Supporting Information

Unexpected Ester and Phosphonate Radical Generation by Hypervalent Iodine Compounds for Synthesizing 6-Phenanthridine Derivatives

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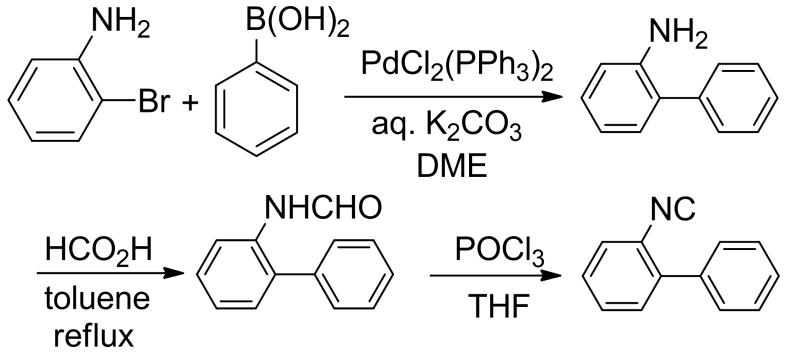
I. Materials and Methods.

All reagents were used as purchased and used with no further purification. Ethyl diazoacetate, (≥13 wt. % dichloromethane) was purchased from Aldrich and used without further purification. Anhydrous solvents were dried by passing through an activated alumina column on a PureSolvTM solvent purification system. Analytical thin layer chromatography (TLC) was carried out using aluminum sheets with 0.2 mm of silica gel (Merck GF234). Visualization of the developed chromatogram was performed by irradiation with UV light. Flash column chromatography was performed on silica gel. Organic solutions were concentrated under reduced pressure on an IKA rotatory evaporator. Unless otherwise stated, reactions were carried out under nitrogen atmosphere. Yields refer to purified compounds unless otherwise noted. NMR spectra were recorded at 298 K on Bruker Avance 400. Chemical shifts (δ) are quoted in ppm relative to residual solvent signals, $CDCl_3$ referenced at δ 7.26 and 77.16 ppm, DMSO-*d*6 referenced at δ 2.50 and 39.52 ppm. Coupling constants (*J*) are quoted in hertz (Hz). Multiplicity is reported with the following abbreviations: s = singlet, brs = broad singlet, d = doublet, t = triplet, q = quartet, dt = doublet of triplets, td = triplet of doublets, tt = triplet of triplets, sp = septet, m = multiplet, app = apparent. Mass spectra were recorded on a Waters LCT Premier spectrometer. Liquid chromatography-mass spectrometry (LC-MS).

II. Experimental Section

1. Synthesis of isonitrile 1:

A typical procedure (synthesis of isonitrile **1**) is shown below:[1]



2-Bromo-aniline (1.72 g, 10 mmol, 1.0 equiv), boronic acid (1.34 g, 11.0mmol, 1.1 equiv) and an aqueous solution of K_2CO_3 (2 M, 25 mL) were placed in a three necked flask under N_2. Then, DME (25 mL) was added and the mixture was stirred for 10-30 min. To the stirred mixture, $PdCl_2(PPh_3)_2$ (71.1 mg, 0.1mmol, 0.01 equiv) was added and the mixture was stirred at 80 ℃ for 6h. The mixture was then cooled to room temperature and diluted with EtOAc. The organic layer was washed with water and dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel by using a 20:1 mixture of pentane/EtOAc as an eluent to provide 2-phenylaniline as white solid (1.44 g, 84%).

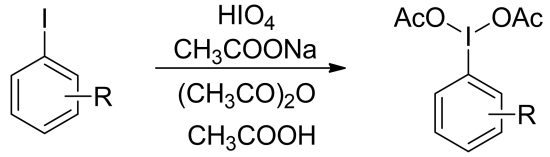
Asolution of 2-phenylaniline (1.70 g, 10mmol) and formic acid (0.9 mL) in toluene (15mL) is refluxed under N_2 atmosphere. The reaction was monitored by TLC. After the reaction, volatile materials were evaporated under reduced pressure. Crude product was purified by flash column chromatography on silica gel to afford pure 2-phenylformanilide (1.88 g, 95%).

A THF solution (20 mL) of 2-phenylformanilide (1.88 g, 9.5 mmol) and NEt_3 (7 mL, 50 mmol) was cooled to 0 ℃. Then, $POCl_3$ (1.2 mL, 11 mmol) was added dropwise and the mixture was stirred at 0 ℃ for 1 h. After the reaction was completed, the mixture was quenched by aqueous saturated Na_2CO_3 solution and stirred for 0.5 h. The mixture was extracted with EtOAc for three times. The combined organic layer

was dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure and the crude product was purified by flash column chromatography on silica gel (pentane/EtOAc = 50:1) to provide analytical pure isocyanide product as pale oil (1.50 g, 88%).

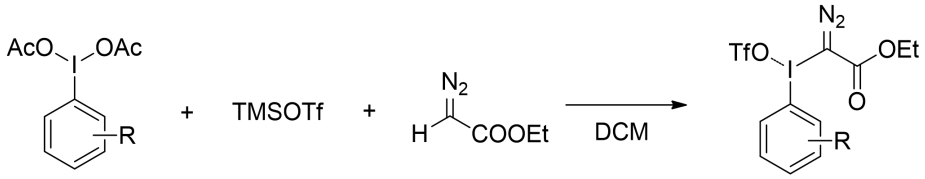
2. Preparation of hyperiodine reagents 2 and their derivatives

Diacetoxyiodoarenes were prepared from the corresponding iodoarene according to previously reported synthetic procedures. [2]



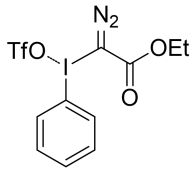
A stirred suspension of $NaIO_4$ (4.40 g, 20.5 mmol) and AcONa (3.60 g, 44.0 mmol) in glacial AcOH (40 mL) was added Ac_2O (3.0 mL). The mixture was treated with iodoarene (20.0 mmol) and refluxed for 2-8h. The reaction mixture was then poured into water (50 mL). The resulting mixture was extracted three times with DCM (50 mL). The combined extraction was washed with water and concentrated on rotary evaporator to provide corresponding diacetoxyiodoarenes. Hexane was added to the obtained products if impurities are observed. The solids were filtered and washed with excess of hexane to provide pure products.

The preparation of hypervalent iodine reagents was performed according to a literature reference. [3]

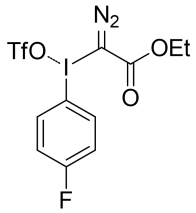


A solution of the corresponding aryliodoso diacetate (5.0 mmol, 1.0 equiv) dichloromethane (0.25 M) was treated with trimethylsilyl trifluoromethanesulfonate (0.90 mL, 5.0 mmol, 1.0 equiv) at room temperature. After this, ethyl diazoacetate (1.26 mL, 12.0 mmol, 2.4 equiv) was added dropwise during 10 minutes. Nitrogen

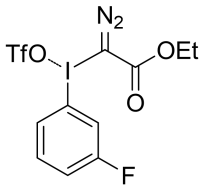
evolution was observed and the resulting yellow reaction mixture was stirred for 1 hour at room temperature. Solvent was removed under vacuum and the crude was recrystallized from a mixture of diethyl ether/dichloromethane (5/1) during 12 hours at -30 °C. The product was collected by filtration, washed with cold diethyl ether (200 mL), dried under high vacuum and stored at -20 °C.



2a: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.07-8.09 (d, *J* = 7.6 Hz, 2H), 7.60-7.66 (m, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 4.31 (q, *J* = 7.2 Hz, 2H), 1.29 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 161.7, 137.5, 135.1, 132.8, 131.8, 130.3, 64.0,14.2. The values of the NMR spectra are in accordance with reported literature data.[3]

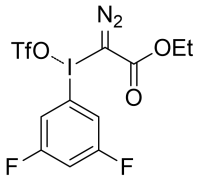


2b: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.10-8.17 (m, 2H), 7.15 (t, *J* = 8.4 Hz, 2H), 4.31 (q, *J* = 7.2 Hz, 2H), 1.29 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.5, 164.0, 138.3, 119.4(dd, *J* = 23 Hz, 3.4 Hz), 110.4, 64.0,14.2; HRMS (ESI) calculated for $C_{10}H_9FIN_2O_2^+$ $[M\text{-}OTf]^+$ m/z: 334.9693, found: 334.9687.

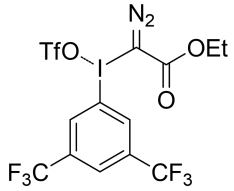


2c: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.88-7.95 (m, 2H), 7.44-7.51 (m, 1H), 7.29-7.36 (m, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 1.30 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.7, 161.1, 132.7, 131.2, 122.8, 121.5, 120.4, 119.4 (q, *J* =

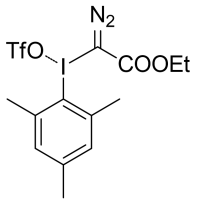
324 Hz), 64.2,14.2; HRMS (ESI) calculated for $C_{10}H_9FIN_2O_2^+$ [M-OTf]$^+$ m/z: 334.9687, found: 334.9681.



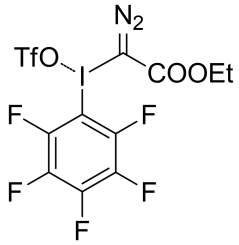
2d: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.72-7.76 (m, 2H), 7.05 (t, *J* = 8.4 Hz, 1H), 4.33 (q, *J* = 7.2 Hz, 2H), 1.31 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 164.1 (d, *J* = 5.7 Hz), 161.5 (d, *J* = 5.8 Hz), 119.6 (d, *J* = 158 Hz), 119.0 (d, *J* = 7.4 Hz), 109.1(d, *J* = 4.6 Hz), 64.2,14.2; HRMS (ESI) calculated for $C_{10}H_8F_2IN_2O_2^+$ [M-OTf]$^+$ m/z: 352.9593, found:352.9591.



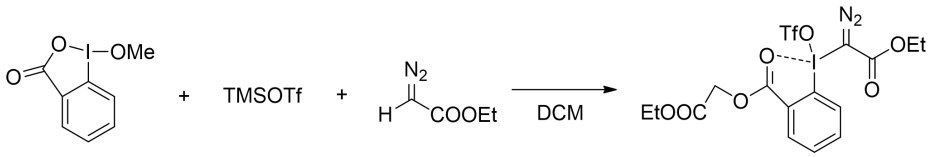
2e: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.60 (s, 2H), 8.00 (s, 1H), 4.24 (q, *J* = 7.2 Hz, 2H), 1.22 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 161.3, 155.8, 134.0 (q, *J* = 35 Hz), 126.4, 123.2, 120.5, 117.5, 64.2,14.1; HRMS (ESI) calculated for $C_{12}H_8F_6IN_2O_2^+$ [M-OTf]$^+$ m/z: 452.9529, found: 452.9519.



2f: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.60 (s, 2H), 8.00 (s, 1H), 4.24 (q, *J* = 7.2 Hz, 2H), 1.22 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 161.3, 144.3, 142.4, 130.1, 124.0, 64.0, 26.7, 21.1, 14.2; HRMS (ESI) calculated for $C_{13}H_{16}IN_2O_2^+$ [M-OTf]$^+$ m/z: 359.0250, found: 359.0250.

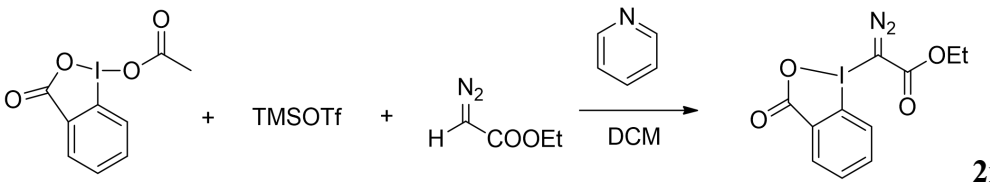


2g: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.31 (q, *J* = 7.2 Hz, 2H), 1.32 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 159.9, 147.1-147.7 (m, 1C), 144.5-145.2 (m, 1C), 138.6-138.8 (m, 1C), 135.9-136.2 (m, 1C), 119.1(q, *J* = 317 Hz), 64.7, 14.0; HRMS (ESI) calculated for $C_{10}H_5F_5IN_2O_2^+$ $[M\text{-}OTf]^+$ m/z: 406.9394, found:. 406.9281.



A solution of 1-methoxy-1,2-benziodoxol-3(1H)-one (4.0 g, 14.38 mmol, 1.0 equiv) in dichloromethane (20 mL) was treated with trimethylsilyl trifluoromethanesulfonate (2.60 mL, 14.38 mmol, 1.0 equiv) at room temperature. After 30 minutes, a cloudy suspension was observed and then the corresponding diazoacetate (31.64 mmol, 2.2 equiv) was added dropwise during 15 minutes. Nitrogen evolution was observed and the resulting reaction mixture was stirred at room temperature until a clear yellow solution was observed (usually 3 hours). Solvent was removed under vacuum and the crude was recrystallized from a mixture of diethyl ether/dichloromethane (5/1) during 12 hours at -30 °C (Note: the recrystallization process may be repeated if impurities are observed). The desired product was collected by filtration, washed with cold diethyl ether (500 mL), dried under high vacuum and stored at -30 °C.[3]

2h: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.41 (dd, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.98 (dt, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.89 (d, *J* = 7.6 Hz,1H), 7.83 (t, *J* = 7.6 Hz,1H), 5.01 (s, 2H), 4.26- 4.37 (m, 4H), 1.28-1.35 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 169.4, 165.9, 161.1, 138.4, 133.6, 132.1, 130.0, 125.4, 116.0, 64.4, 63.8,62.4, 14.2, 14.1. The values of the NMR spectra are in accordance with reported literature data.[3]

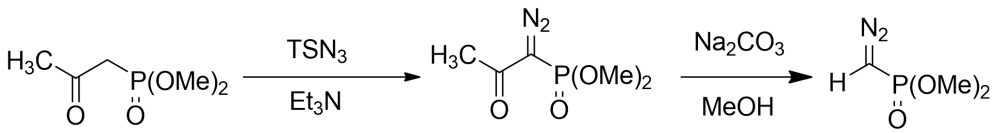


2i

A solution of 1-acetoxy-1,2-benziodoxol-3(1H)-one (3.06 g, 10.0 mmol, 1.0 equiv) in dichloromethane (0.5 M) was treated with trimethylsilyl trifluoromethanesulfonate (1.8 mL, 10 mmol, 1.0 equiv) at room temperature. After 10 minutes, a solution of pyridine (0.88 mL, 11.0 mmol, 1.1 eq) in dichloromethane (2.0 mL) was added dropwise over 10 minutes and the resulted suspension was stirred for 1 hour at room temperature. A solution of the corresponding diazo compound (12 mmol, 1.2 eq) in dichloromethane (2.0 mL) was added dropwise over 10 minutes and the resulting reaction mixture was stirred until a clear yellow solution was obtained (1-6 hours). After this, the solution was washed with distilledwater (200 mL x 2, no vigorously shaking!) and dried with anhydrous sodium sulfate. Solvent was removed under vacuum and the residue was recrystallized from a mixture of diethyl ether/dichloromethane (5/1) during 12 hours at -30 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ: 8.41 (d, *J* = 7.6 Hz, 1H), 7.93-7.98 (m, 1H), 7.80-7.87 (m, 2H), 4.36 (q, *J* = 7.2, 2H), 1.32 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 169.3, 165.8, 138.2, 133.6, 132.0, 128.9, 125.5, 116.1, 63.8, 14.2. The values of the NMR spectra are in accordance with reported literature data.[3]

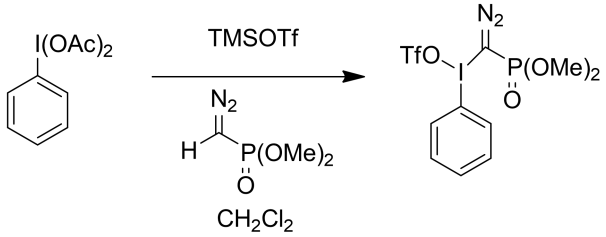
(Note: the recrystallization process may be repeated if impurities are observed). The desired product was collected by filtration, washed with cold diethyl ether (200 mL), dried under high vacuum and stored at -30°C.

The preparation of phosphonate bearing hypervalent iodine reagents was performed according to a literature reference. [4]

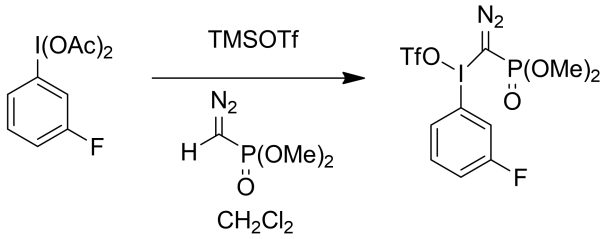


Following a reported procedure, a mixture of dimethyl (2-oxopropyl)phosphonate (6.00 mmol, 1.00 equiv), tosyl azide (1.2 g, 6.6 mmol, 1.1 equiv) and triethylamine (6 mL) was stirred at room temperature for 18 h. After evaporation of triethylamine under reduced pressure, the residue was dissolved in diethyl ether (50 mL). The precipitate was filtered off, the filtrate was evaporated and the residue was purified by column chromatography using 1:1 EtOAc:pentane as mobile phase affording the corresponding dimethyl (1-diazo-2-oxopropyl)phosphonate as a yellow oil (0.75 g, 3.90 mmol, 65%). TLC (EtOAc:pentane, 1:3 v/v): R_f = 0.41, $KMnO_4$; ^{1}H NMR (400 MHz, $CDCl_3$): δ: 3.80 (s, 3H), 3.77 (s, 3H), 2.20 (s, 3H); ^{13}C NMR (100 MHz, CDCl 3): 189.8, 53.6, 53.5, 27.1.

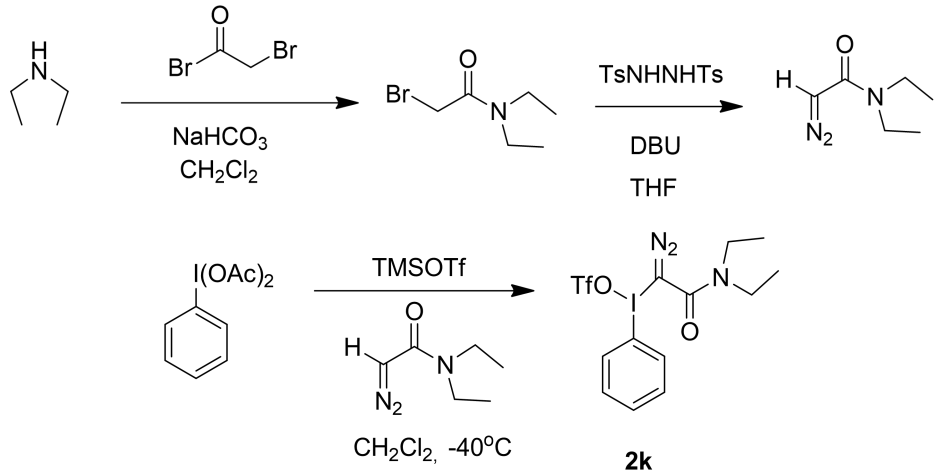
To a solution of dimethyl (1-diazo-2-oxopropyl)phosphonate (1.15 g, 6.0mmol, 1.00 equiv) in MeOH (10 mL) was added Na_2CO_3 (826 mg, 7.8 mmol, 1.3 equiv). The mixture was stirred at room temperature for 15 min. The precipitate was filtered off, the filtrate was evaporated and the residue was purified by column chromatography using 1:1 EtOAc:pentane as mobile phase affording the corresponding dimethyl (diazomethyl)phosphonate as a yellow oil (855 mg, 95%). ^{1}H NMR (400 MHz, $CDCl_3$): δ: 3.80 (s, 3H), 3.78 (d, *J* = 10.8 Hz, 1H), 3.77 (s, 3H); ^{13}C NMR (100 MHz, CDCl 3): 53.1, 53.0.



2j: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.12 (d, *J* = 7.6 Hz, 2H), 7.68 (t, *J* = 7.6 Hz, 1H); 7.52 (d, *J* = 7.6 Hz, 2H), 3.71 (s, 3H), 3.68 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 162.4, 132.8, 131.2, 122.6, 120.6, 116.2, 54.4, 54.3; HRMS (ESI) calculated for $C_9H_{11}IN_2O_3P^+$ $[M\text{-}OTf]^+$ m/z: 352.9552, found: 352.9540.



2l: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.93-7.98 (m, 2H), 7.50-7.56 (m, 1H), 7.36-7.51 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 162.4 (d, J = 255 Hz), 132.7 (d, J = 7.4 Hz), 131.2 (d, J = 3.5 Hz), 122.6 (d, J = 26 Hz), 120.6 (d, J = 20.8 Hz), 116.2 (d, J = 7.4 Hz), 54.4, 54.3; HRMS (ESI) calculated for $C_9H_{10}FIN_2O_3P^+$ [M-OTf]$^+$ m/z: 370.9452, found: 370.9443.

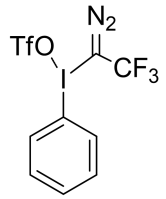


Following a reported procedure, diethyl amine (2.20 g, 30 mmol, 1.0 equiv) and $NaHCO_3$ (7.56 g, 90.0 mmol, 3.0 equiv) were dissolved in dry CH_2Cl_2 (50 mL) and bromoacetyl bromide (7.89 g, 90.0 mmol, 3.0 equiv) was added slowly at 0 °C and stirred for 6 h at room temperature, the reaction was quenched with 100 mL of H_2O and the solution was extracted with CH_2Cl_2 (3 x 50 mL). The combined organic layers were washed with water (100 mL) and dried over $MgSO_4$, the solvent was evaporated and the residue was used in the next step without purification. The resulting bromoacetamide and N,N'-ditosylhydrazine (1.94 g, 10.0 mmol, 1.0 equiv) were dissolved in dry THF (20 mL) and cooled down to 0 ˚C, then DBU (2.30 mL, 15.0 mmol, 1.5 equiv) was added dropwise and stirred at room temperature for 1 h and then quenched with saturated solution of $NaHCO_3$ (50 mL) and extracted with diethyl ether (3 x 50 mL). The combined organic layers were dried over anhydrous $MgSO_4$.

The solvent was removed under reduced pressure and purified by flash column chromatography using 1:4 EtOAc:pentane as mobile phase affording the corresponding 2-diazo- N,N′-diethylacetamide (1e) as a yellow oil (0.84 g, 6.0 mmol, 60%). 1 H NMR (400 MHz, $CDCl_3$): δ 4.97 (s, 1H), 3.25-3.29 (br s, 4H), 1.15 (t, J = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): 164.8, 46.3, 41.4, 13.8. The values of the NMR spectra are in accordance with reported literature data. [3b]

(1-Diazo-2-oxo-2-(diethyl-1-yl)ethyl)(phenyl)iodonium Triflate. [3b] Diacetoxyiodobenzene (1.93 g, 6.0 mmol, 1.0 equiv) was dissolved in dry CH_2Cl_2 (10 mL) and the solution cooled to -40 °C. To this solution was added (TMS)OTf (1.17 mL, 6.0 mmol, 1.0 equiv) in one portion, followed by dropwise addition of 2-diazo-1-(diethyl-1-yl)ethanone (0.85 g, 6.0 mmol, 1.0 equiv) dissolved in dry CH_2Cl_2 (10 mL). The mixture turned red, and some gas evolution occurred. The red solution was stirred at -40 °C for 15 min. Et_2O was added until the mixture became cloudy. At this point the temperature was raised to 0 °C, and more Et_2O was added in small portions until precipitation occurred. The mixture was stirred at 0 °C for 15 min and then cooled again to -40 °C to complete precipitation. The solid was filtered off quickly with suction and washed with -40 °C cooled Et_2O to afford 2.04 g (4.14 mmol, 69%) of (1-Diazo-2-oxo-2-(diethyl-1-yl)ethyl)(phenyl)iodonium Triflate as an orange solid. *The product must be kept below -20 °C to avoid thermal decomposition.*

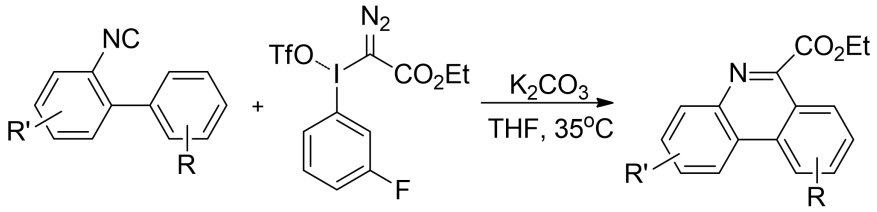
2k: Orange solid;^1H NMR (400 MHz, $CDCl_3$): δ: 8.17 (d, *J* = 8.0 Hz, 2H), 7.65 (t, J = 7.6 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 2H), 3.38 (q, *J* = 7.2 Hz, 4H), 1.16 (t, *J* = 7.2Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ: 159.3, 137.5, 135.9, 132.9, 131.8, 130.3, 127.5, 115.8, 42.9, 13.0.



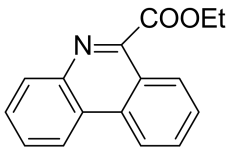
2m: Yellow solid; ^1H NMR (400 MHz, $CDCl_3$) δ: 8.10 (d, J = 8.0 Hz, 2H), 7.71 (t, J =

7.6Hz, 1H), 7.53 (t, J = 7.6Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 137.5, 135.1, 132.9 (d, *J* = 204 Hz), 130.3, 127.5. The values of the NMR spectra are in accordance with reported literature data.[3]

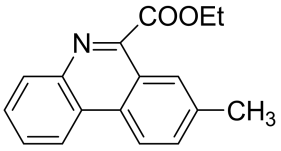
3. Synthesis of phenanthridine-6-carboxylate 3.



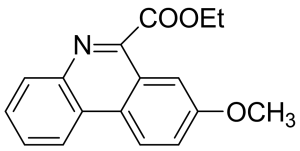
A 15 mL tube was charged with hypervalent iodine reagent **2c** (0.20 mmol) and K_2CO_3 (0.30 mmol). The tube was sealed and filled with N_2. A solution of 2-isocyanodiphenyl **1** (0.44 mmol) in THF (2.0 mL) was preheated to 40°C and rapidly transferred to the tube using syringe. The reaction mixture was stirred at 40°C in an oil bath for 1.5 h. After removal of the solvent, the resulting product was isolated by column chromatography on silica gel using ethylacetate-petroleum ether mixture as eluent.



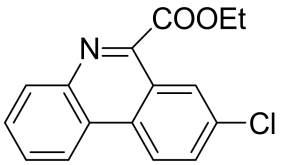
3a: White solid (35 mg, 70%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.61 (d, *J* = 8.4 Hz, 1H), 8.53 (d, *J* = 8.0 Hz, 1H), 8.47 (t, *J* = 8.4 Hz,1H), 8.22 (d, J = 8.4 Hz, 1H), 7.83 (t, *J* = 8.0 Hz, 1H), 7.64-7.74 (m, 3H), 4.57 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.4, 151.2, 142.8, 133.5, 131.2, 131.0, 129.1, 128.6, 127.9, 127.3, 124.9, 123.4, 122.2, 122.1, 62.4, 14.4; HRMS (ESI) calculatedfor $C_{16}H_{14}NO_2^+$ $[M+H]^+$ m/z: 252.1025, Found: 252.1021；Anal. Calcd for $C_{16}H_{13}NO_2$: C, 76.48; H, 5.21; N, 5.57; Found: C, 74.71; H, 5.66; N, 6.300.



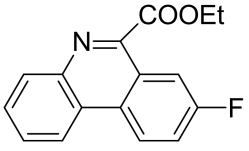
3b: White solid (37 mg, 71%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.46 (d, *J* = 8.4 Hz, 2H), 8.21 (s, 1H), 8.18 (d, *J* = 8.4 Hz,1H), 7.60-7.68 (m, 3H), 4.56 (q, *J* = 7.2 Hz, 2H), 2.52 (s, 3H), 1.46 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.5, 150.9, 142.4, 138.0, 133.0, 131.4, 130.9, 128.6, 128.5, 126.5, 124.9, 123.6, 122.1, 121.9, 62.3, 21.9, 14.4; HRMS (ESI) calculated for $C_{17}H_{16}NO_2^+$ $[M+H]^+$ m/z: 266.1181, Found 266.1179.



