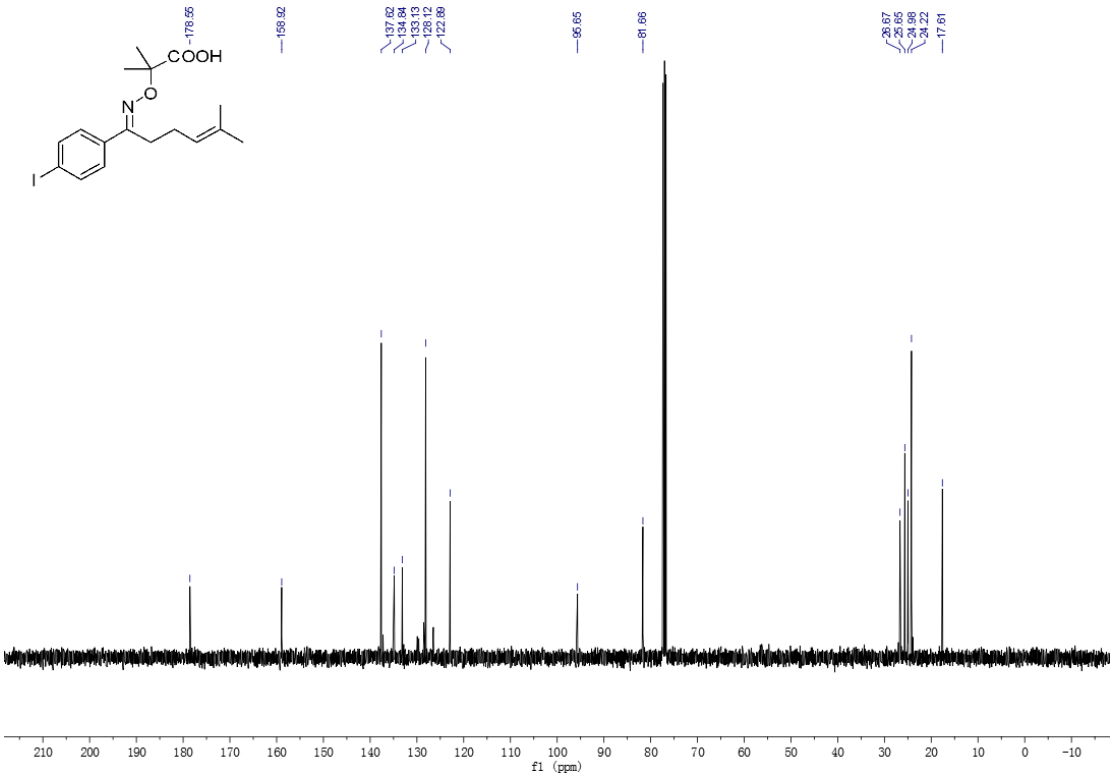
^{13}C NMR of **1ad**



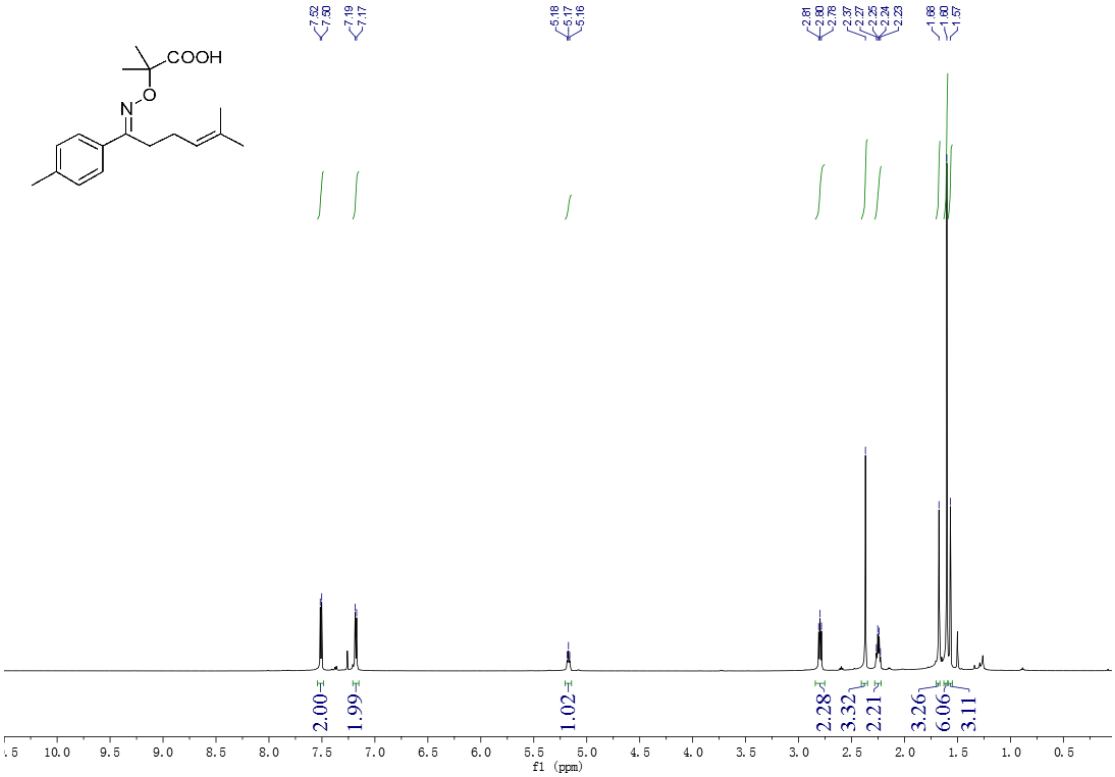
^{1}H NMR of **1ae**



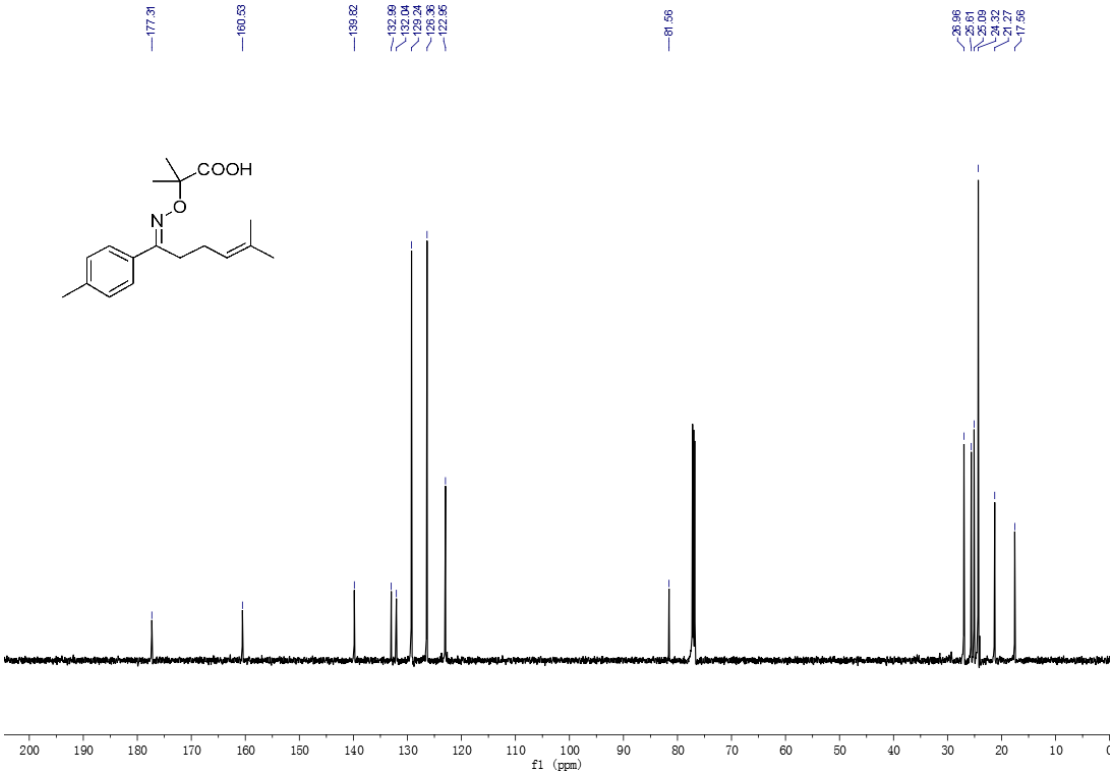
^{13}C NMR of **1ae**



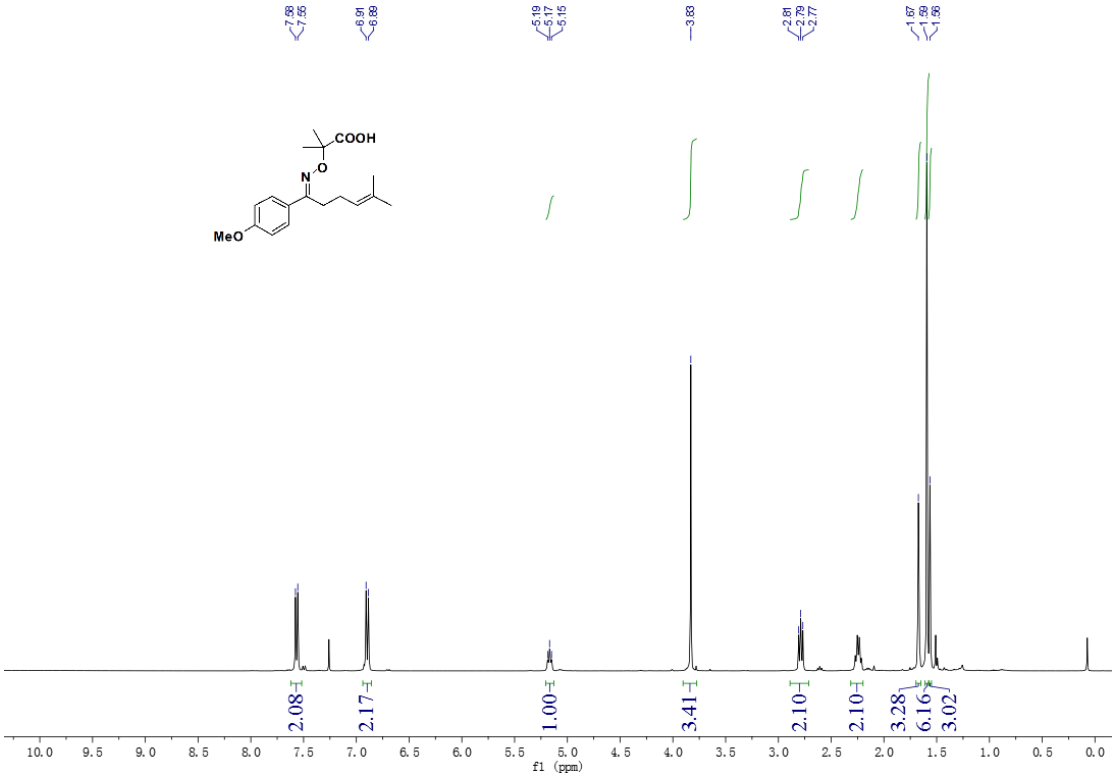
^{1}H NMR of **1af**



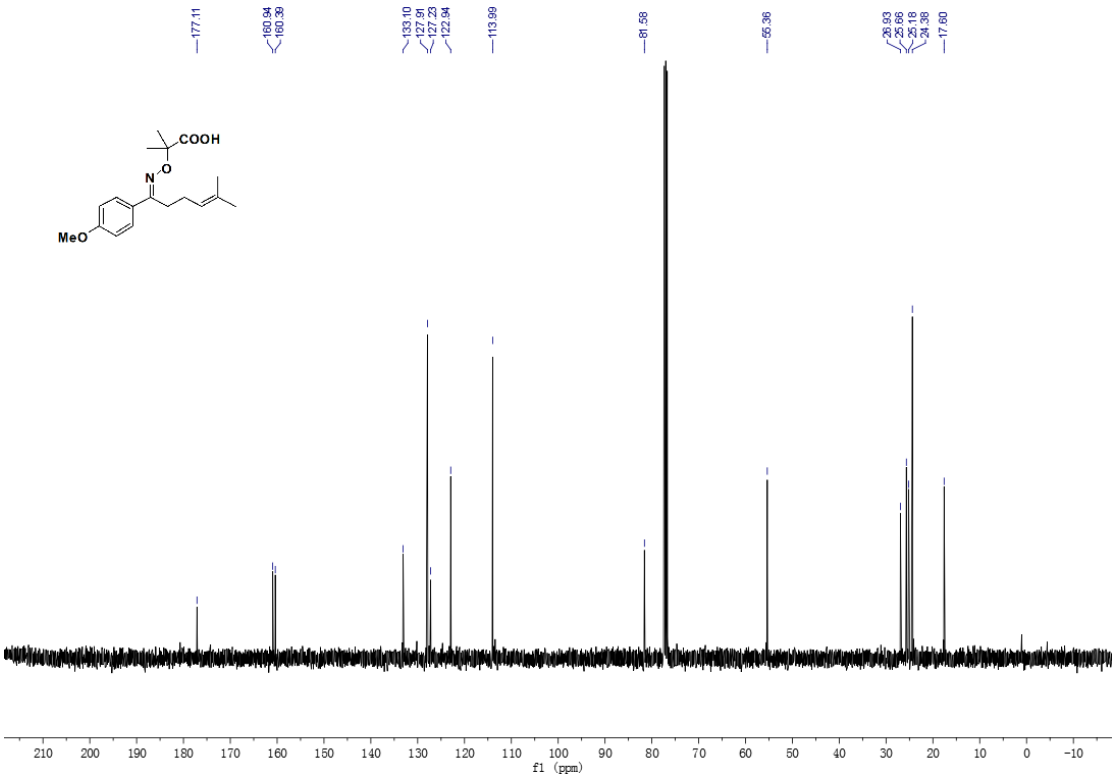
^{13}C NMR of **1af**



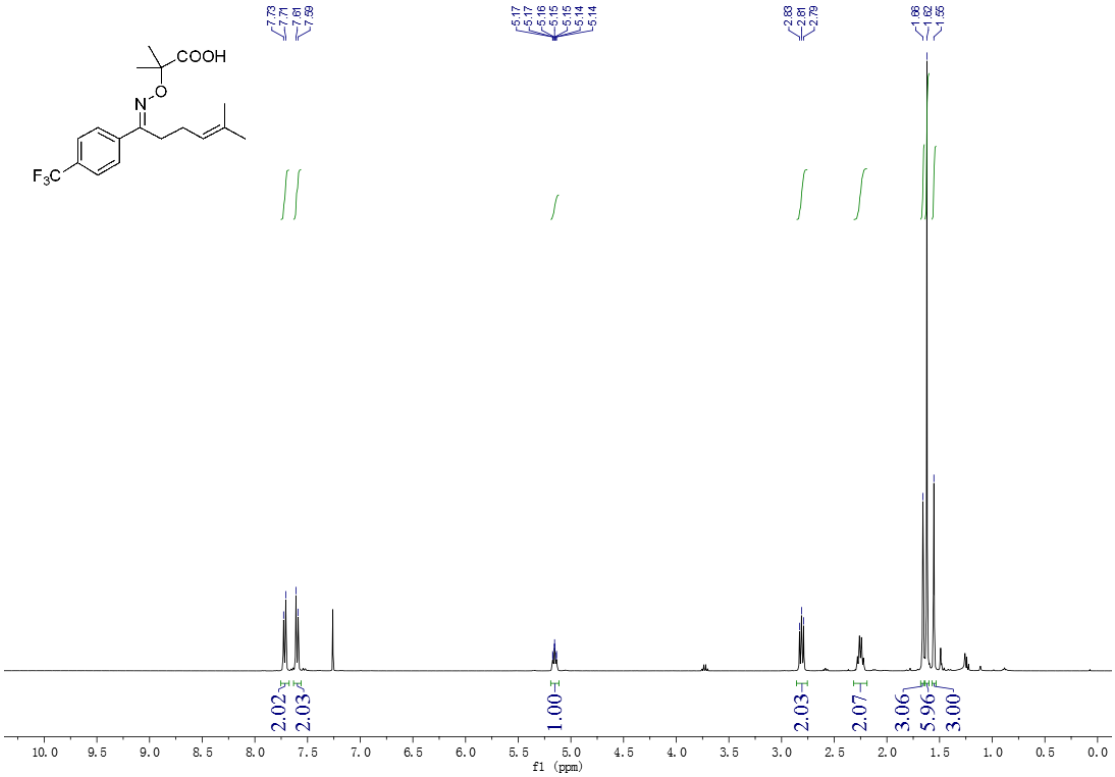
^{1}H NMR of **1ag**



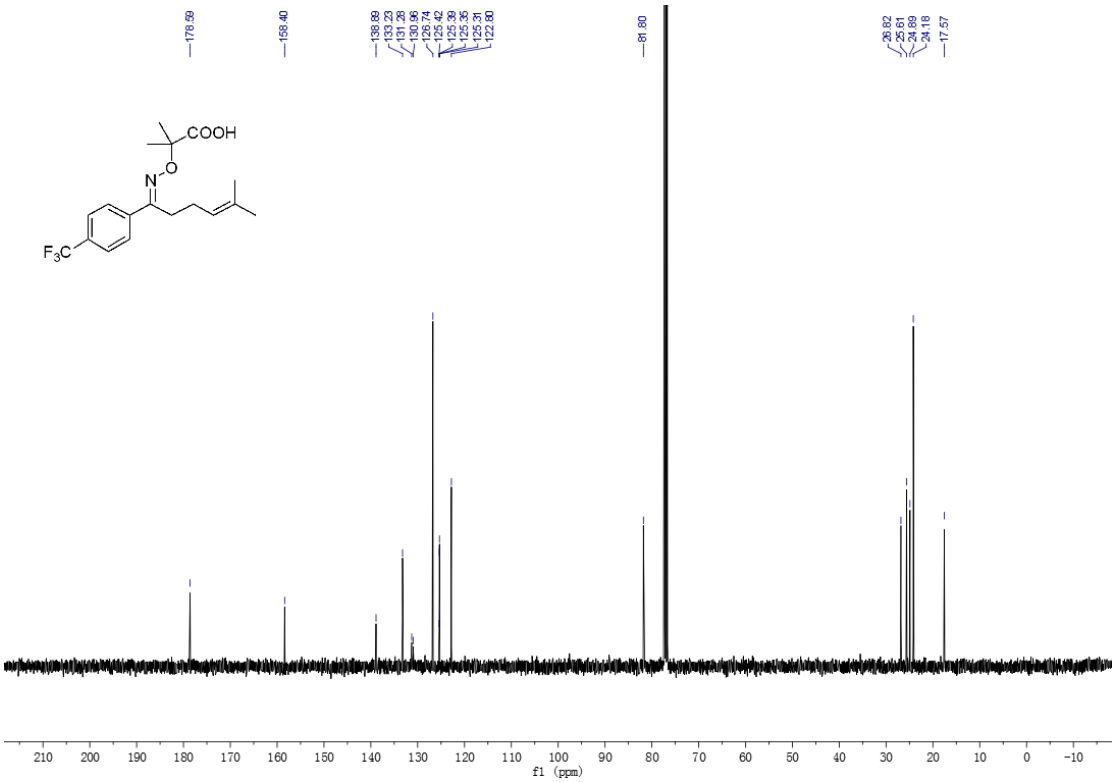
^{13}C NMR of **1ag**



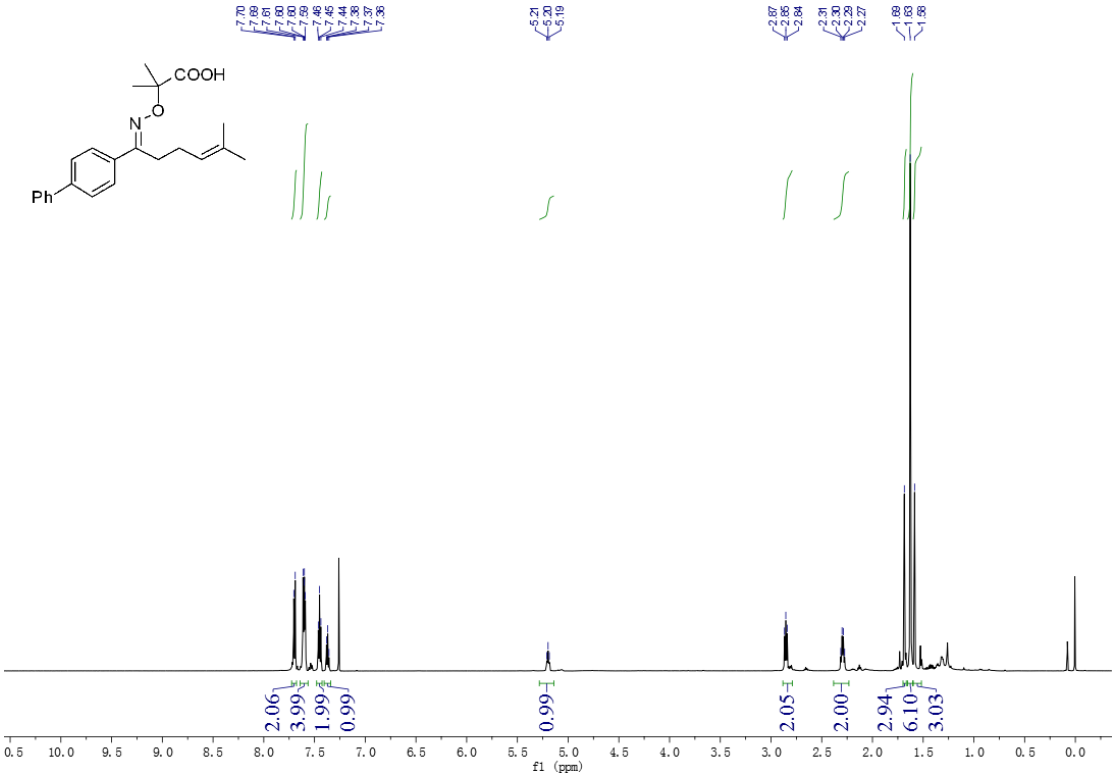
^{1}H NMR of **1ah**



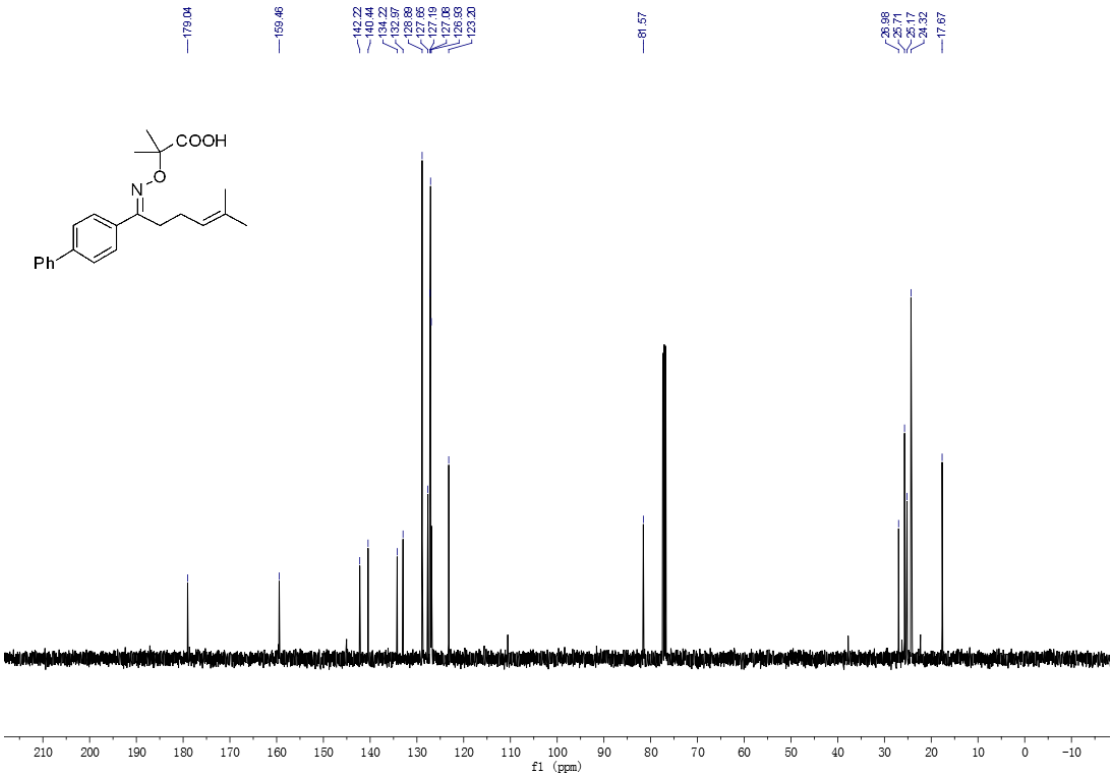
^{13}C NMR of **1ah**



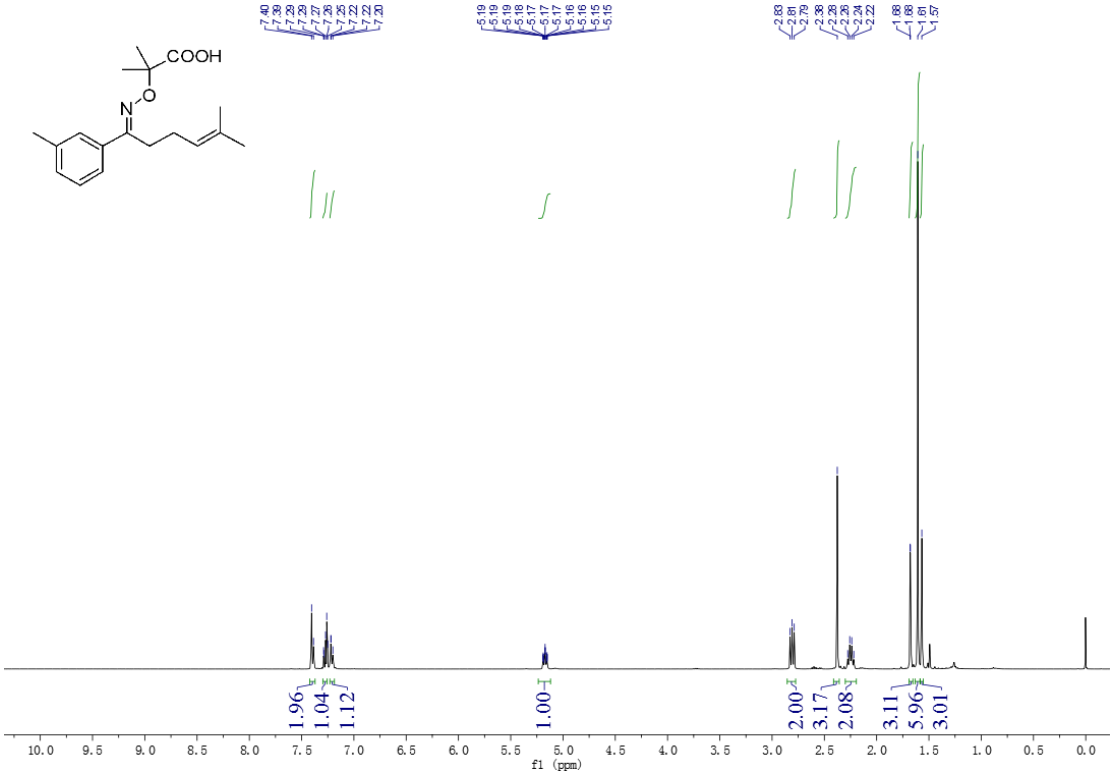
^{1}H NMR of **1ai**



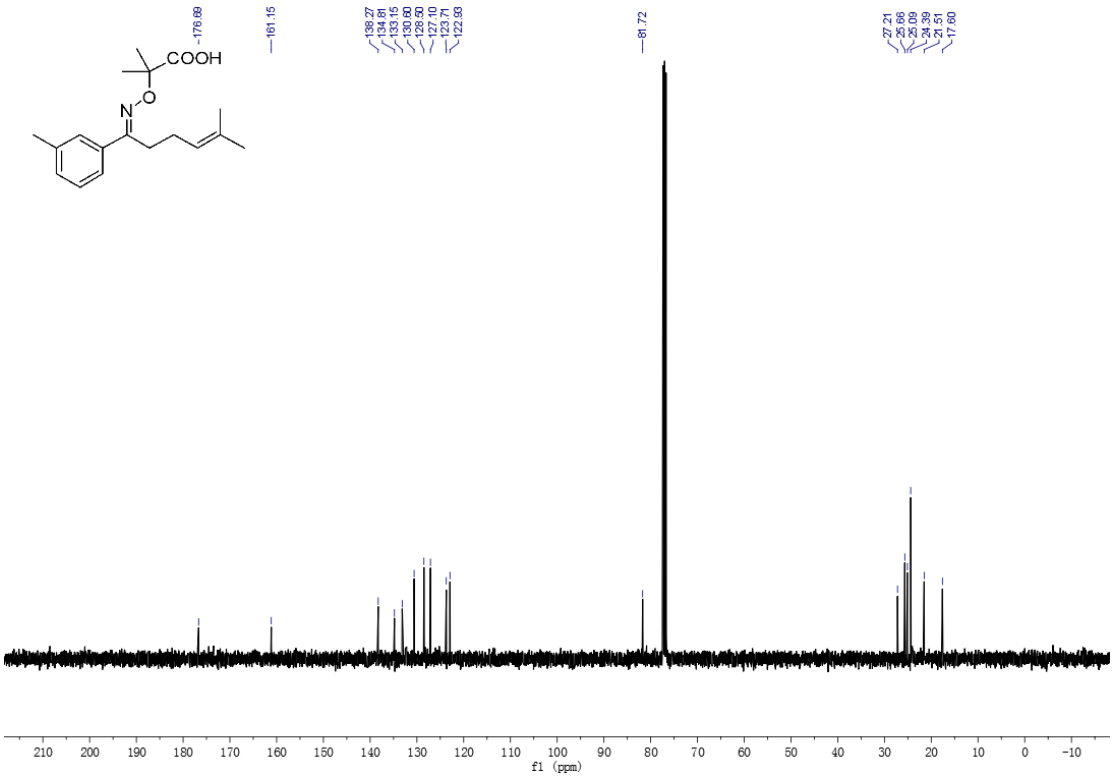
^{13}C NMR of **1ai**



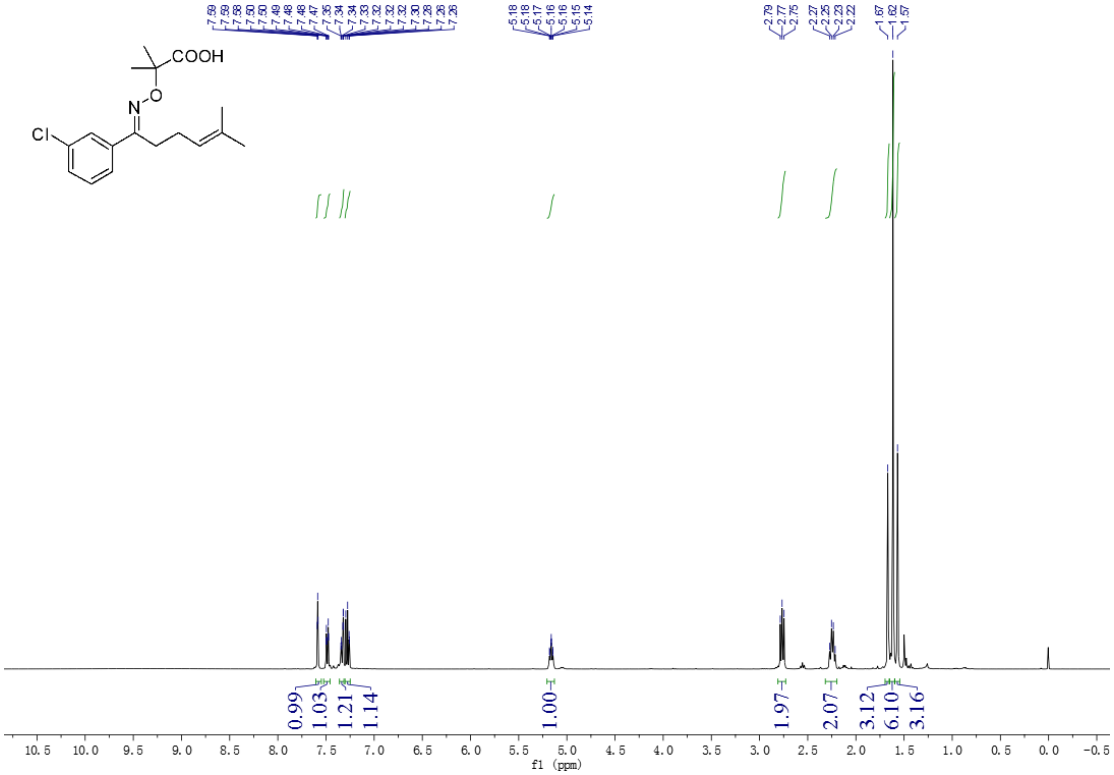
^{1}H NMR of **1aj**



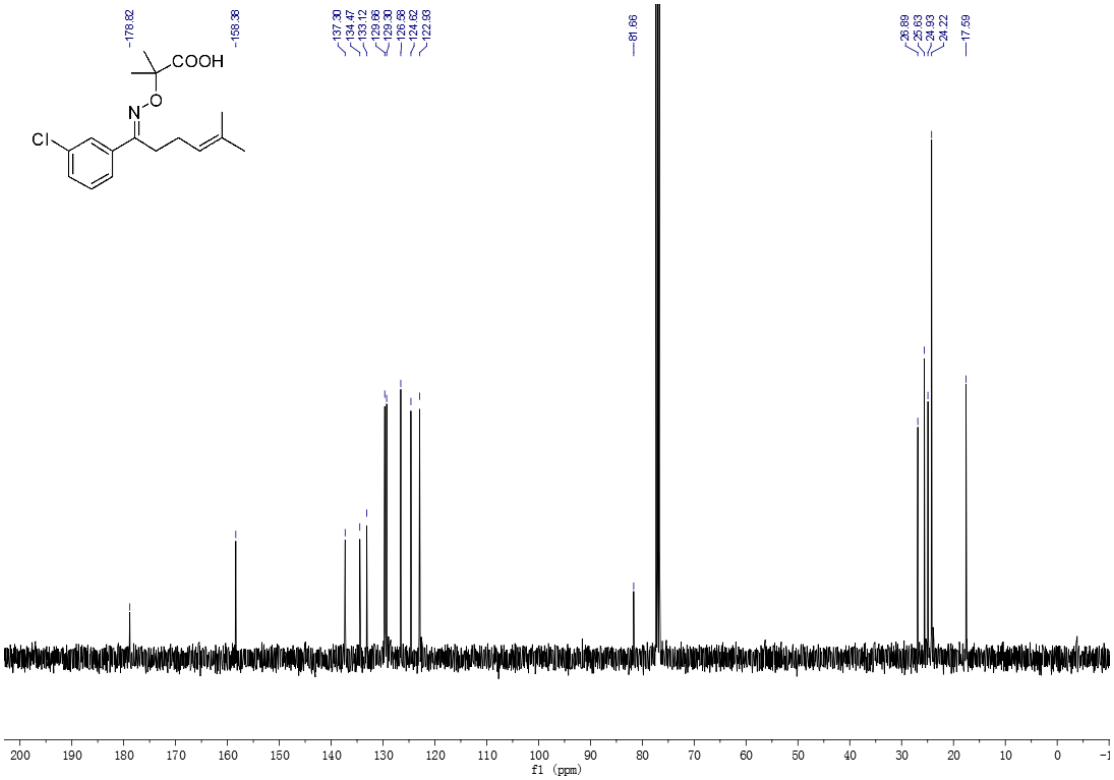
^{13}C NMR of **1aj**



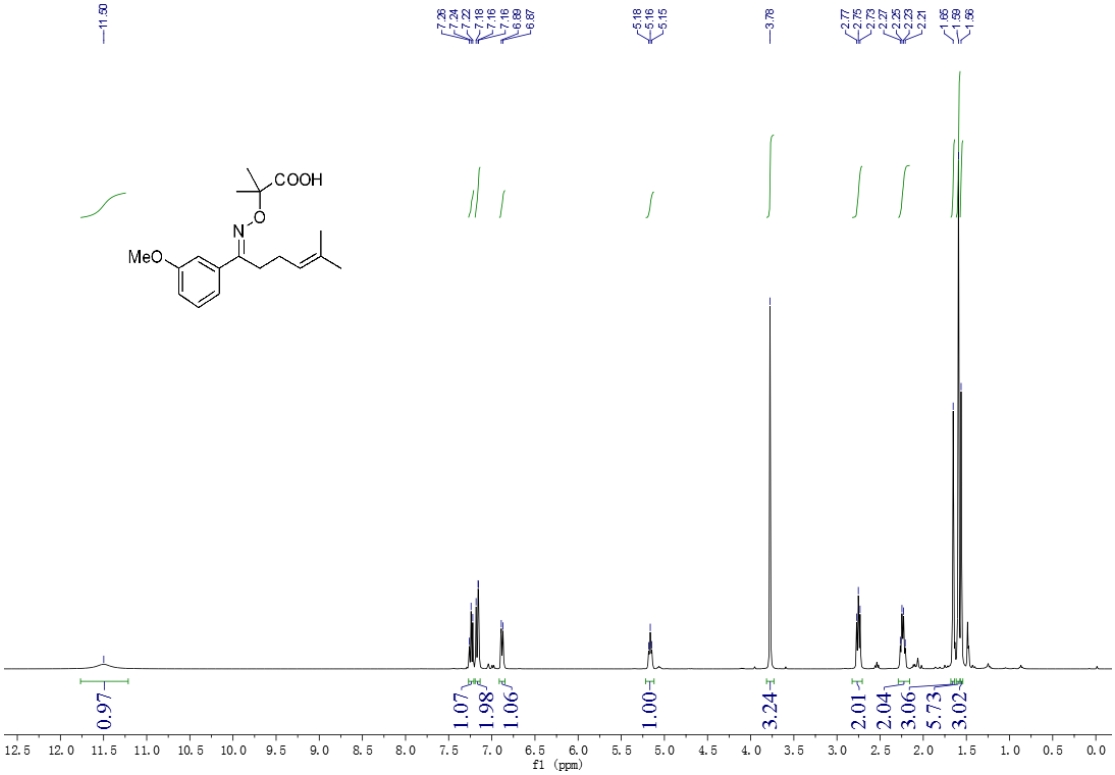
^{1}H NMR of **1ak**



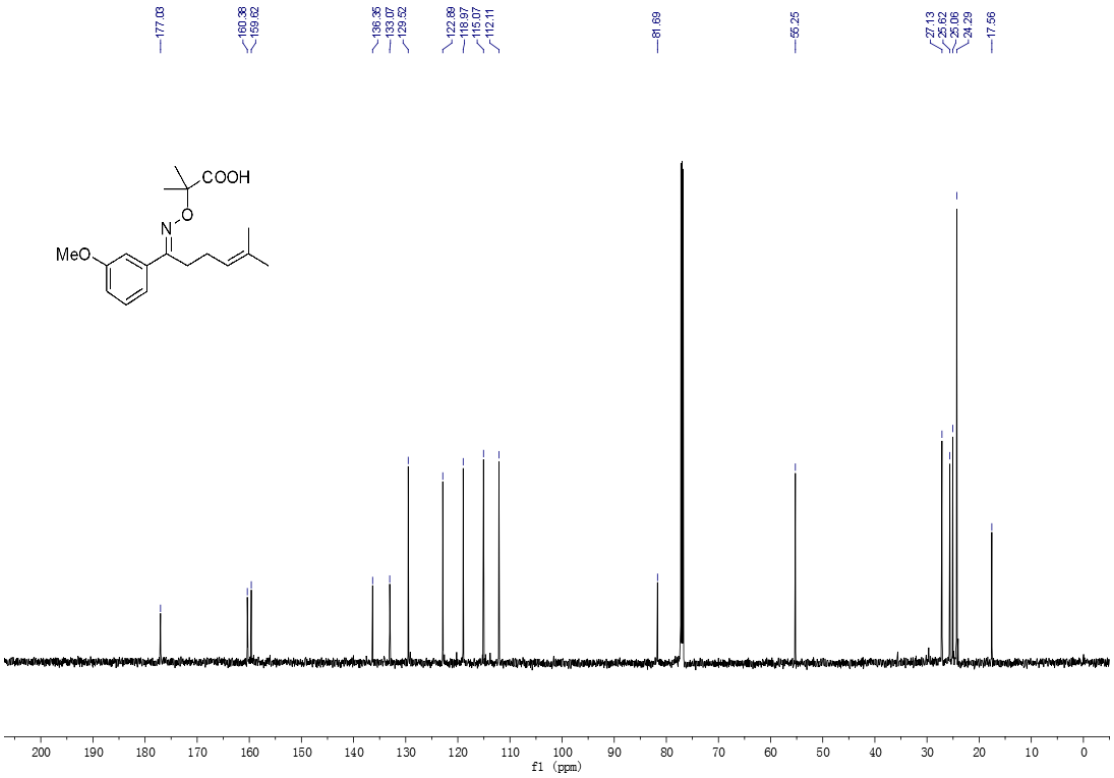
^{13}C NMR of **1ak**



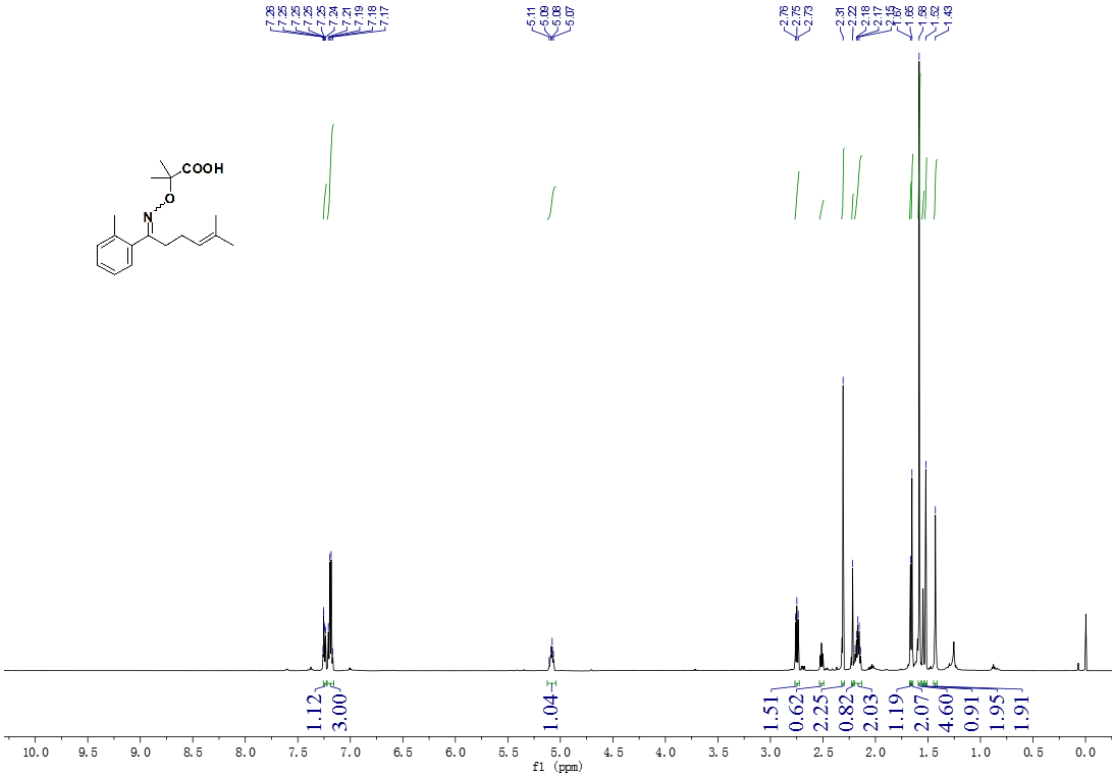
^{1}H NMR of **1al**



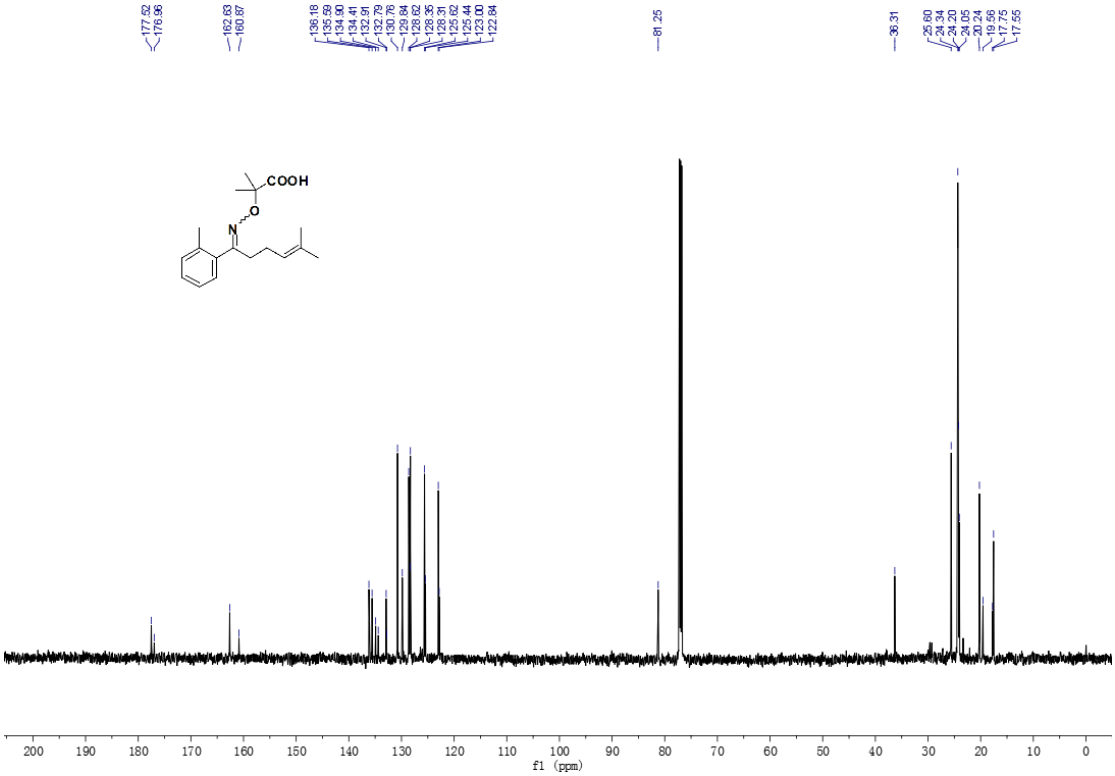
^{13}C NMR of **1al**



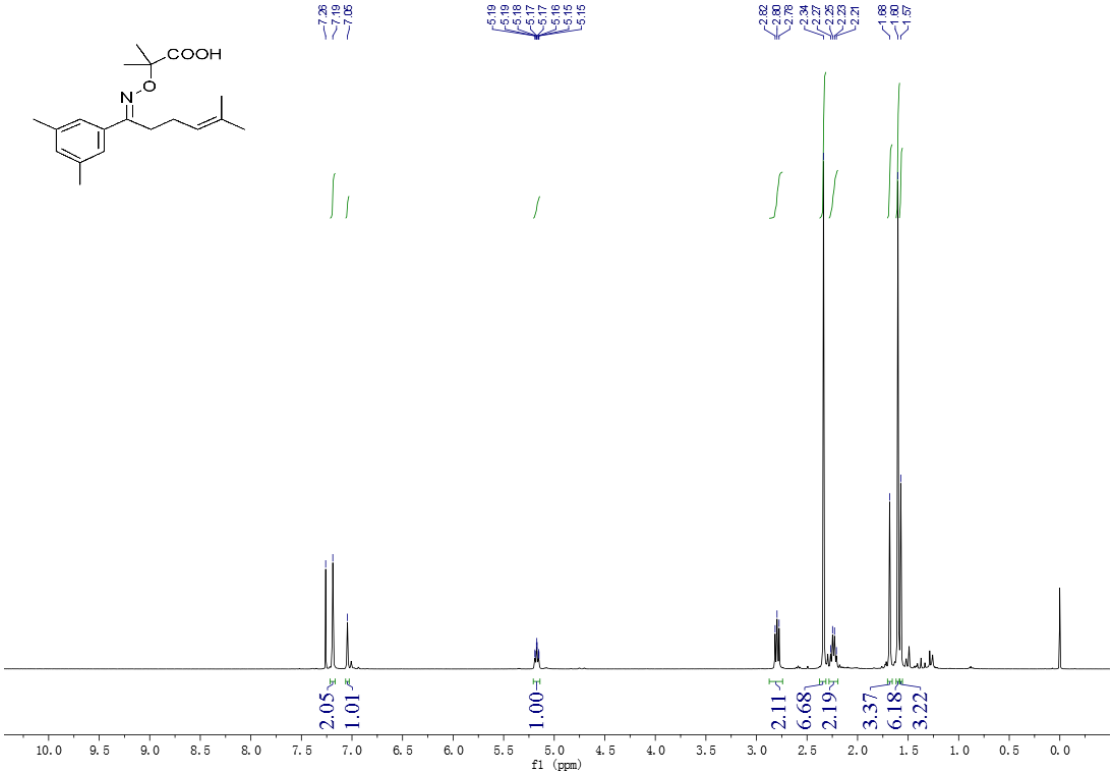
^{1}H NMR of **1am**



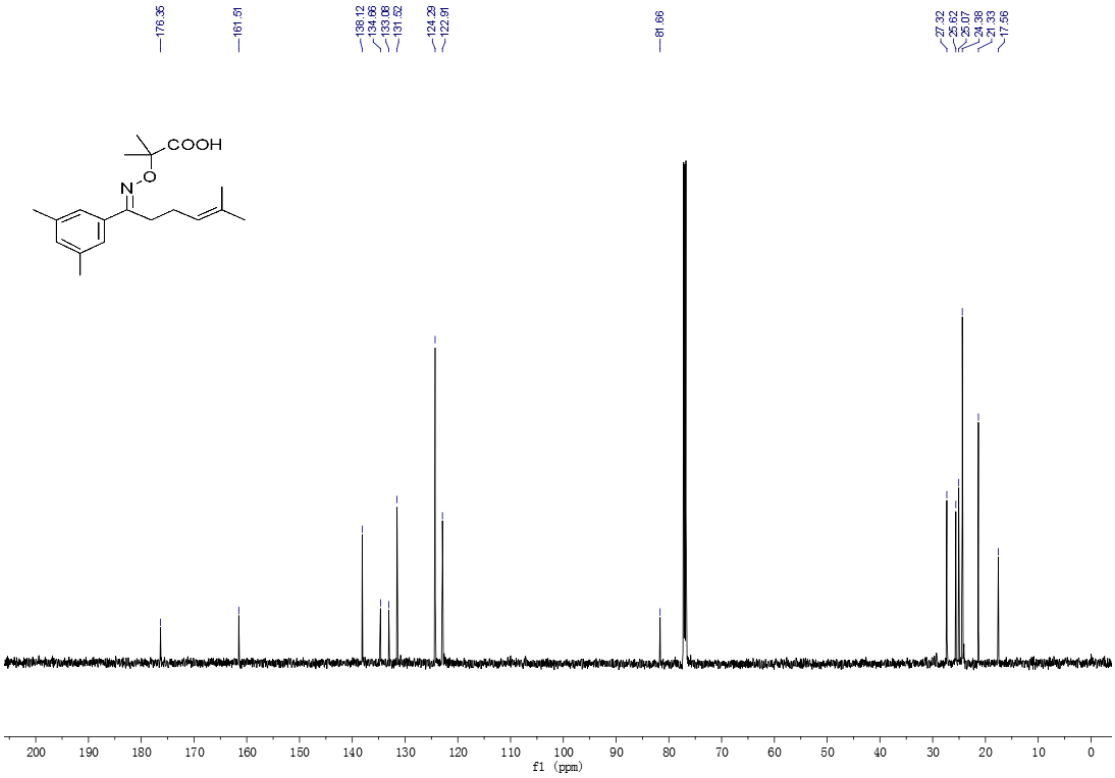
^{13}C NMR of **1am**



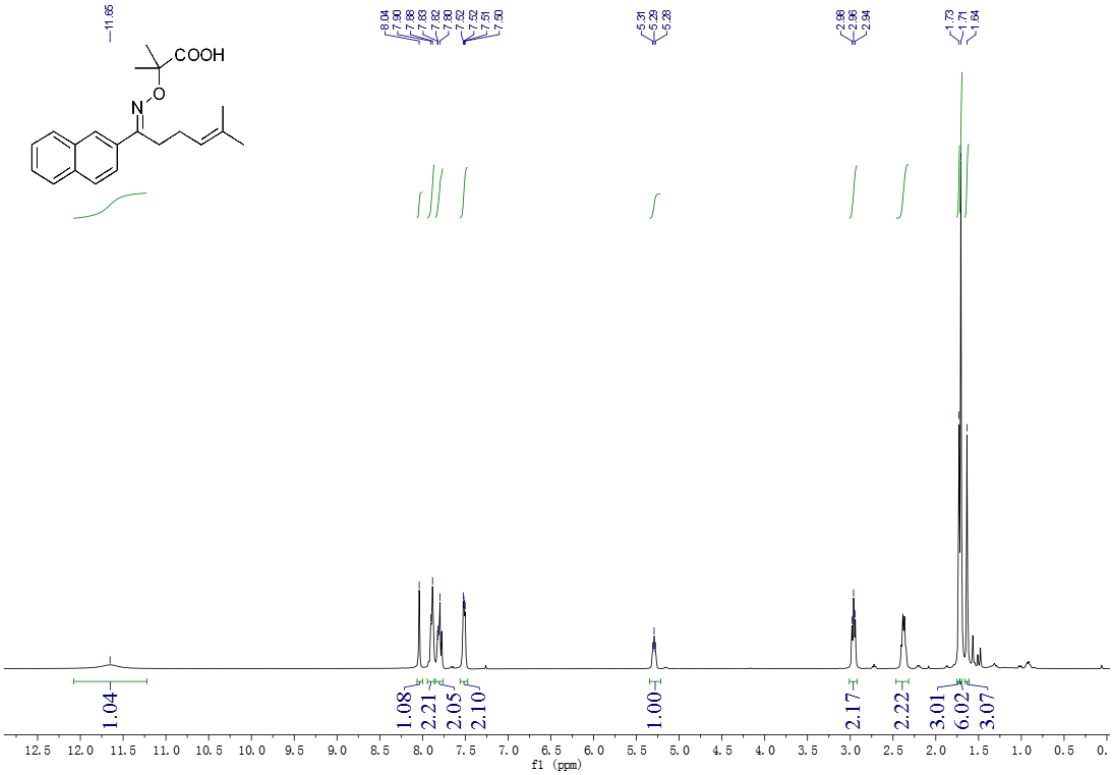
^{1}H NMR of **1an**



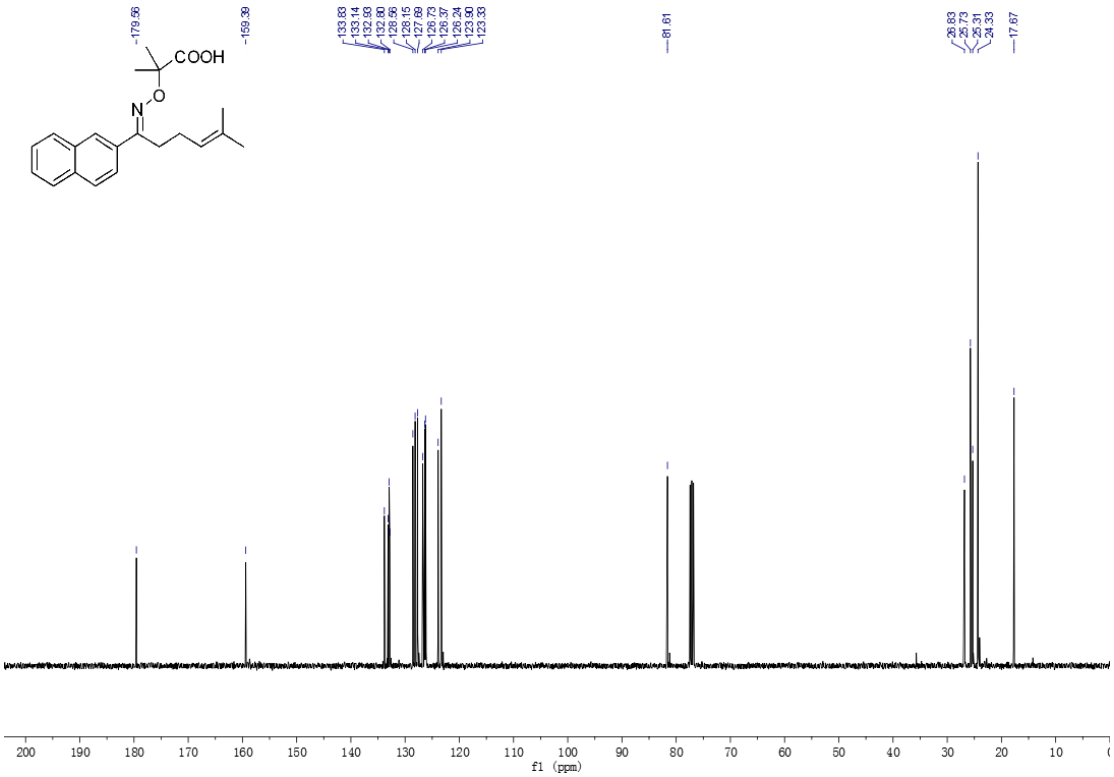
^{13}C NMR of **1an**



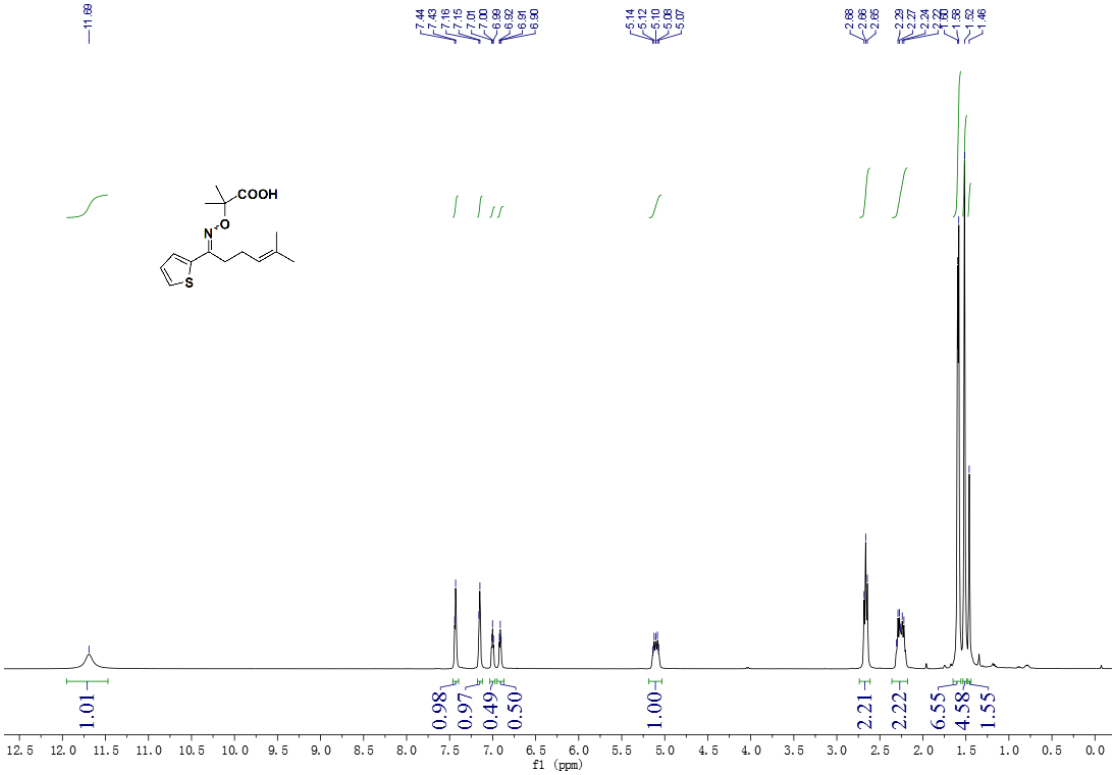
^{1}H NMR of **1ao**



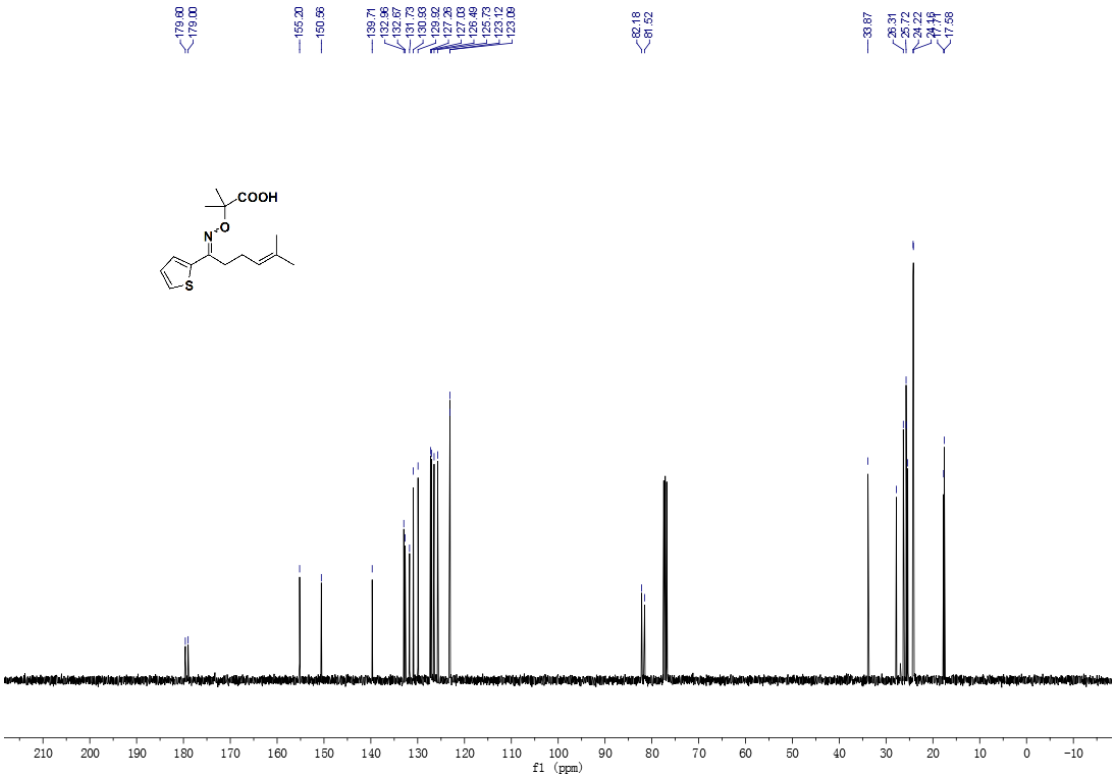
^{13}C NMR of **1ao**



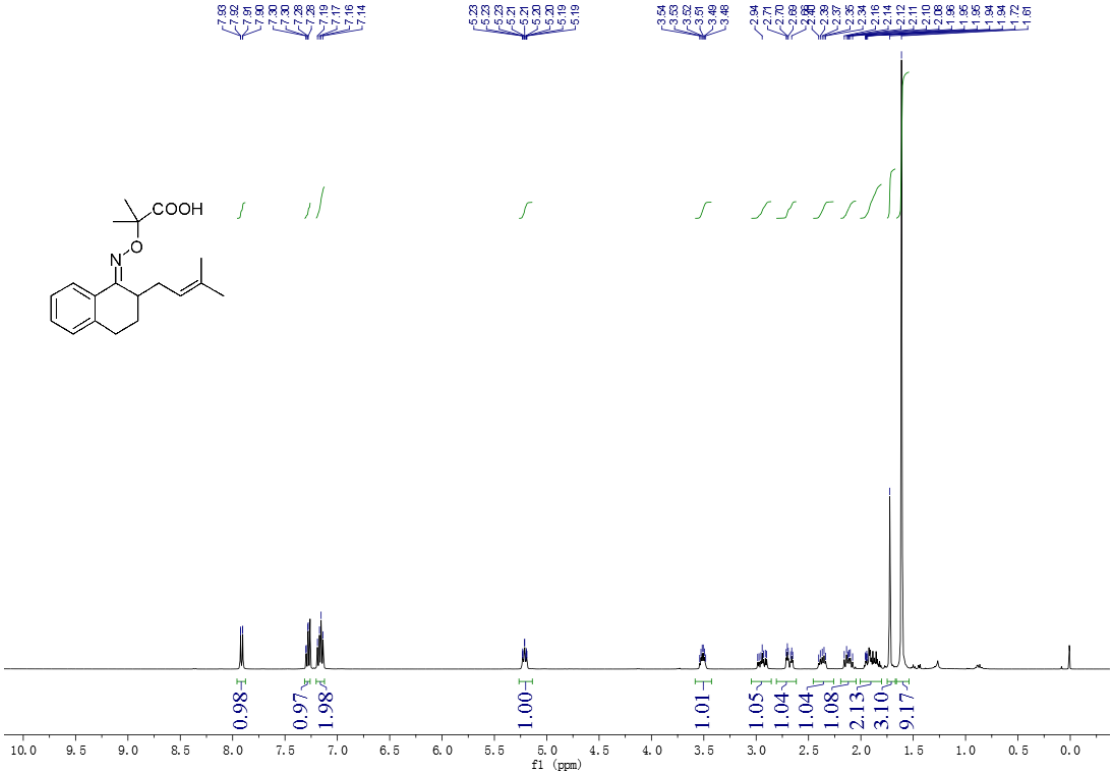
^{1}H NMR of **1ap**



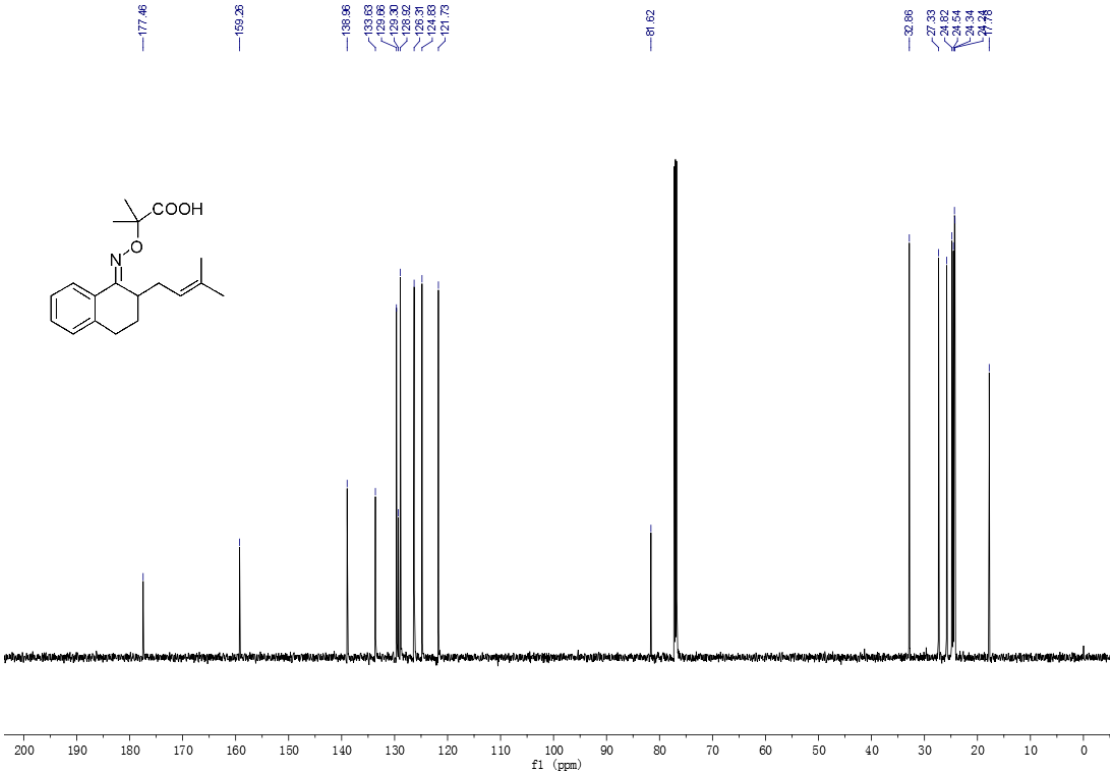
^{13}C NMR of **1ap**



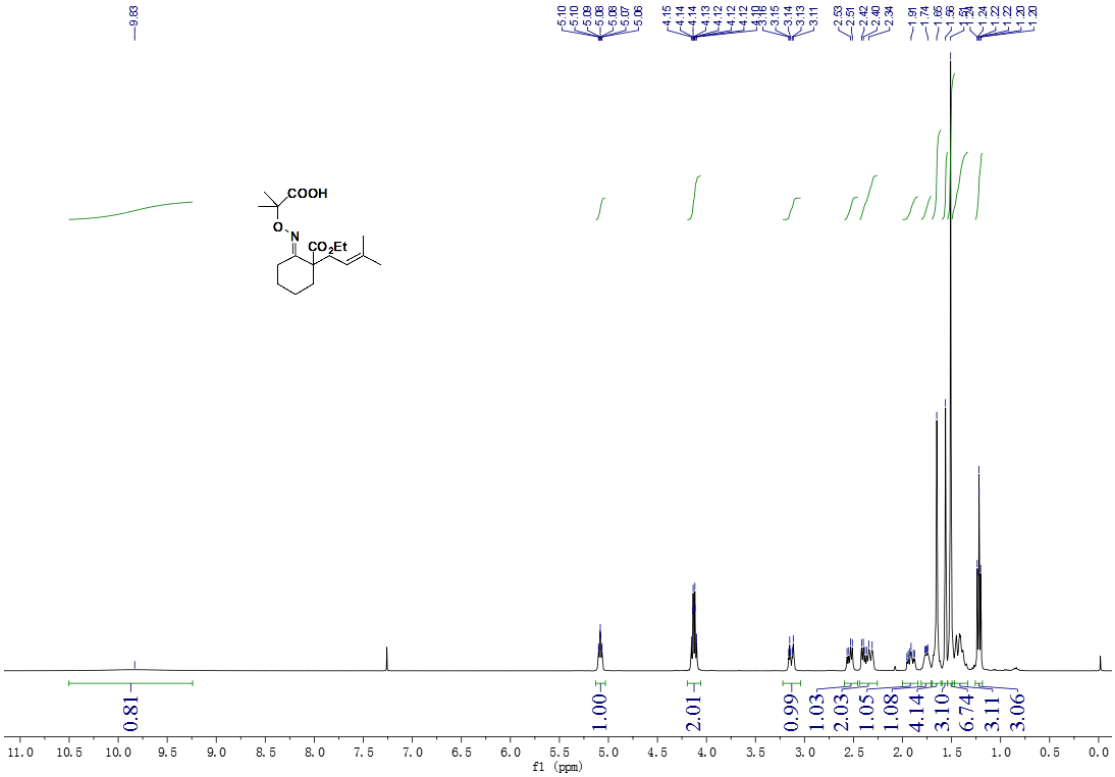
^{1}H NMR of **1aq**



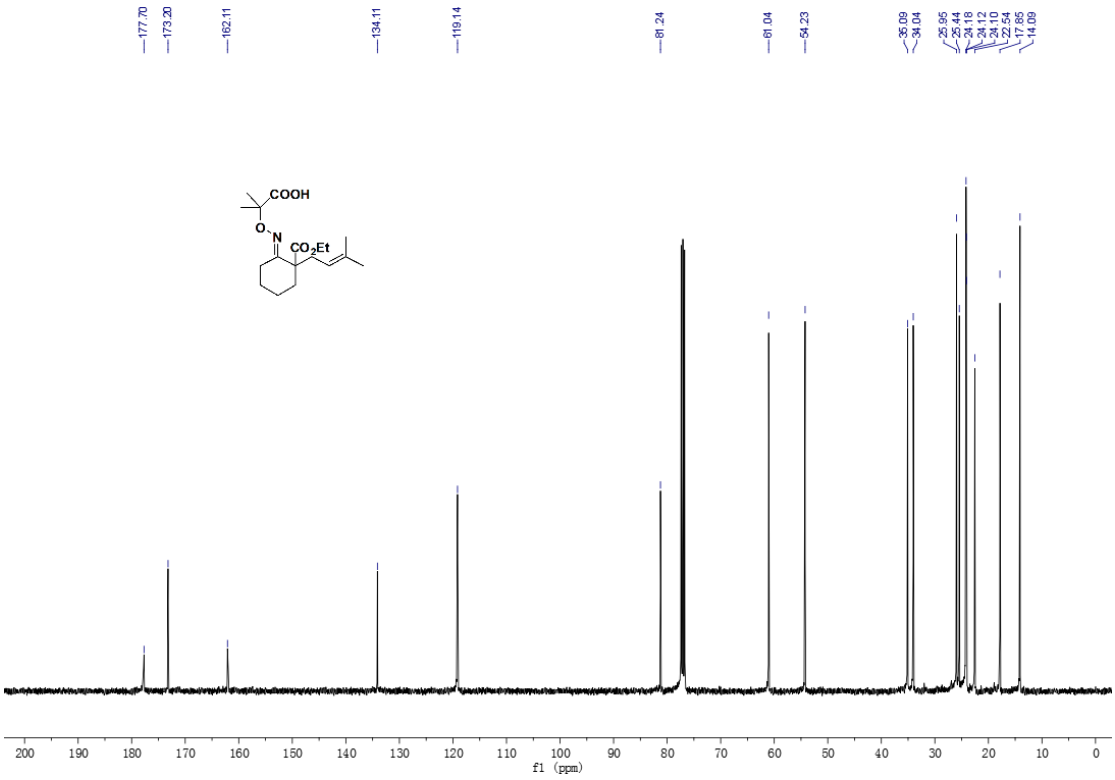
^{13}C NMR of **1aq**



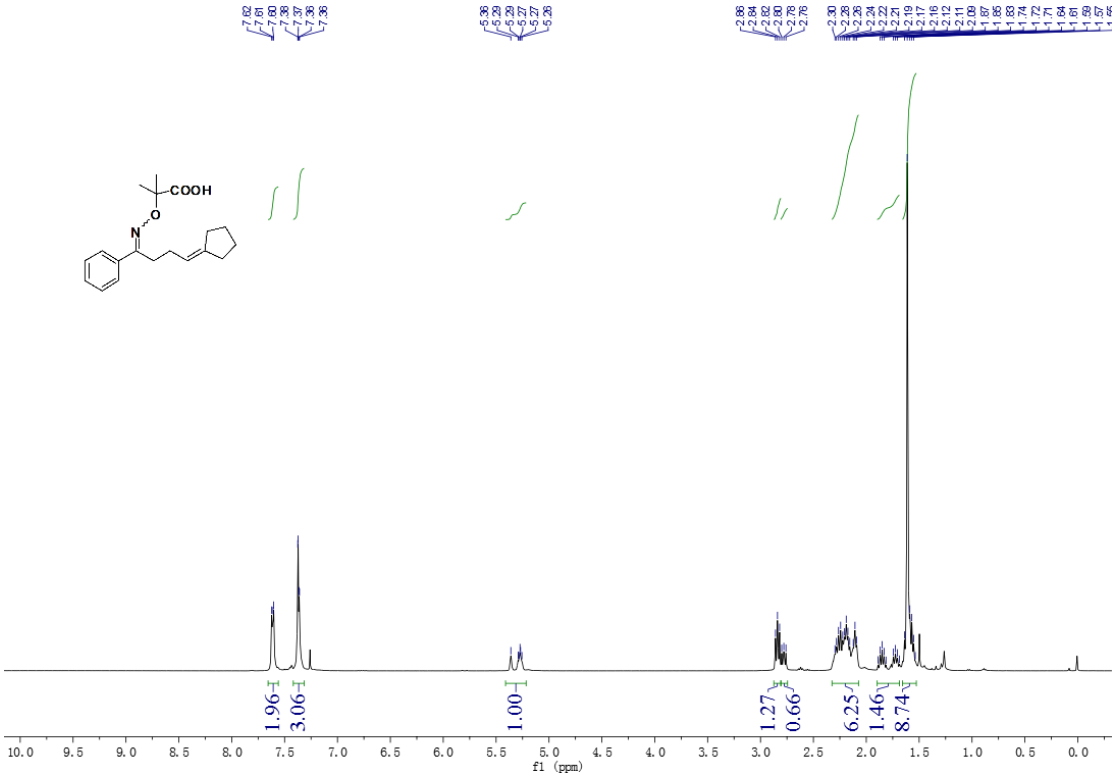
^{1}H NMR of **1ar**



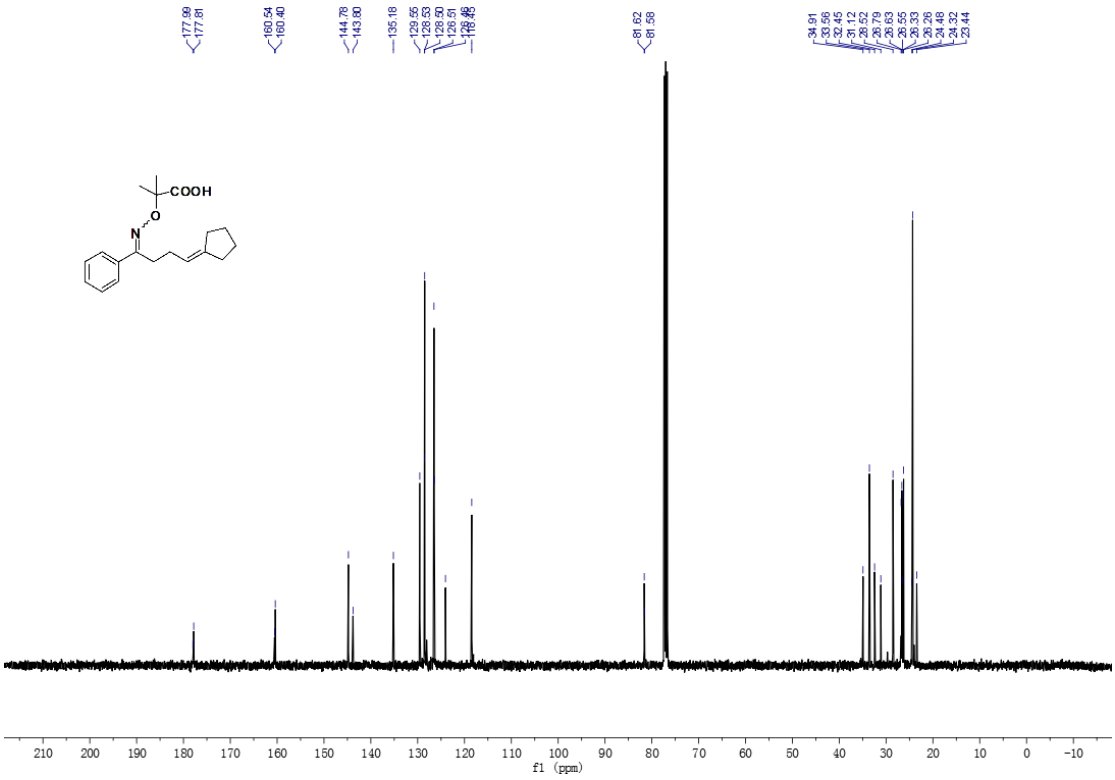
^{13}C NMR of **1ar**



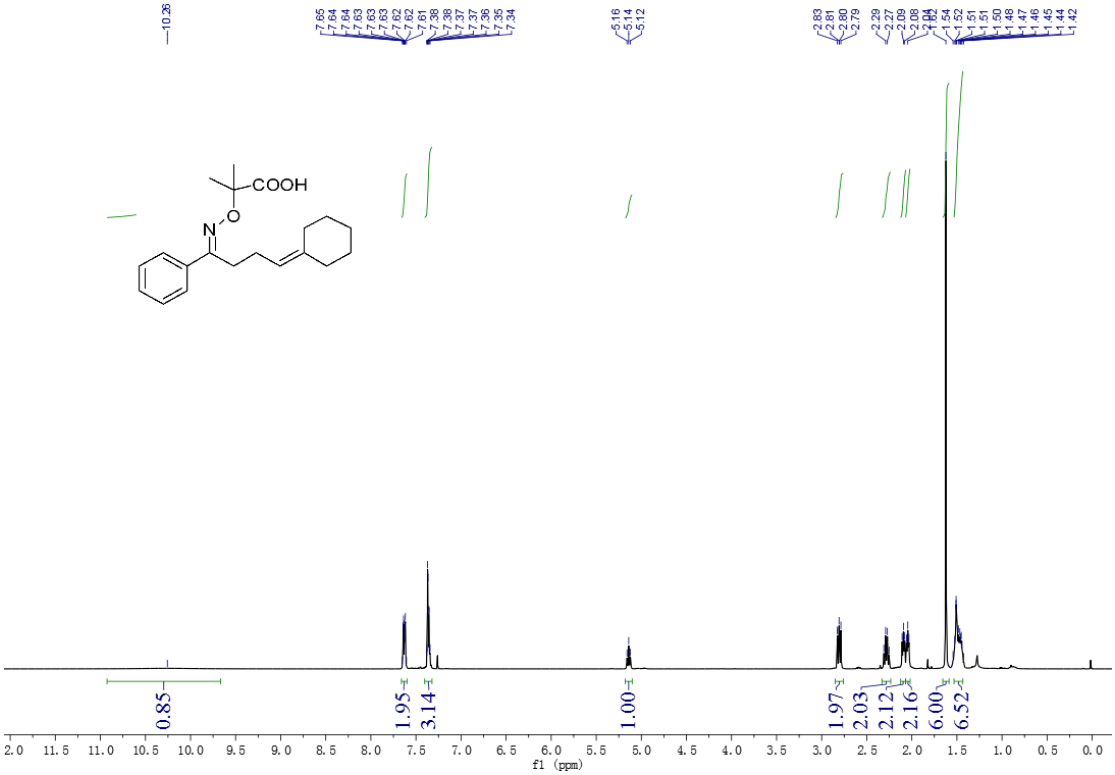
^{1}H NMR of **1as**



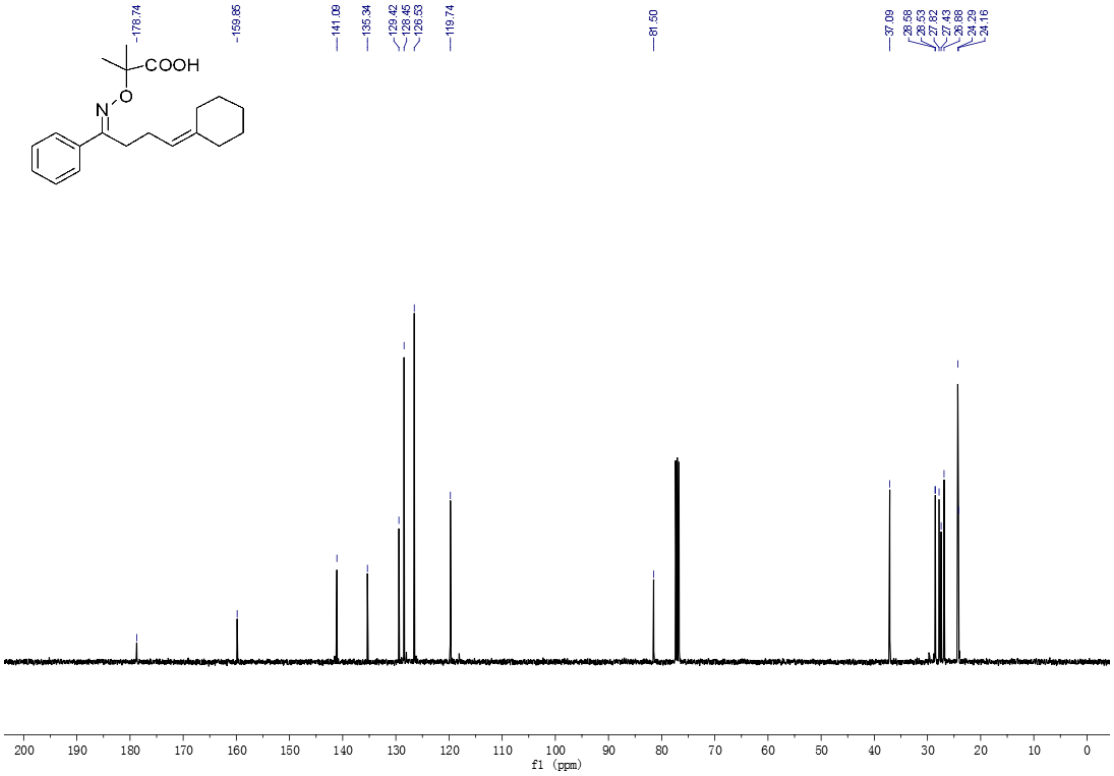
^{13}C NMR of **1as**



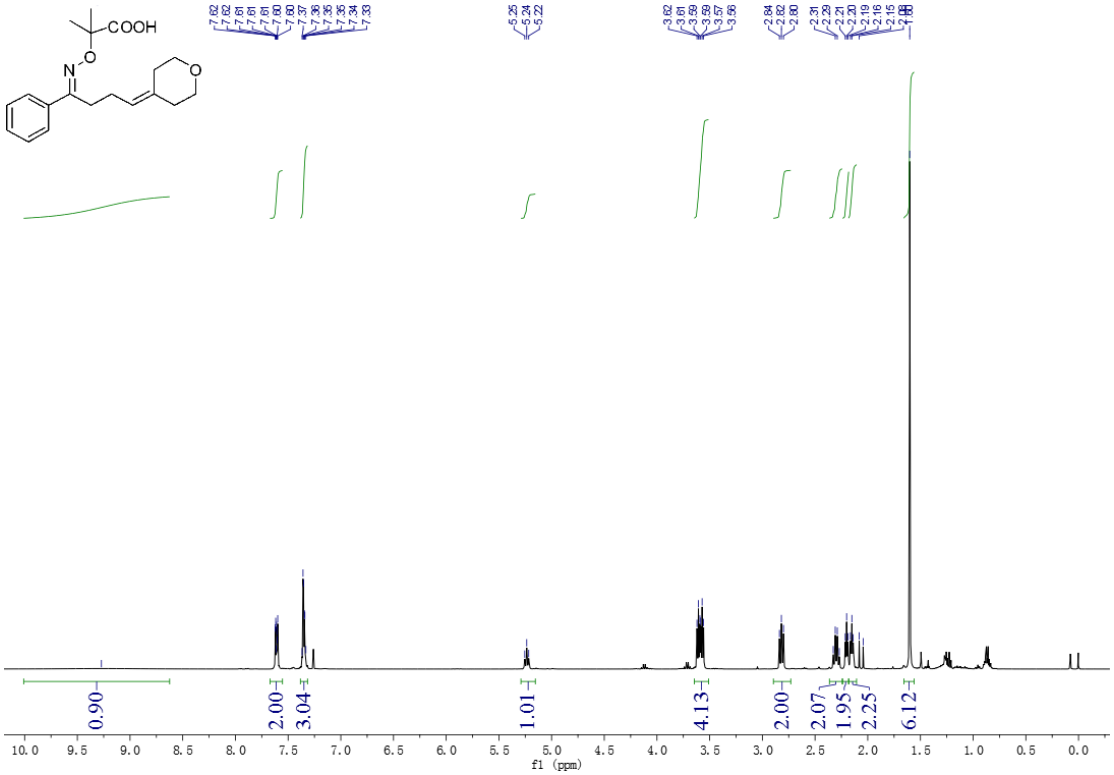
^{1}H NMR of **1at**



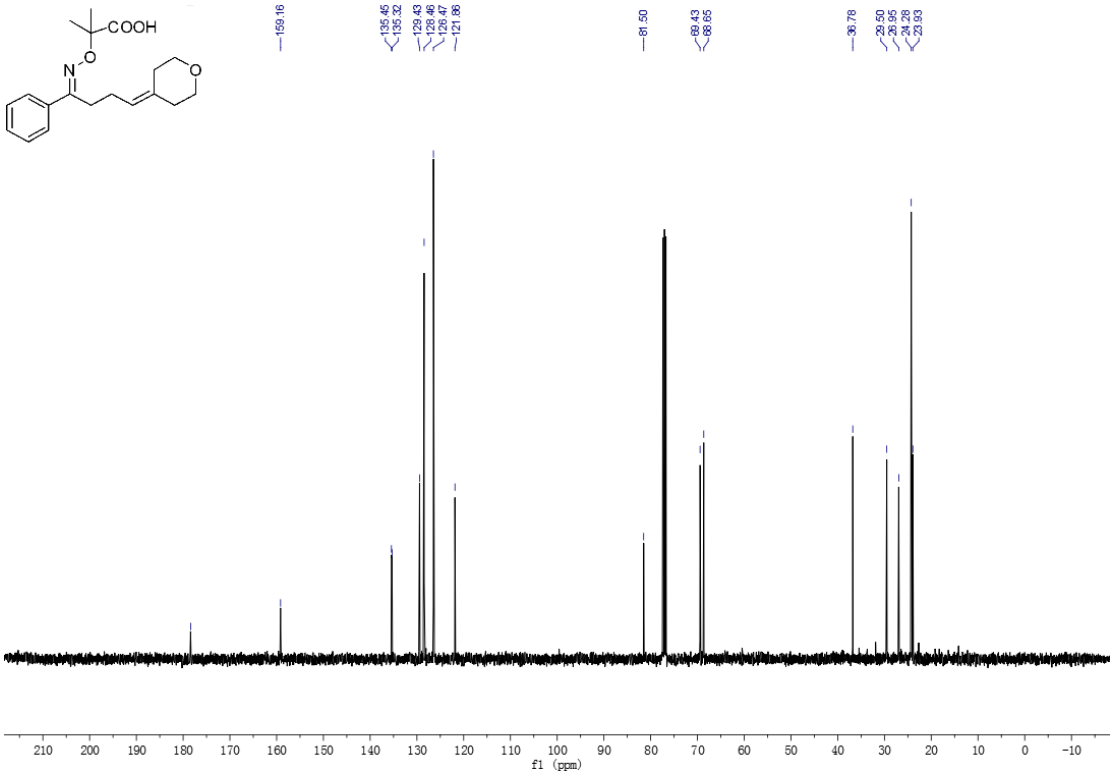
^{13}C NMR of **1at**



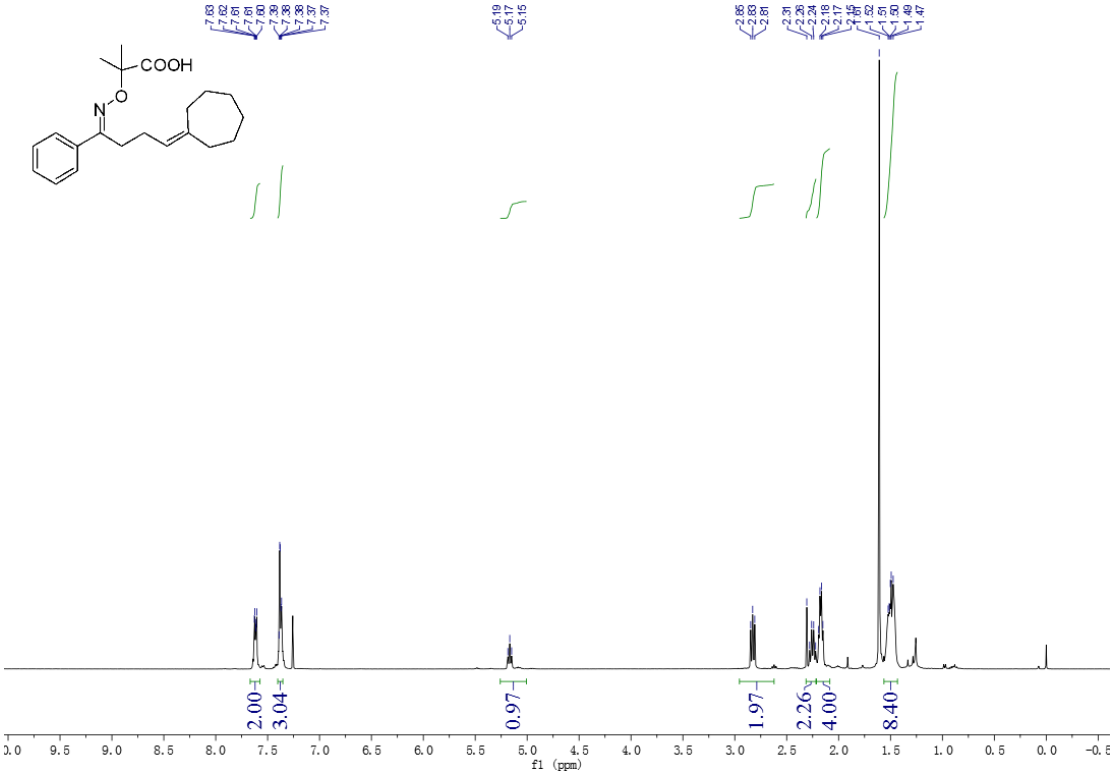
^{1}H NMR of **1au**



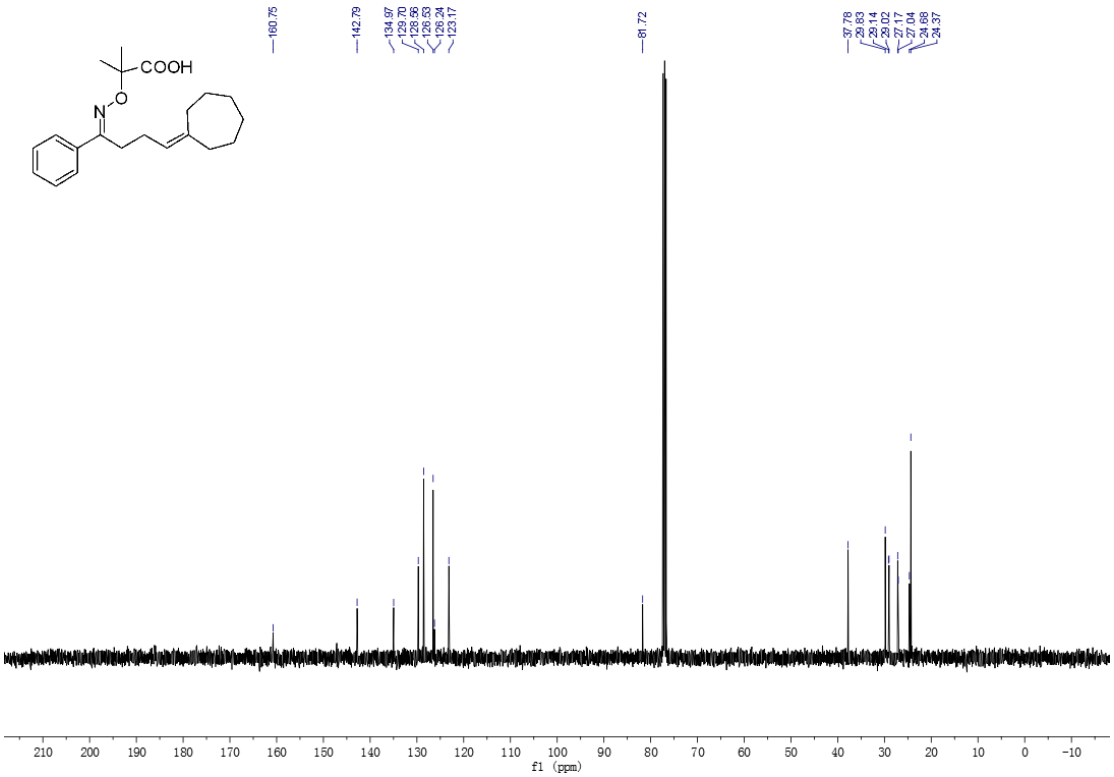
^{13}C NMR of **1au**



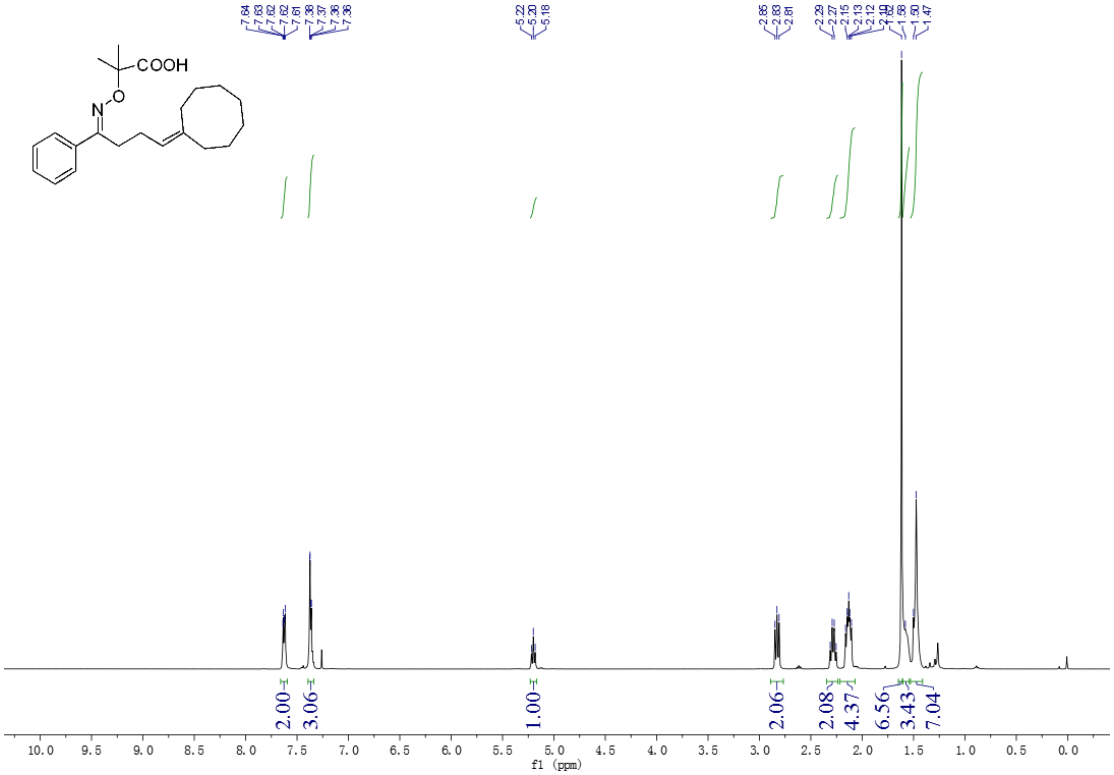
^{1}H NMR of **1av**



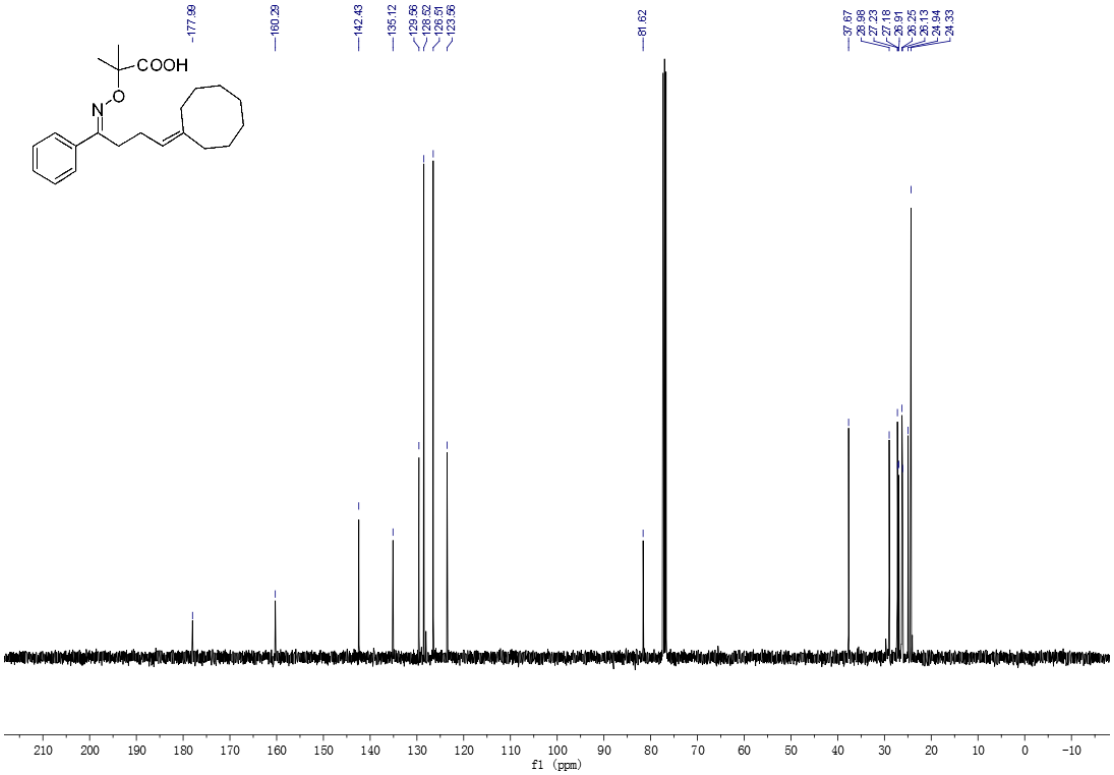
^{13}C NMR of **1av**



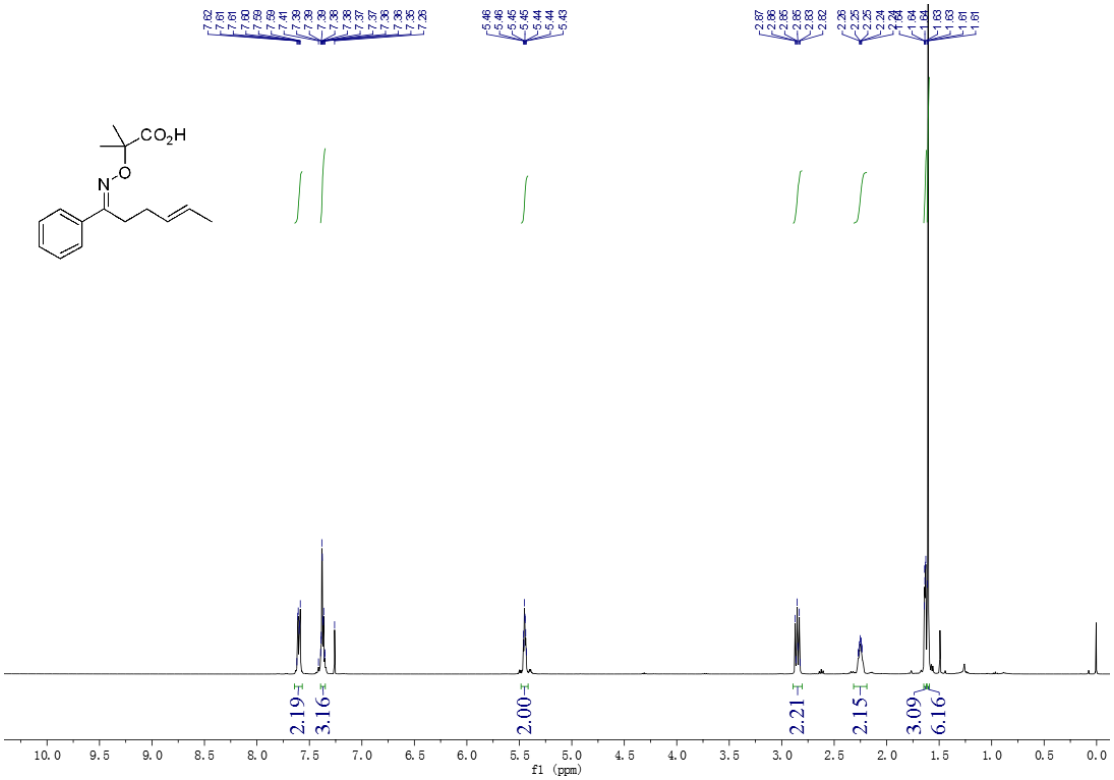
^{1}H NMR of **1aw**



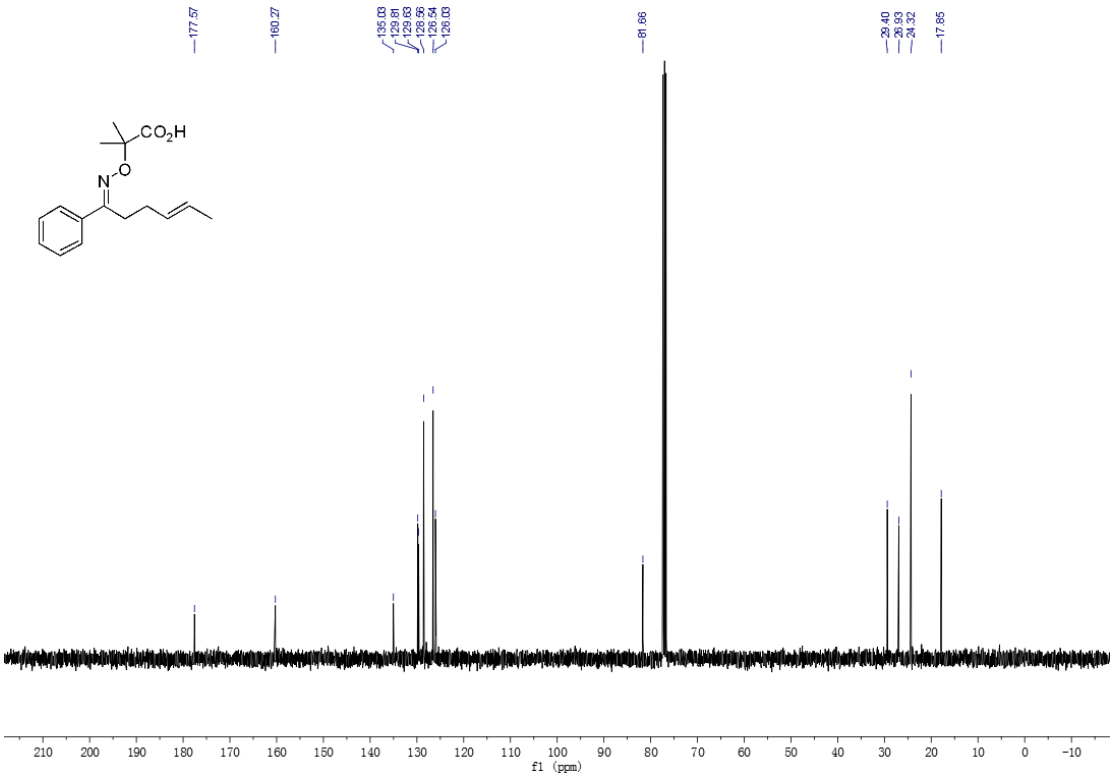
^{13}C NMR of **1aw**



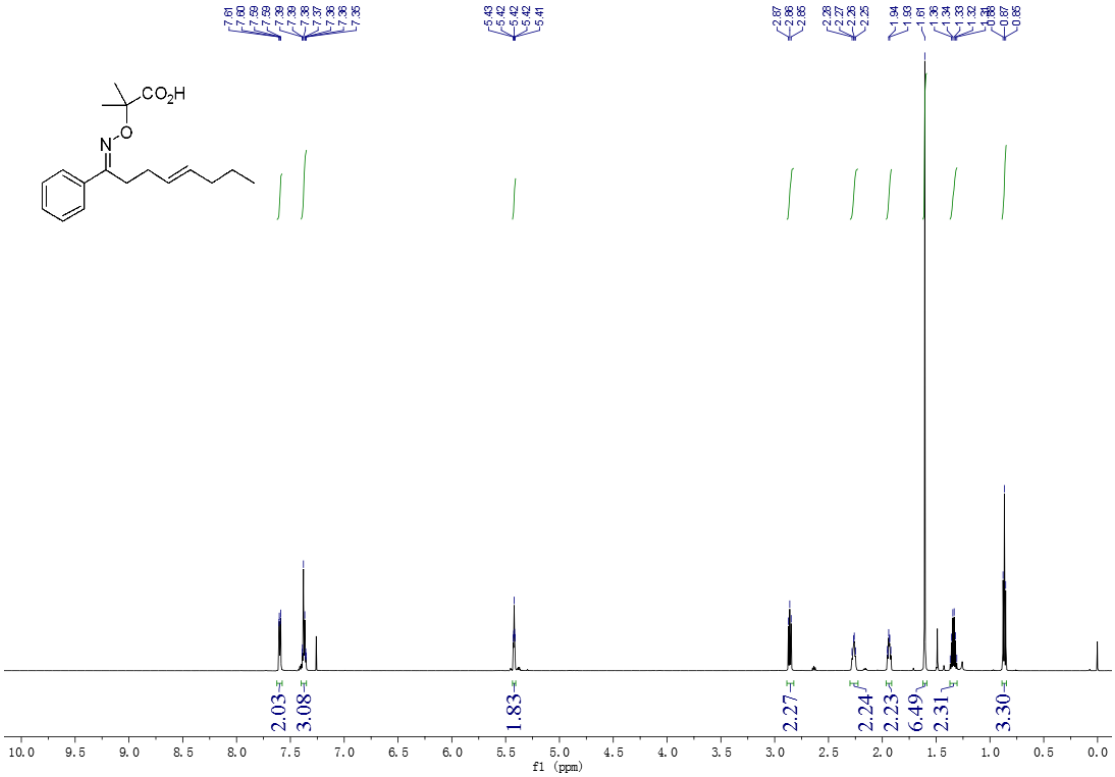
^{1}H NMR of **1ba**



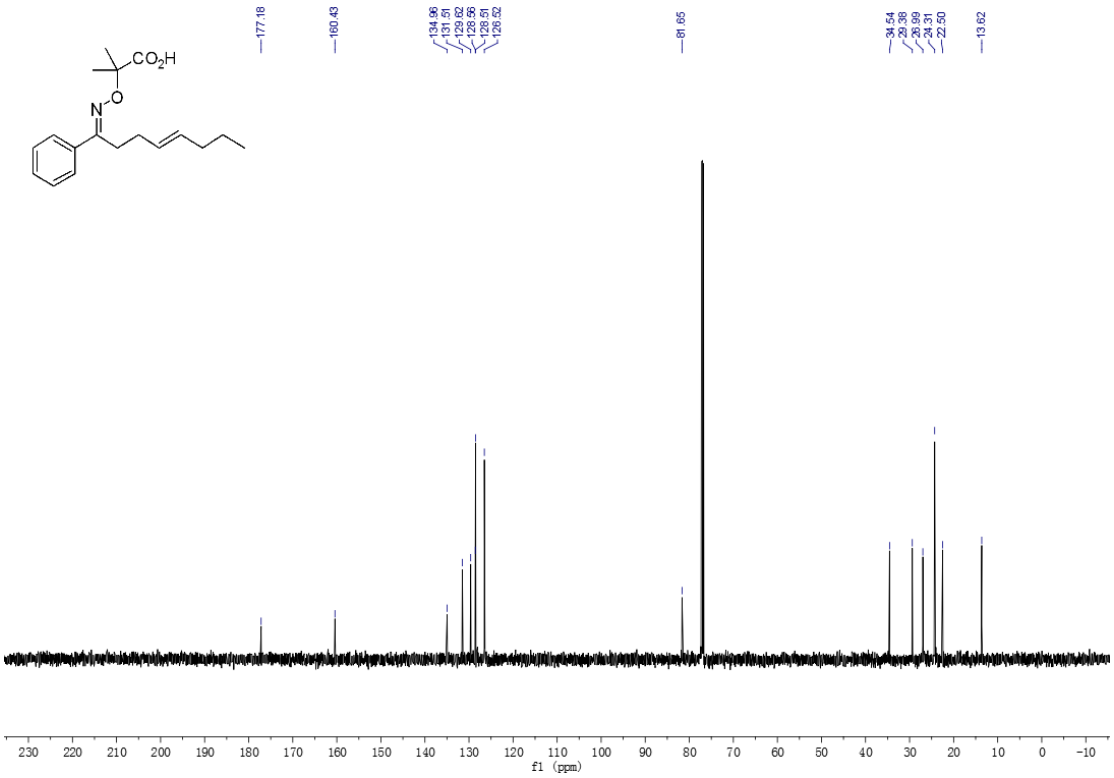
^{13}C NMR of **1ba**



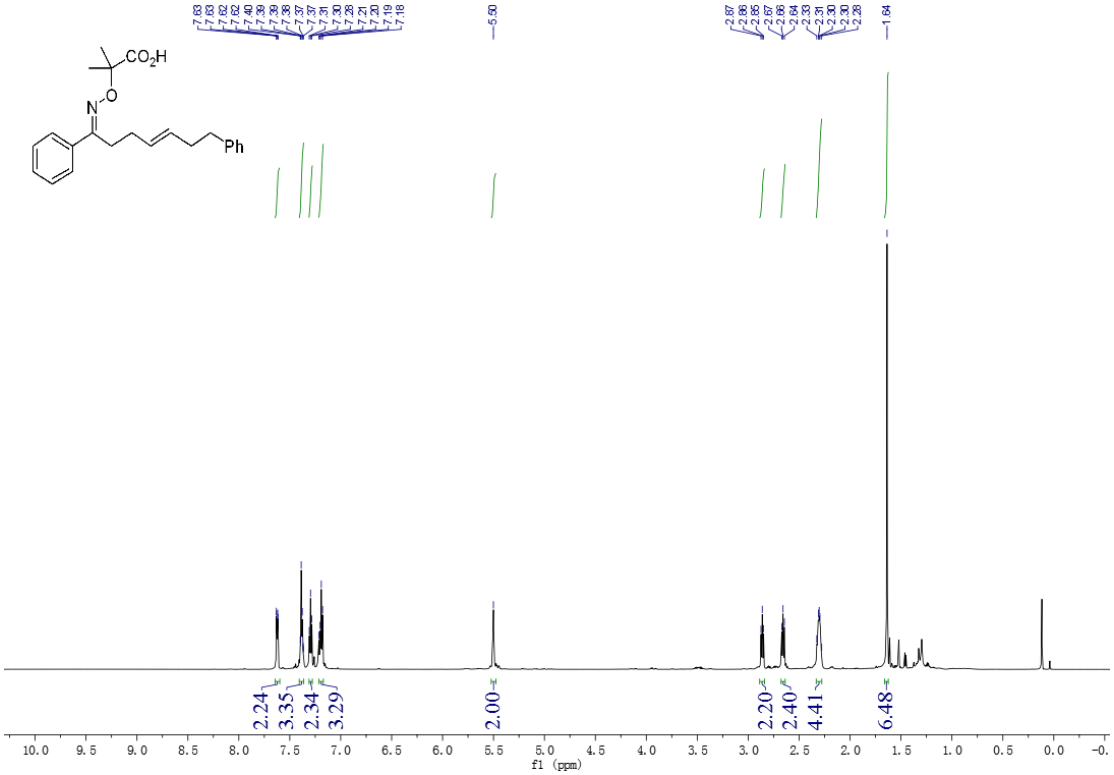
^{1}H NMR of **1bb**



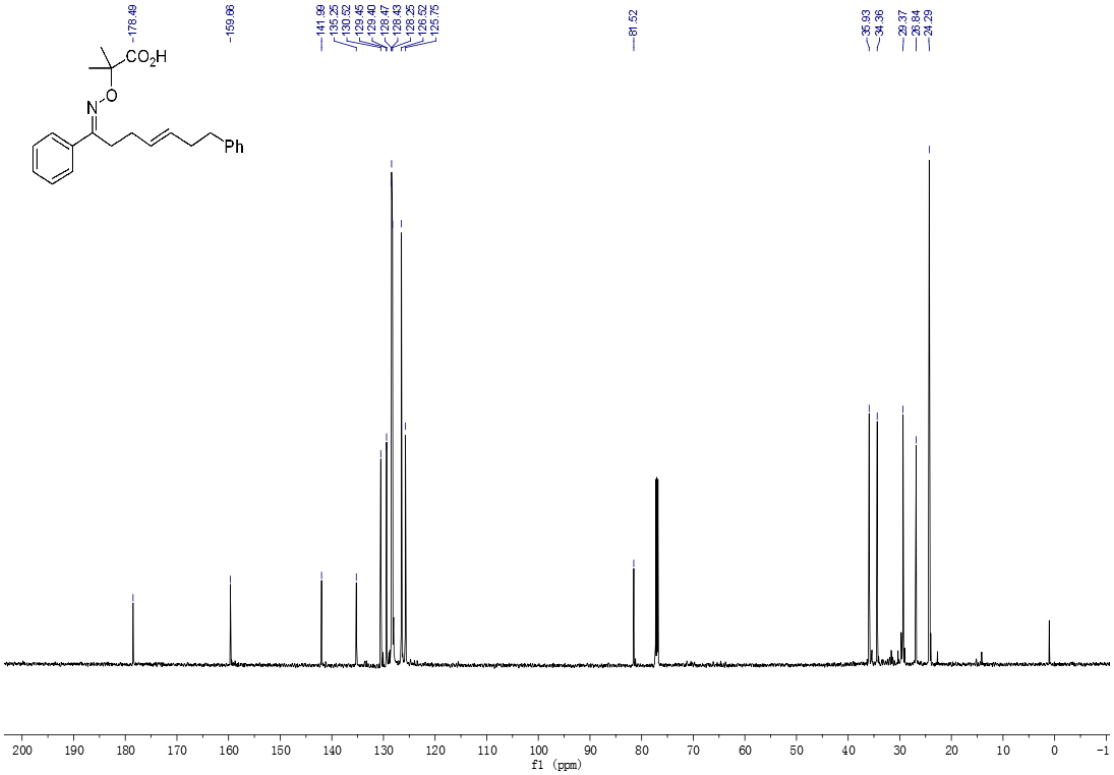
^{13}C NMR of **1bb**



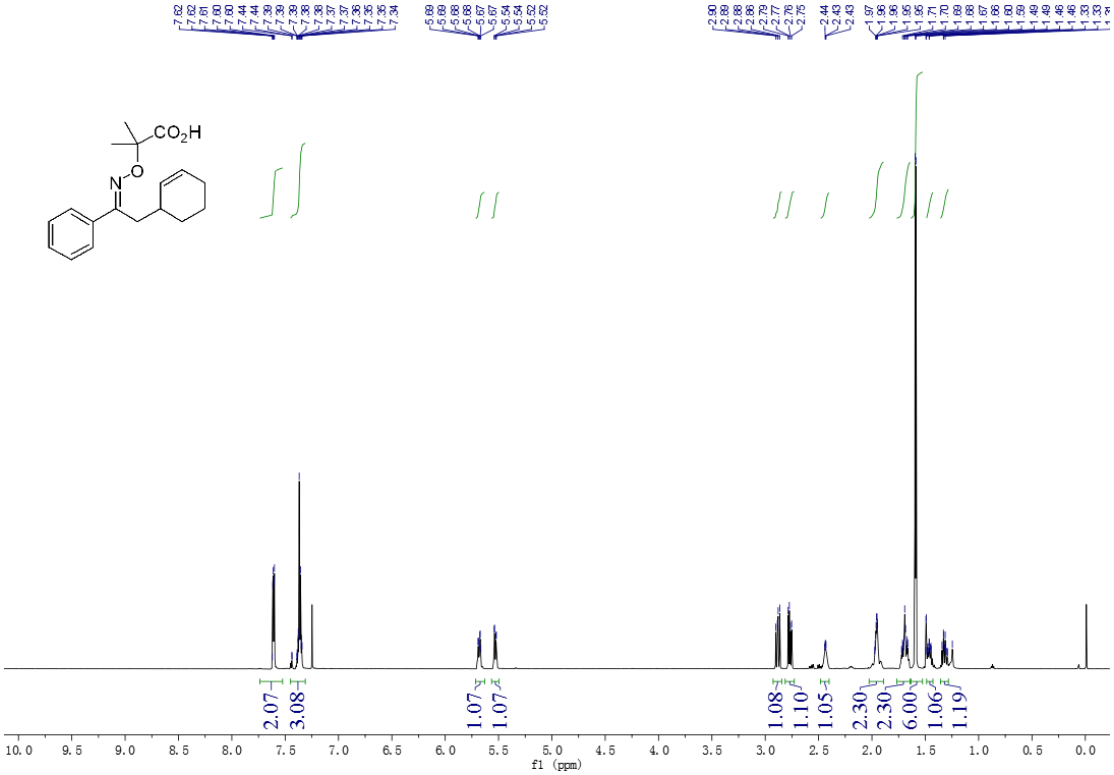
^{1}H NMR of **1bc**



^{13}C NMR of **1bc**



^{1}H NMR of **1bd**



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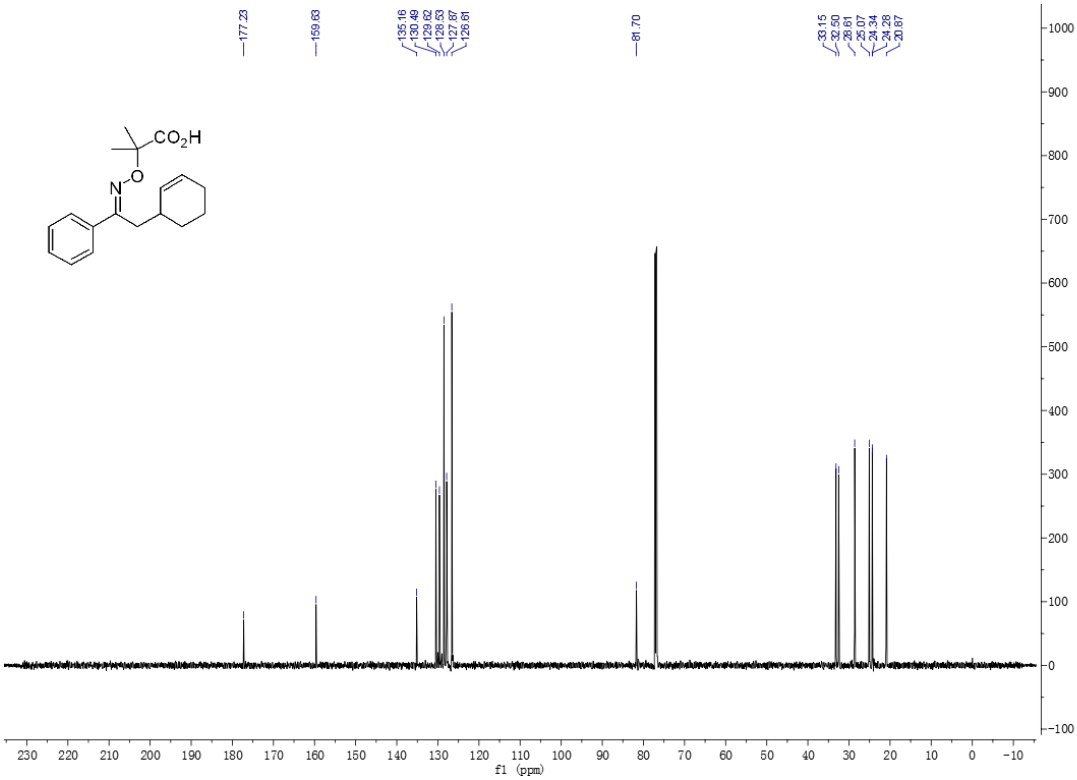

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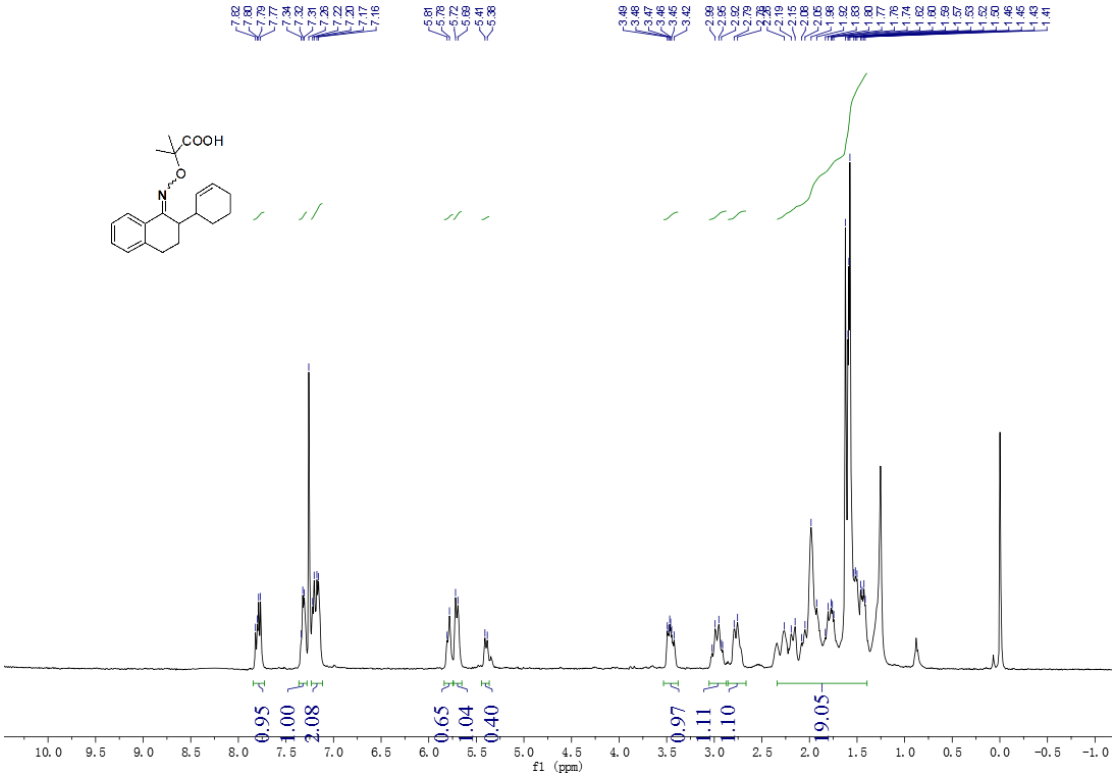

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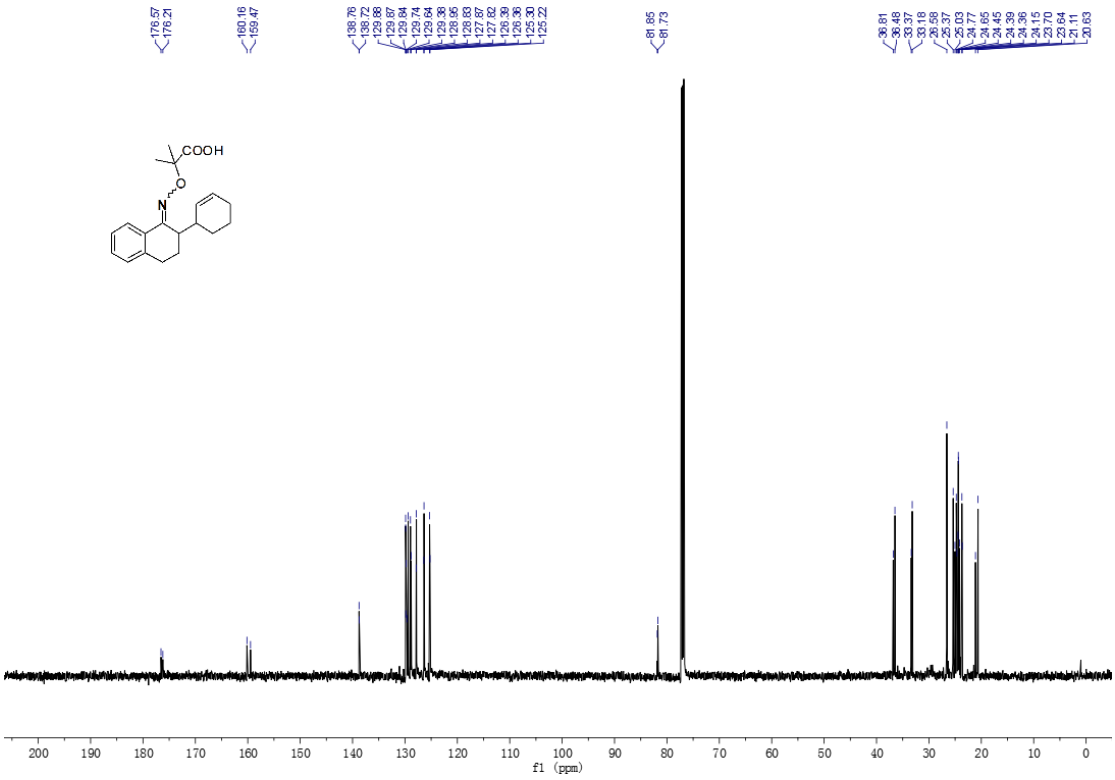

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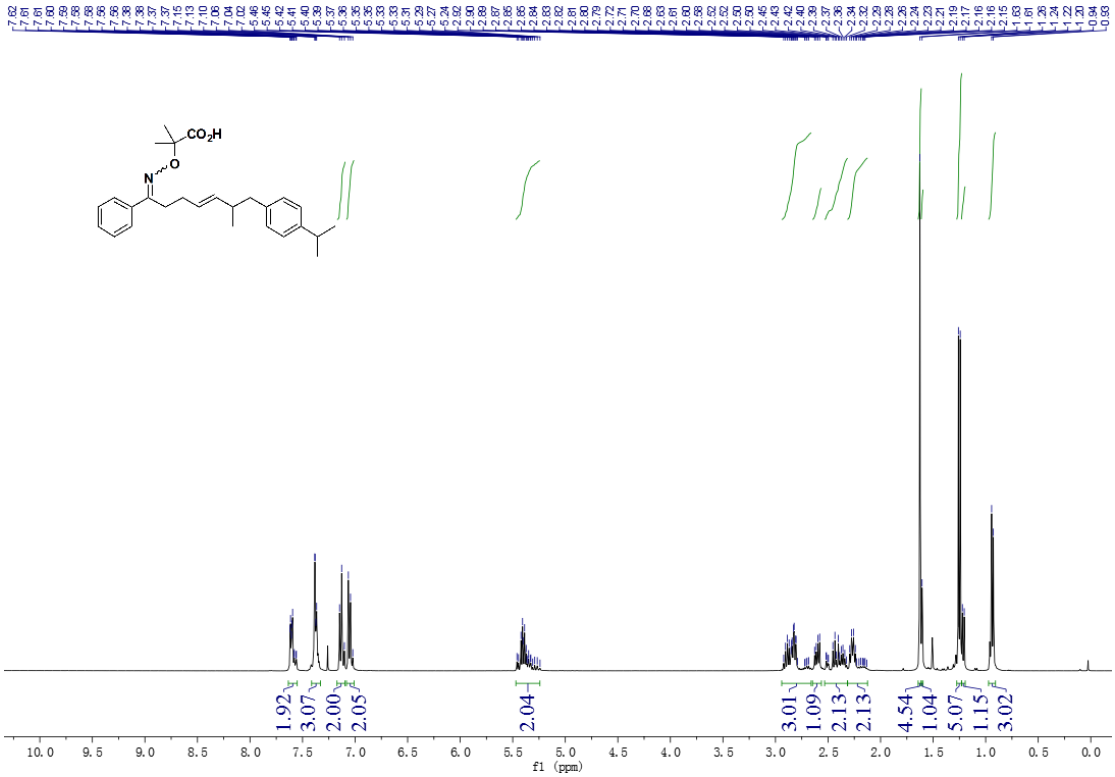

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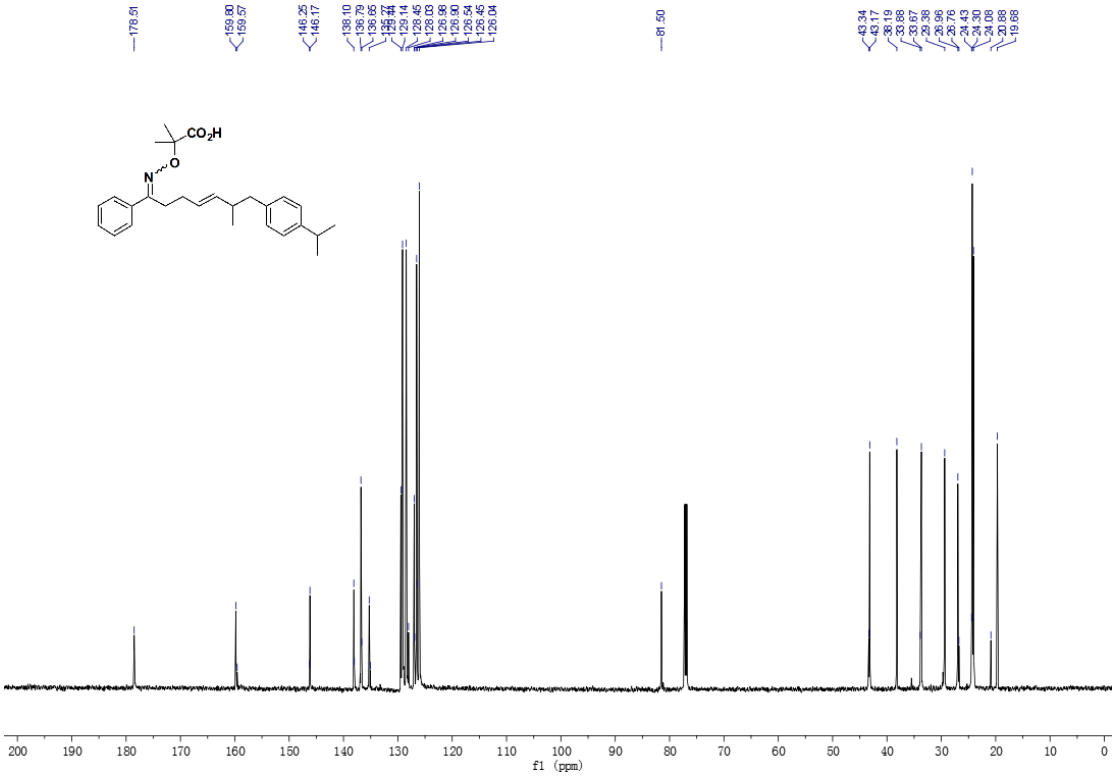

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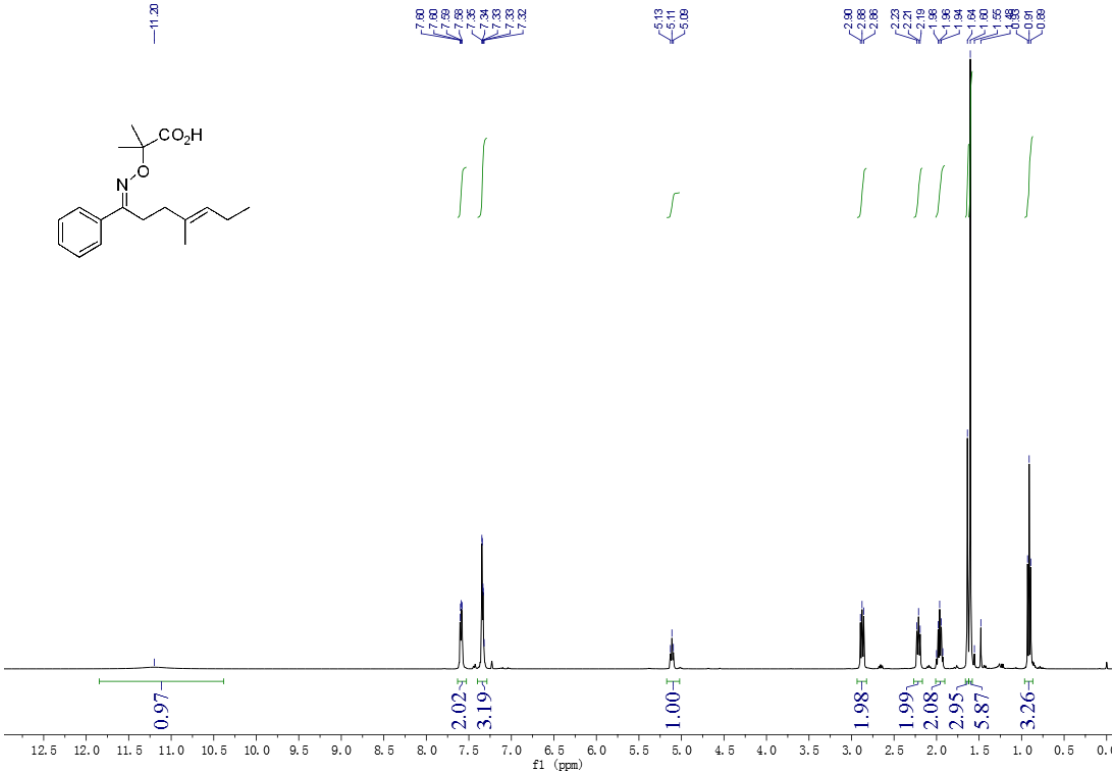

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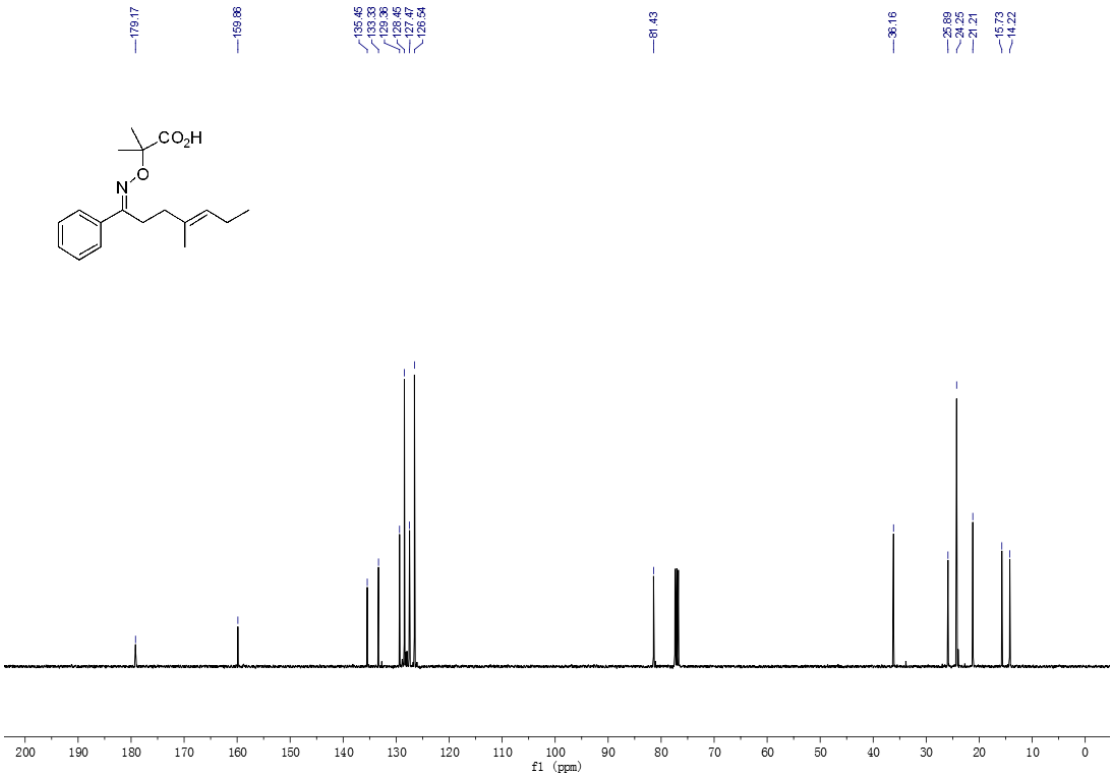

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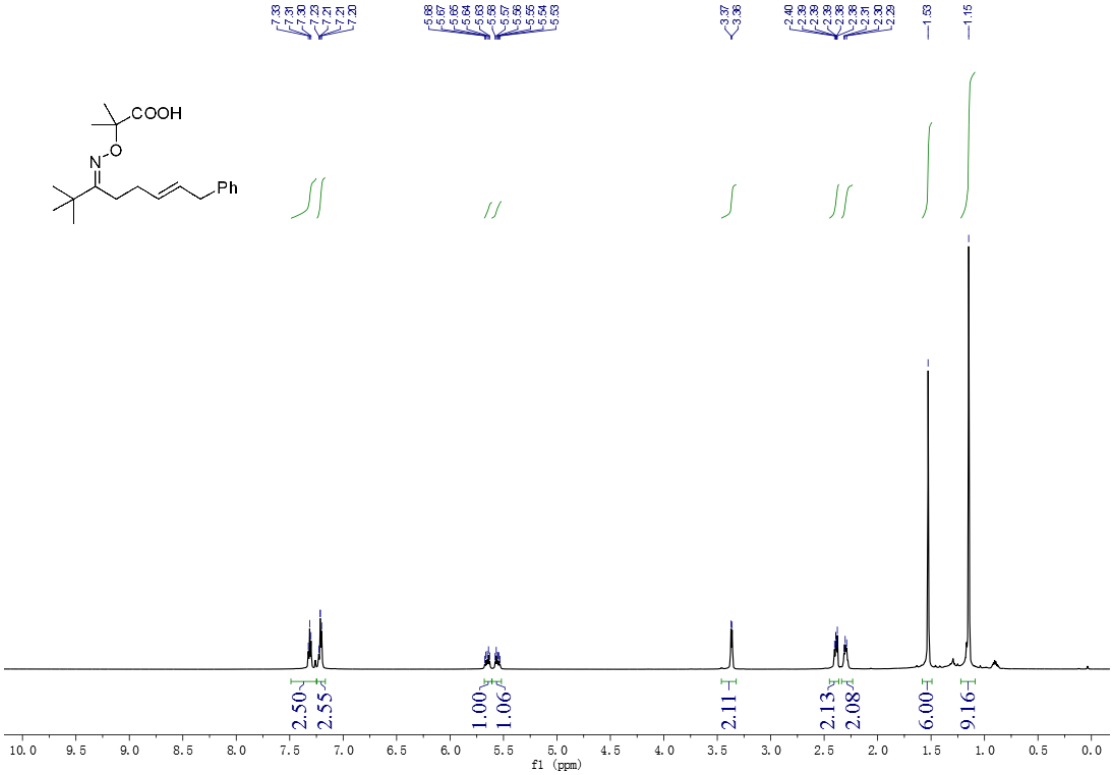

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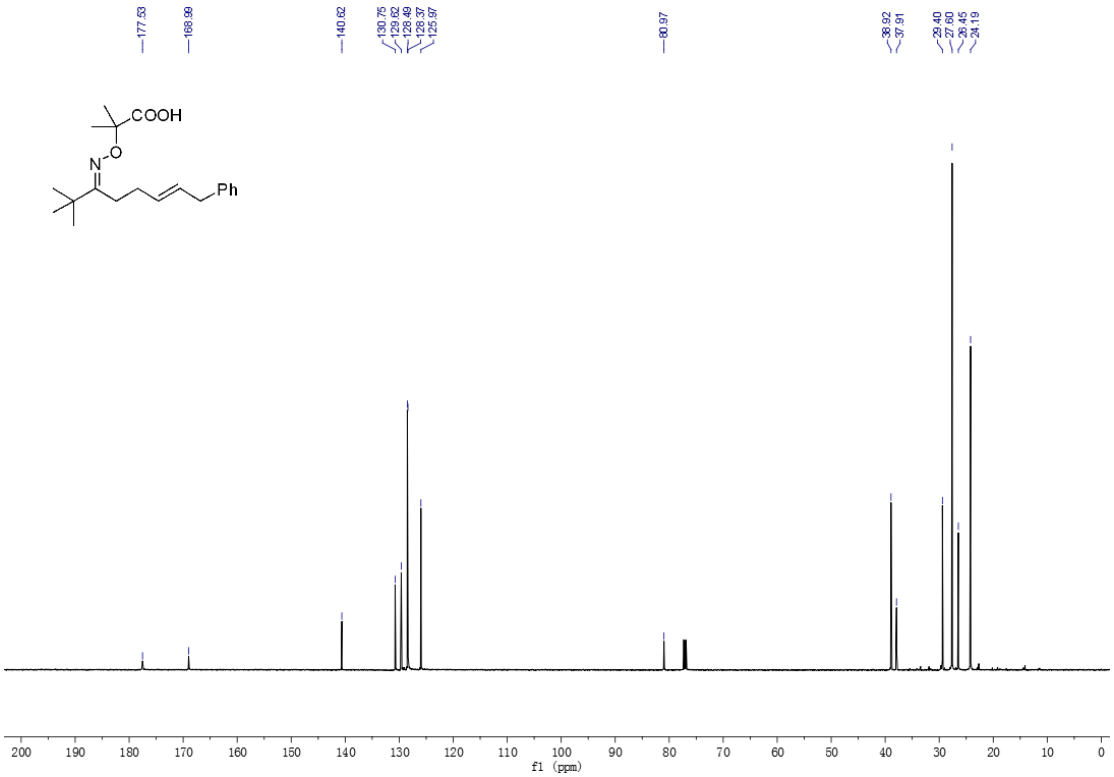

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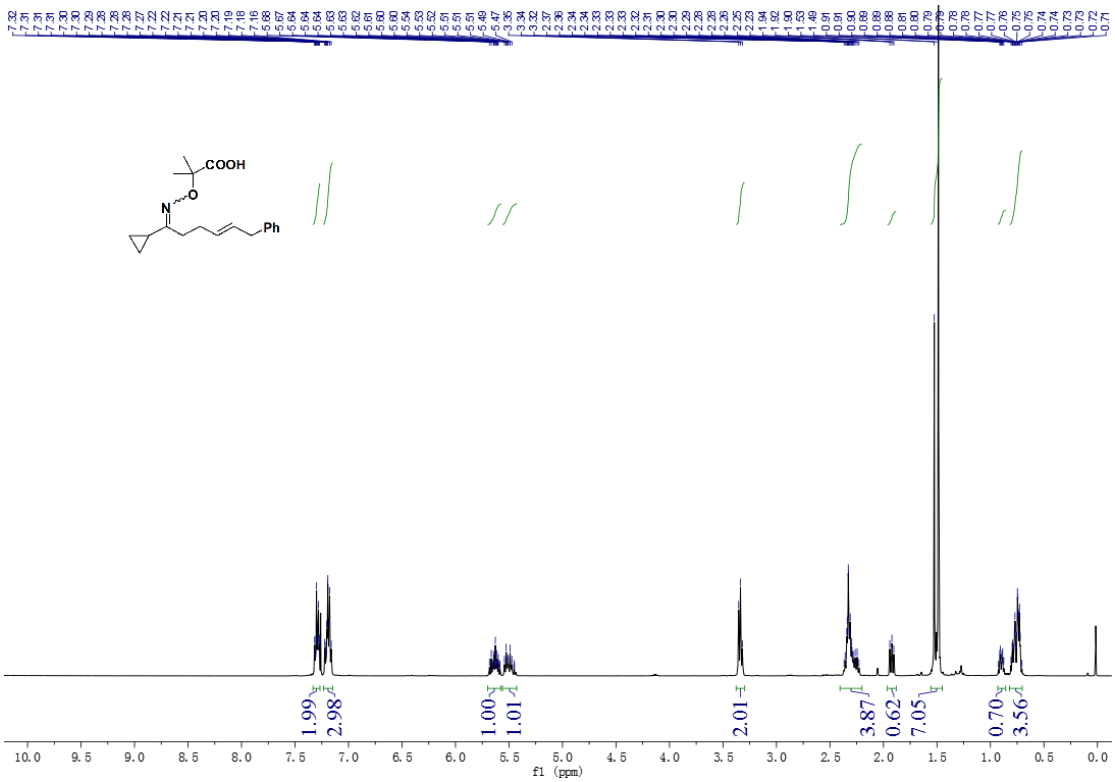