3c: White solid (32 mg, 57%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.51 (d, *J* = 8.8 Hz, 1H), 8.42-8.45 (m, 1H), 8.18-8.21 (m,1H), 7.97 (d, *J* = 2.8 Hz, 1H), 7.63-7.67 (m, 2H), 7.44 (dd, *J* = 8.8 Hz, 2.8 Hz, 1H), 4.56 (q, *J* = 7.2 Hz, 2H), 3.92 (s, 3H), 1.47 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.4, 159.0, 149.5, 141.9, 131.0, 128.7, 128.1, 128.0, 125.1, 125.0, 123.8, 122.4, 121.6, 106.8, 62.3, 55.5, 14.4; HRMS (ESI) calculatedfor $C_{17}H_{16}NO_3^+$ $[M+H]^+$ m/z: 282.1130, Found 282.1132.

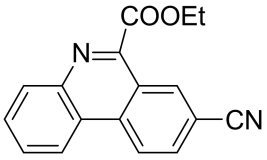


3d: White solid (31mg, 55%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.57 (d, *J* = 2.0 Hz, 1H), 8.54 (d, *J* = 8.8 Hz, 1H), 8.47-8.48 (m, 1H), 8.22-8.25 (m, 1H), 7.69-7.79 (m, 3H), 4.58 (q, *J* = 7.2 Hz, 2H), 1.47 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 165.8, 149.5, 142.6, 134.1, 131.8, 131.7, 131.2, 129.4, 129.2, 126.7, 124.4, 123.9, 121.9, 62.6, 14.4; HRMS (ESI) calculated for $C_{16}H_{13}ClNO_2^+$ $[M+H]^+$ m/z: 286.0635, Found: 286.0639.

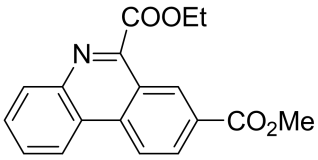


3e: White solid (31 mg, 58%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.67-8.72 (m, 1H), 8.54-8.57 (m, 1H), 8.31-8.39 (m, 2H), 7.78-7.81 (m, 2H), 7.64-7.69 (m, 1H), 4.67 (q,

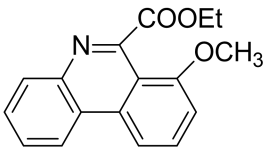
J = 7.2 Hz, 2H), 1.57 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 165.9, 149.6 (d, *J* = 3.9 Hz), 142.4, 131.2, 130.2, 129.1, 128.9, 124.8, 124.7, 121.8, 120.5 (d, *J* = 24 Hz), 112.3, 112.0, 62.5, 55.5, 14.3; HRMS (ESI) calculatedfor $C_{16}H_{13}NO_2^+$ $[M+H]^+$ m/z: 270.0924, Found: 270.0923.



3f: White solid (27 mg, 49%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.00 (s, 1H), 8.66 (d, *J* = 8.4 Hz, 1H), 8.51 (d, *J* = 8.0 Hz, 1H), 8.26 (d, *J* = 8.0 Hz, 1H), 7.96 (d, *J* = 8.4 Hz, 1H), 7.74-7.84 (m, 2H), 4.58 (q, *J* = 7.2 Hz, 2H), 1.48 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 165.4, 149.5, 143.6, 135.7, 133.1, 132.2, 131.4, 130.8, 129.6, 123.7, 123.5, 123.0, 122.6, 118.3, 111.6, 62.9, 14.3; HRMS (ESI) calculated for $C_{17}H_{13}N_2O_2^+$ $[M+H]^+$ m/z: 277.0977, Found: 277.0968.

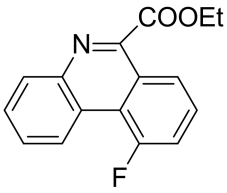


3g: White solid (28 mg, 46%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.32 (s, 1H), 8.77 (d, *J* = 8.8 Hz, 1H), 8.67 (d, *J* = 8.0 Hz, 1H), 8.53 (d, *J* = 8.8 Hz, 1H), 8.35 (d, *J* = 8.0 Hz, 1H), 7.81-7.90 (m, 2H), 4.70 (q, *J* = 7.2 Hz, 2H), 4.06 (s, 3H), 1.59 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ:166.4, 165.9, 151.3,143.5, 136.3, 131.2, 130.9, 130.2, 129.8, 129.3, 129.1, 124.3, 123.0, 122.7, 122.6, 62.6, 52.6, 14.4 ; HRMS (ESI) calculated for $C_{19}H_{18}NO_4^+$ $[M+H]^+$ m/z: 324.1236, Found: 324.1252.

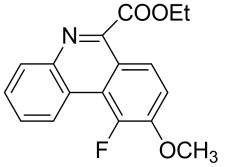


3h: White solid (29 mg, 52%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.47 (d, *J* = 8.0 Hz, 1H), 8.15 (t, *J* = 8.8 Hz, 2H), 7.74 (t, *J* = 8.0 Hz,1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.03 (d, *J* = 8.0 Hz, 1H), 4.49 (q, *J* = 7.2 Hz, 2H), 3.95 (s, 3H), 1.41 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 168.5, 156.5, 150.8, 143.1, 135.1,

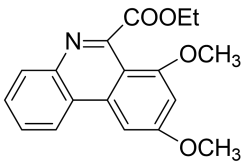
132.1, 130.3, 129.2, 127.8, 123.7, 122.5, 114.6, 114.1, 108.0, 61.9, 56.3, 14.3; HRMS (ESI) calculated for $C_{17}H_{16}NO_3^+$ $[M+H]^+$ m/z: 282.1130, Found: 282.1130.



3i: White solid (30 mg, 57%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.01 (d, *J* = 7.6 Hz, 1H), 8.33 (t, *J* = 8.0 Hz, 2H), 7.78-7.86 (m, 2H), 7.68-7.72 (m, 1H), 7.59-7.65 (m, 1H), 4.67 (q, *J* = 7.2 Hz, 2H), 1.55 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.2, 150.9, 143.1, 130.9, 129.3, 129.1, 128.2, 128.1, 127.1, 126.8, 125.4 (d, *J* = 3.9 Hz), 123.3 (d, *J* = 4.0 Hz), 117.8, 117.6, 62.5, 14.3; HRMS (ESI) calculatedfor $C_{16}H_{13}NO_2^+$ $[M+H]^+$ m/z: 270.0924, Found: 270.0926.

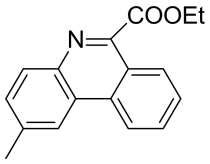


3j: White solid (37 mg, 62%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.93 (d, *J* = 8.0 Hz, 1H), 8.28 (dd, *J* = 8.8, 1.2 Hz, 1H), 8.21 (d, *J* = 8.0 Hz, 1H),7.65-7.75 (m, 2H), 7.39 (t, *J* = 8.4 Hz, 1H), 7.19 (s, 1H), 4.56 (q, *J* = 7.2 Hz, 2H), 4.04 (s, 3H), 1.46 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.3, 150.5 (d, *J* = 18 Hz), 149.3 (d, *J* = 11 Hz), 147.9, 143.2, 130.8, 129.4, 128.2, 128.7, 127.2, 124.0 (d, *J* = 5.0 Hz), 122.3, 118.9 (d, *J* = 2.9 Hz), 114.0, 62.5, 56.8, 14.3; HRMS (ESI) calculated for $C_{17}H_{15}FNO_3^+$ [M+H]+ m/z: 300.1030, Found: 300.1029.

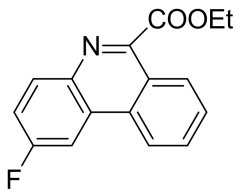


3k: White solid (50 mg, 81%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.37 (d, *J* = 8.0 Hz, 1H), 8.09 (t, *J* = 8.0 Hz, 1H), 7.66 (t, *J* = 8.0 Hz, 1H), 7.57 (t, *J* = 8.0 Hz, 1H), 7.46 (s, 1H), 6.59 (s, 1H), 4.47 (q, *J* = 7.2 Hz, 2H), 3.97 (s, 3H), 3.90 (s, 3H), 1.39 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 168.6, 163.0, 158.0, 150.4, 143.6, 136.8, 130.3, 129.3, 127.2, 123.5, 122.4, 109.7, 99.2, 95.1, 61.8, 56.2, 55.7, 14.3; HRMS

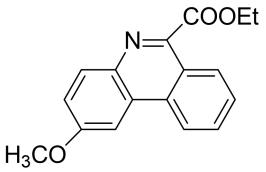
(ESI) calculated for $C_{18}H_{18}NO_4^+$ [M+H]+ m/z: 312.1230, Found: 312.1223.



3l: White solid (36 mg, 68%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.69 (d, *J* = 8.4 Hz, 1H), 8.59 (d, *J* = 8.0 Hz, 1H), 8.40 (s, 1H), 8.21 (d, *J* = 8.4 Hz, 1H), 7.90 (t, *J* = 7.6 Hz, 1H), 7.74 (t, *J* = 8.0 Hz, 1H),7.63 (d, *J* = 8.0 Hz, 1H), 4.65 (q, *J* = 7.2 Hz, 2H), 2.67 (s, 3H), 1.56 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.4, 150.1, 141.1, 138.8, 133.1, 130.9, 130.8, 130.7, 127.7, 127.3, 124.8, 123.6, 122.2, 121.7, 62.3, 22.2, 14.4; HRMS (ESI) calculated for $C_{17}H_{16}NO_2^+$ [M+H]+ m/z: 266.1181, Found:266.1184.

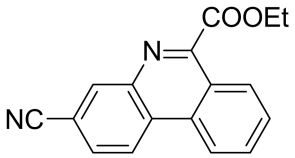


3m: White solid (25 mg, 47%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.60 (d, *J* = 8.4 Hz, 1H), 8.54-8.57 (m, 1H), 8.28-8.33 (m, 1H), 8.18-8.22 (m, 1H), 7.90-7.94 (m, 1H), 7.79 (t, *J* = 8.0 Hz, 1H), 7.51-7.57 (m, 1H), 4.66 (q, *J* = 7.2 Hz, 2H), 1.56 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.2, 150.3, 139.5, 133.4 (d, *J* = 9.3 Hz), 132.9, 131.2, 128.6, 127.5, 126.5, 123.5, 122.4, 118.3, 118.1, 107.0 (d, *J* = 23 Hz), 62.5, 14.4; HRMS (ESI) calculatedfor $C_{16}H_{13}NO_2^+$ [M+H]$^+$ m/z: 270.0924, Found: 270.0923.

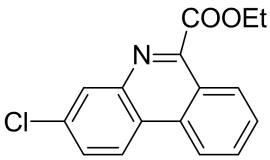


3n: White solid (31 mg, 55%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.56 (d, *J* = 8.4 Hz, 1H), 8.51 (d, *J* = 8.4 Hz, 1H), 8.14 (d, *J* = 8.8 Hz, 1H), 7.83 (d, *J* = 3.6 Hz, 1H), 7.90 (t, *J* = 8.0 Hz, 1H), 7.66 (t, *J* = 7.6 Hz, 1H), 7.33 (dd, *J* = 8.8, 3.6 Hz, 1H), 4.55 (q, *J* = 7.2 Hz, 2H), 3.97 (s, 3H), 1.46 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ:

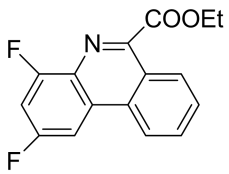
166.4, 159.8, 148.1, 138.0, 132.8, 132.6, 130.6, 128.0, 127.4, 126.3, 123.8, 122.2, 119.2, 102.7, 62.2, 55.7, 14.4; HRMS (ESI) calculated for $C_{17}H_{16}NO_3^+$ [M+H]+ m/z: 282.1130, Found: 282.1126.



3n: White solid (25 mg, 46%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.94 (s, 1H), 8.65 (d, *J* = 8.4 Hz, 1H), 8.53 (d, *J* = 8.0 Hz, 1H), 8.36 (d, *J* = 8.4 Hz, 1H), 7.94-8.02 (m, 2H), 7.85 (t, *J* = 8.0 Hz, 1H), 4.65 (q, *J* = 7.2 Hz, 2H), 1.54 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 165.7, 154.2, 144.4, 132.5,132.3, 132.1, 130.5, 129.2, 127.8, 127.7, 124.9, 123.6, 122.2, 118.7, 112.0, 62.7, 14.3; HRMS (ESI) calculated for $C_{17}H_{13}N_2O_2^+$ [M+H]+ m/z: 277.0977, Found: 277.0968.

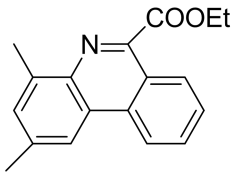


3o: White solid (28 mg, 49%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.61 (d, *J* = 8.4 Hz, 1H), 8.52 (d, *J* = 7.2 Hz, 1H), 8.51 (d, *J* = 8.0 Hz, 1H), 8.28 (d, *J* = 2.4 Hz, 1H), 7.90 (dt, *J* = 8.0 Hz, 3.2 Hz, 1H), 7.75 (t, *J* = 8.0 Hz, 1H), 8.28 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 4.63 (q, *J* = 7.2 Hz, 2H), 1.53 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.1, 152.4, 143.4, 134.8, 133.1, 131.6, 130.1, 129.2, 129.1, 128.2, 127.5, 123.4, 123.3, 122.1, 62.5, 14.3; HRMS (ESI) calculated for $C_{16}H_{13}ClNO_2^+$ [M+H]+ m/z: 286.0635, Found: 286.0631.



3q: White solid (24 mg, 42%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.51 (d, *J* = 8.4 Hz, 1H), 8.44 (d, *J* = 8.4Hz, 1H), 7.94 (d, *J* = 9.2Hz, 1H), 7.86 (t, *J* = 7.6 Hz, 1H), 7.74 (t, *J* = 8.0 Hz, 1H), 7.19-7.24 (m, 1H), 4.56 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H);

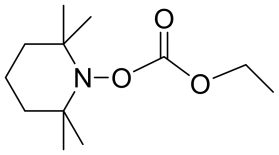
^{13}C NMR (101 MHz, $CDCl_3$) δ: 165.8, 131.7, 129.2, 127.7, 123.9, 122.6, 104.9, 104.7, 104.6, 104.4, 103.2, 103.1, 103.0, 102.9, 62.5, 14.3; HRMS (ESI) calculated for $C_{16}H_{12}NO_2^+$ [M+H]+ m/z: 288.0836, Found: 288.0835.



3r: White solid (35 mg, 63%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.57 (d, *J* = 8.0 Hz, 1H), 8.44 (d, *J* = 8.4 Hz, 1H), 8.15 (s, 1H), 7.76 (t, *J* = 7.6 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.39 (s, 1H), 4.54 (q, *J* = 7.2 Hz, 2H), 2.79 (s, 3H), 2.53 (s, 3H), 1.46 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.9, 148.6, 140.0, 138.4, 133.4, 131.6, 130.5, 129.0, 127.5, 127.0, 124.7, 123.3, 122.4, 119.4, 61.9, 22.1, 18.1, 14.4; HRMS (ESI) calculated for $C_{18}H_{18}NO_2^+$ [M+H]+ m/z: 280.1338, Found: 280.1335.

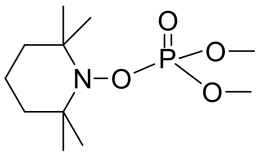
4. Synthesis of TEMPO-diazomethane adducts 4-6.

A 15 mL tube was charged with hypervalent iodine reagent **2a (**or **2j or 2k)** (0.20 mmol), 2,2,6,6-tetramethylpyperidine oxide (TEMPO) (0.24 mmol) and K_2CO_3 (0.30 mmol). The tube was sealed and filled with N_2. A solution of 2-isocyanodiphenyl **1** (0.44 mmol) in THF (2.0 mL) was preheated to 40ºC and rapidly transferred to the tube using syringe. The reaction mixture was stirred at 40ºC in an oil bath for 0.5 h. After removal of the solvent, the resulting product was isolated by column chromatography on silica gel using ethylacetate-petroleum ether mixture as eluent.

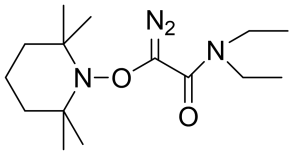


4: Yellowish oil (40 mg, 86%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.20 (q, *J* = 7.2 Hz, 2H), 1.67 (s, 6H), 1.41 (s, 12H), 1.28 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 164.8, 164.4, 61.7, 57.1, 37.7, 29.1, 14.9, 13.8; HRMS (ESI) calculated for

$C_{12}H_{24}NO_3^+$ $[M+H]^+$ m/z: 230.1756, Found: 230.1760; Anal. Calcd for $C_{12}H_{23}NO_3$: C, 62.5; H, 10.5; N, 6.08; Found: C, 62.8; H, 6.8; N, 5.5.



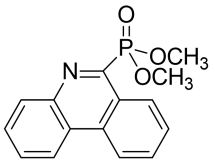
5: Yellowish oil (42 mg, 79%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 3.79 (s, 3H), 3.76 (s, 3H), 1.71 (s, 6H), 1.52 (s, 12H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 58.3, 58.2, 54.3, 54.2, 35.9, 29.3, 14.2; Anal. Calcd for $C_{11}H_{24}NO_4P$: C, 49.8; H, 9.1; N, 5.3; Found: C, 46.7; H, 5.7; N, 4.4.



6: Yellowish oil (20 mg, 35%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 3.43 (q, J = 7.2 Hz, 2H), 3.18 (q, J = 7.2 Hz, 2H), 1.85-1.87 (m, 2H), 1.62-1.80 (m, 4H),1.62 (s, 6H), 1.41 (s, 6H), 1.18 (t, J = 7.2 Hz, 3H), 1.15 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 179.1, 167.6, 66.2, 65.3, 41.9, 41.7, 38.2, 37.9, 31.3, 25.8, 15.8, 13.9, 13.0; HRMS (ESI) calculated for $C_{15}H_{29}N_4O_2^+$ $[M+H]^+$ m/z: 297.2291, Found: 297.2286; Anal. Calcd for $C_{15}H_{28}N_4O_2$: C, 60.8; H, 9.5; N, 18.8; Found: C, 56.7; H, 6.2; N, 15.9.

5. Synthesis of phenanthridine-6-phosphonate 7

A 15 mL tube was charged with hypervalent iodine reagent **2l** (0.20 mmol), and K_2CO_3 (0.30 mmol). The tube was sealed and filled with N_2. A solution of 2-isocyanodiphenyl **1** (0.44 mmol) in THF (2.0 mL) was preheated to 40ºC and rapidly transferred to the tube using syringe. The reaction mixture was stirred at 40ºC in an oil bath for 0.5 h. After removal of the solvent, the resulting product phenanthridine-6-phosphonate **7** was isolated by column chromatography on silica gel using ethylacetate-petroleum ether mixture (1:1) as eluent.



7: Colorless solid (32 mg, 56%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.87 (d, J = 8.4 Hz, 1H), 8.62 (d, J = 8.4 Hz, 1H), 8.55 (d, J = 8.4 Hz, 1H), 8.25 (d, J = 8.4 Hz, 1H), 7.83 (t, J = 7.6 Hz, 2H), 7.68-7.73 (m, 3H), 3.98 (s, 3H), 3.95 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 143.2, 132.7, 131.4, 131.2, 129.1, 128.9, 128.0, 126.4, 126.1, 124.7, 122.2, 122.1, 54.1, 54.0; HRMS (ESI) calculated for $C_{15}H_{15}NO_3P^+$ [M+H]+ m/z: 288.0784, Found: 288.0780.

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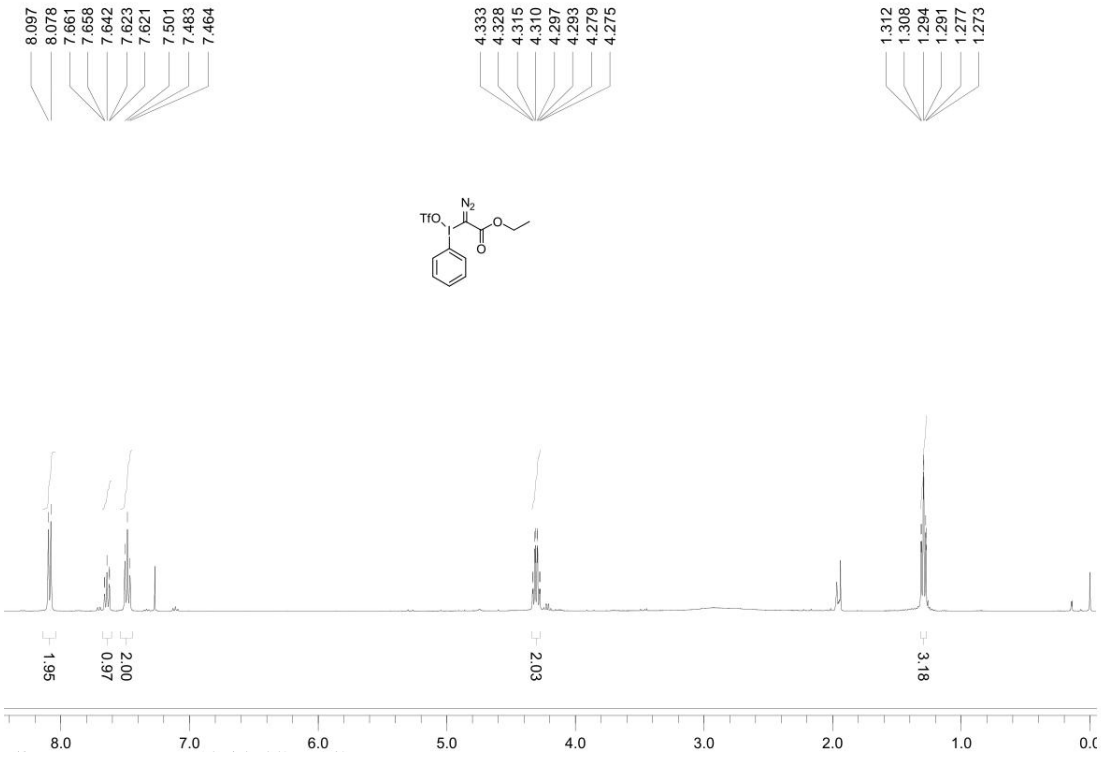
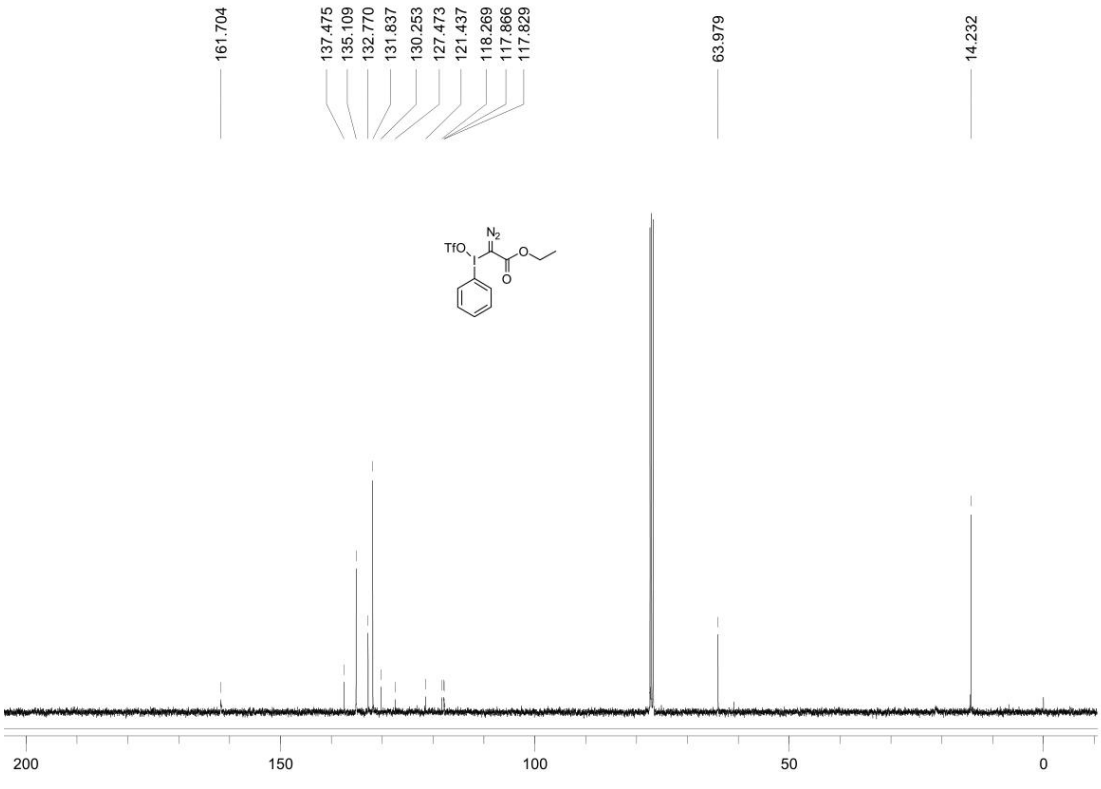
1. Zhang. B.; Christian, M. L; Constantin. G. D.; Armido. S. *Angew. Chem. Int. Ed.*, **2013**, *125*, 10992-10995.
2. (a) Kazmierczak, P.; Skulski, L.; Kraszkiewicz, L. *Molecules*., **2001**, *6*, 881-891. (b) Kamalkishor. P. L.; Keun S. J.; Sang Y. L.; Dae Y.C. *J. Org. Chem*., **2012**, *77*, 5705-5713.
3. (a) Wang. Z.; Herraiz. A. G.; Suero. M. G. *Nature.*, **2018**, *554*, 86-91. (b) Christain. S.; Martin. H.; Tore. B. H. *J. Org. Chem*., **2013**, *78*, 7488-7497. (c) Wang. Z.; Jiang. L.; Suero. M. G. *J. Am. Chem. Soc*., **2019**, *141*, 15509-15514. (d) Robert. W.; Jorg. S., Frank H. *Angew. Chem. Int. Ed.*, **1994**, *33*, 1952-1953.
4. Durga. P. H.; Jerome. W. *J. Am. Chem. Soc*., **2016**, *138*, 2190-2193.