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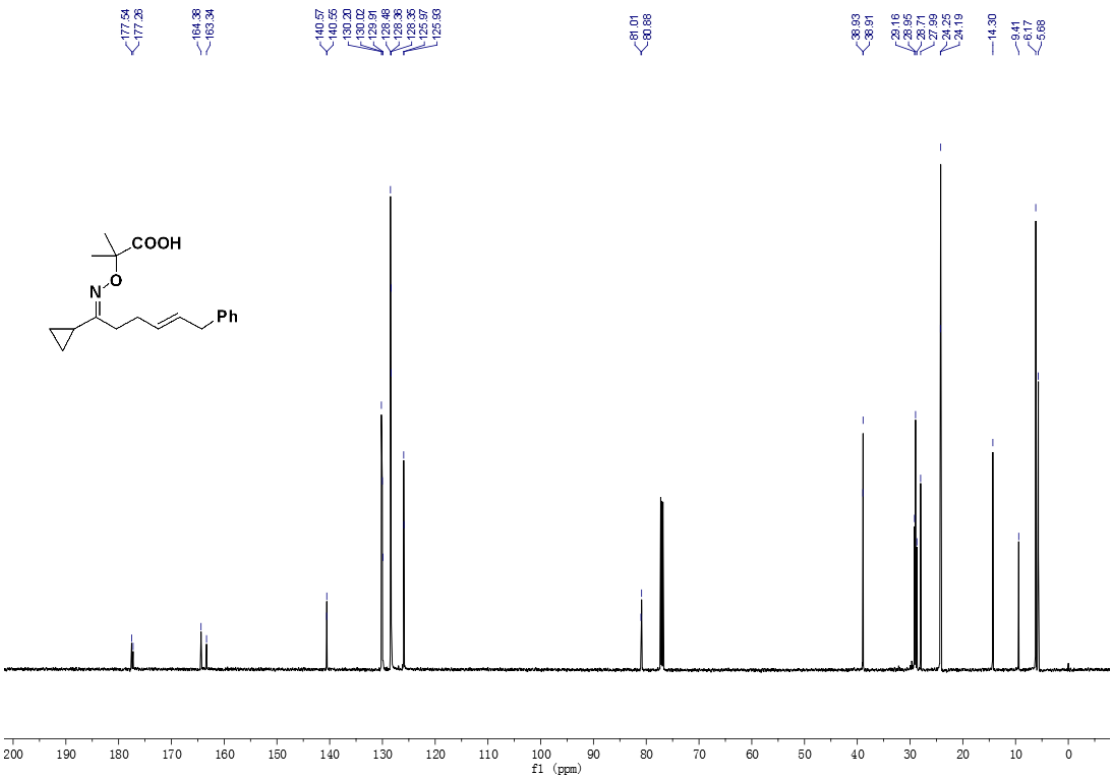

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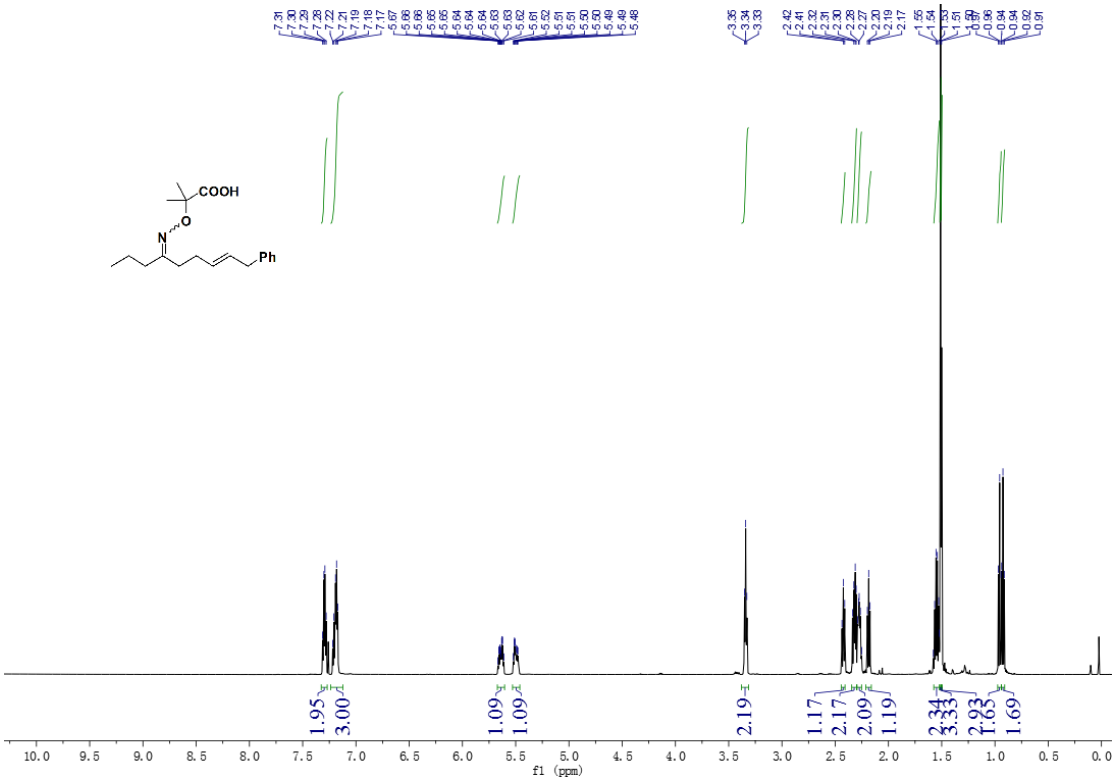

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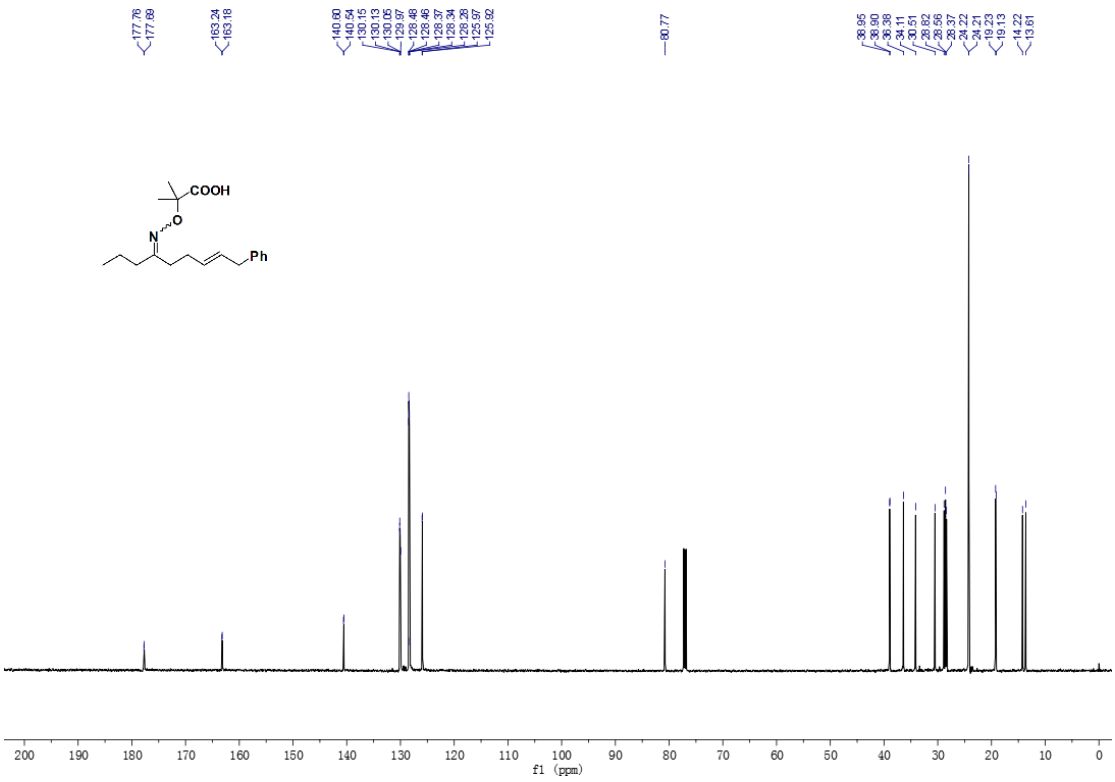
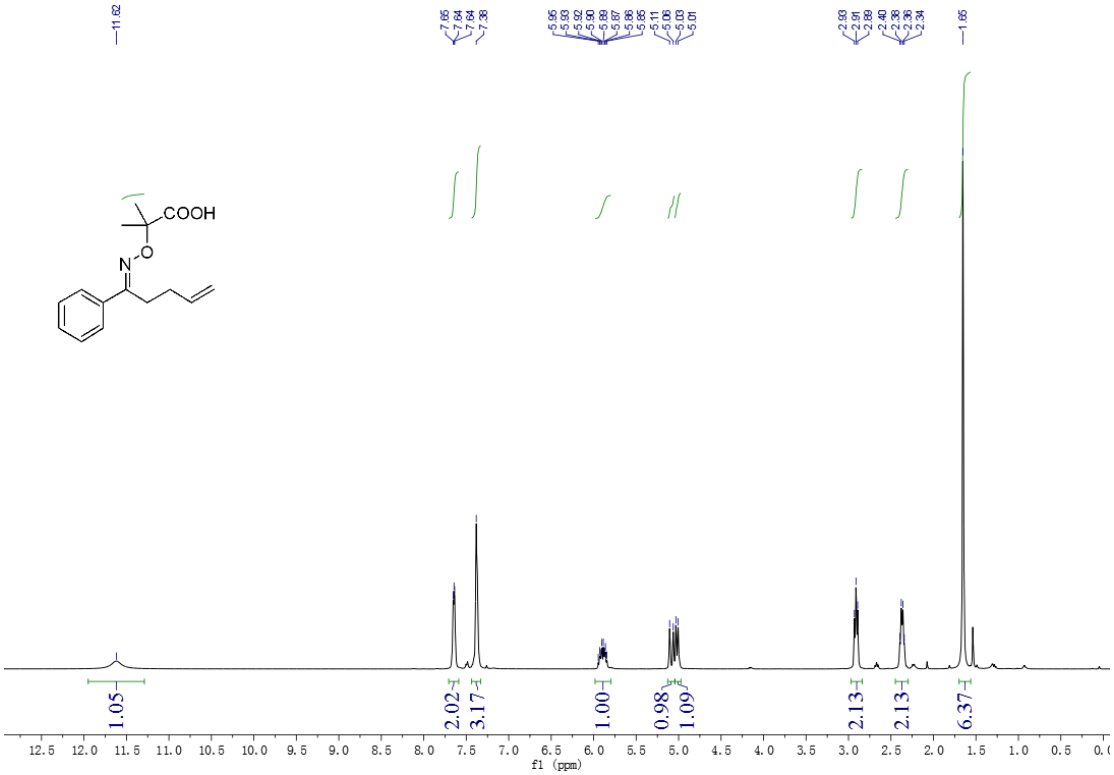
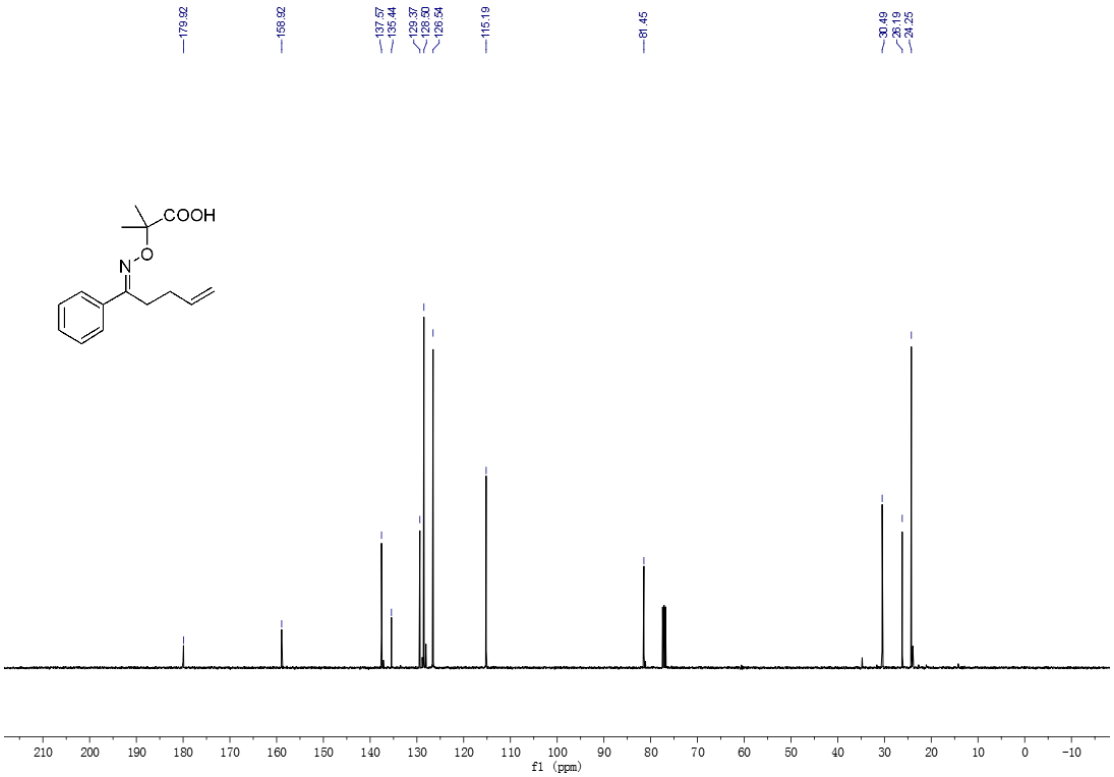


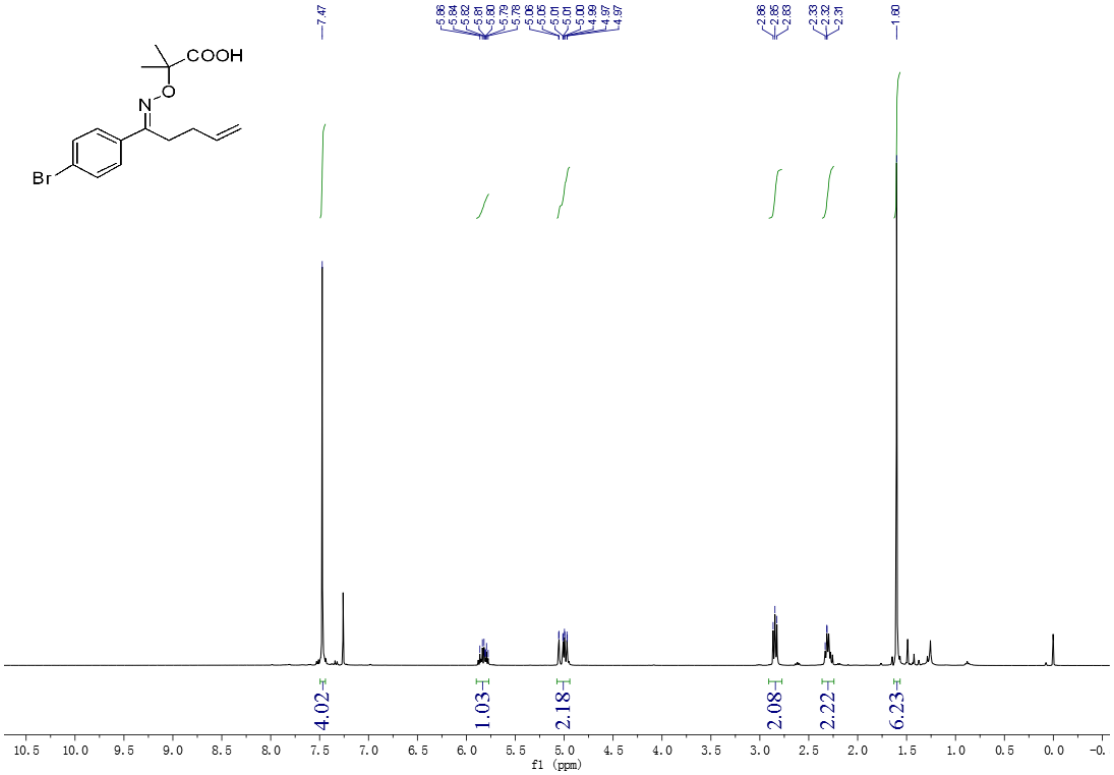
^{1}H NMR of **1ca**



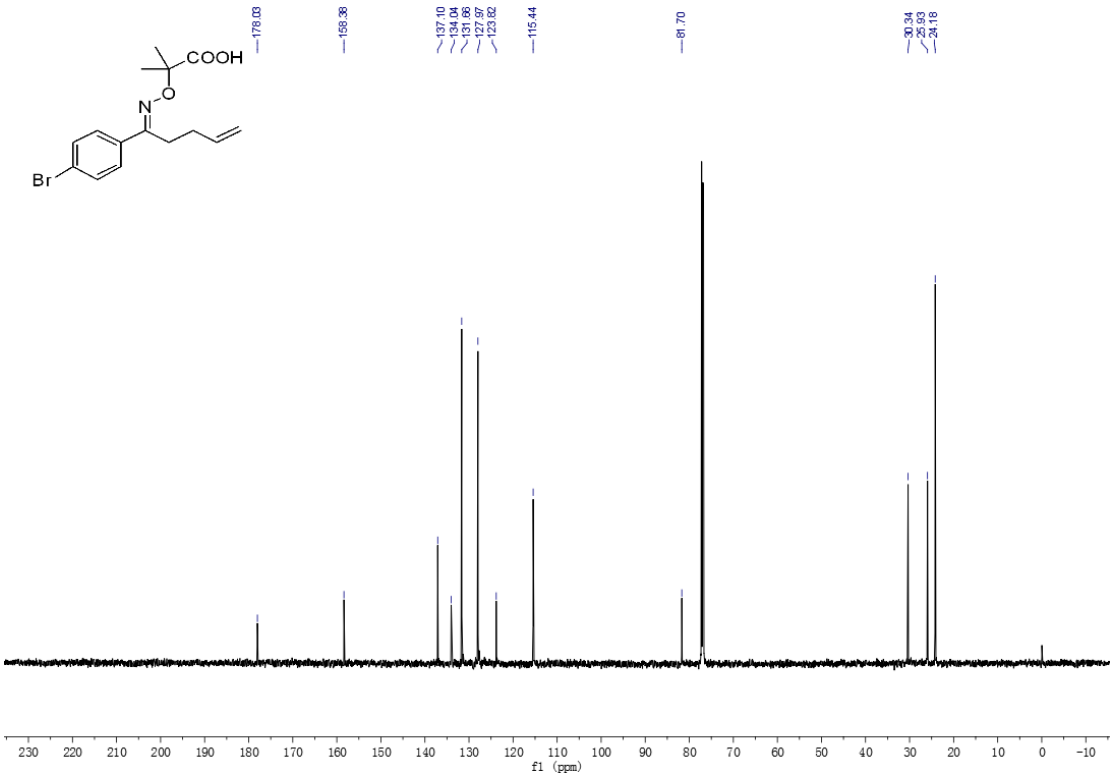
^{13}C NMR of **1ca**



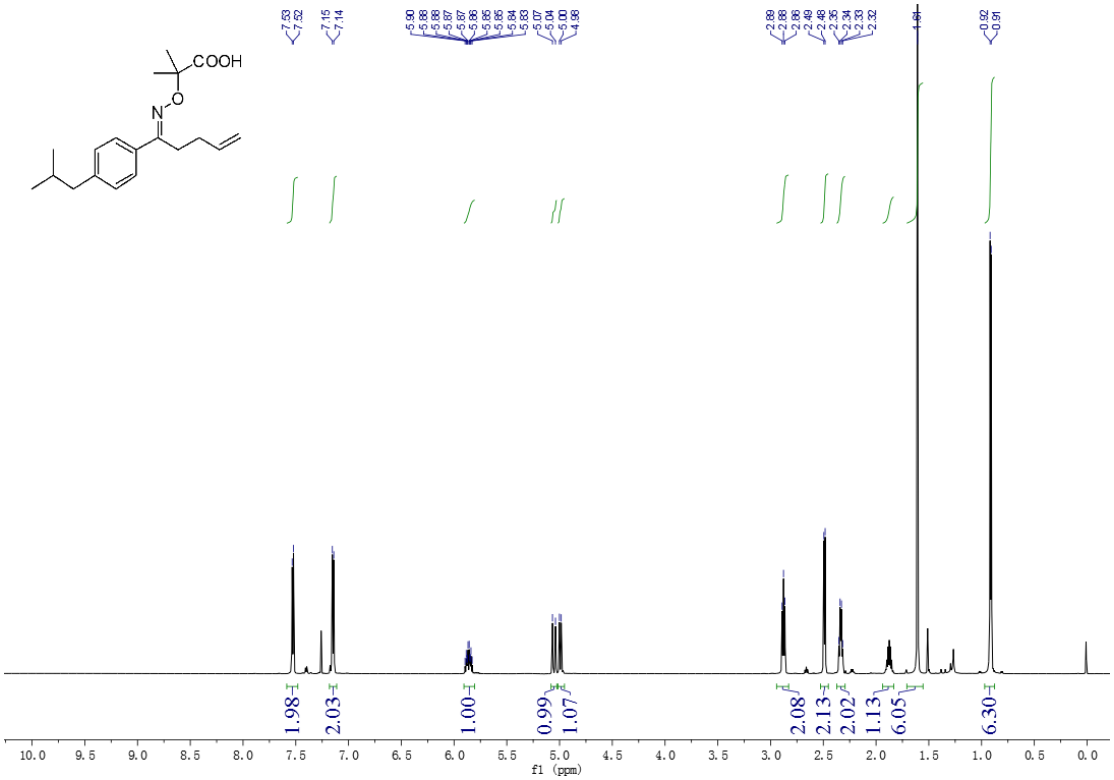
^{1}H NMR of **1cb**



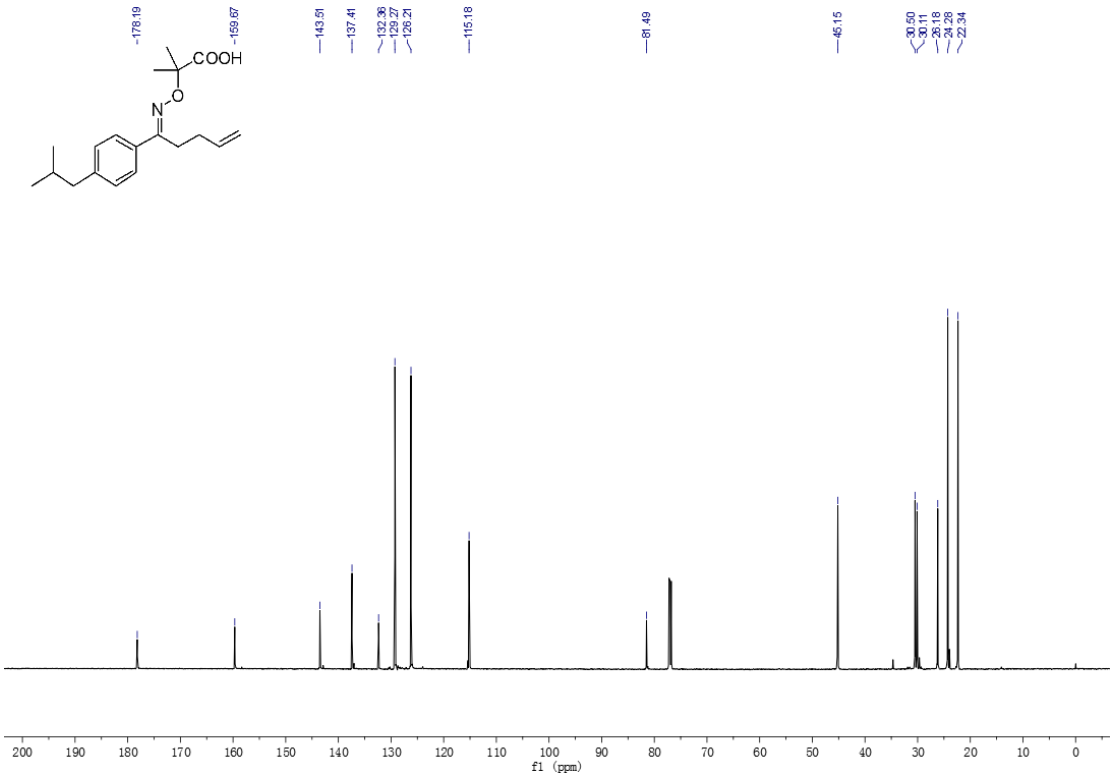
^{13}C NMR of **1cb**



^{1}H NMR of **1cc**



^{13}C NMR of **1cc**



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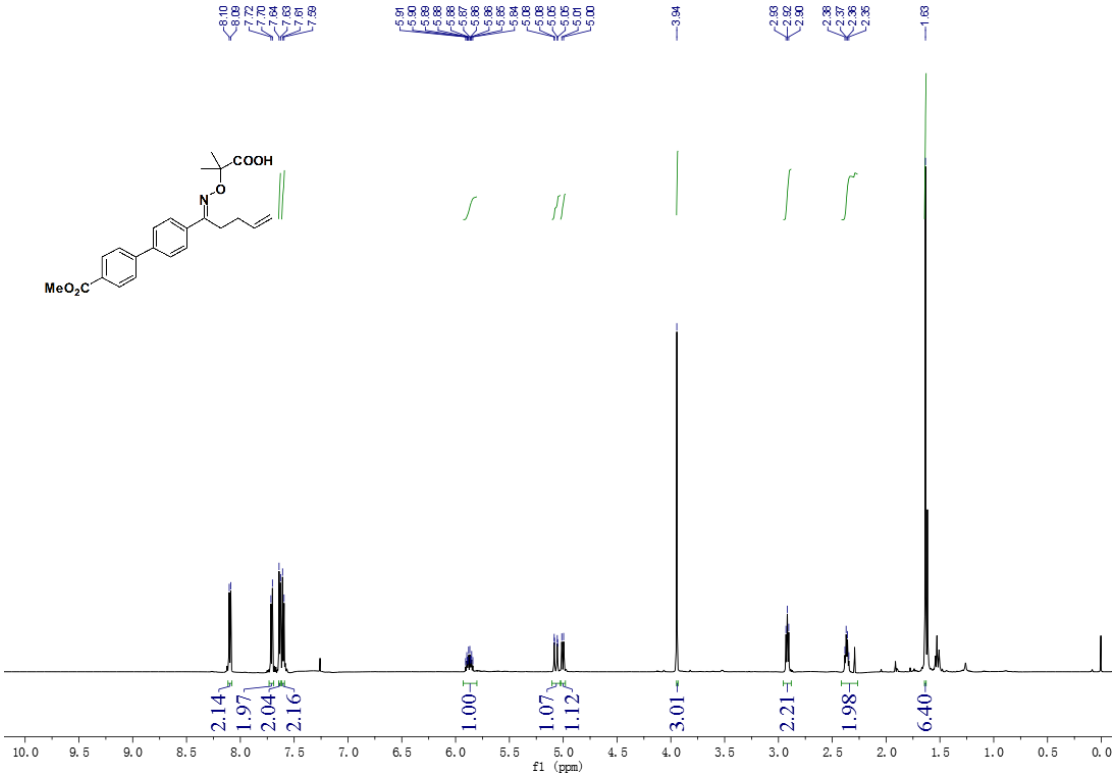

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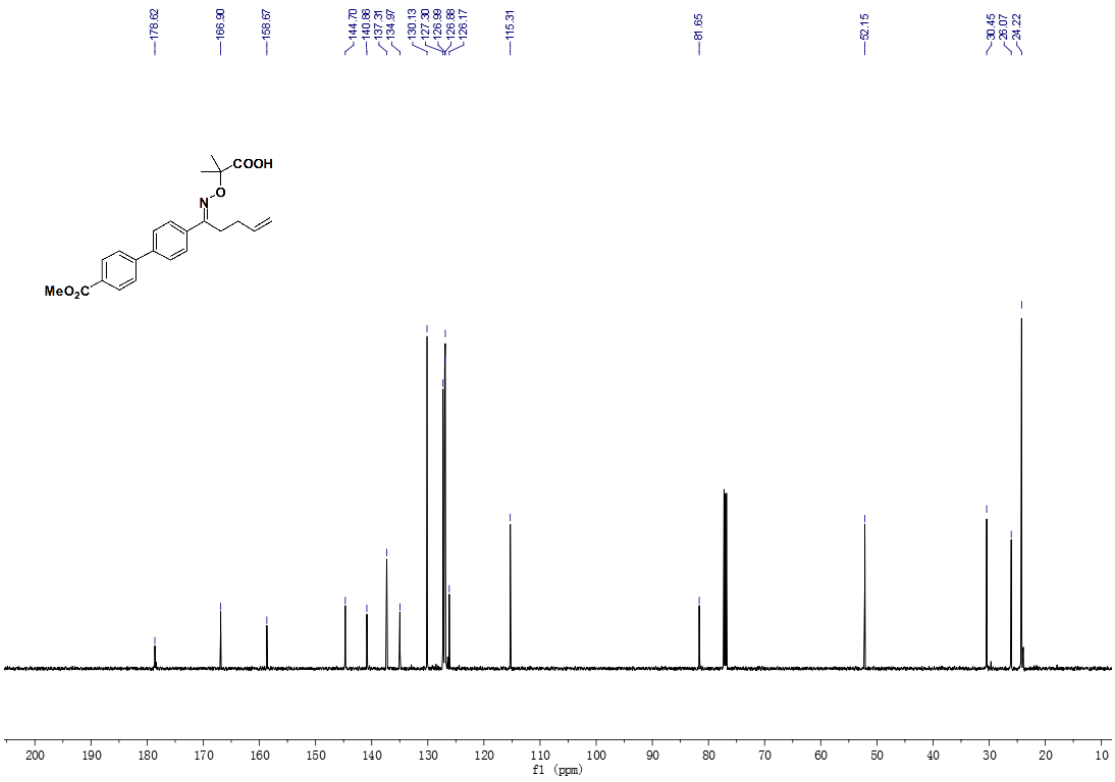
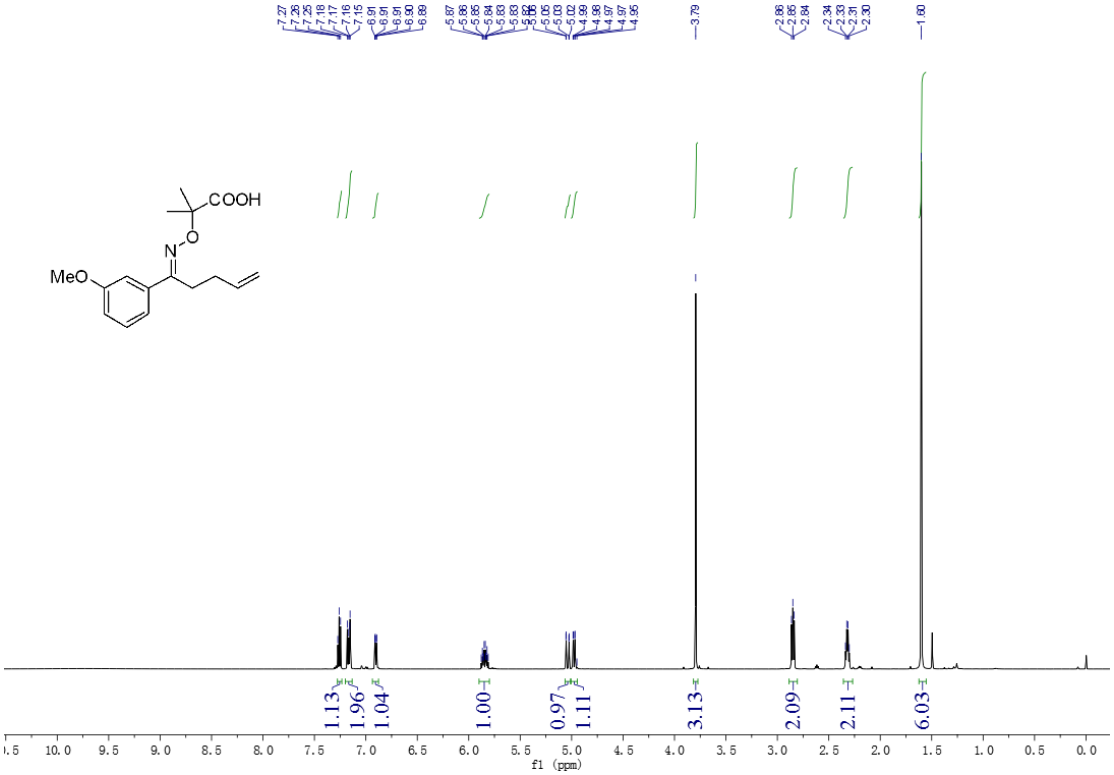
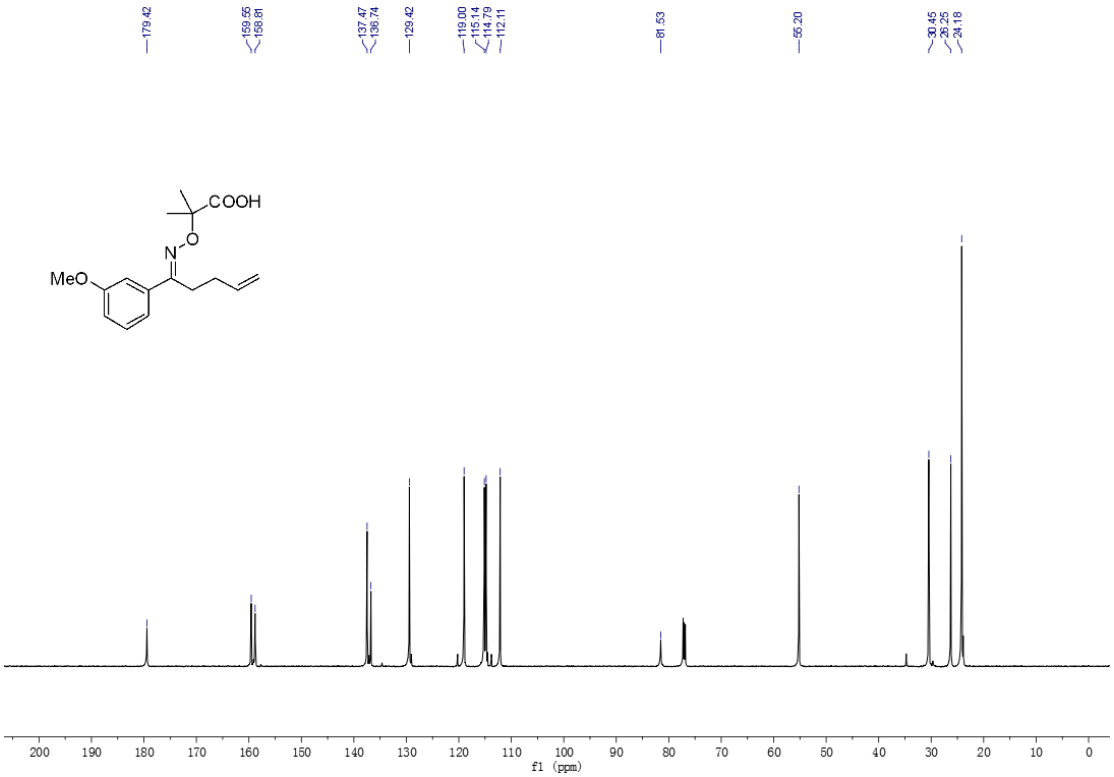


^{1}H NMR of **1ce**



^{13}C NMR of **1ce**



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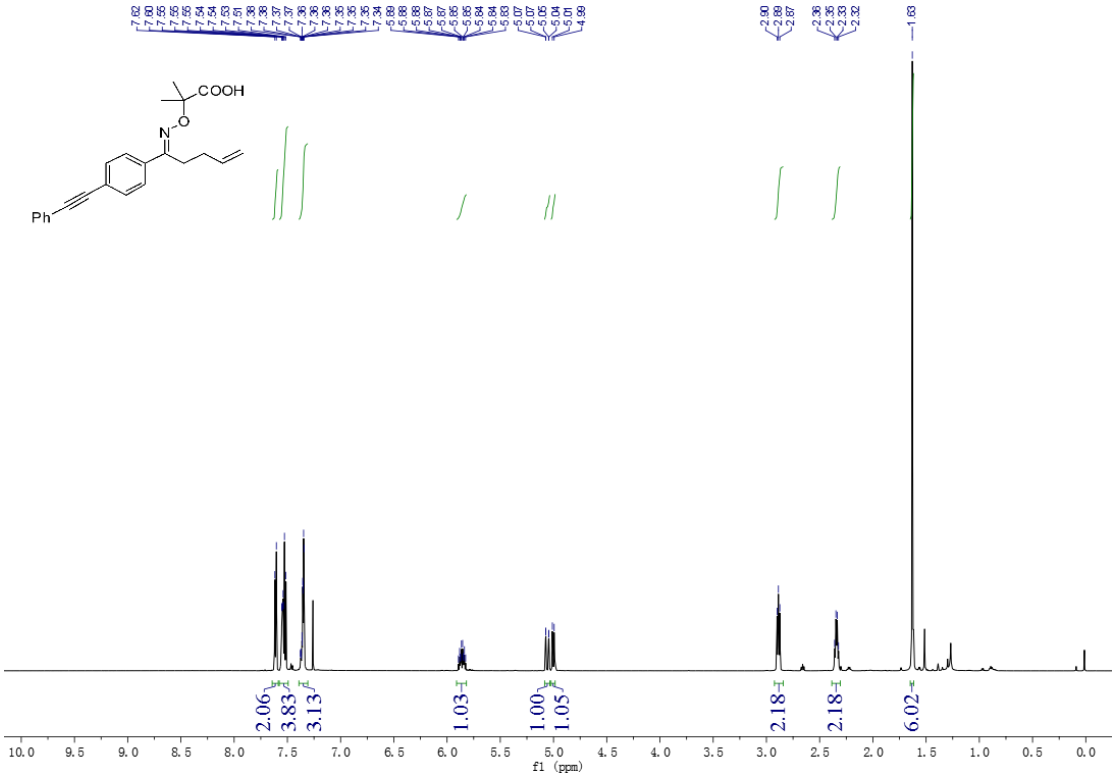

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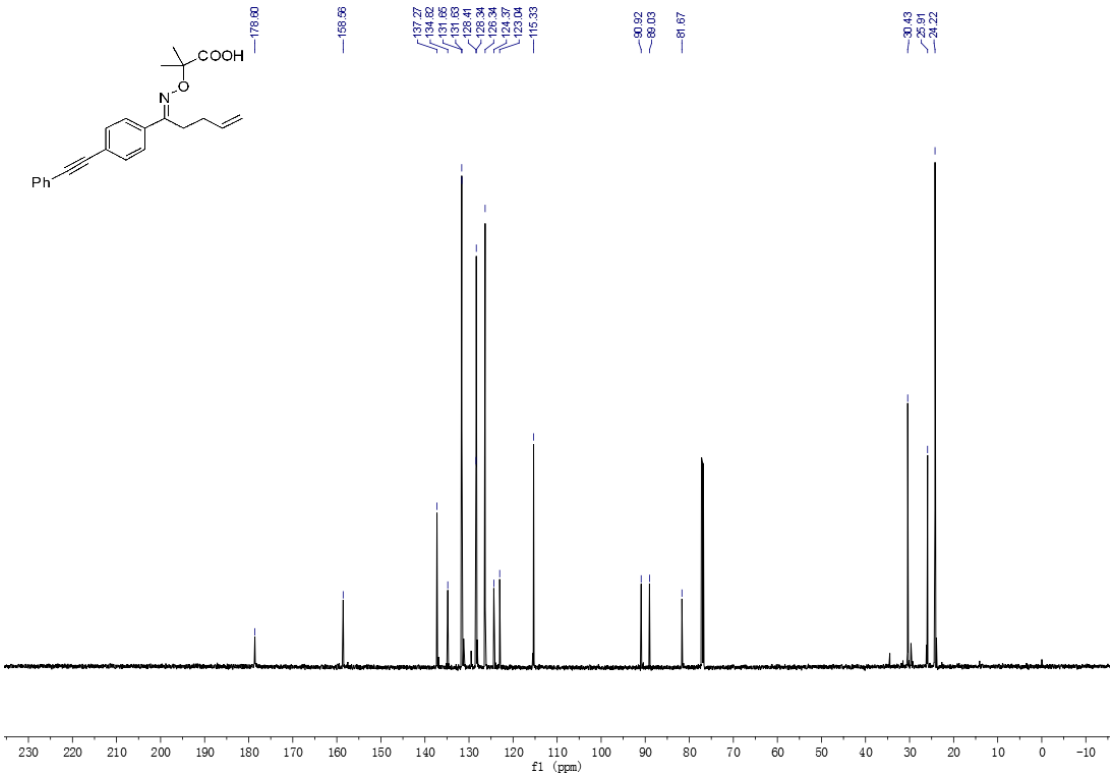

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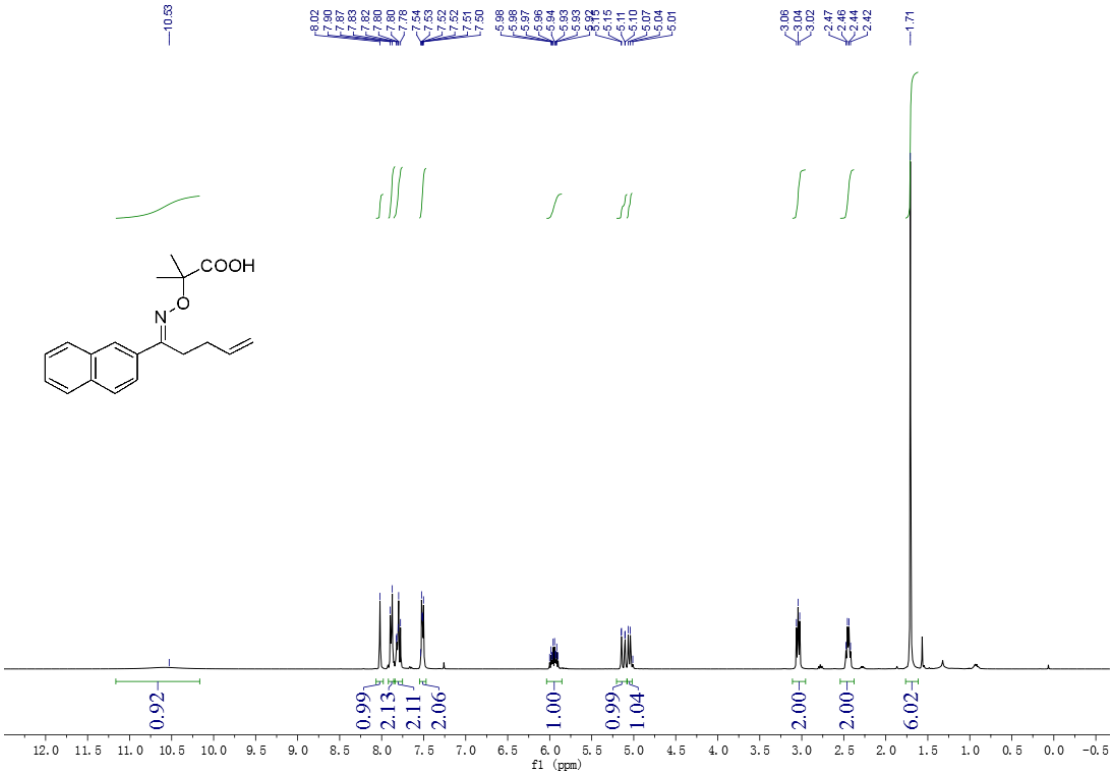

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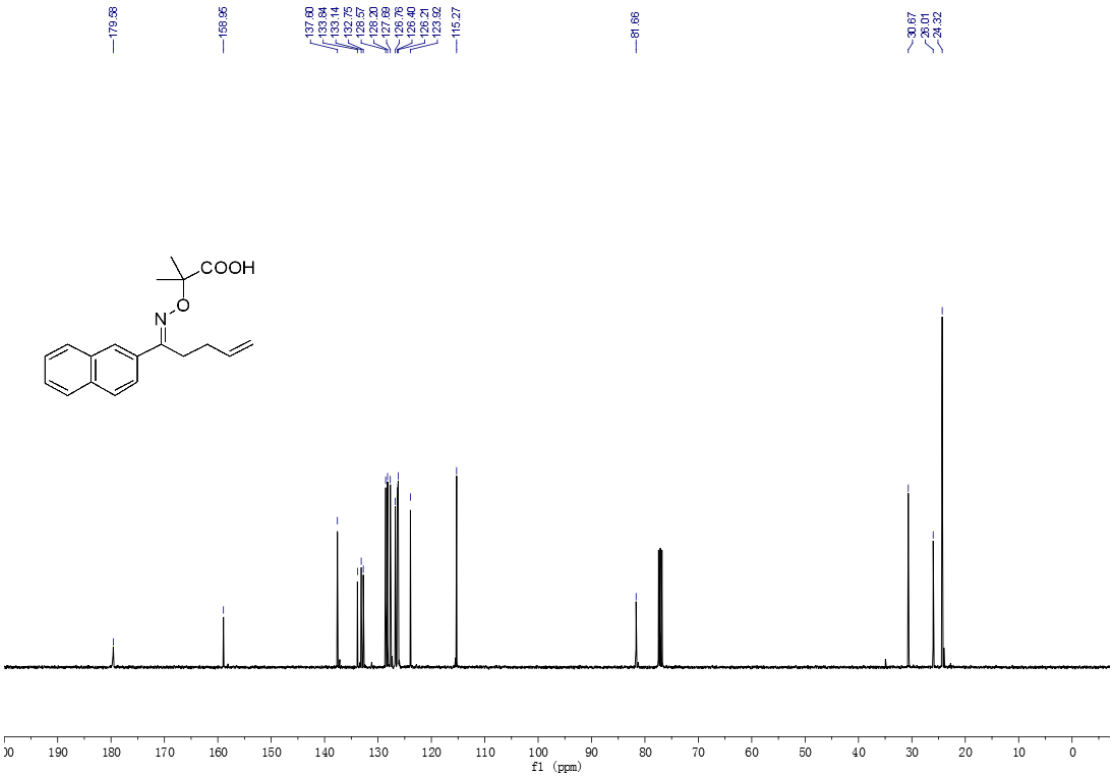
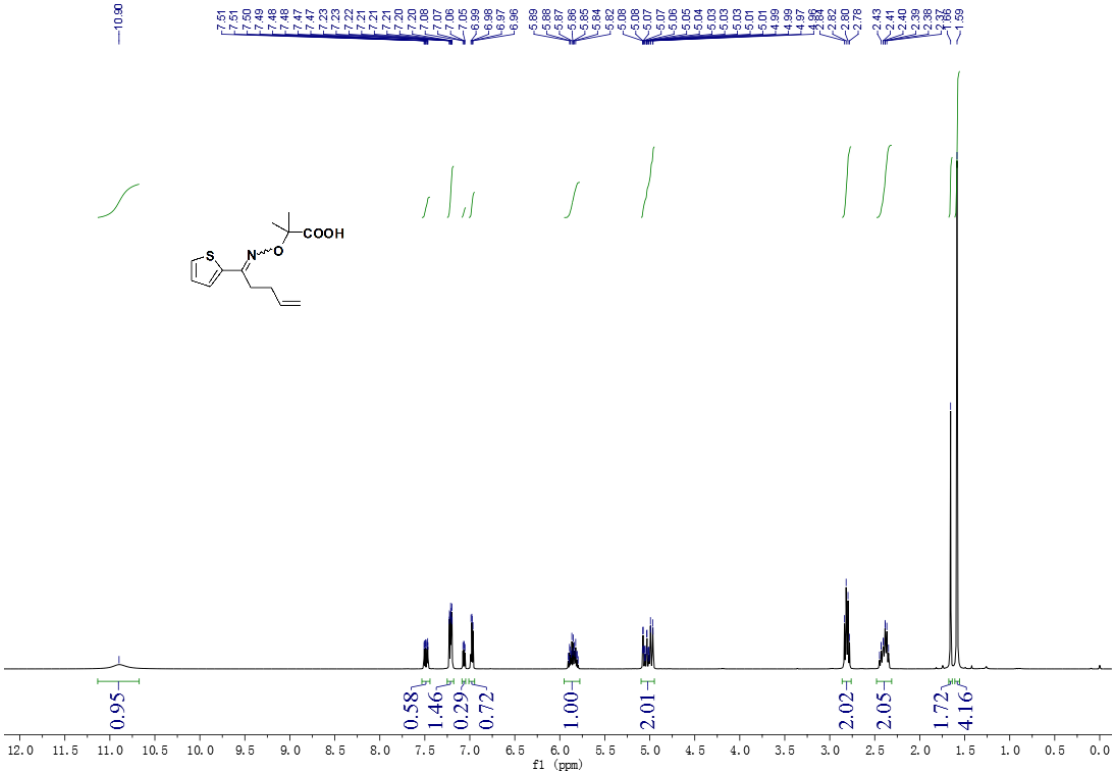


^{1}H NMR of **1ch**



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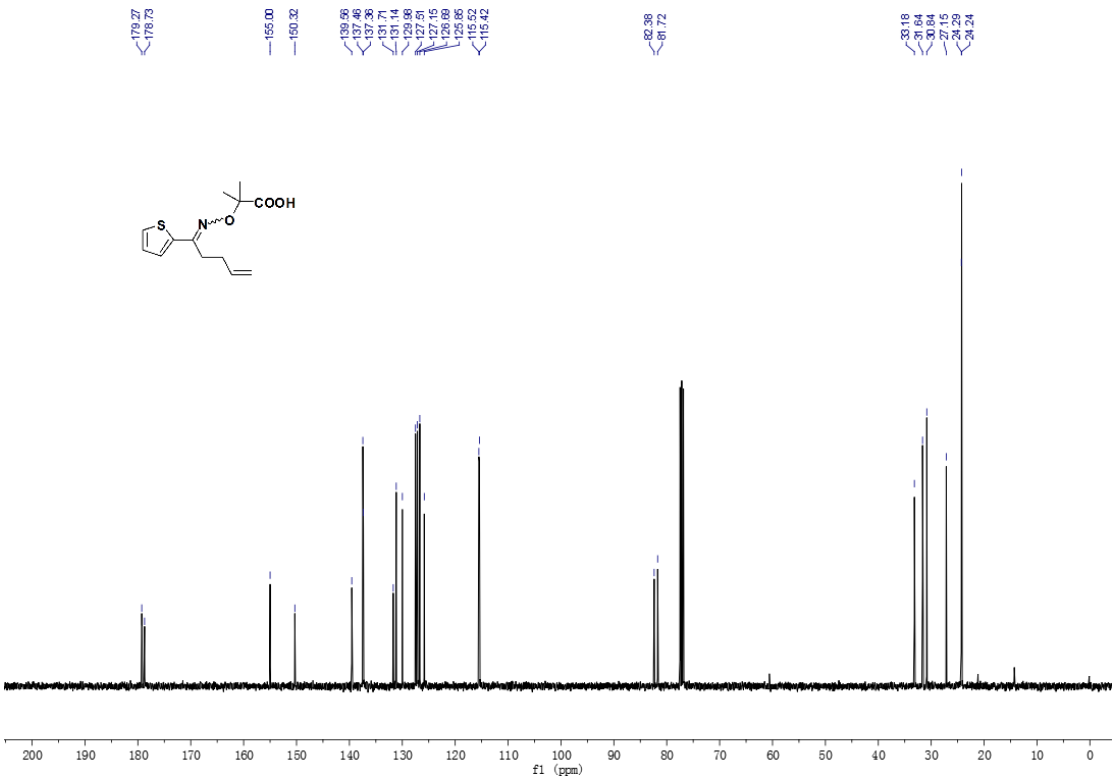
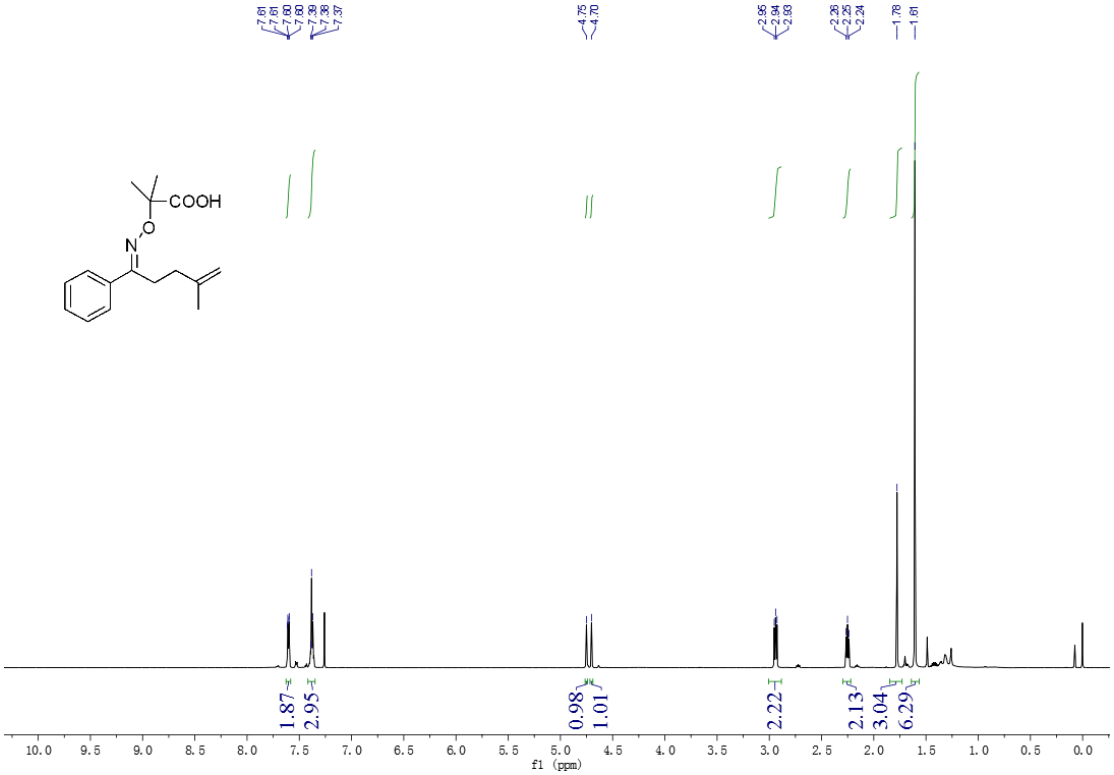


^{1}H NMR of **1ci**



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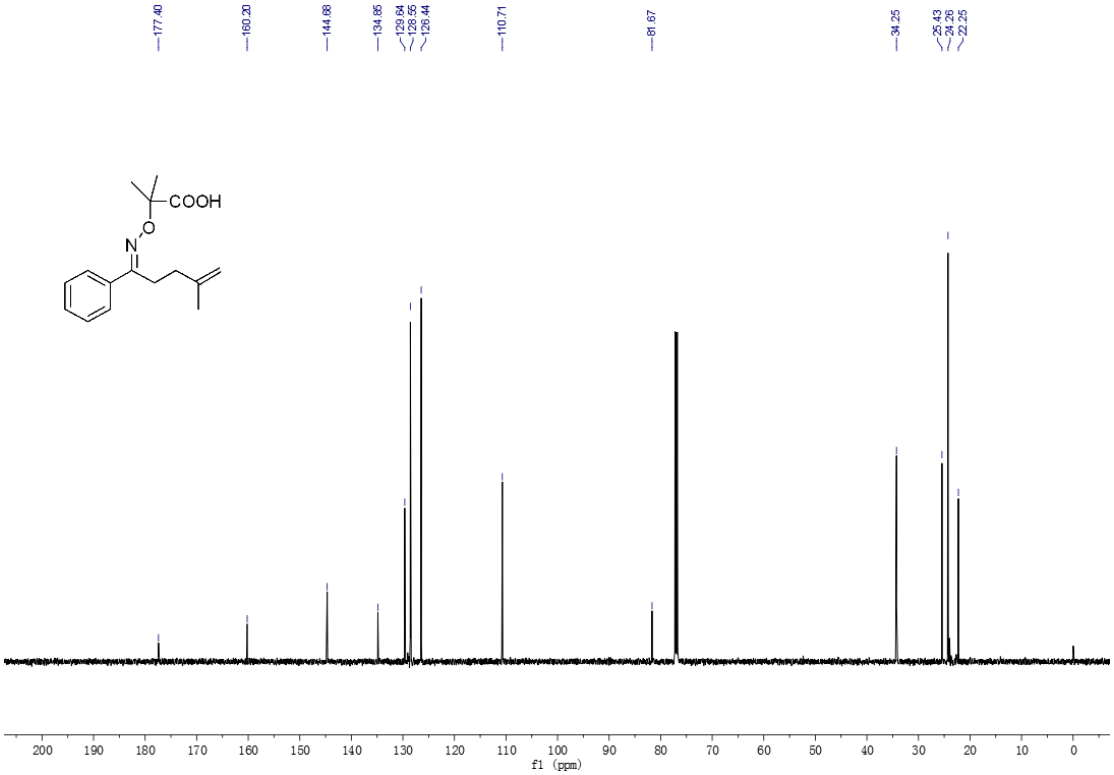
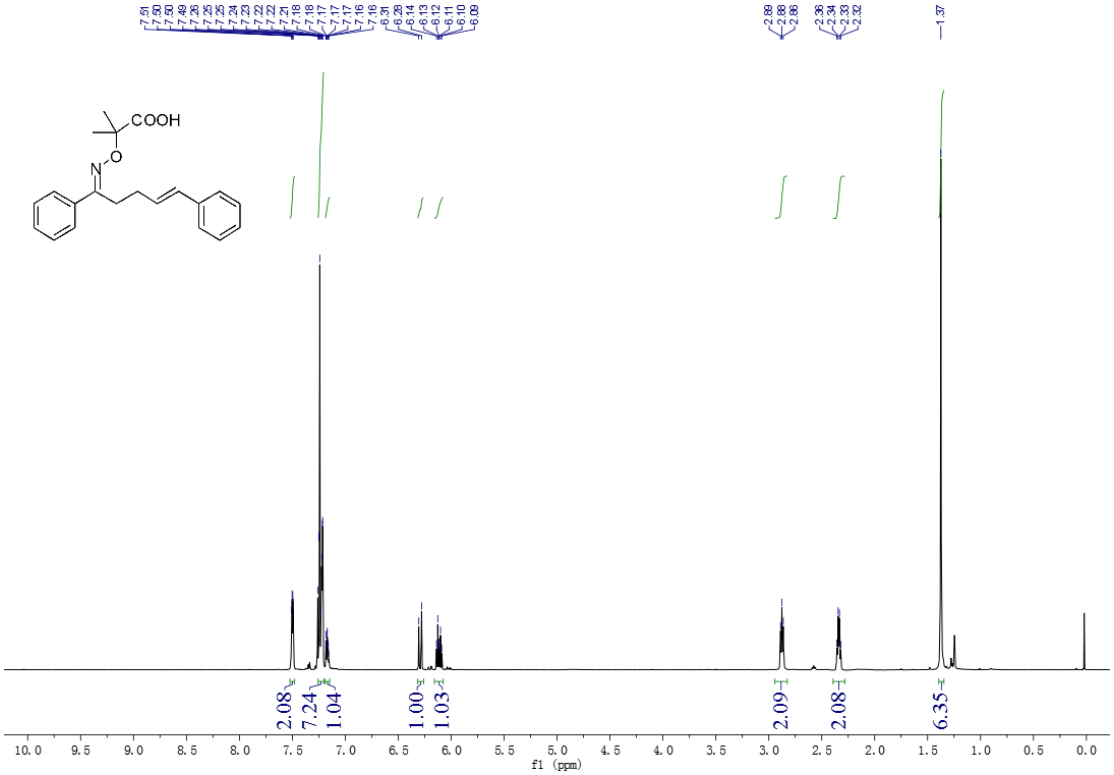


^{1}H NMR of **1cj**



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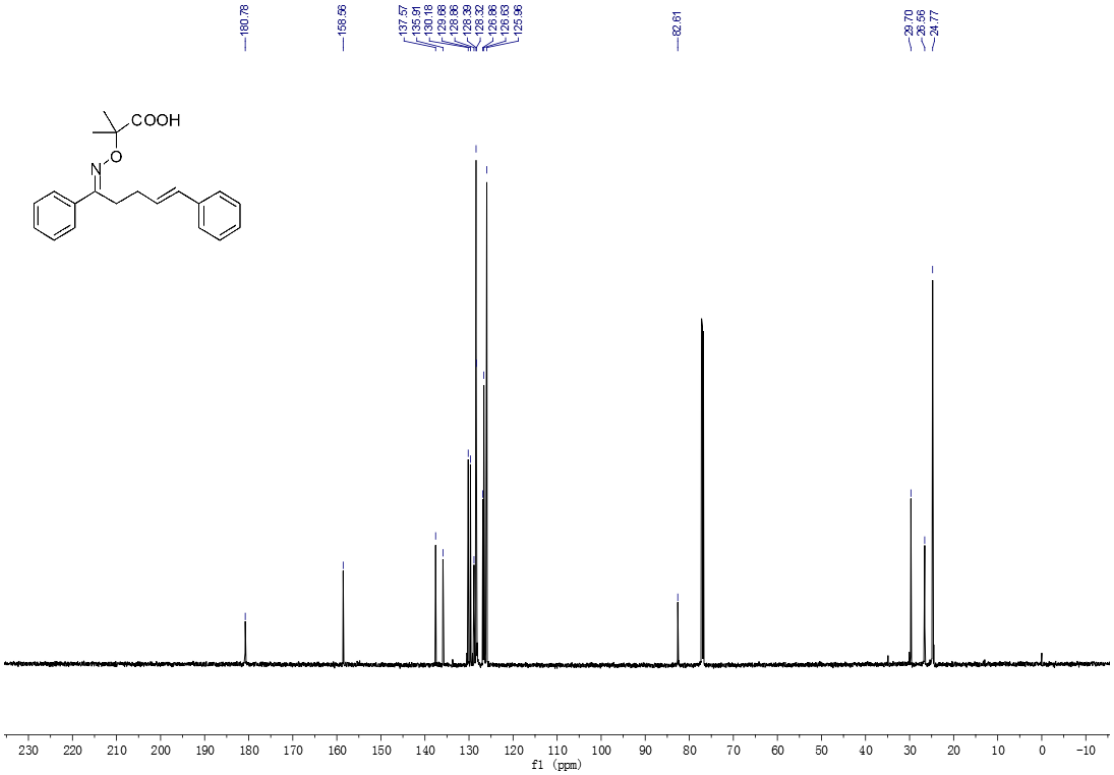
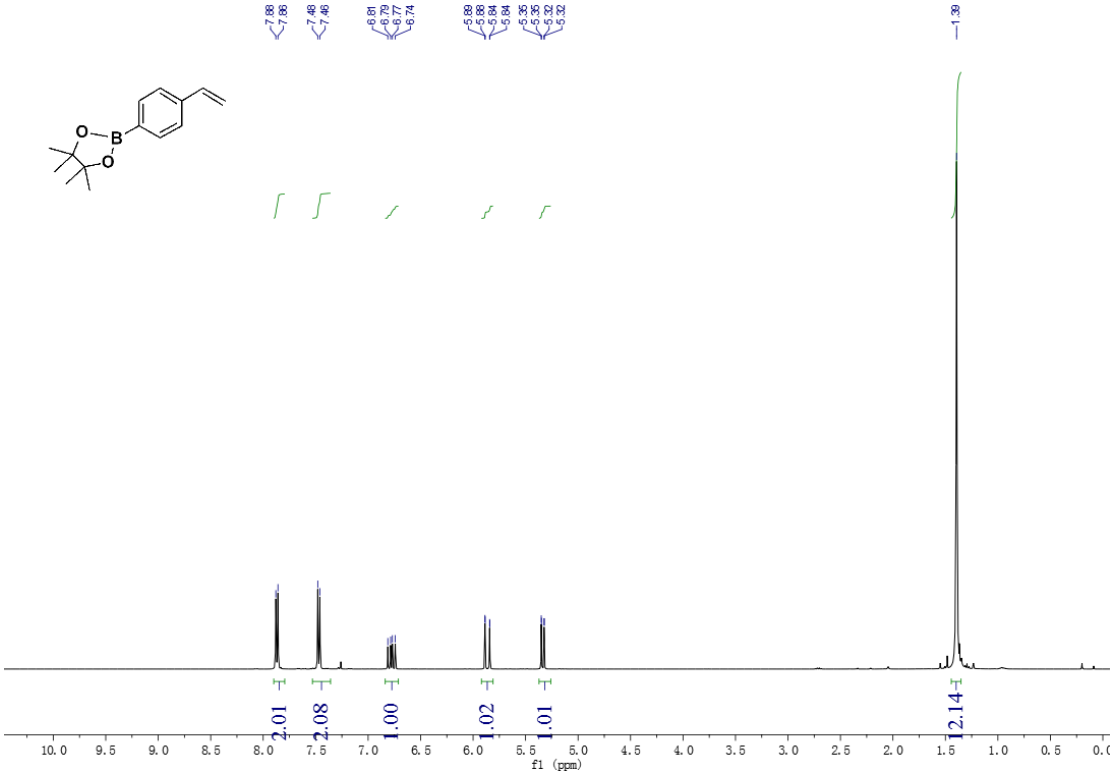
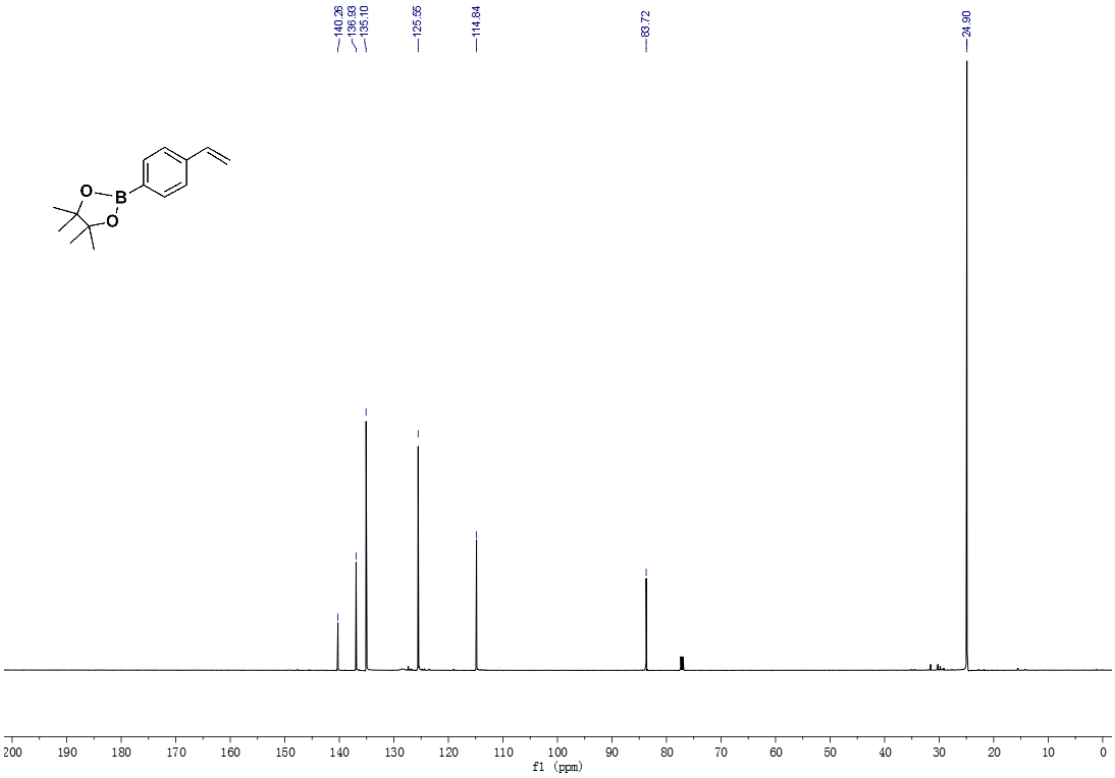