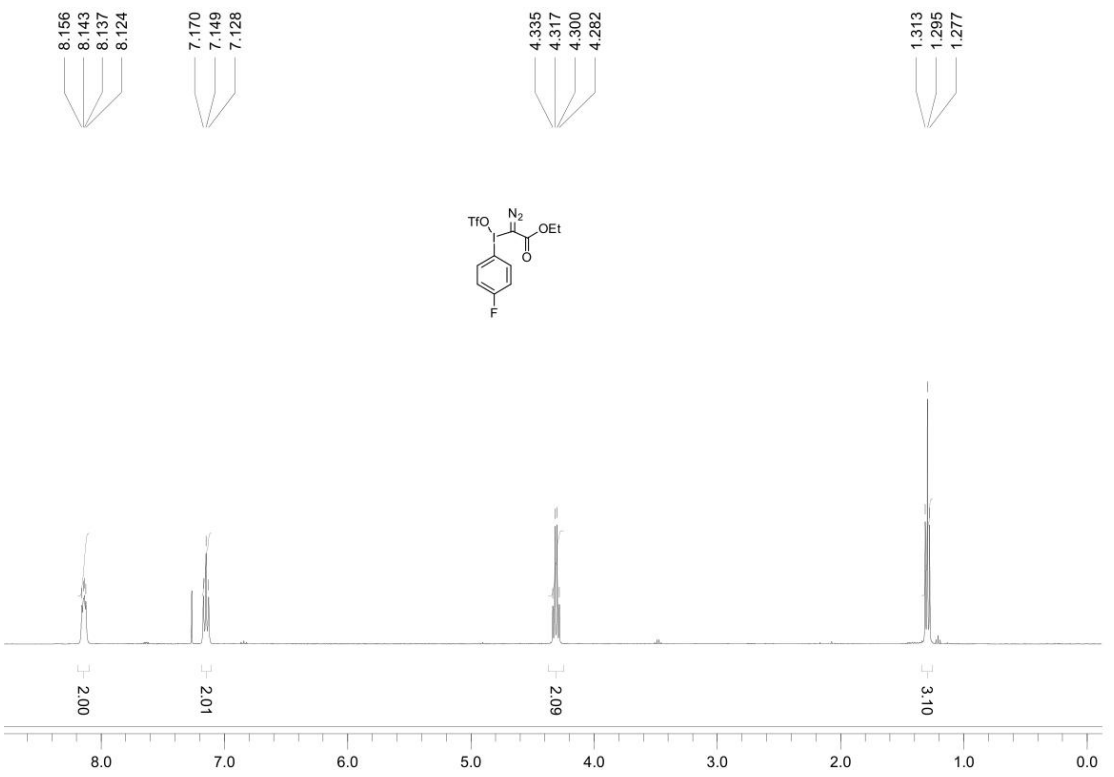
IV NMR Spectrum

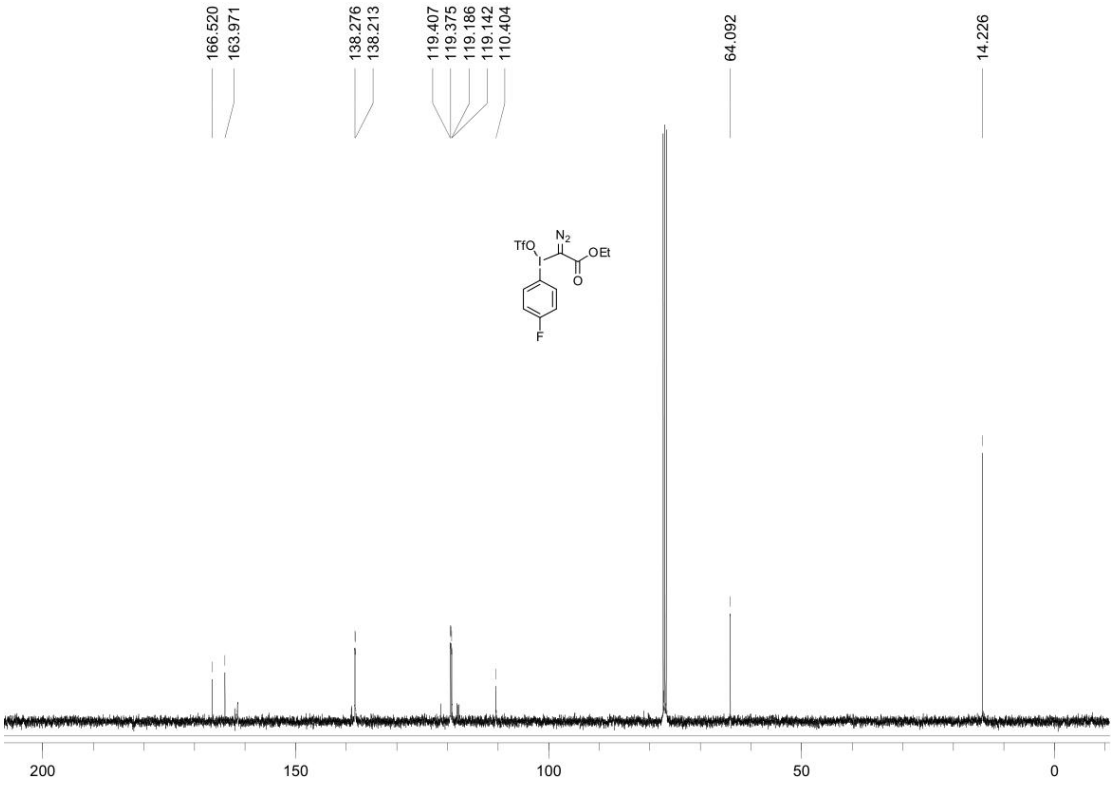
^{1}H NMR and ^{13}C NMR spectrum of **2a**



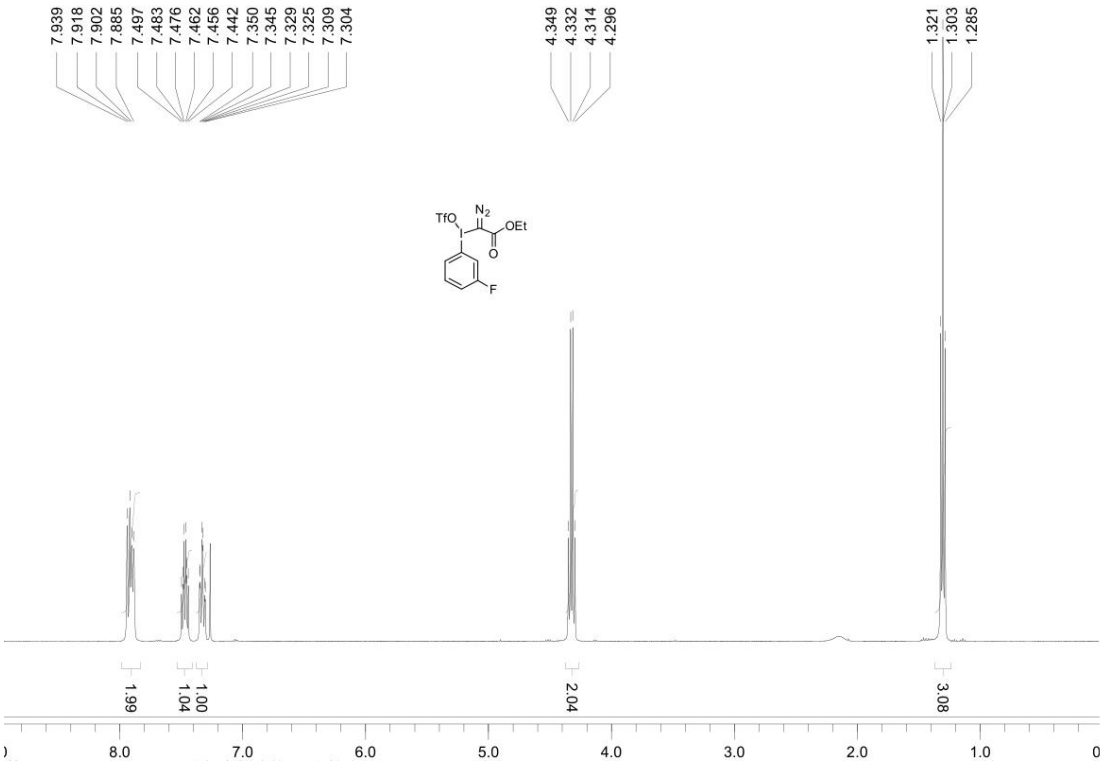


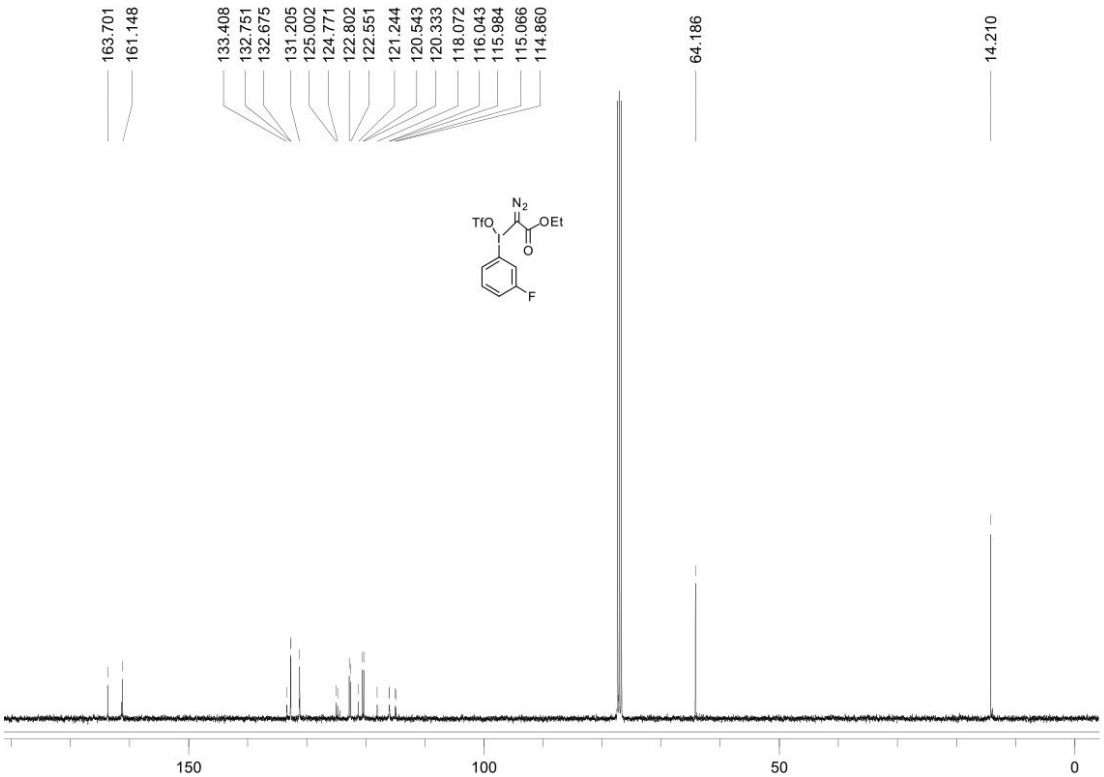
^{1}H NMR and ^{13}C NMR spectrum of **2b**



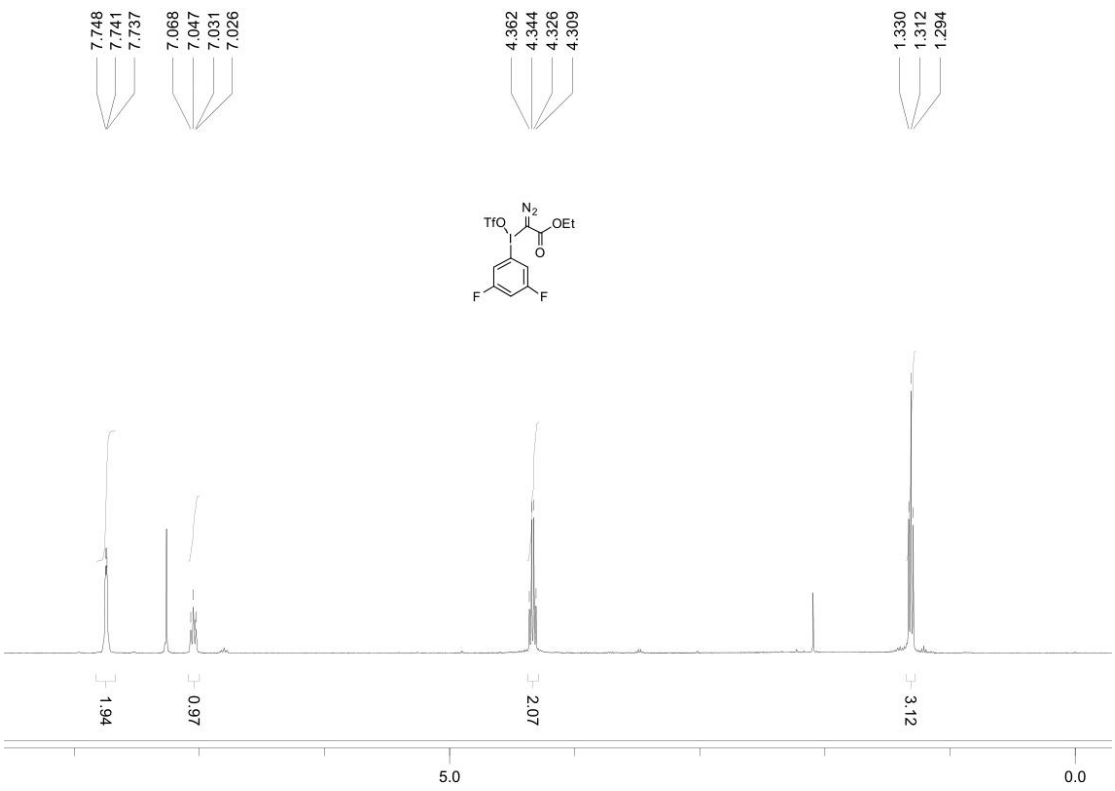


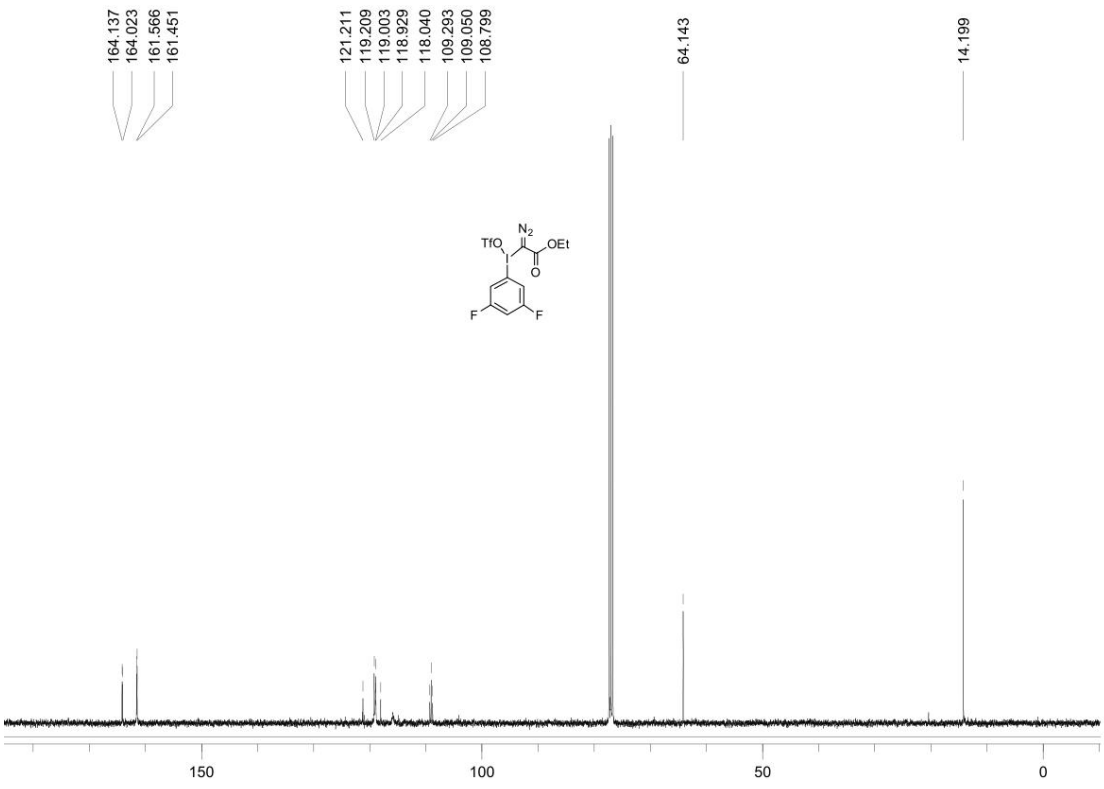
^{1}H NMR and ^{13}C NMR spectrum of **2c**



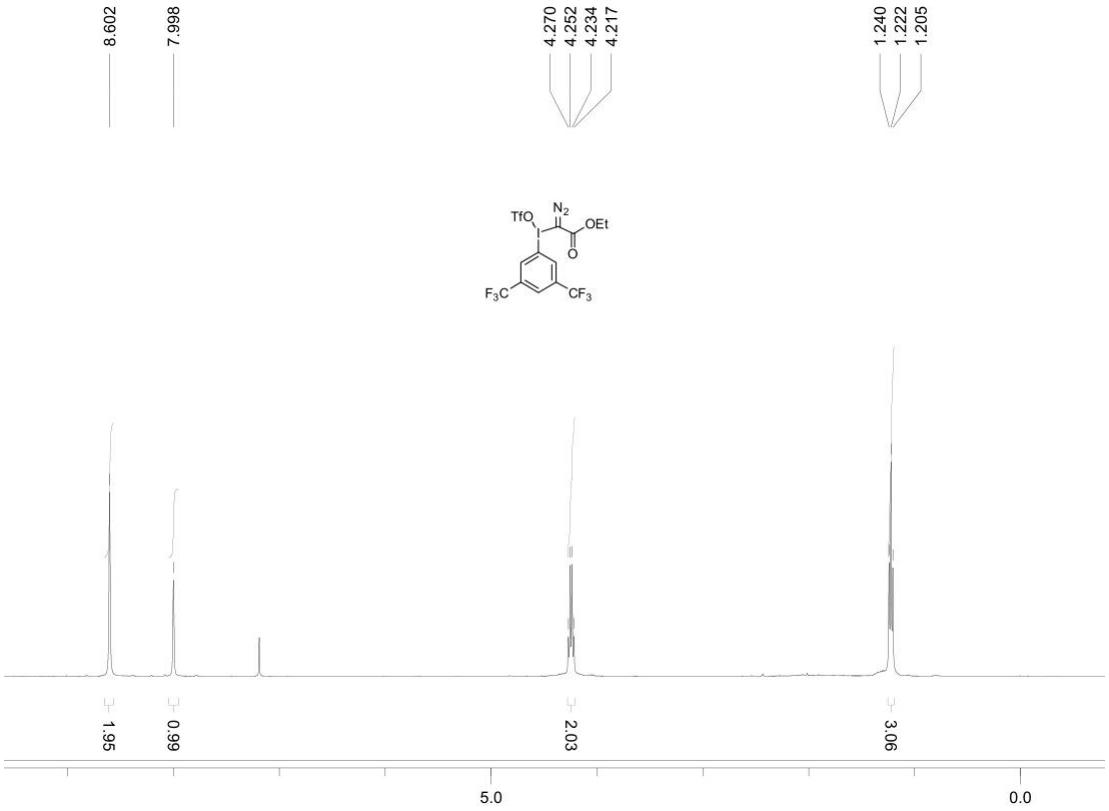


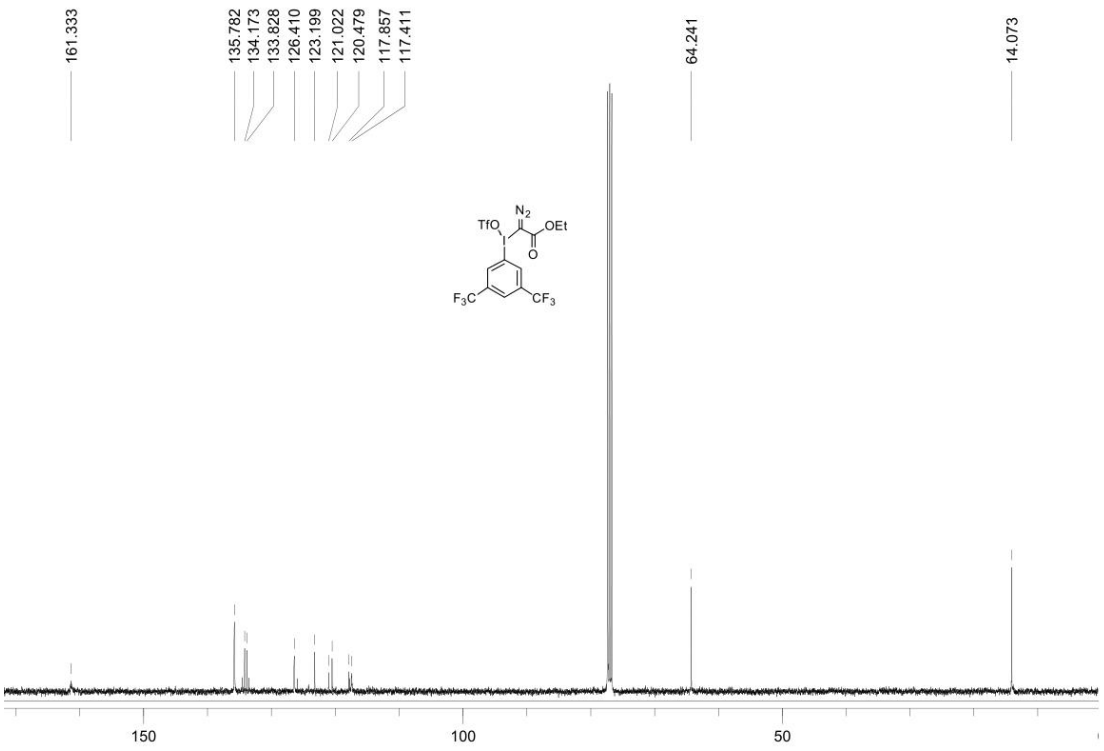
^{1}H NMR and ^{13}C NMR spectrum of **2d**



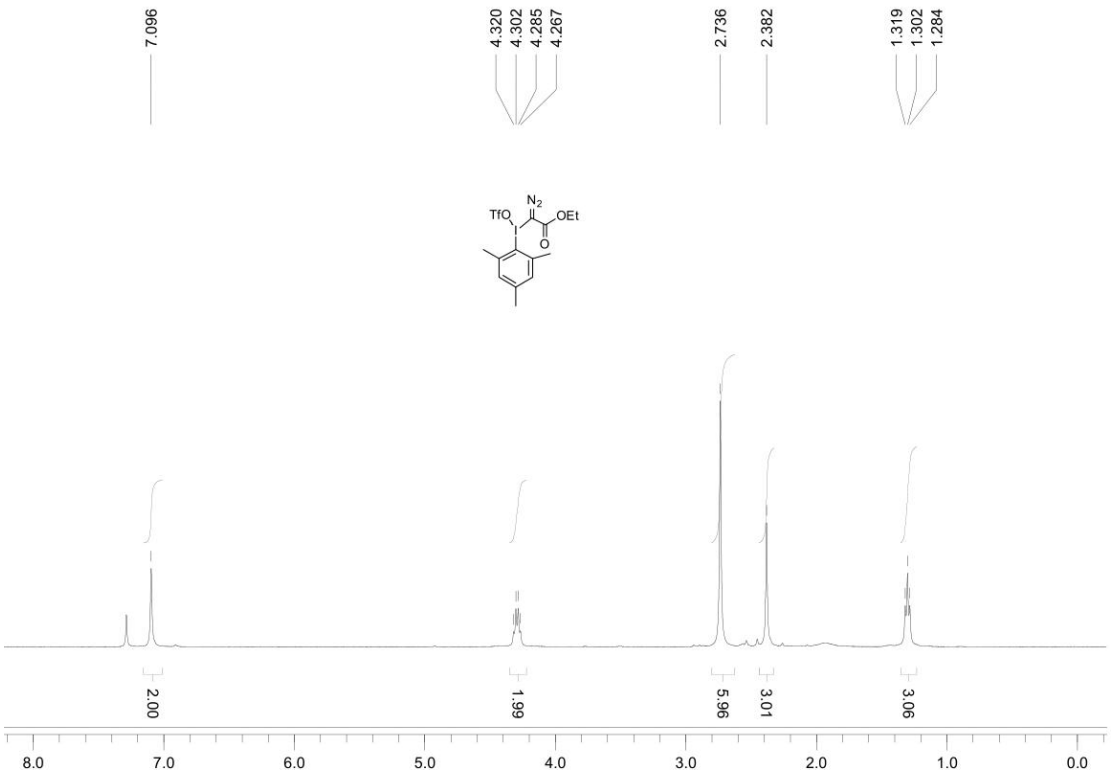


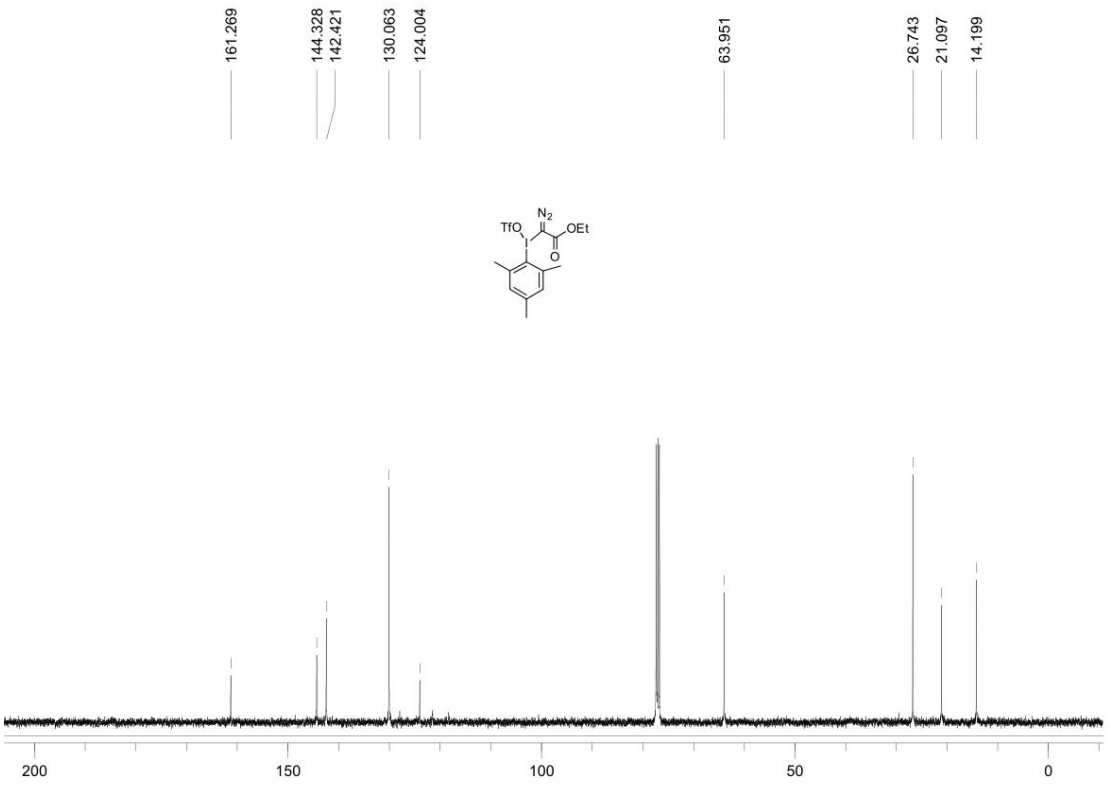
^{1}H NMR and ^{13}C NMR spectrum of **2e**



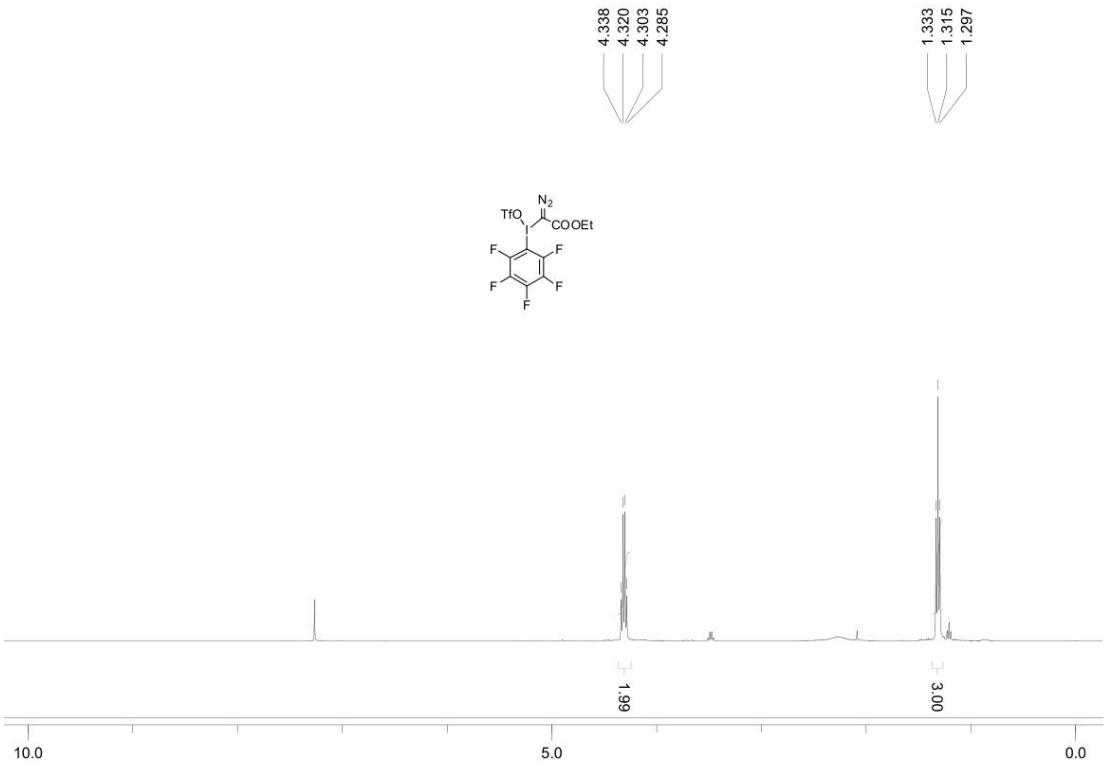


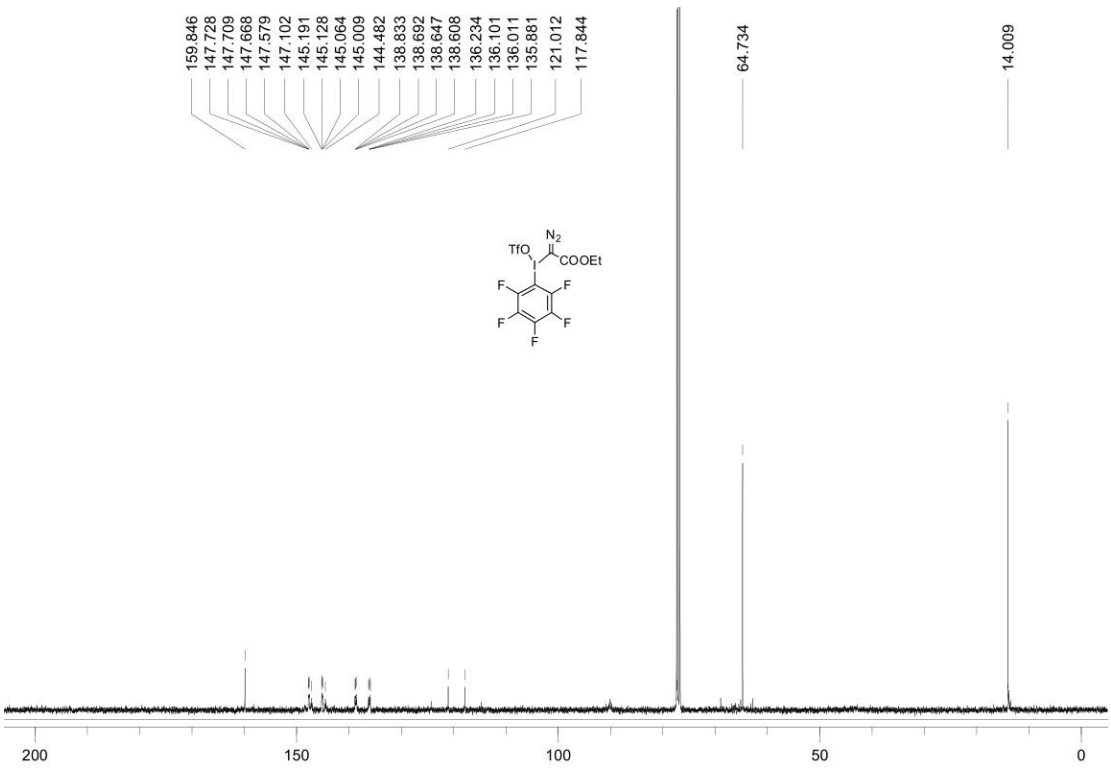
^{1}H NMR and ^{13}C NMR spectrum of **2f**



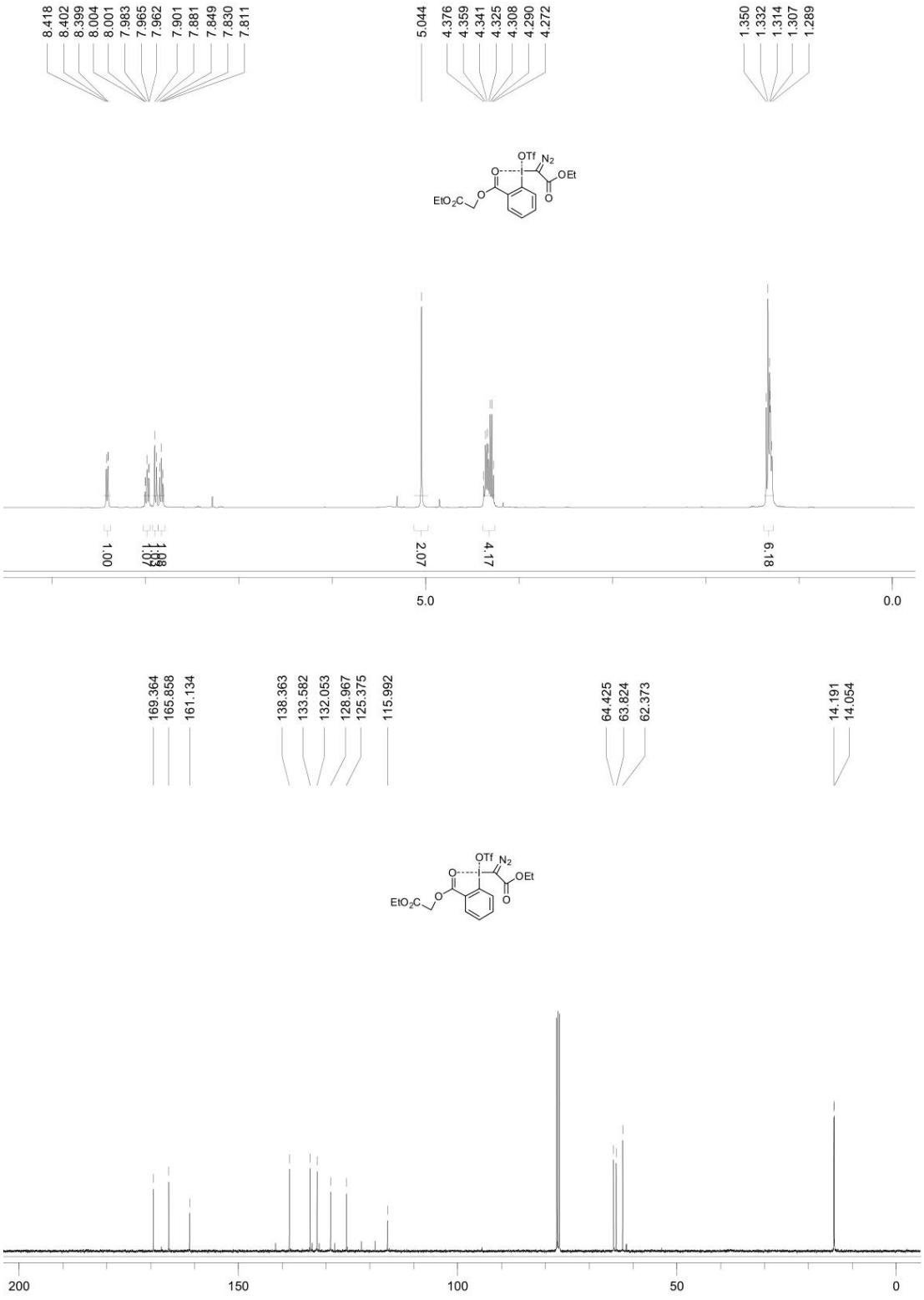


^{1}H NMR and ^{13}C NMR spectrum of **2g**

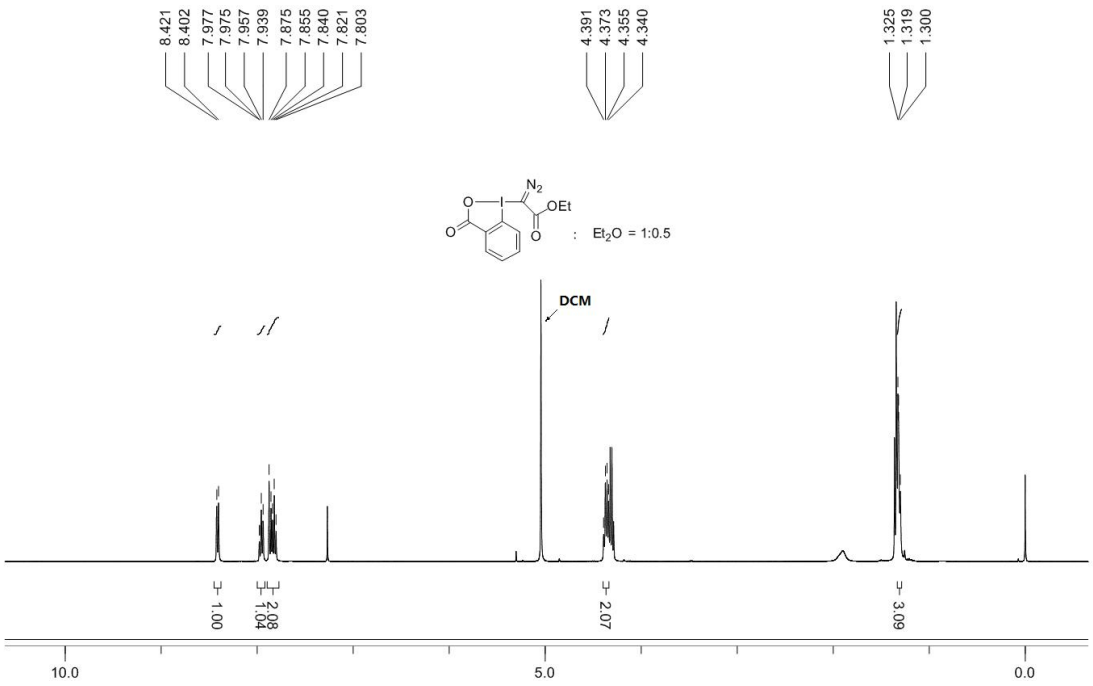


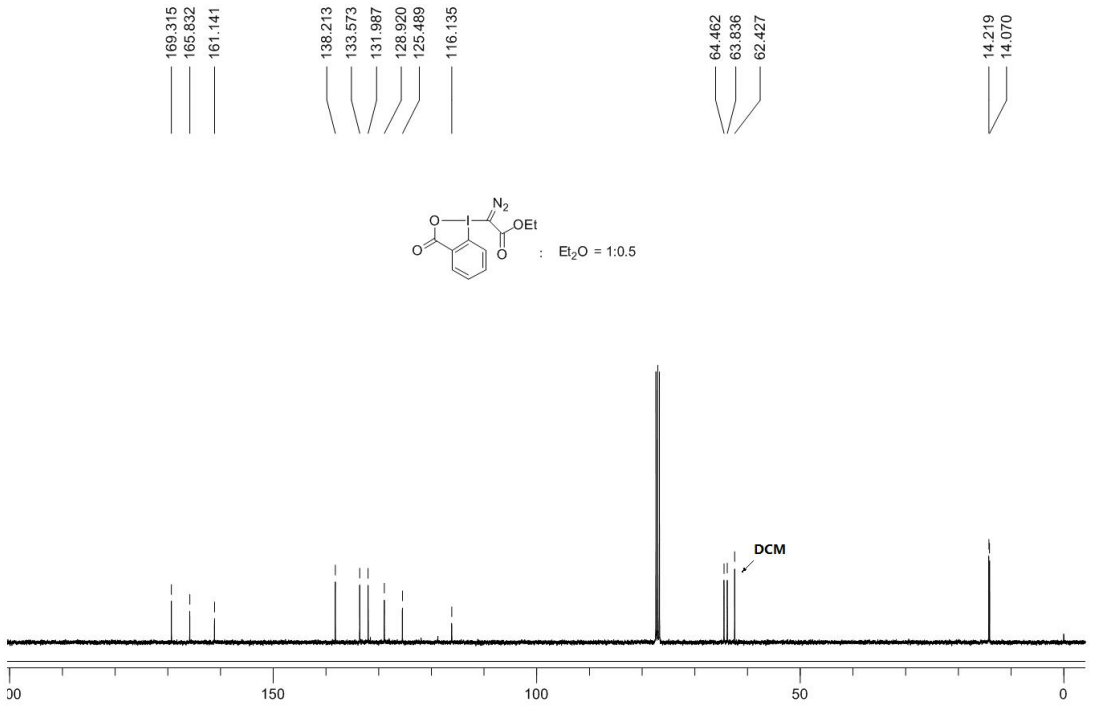


^{1}H NMR and ^{13}C NMR spectrum of **2h**

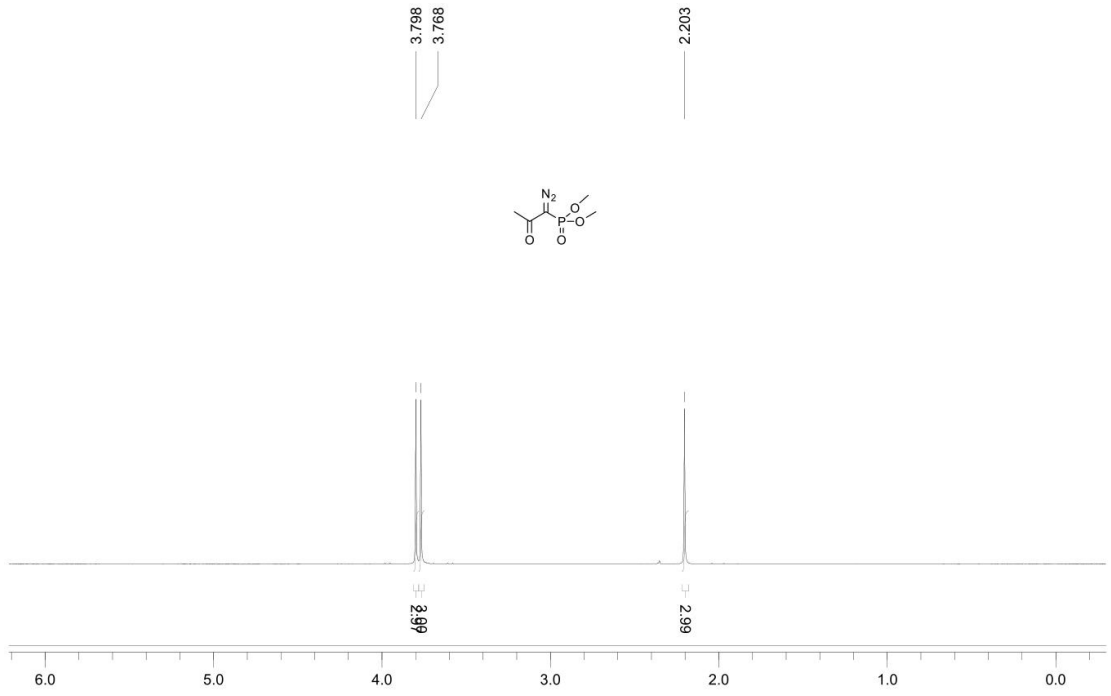


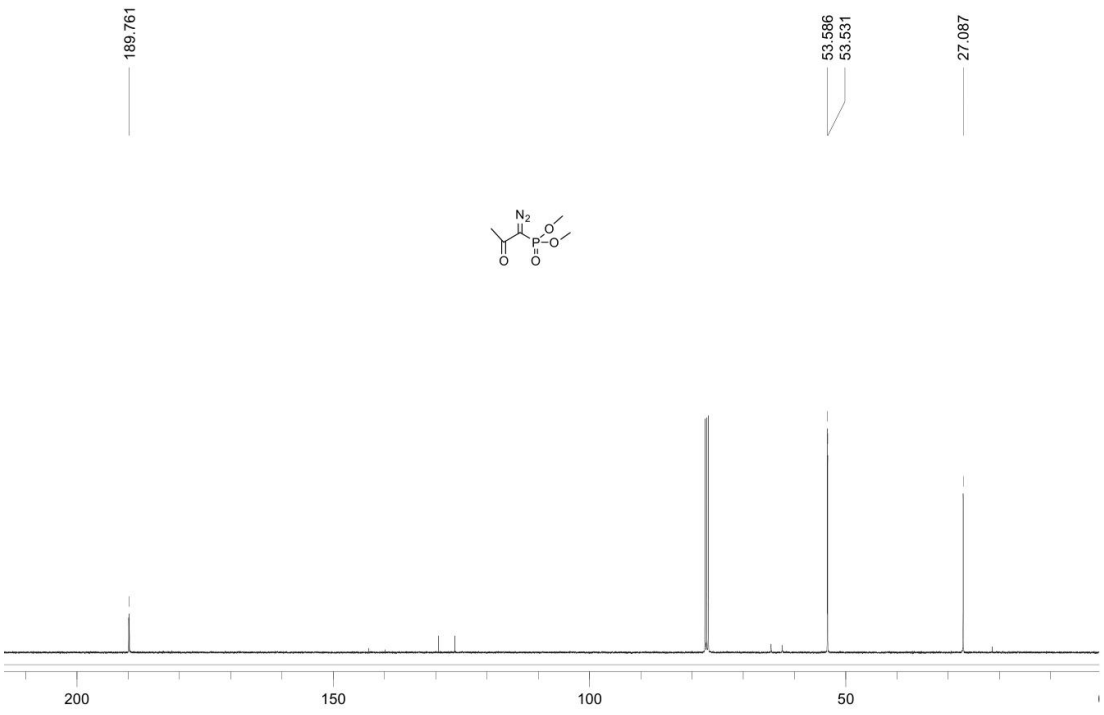
^{1}H NMR and ^{13}C NMR spectrum of **2i**



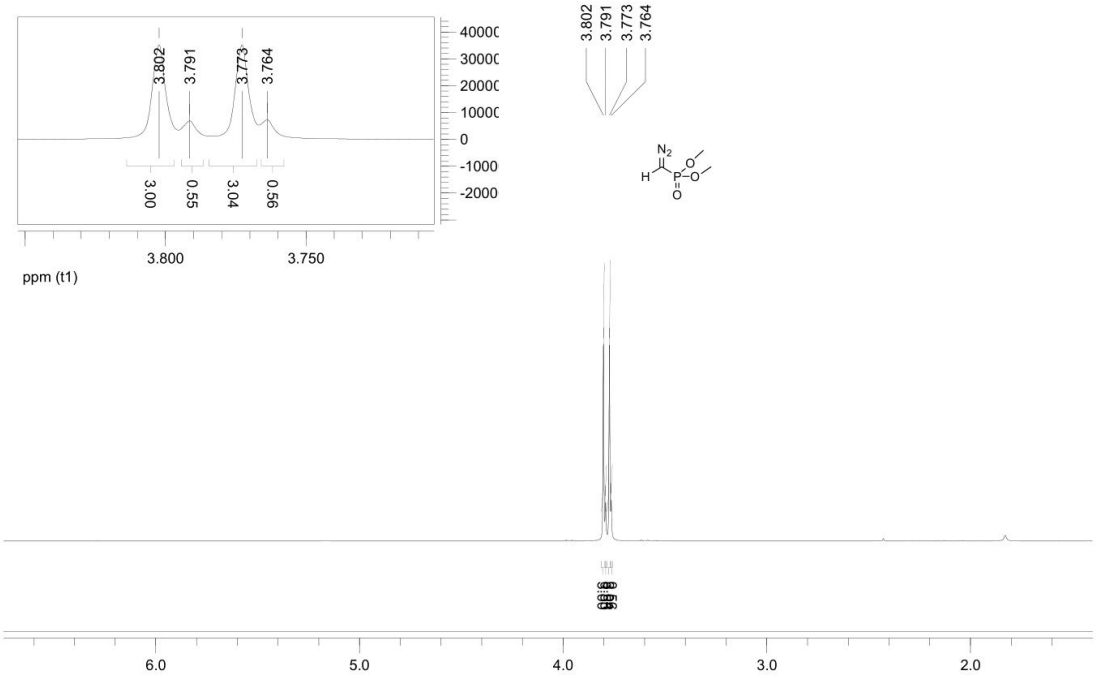


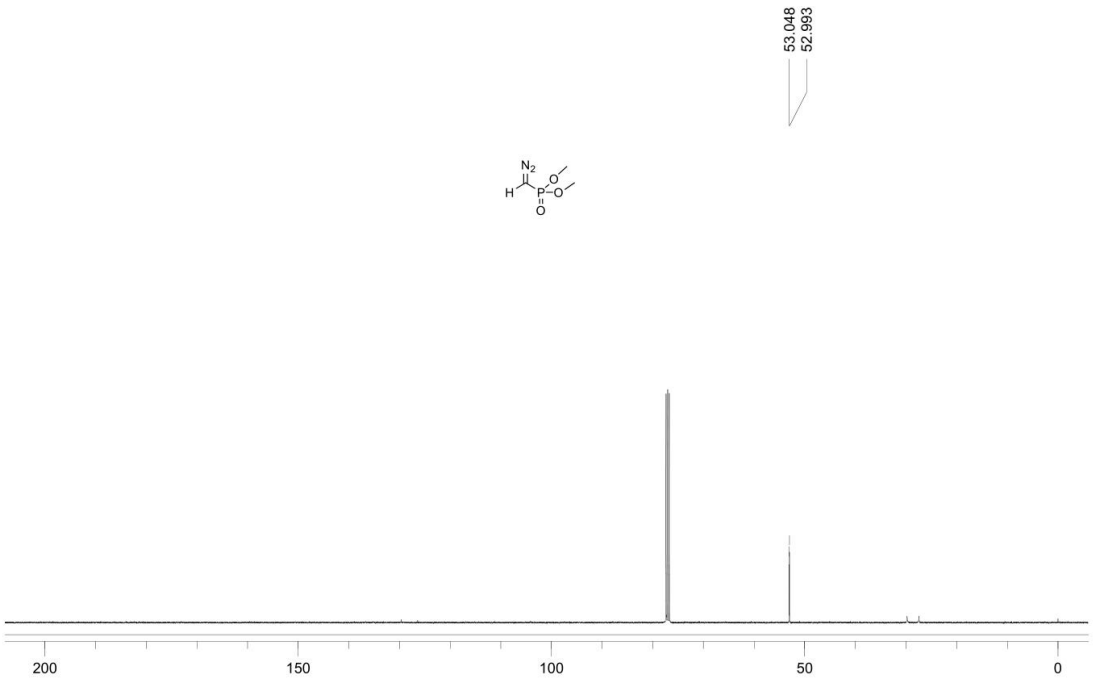
^{1}H NMR and ^{13}C NMR spectrum of the starting material for **2j** and **2l** preparation



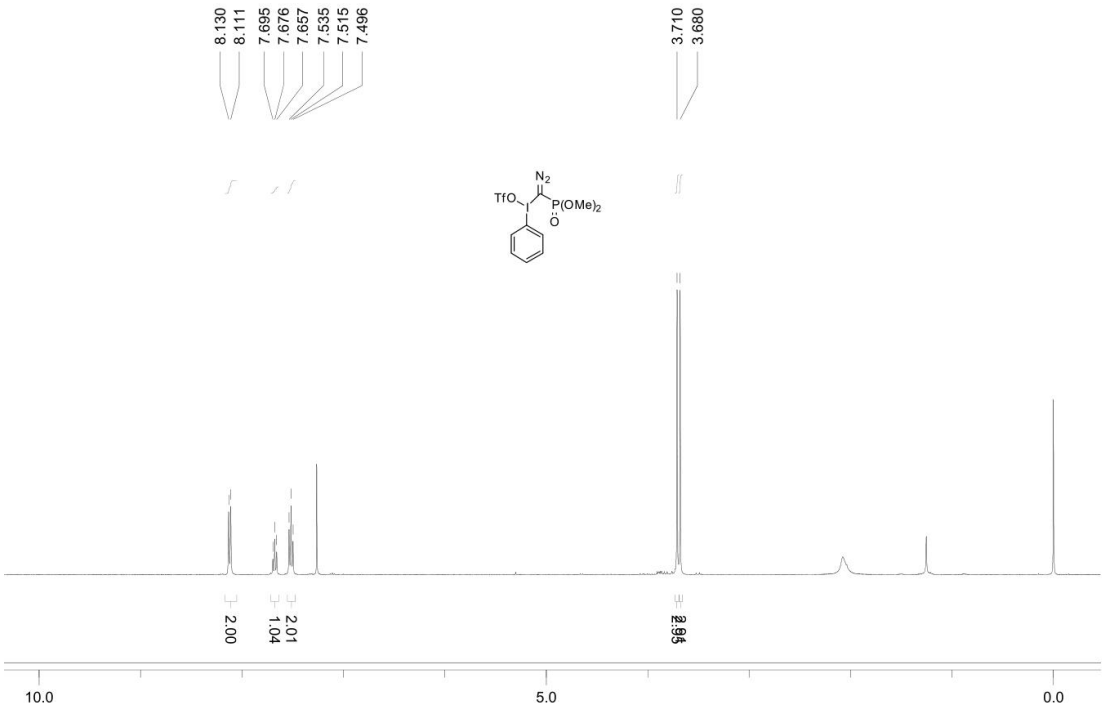


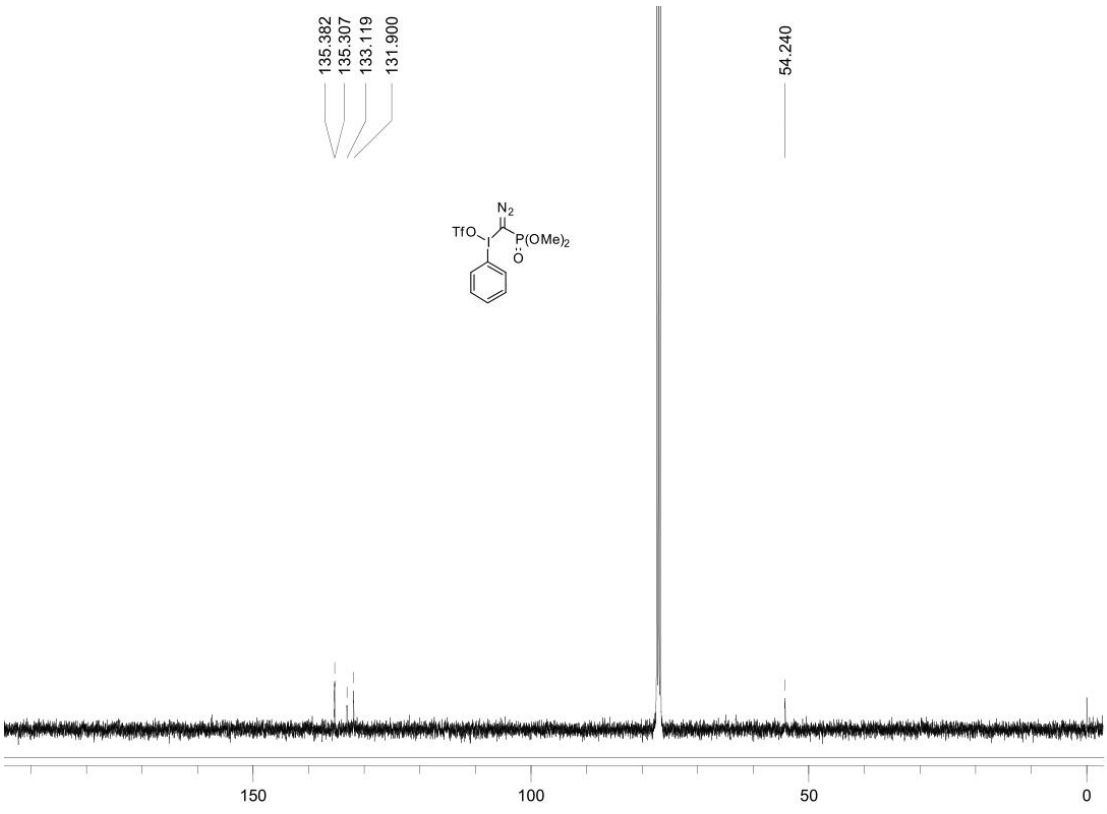
^{1}H NMR and ^{13}C NMR spectrum of the starting material for **2j** and **2l** preparation