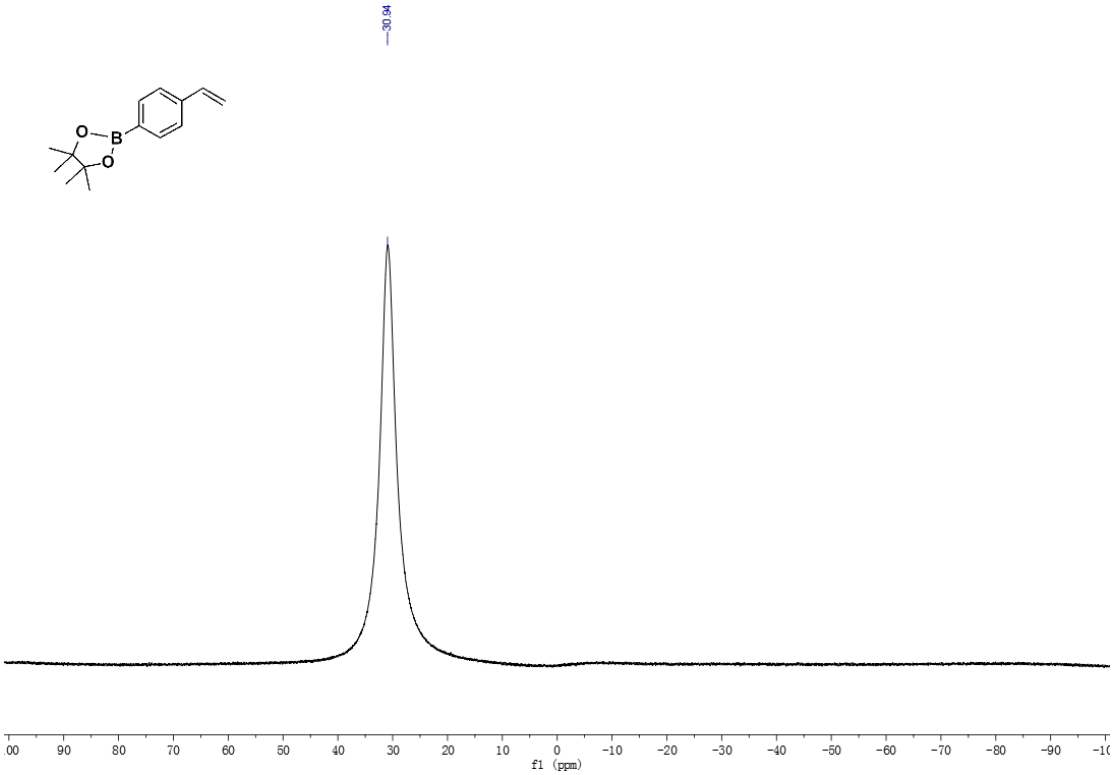
^{1}H NMR of **4a**



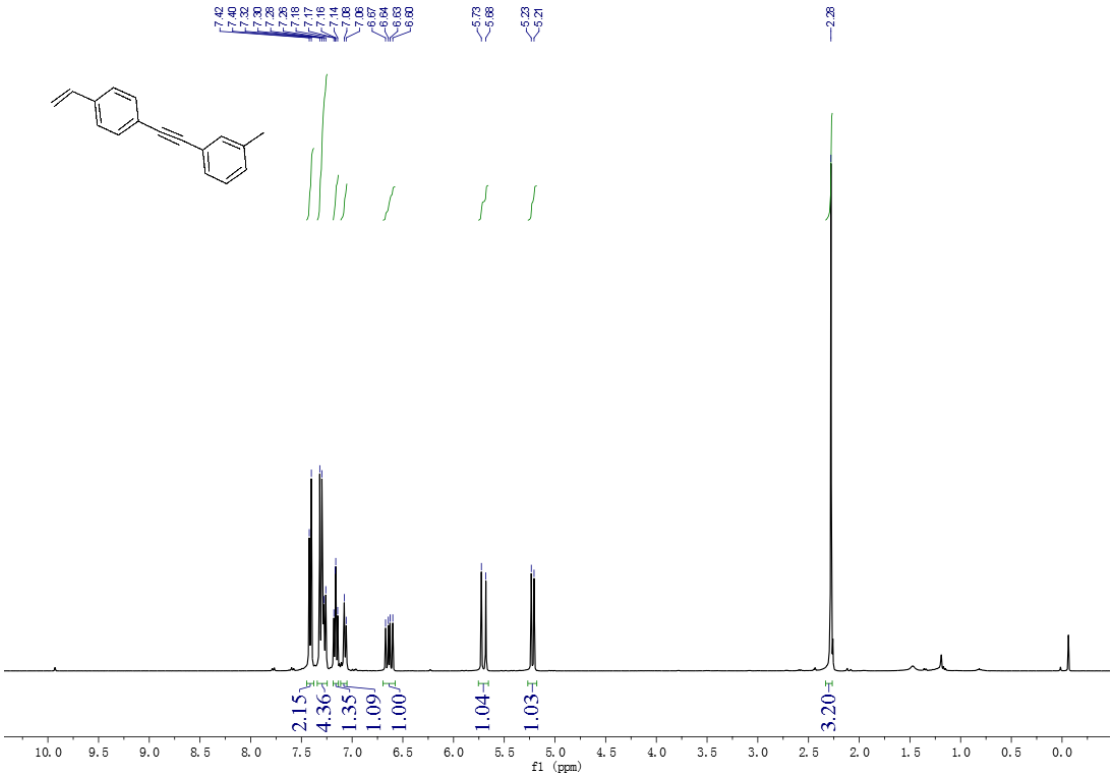
^{13}C NMR of **4a**



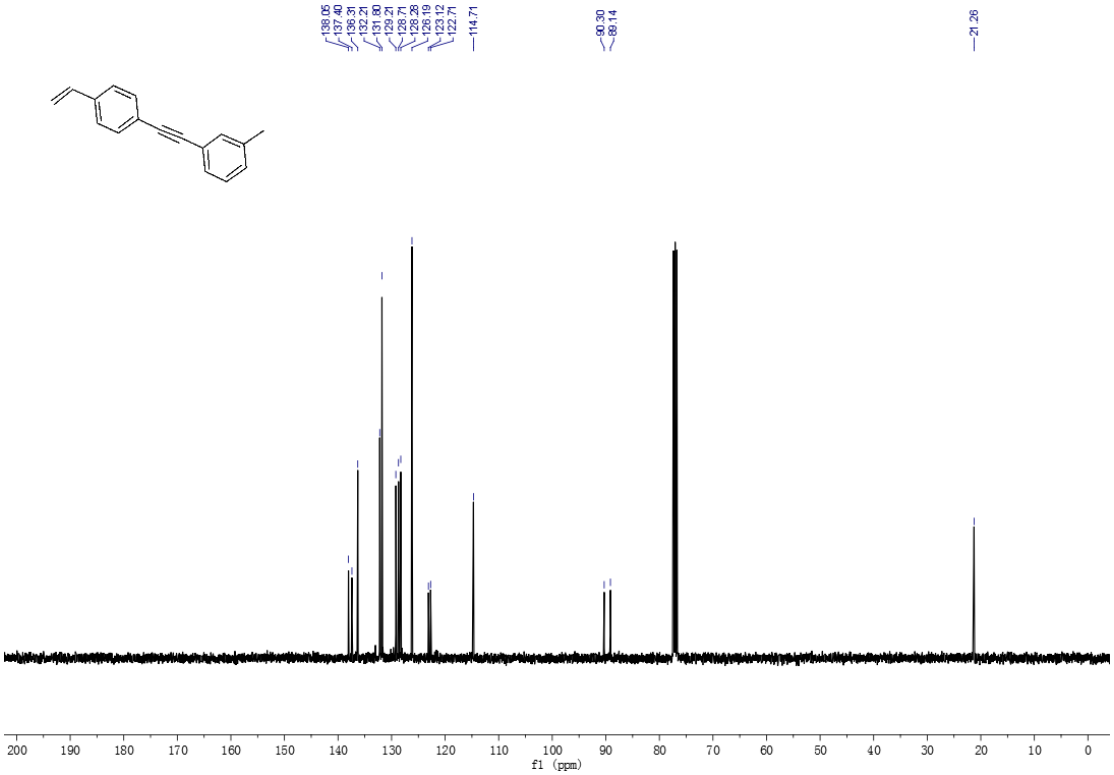
^{11}B NMR of **4a**



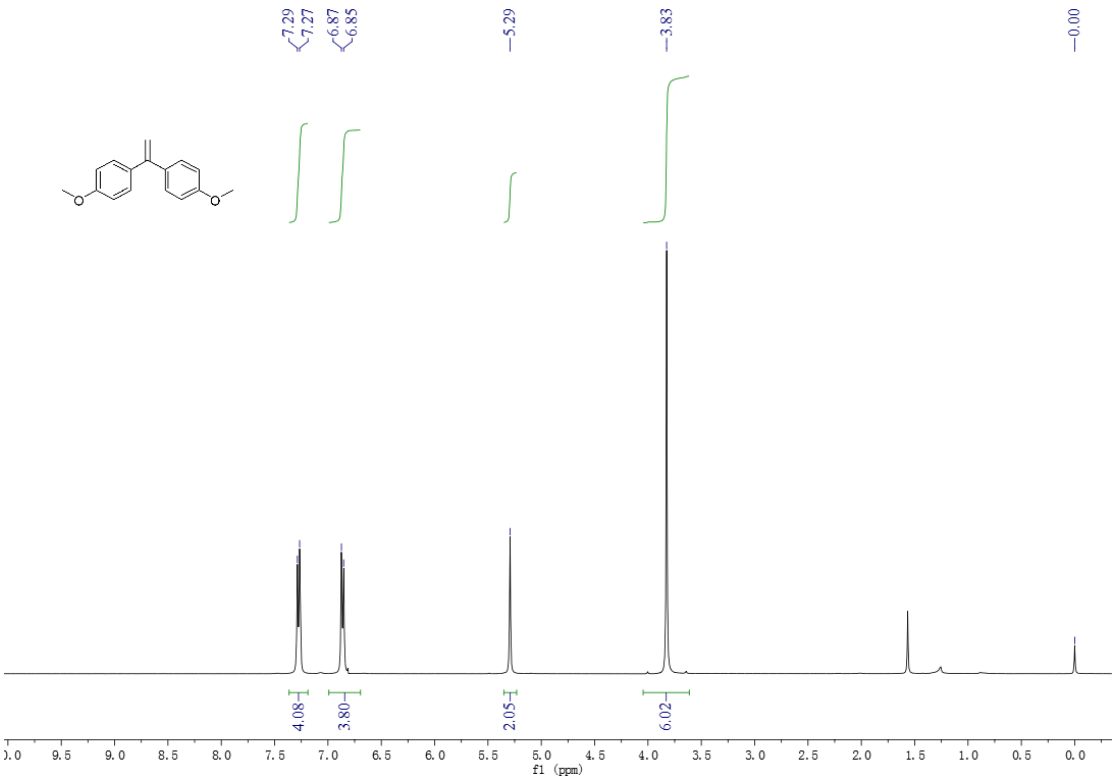
^{1}H NMR of **4b**



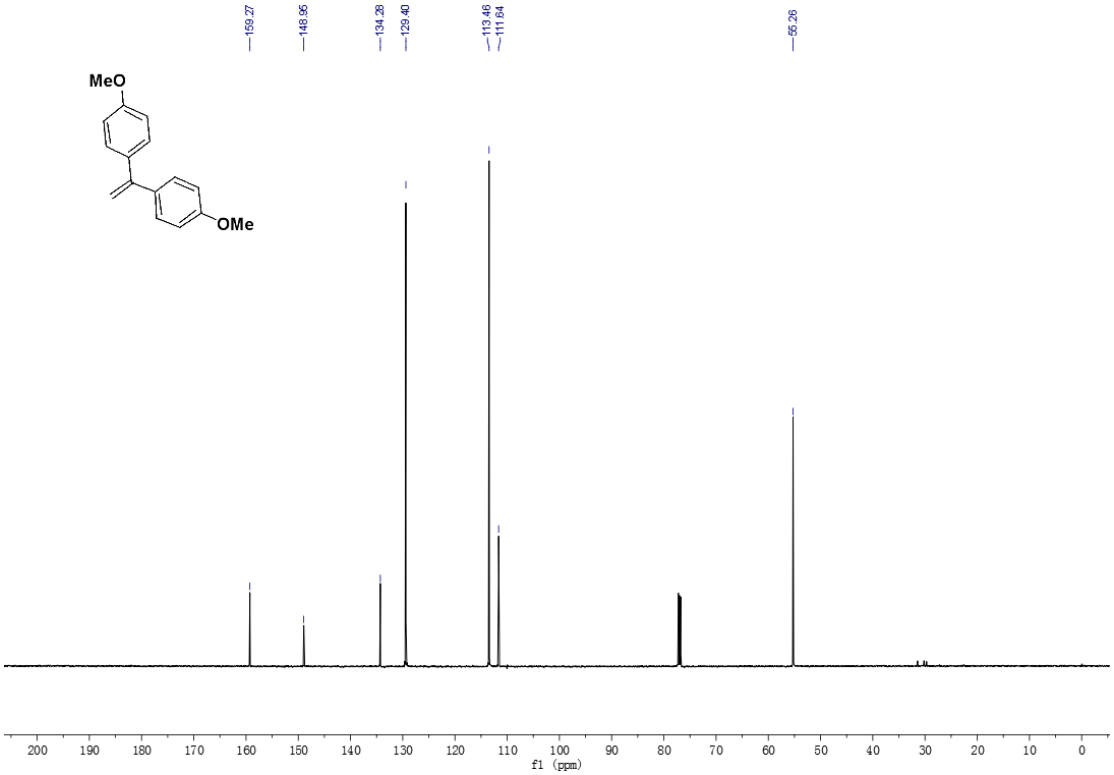
^{13}C NMR of **4b**



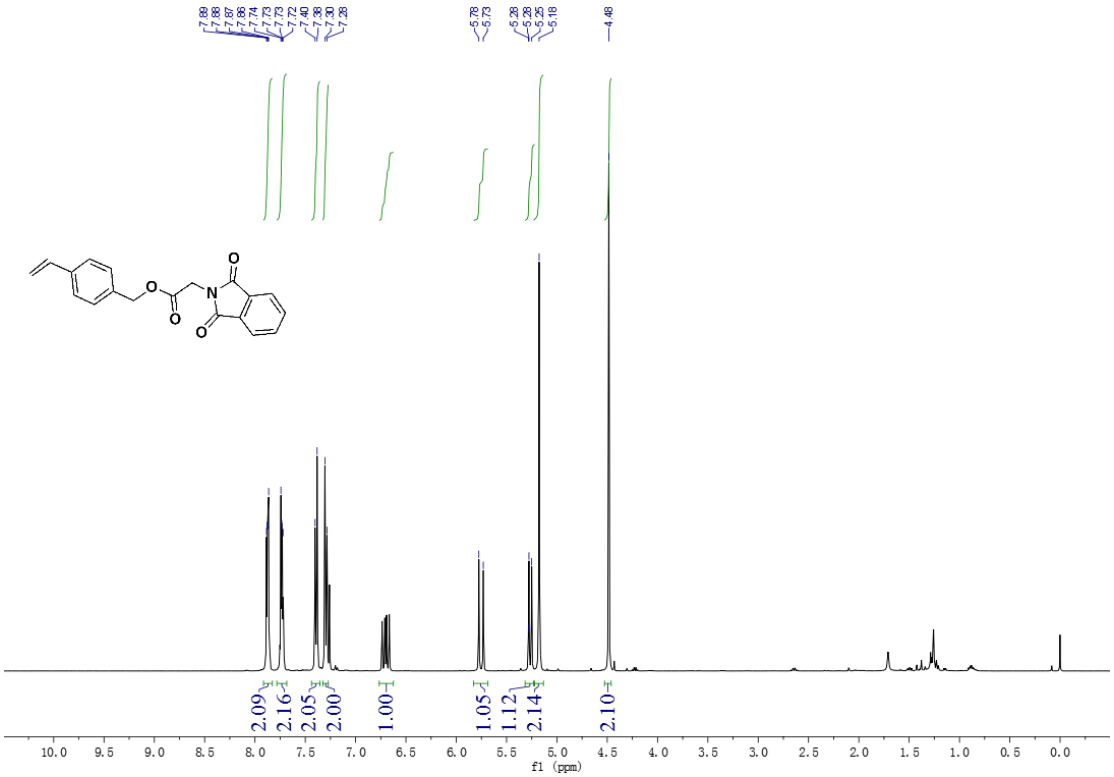
^{1}H NMR of **4c**



^{13}C NMR of **4c**



^{1}H NMR of **4d**



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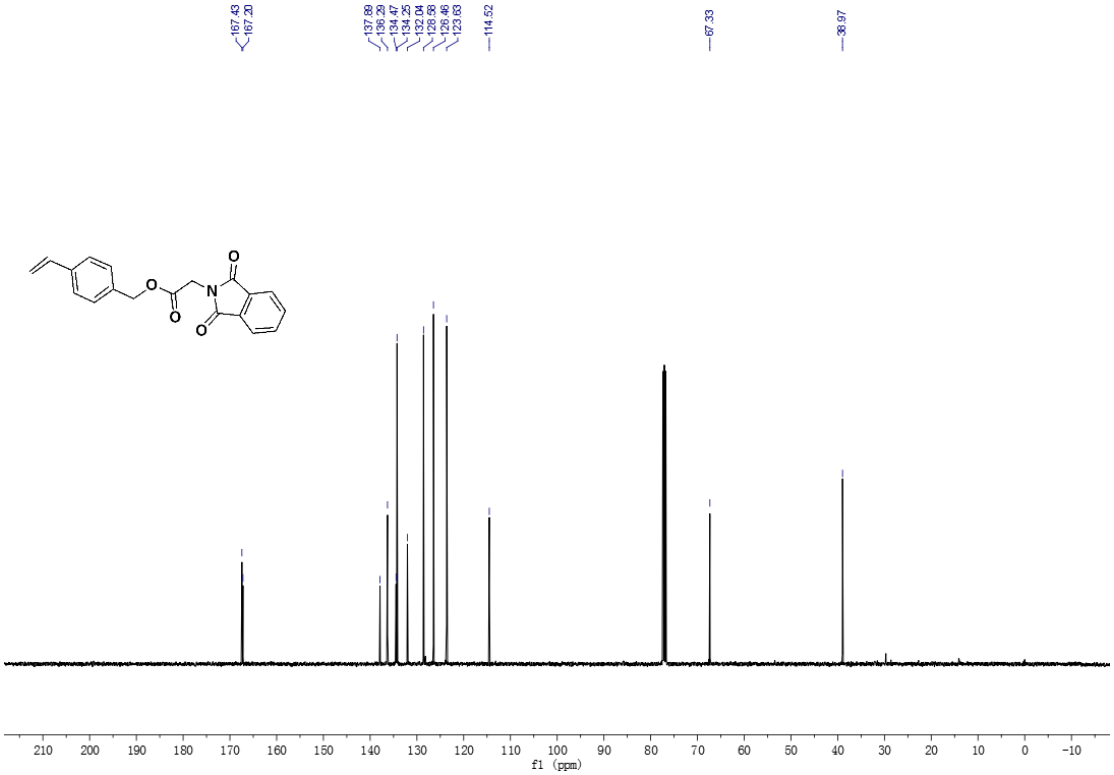

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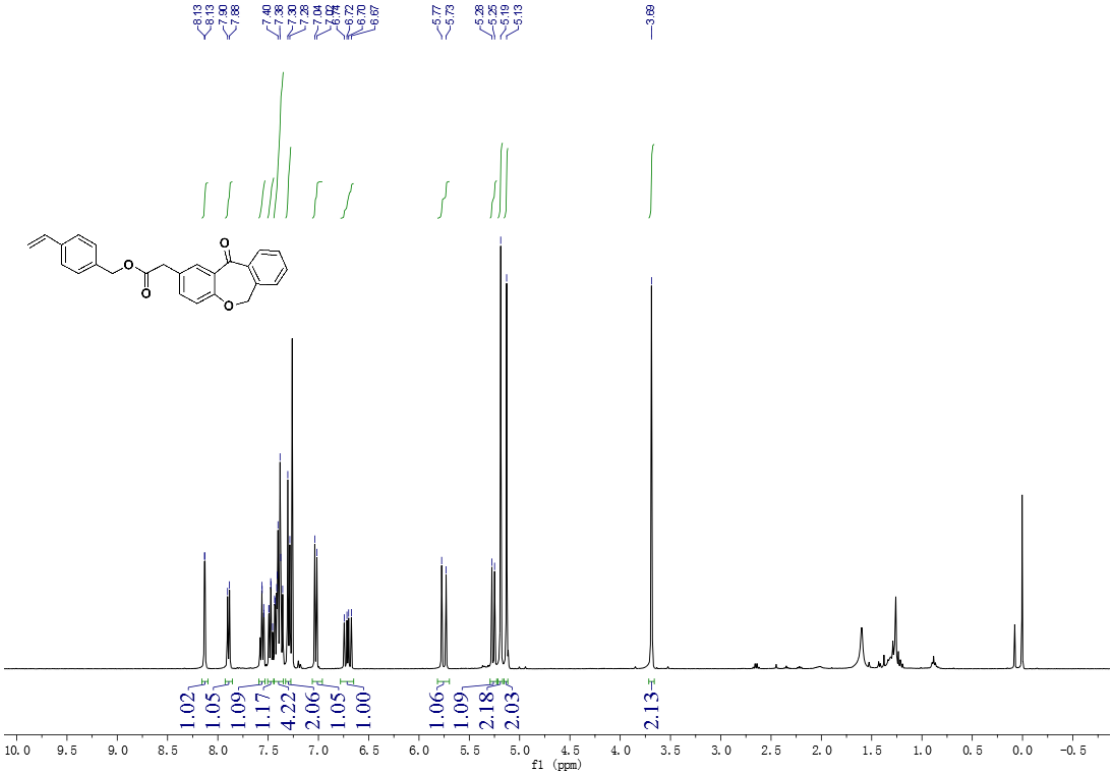

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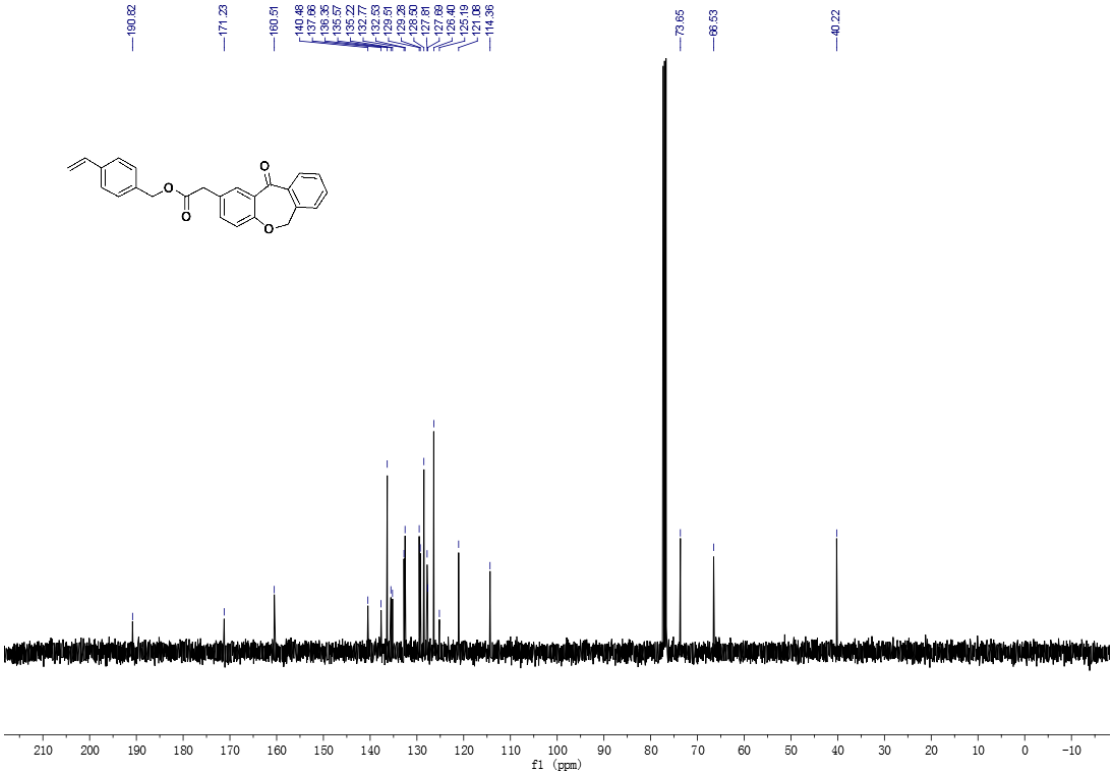
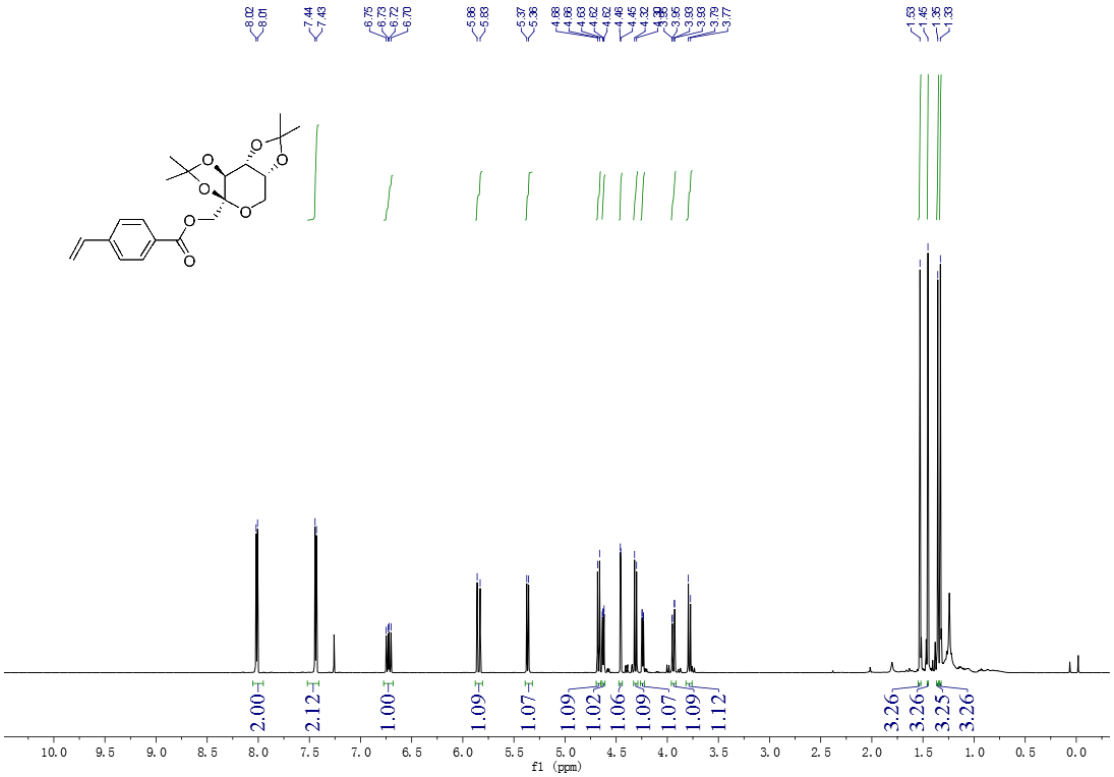


^{1}H NMR of **4f**



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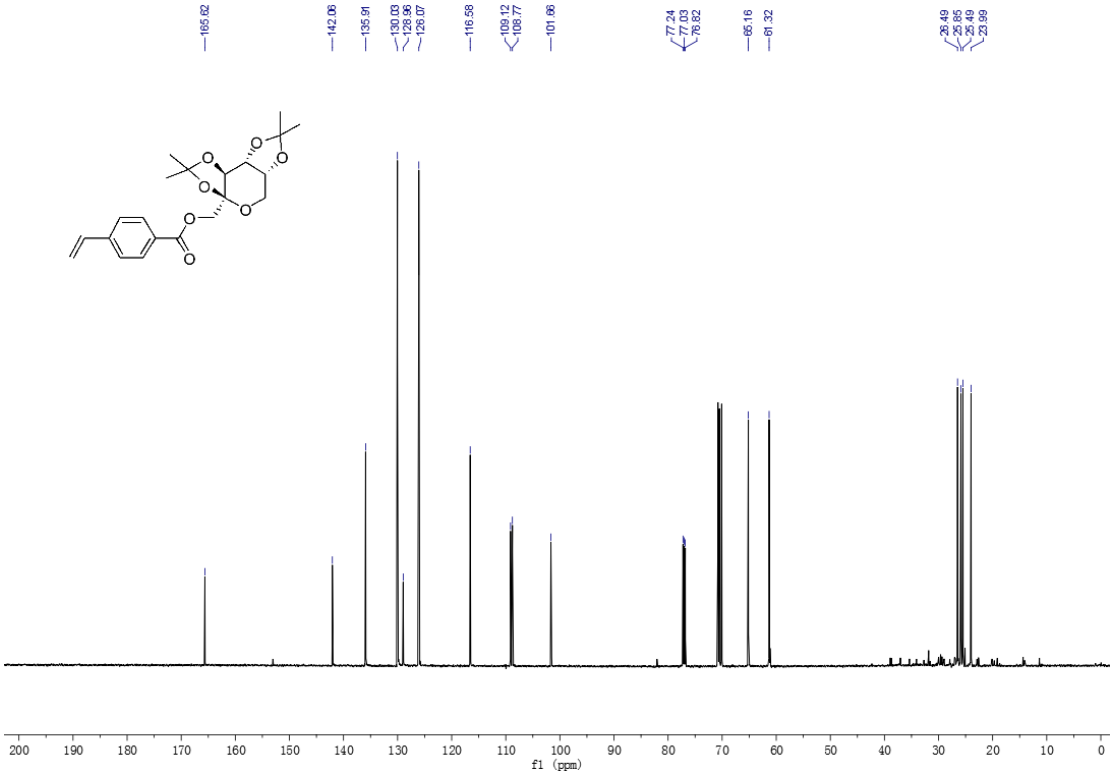
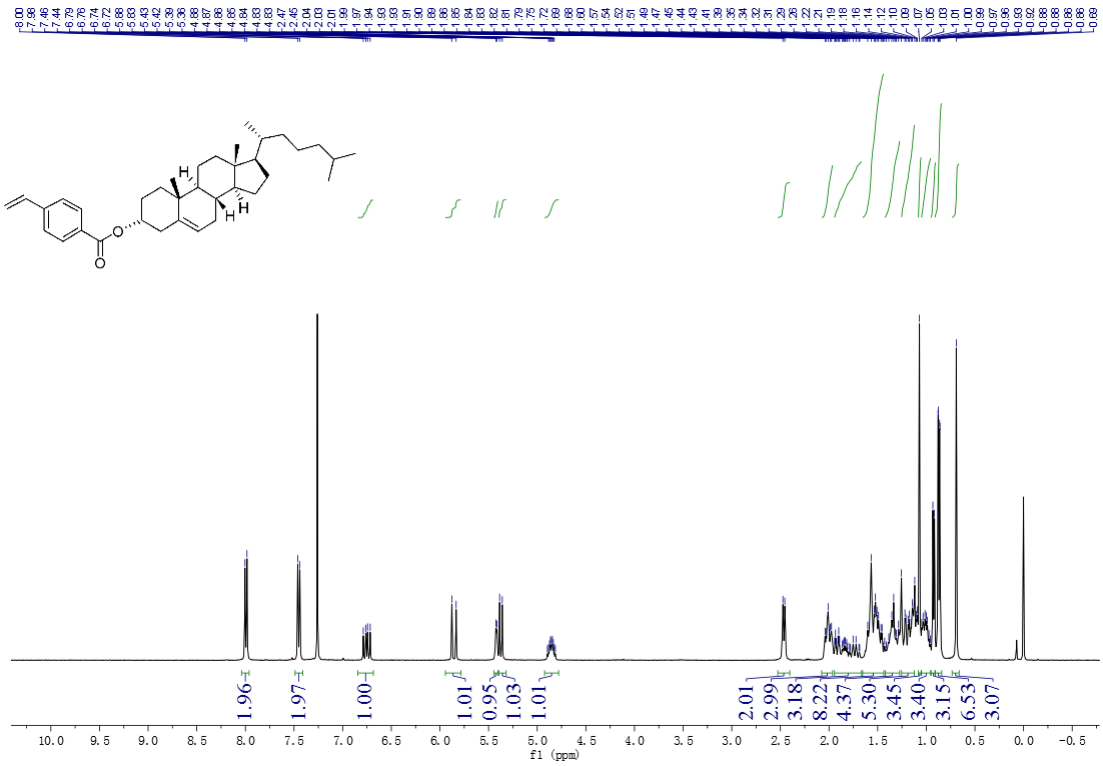


^{1}H NMR of **4g**



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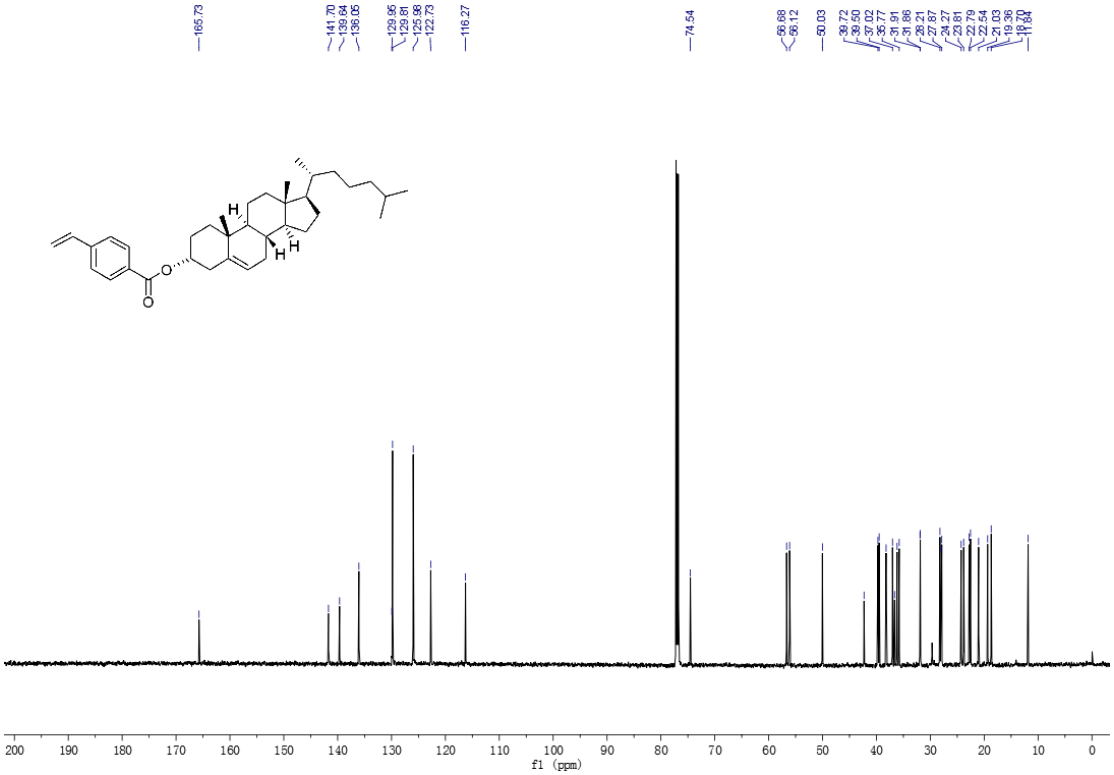
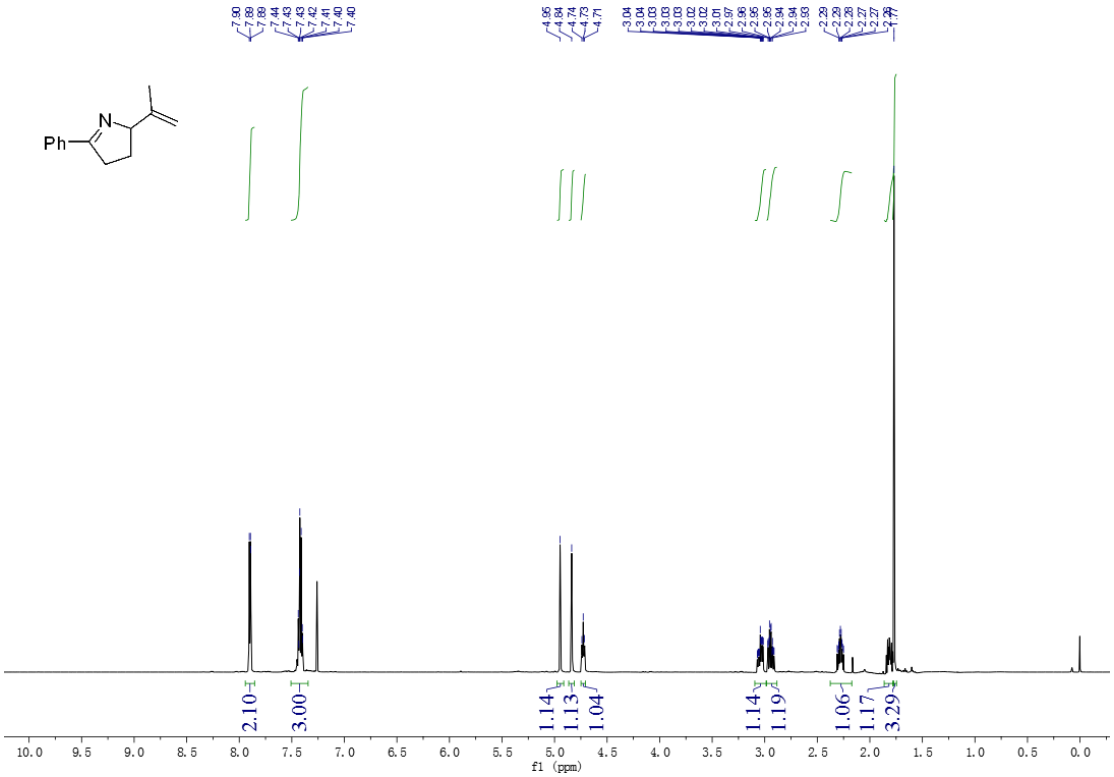
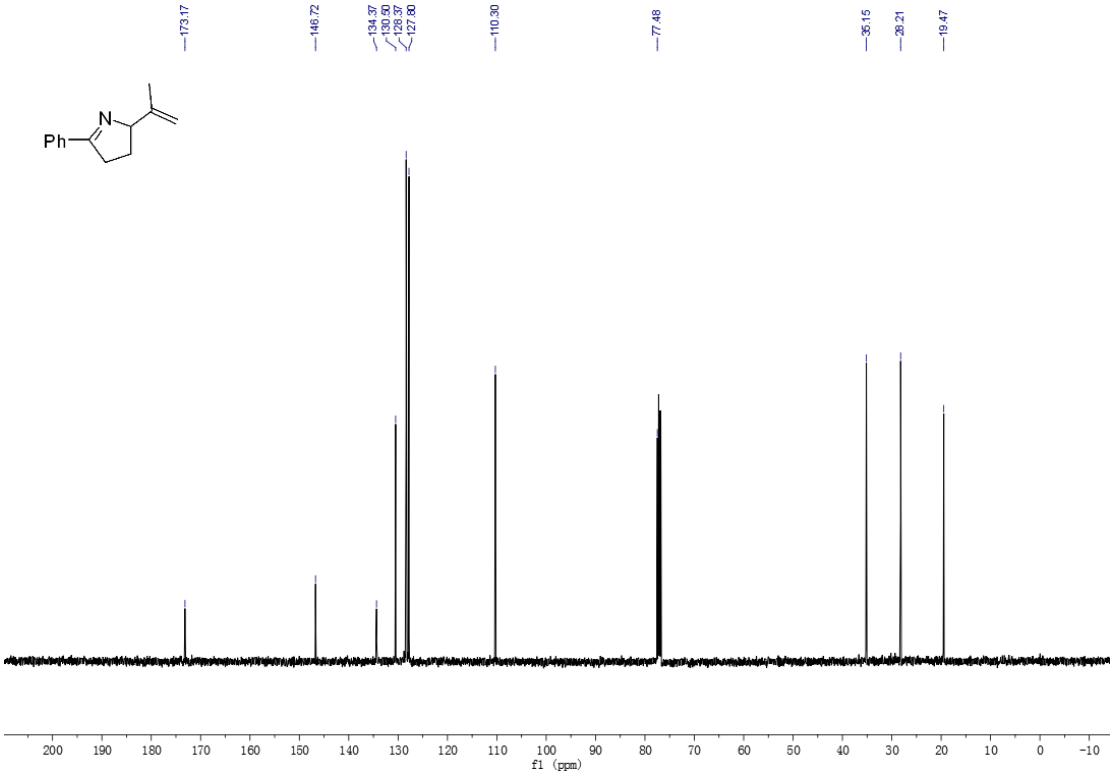


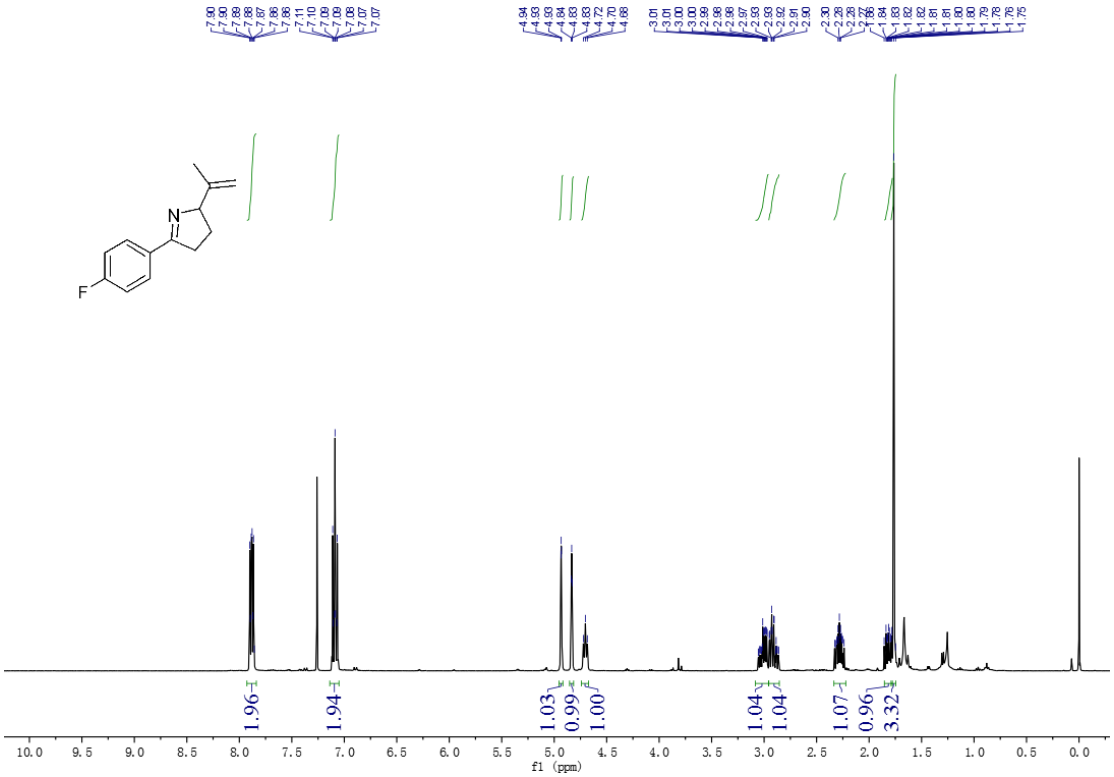
^{1}H NMR of **2a**



^{13}C NMR of **2a**



^{1}H NMR of **2b**



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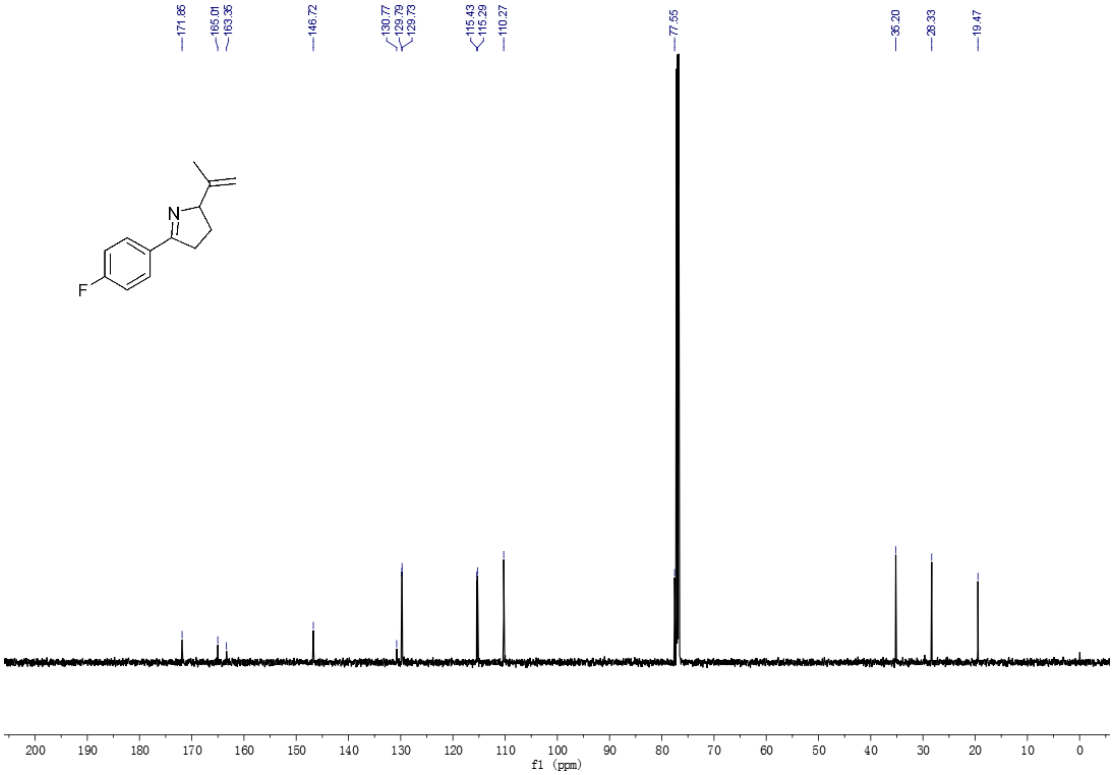

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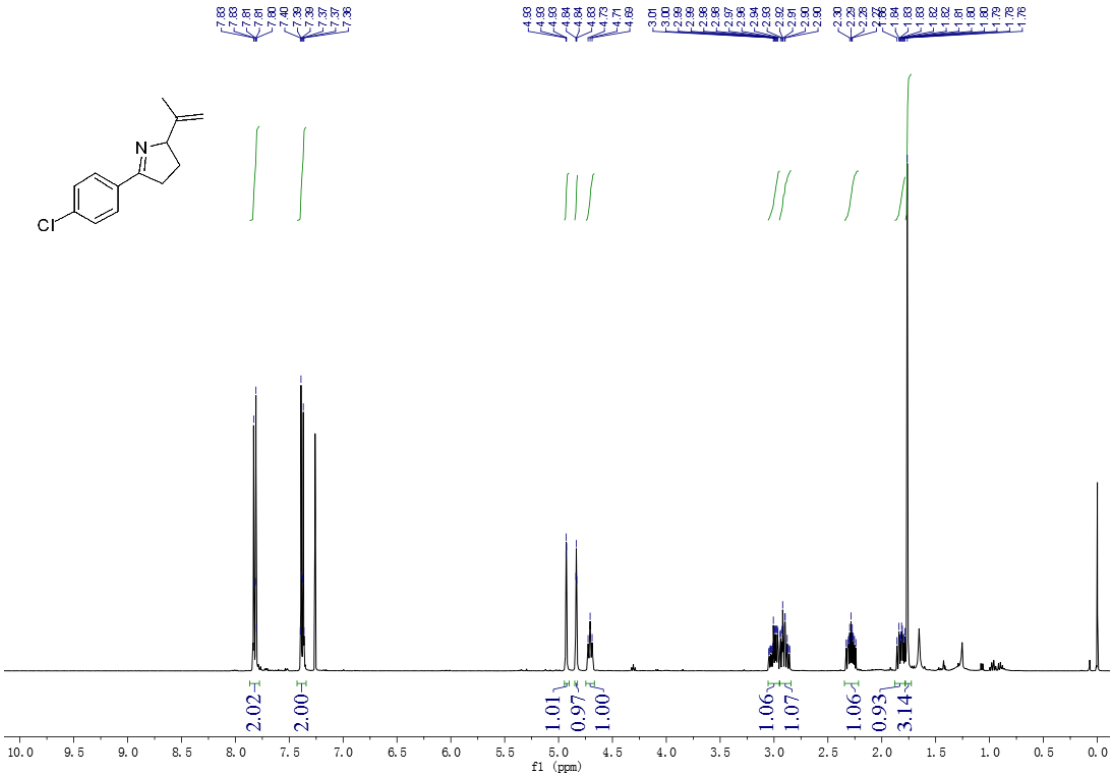
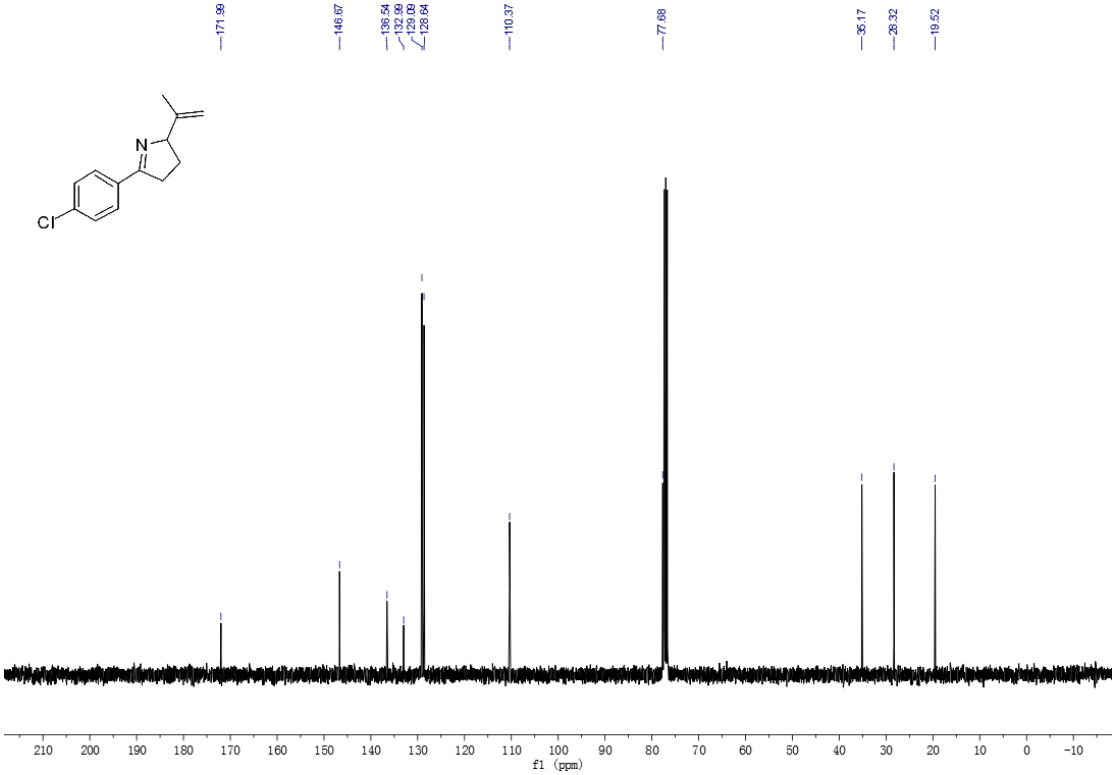
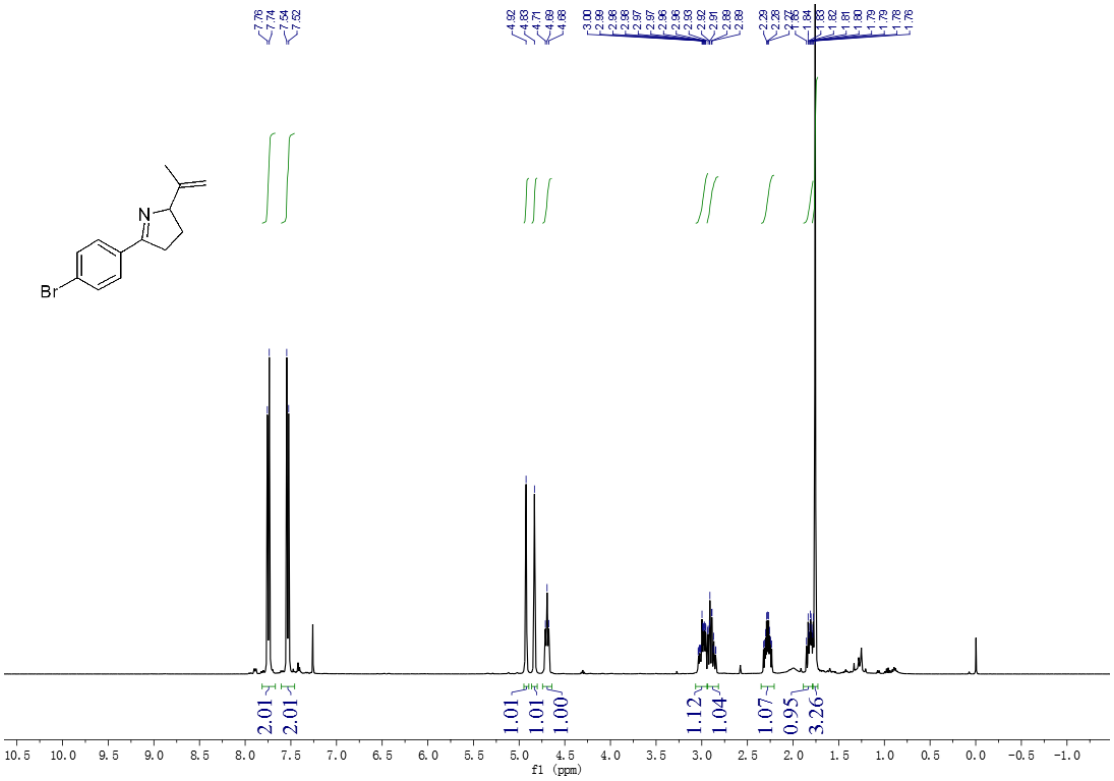


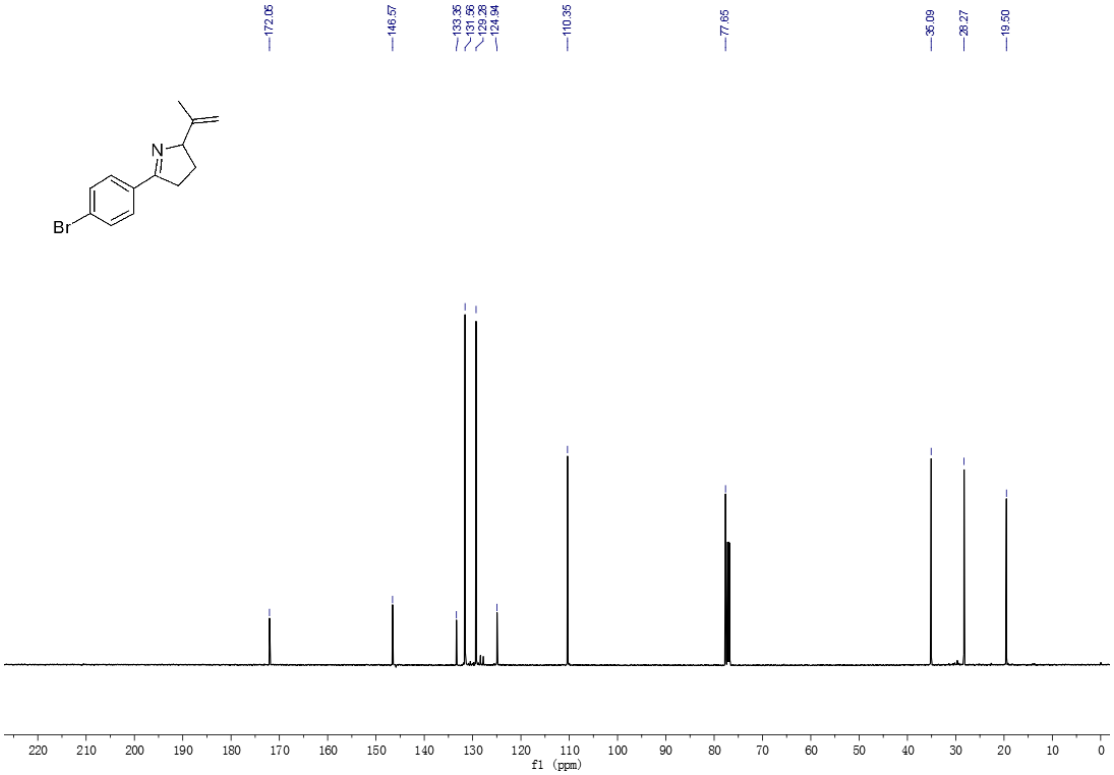
^{13}C NMR of **2c**



^{1}H NMR of **2d**



^{13}C NMR of **2d**



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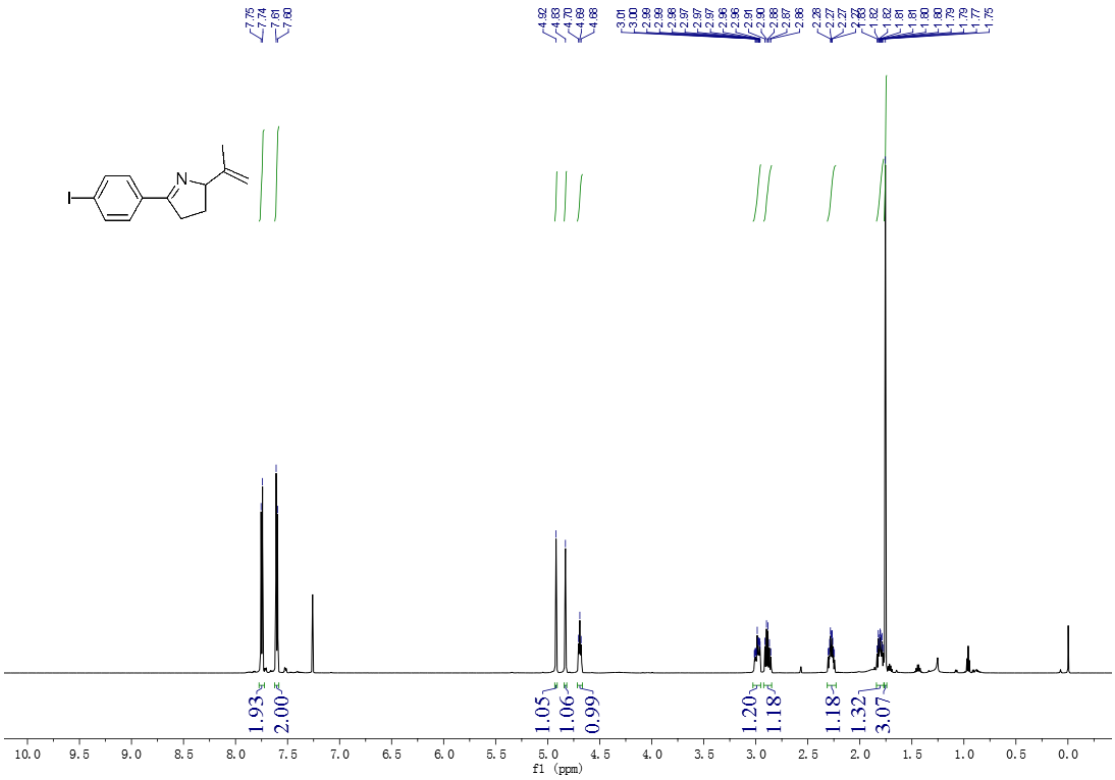
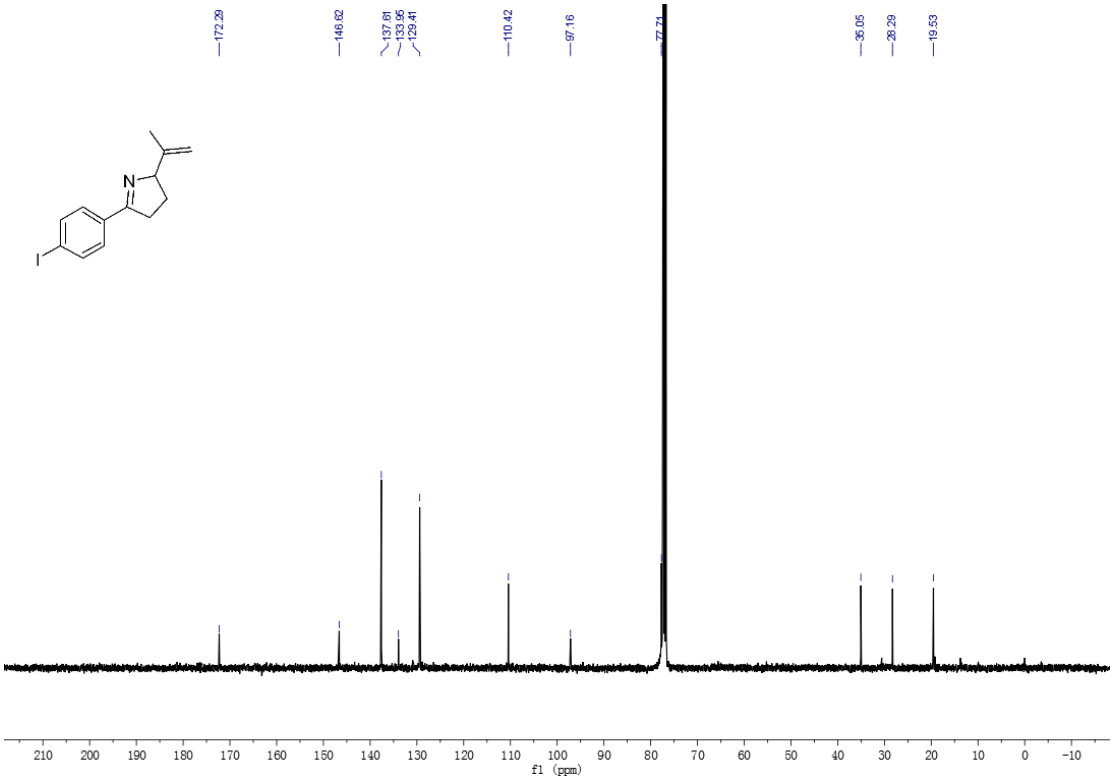
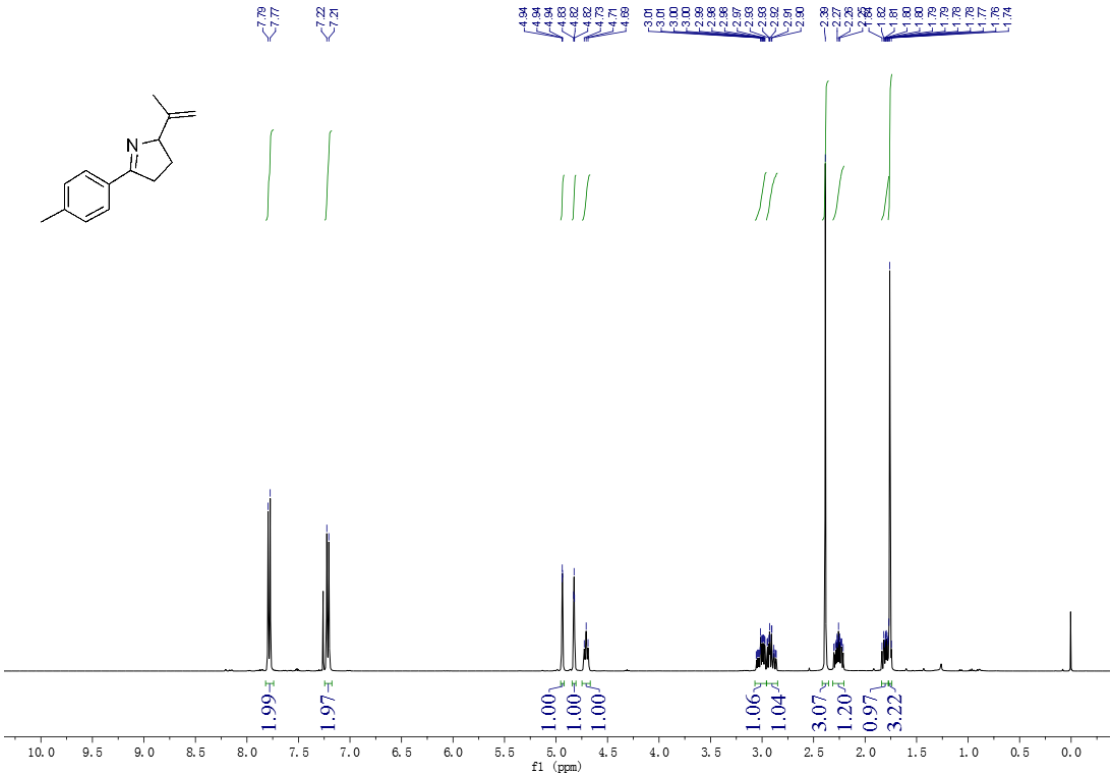


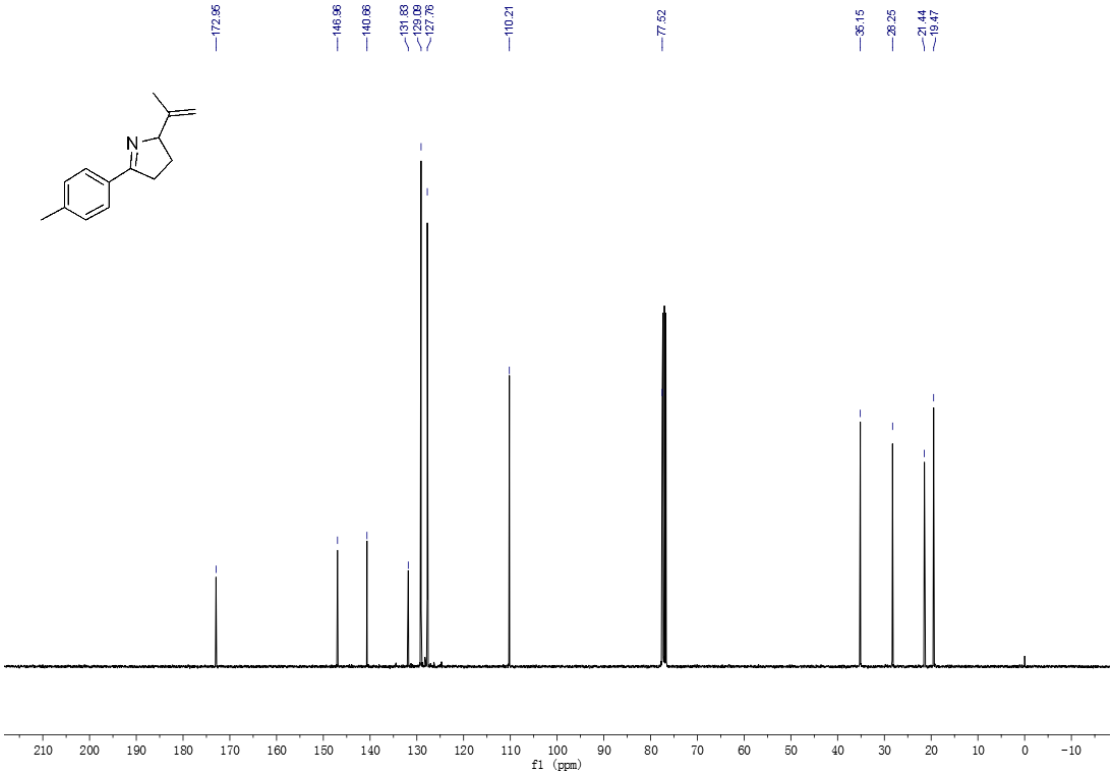
^{13}C NMR of **2e**



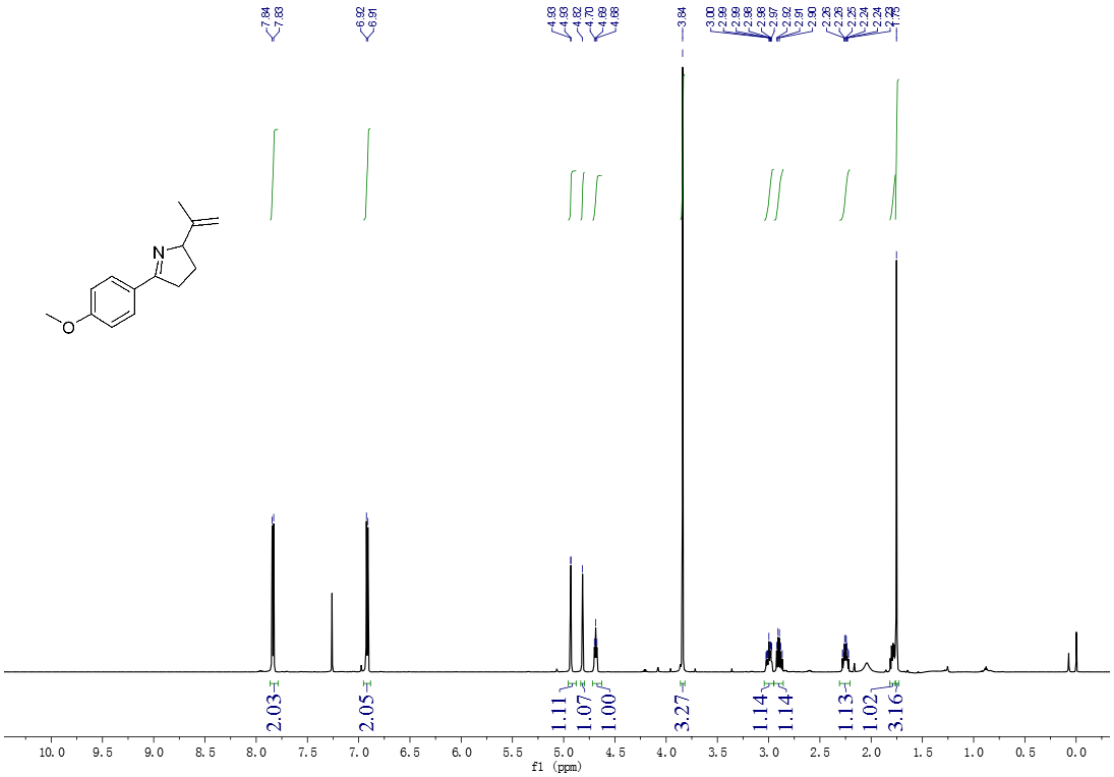
^{1}H NMR of **2f**



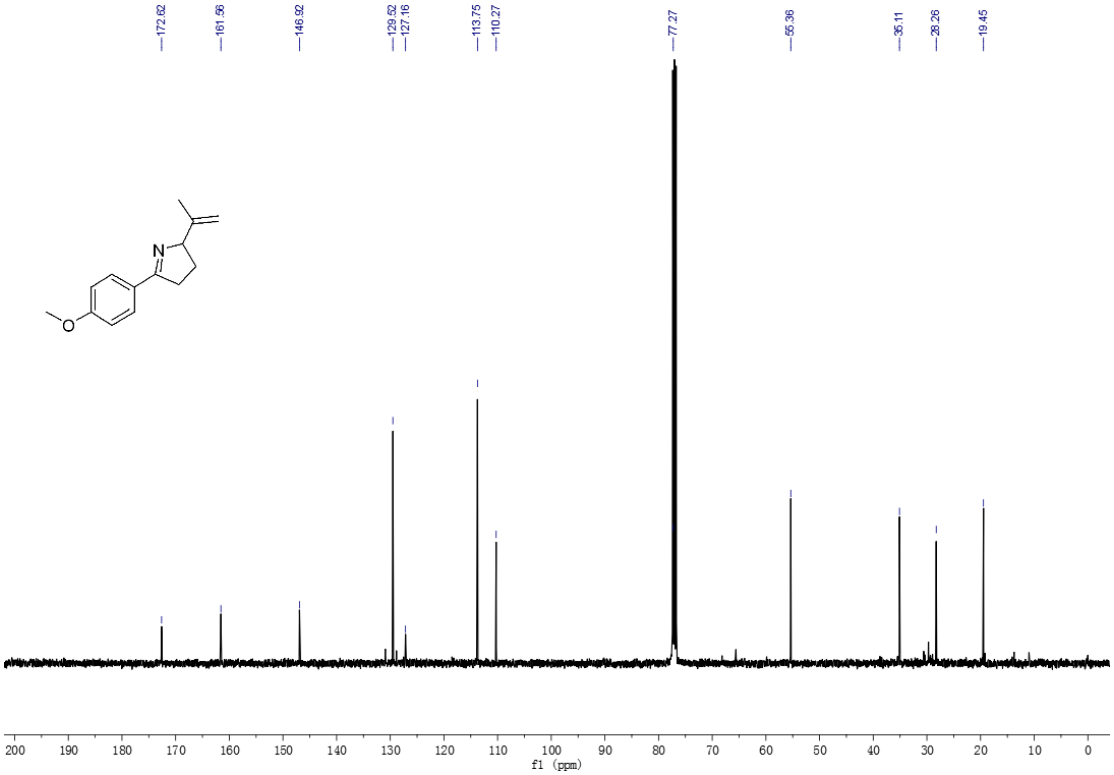
^{13}C NMR of **2f**



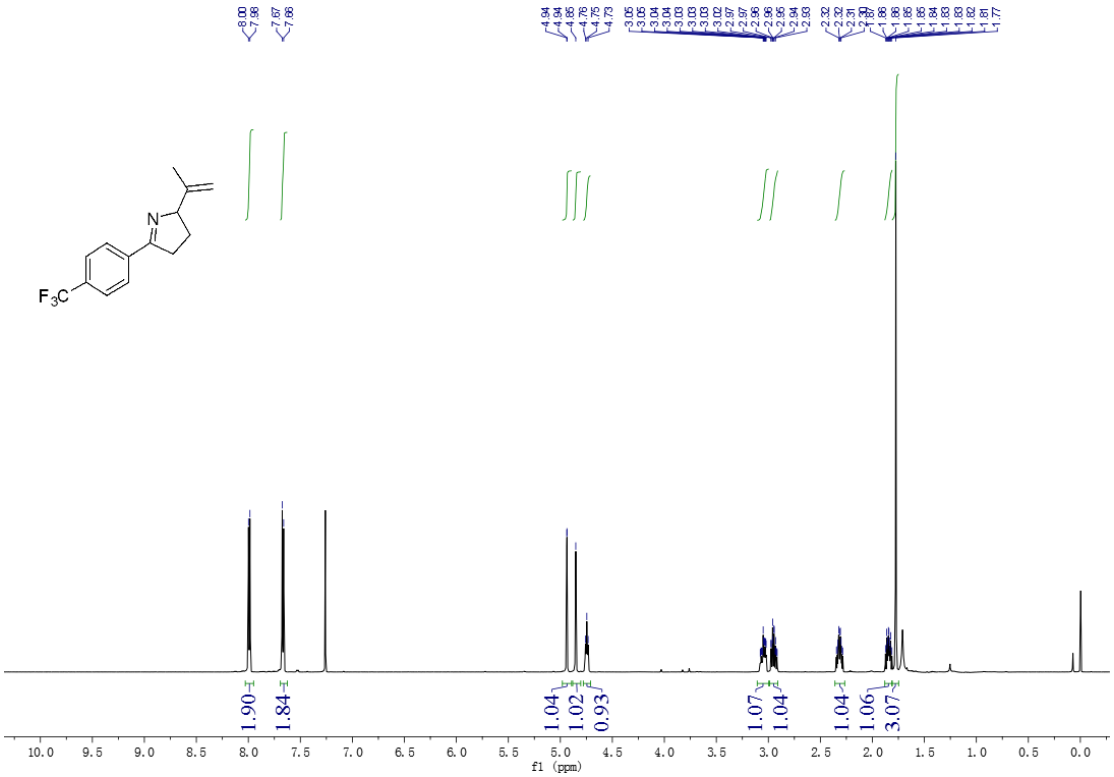
^{1}H NMR of **2g**



^{13}C NMR of **2g**



^{1}H NMR of **2h**



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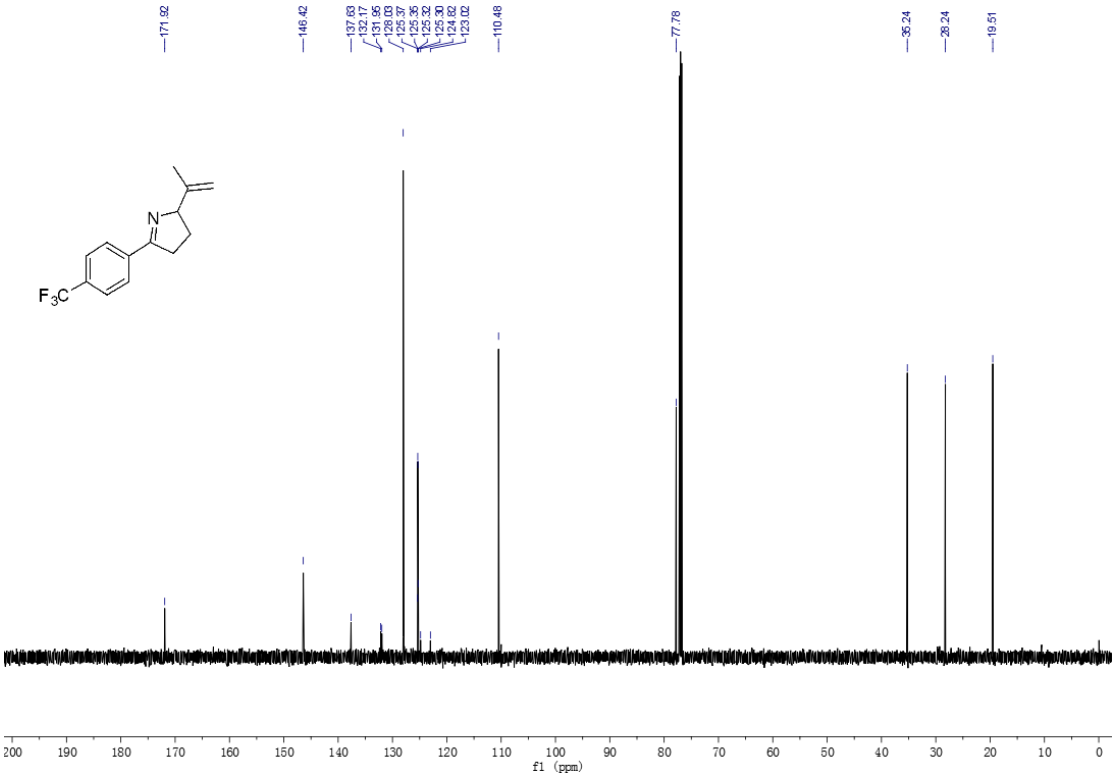
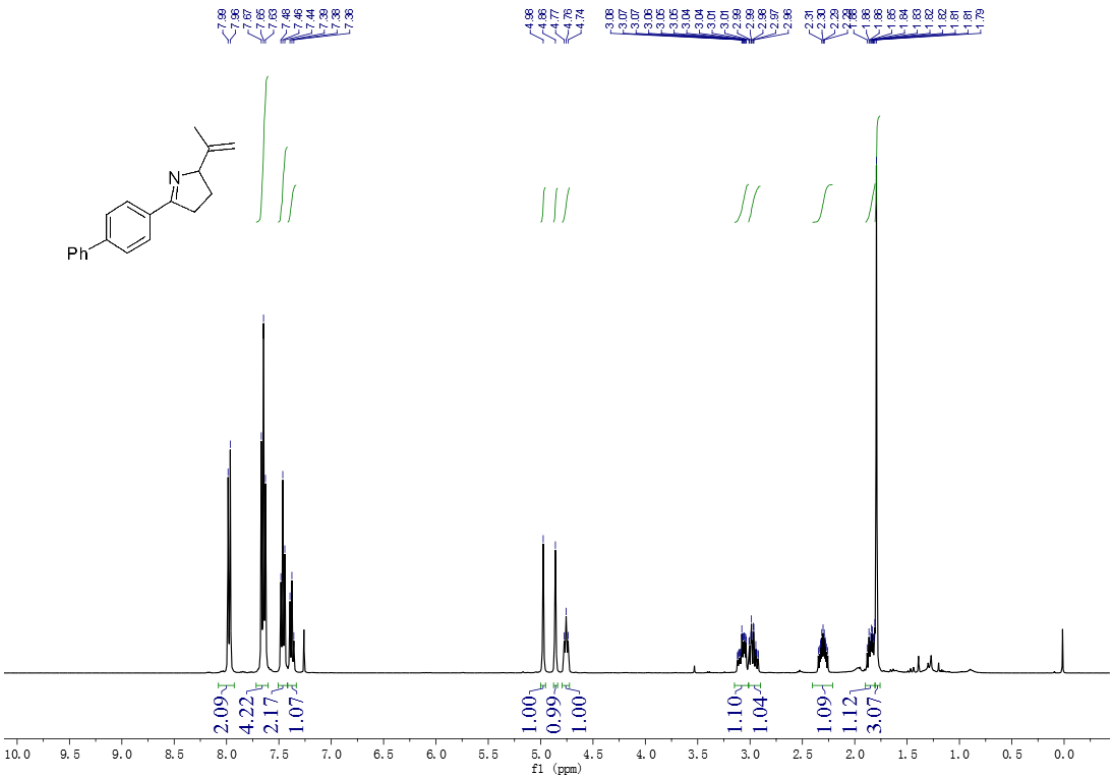
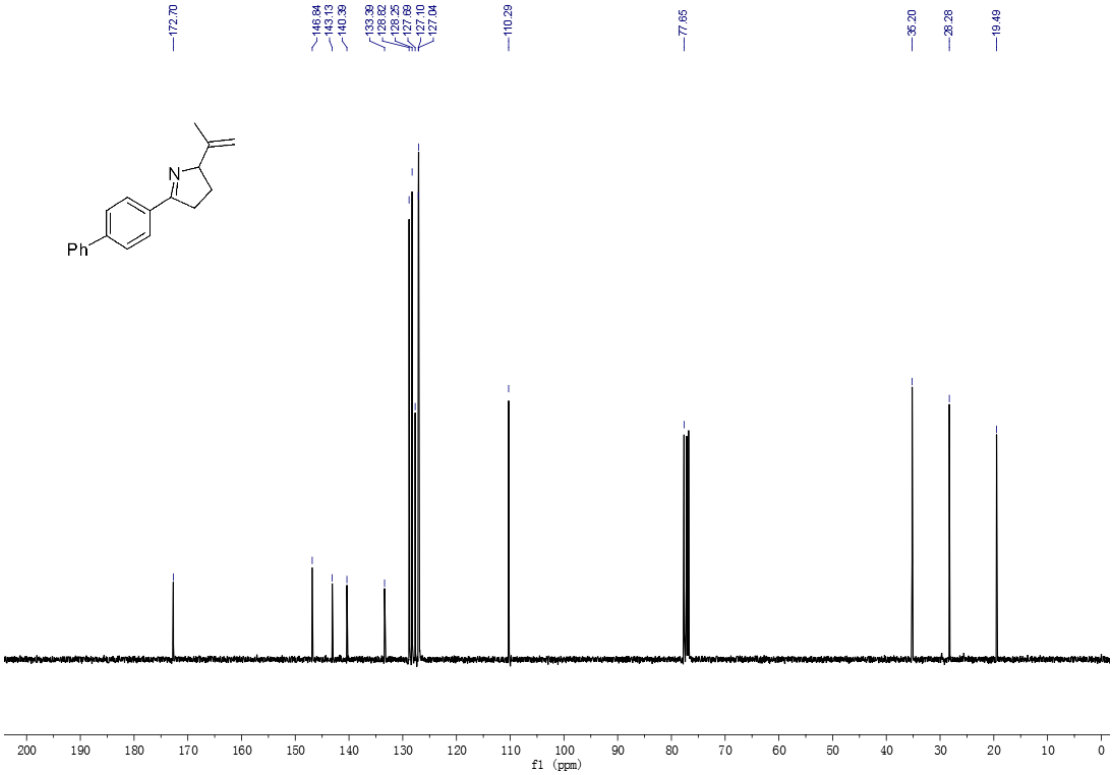