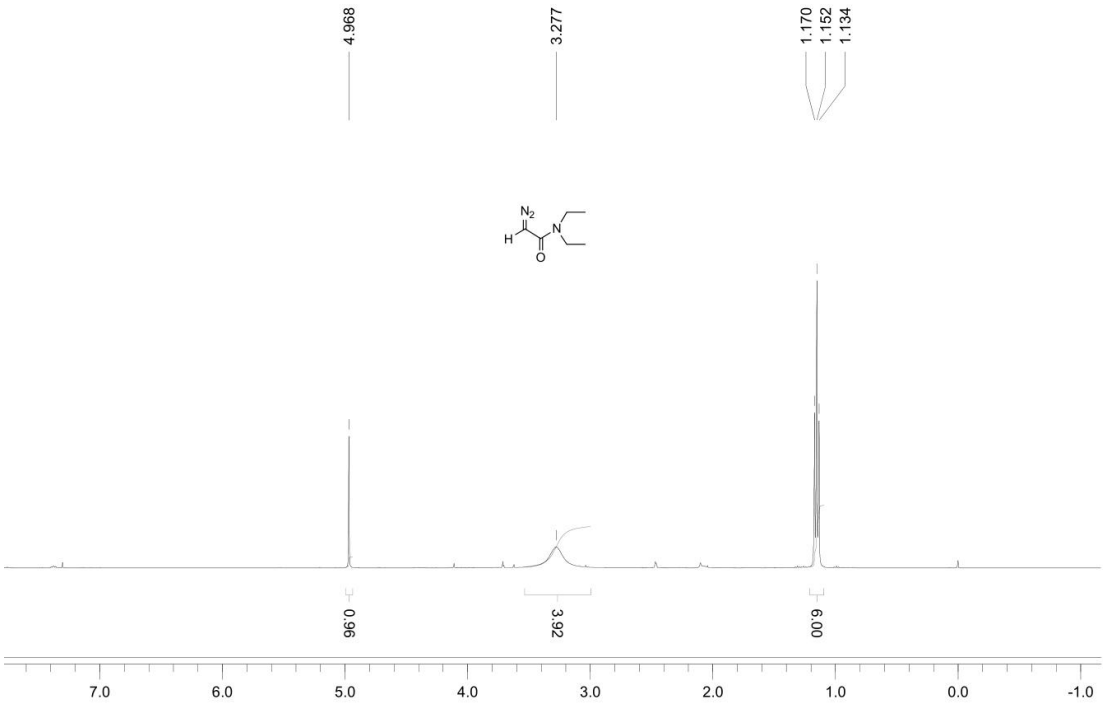


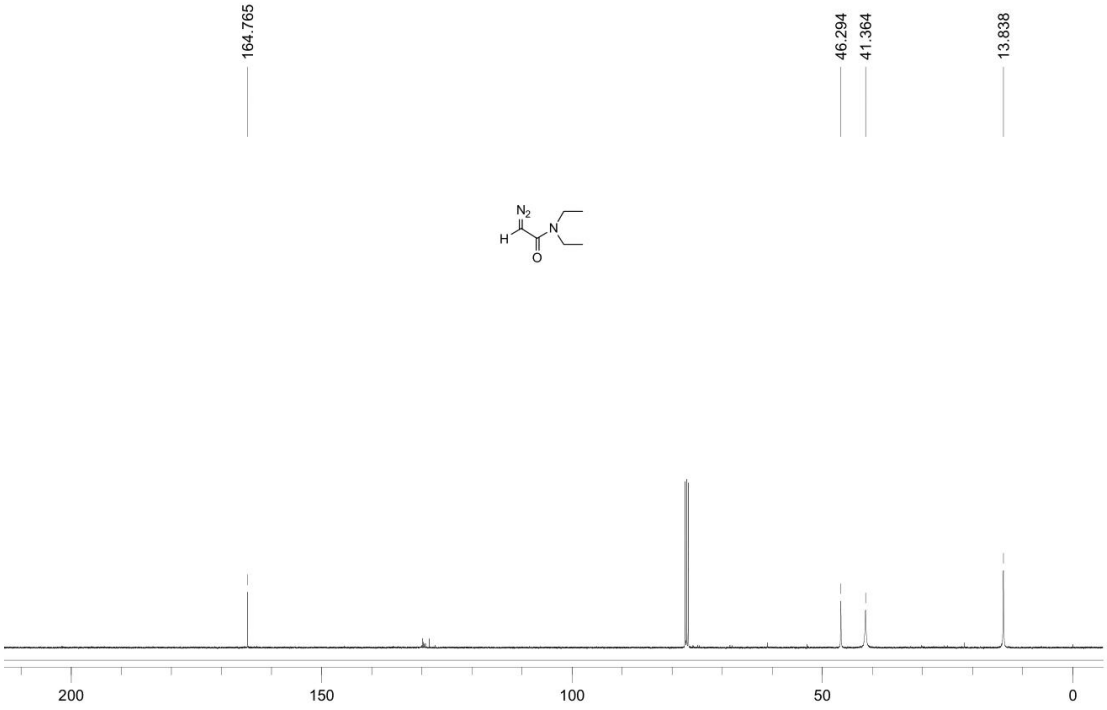
^{1}H NMR and ^{13}C NMR spectrum of **2j**



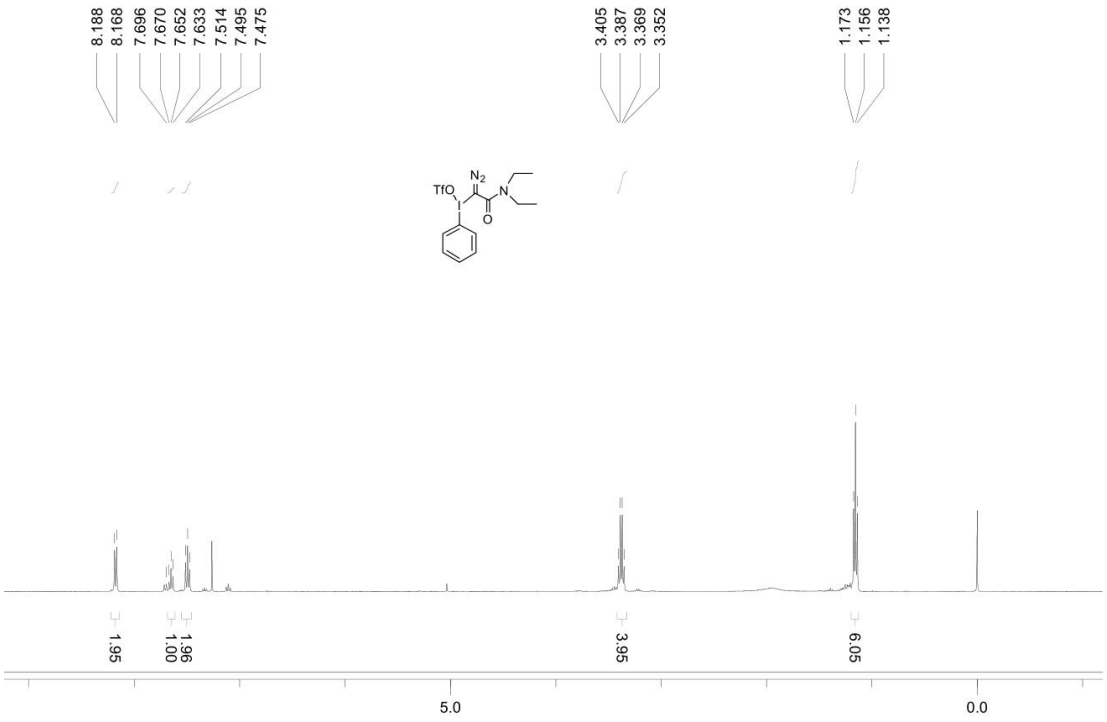


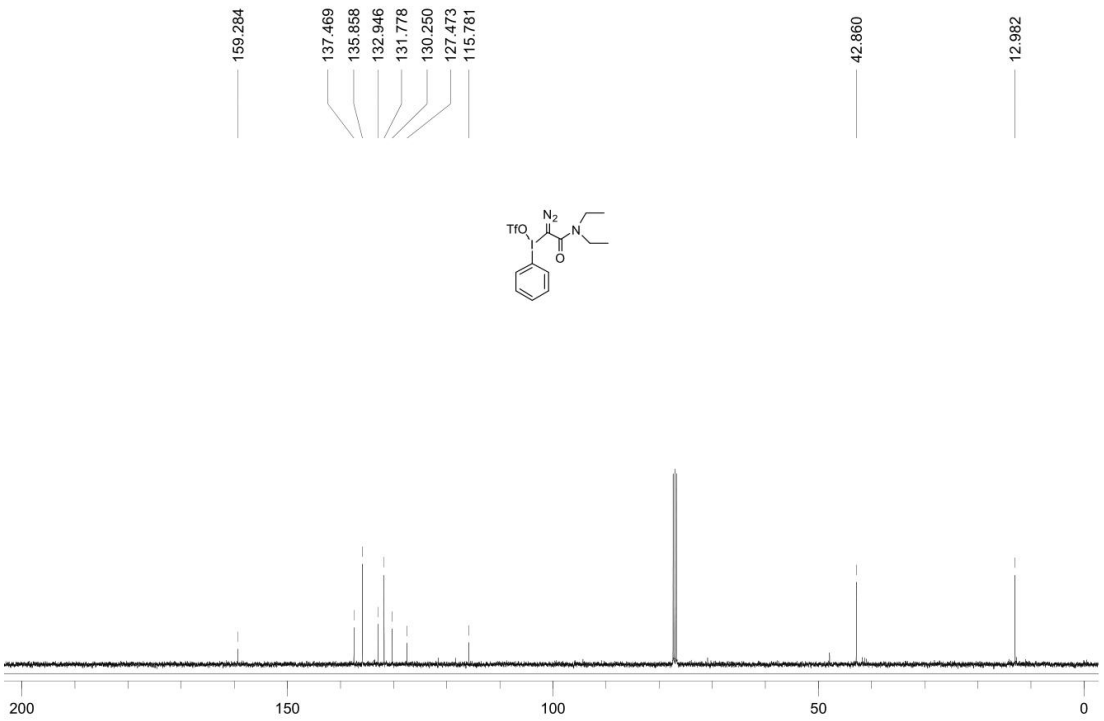
^{1}H NMR and ^{13}C NMR spectrum of the starting material for **2k** preparation



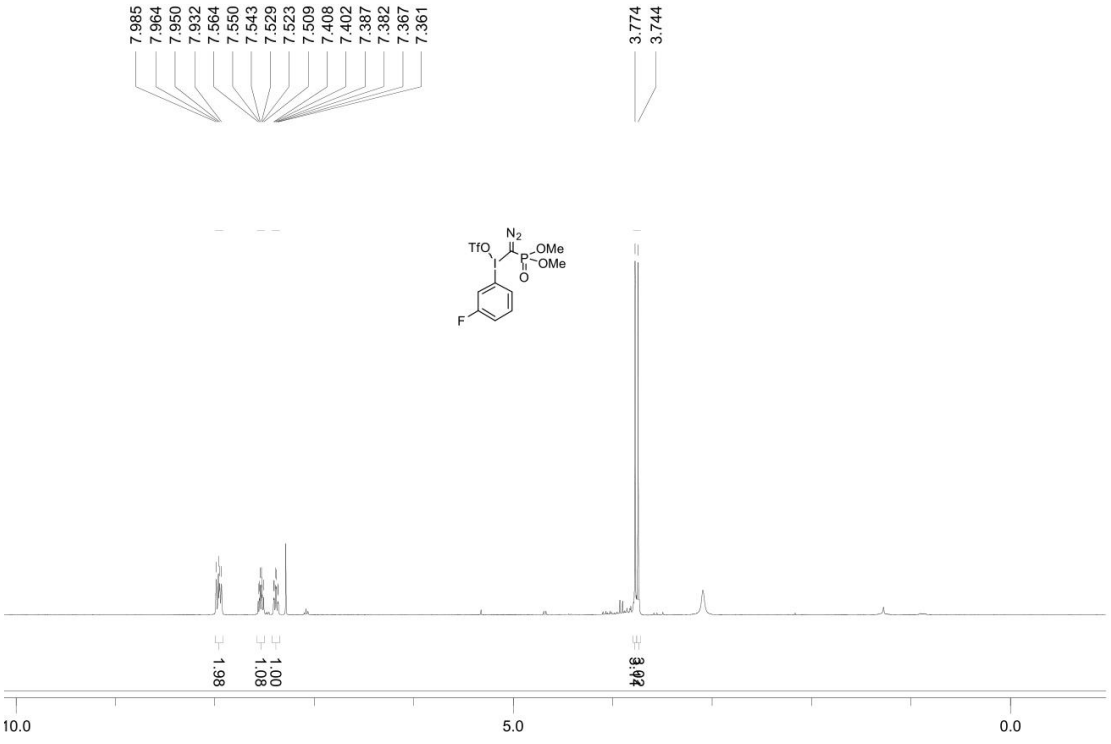


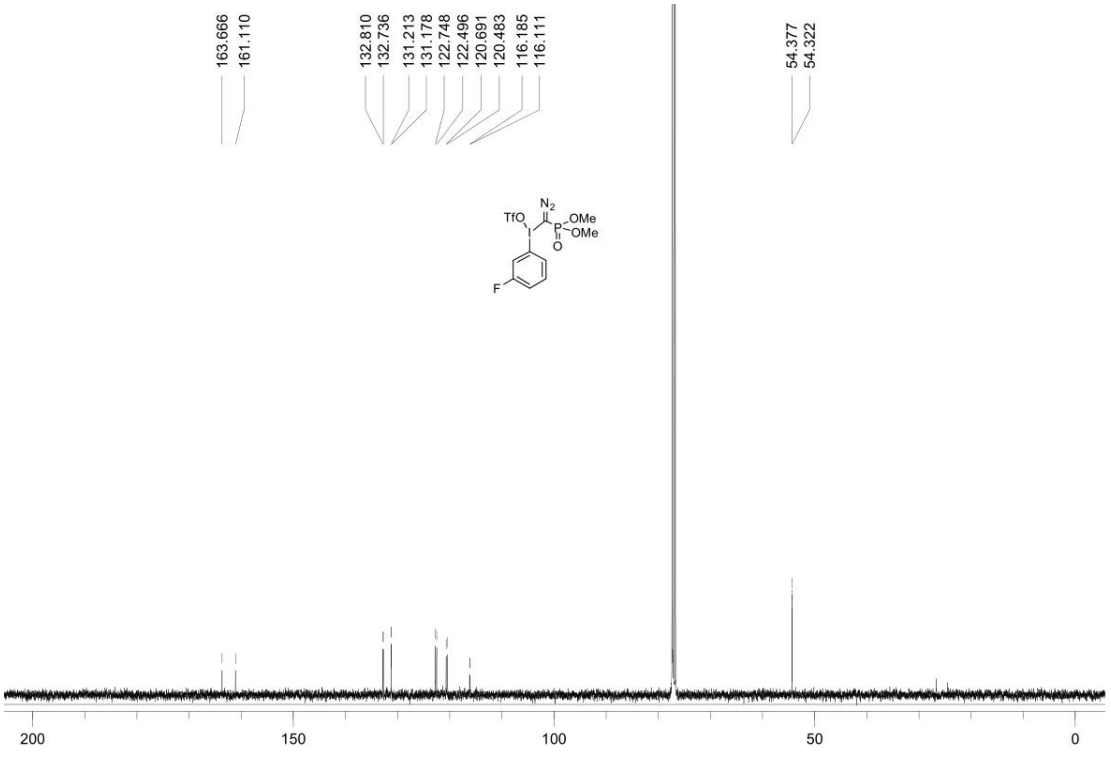
^{1}H NMR and ^{13}C NMR spectrum of **2k**



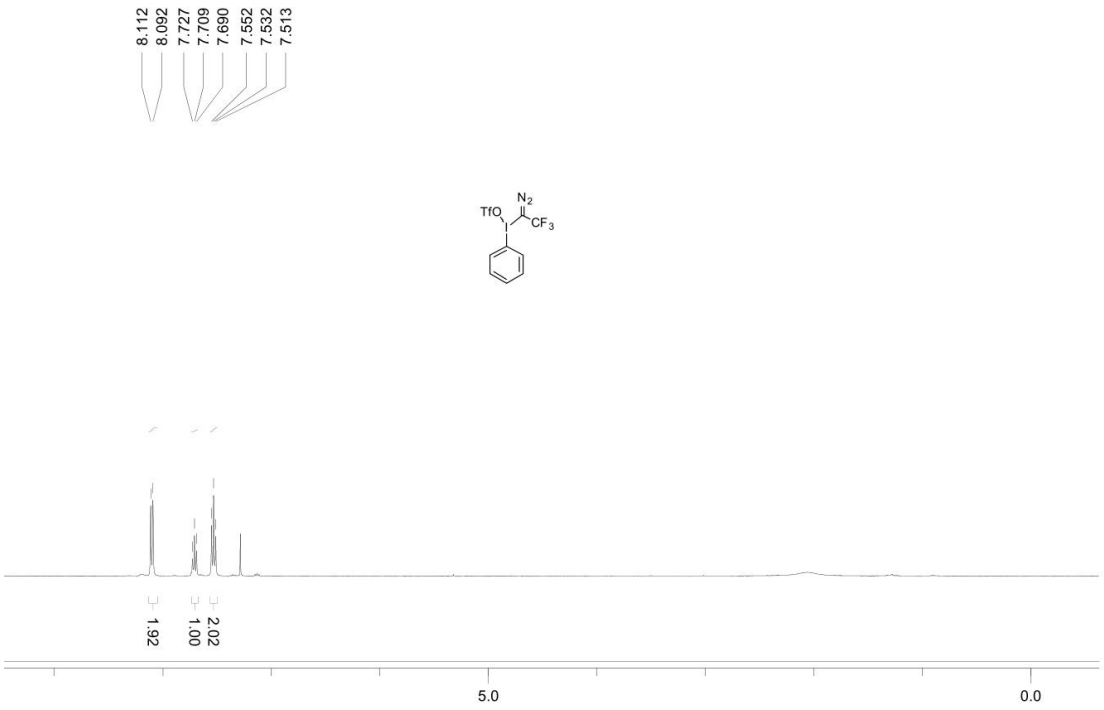


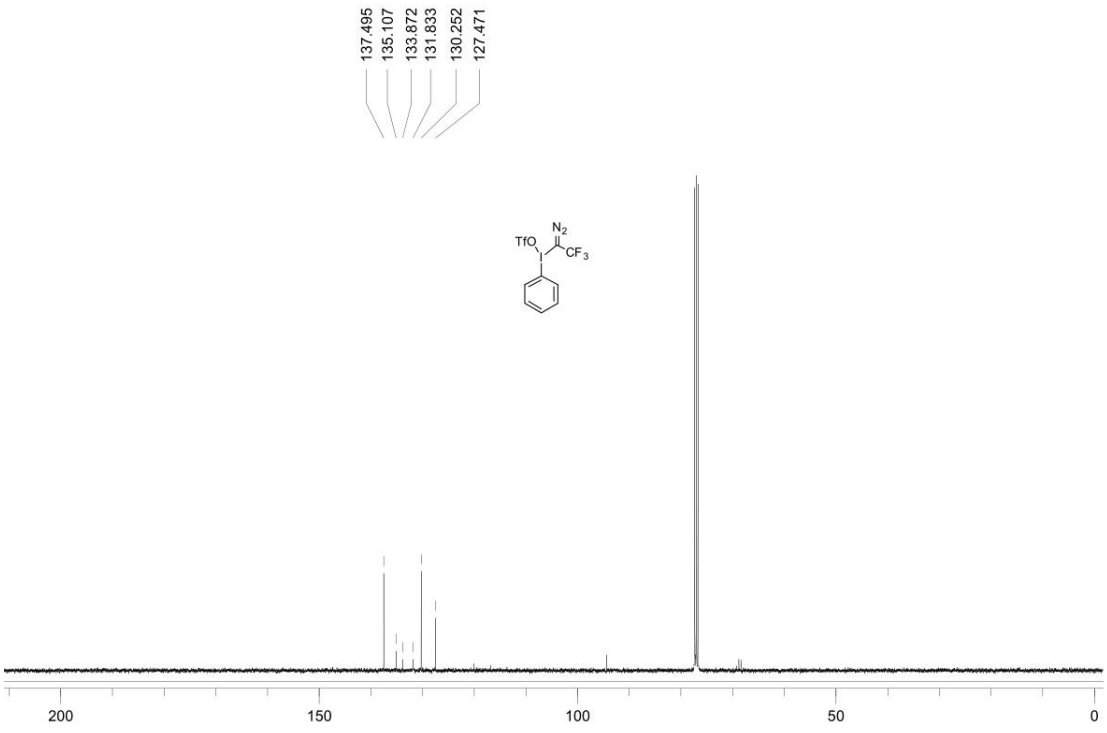
^{1}H NMR and ^{13}C NMR spectrum of **2l**