^{1}H NMR of **2i**



^{13}C NMR of **2i**



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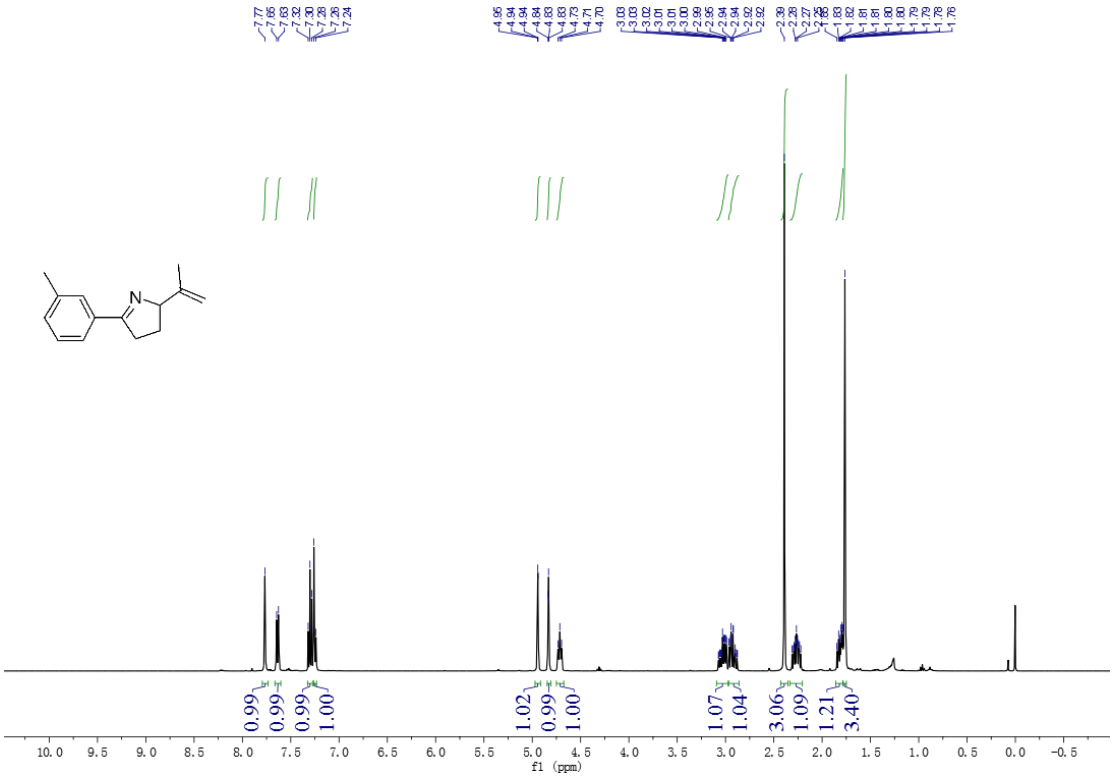

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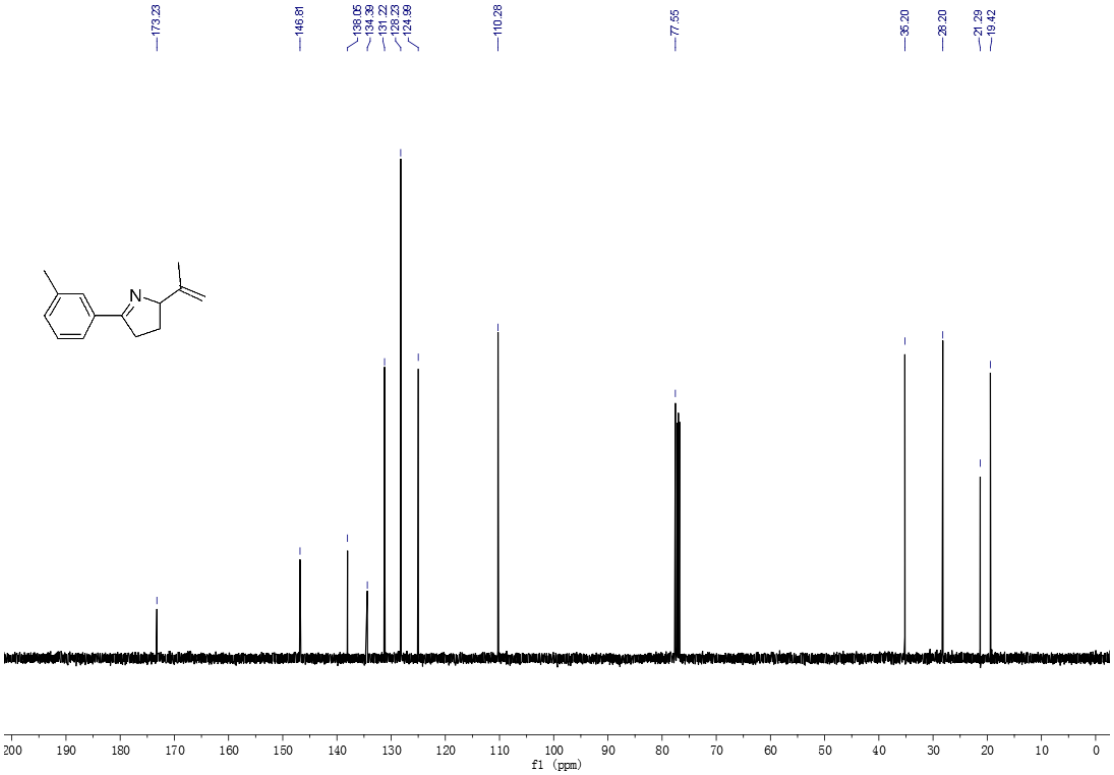
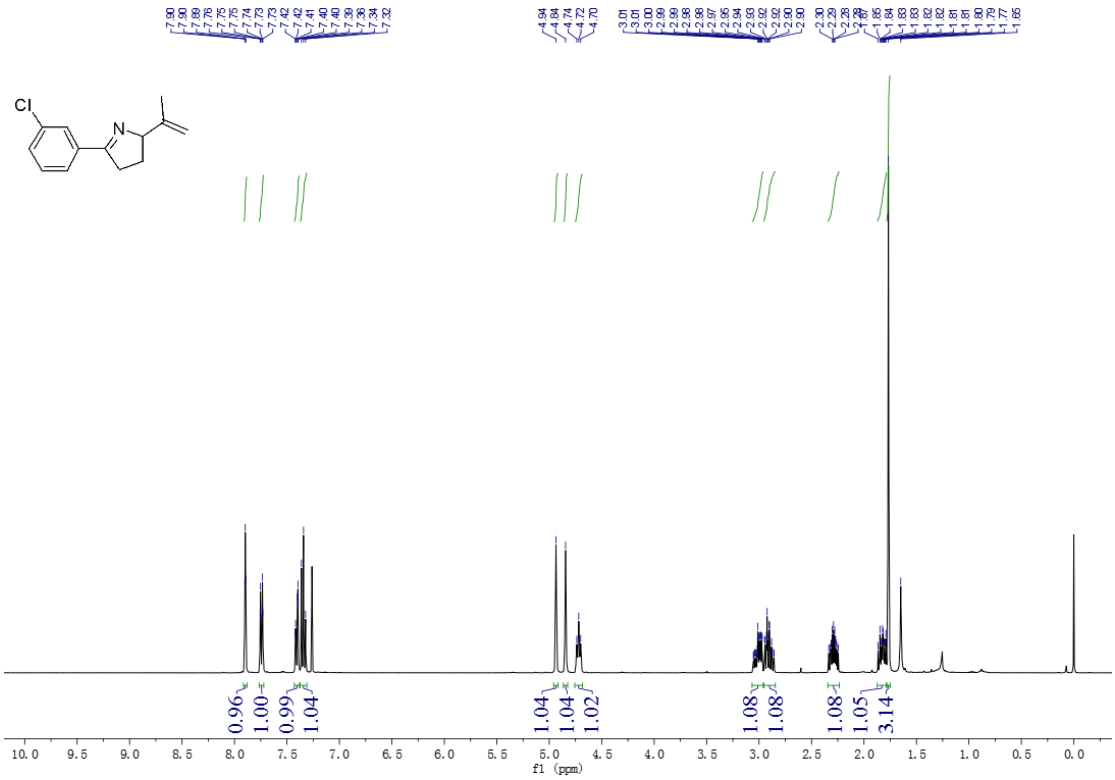


^{1}H NMR of **2k**



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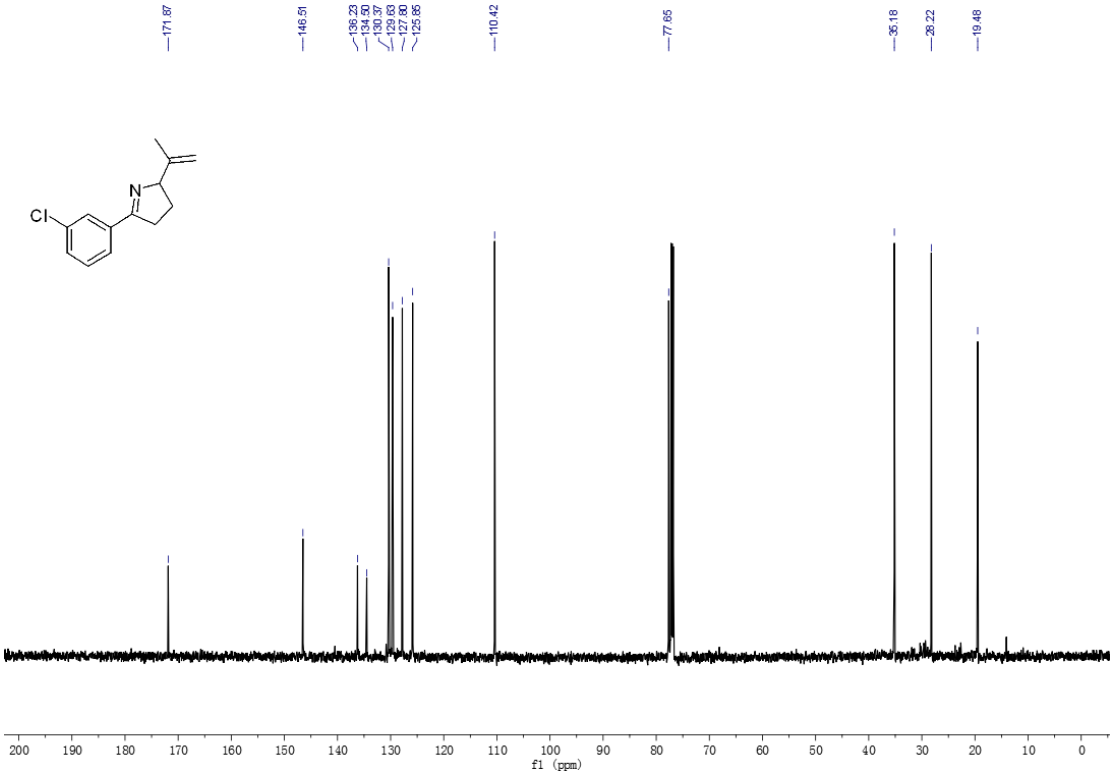
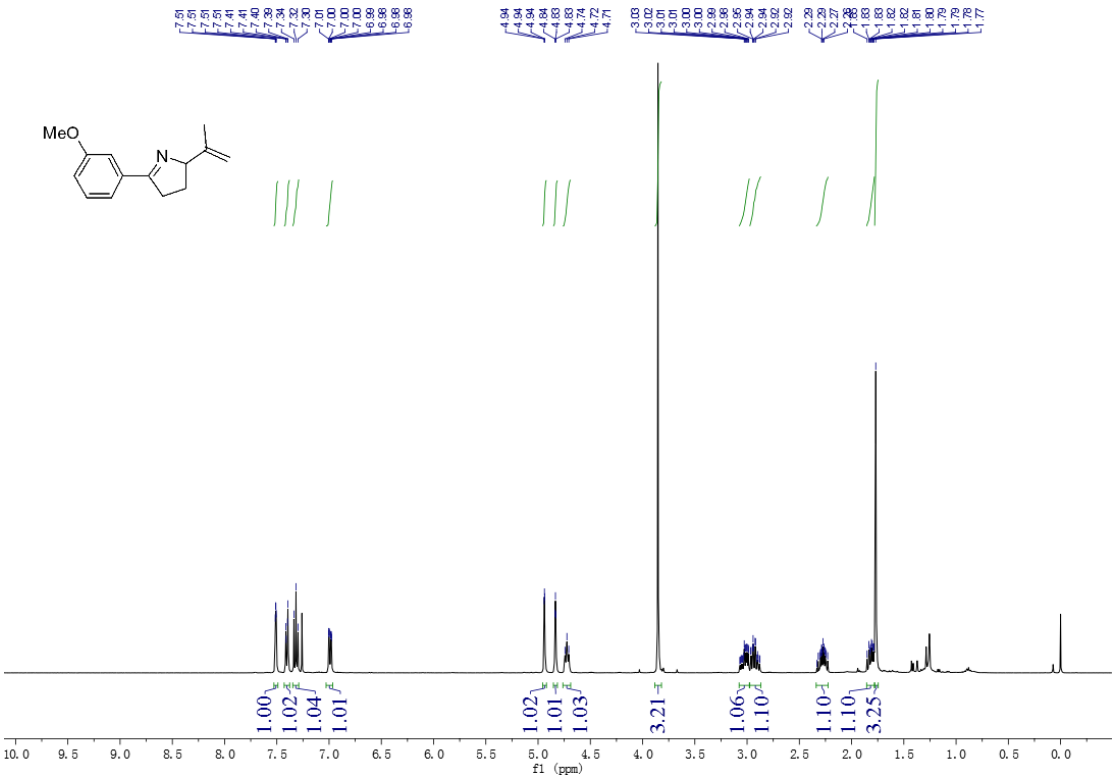
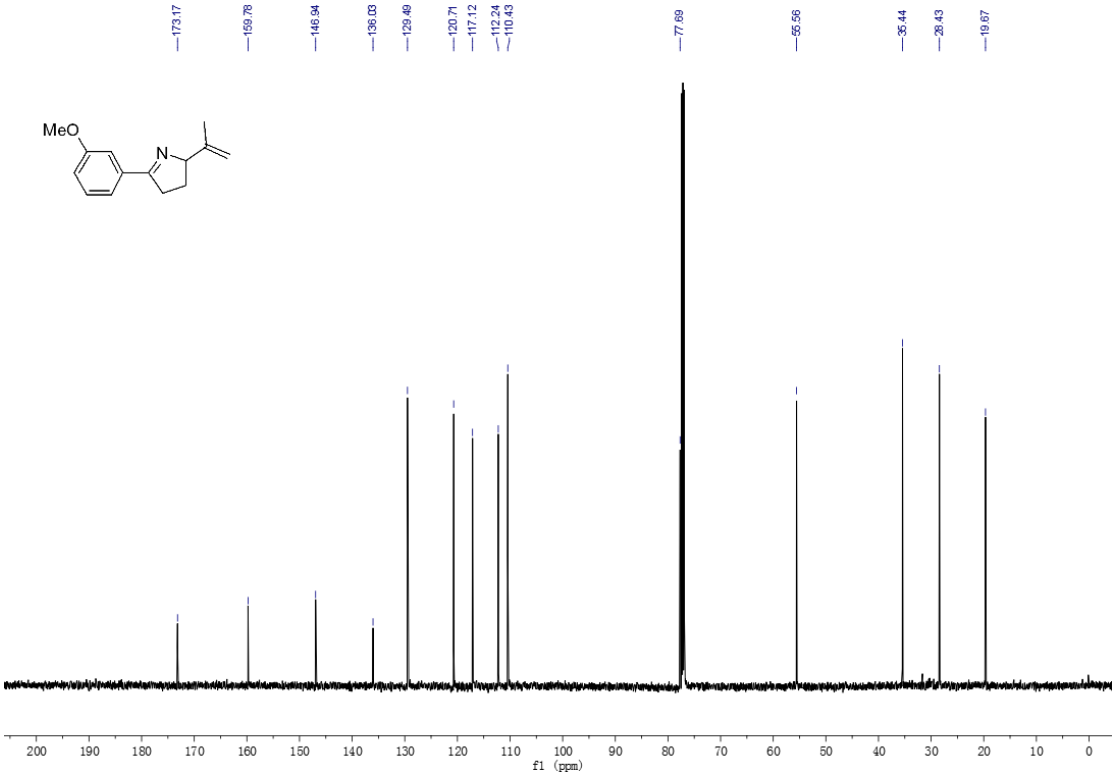


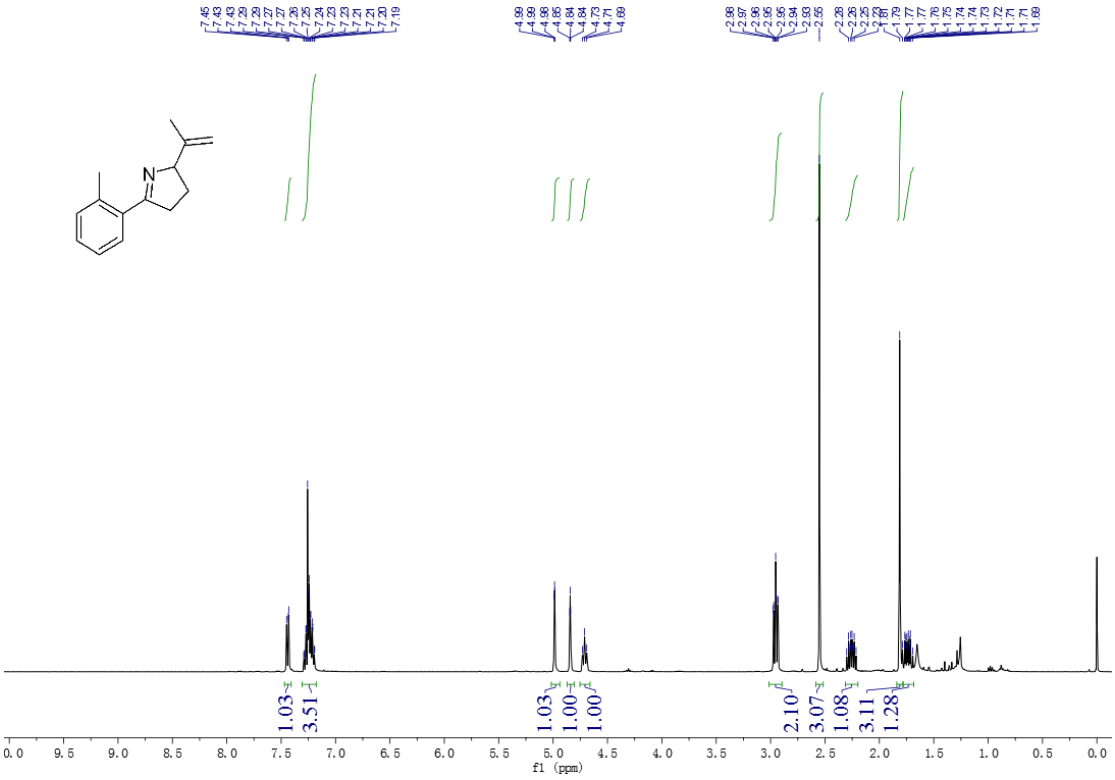
^{1}H NMR of **2l**



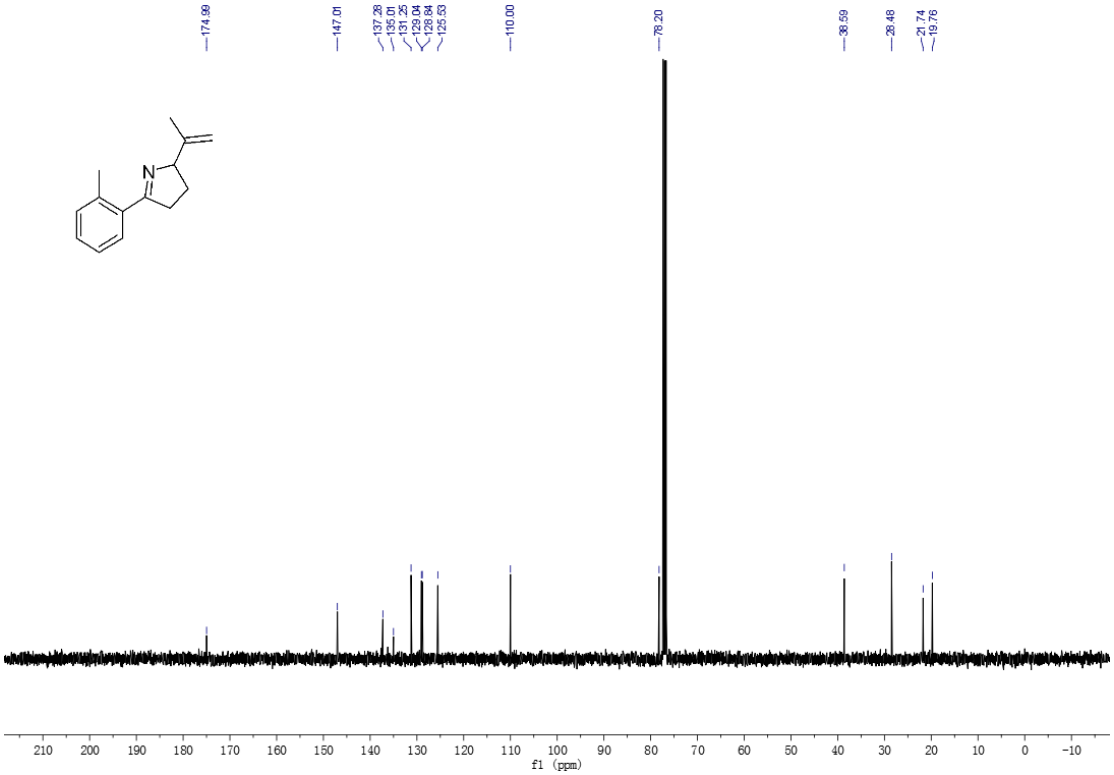
^{13}C NMR of **2l**



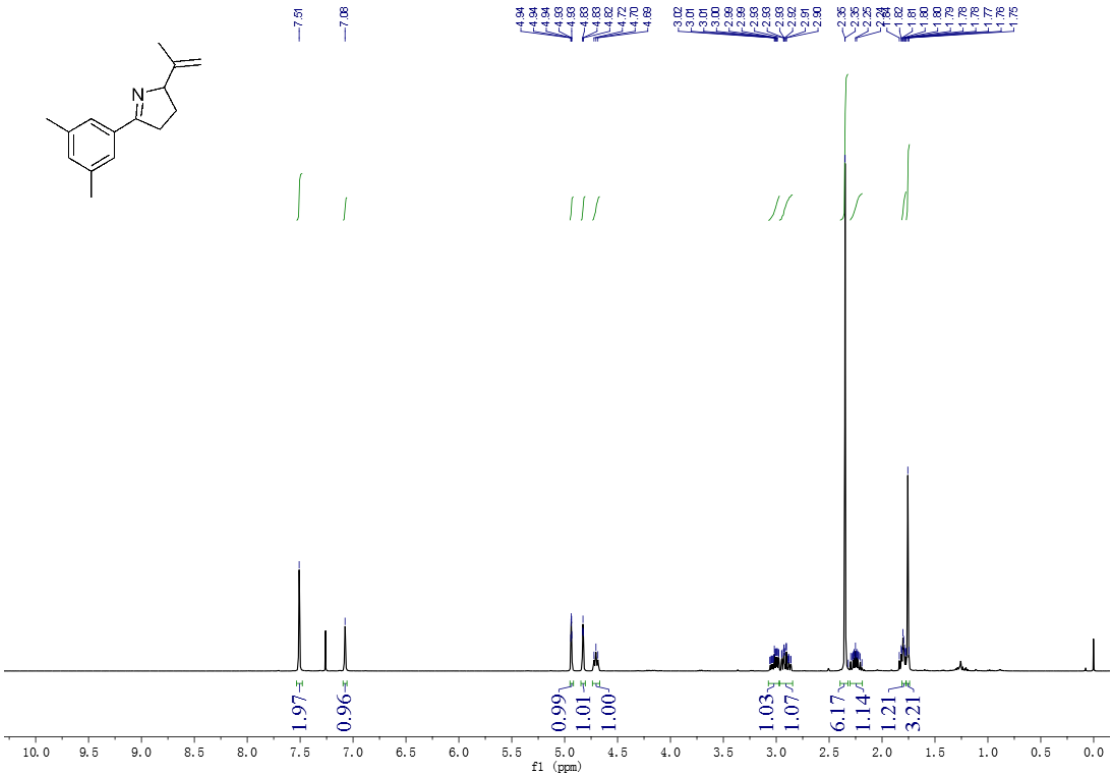
^{1}H NMR of **2m**



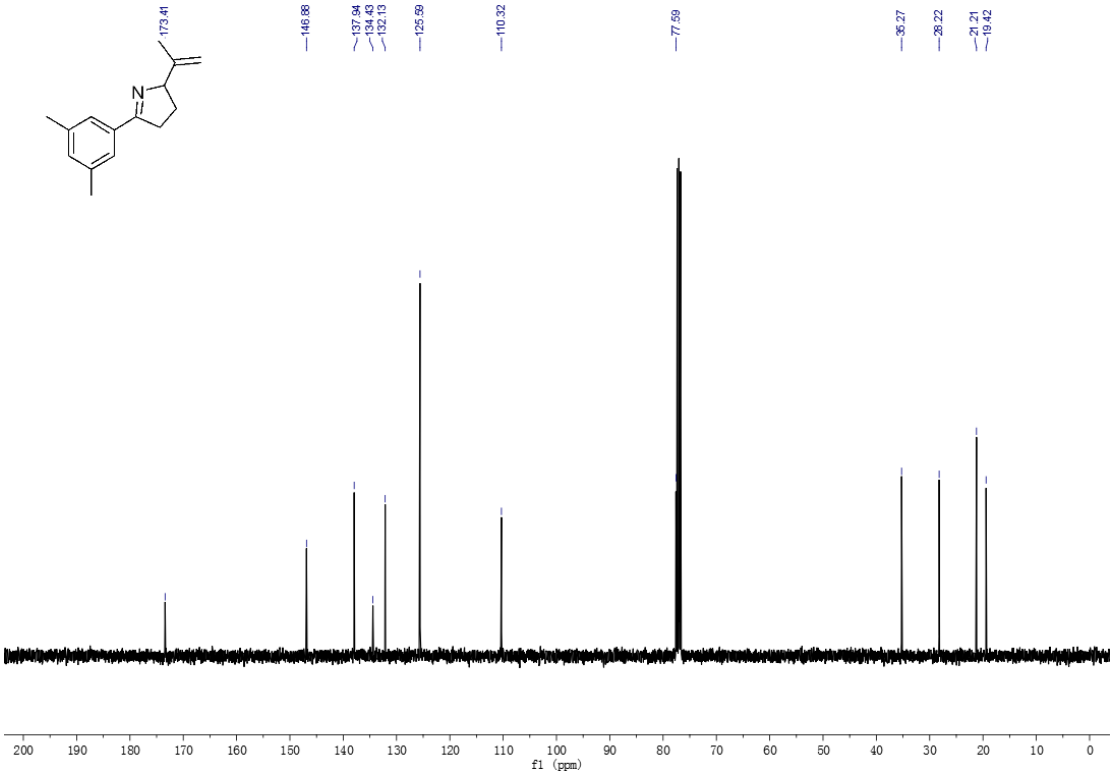
^{13}C NMR of **2m**



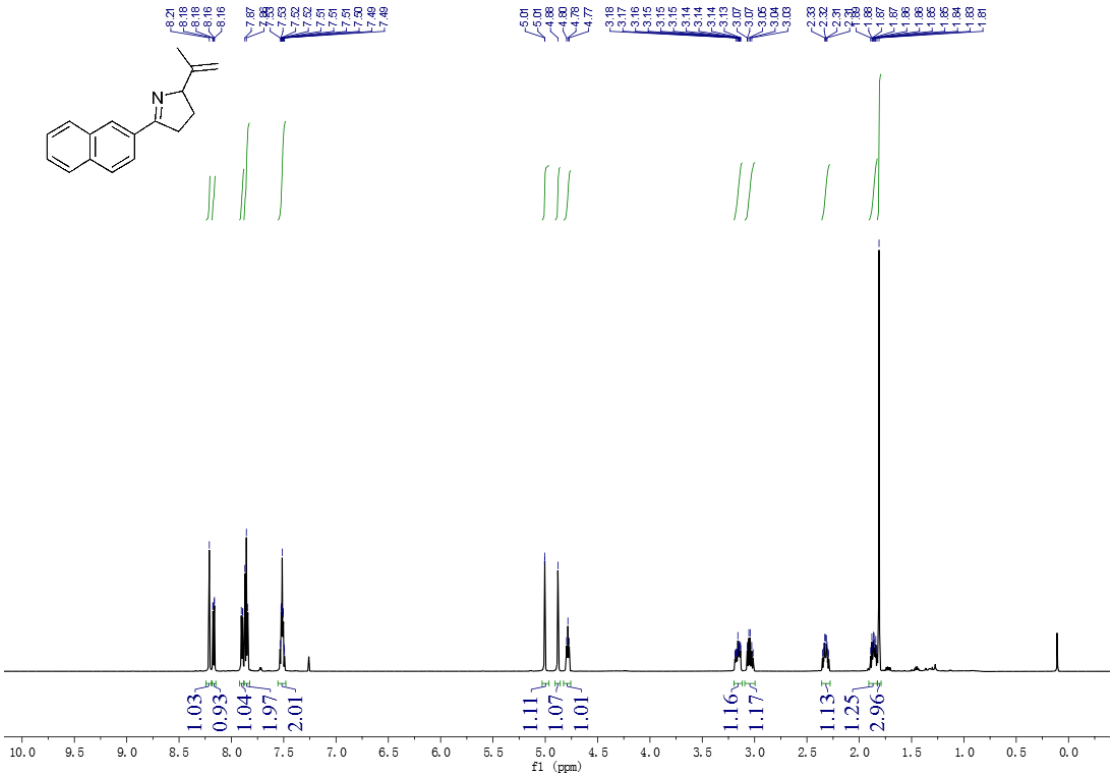
^{1}H NMR of **2n**



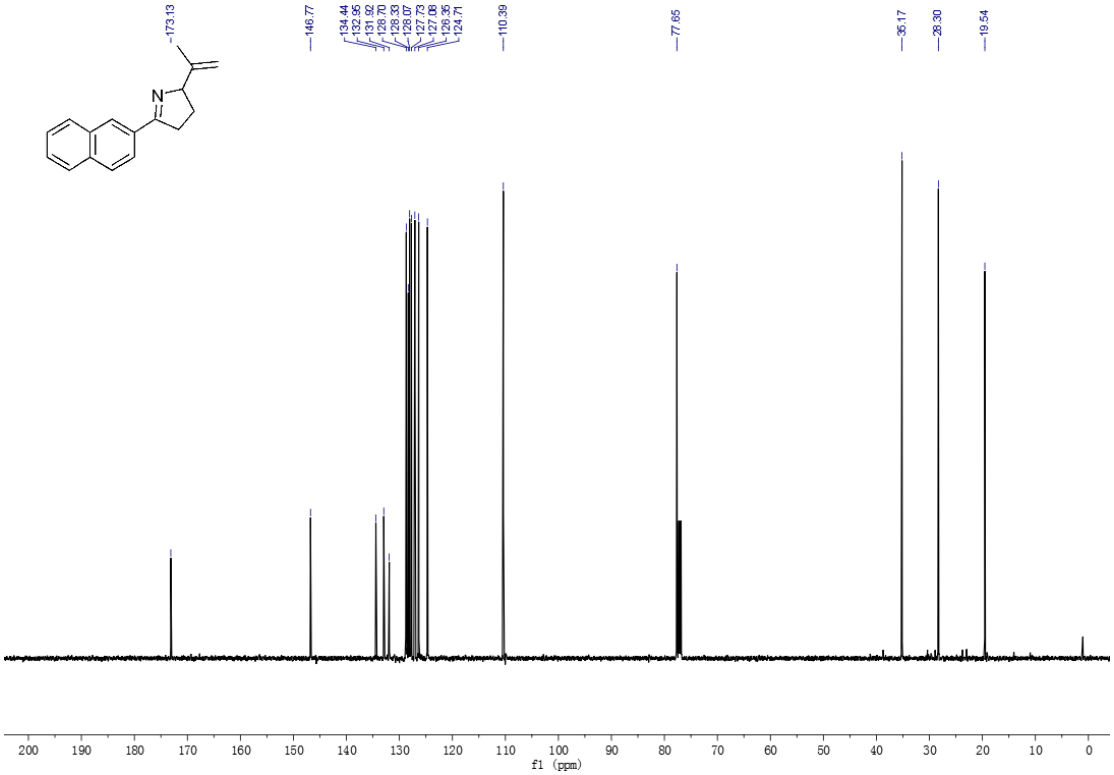
^{13}C NMR of **2n**



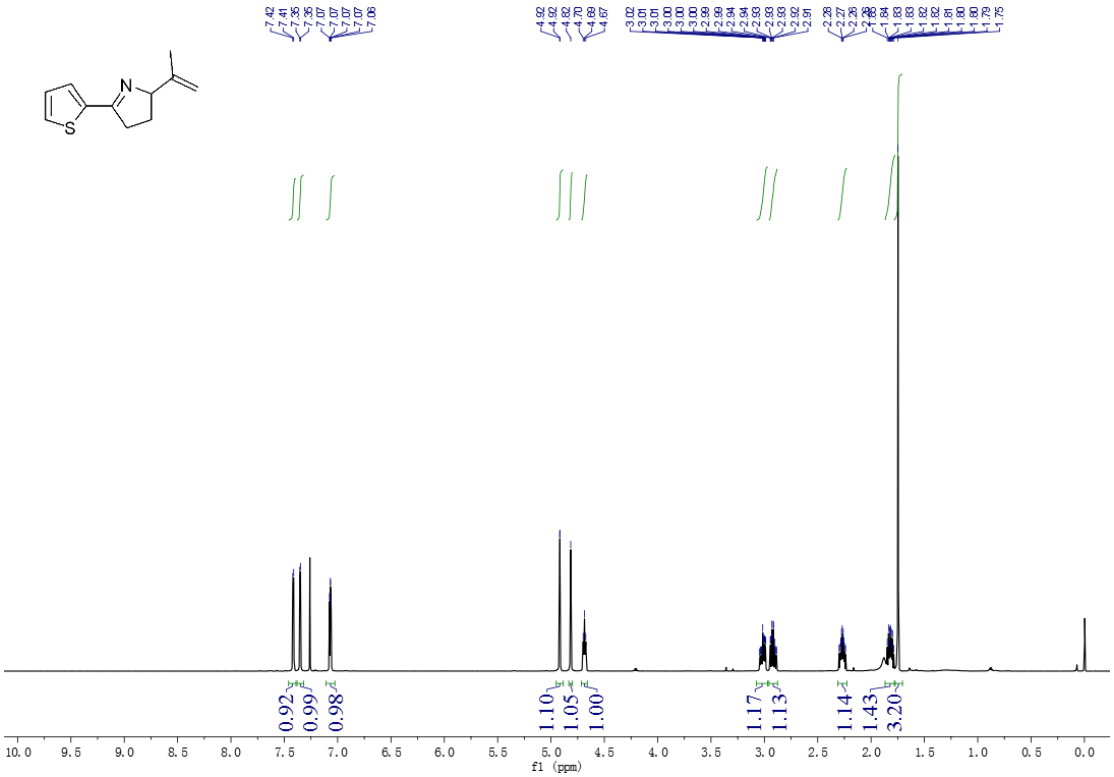
^{1}H NMR of **2o**



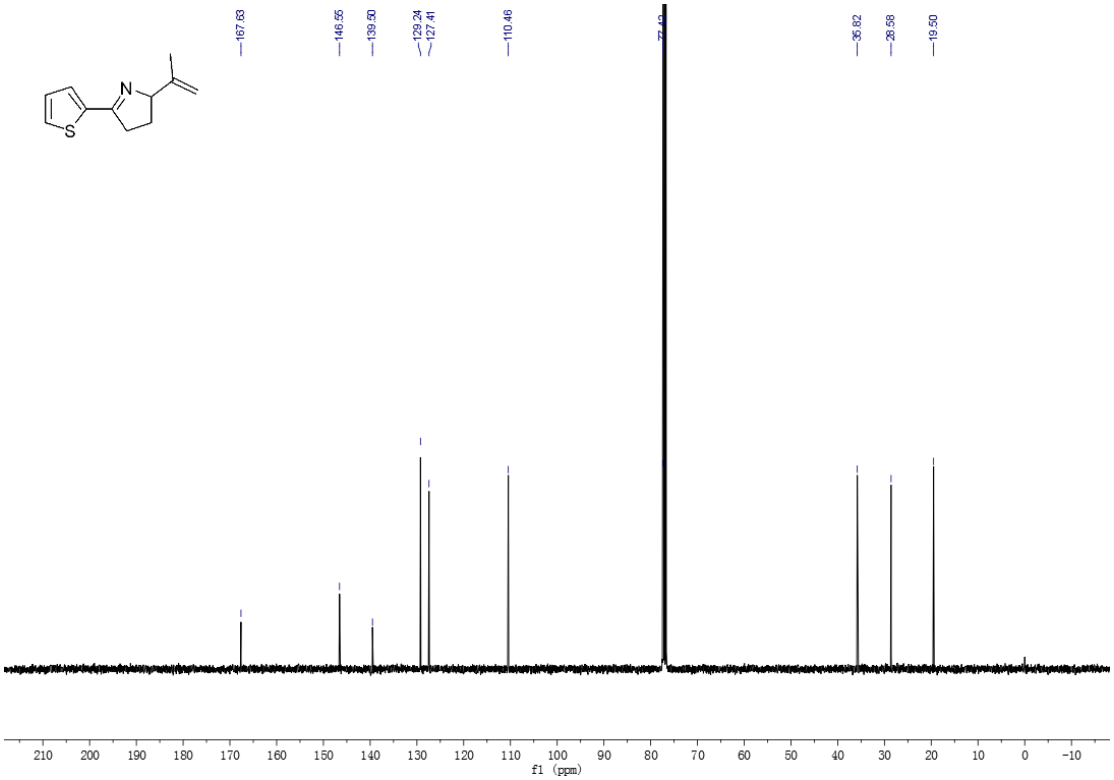
^{13}C NMR of **2o**



^{1}H NMR of **2p**



^{13}C NMR of **2p**



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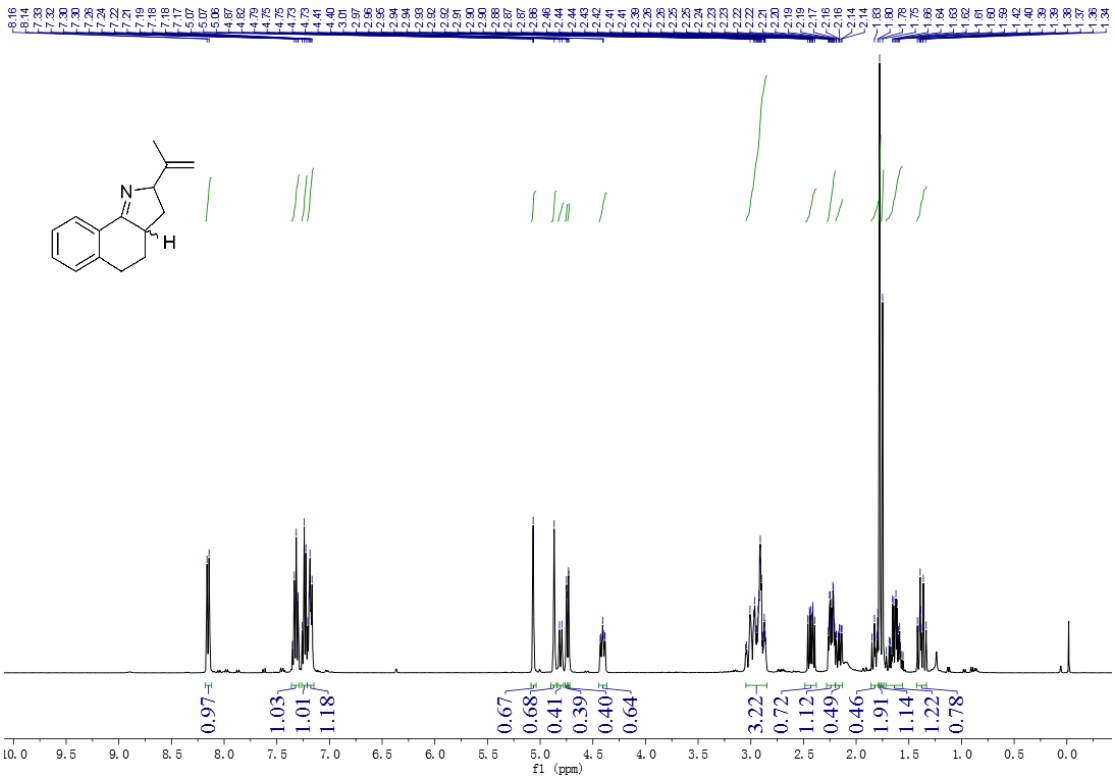
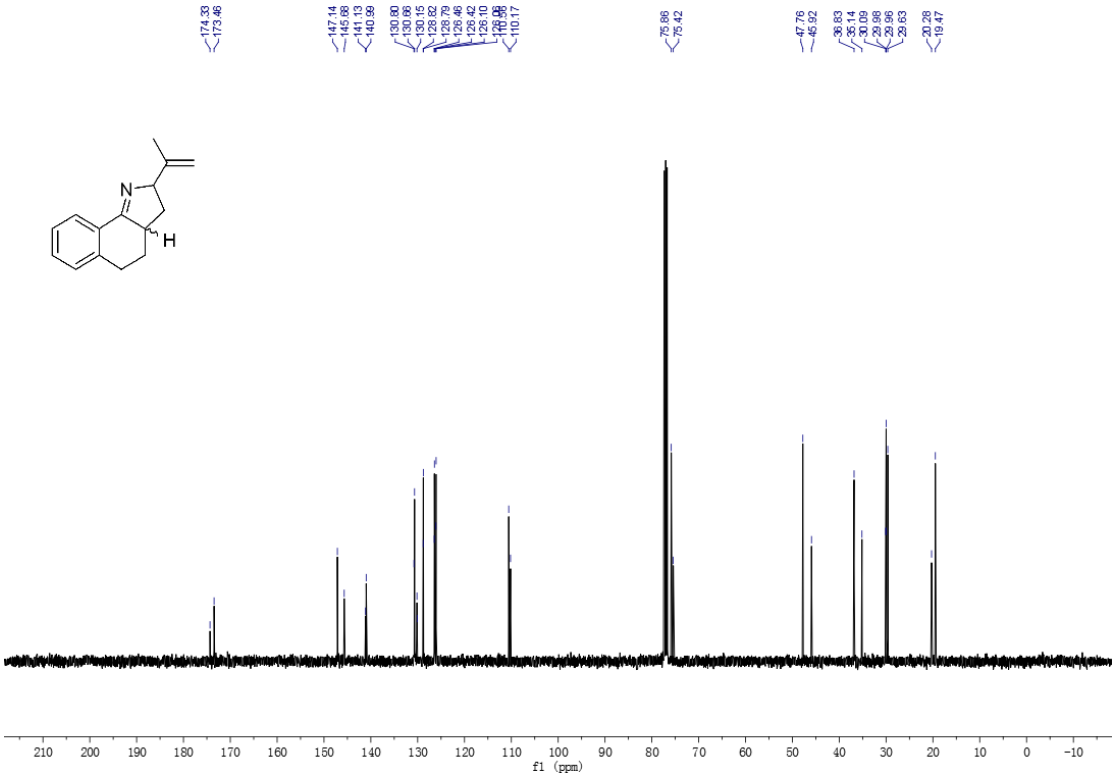
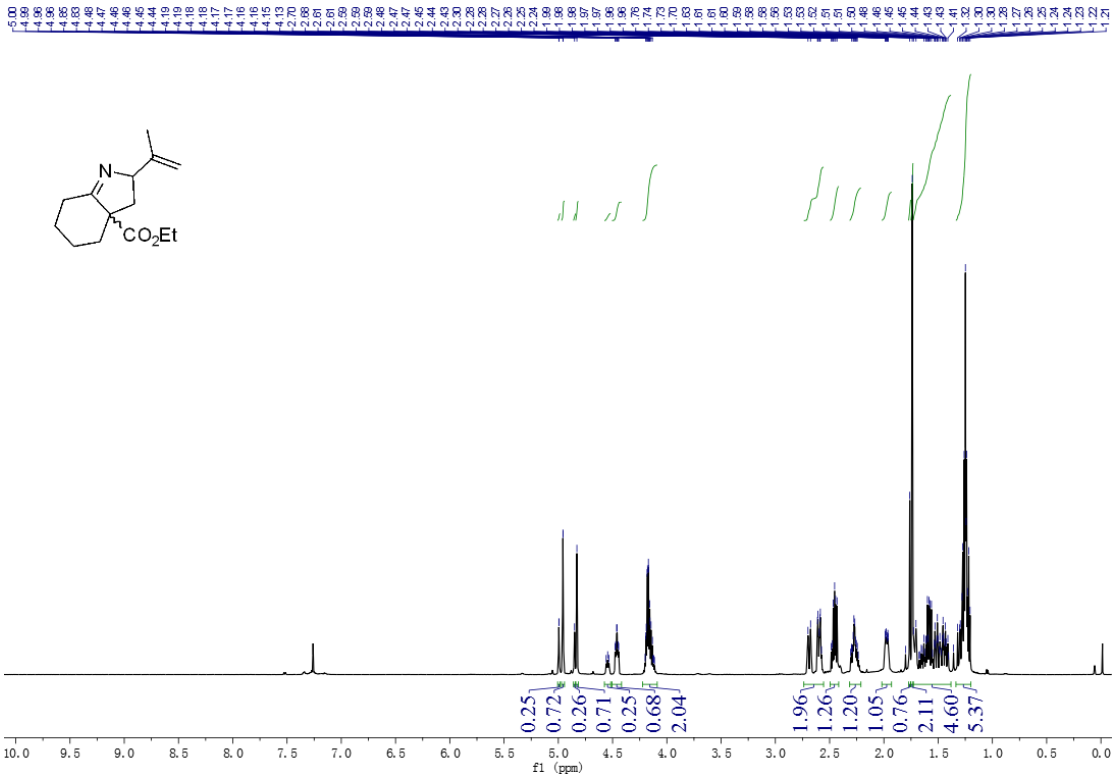


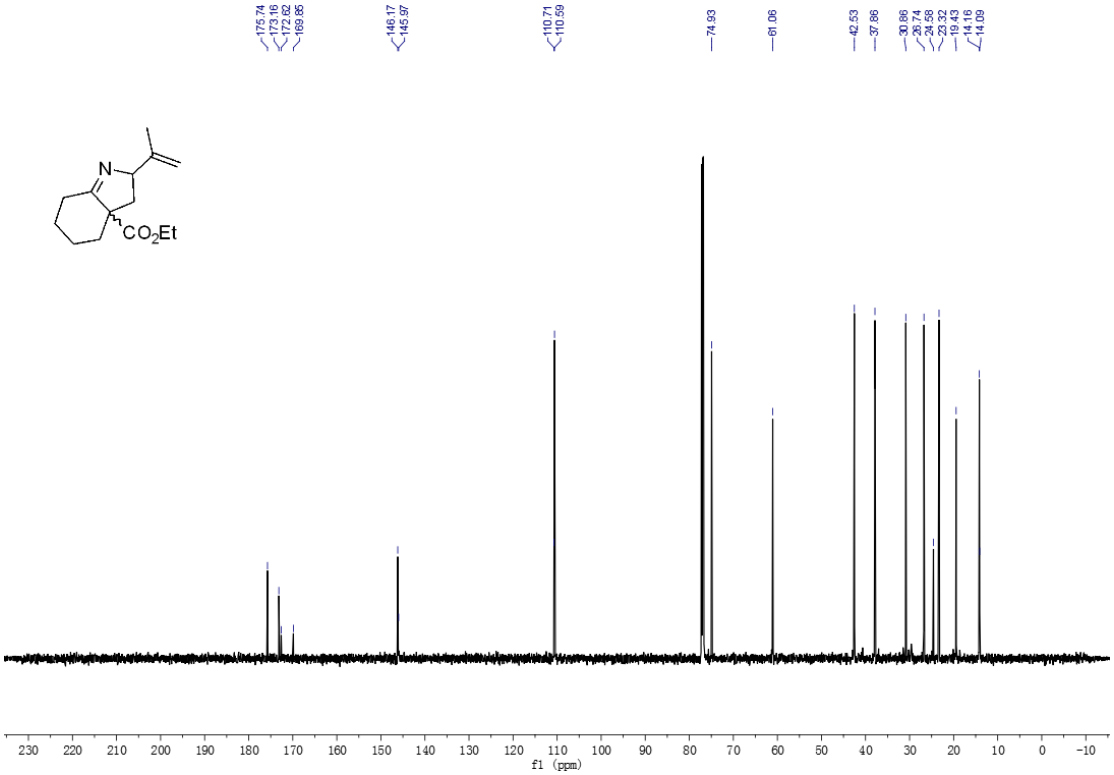
^{13}C NMR of **2q**



^{1}H NMR of **2r**



^{13}C NMR of **2r**



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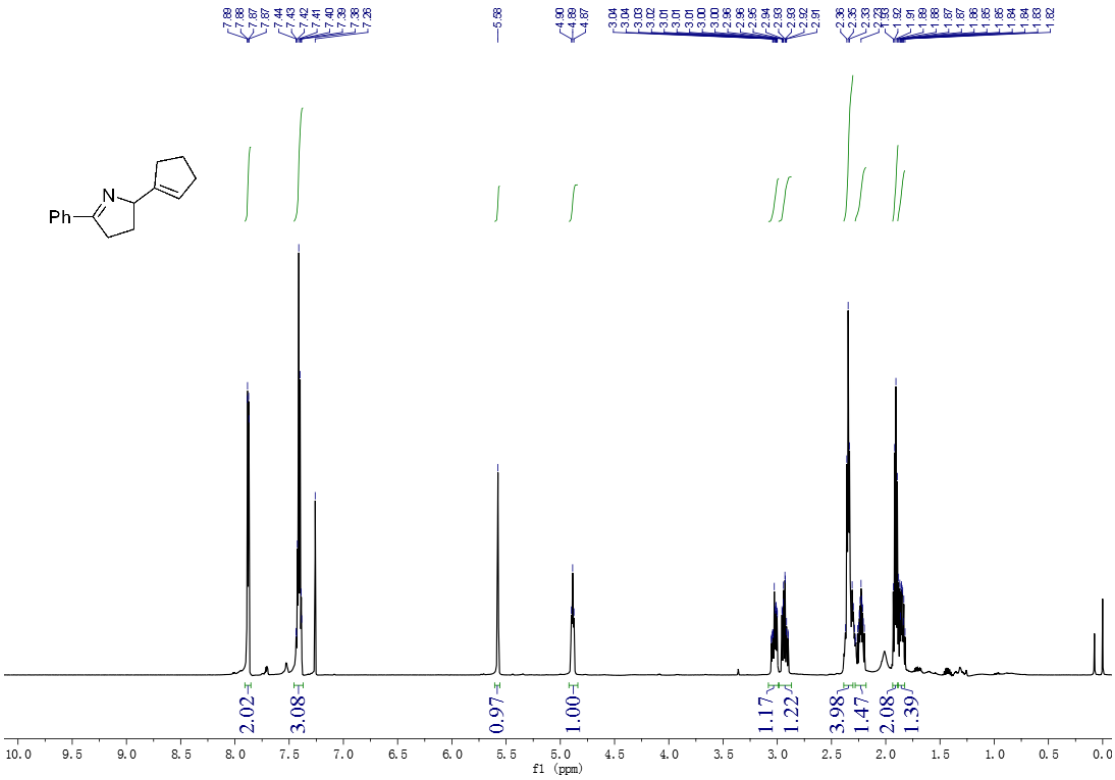
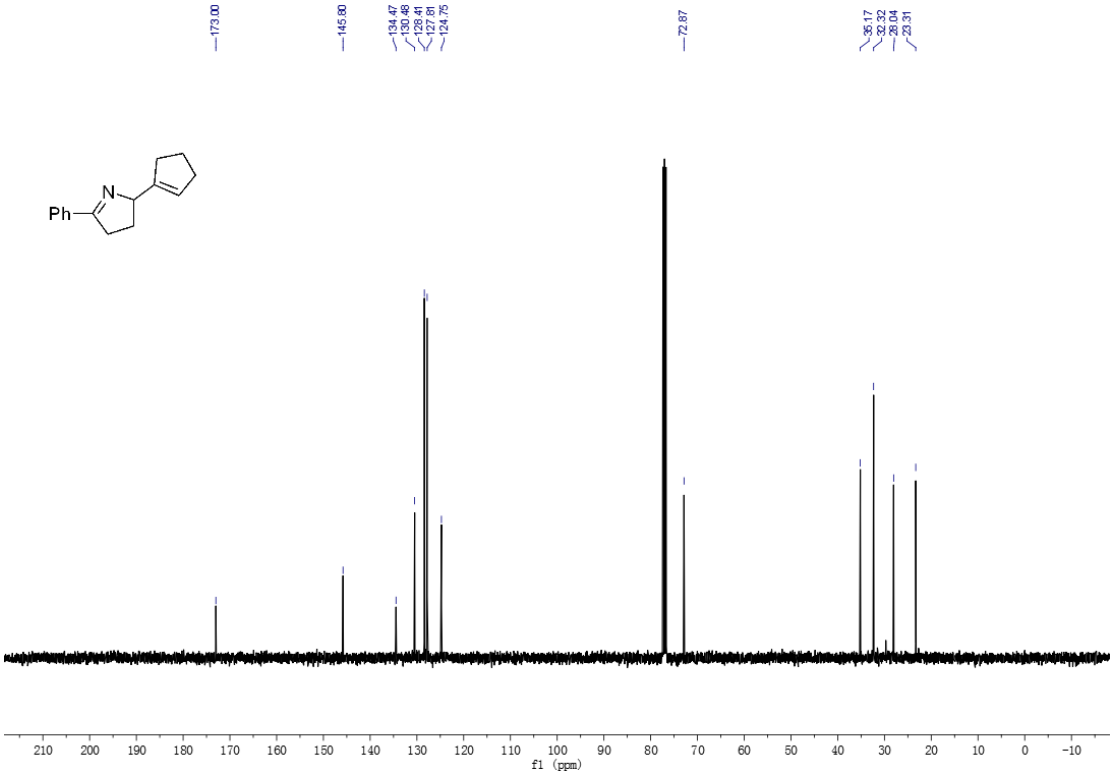


^{13}C NMR of **2s**



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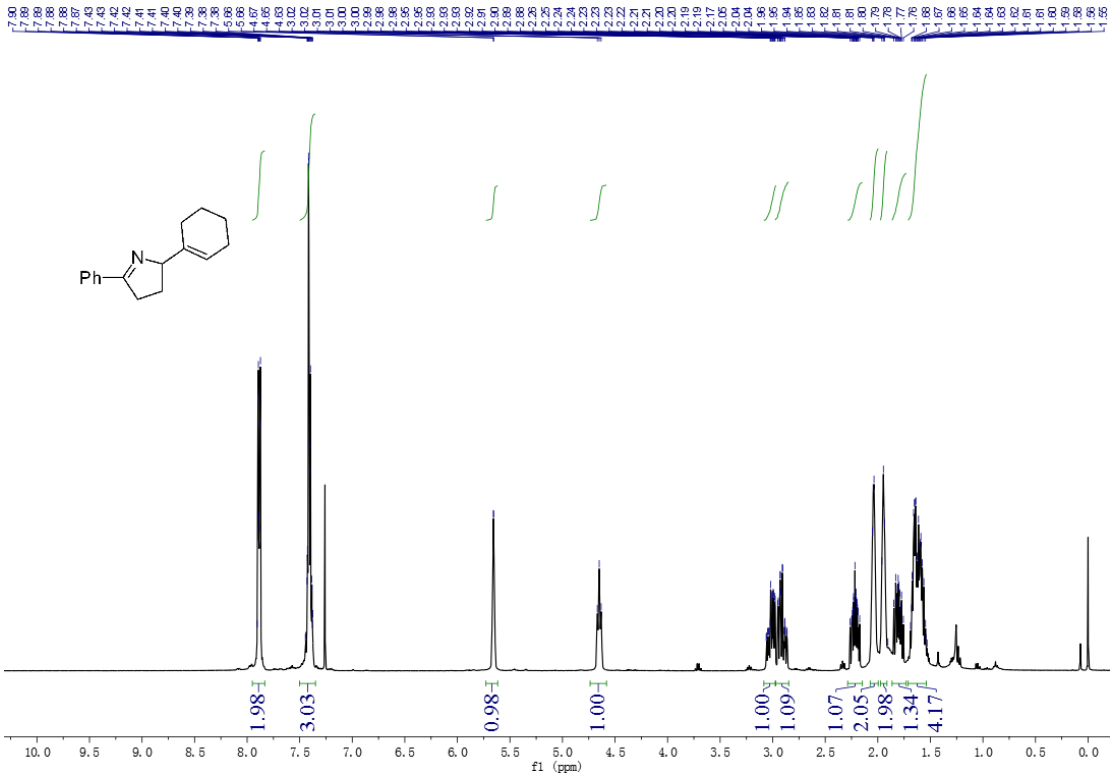

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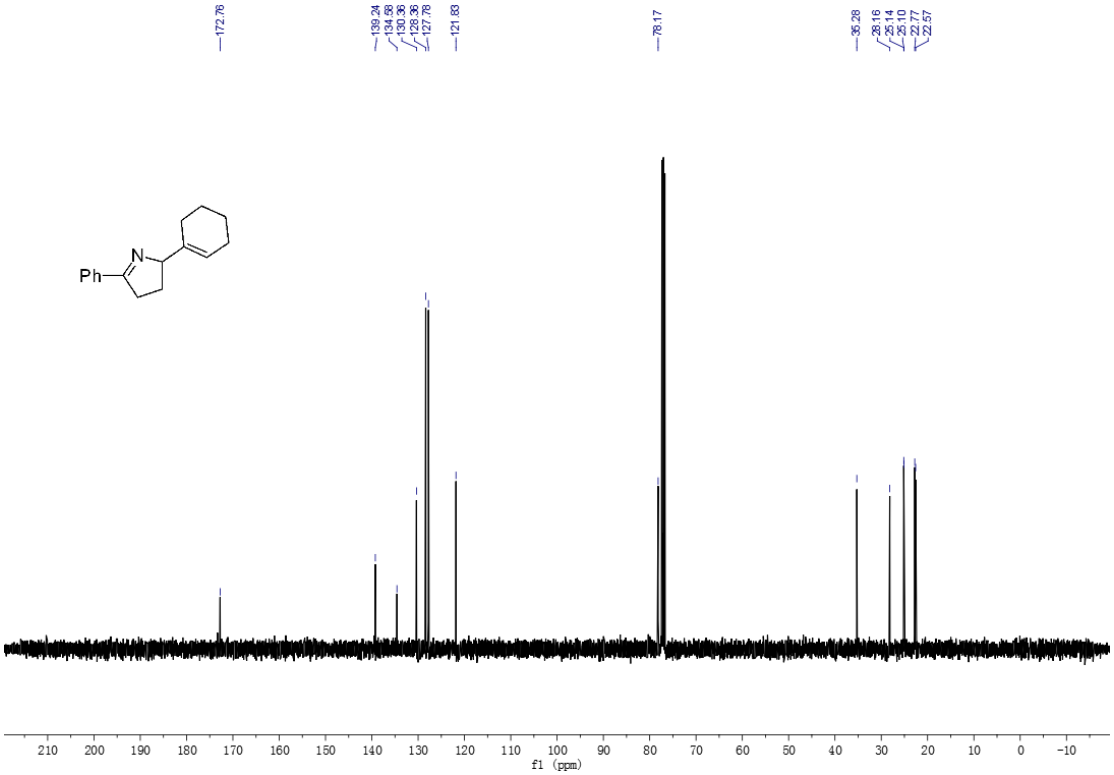

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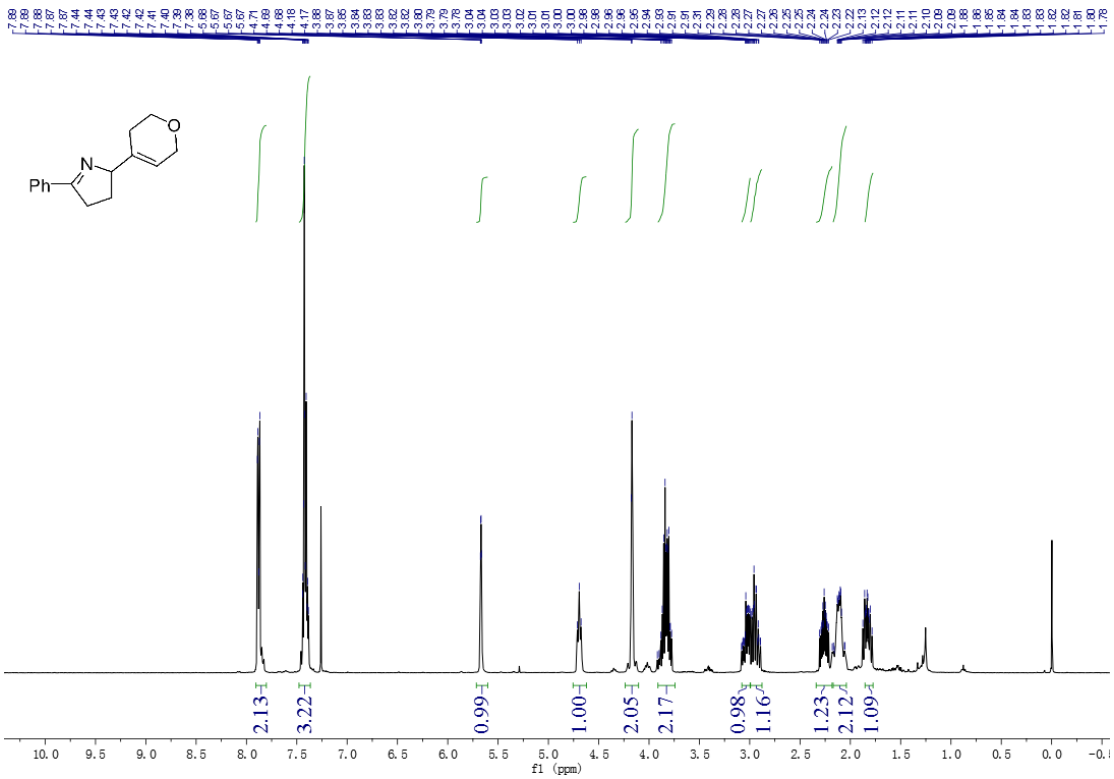
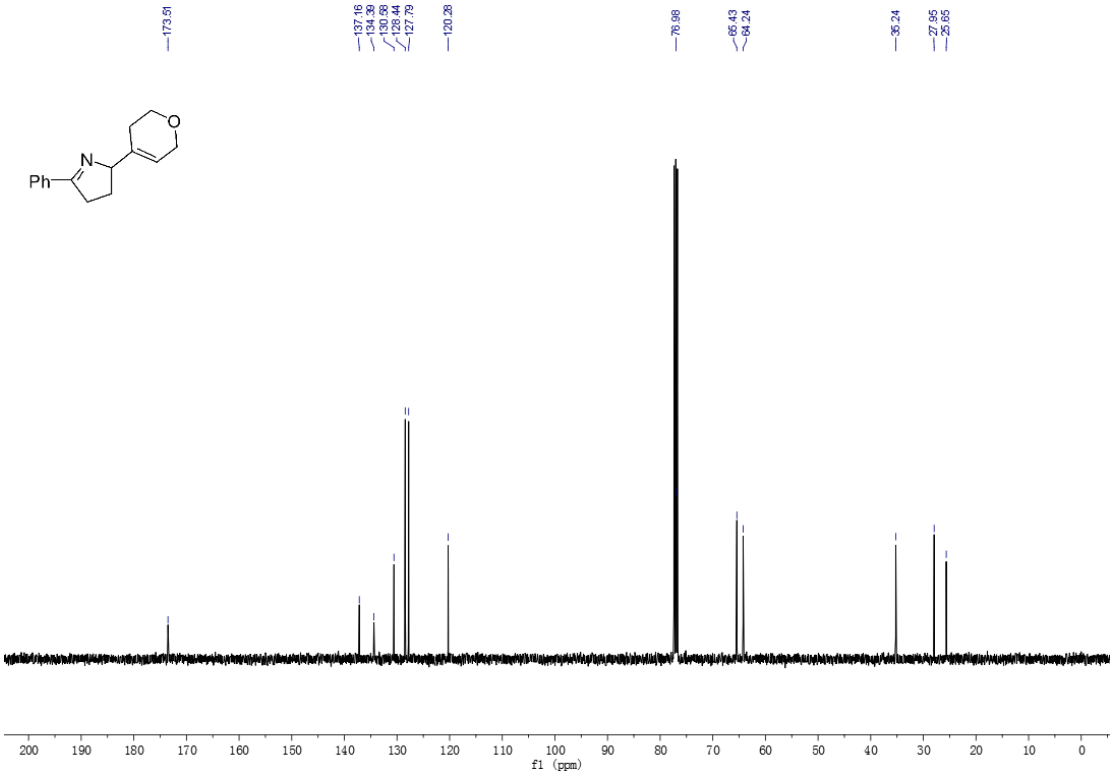
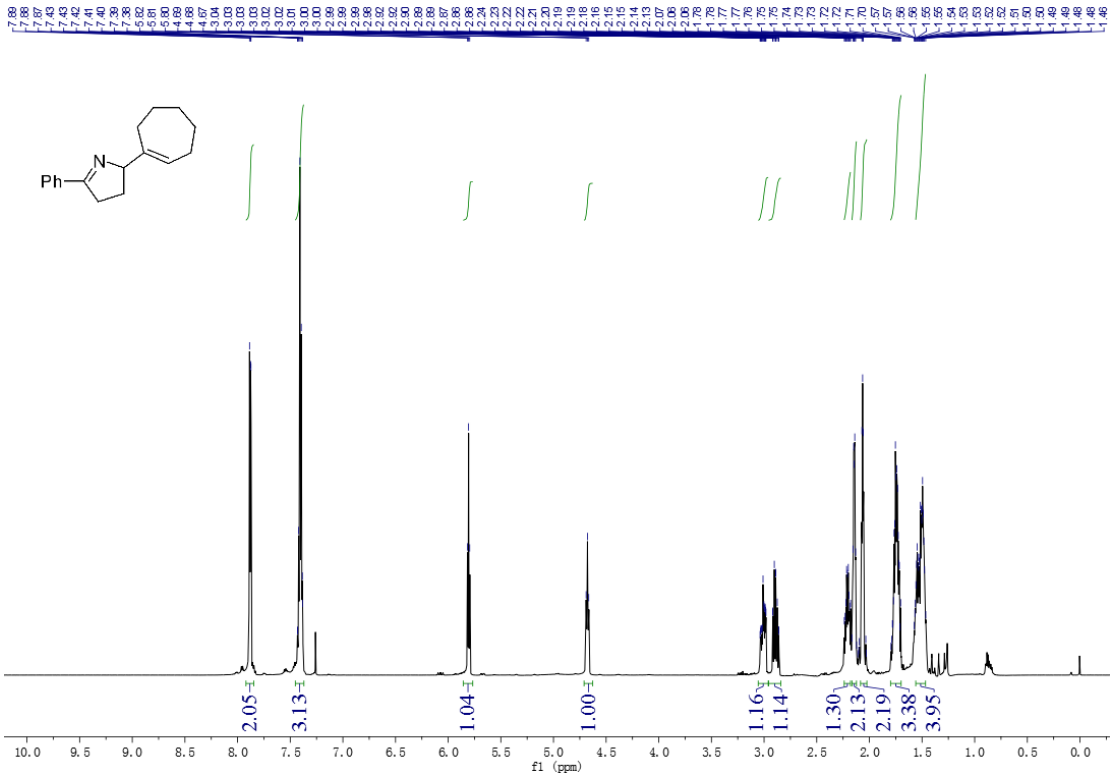


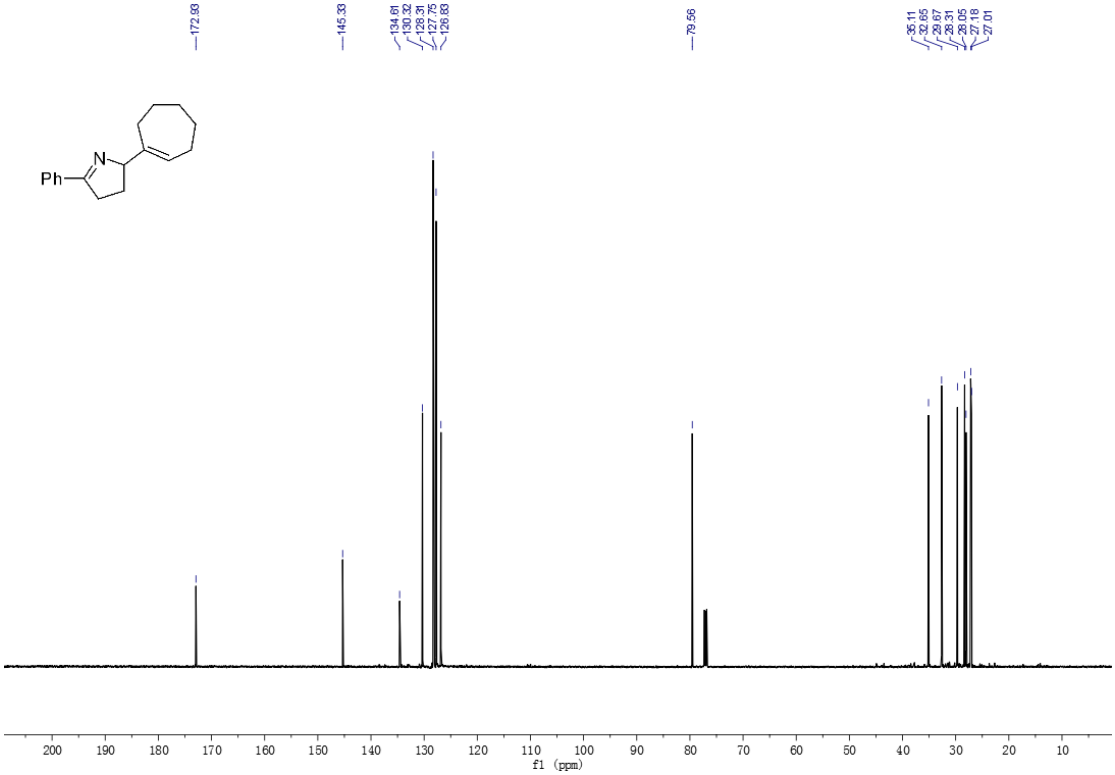
^{13}C NMR of **2u**



^{1}H NMR of **2v**



^{13}C NMR of **2v**



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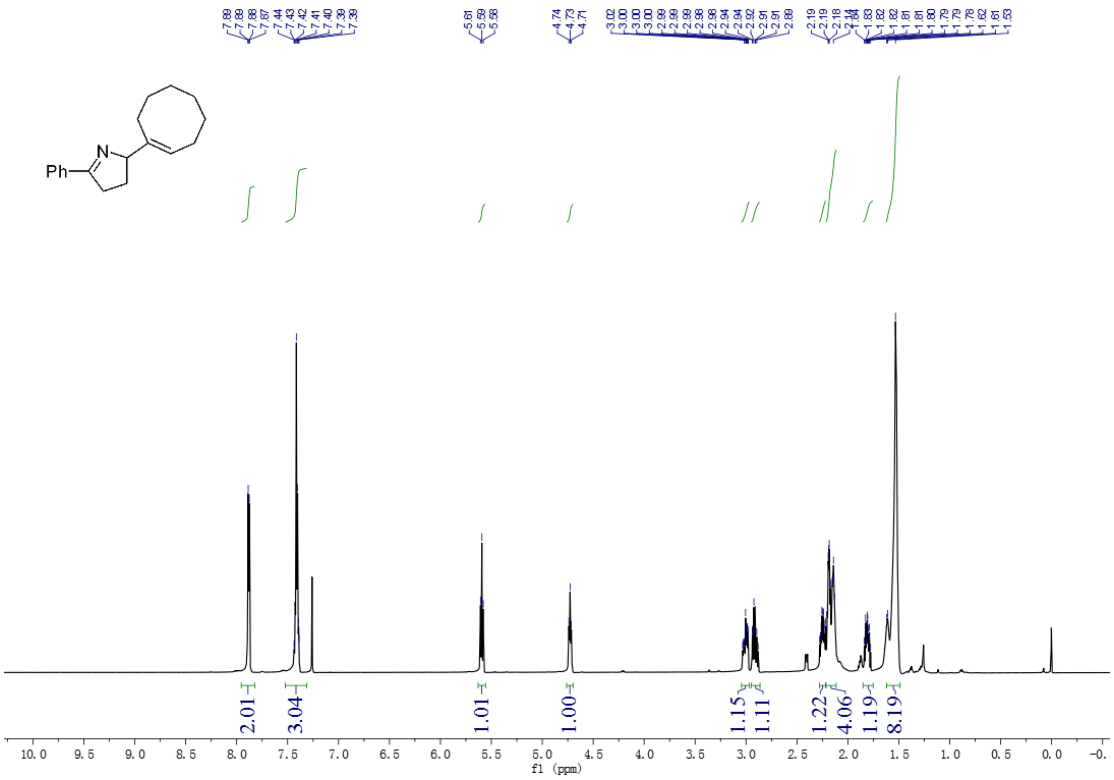
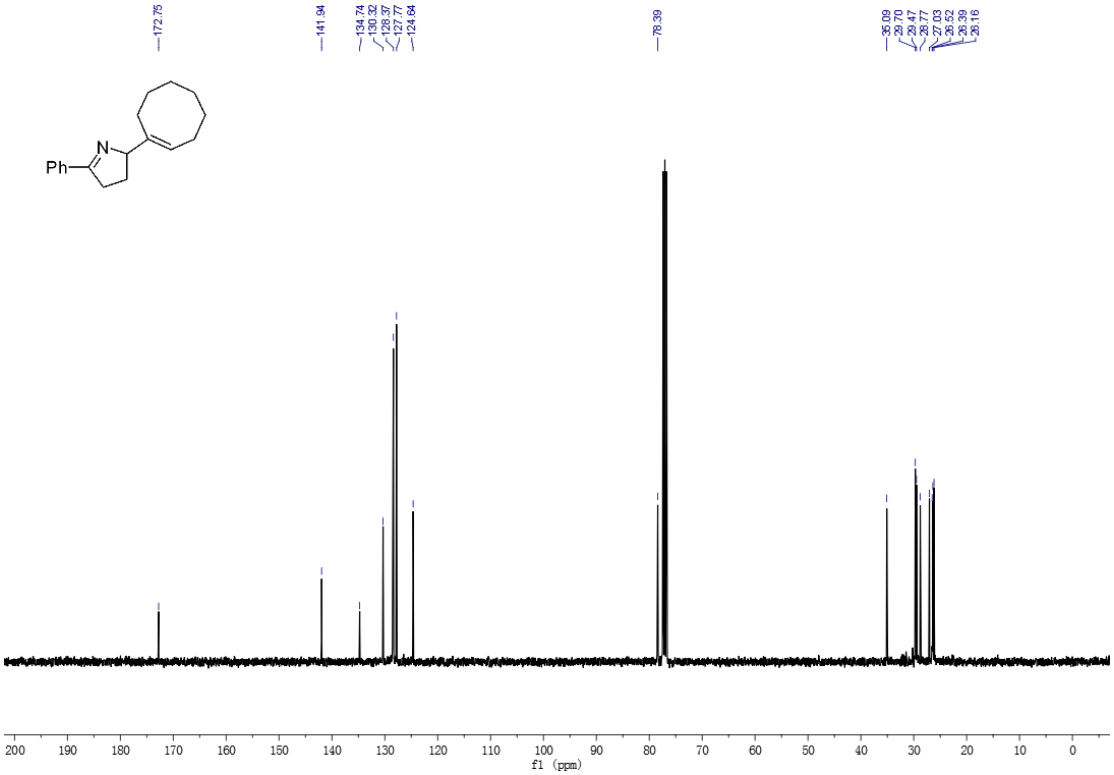
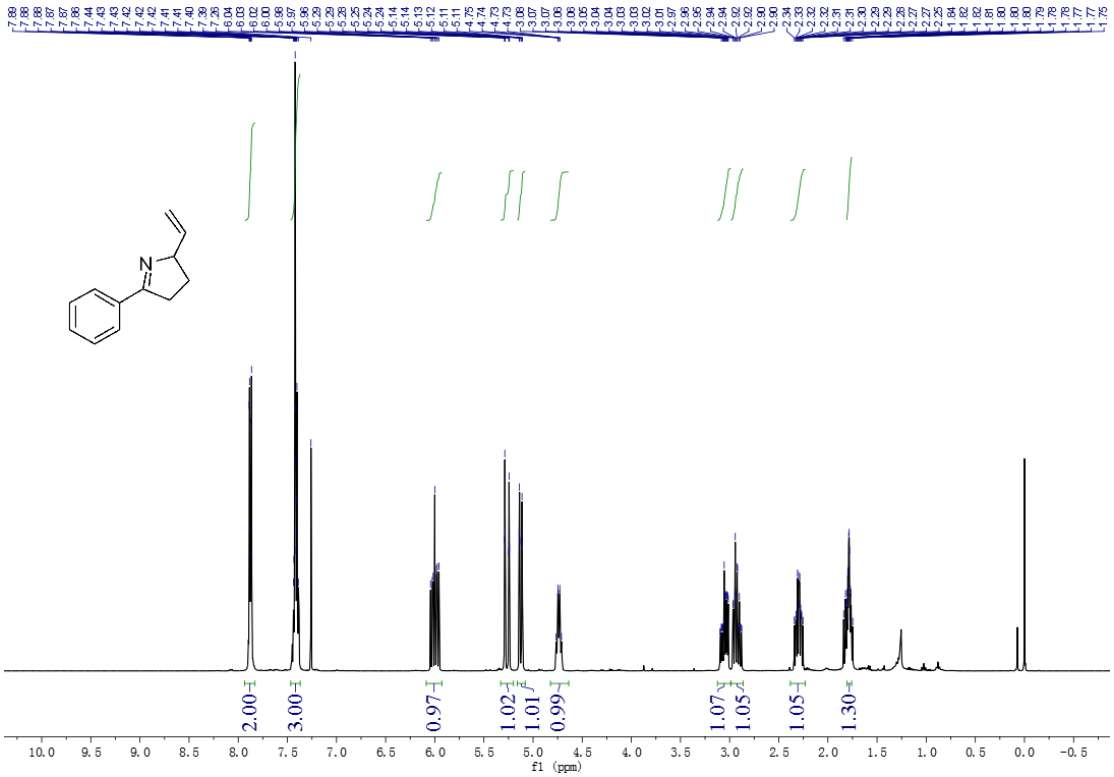


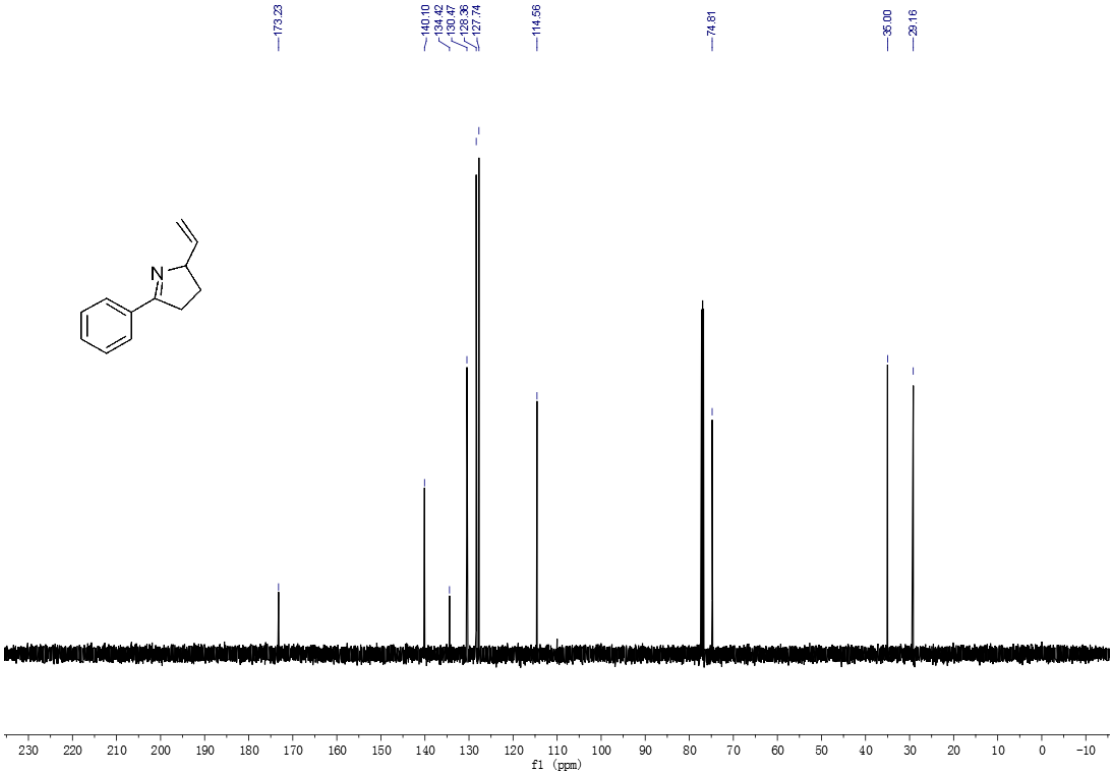
^{13}C NMR of **2w**



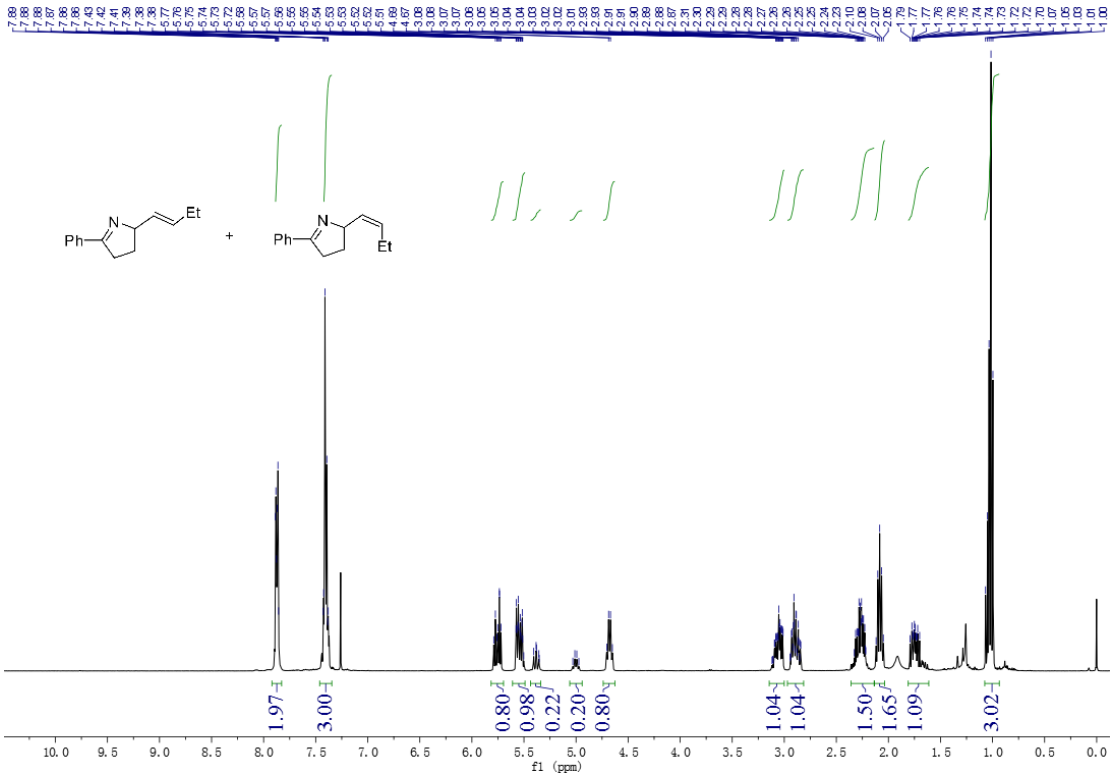
^{1}H NMR of **3a**



^{13}C NMR of **3a**



^{1}H NMR of **3b**



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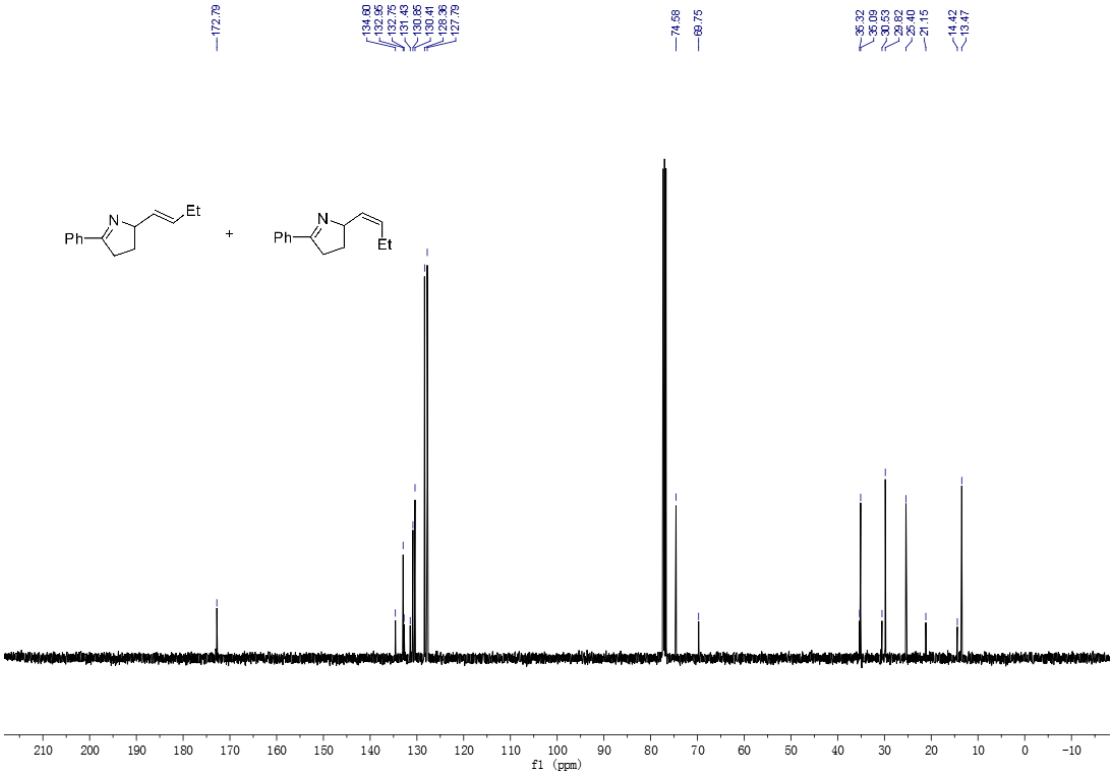
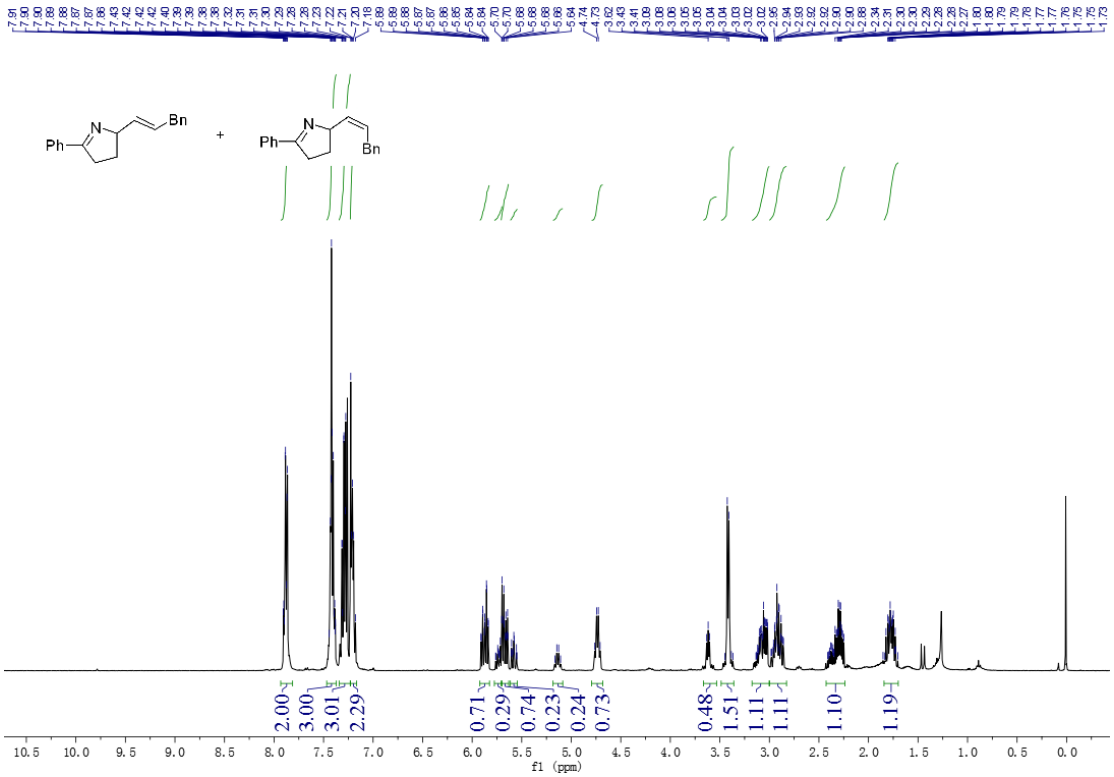


^{1}H NMR of **3c**



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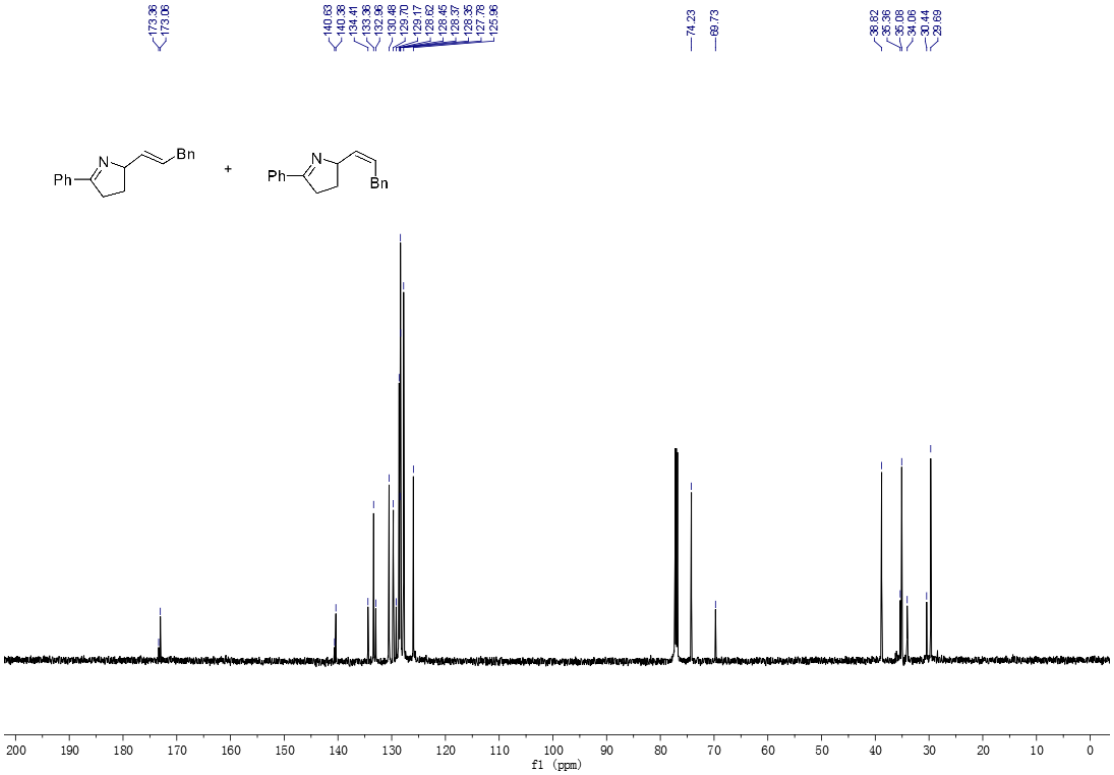
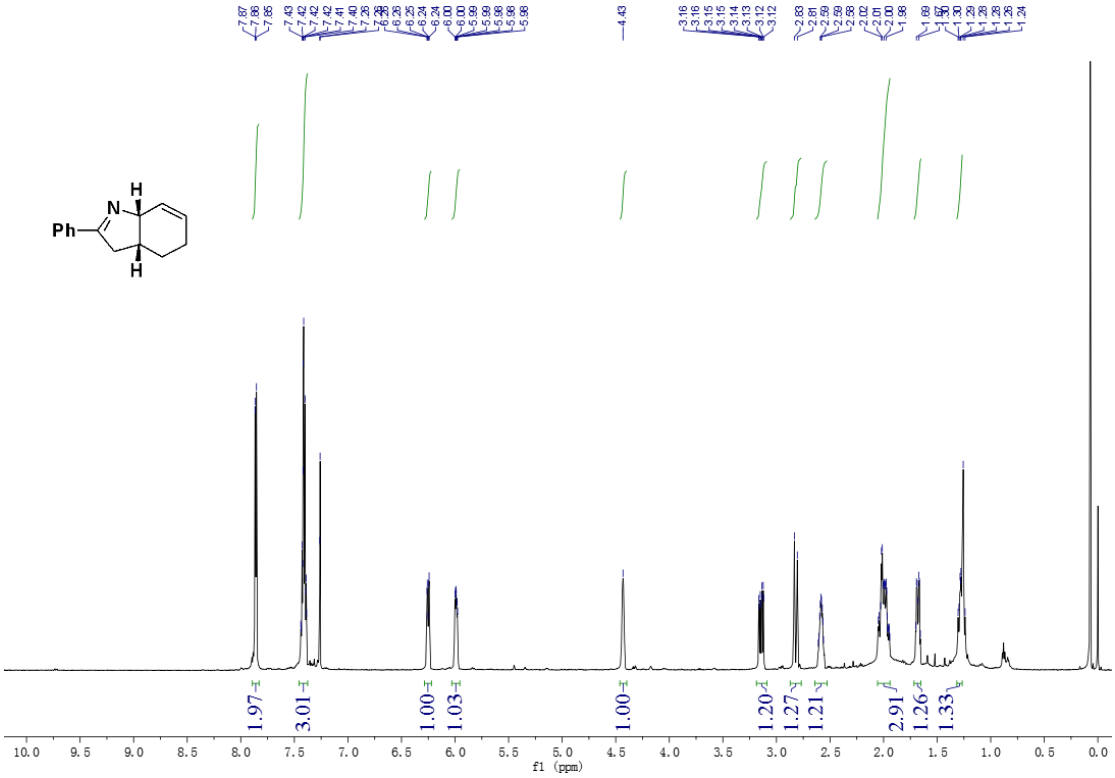
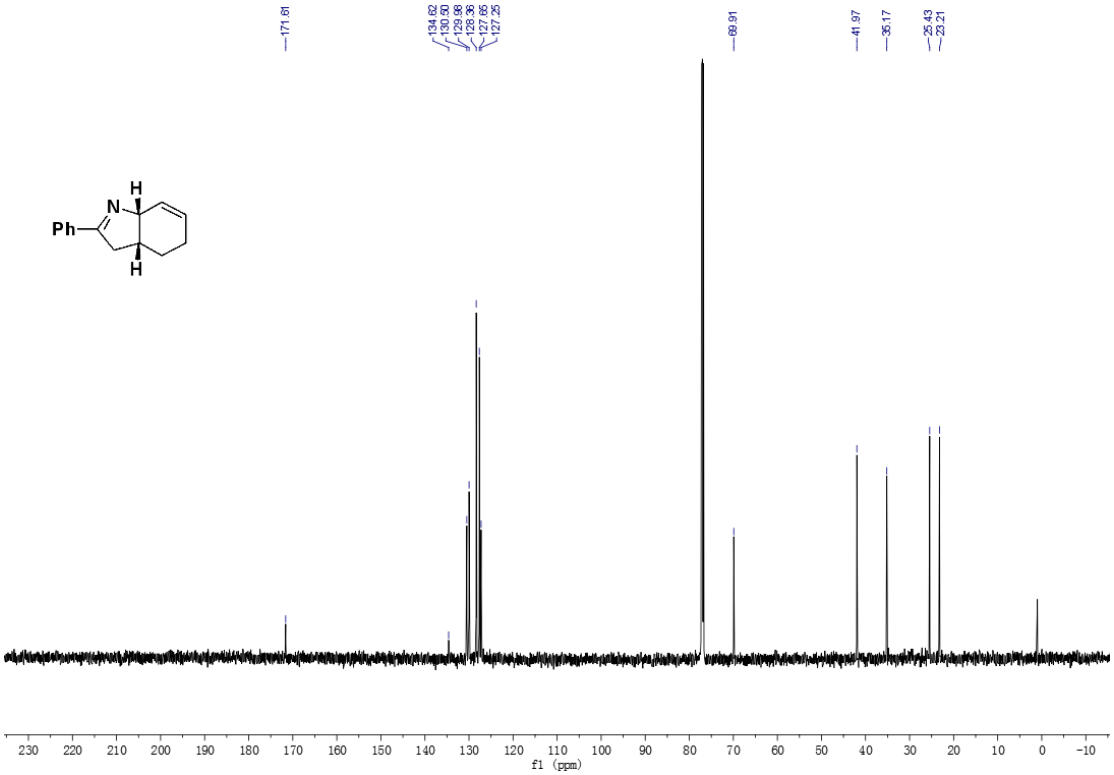


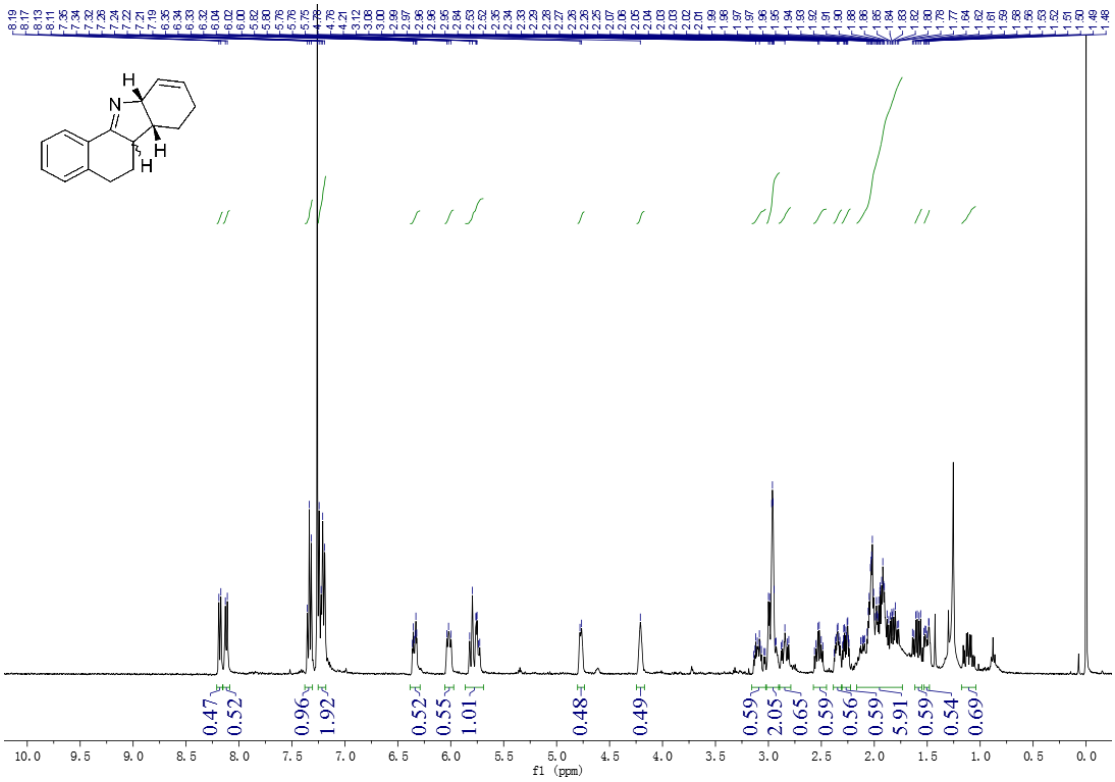
^{1}H NMR of **3d**



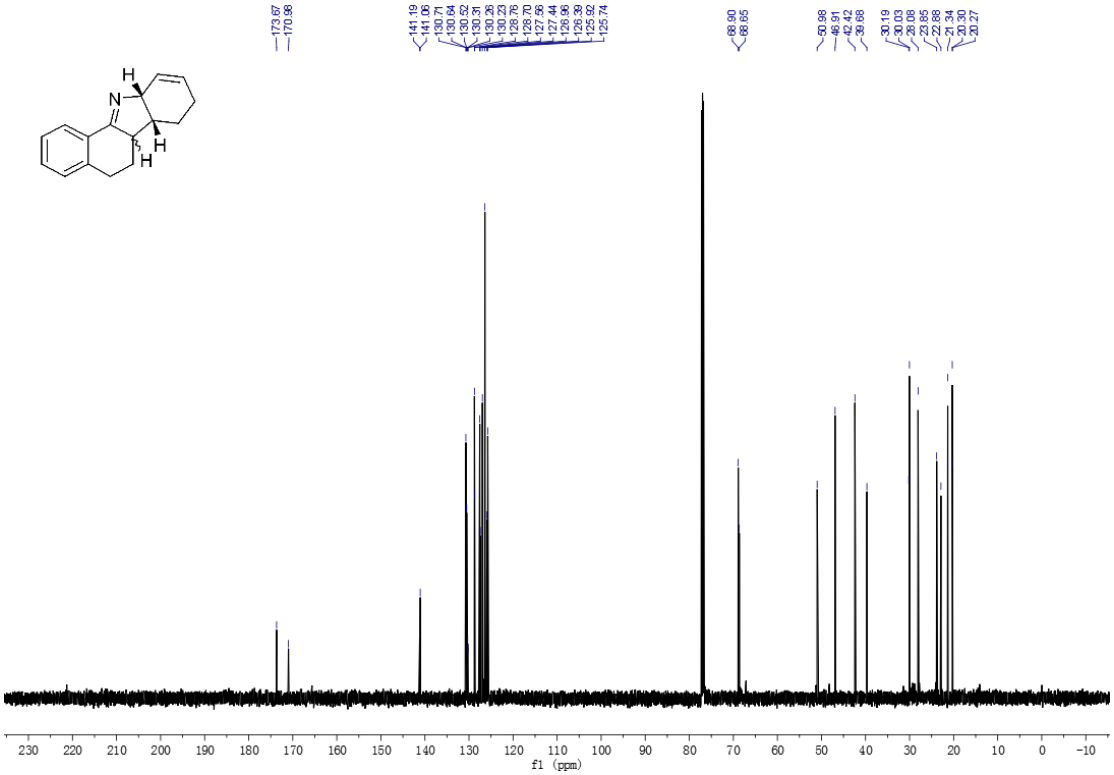
^{13}C NMR of **3d**



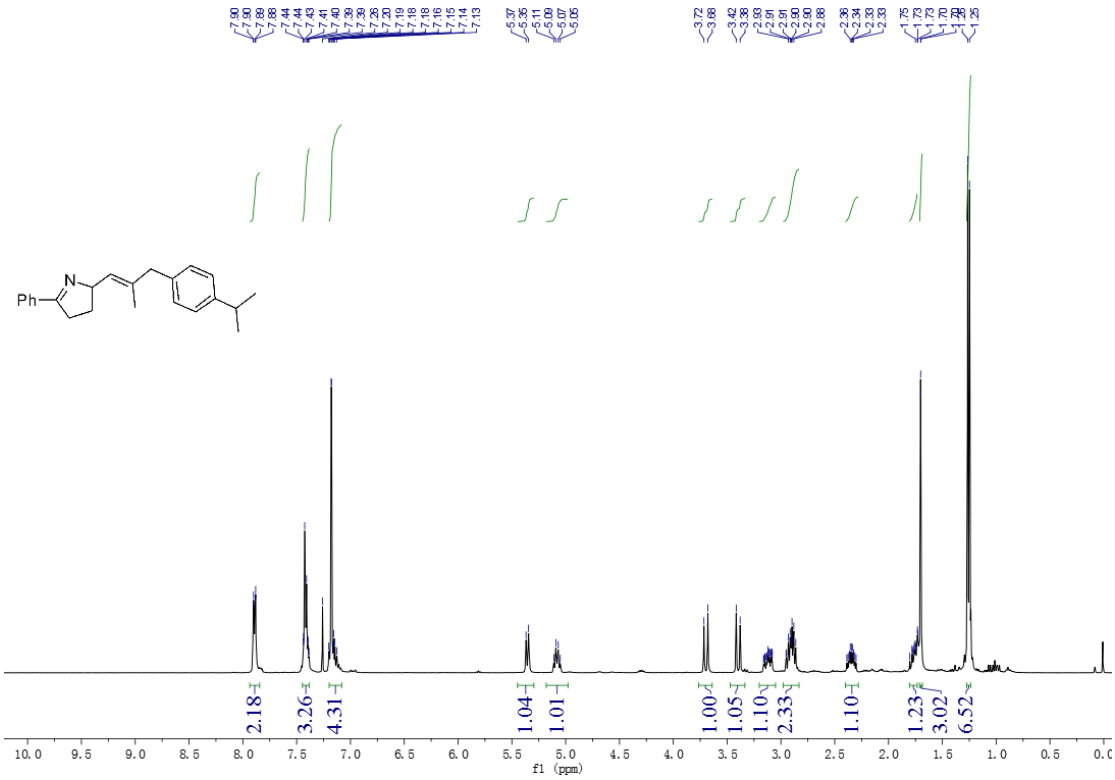
^{1}H NMR of **3e**



^{13}C NMR of **3e**



^{1}H NMR of **3f**



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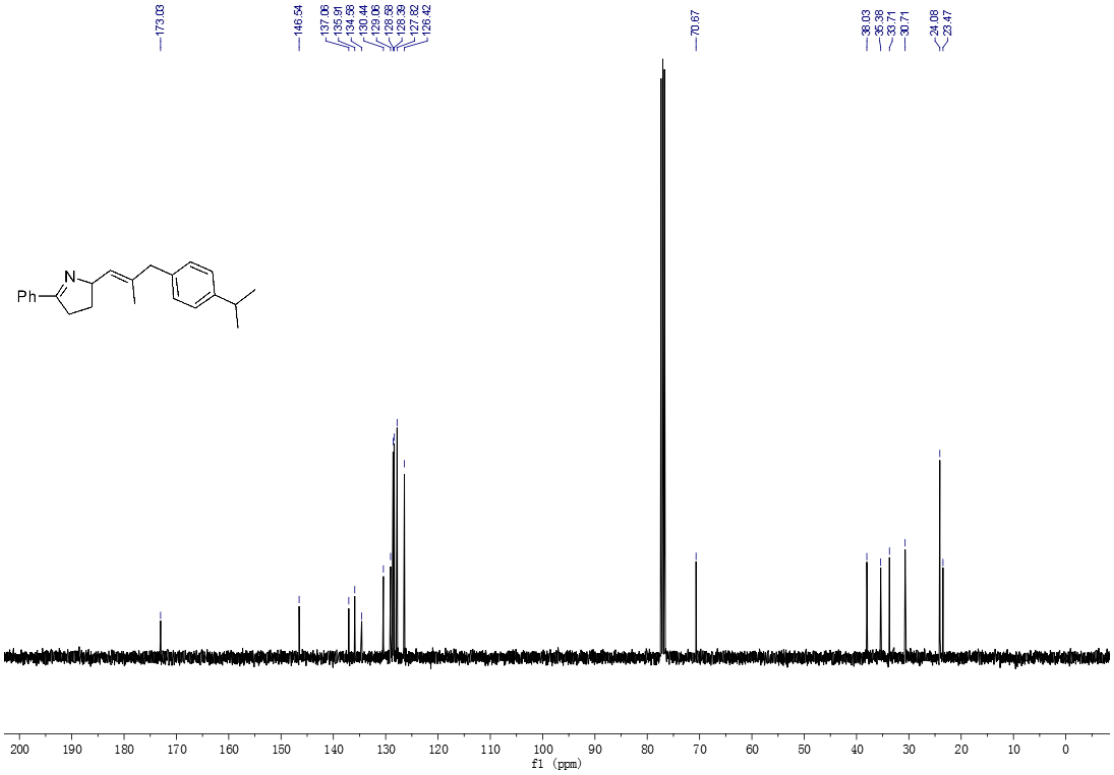
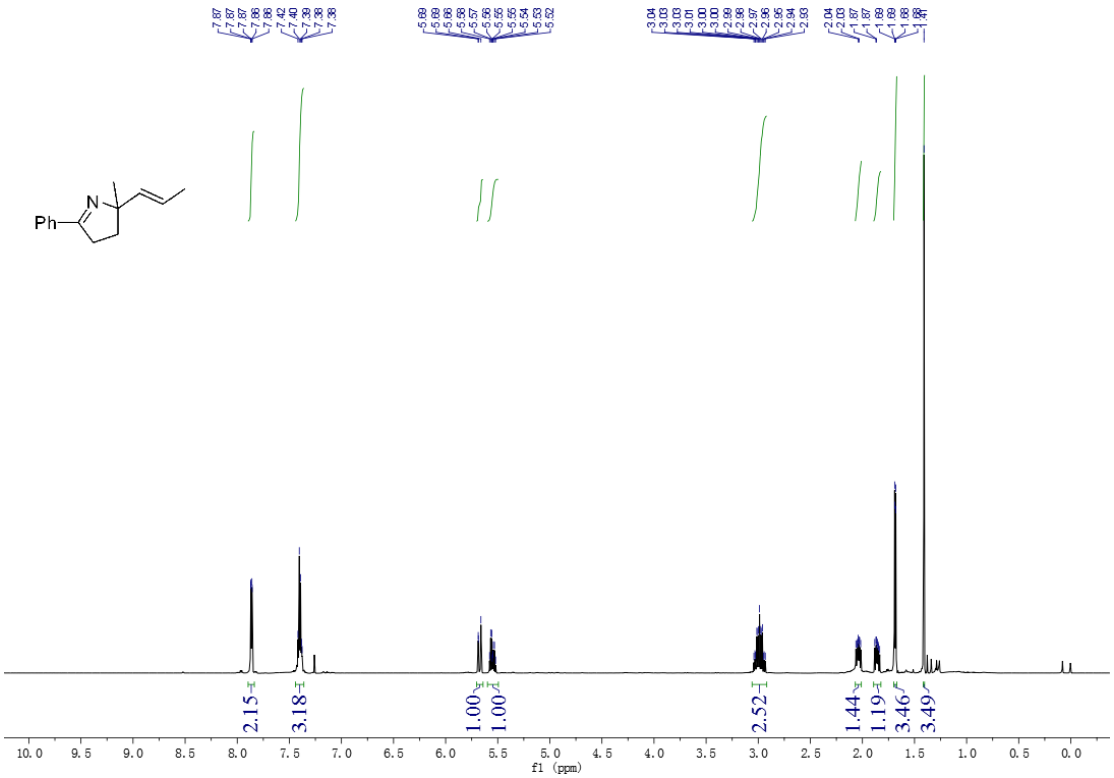
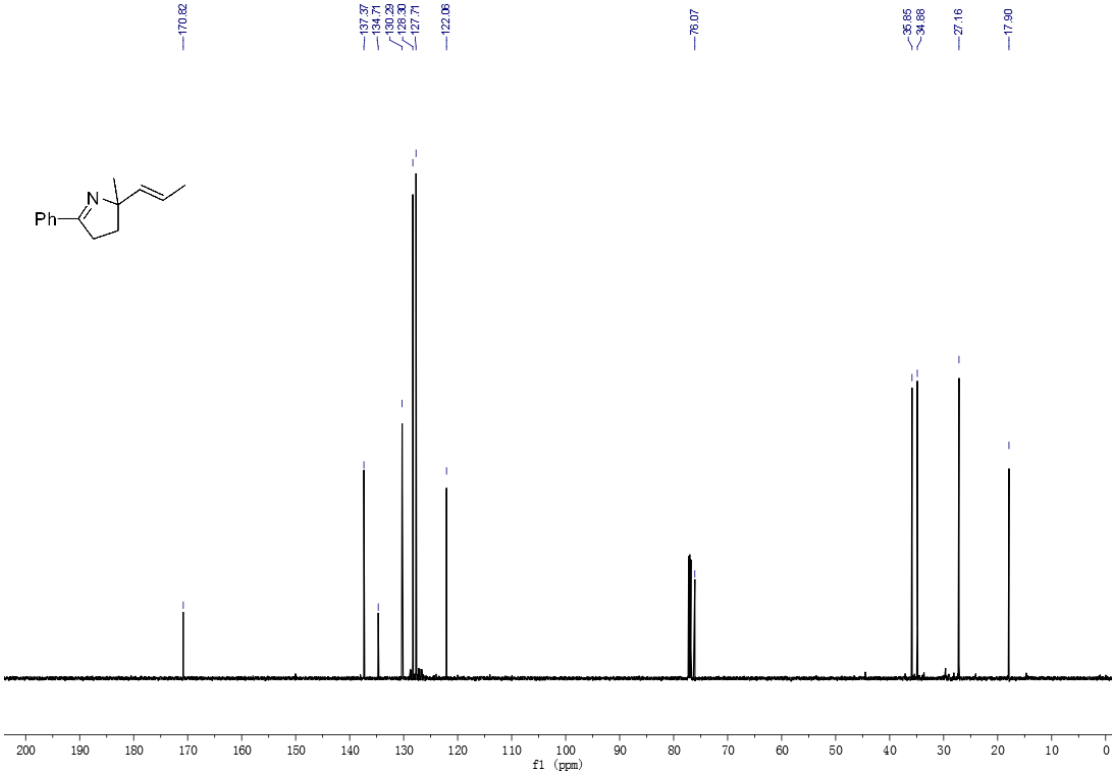


^{1}H NMR of **3g**



^{13}C NMR of **3g**



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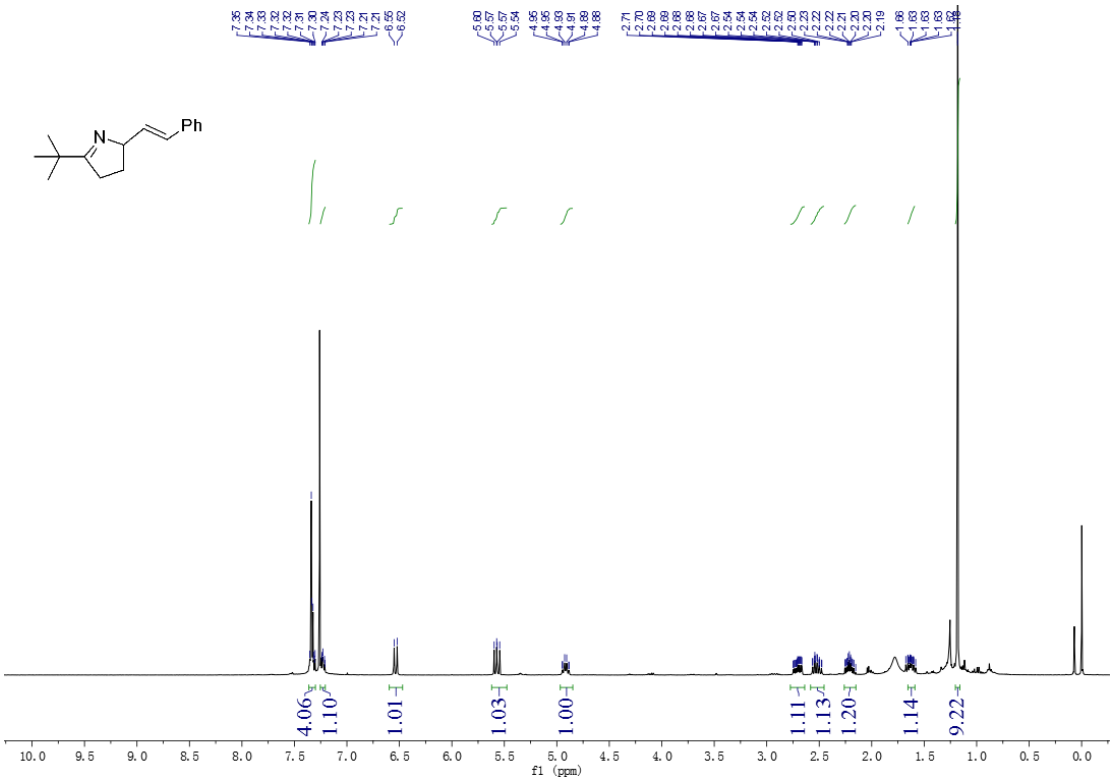

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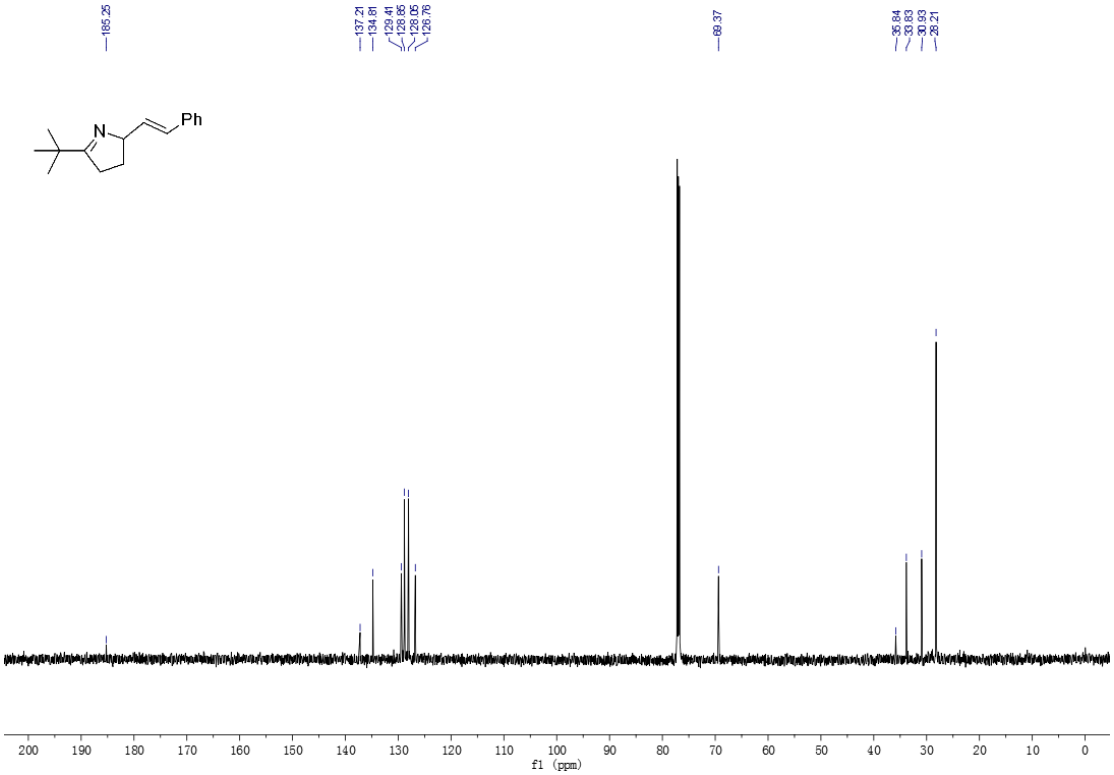
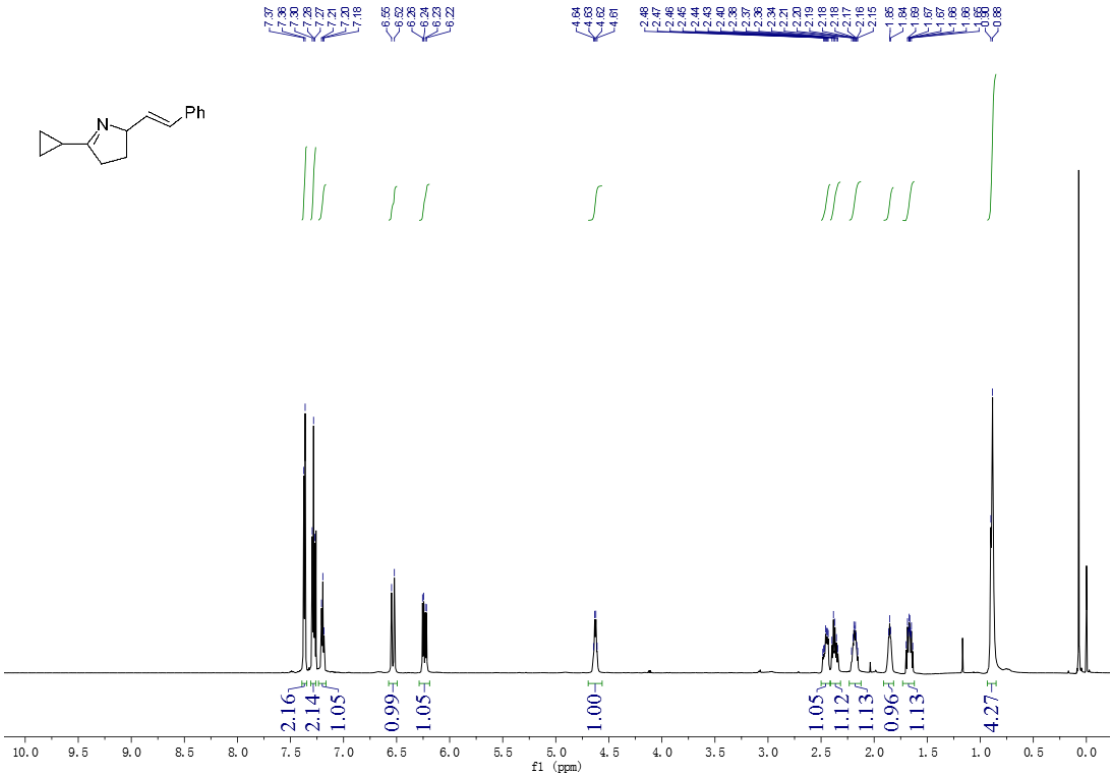


^{1}H NMR of **3i**



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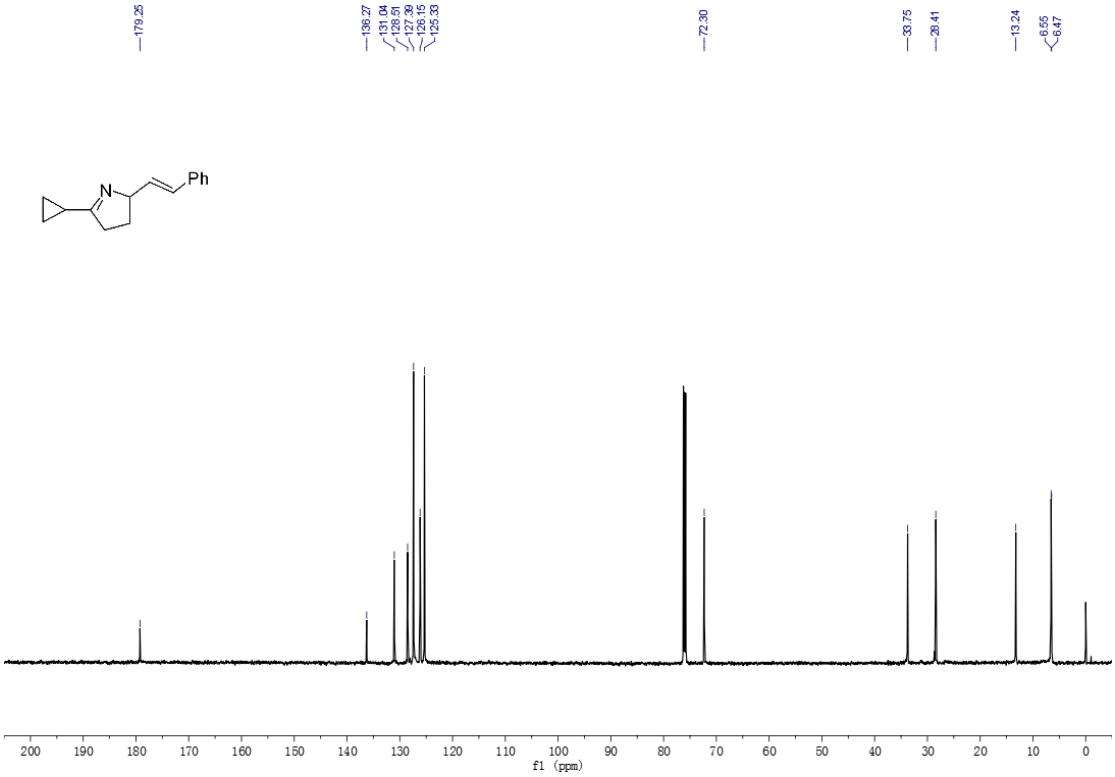
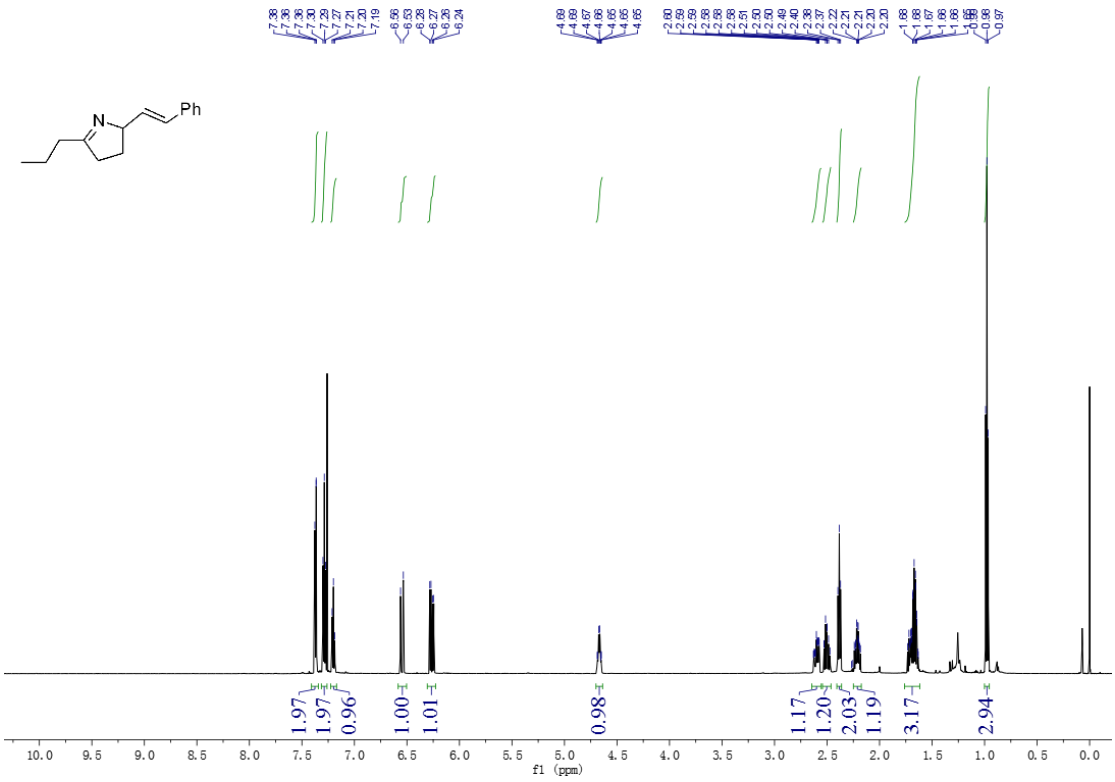
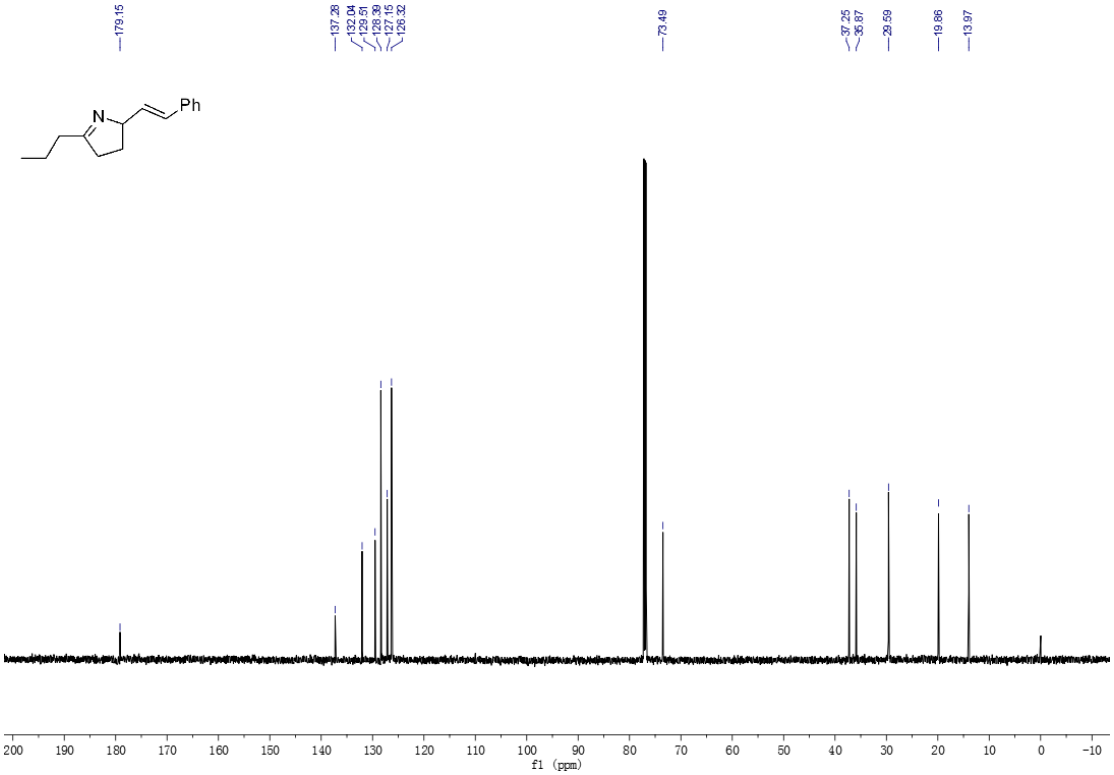


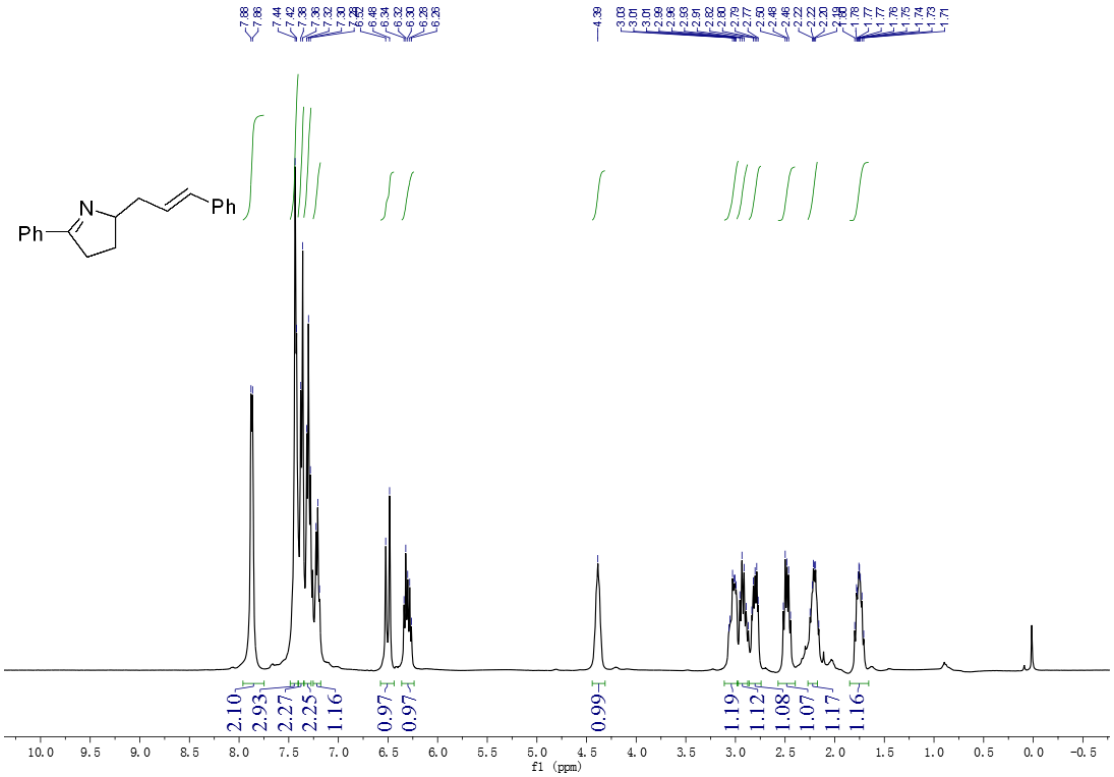
^{1}H NMR of **3j**



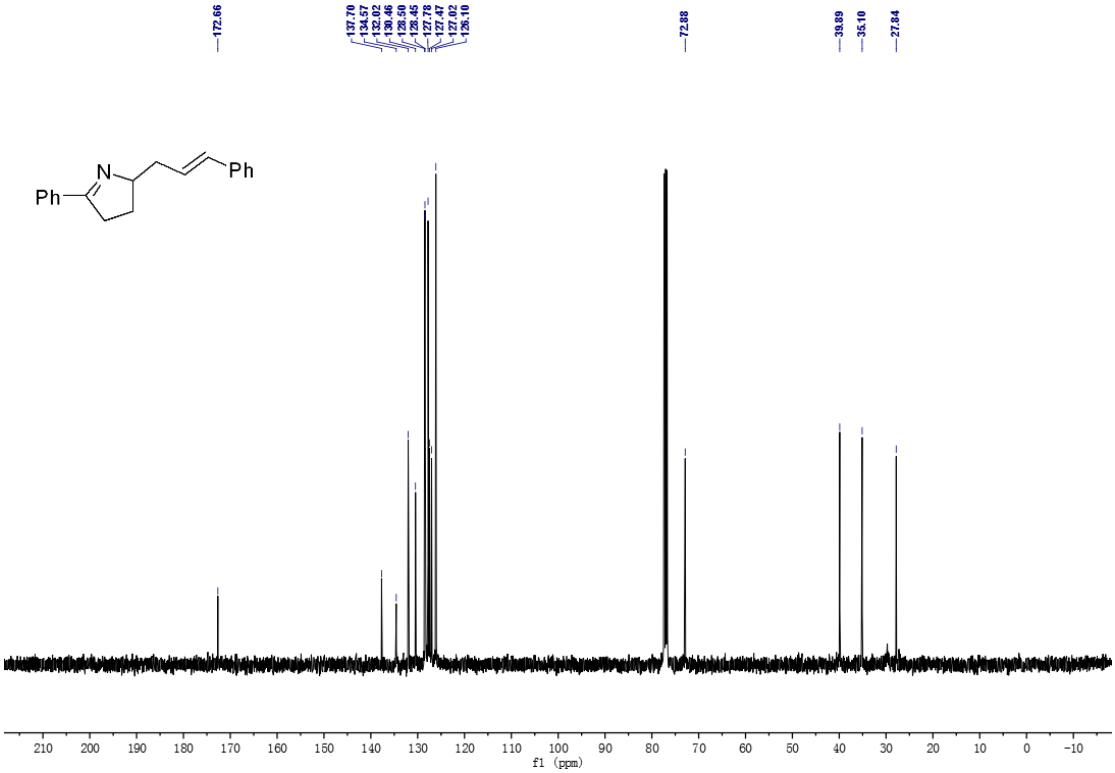
^{13}C NMR of **3j**



^{1}H NMR of **5a**



^{13}C NMR of **5a**



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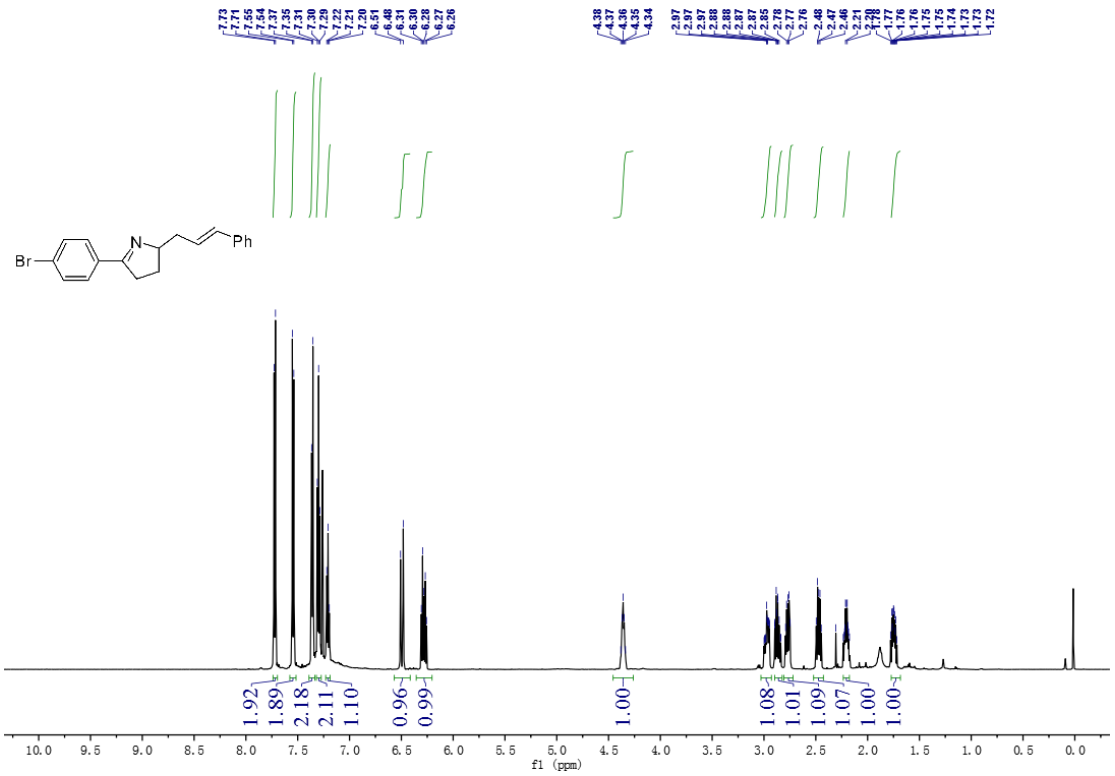

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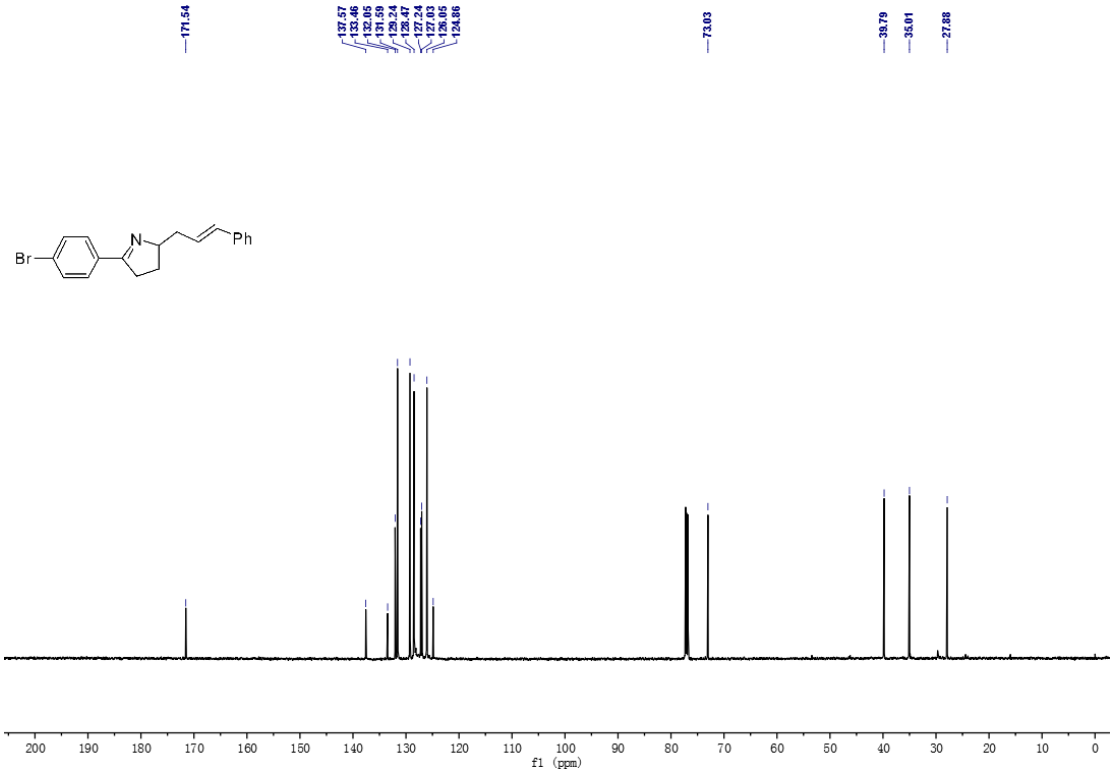