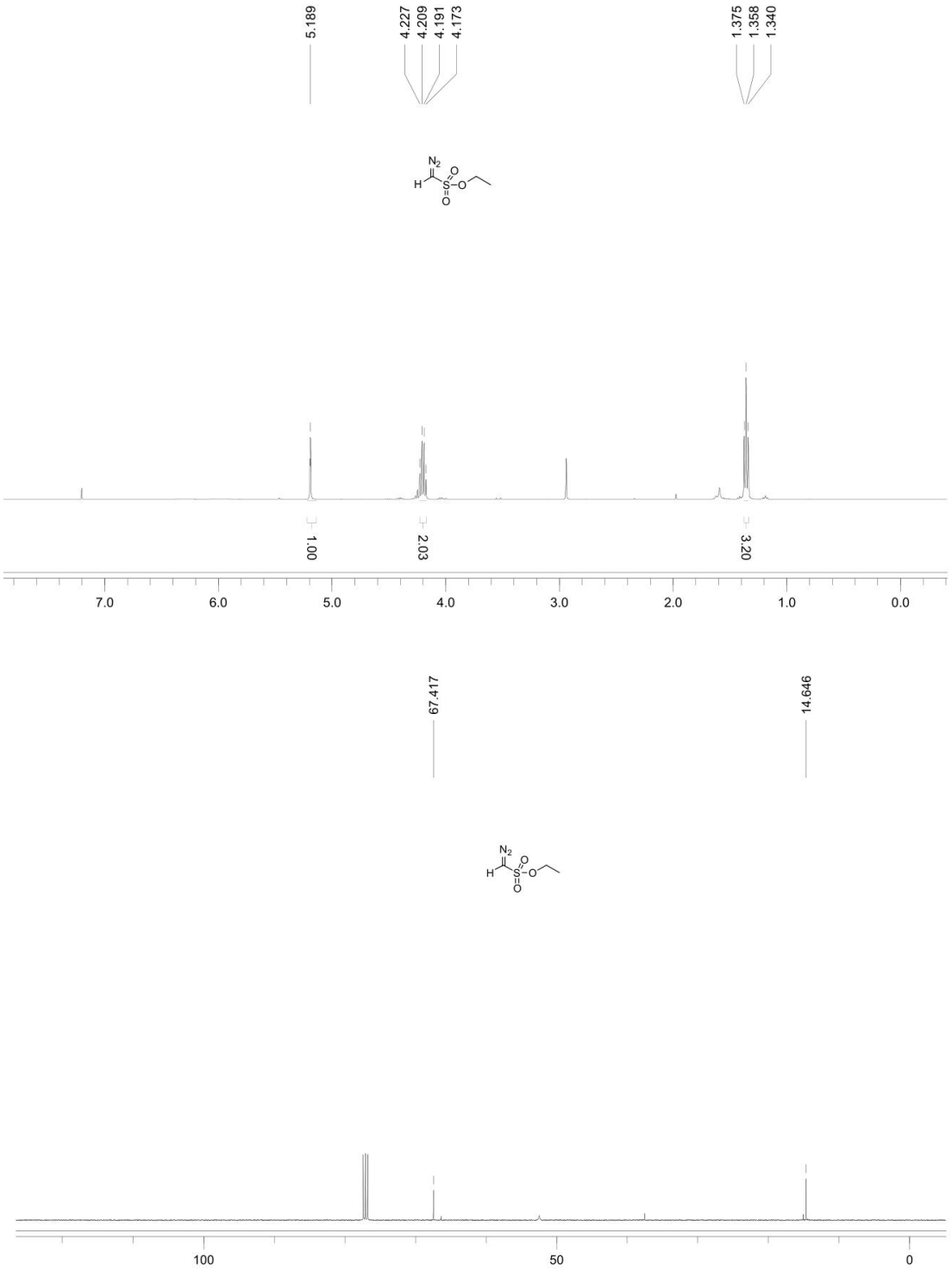


^{1}H NMR and ^{13}C NMR spectrum of **2m**

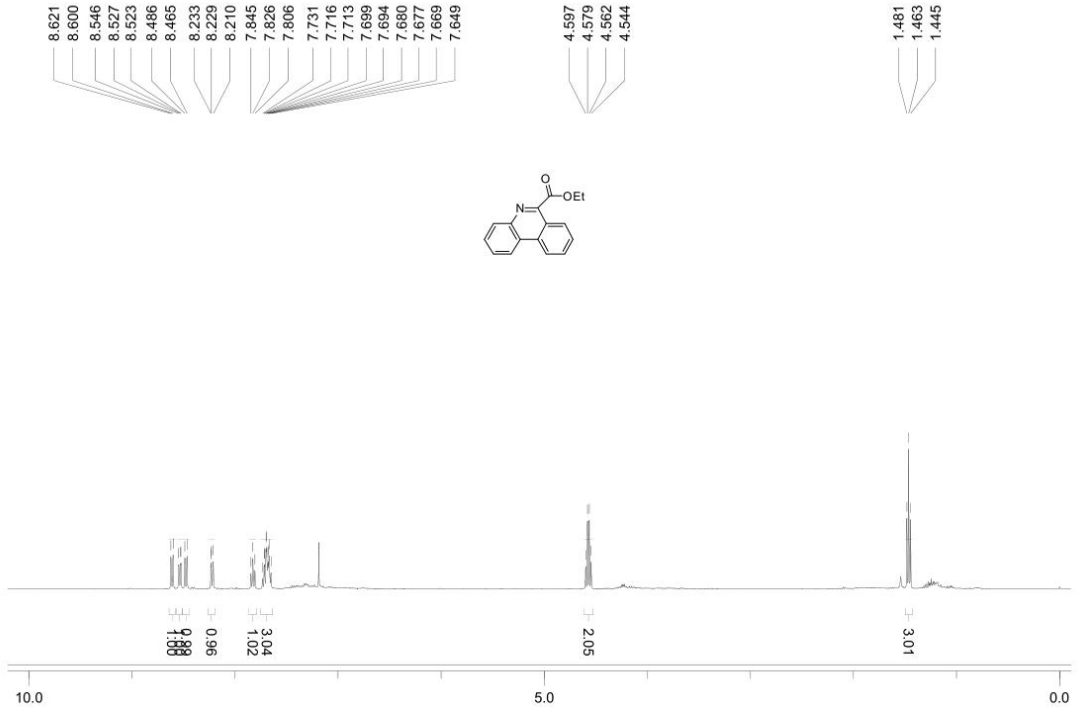


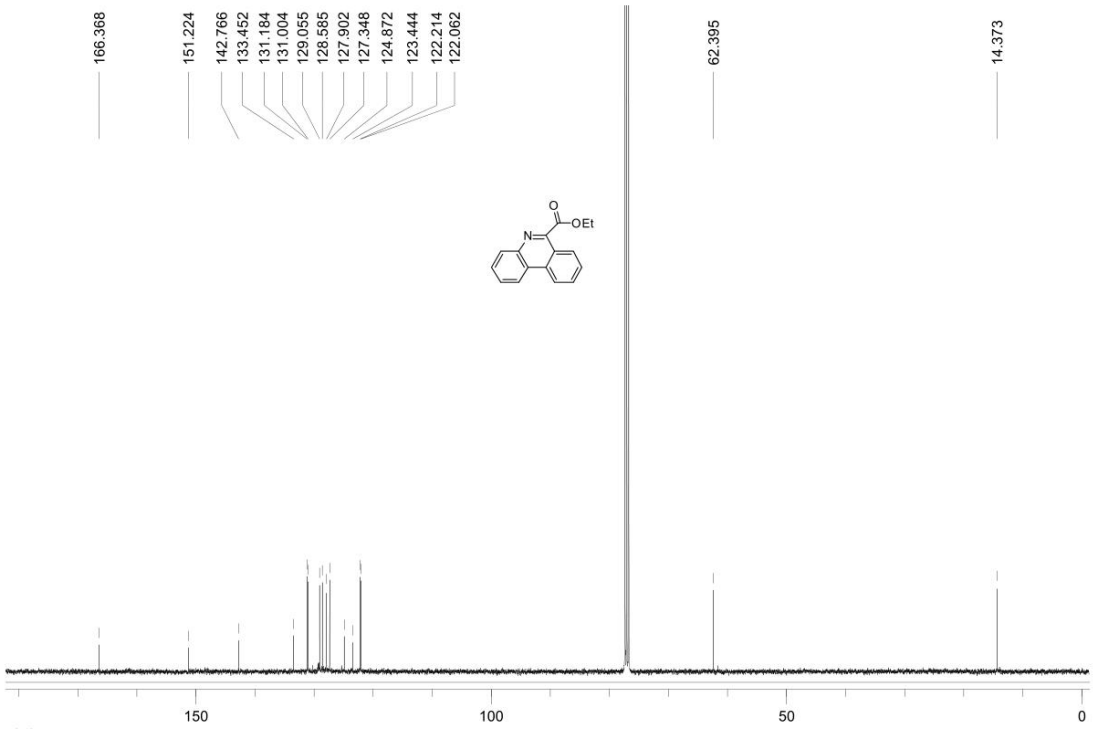


^{1}H NMR and ^{13}C NMR spectrum of Ethyl diazomethanesulfonate

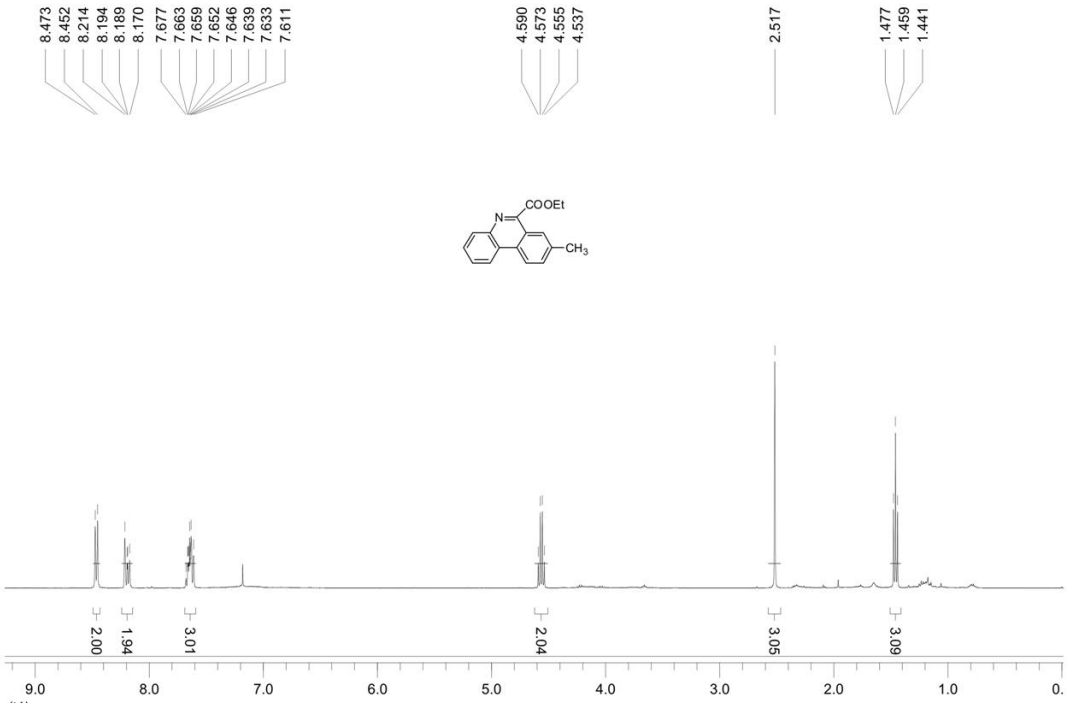


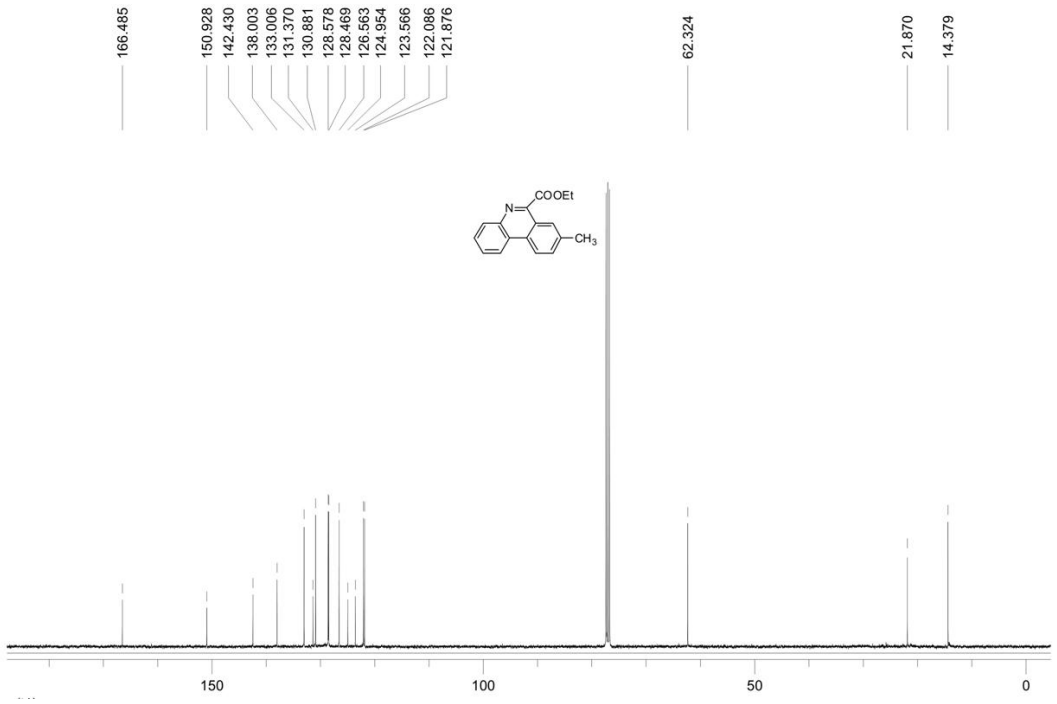
^{1}H NMR and ^{13}C NMR spectrum of **3a**



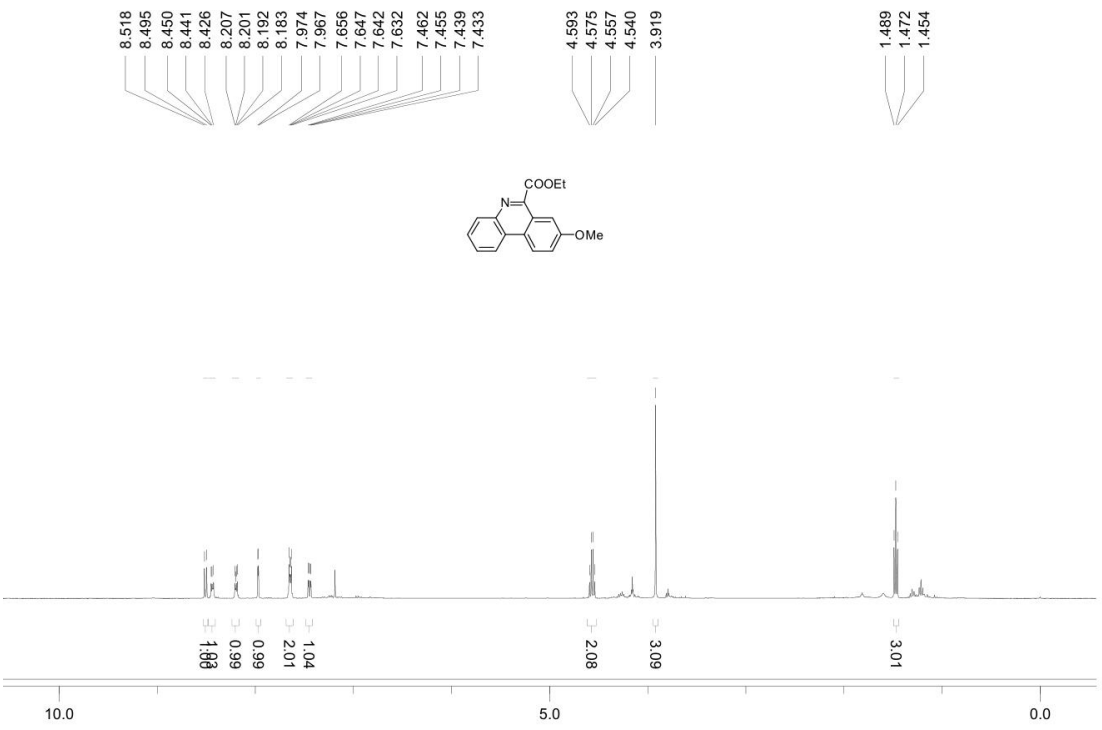


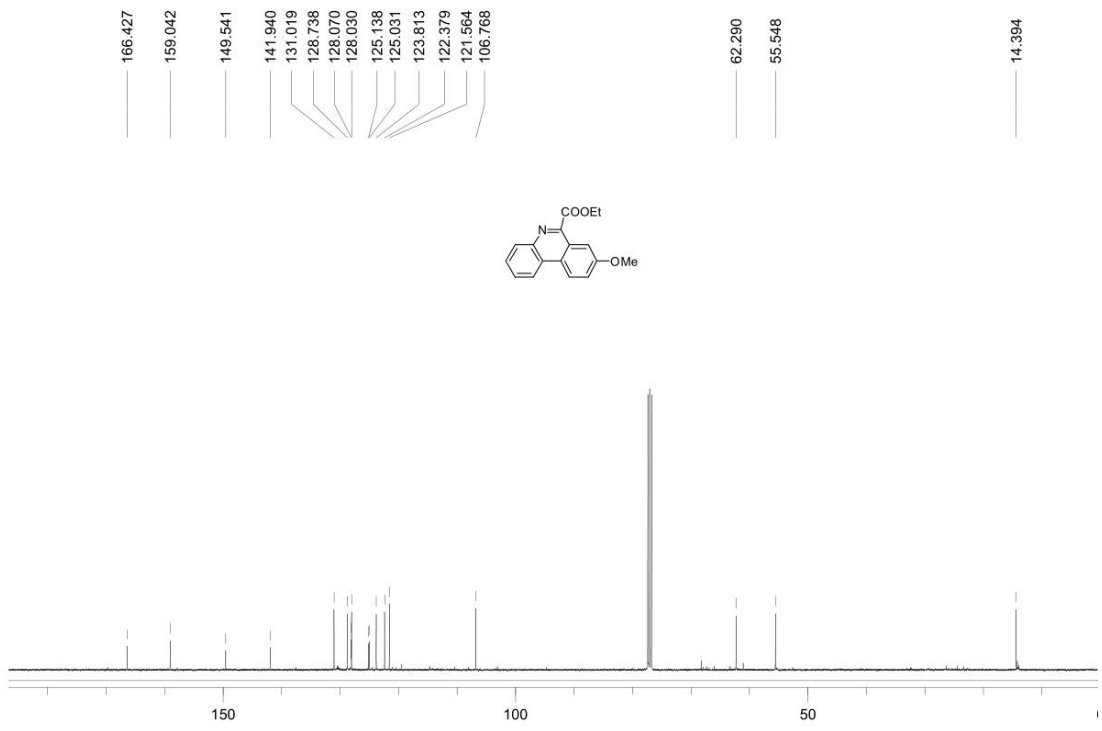
^{1}H NMR and ^{13}C NMR spectrum of **3b**



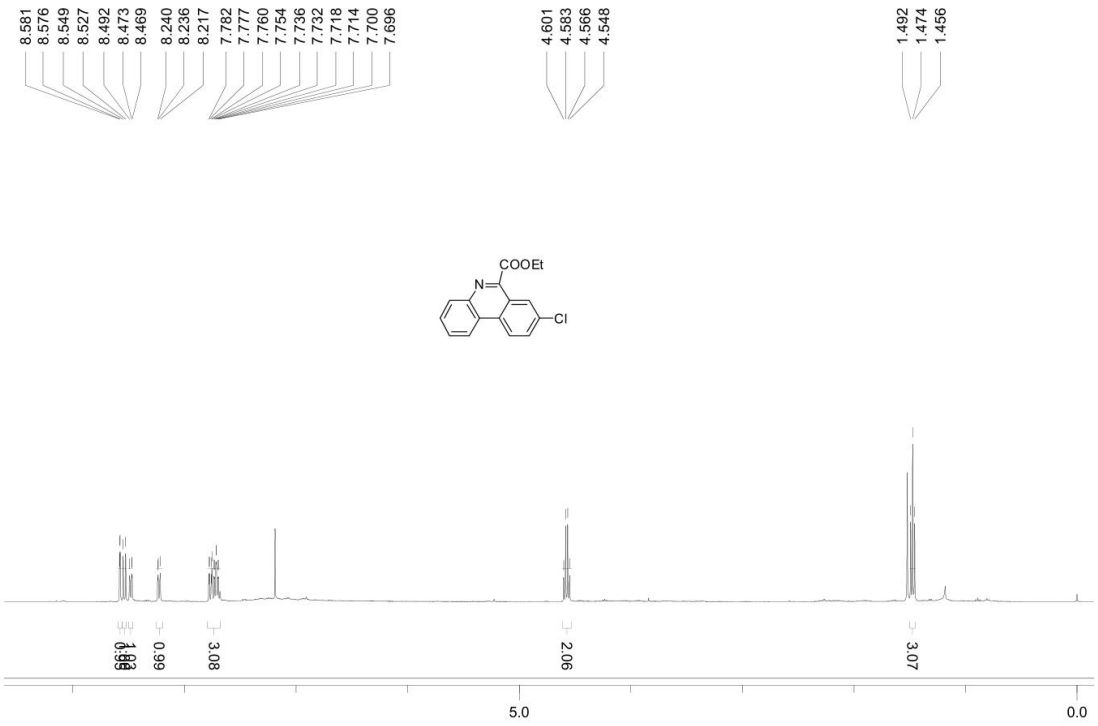


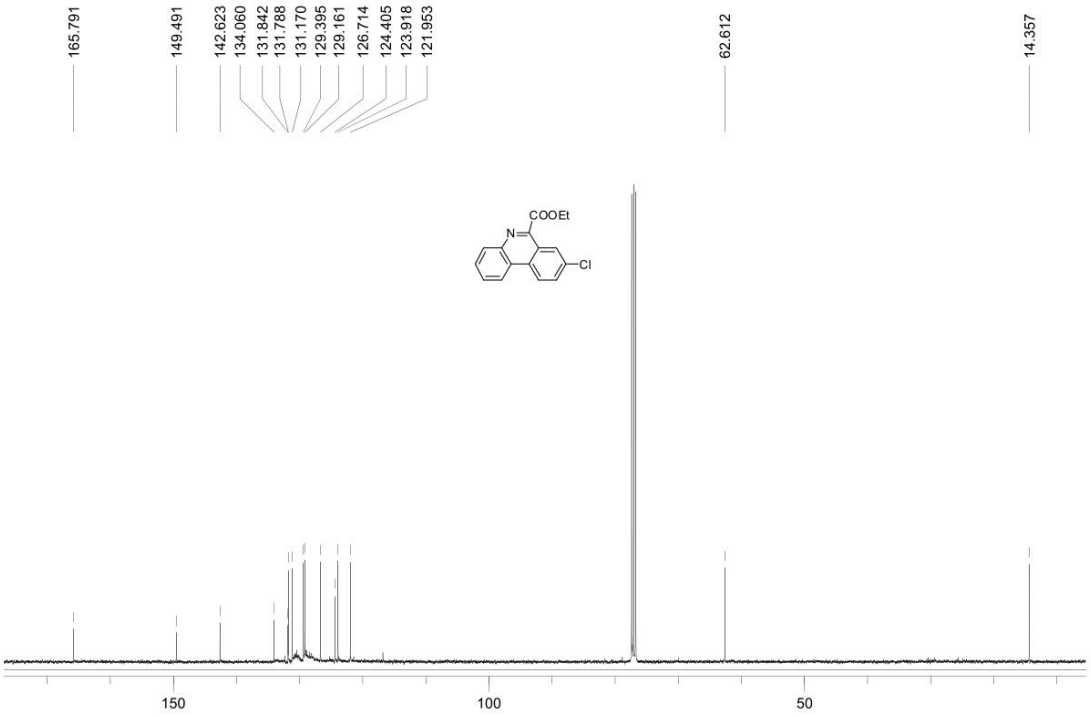
^{1}H NMR and ^{13}C NMR spectrum of **3c**



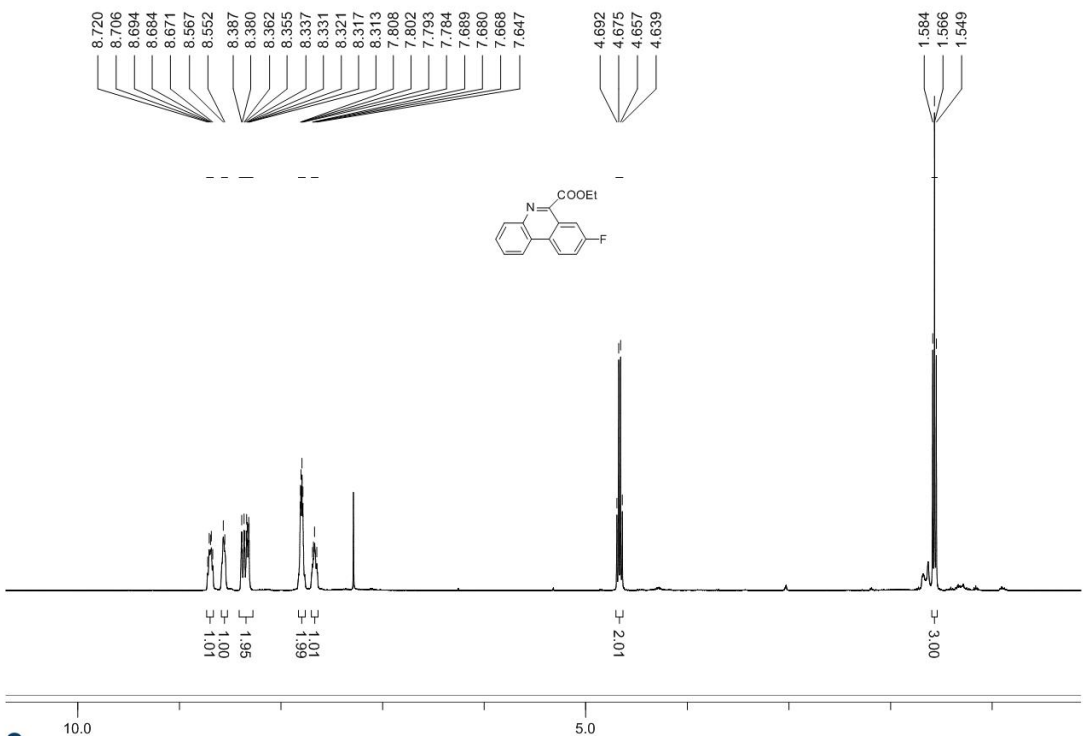


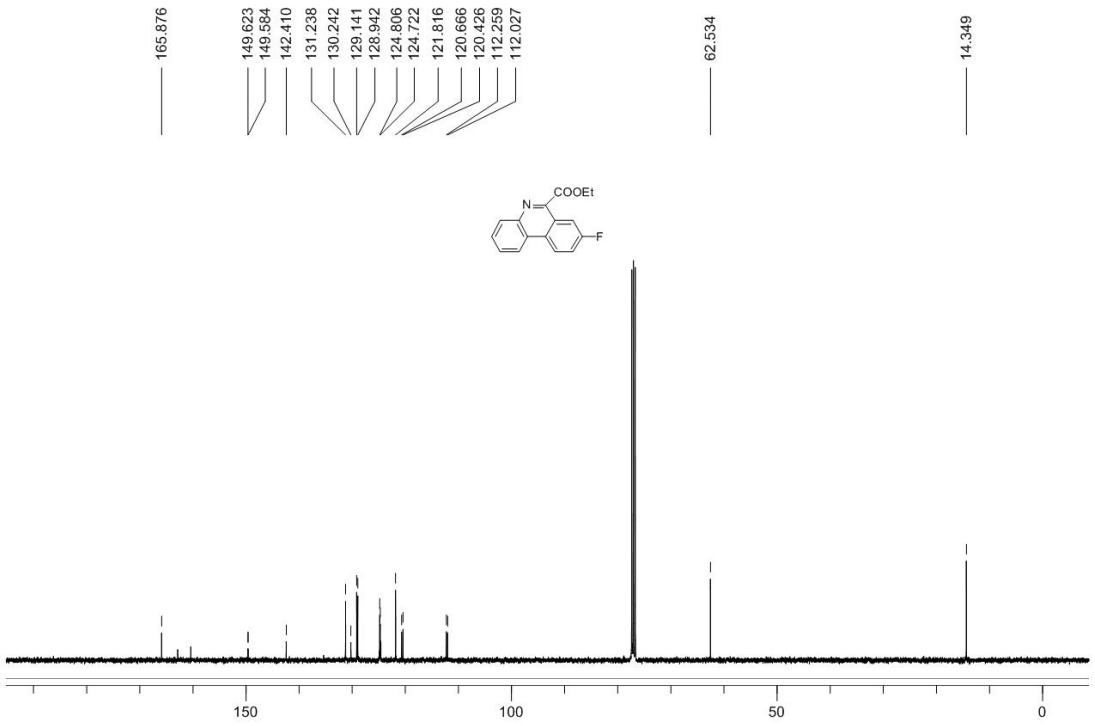
^{1}H NMR and ^{13}C NMR spectrum of **3d**



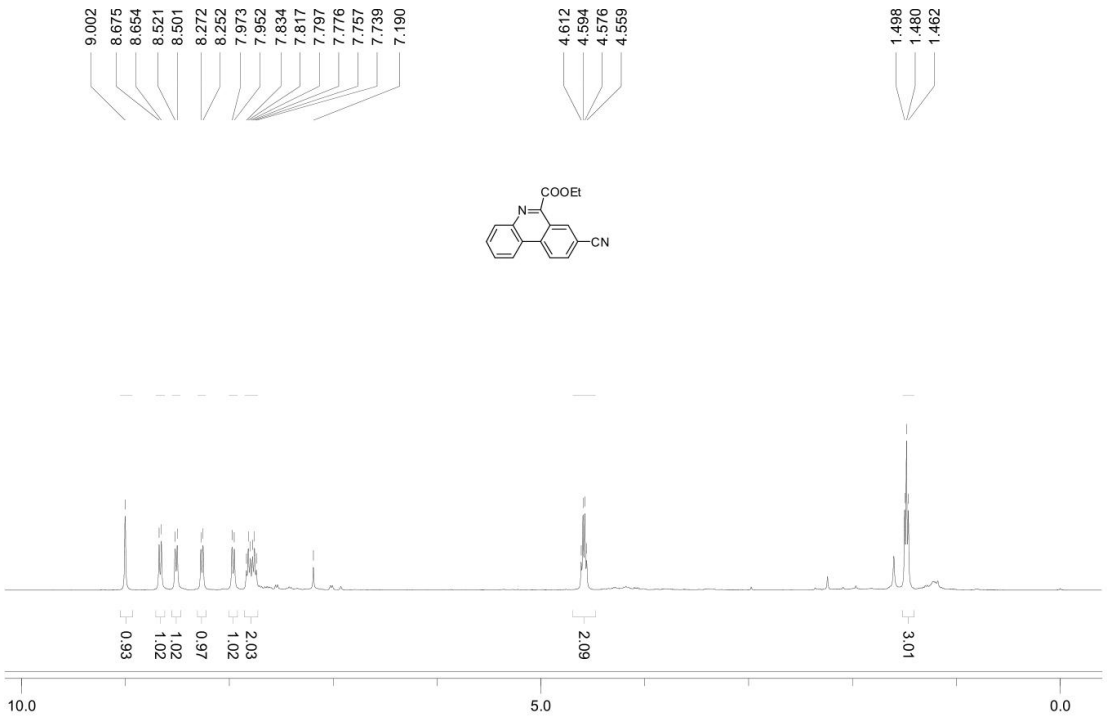


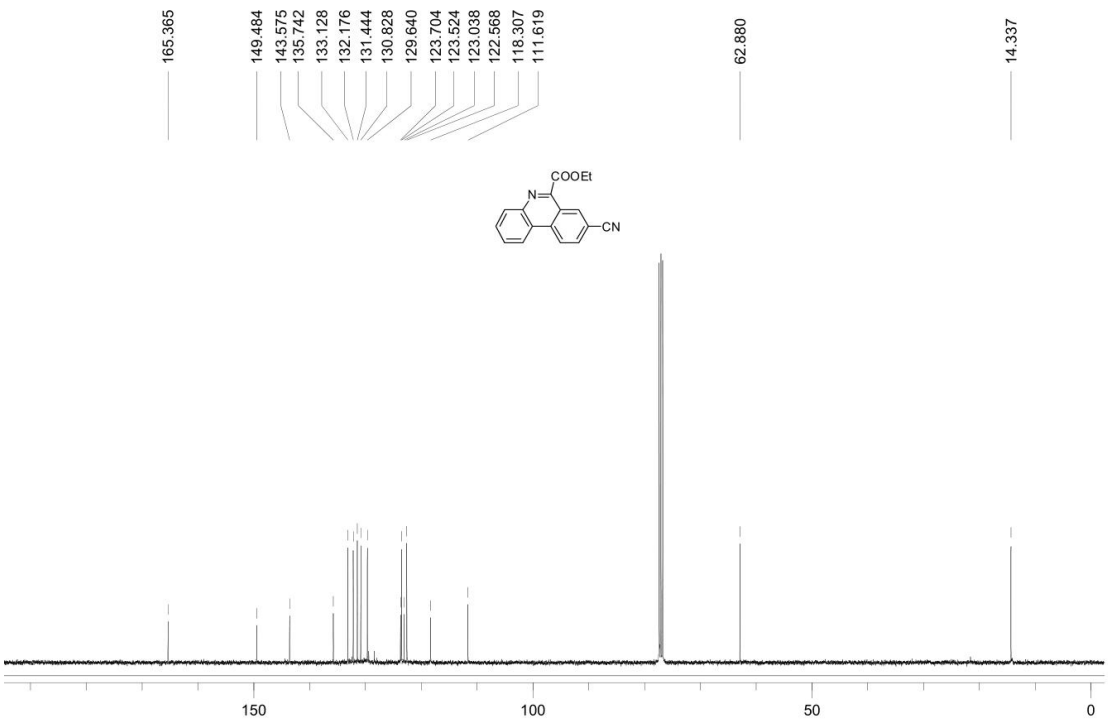
^{1}H NMR and ^{13}C NMR spectrum of **3e**