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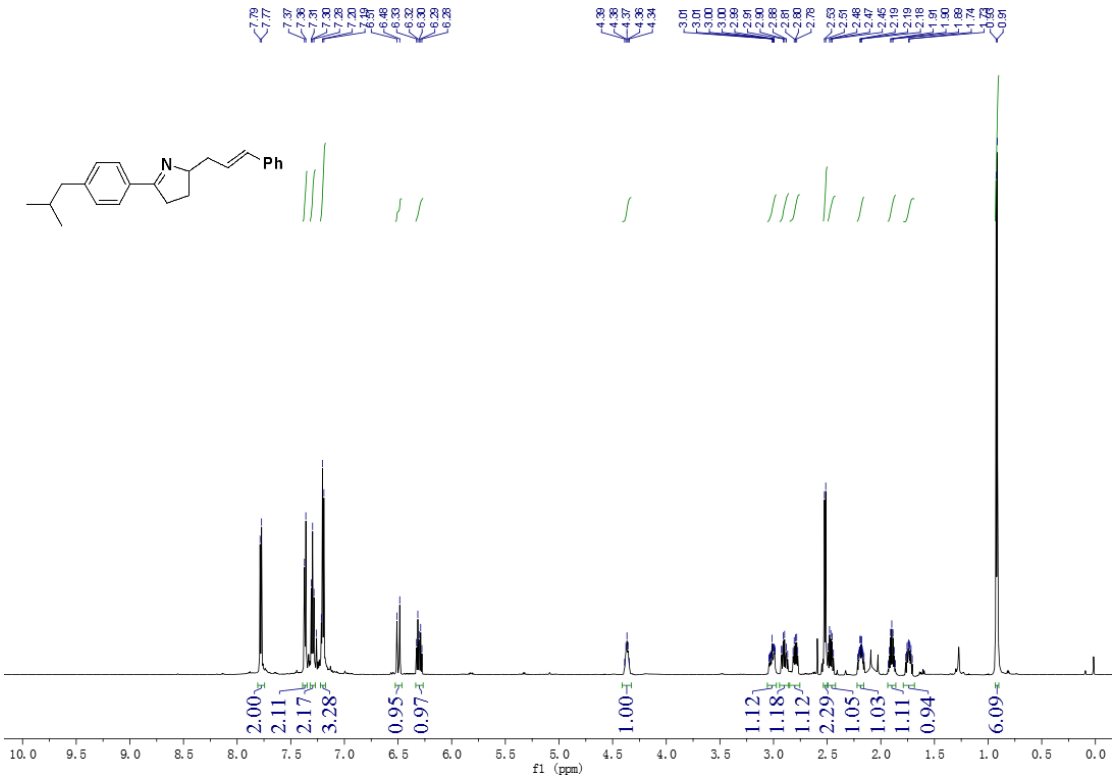
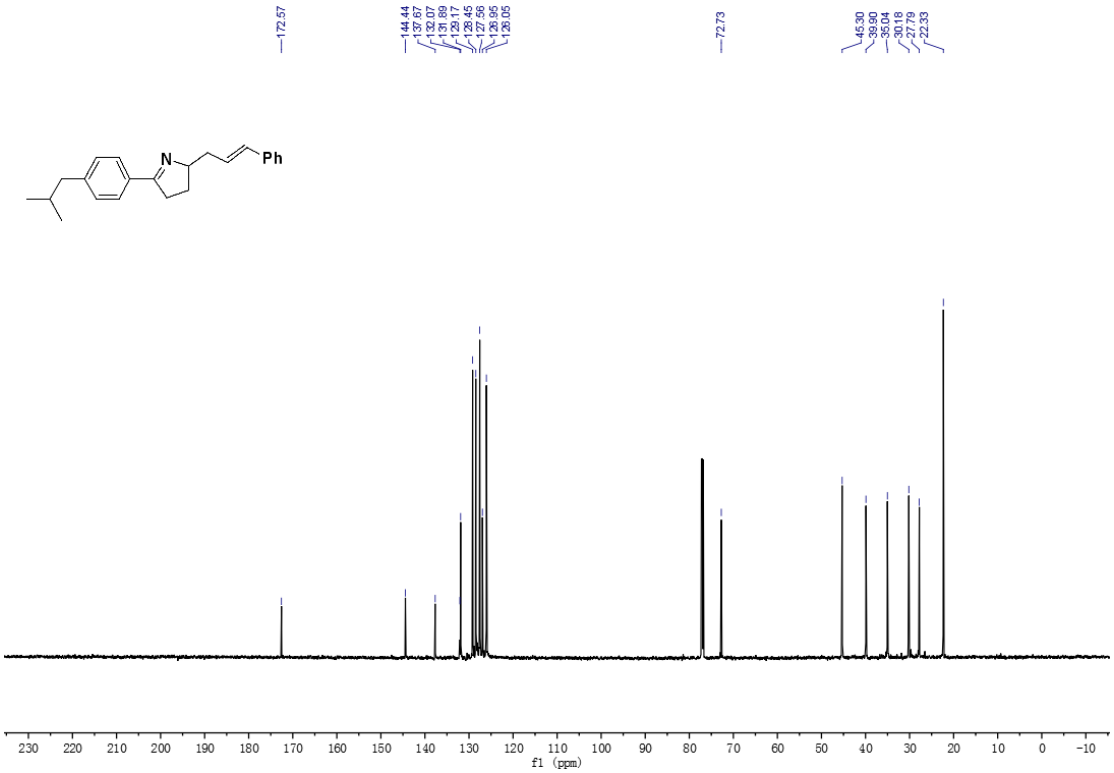
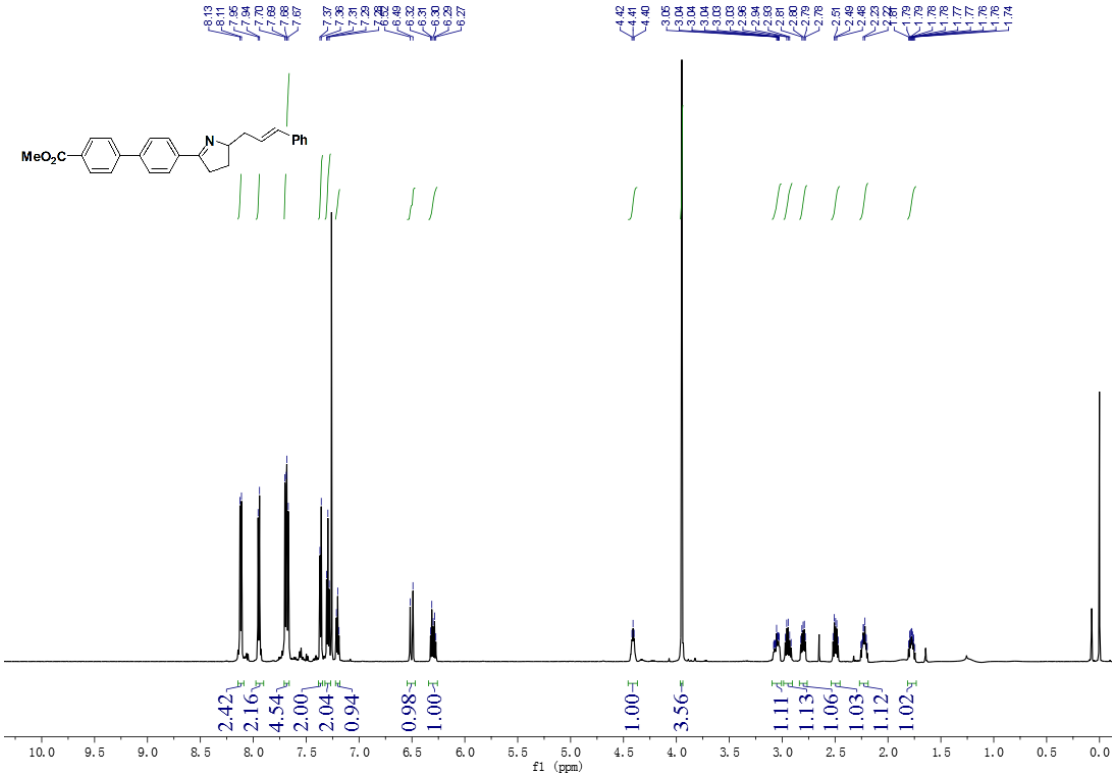


^{13}C NMR of **5c**



^{1}H NMR of **5d**



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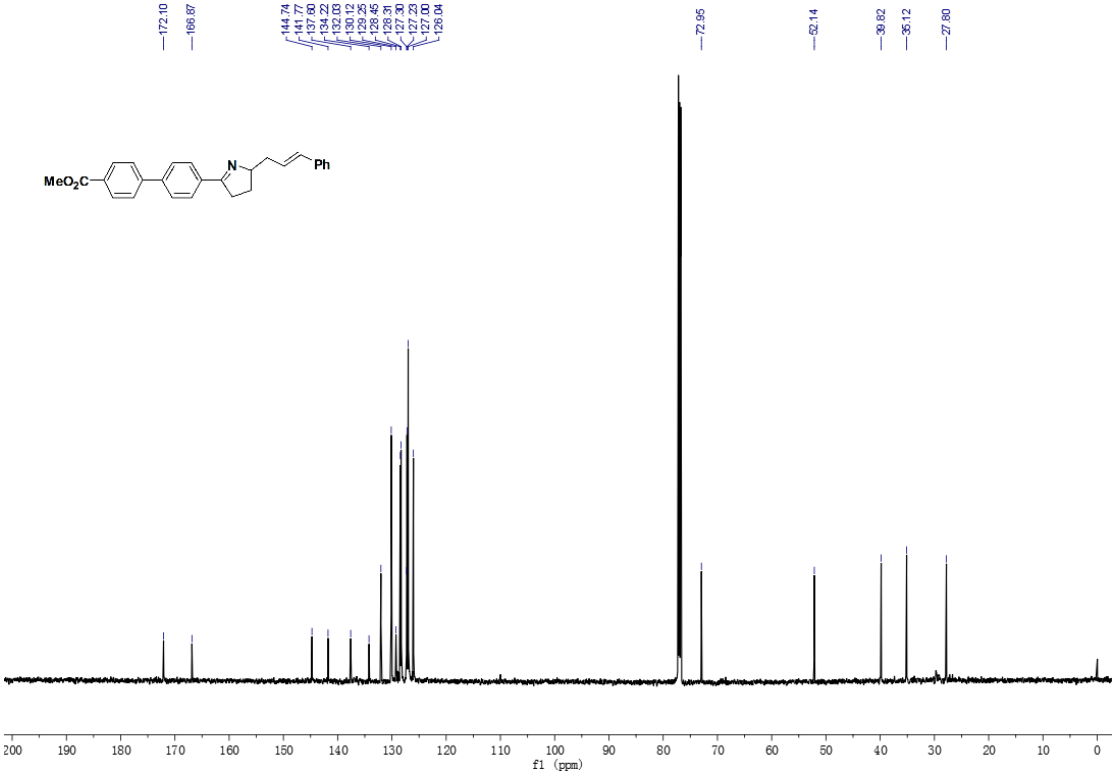
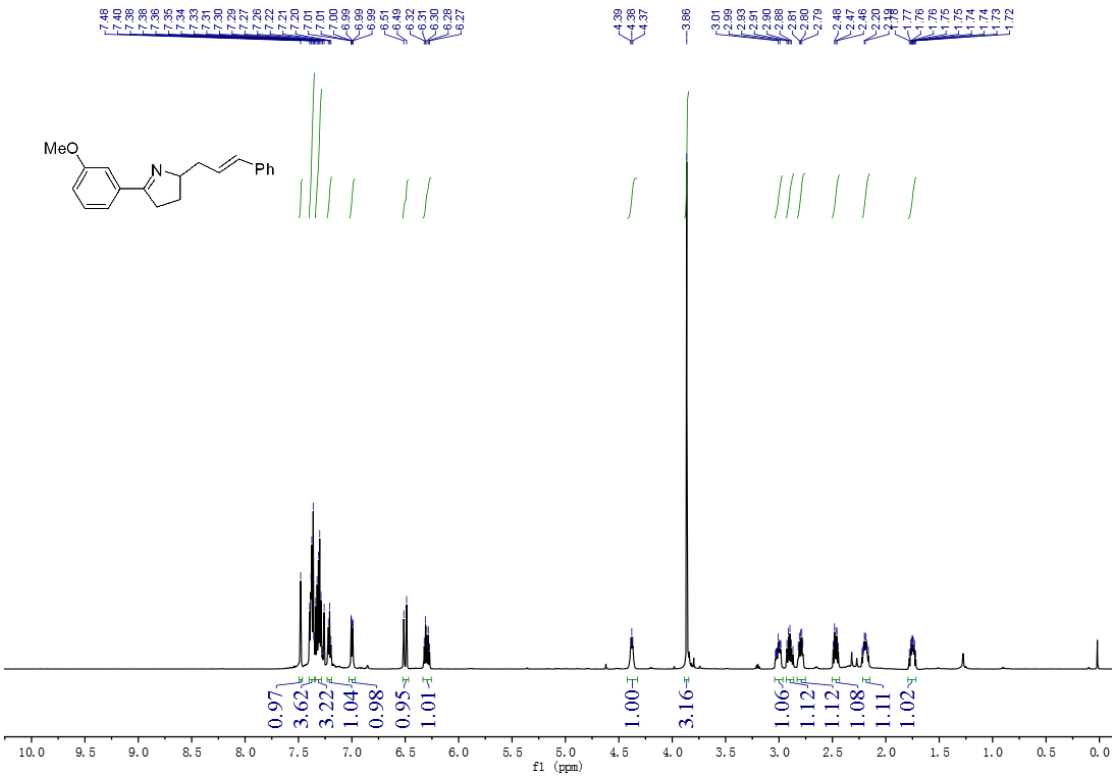


^{1}H NMR of **5e**



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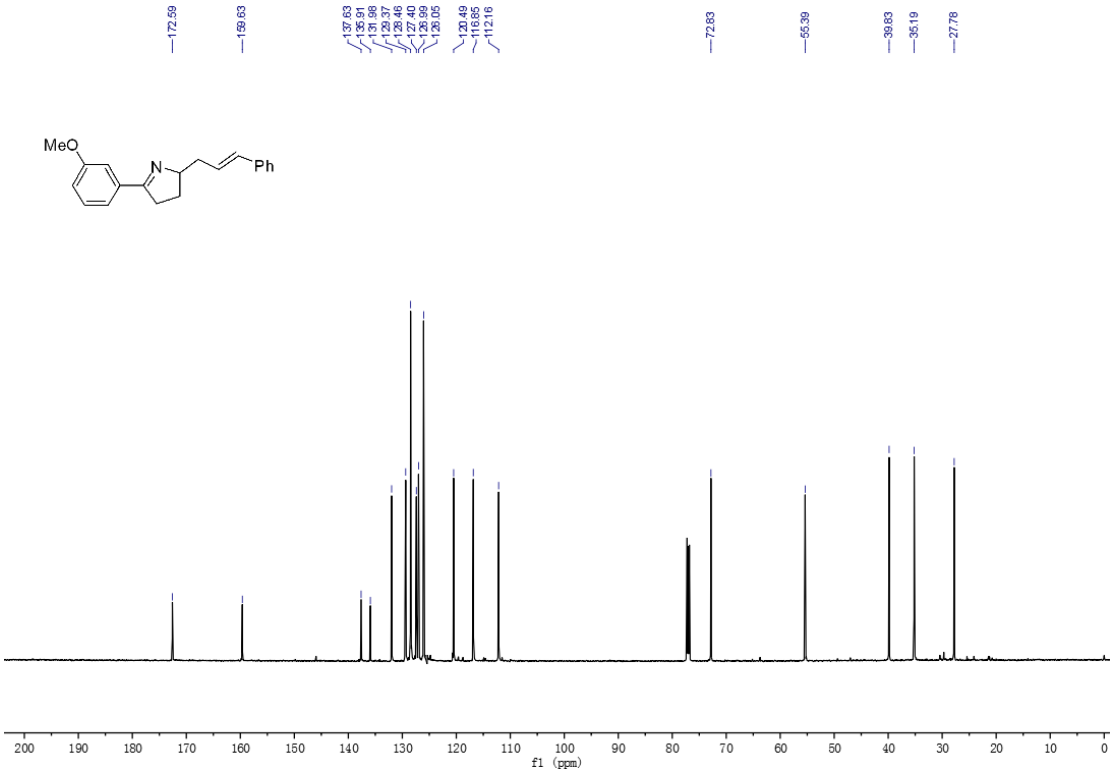

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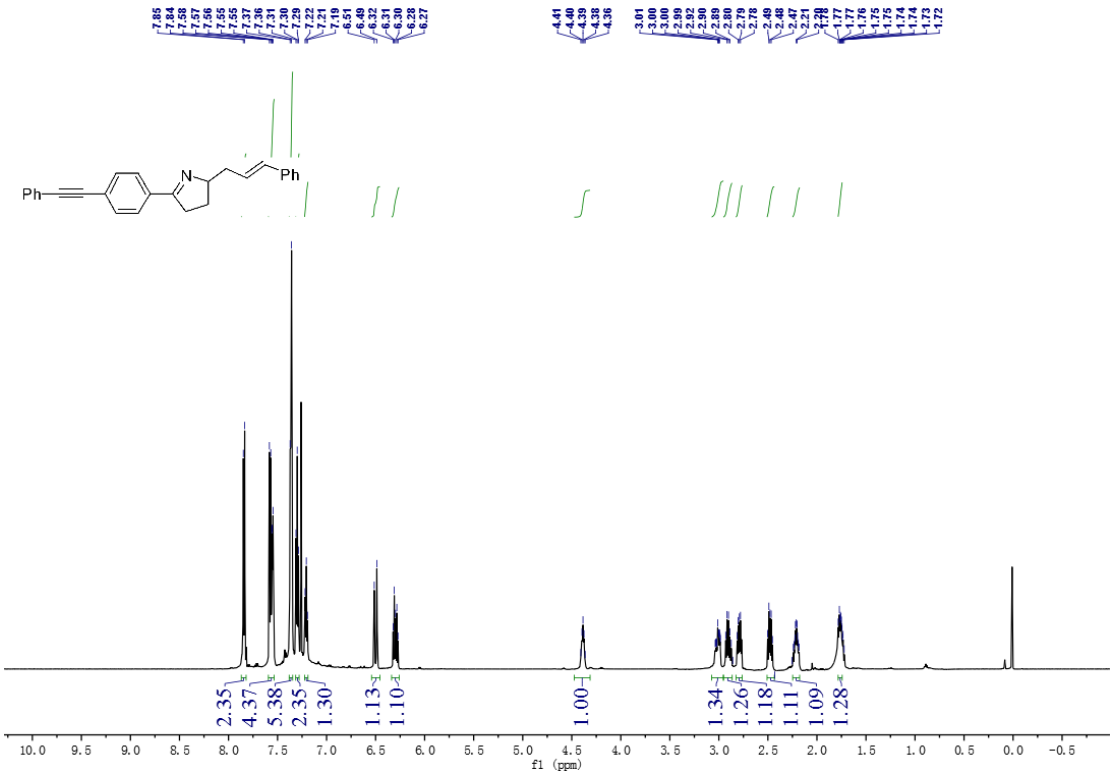
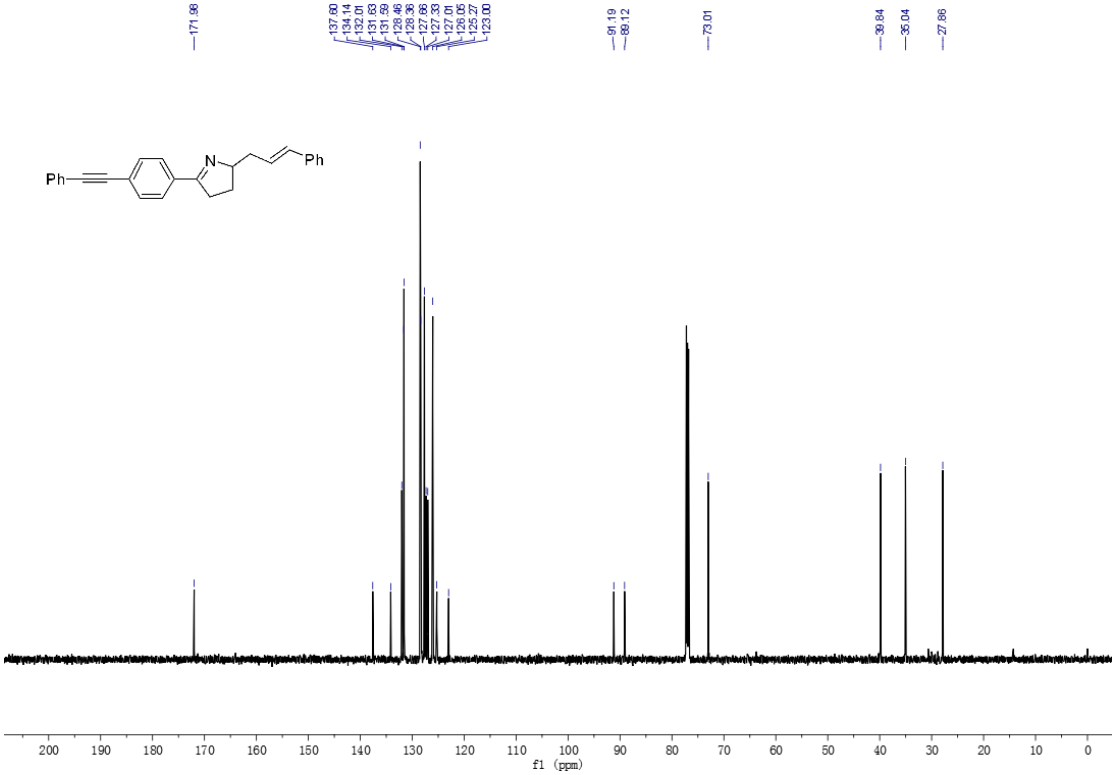


^{13}C NMR of **5f**



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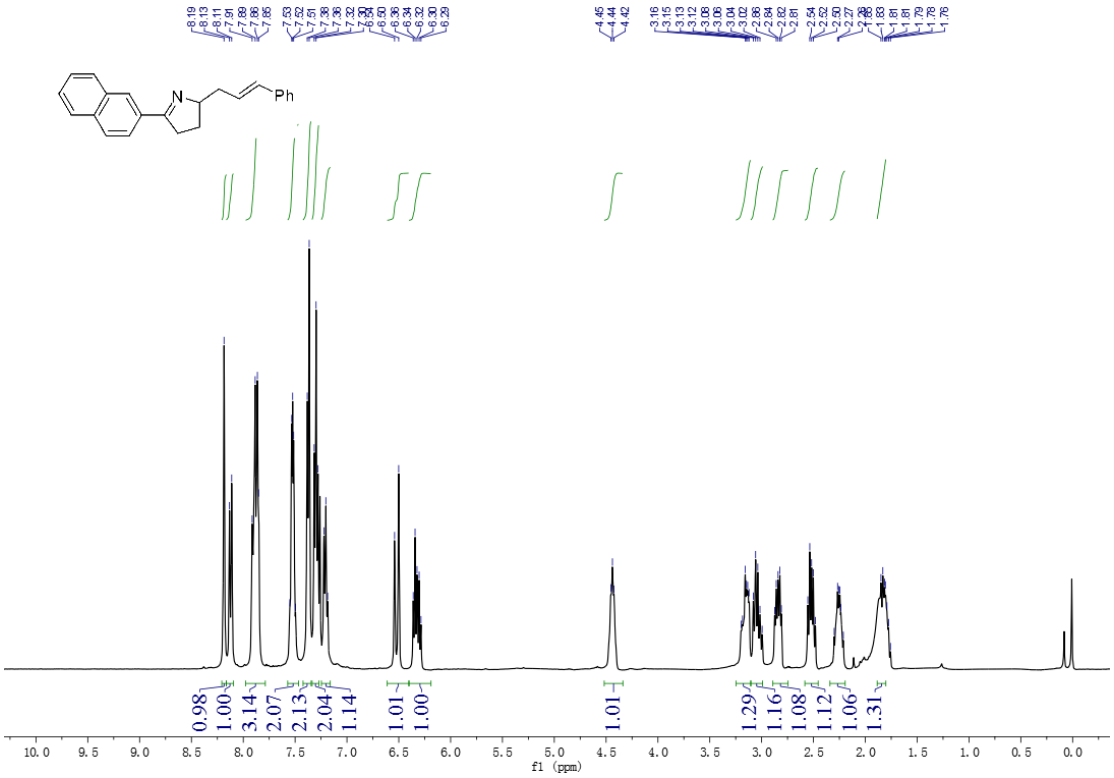
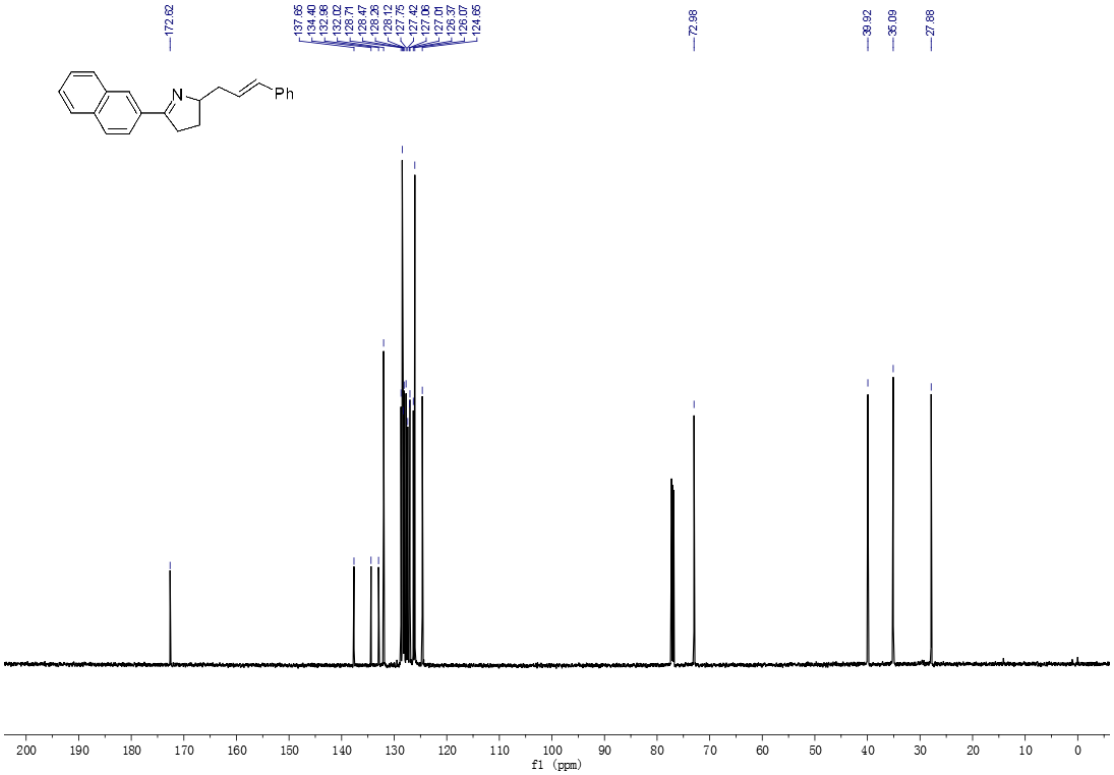
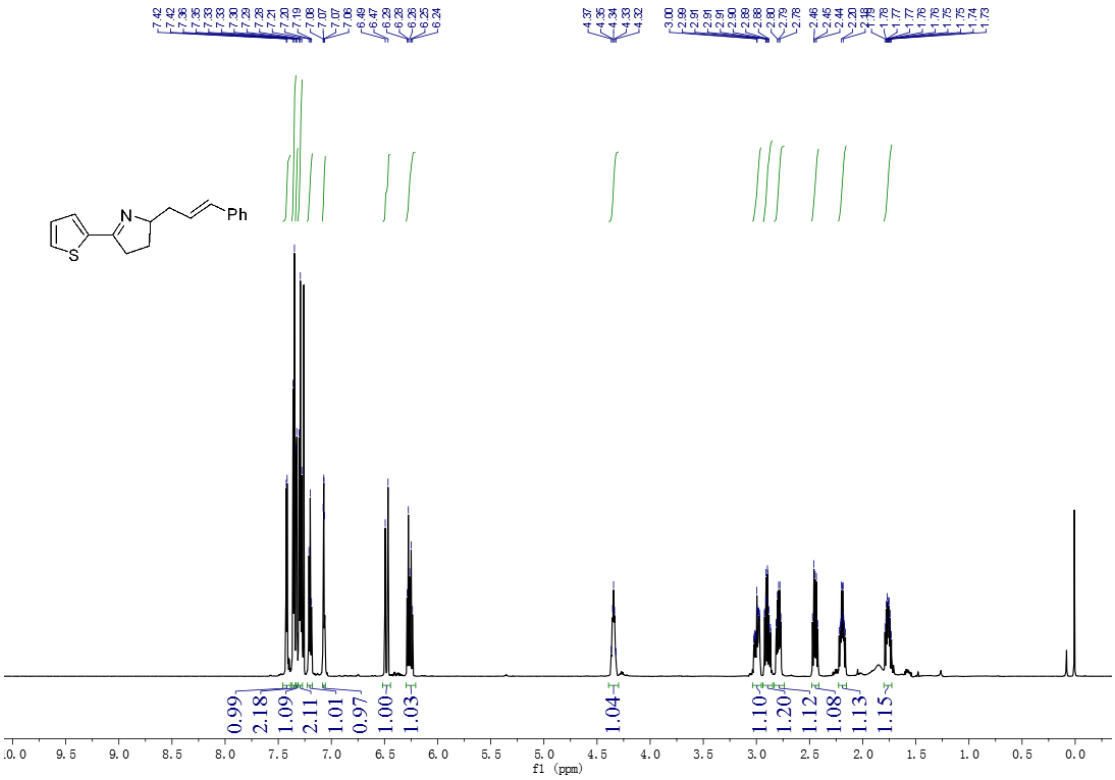