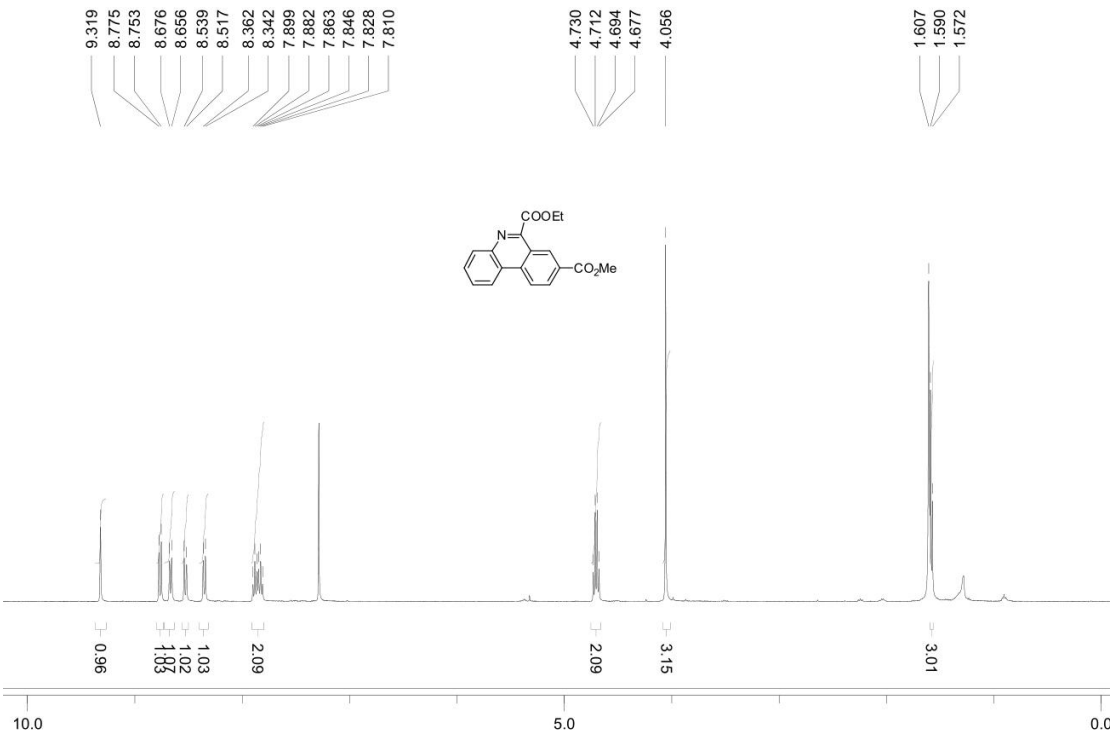


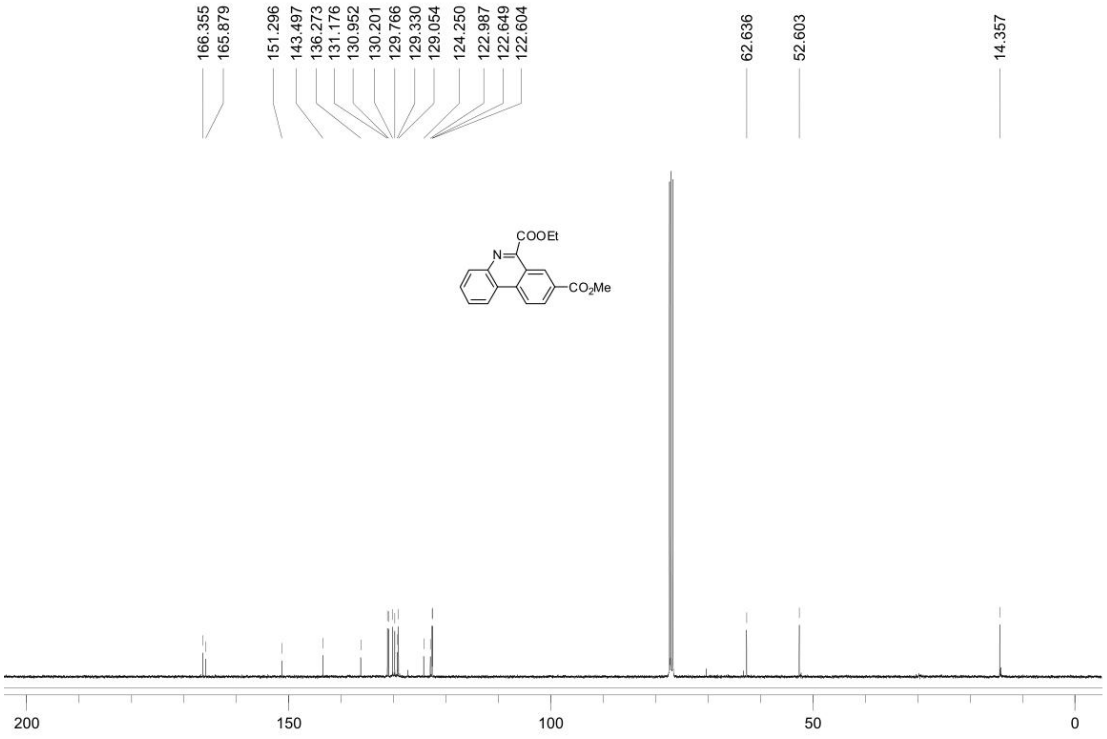
^{1}H NMR and ^{13}C NMR spectrum of **3f**



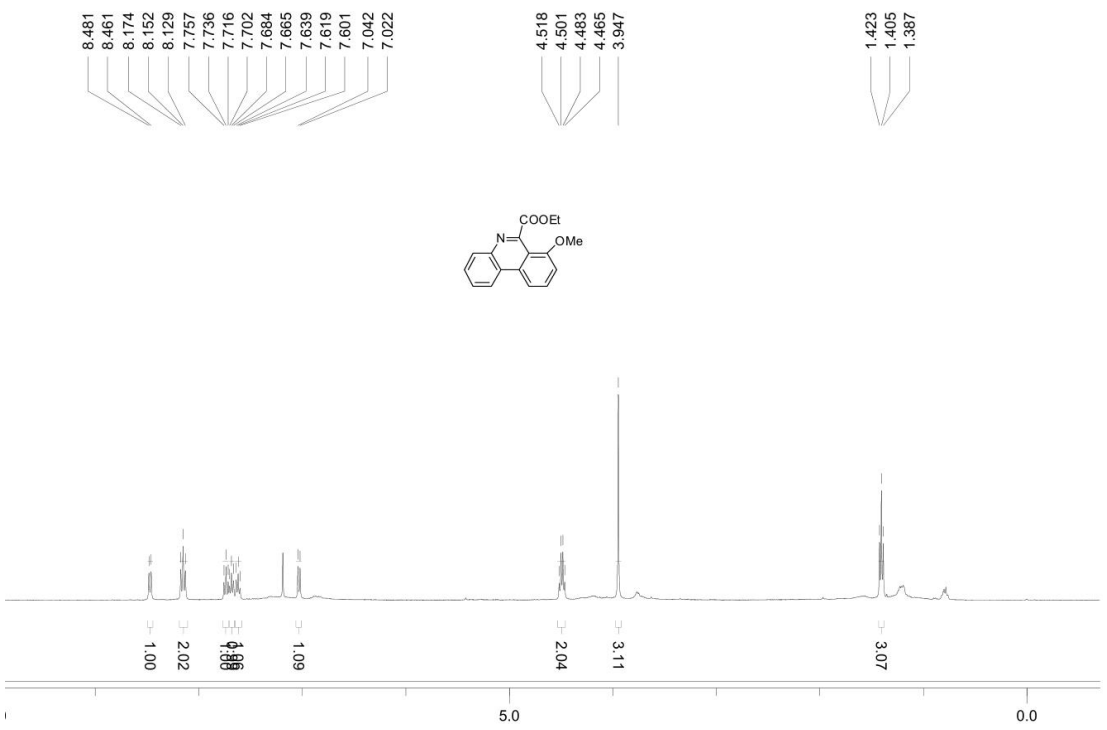


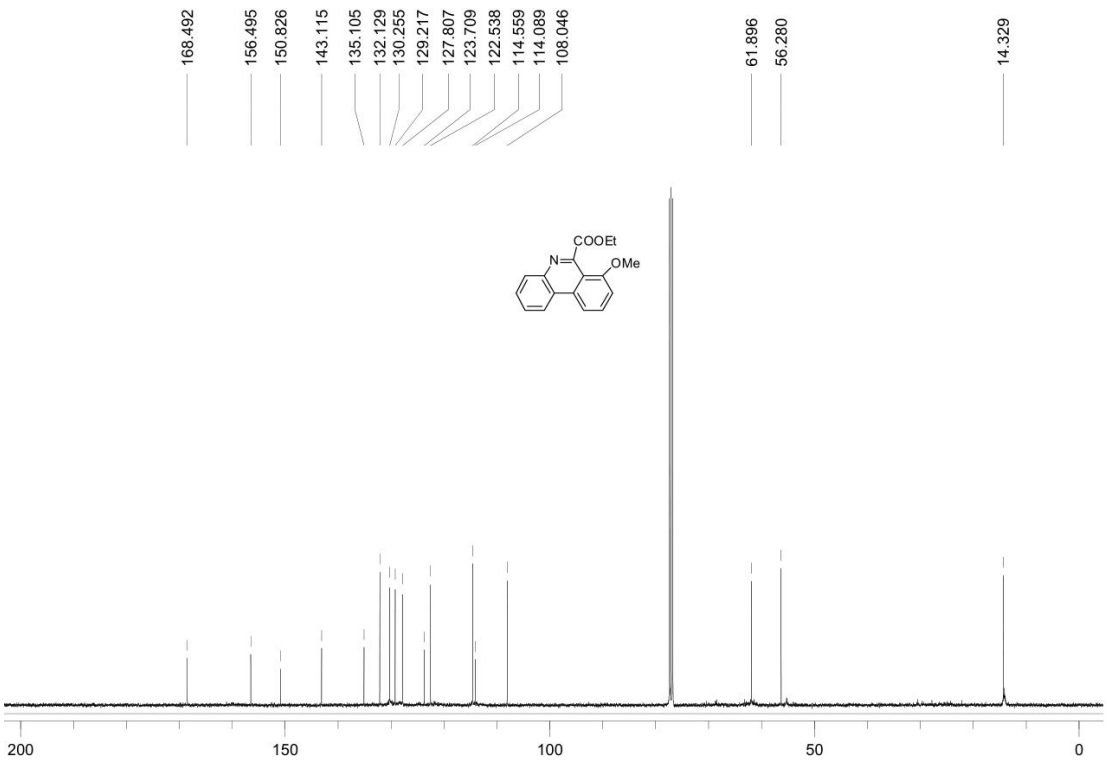
^{1}H NMR and ^{13}C NMR spectrum of **3g**



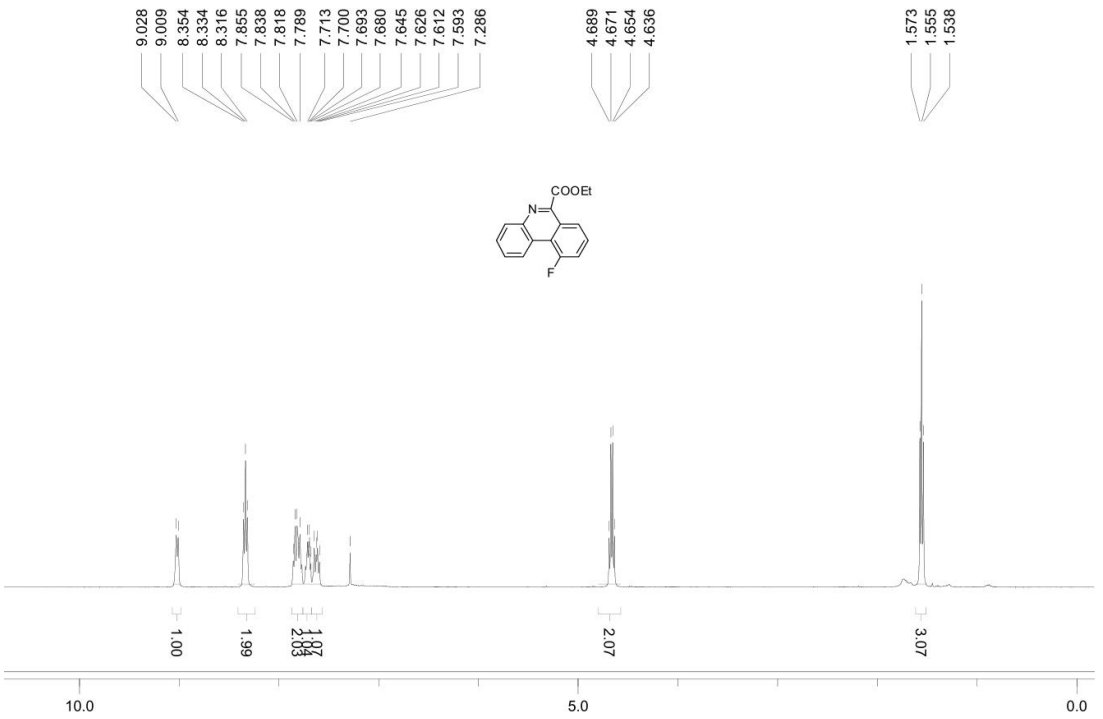


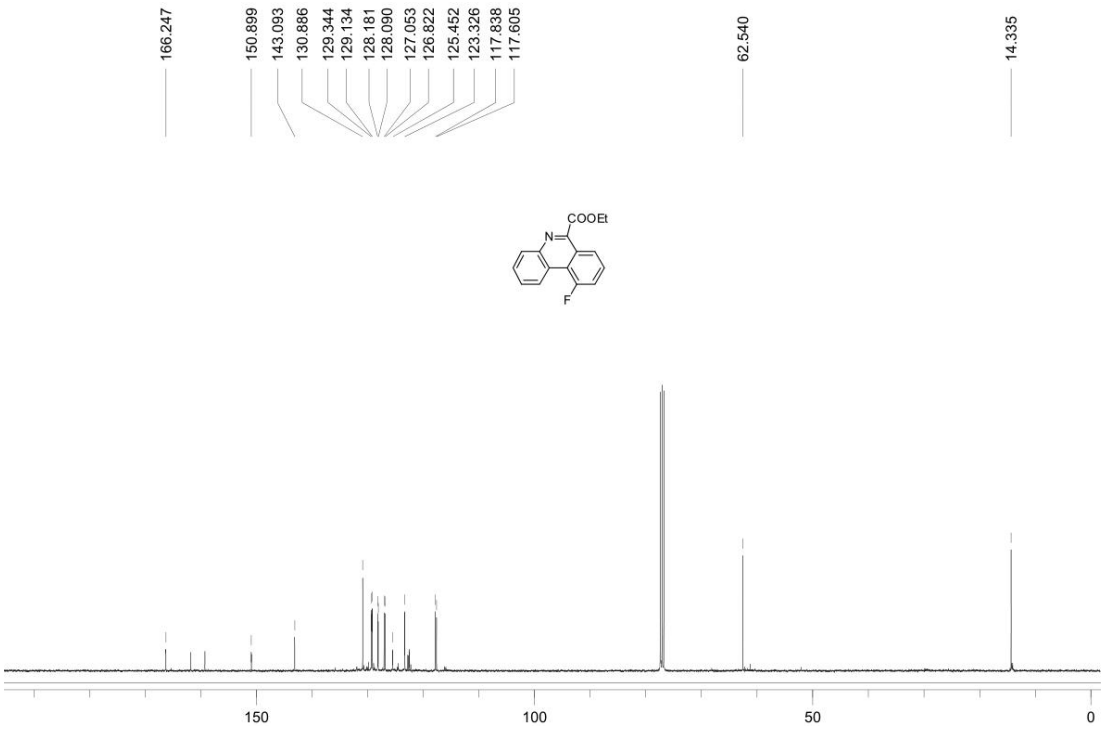
^{1}H NMR and ^{13}C NMR spectrum of **3h**



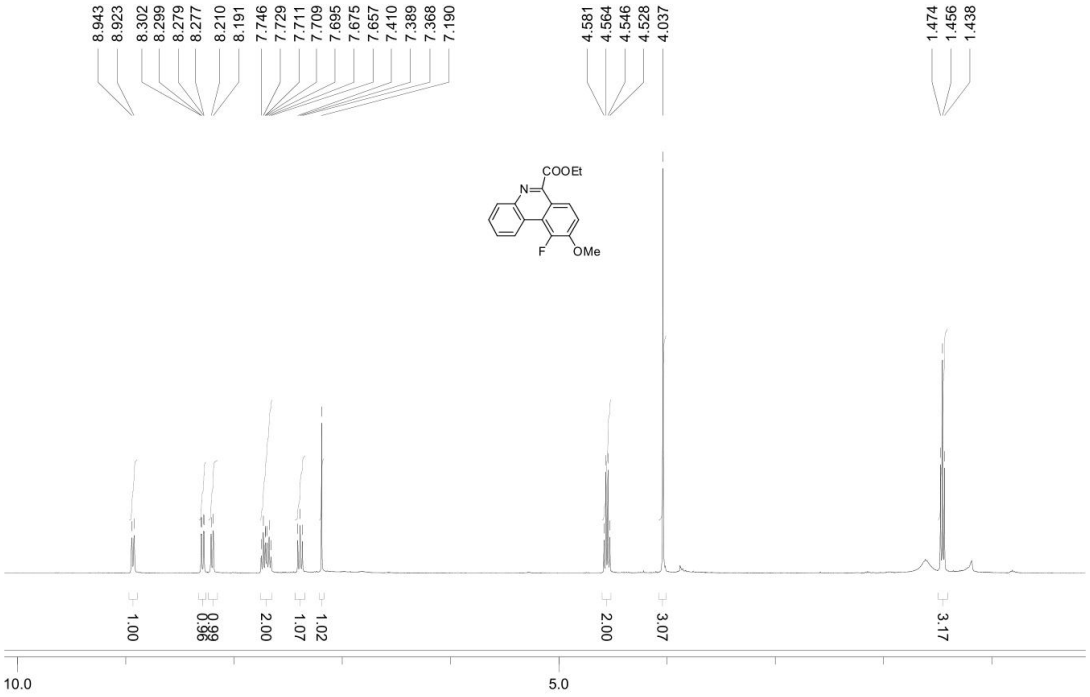


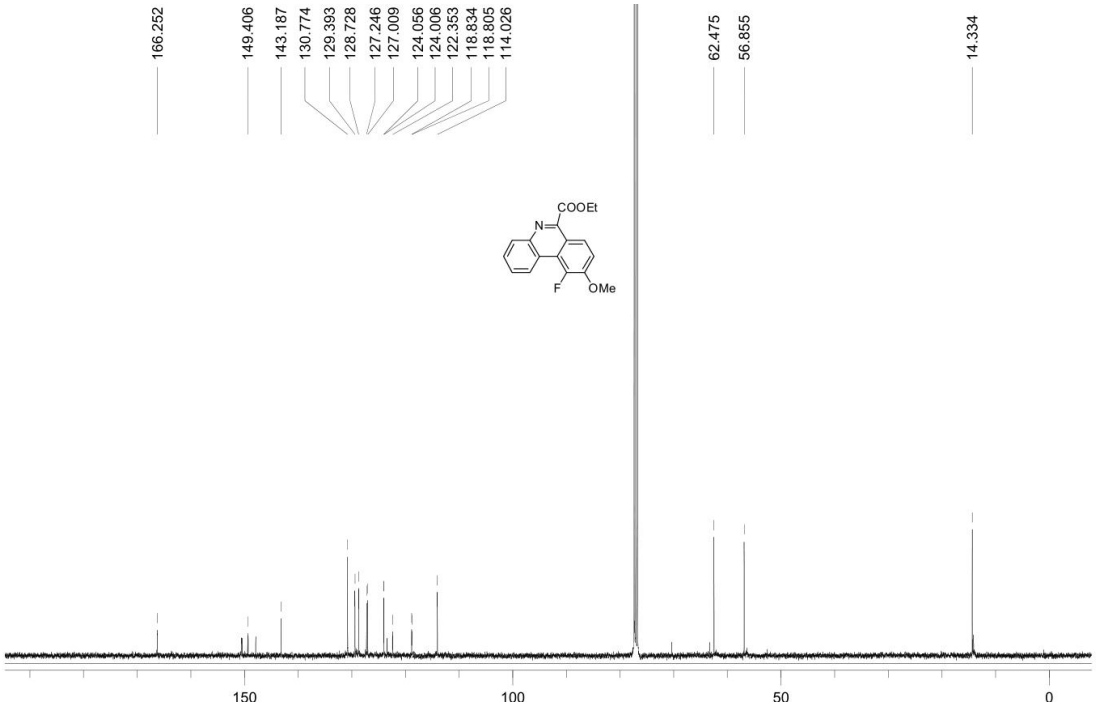
^{1}H NMR and ^{13}C NMR spectrum of **3i**



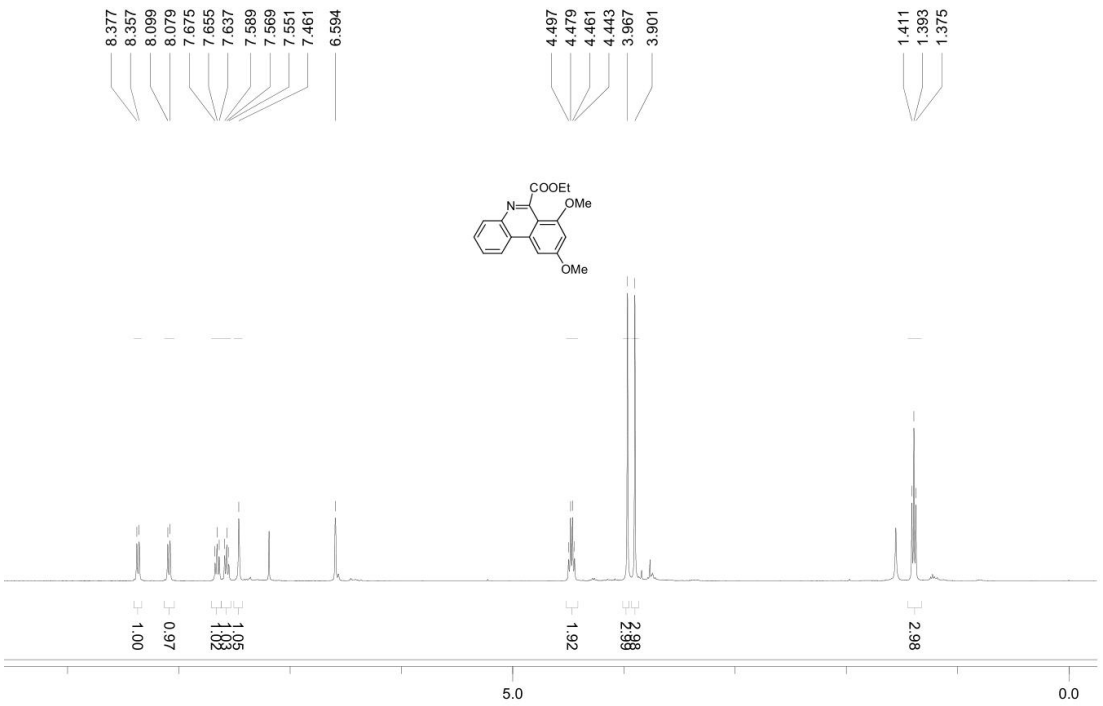


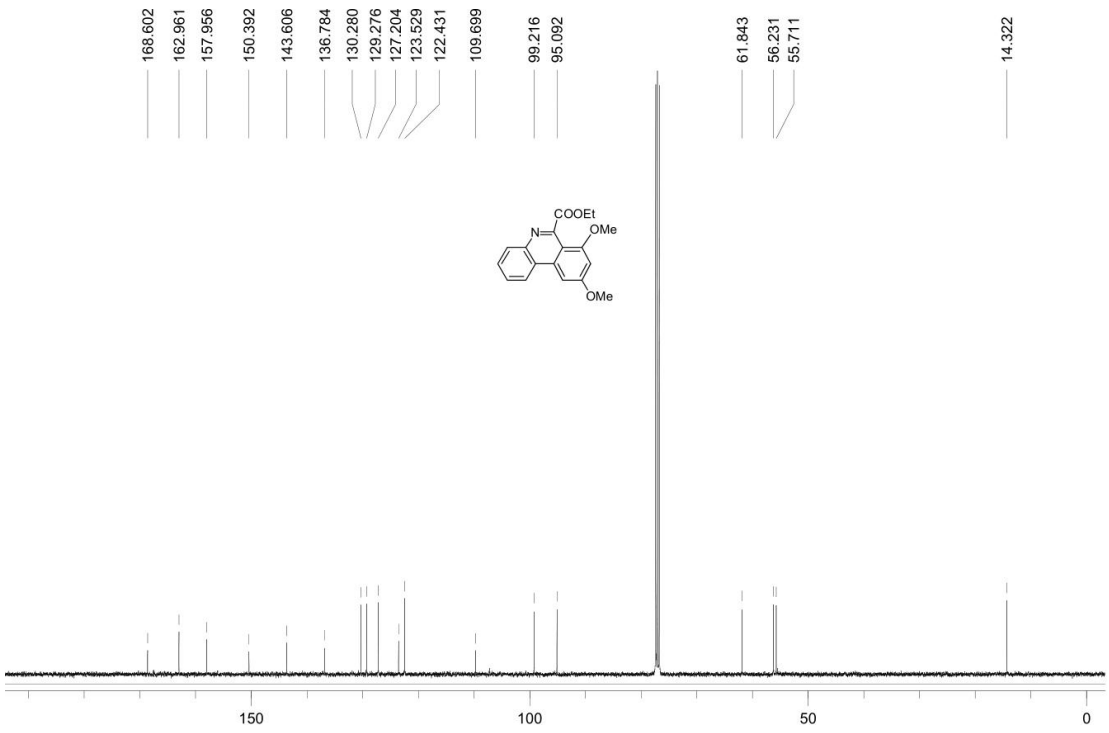
^{1}H NMR and ^{13}C NMR spectrum of **3j**



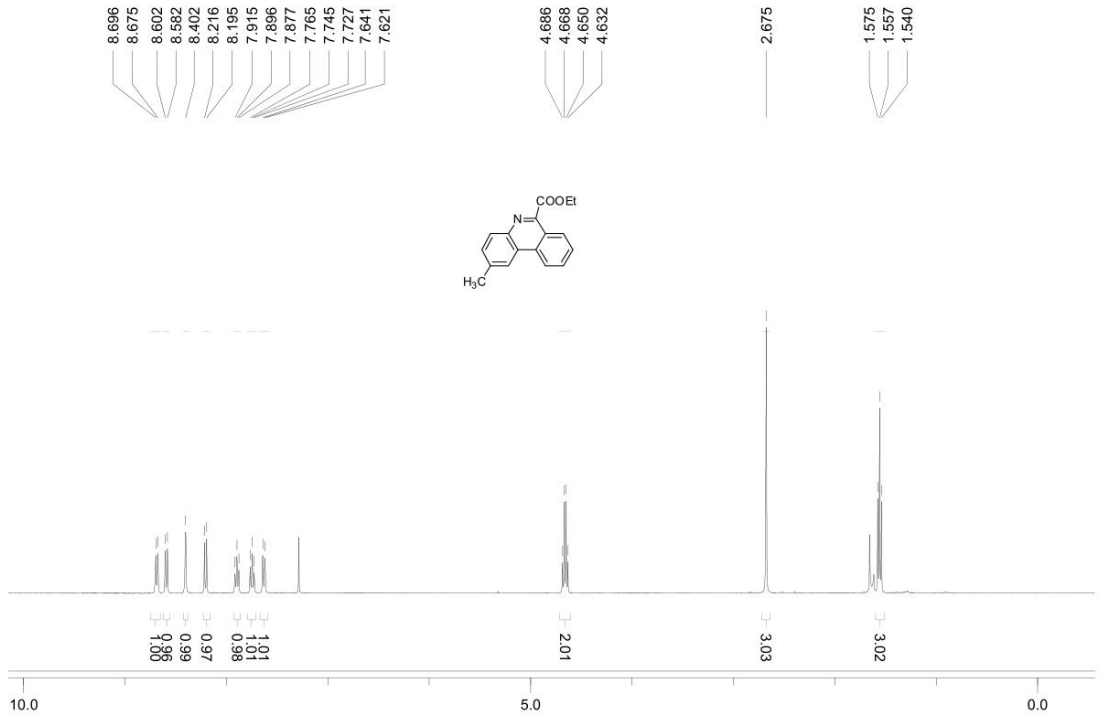


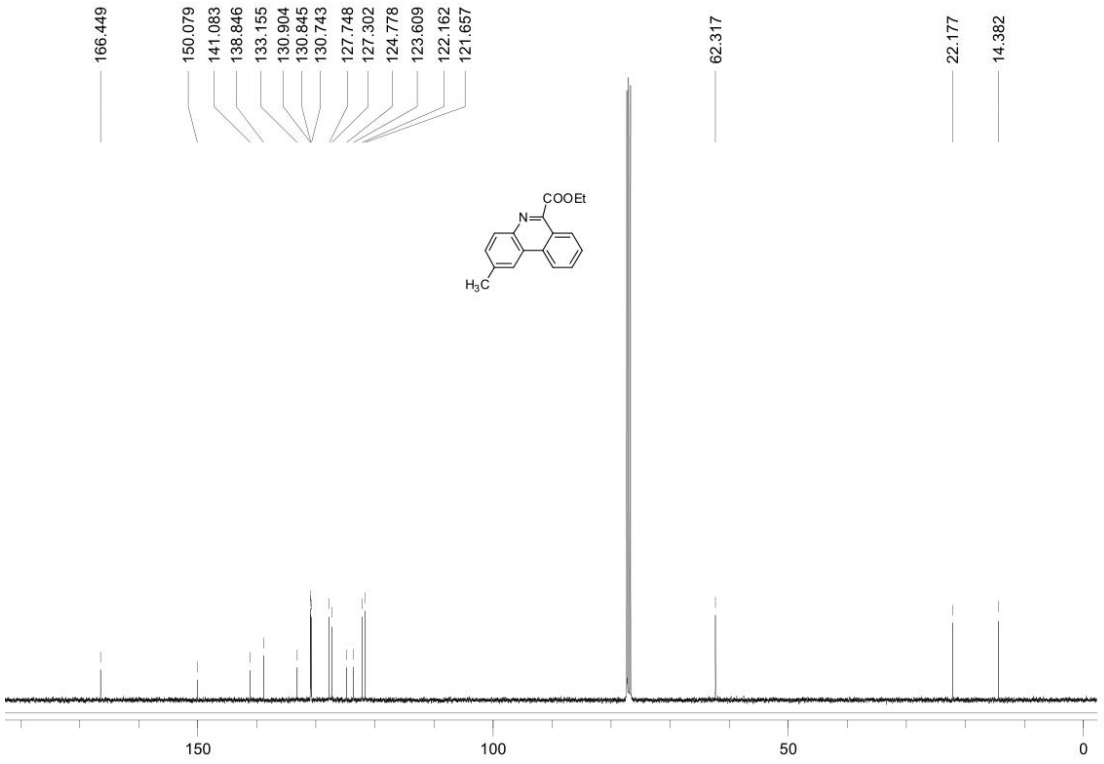
^{1}H NMR and ^{13}C NMR spectrum of **3k**