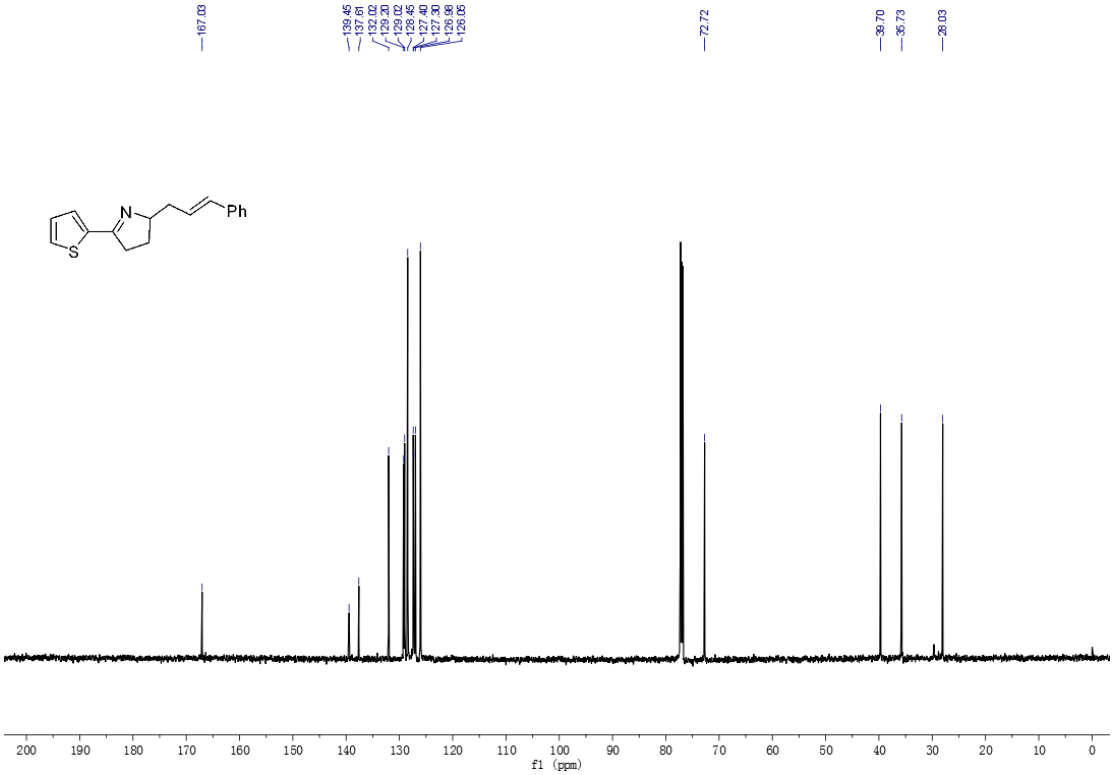
^{13}C NMR of **5g**



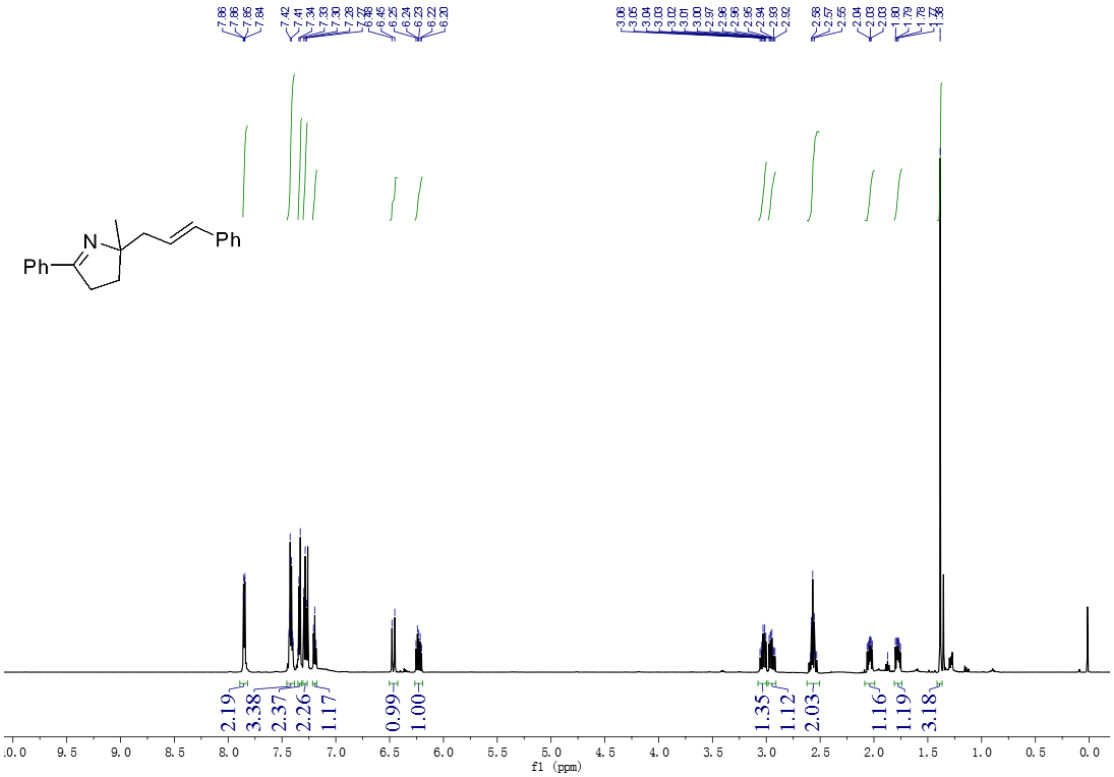
^{1}H NMR of **5h**



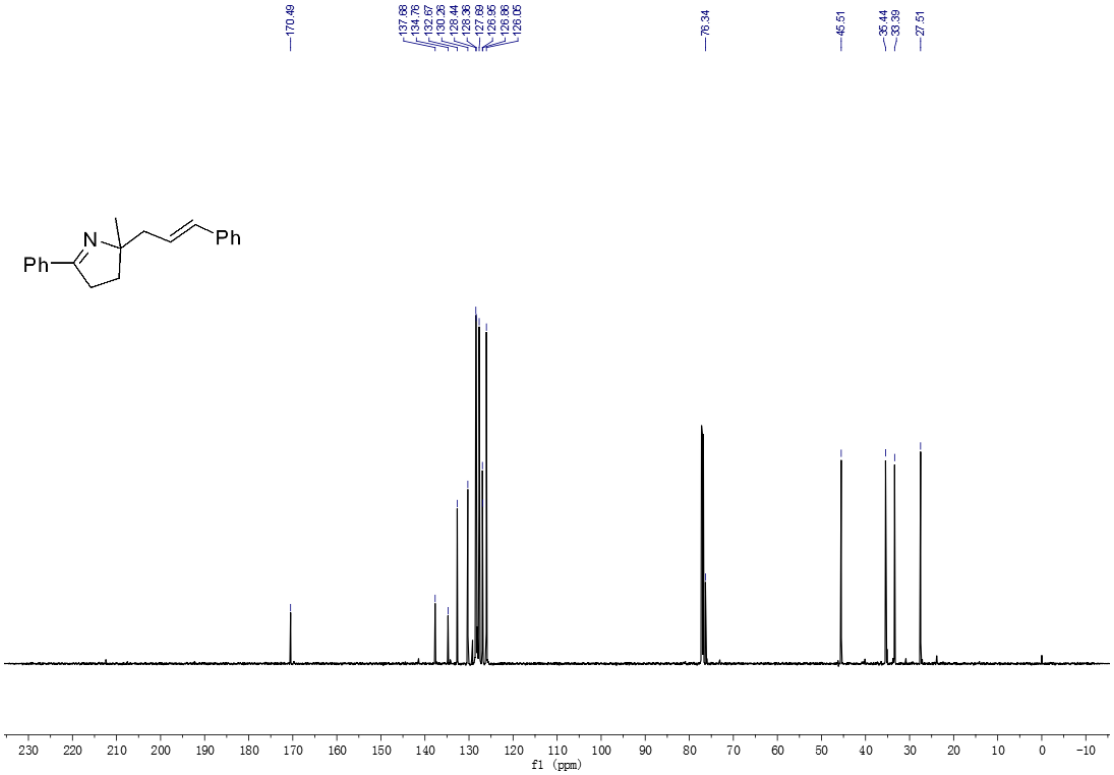
^{13}C NMR of **5h**



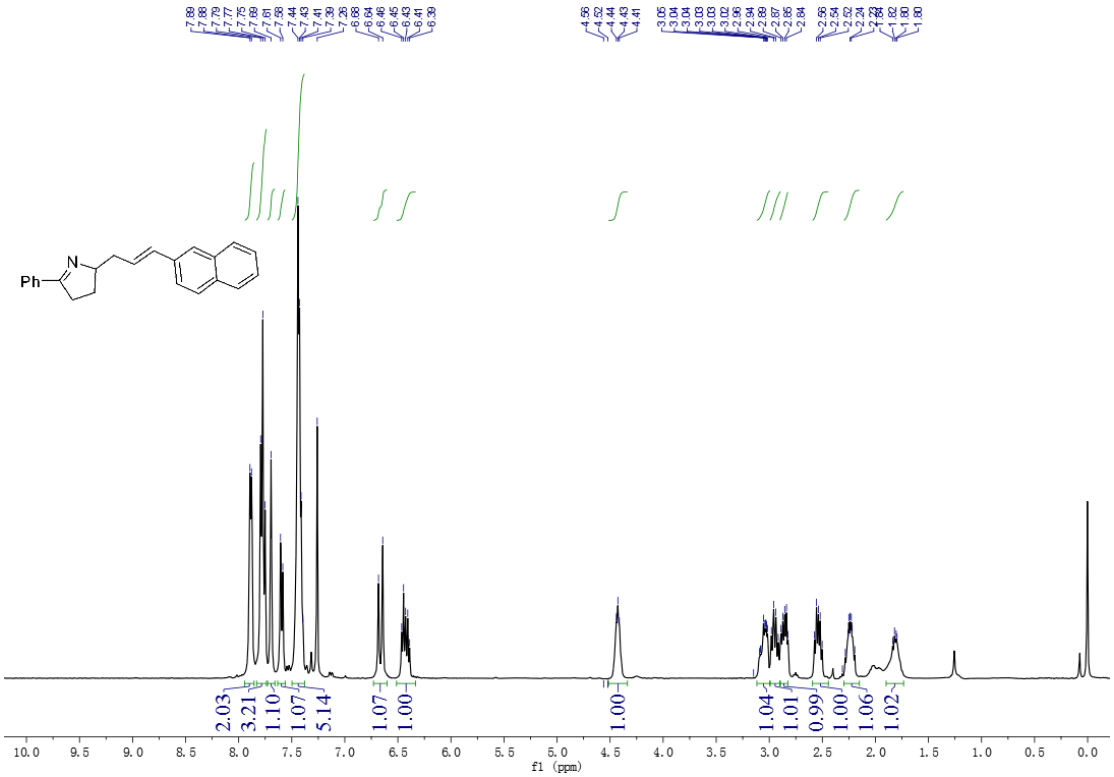
^{1}H NMR of **5i**



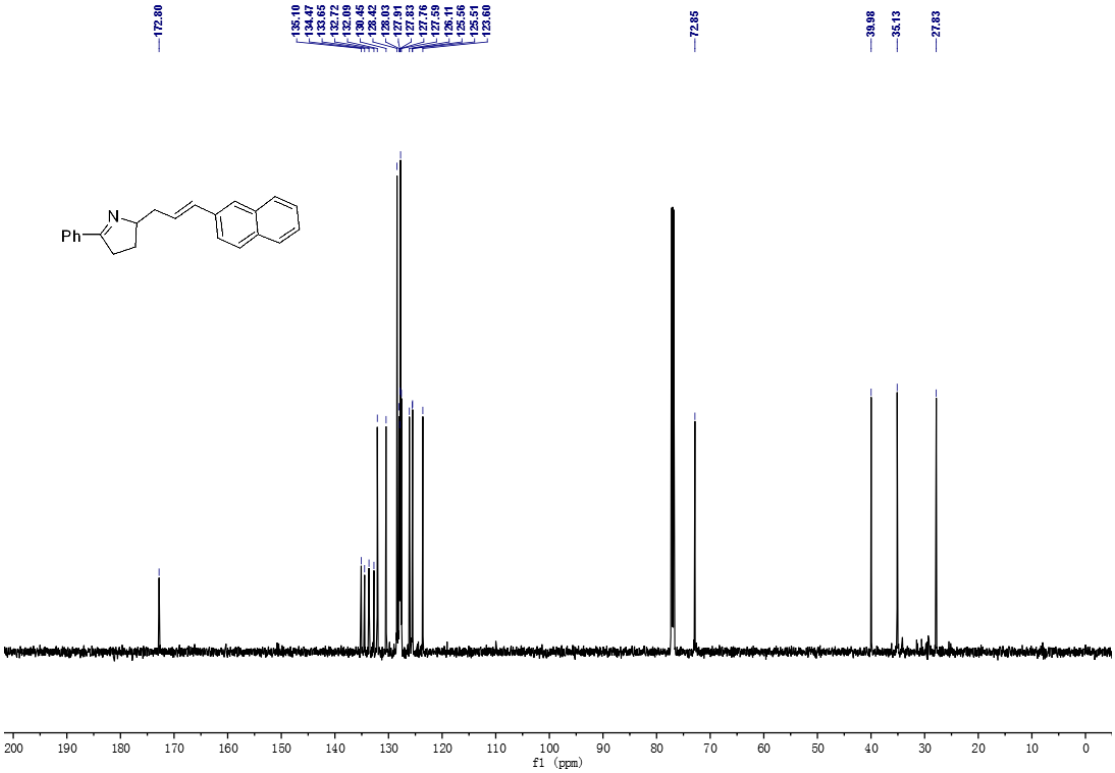
^{13}C NMR of **5i**



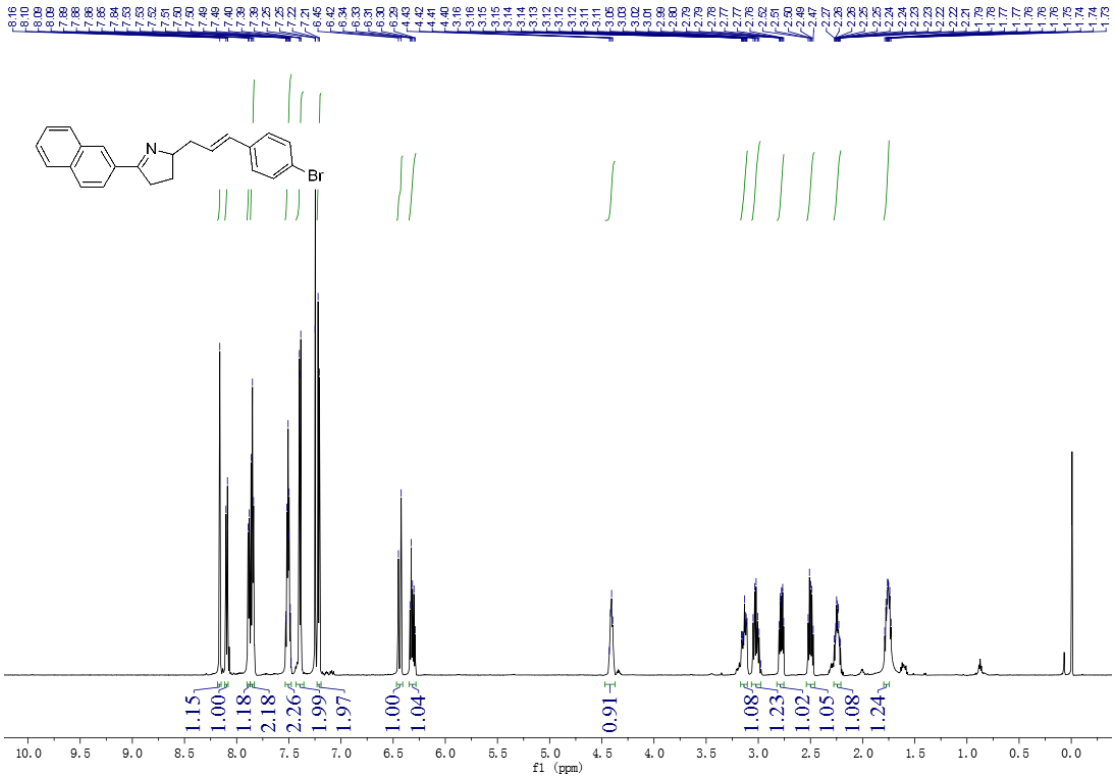
^{1}H NMR of **5j**



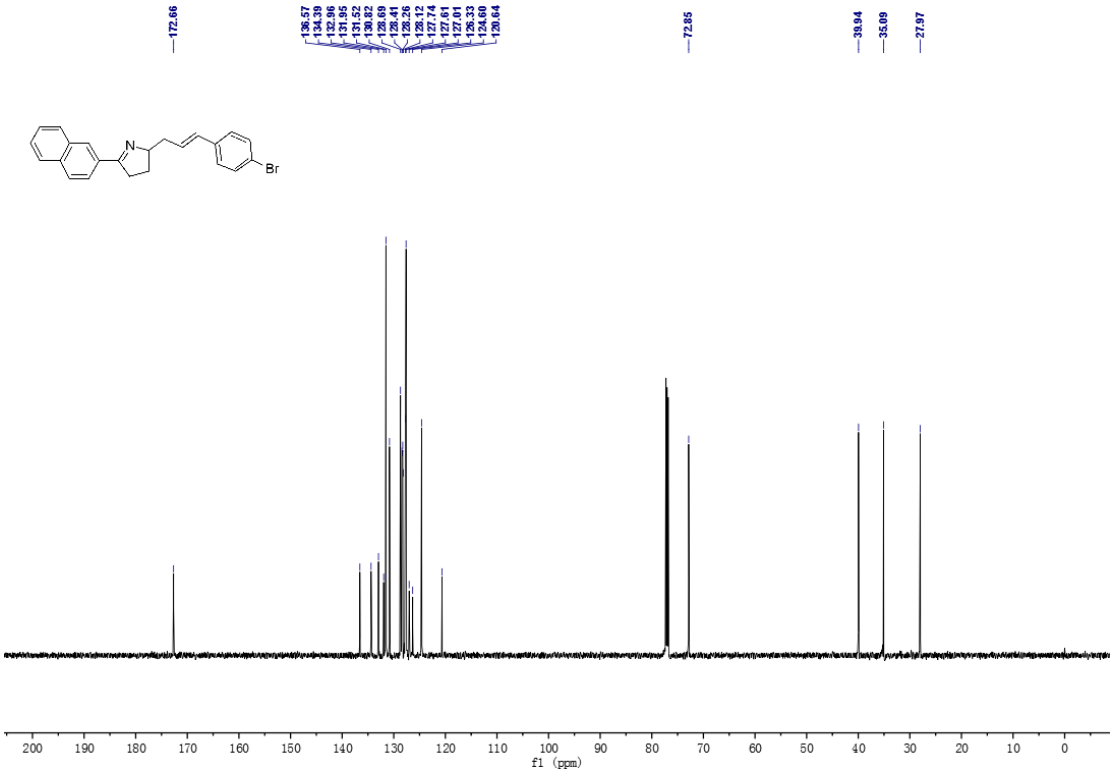
^{13}C NMR of **5j**



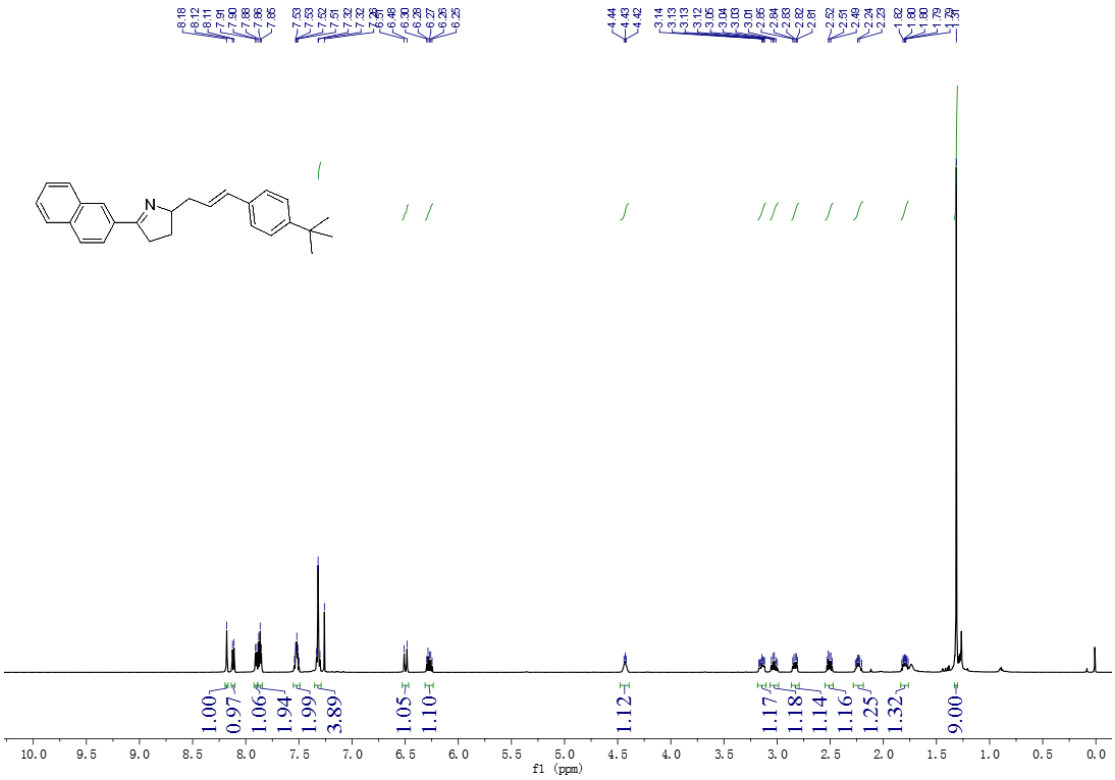
^{1}H NMR of **5k**



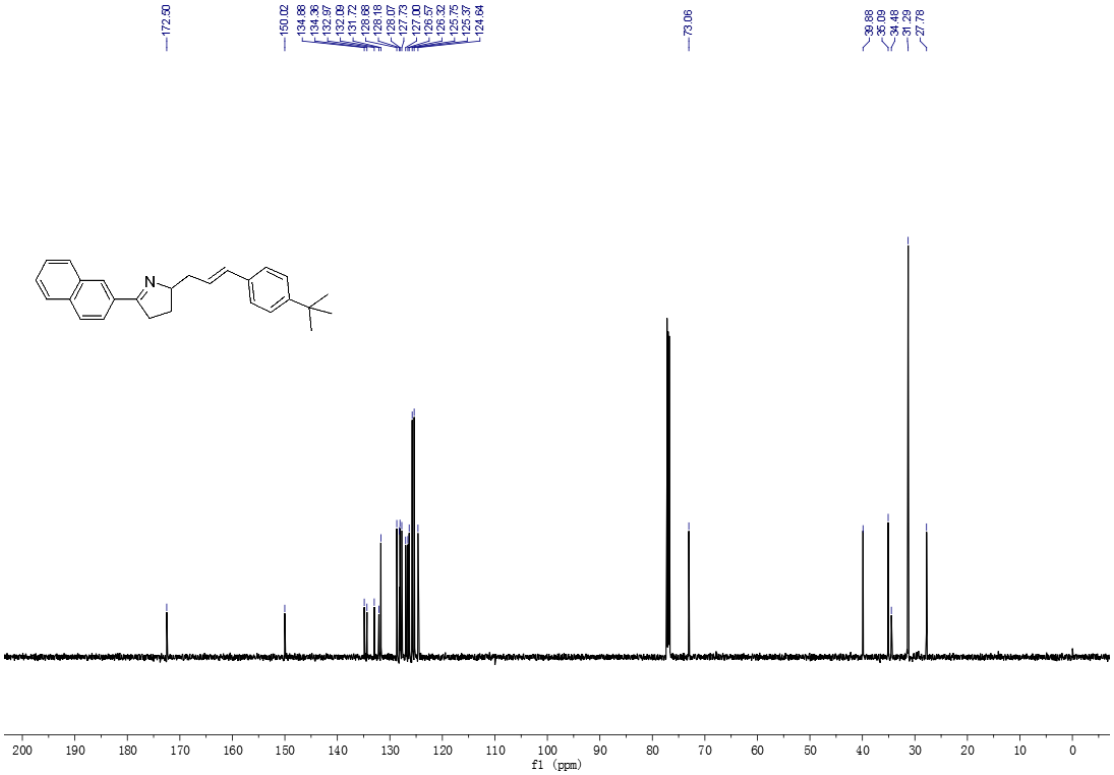
^{13}C NMR of **5k**



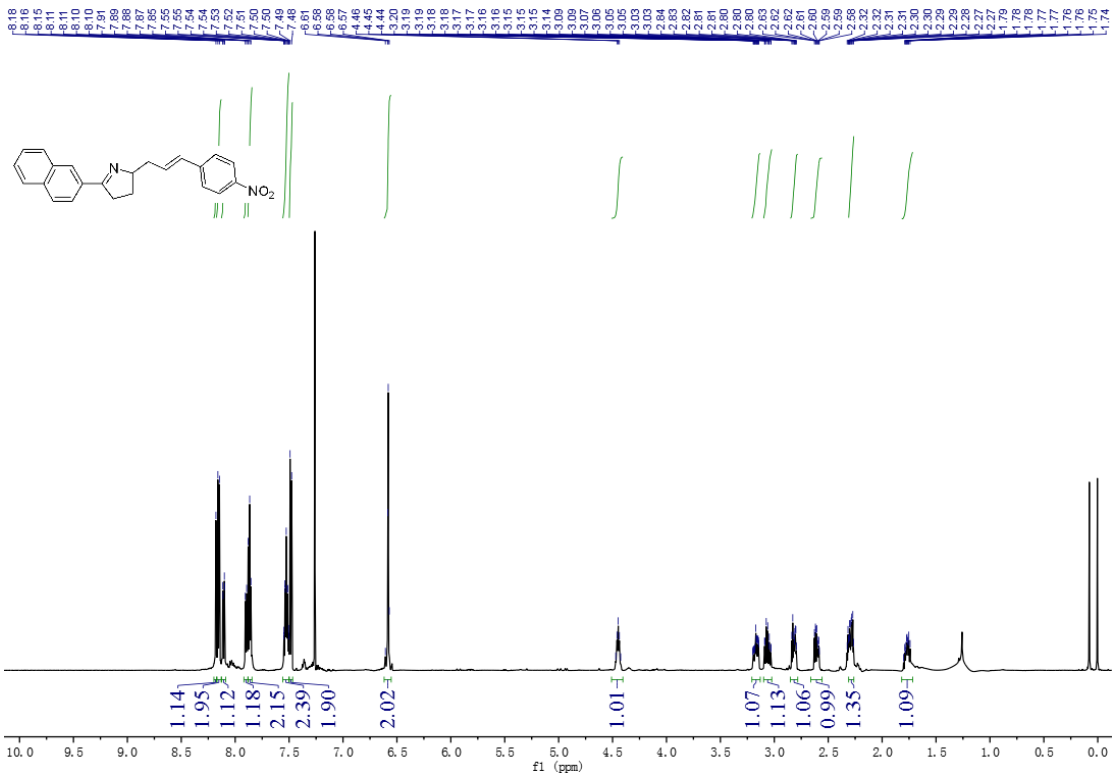
^{1}H NMR of **5l**



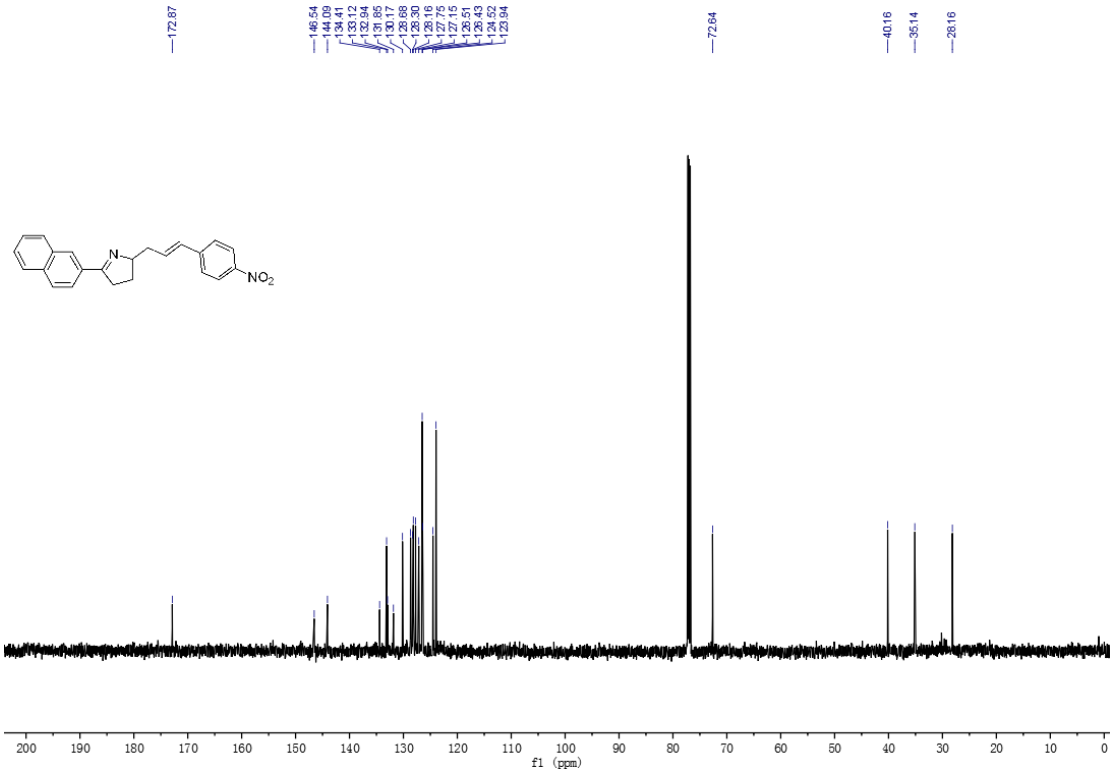
^{13}C NMR of **5l**



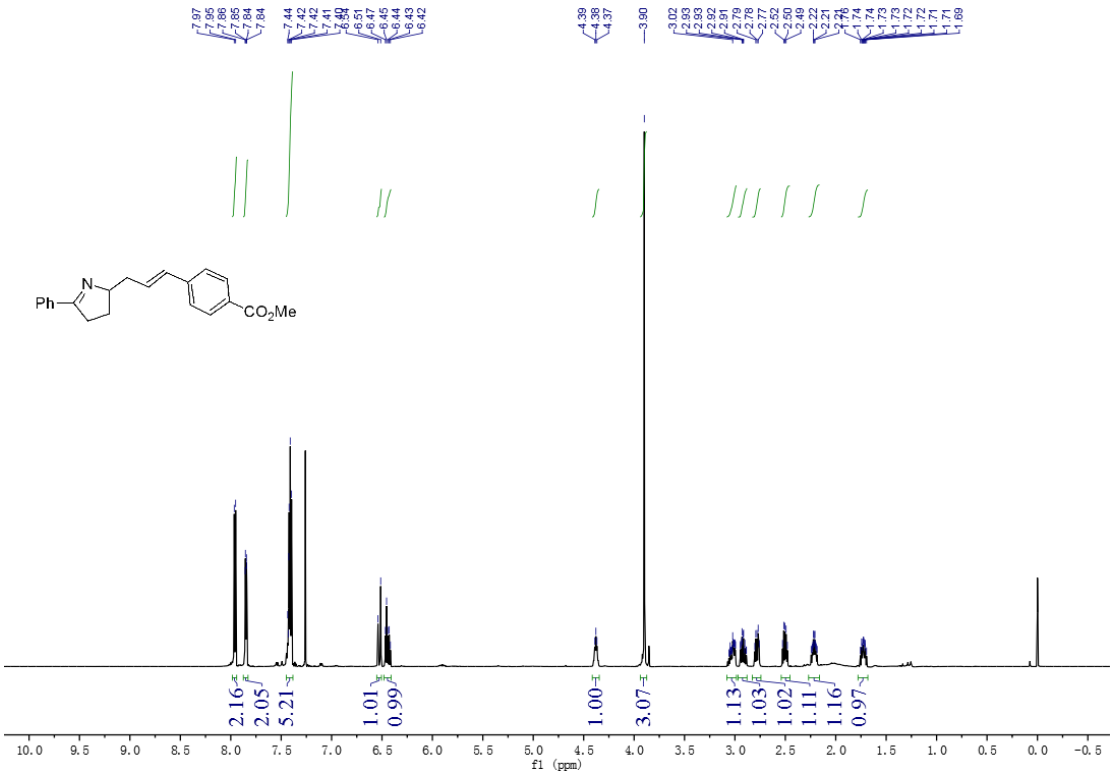
^{1}H NMR of **5m**



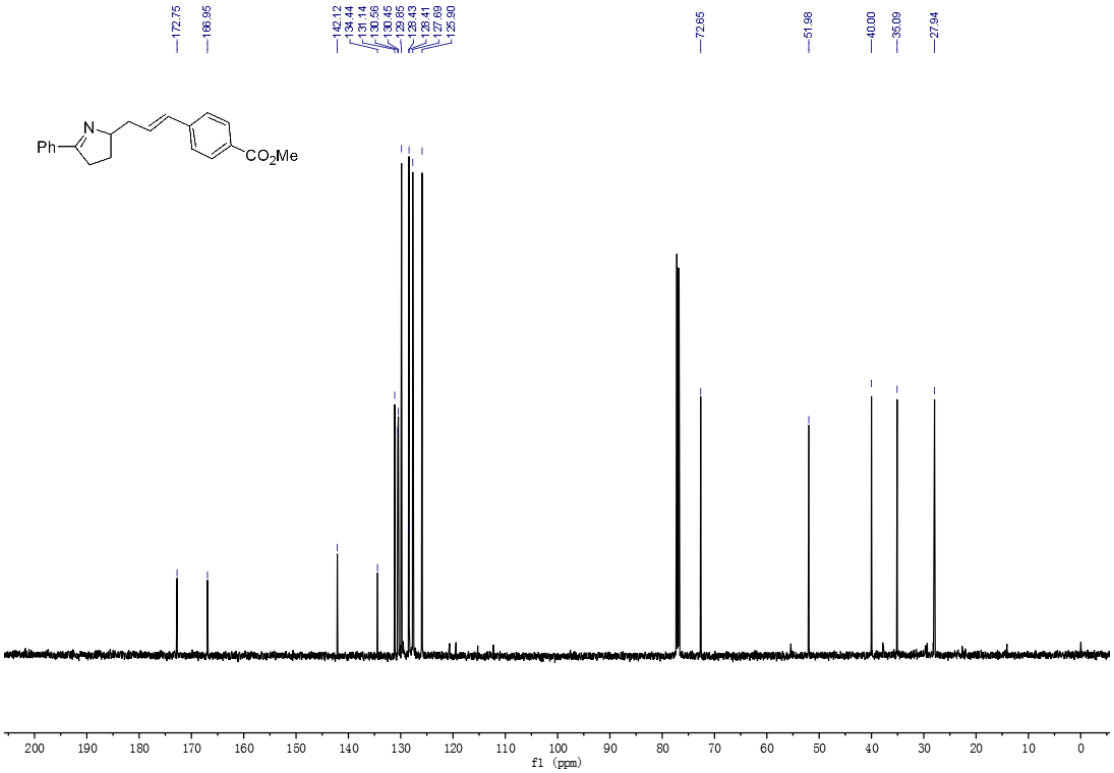
^{13}C NMR of **5m**



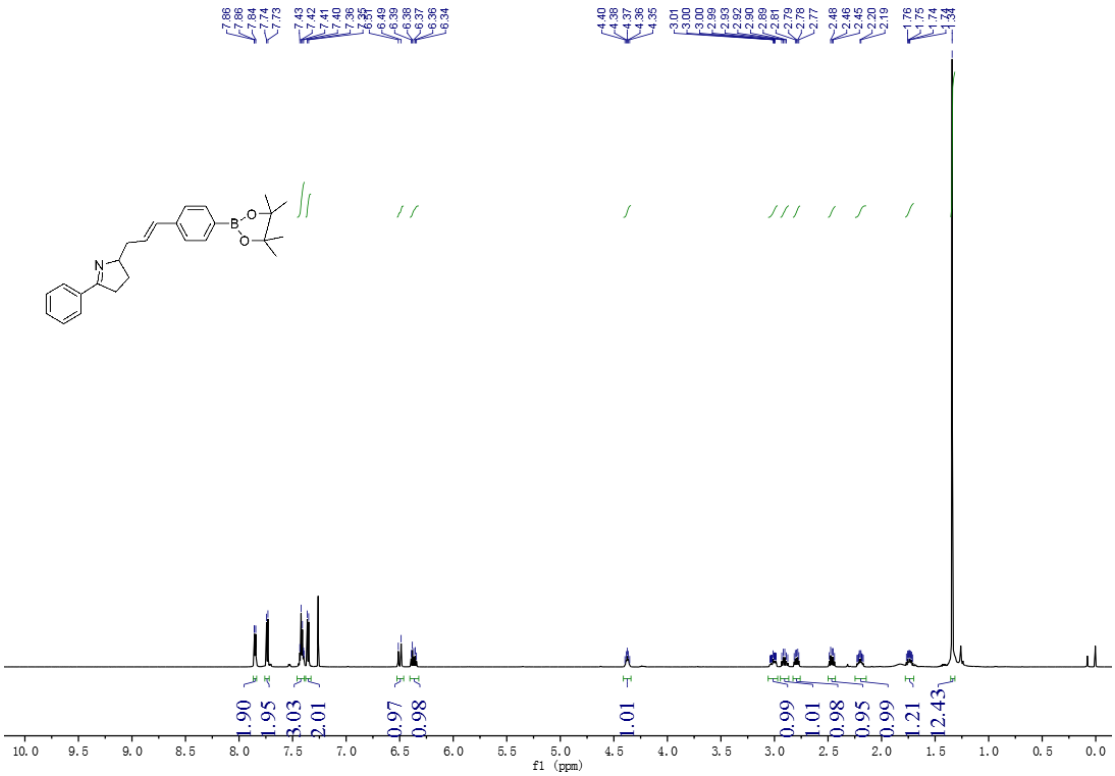
^{1}H NMR of **5n**



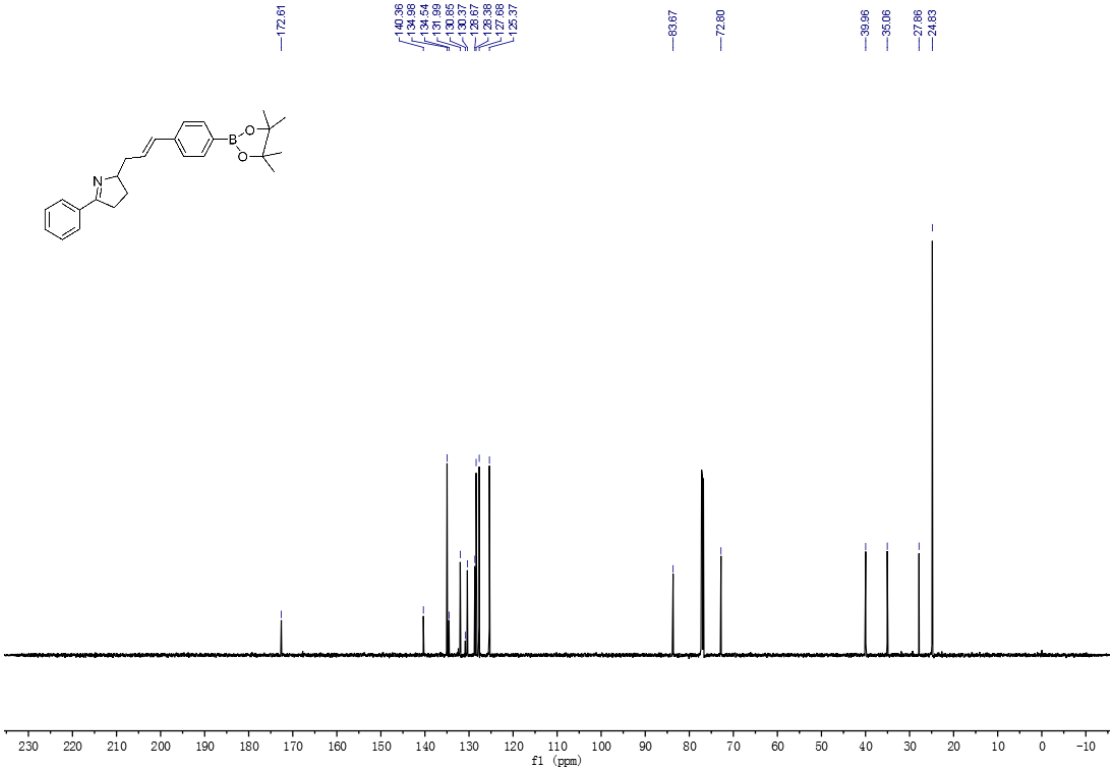
^{13}C NMR of **5n**



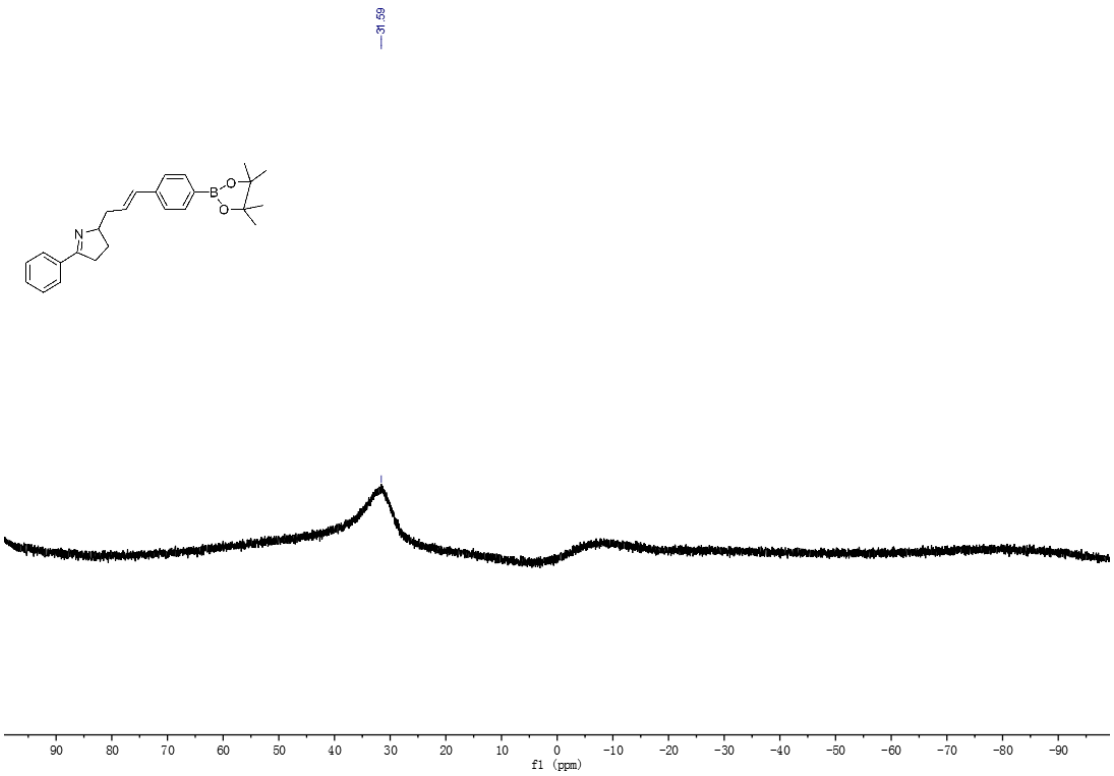
^{1}H NMR of **5o**



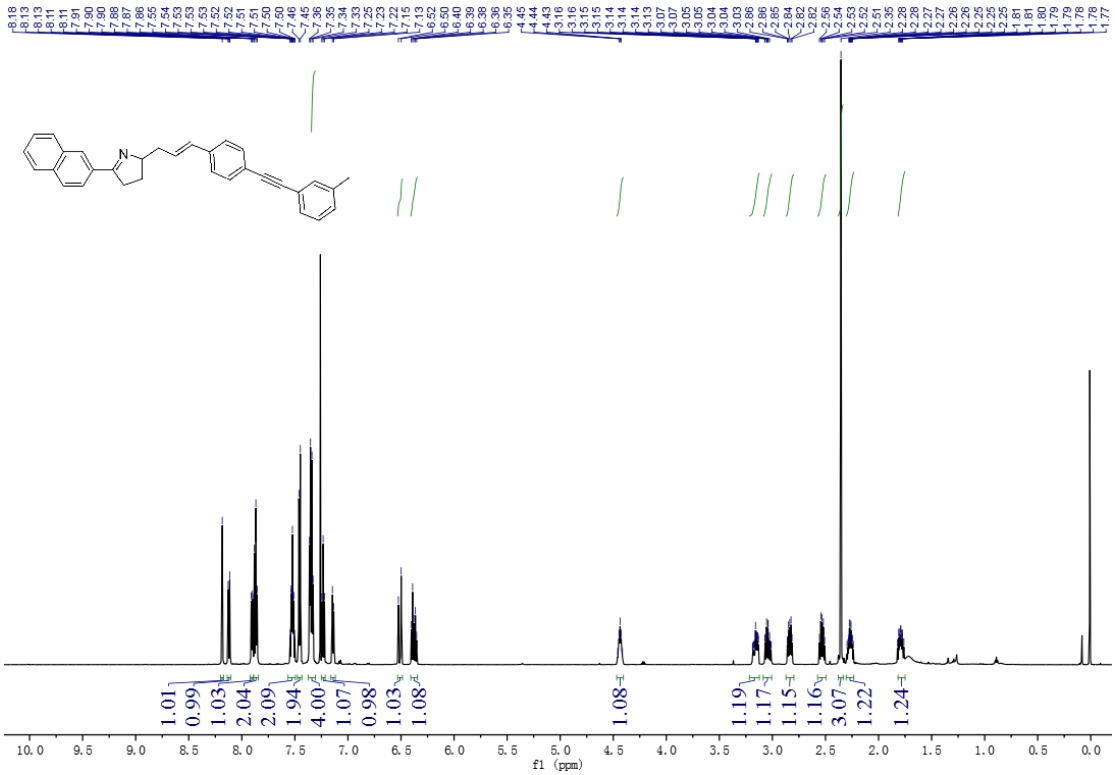
^{13}C NMR of **5o**



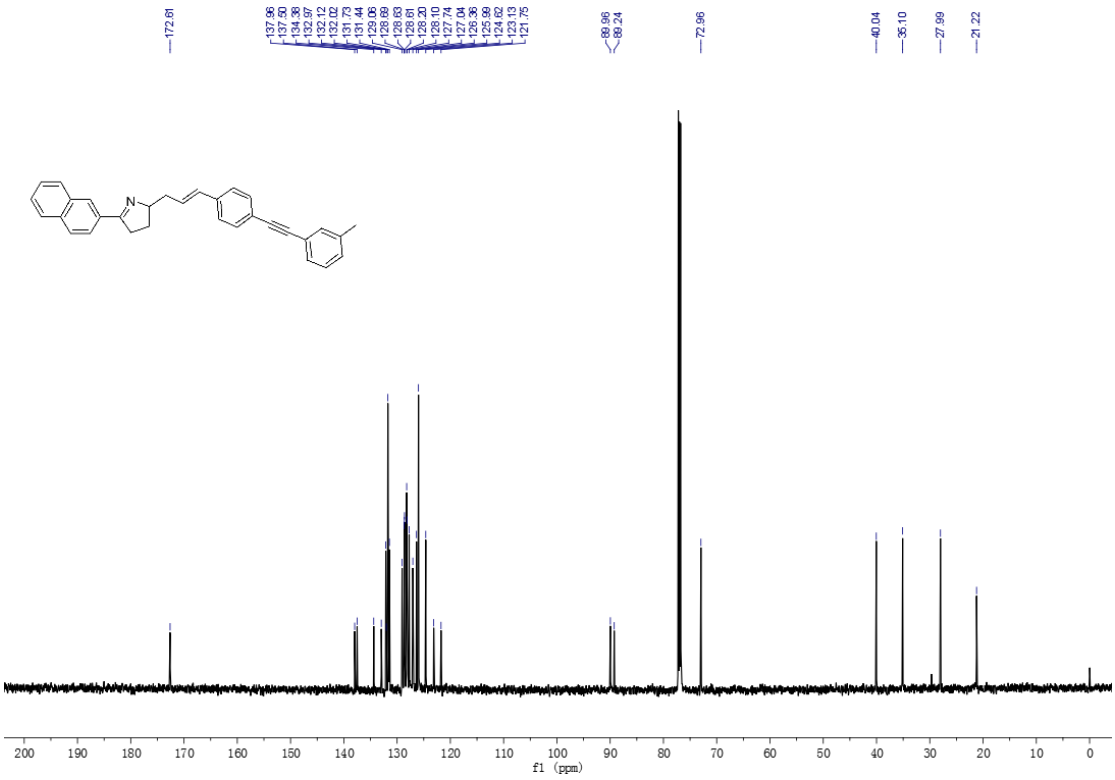
^{11}B NMR of **5o**



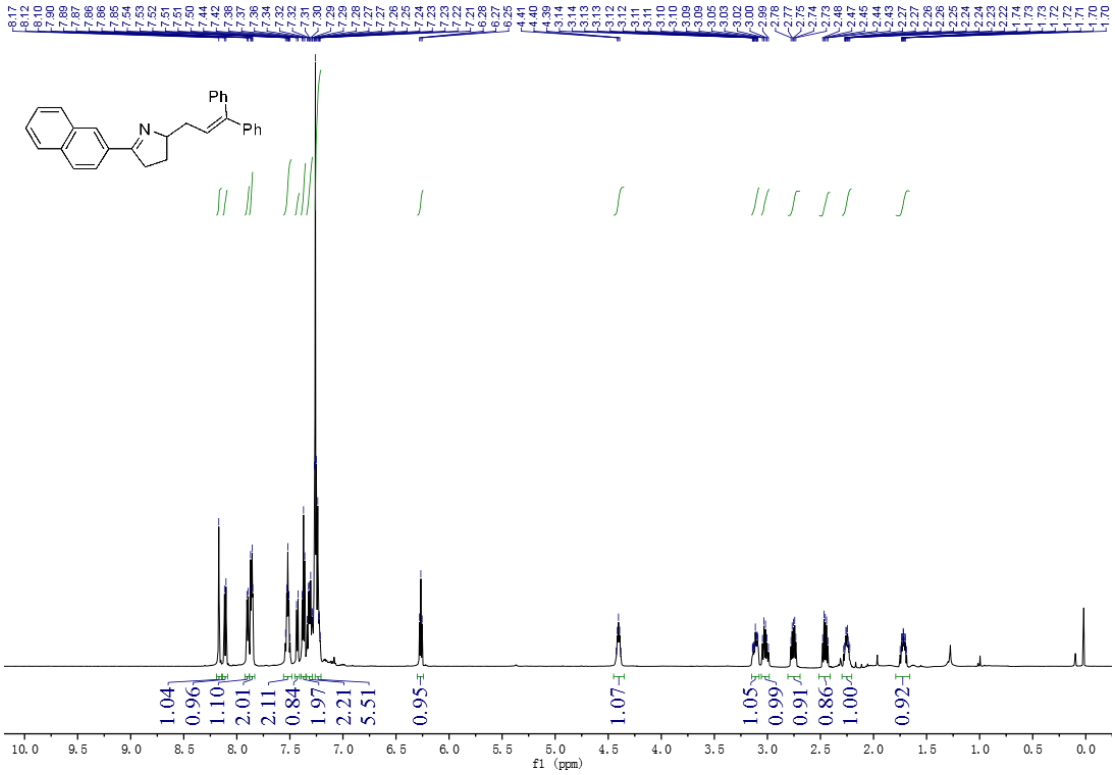
^{1}H NMR of **5p**



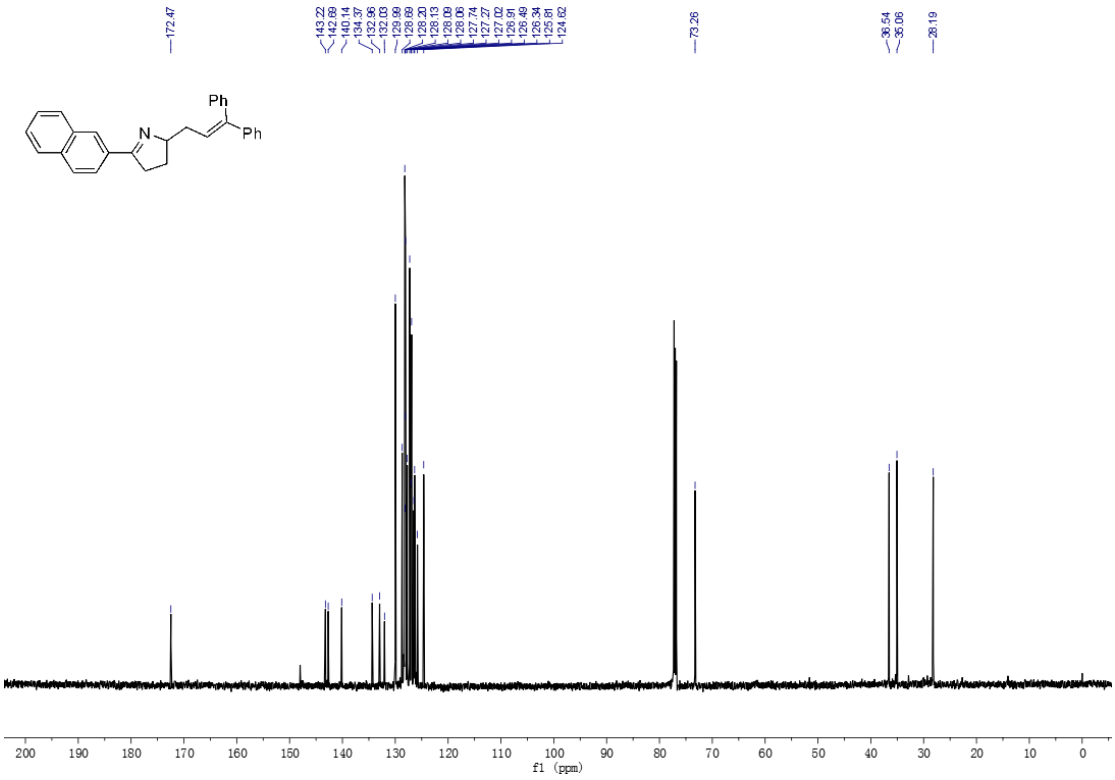
^{13}C NMR of **5p**



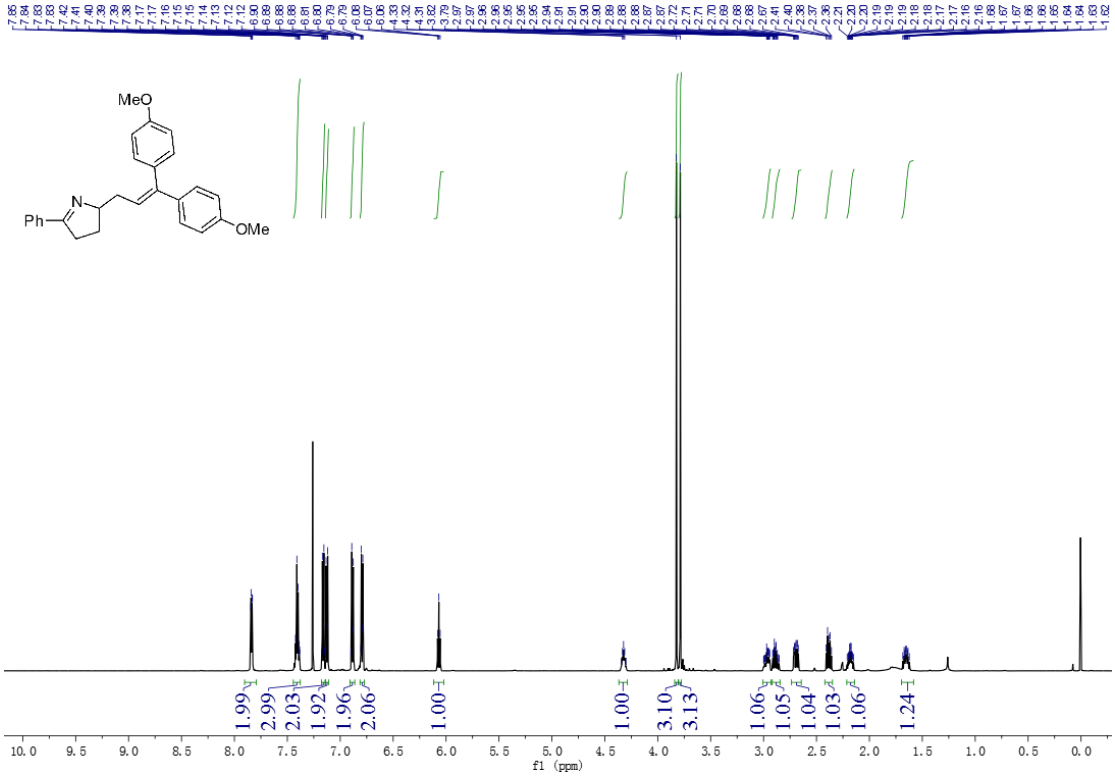
^{1}H NMR of **5q**



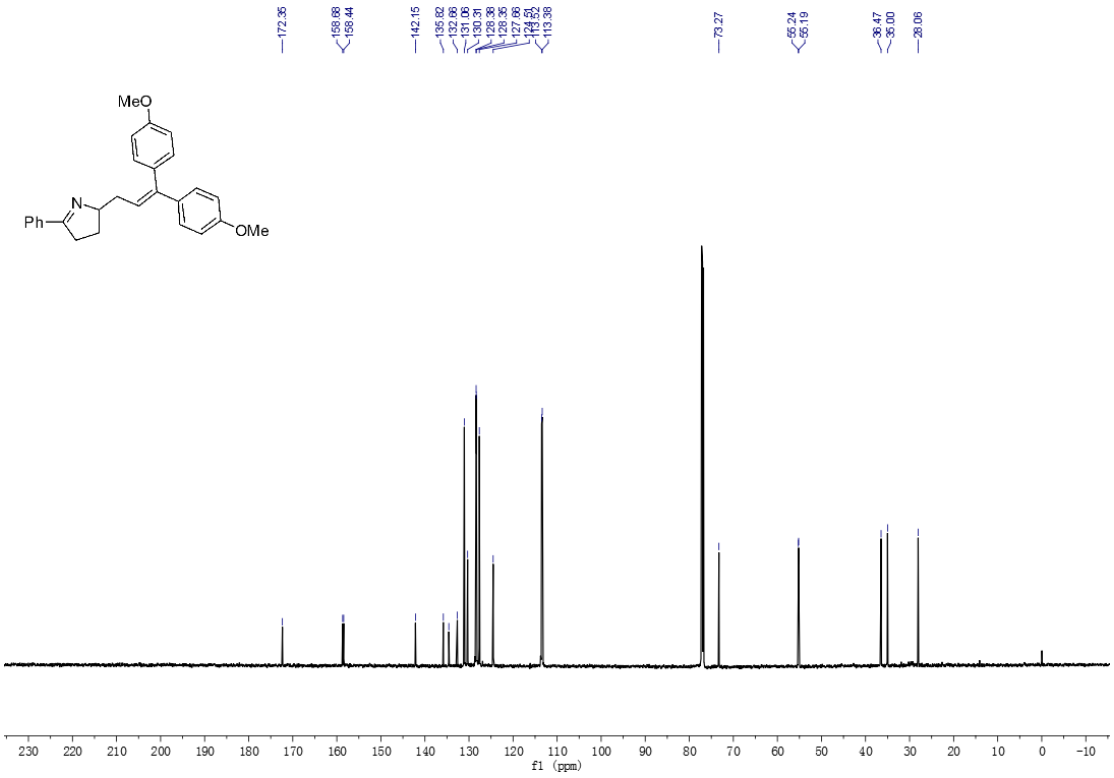
^{13}C NMR of **5q**



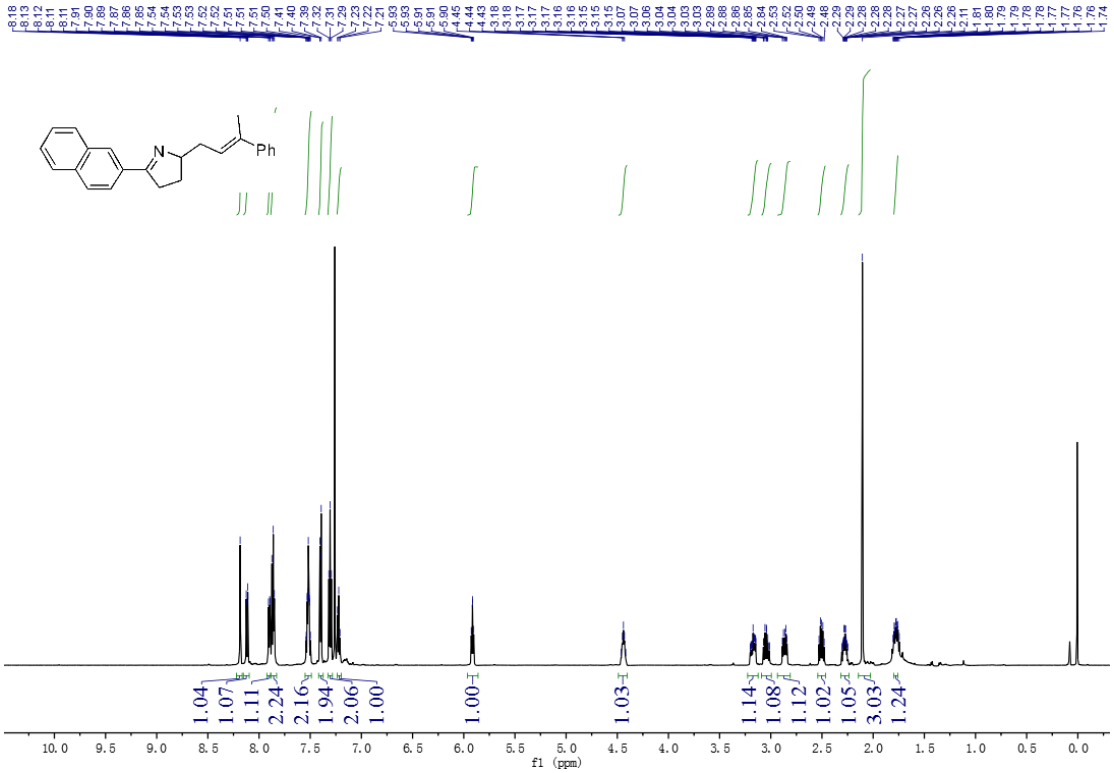
^{1}H NMR of **5r**



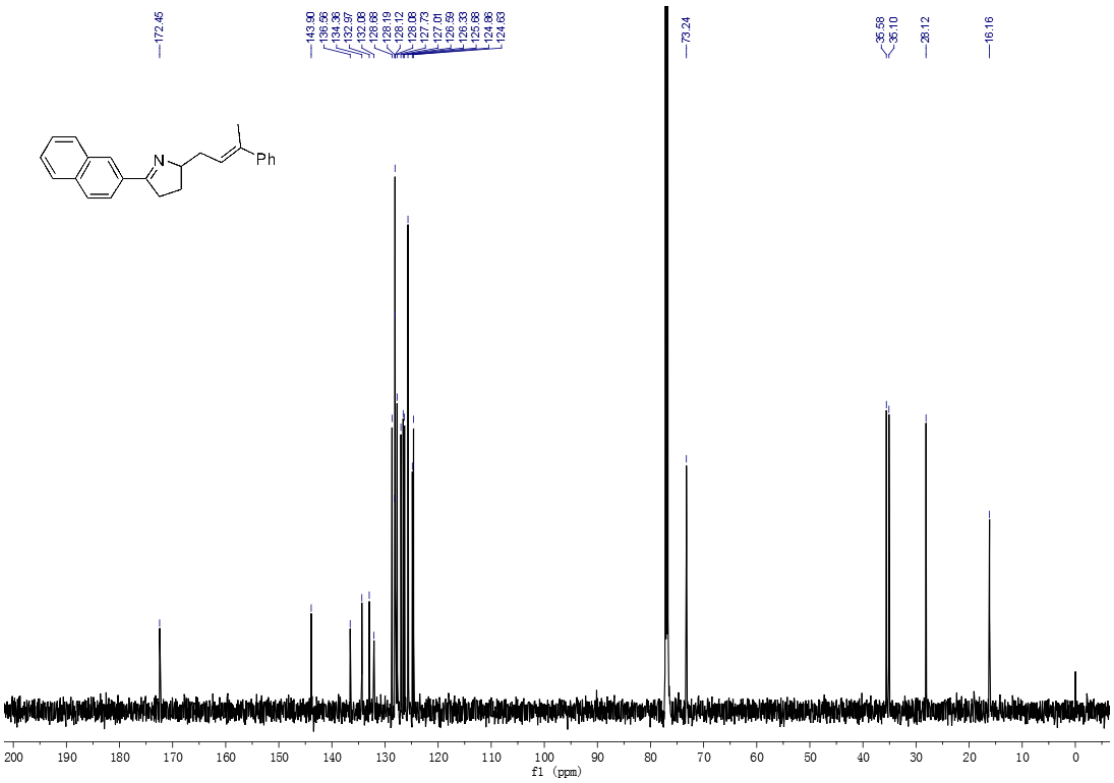
^{13}C NMR of **5r**



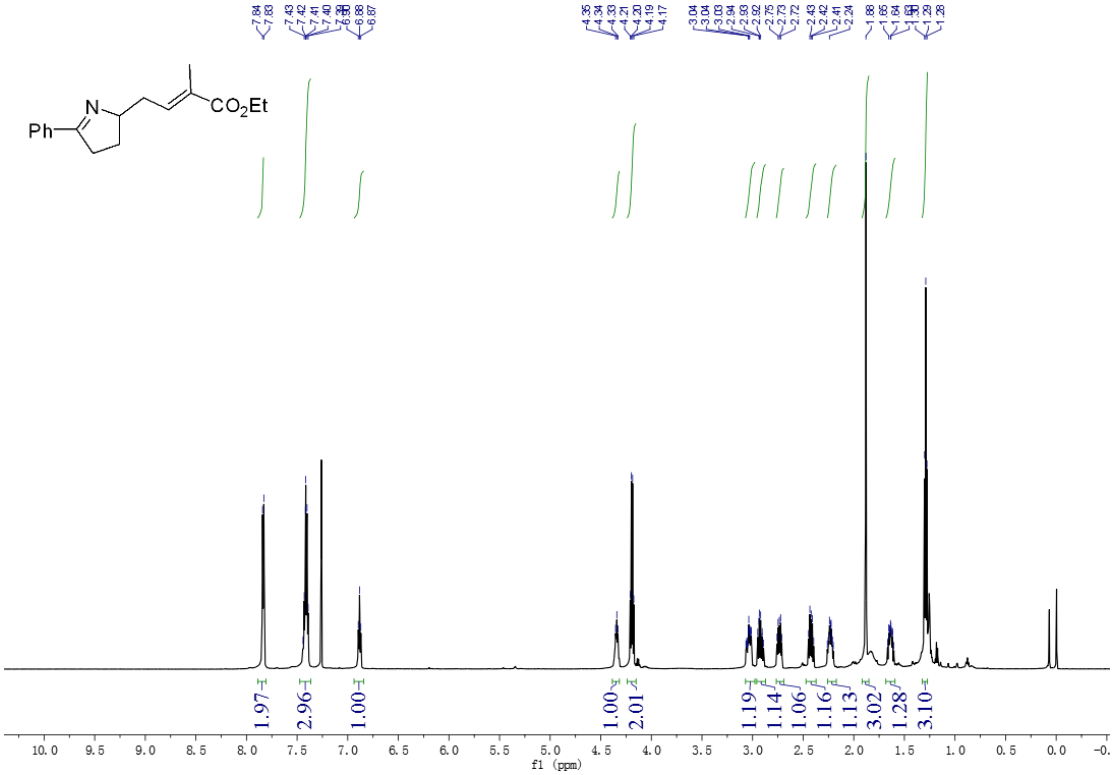
^{1}H NMR of **5s**



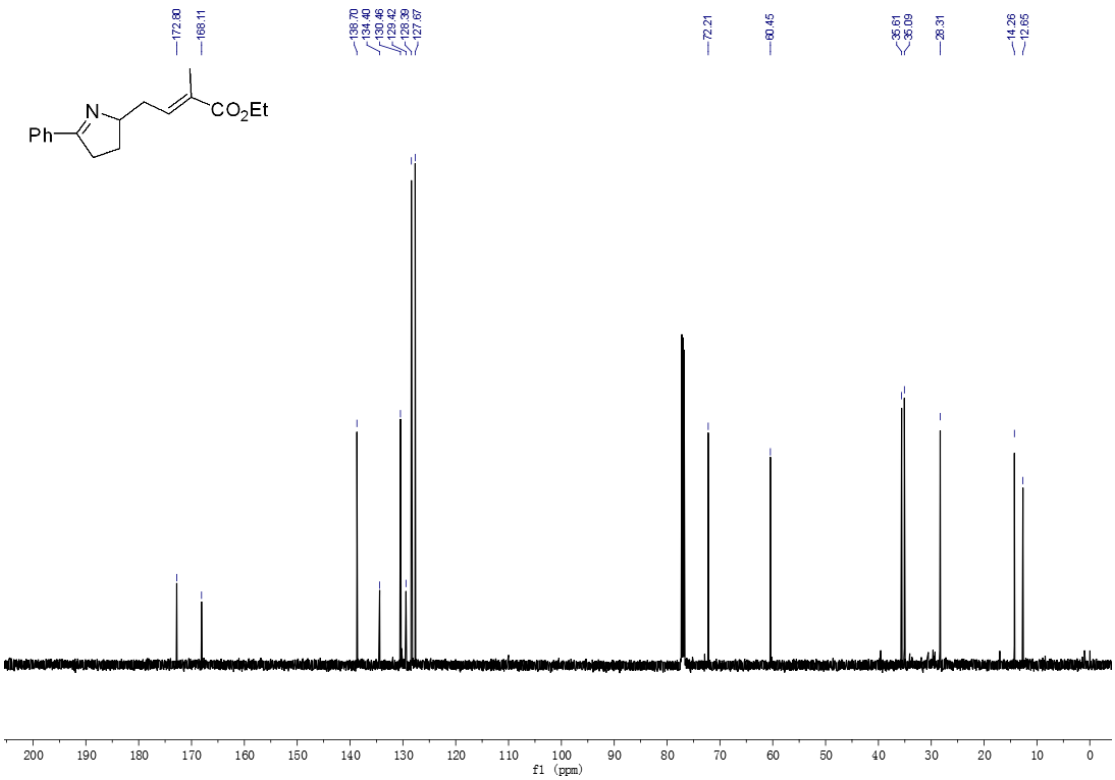
^{13}C NMR of **5s**



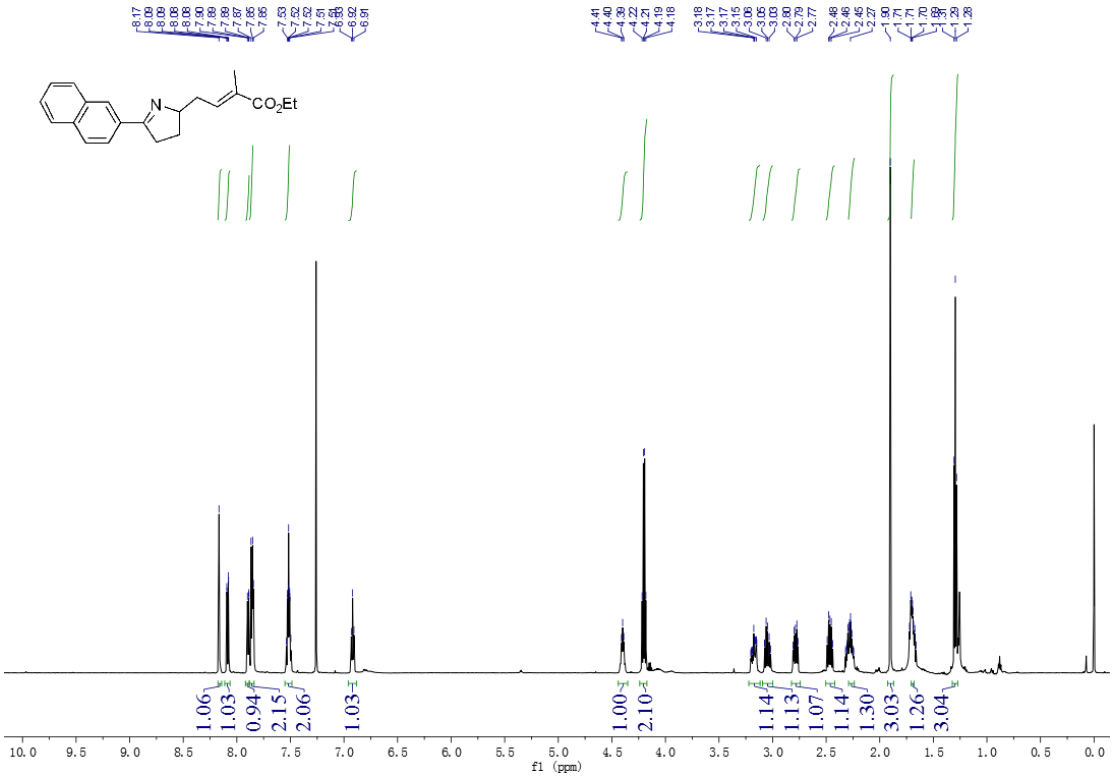
^{1}H NMR of **5t**



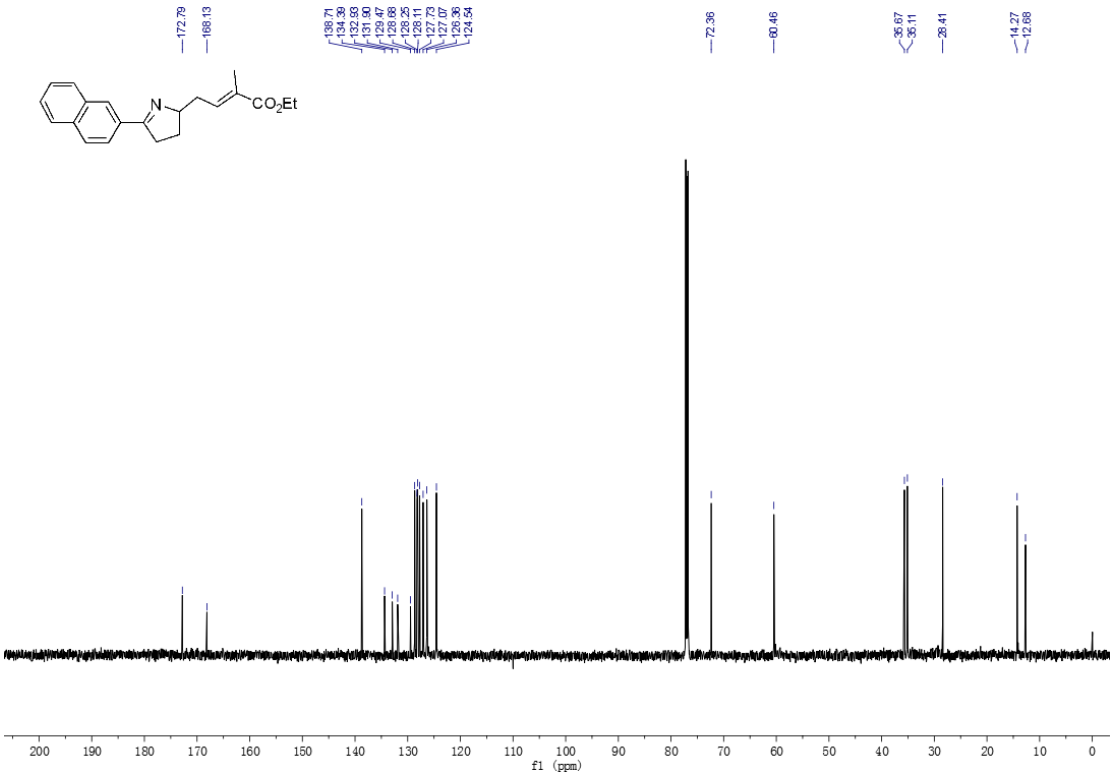
^{13}C NMR of **5t**



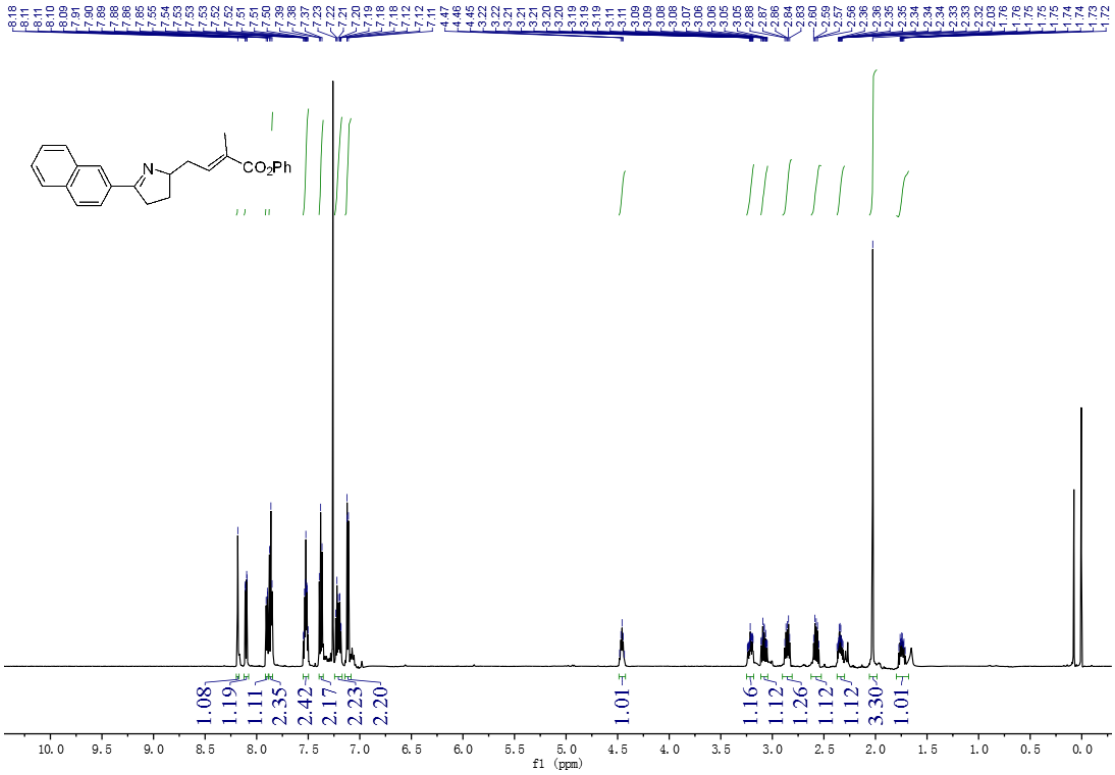
^{1}H NMR of **5u**



^{13}C NMR of **5u**



^{1}H NMR of **5v**



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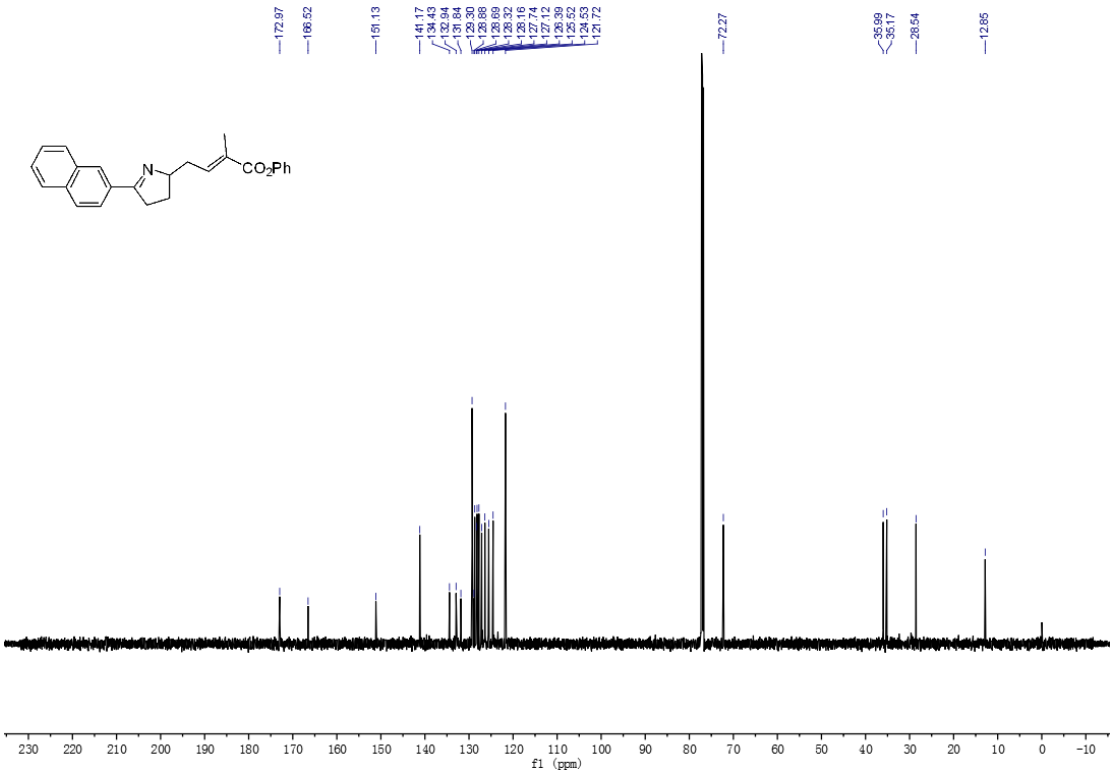
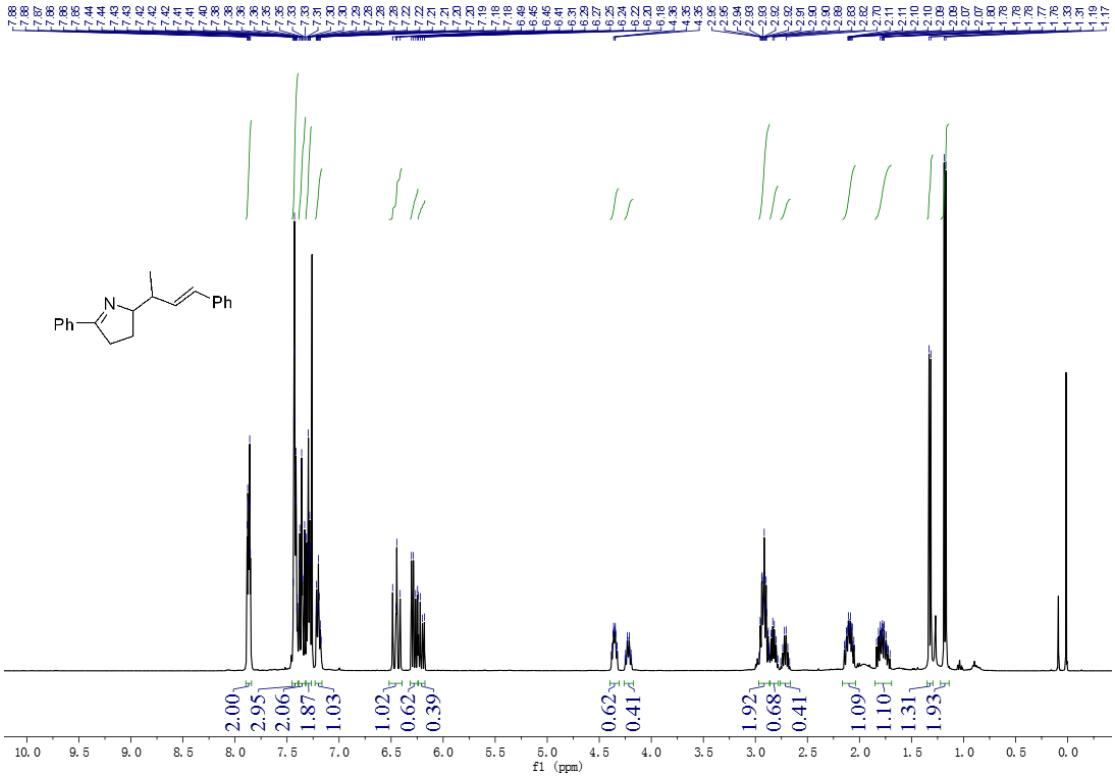
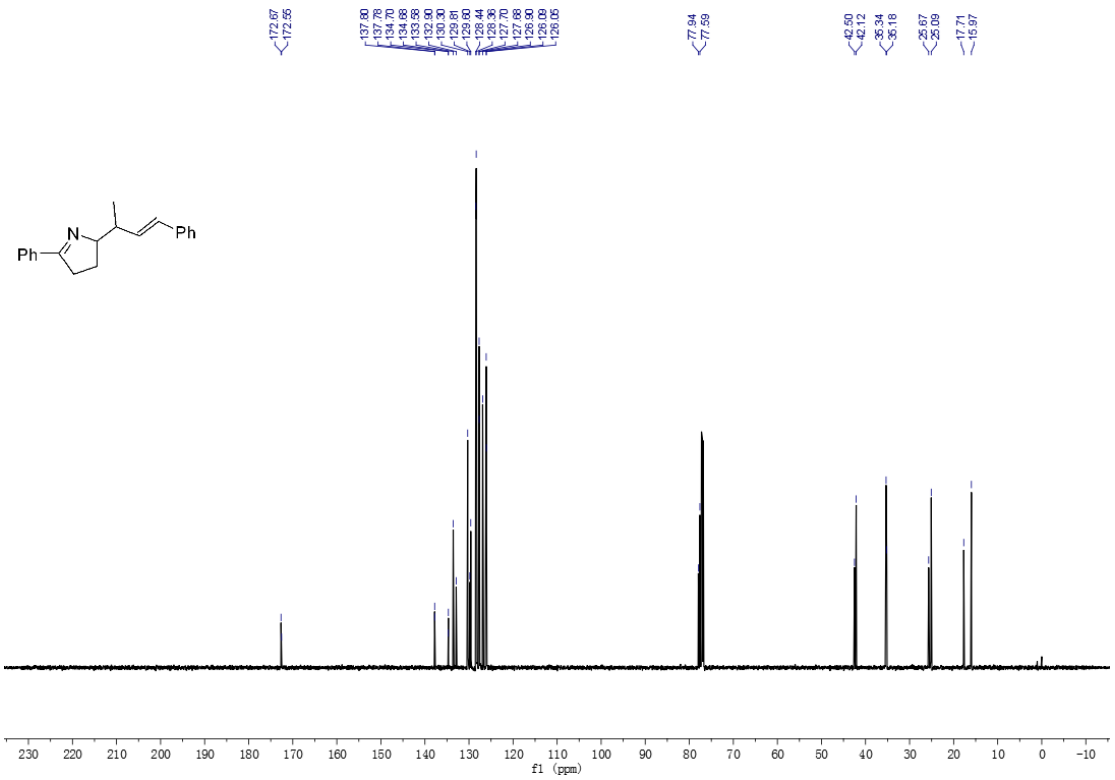


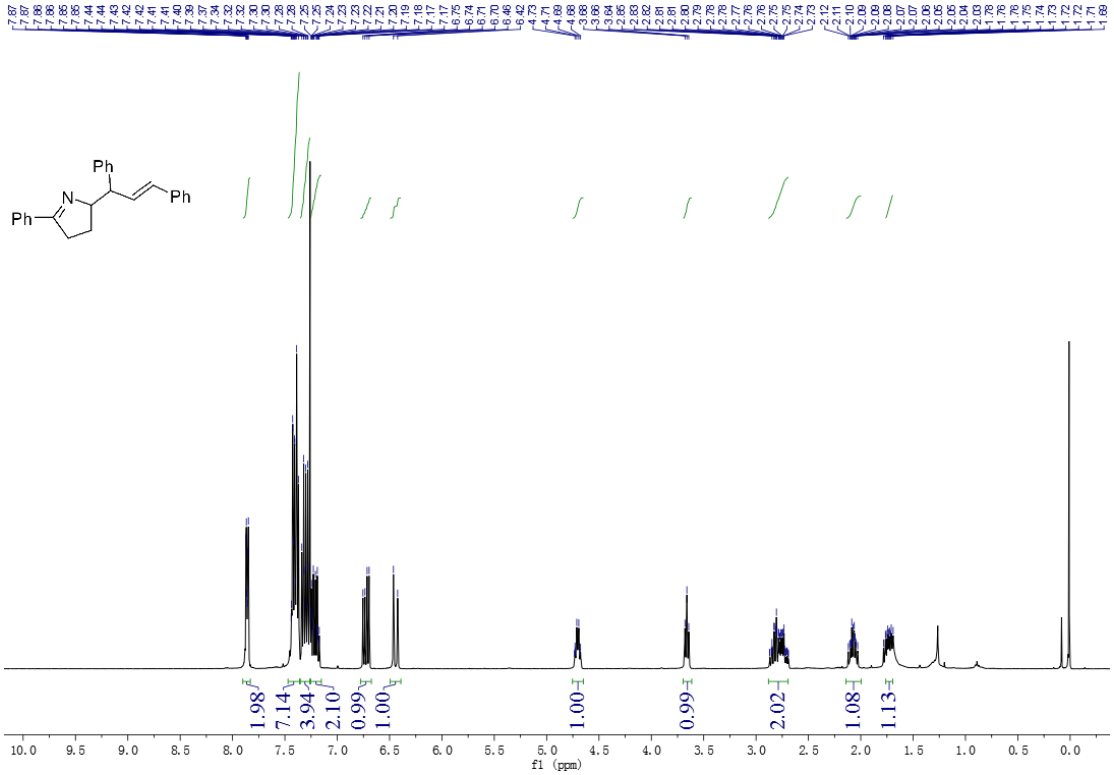
^{1}H NMR of **5w**



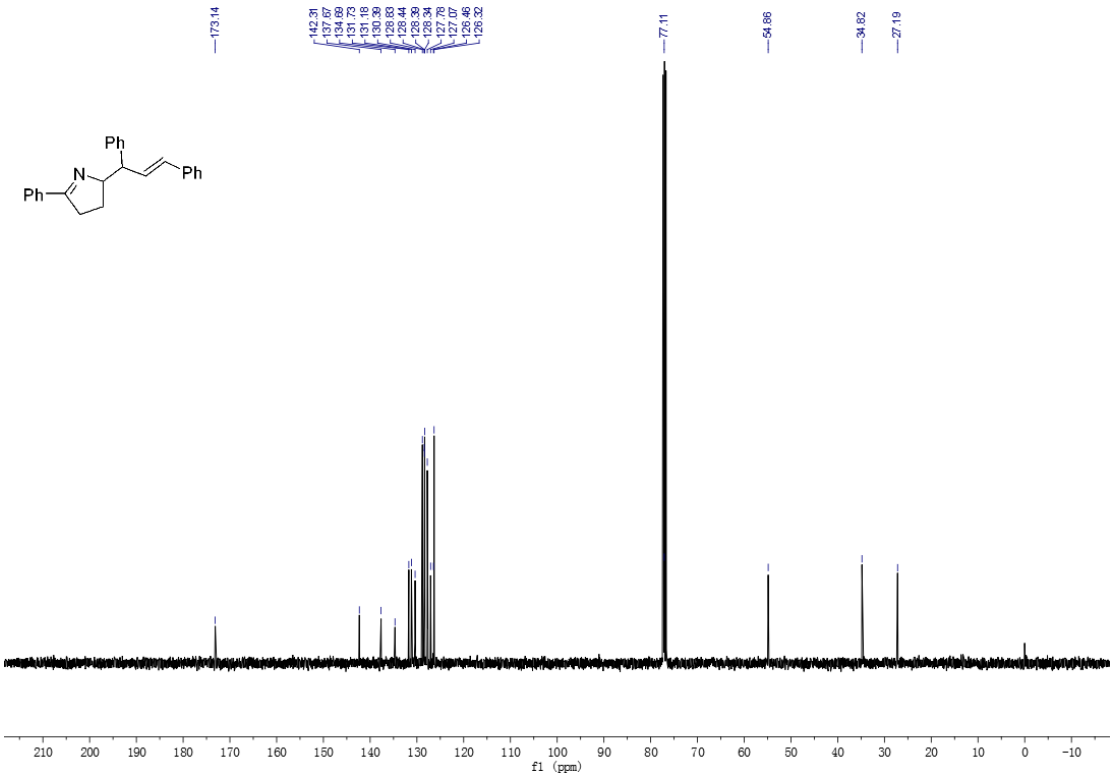
^{13}C NMR of **5w**



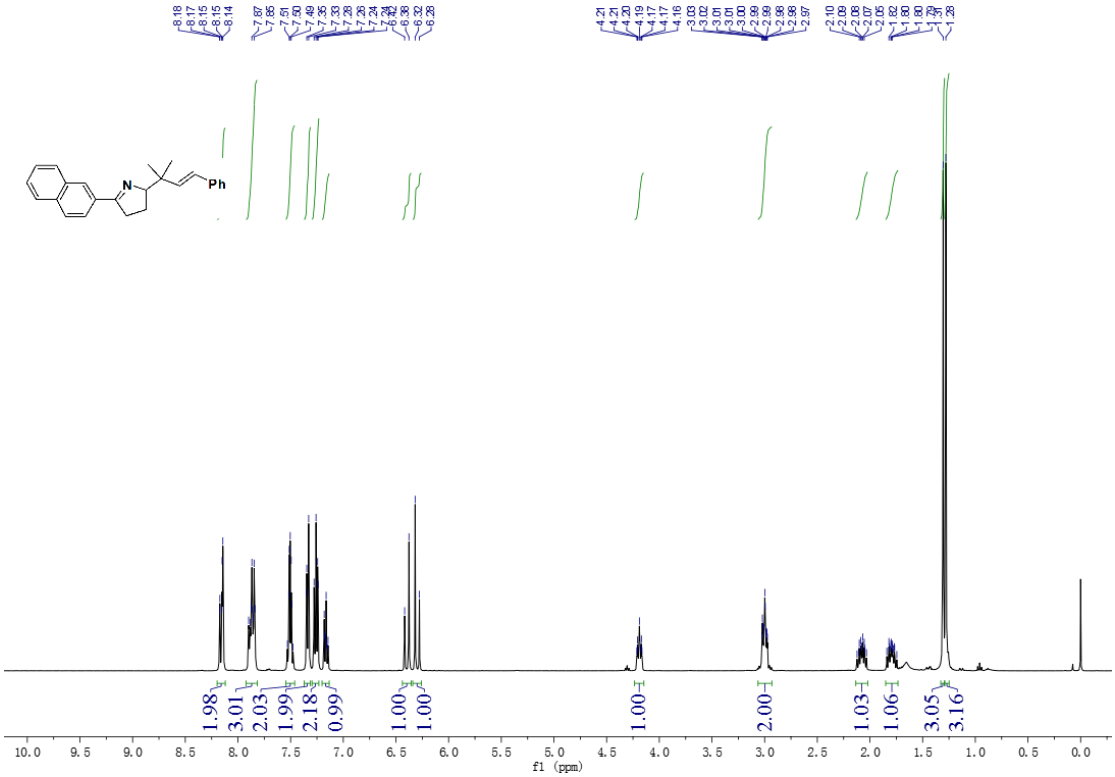
^{1}H NMR of **5x**



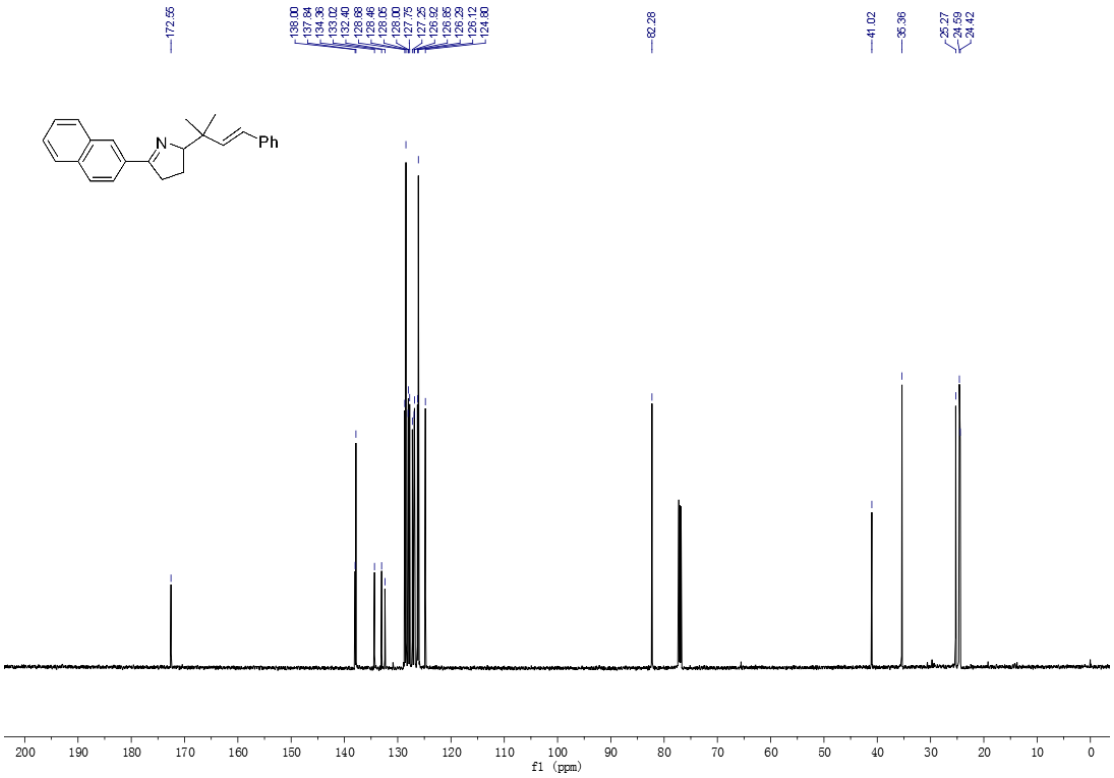
^{13}C NMR of **5x**



^{1}H NMR of **5y**



^{13}C NMR of **5y**



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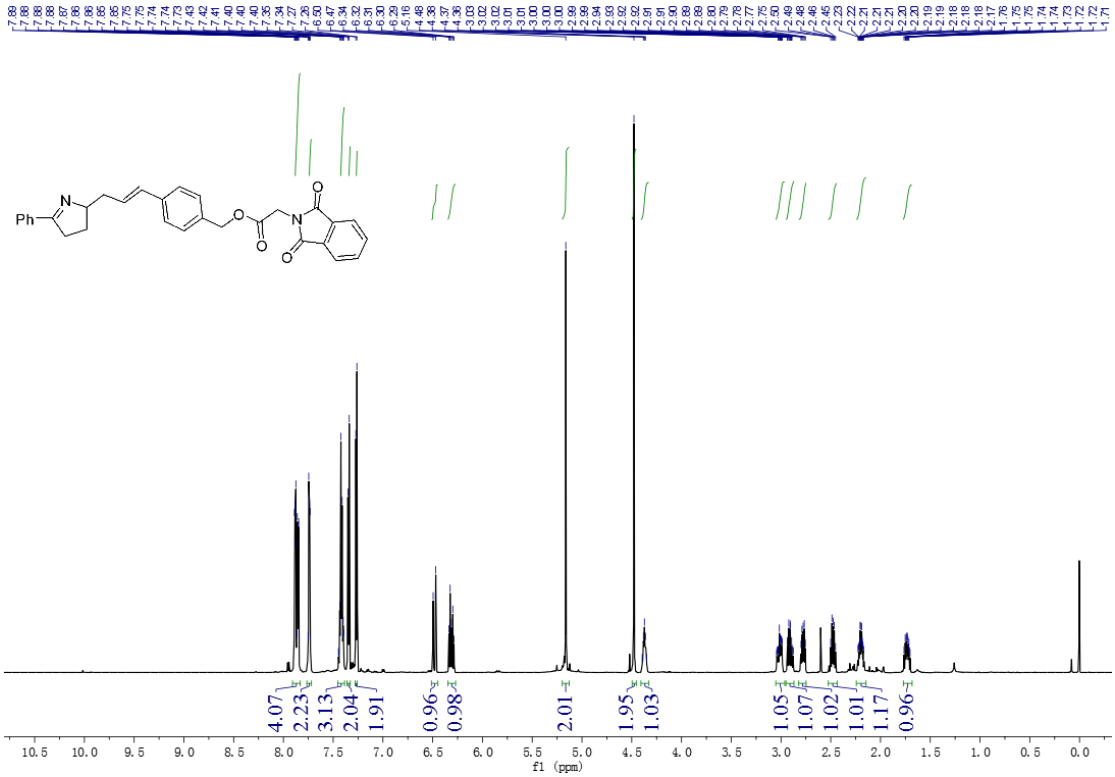

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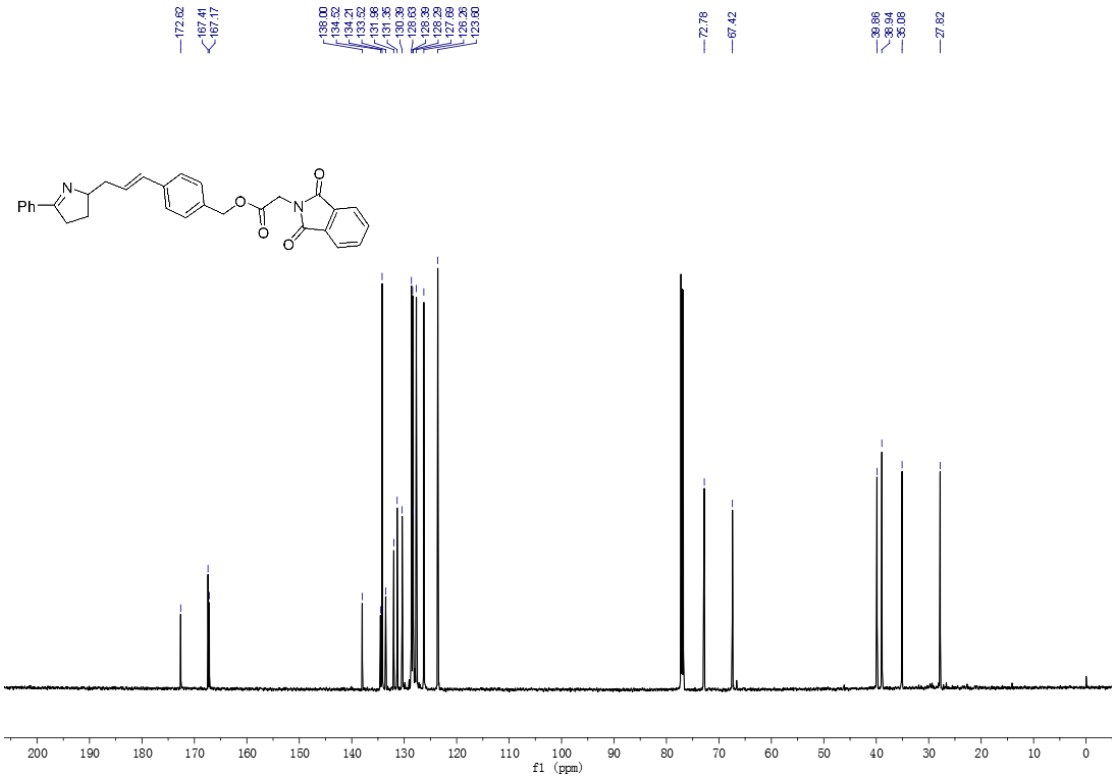
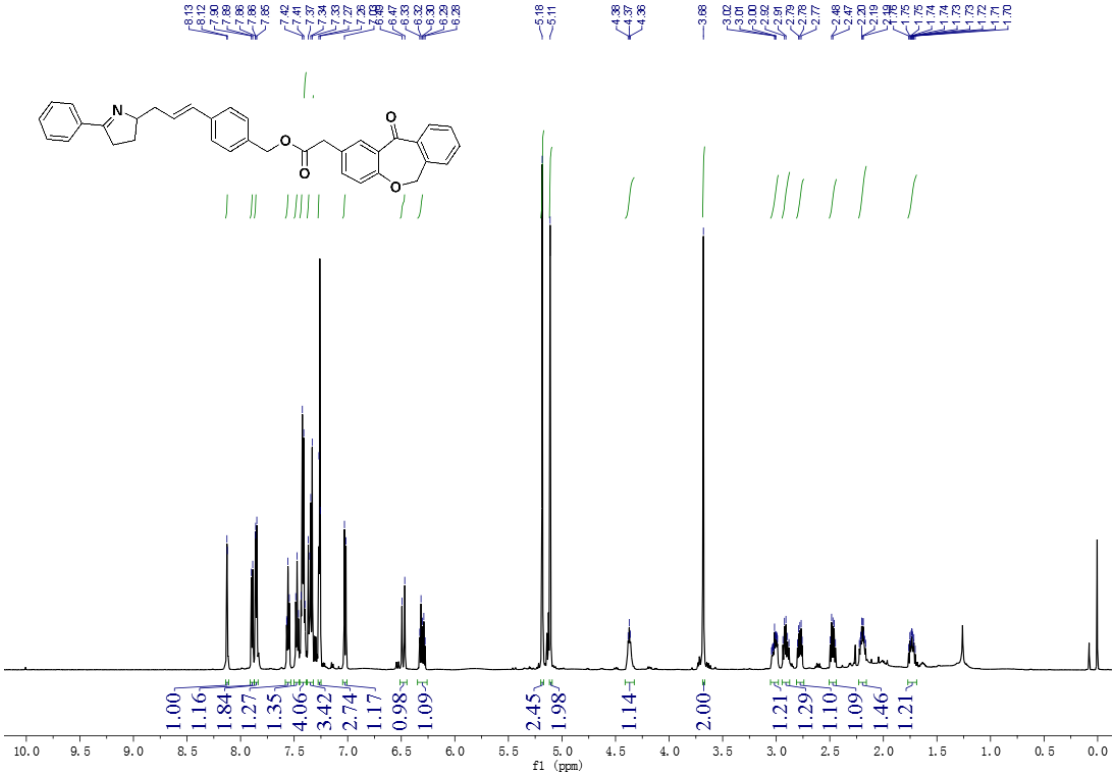


^{1}H NMR of **5ba**



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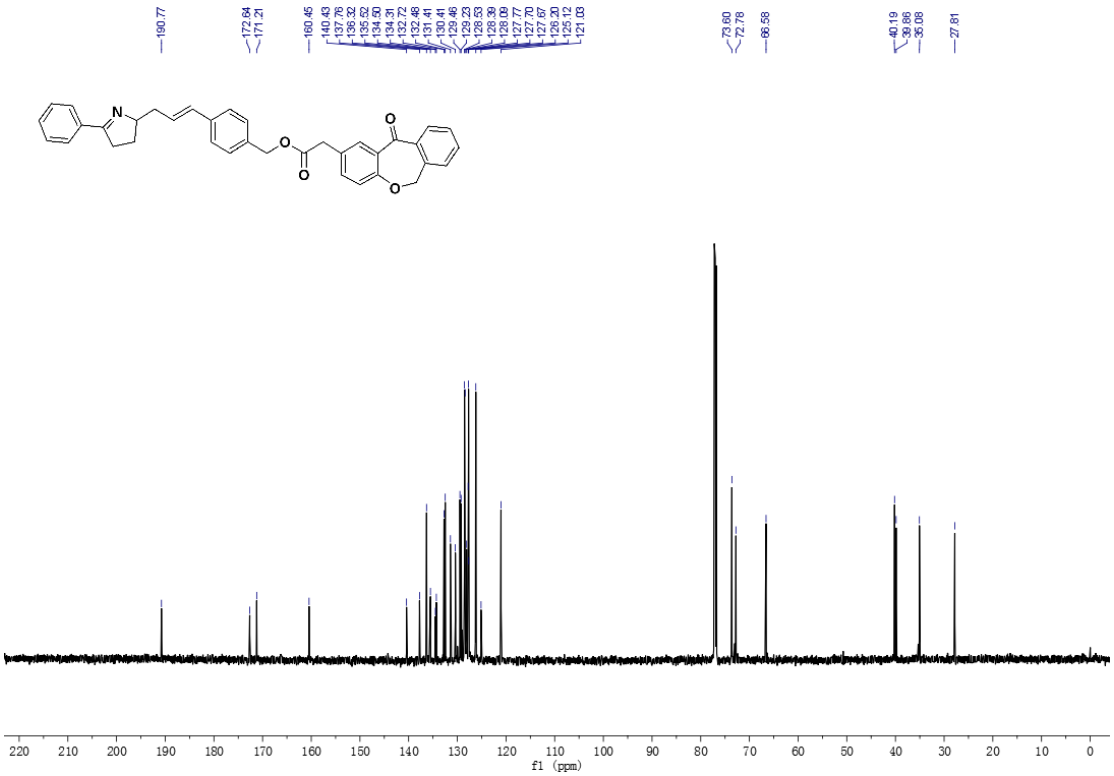
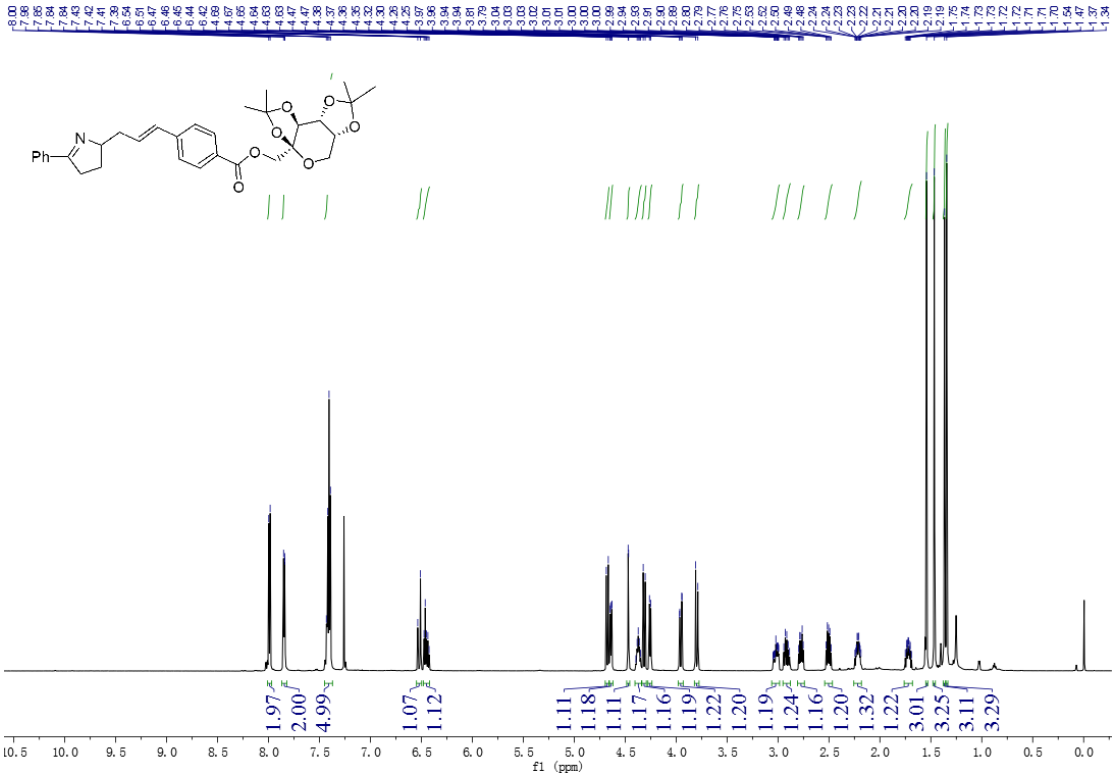
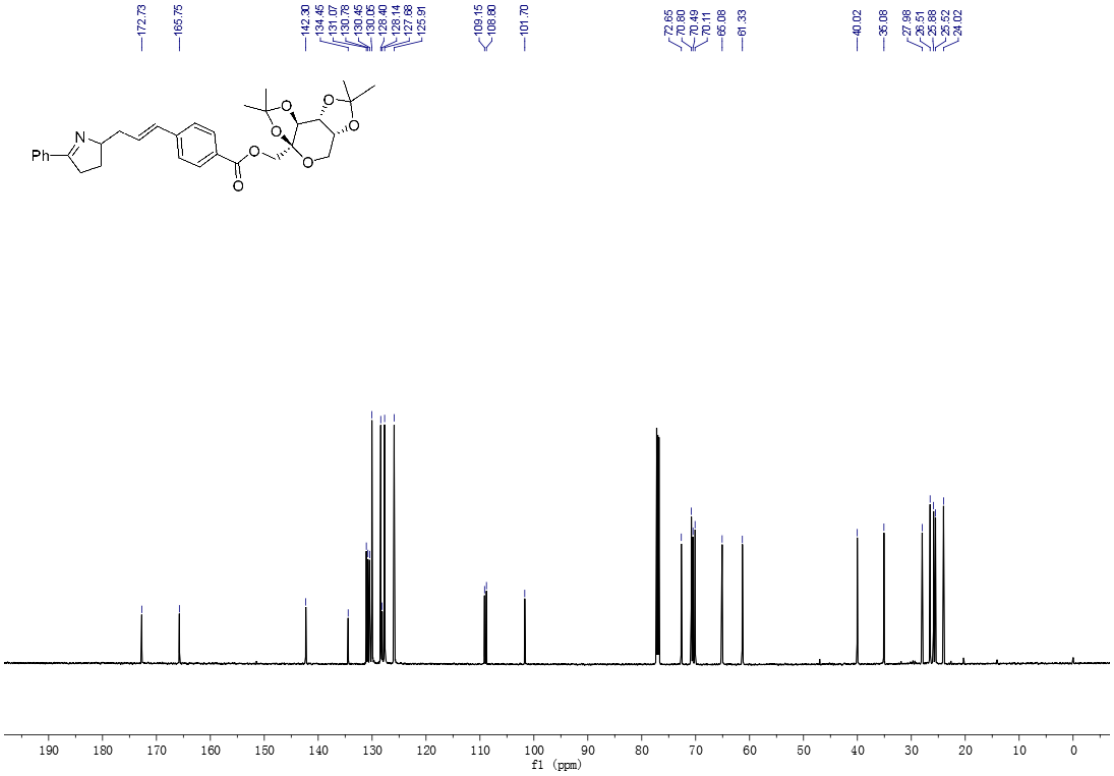


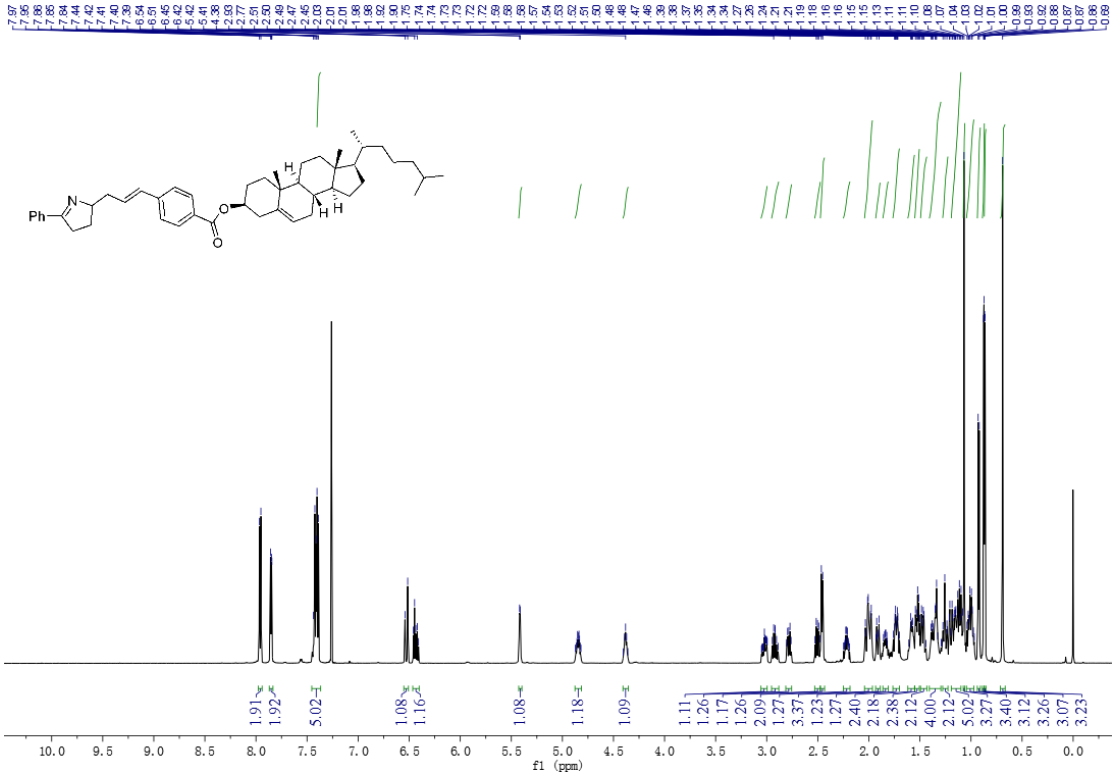
^{1}H NMR of **5bb**



^{13}C NMR of **5bb**



^{1}H NMR of **5bc**



^{13}C NMR of **5bc**