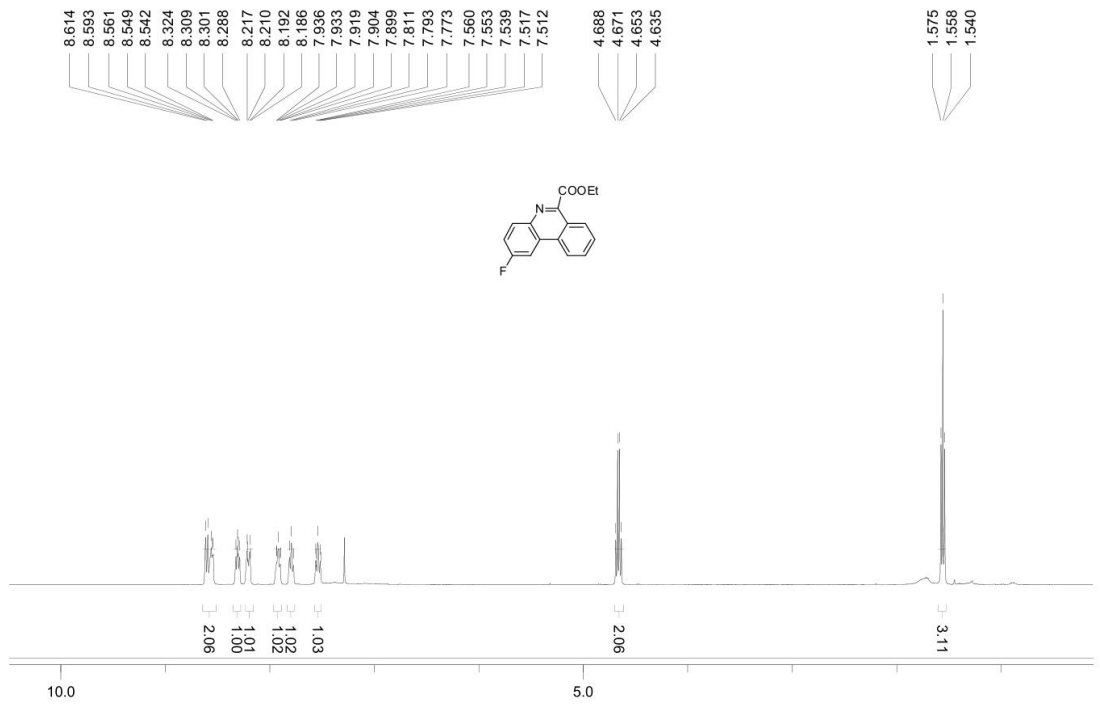


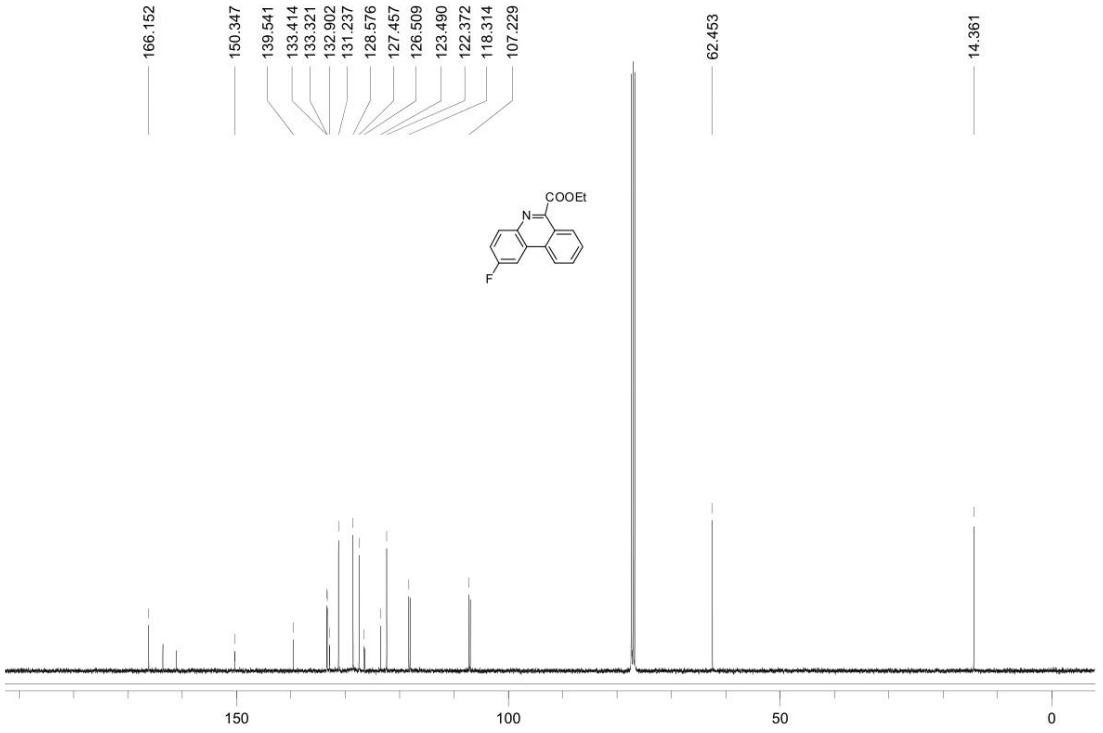
^{1}H NMR and ^{13}C NMR spectrum of **3l**



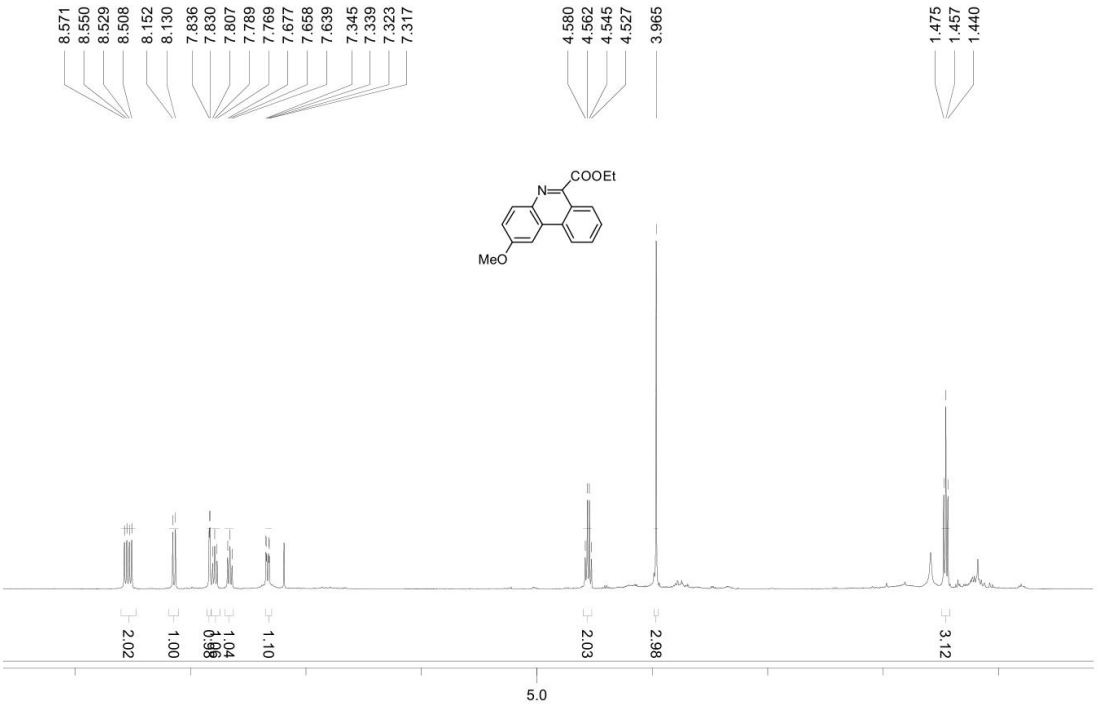


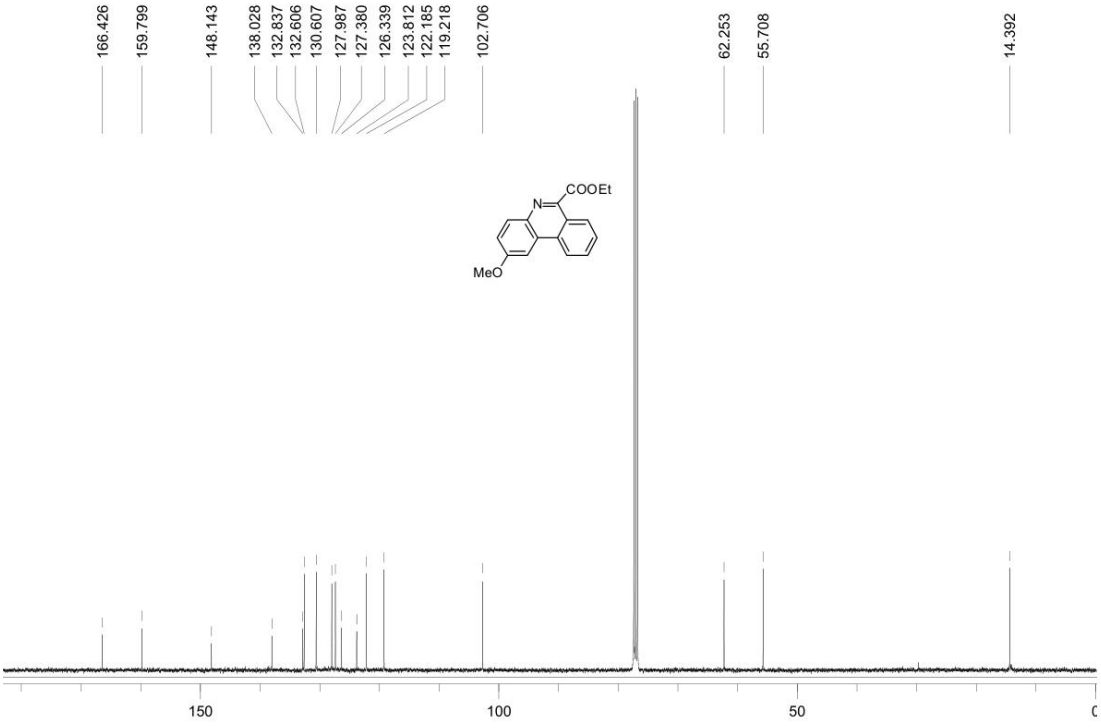
^{1}H NMR and ^{13}C NMR spectrum of **3m**



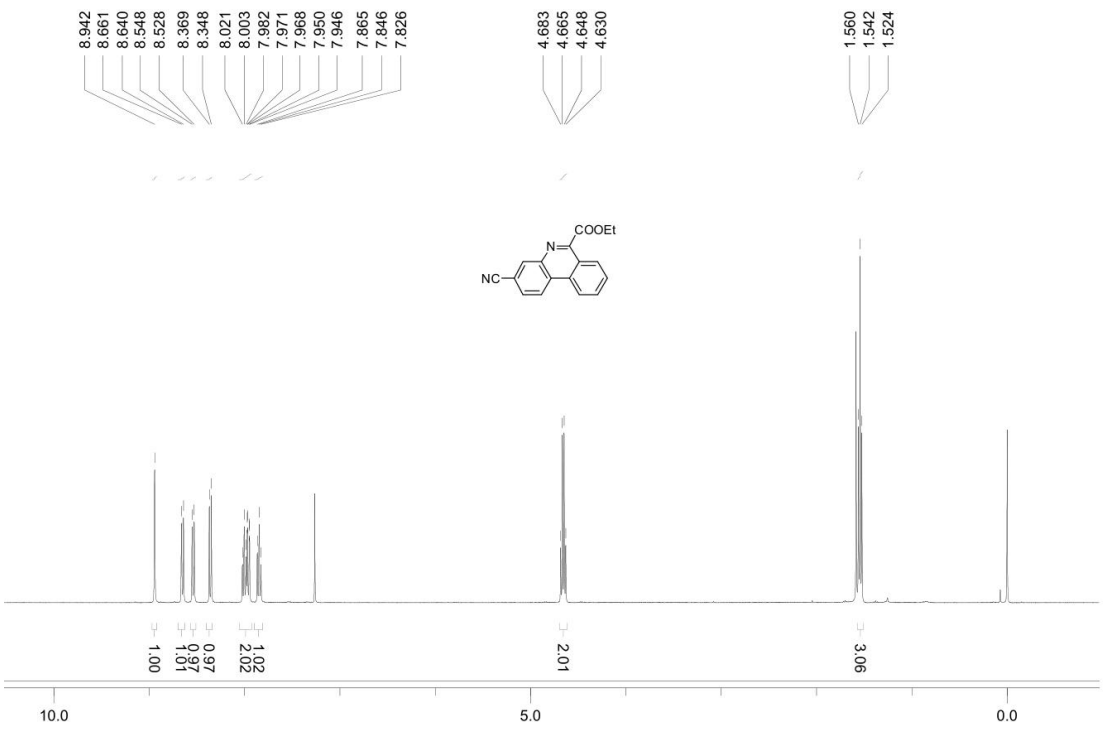


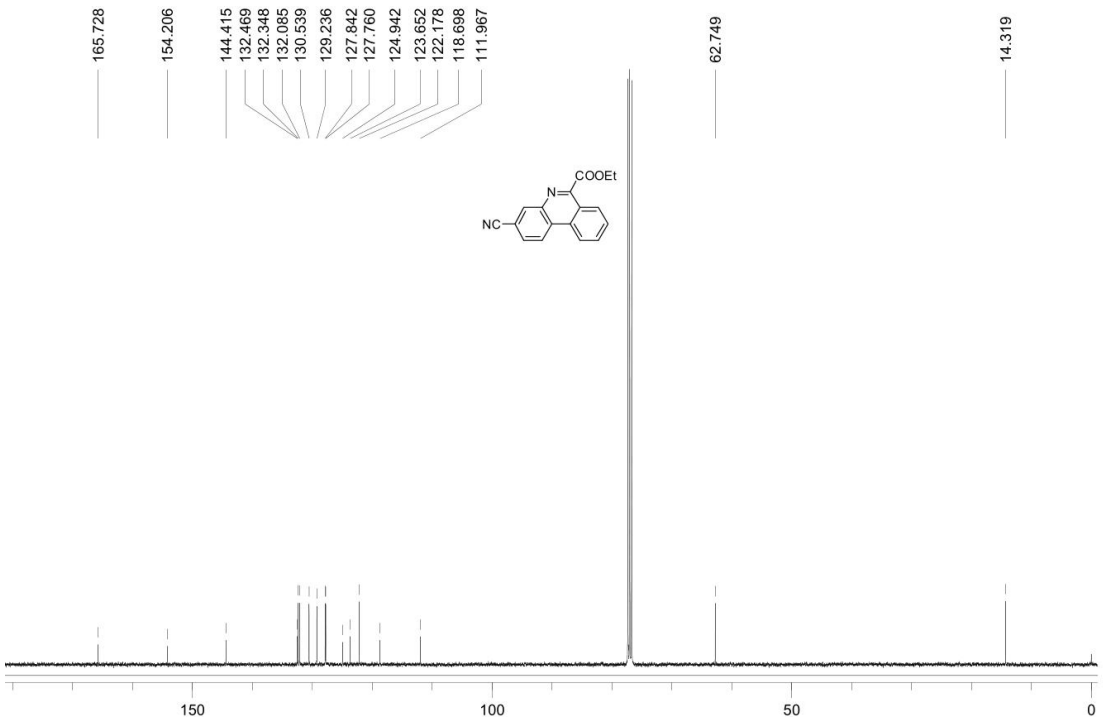
^{1}H NMR and ^{13}C NMR spectrum of **3n**



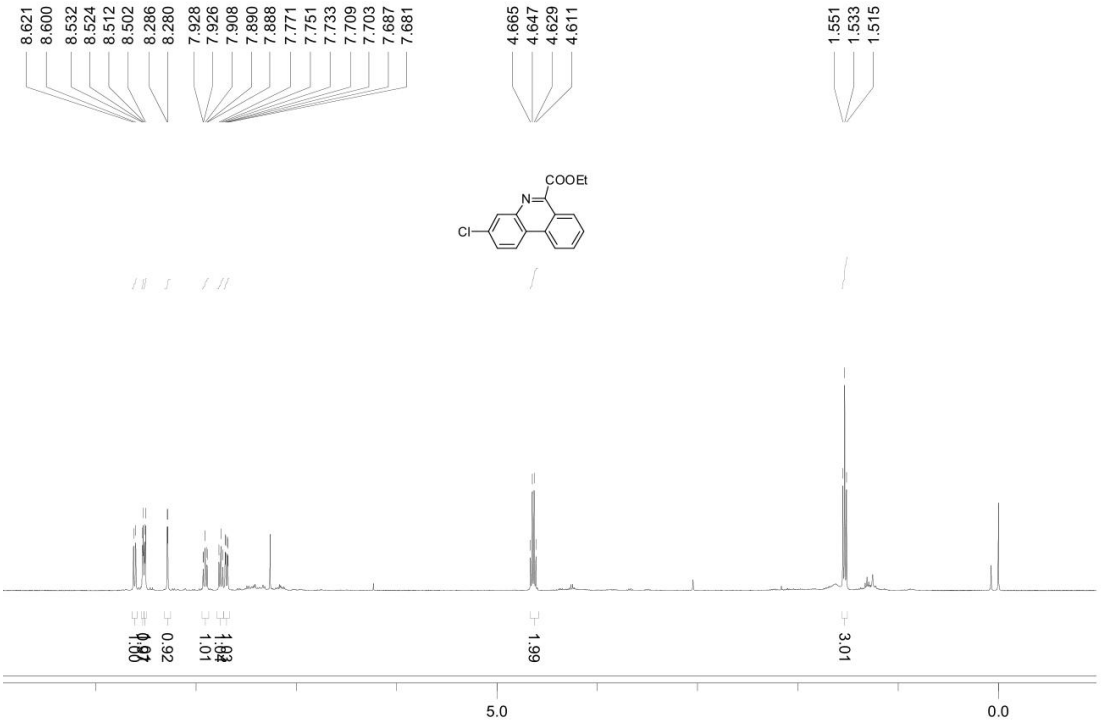


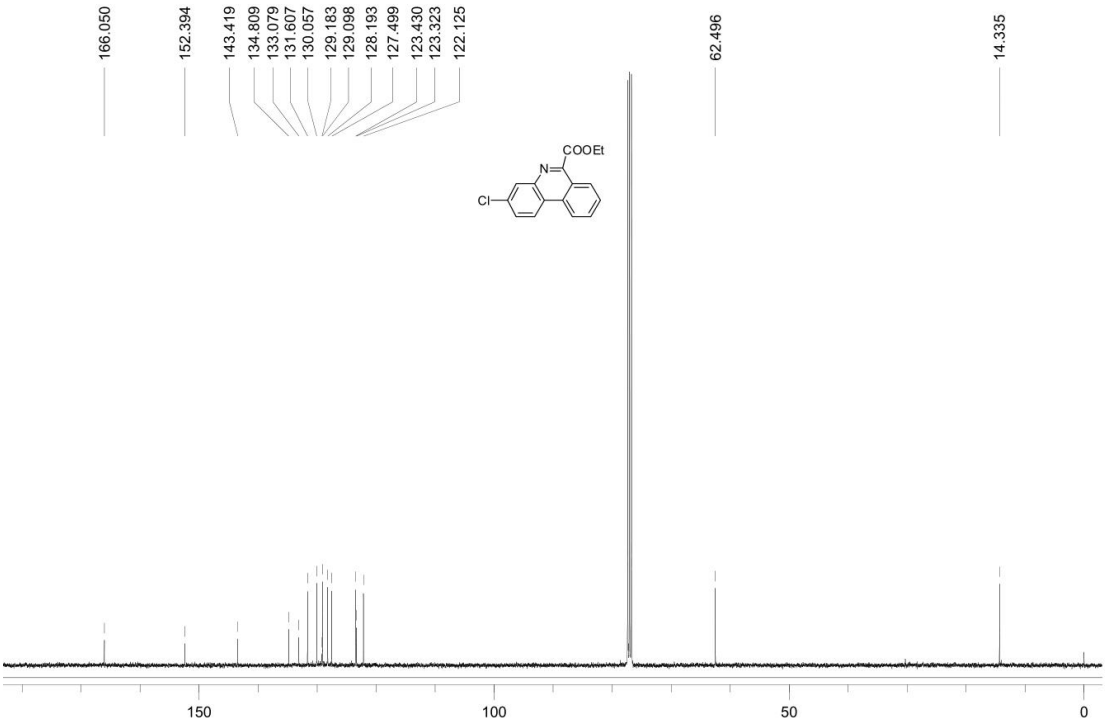
^{1}H NMR and ^{13}C NMR spectrum of **3o**



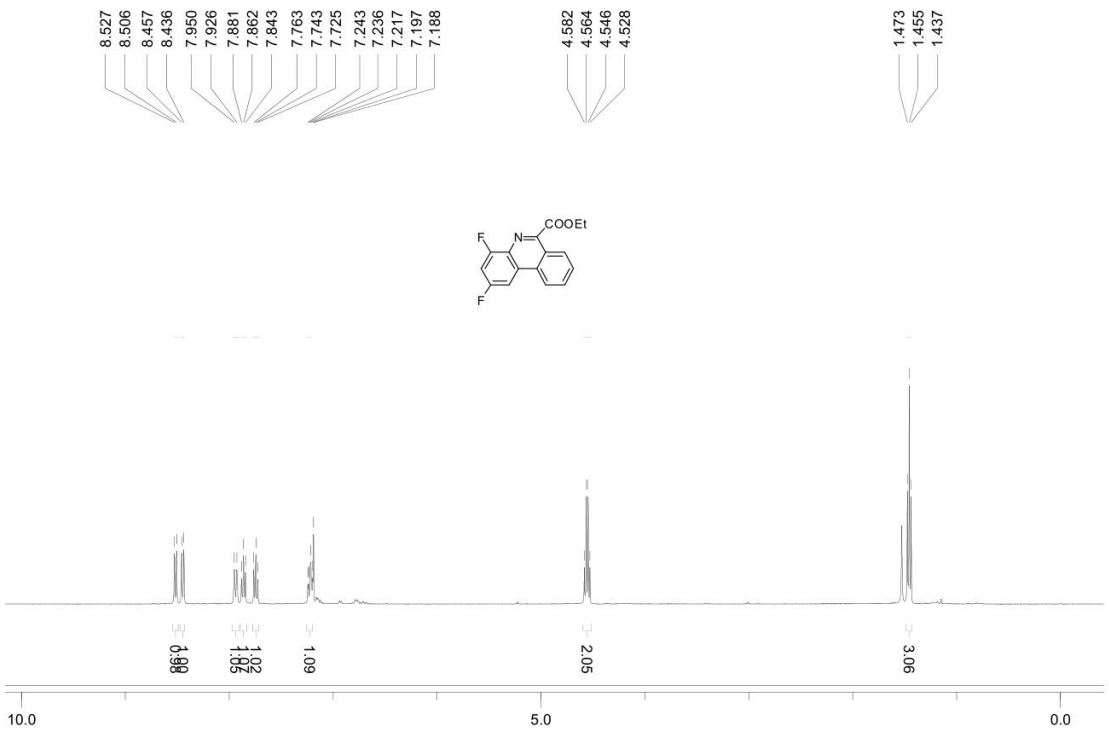


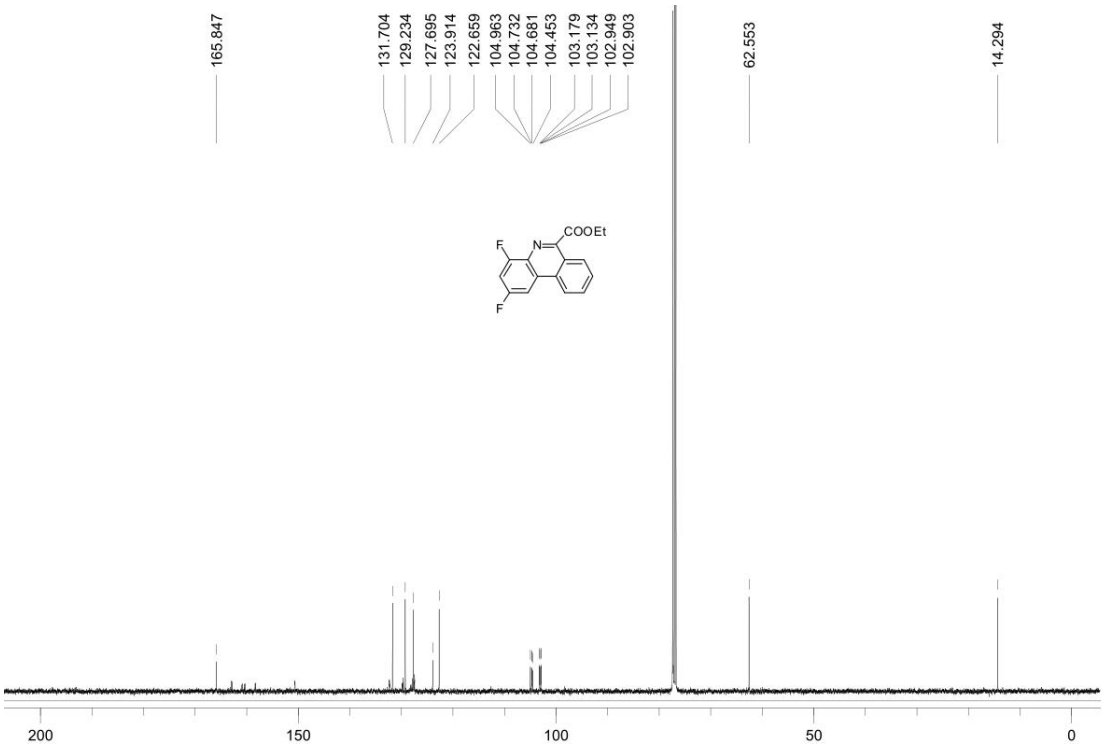
^{1}H NMR and ^{13}C NMR spectrum of **3p**



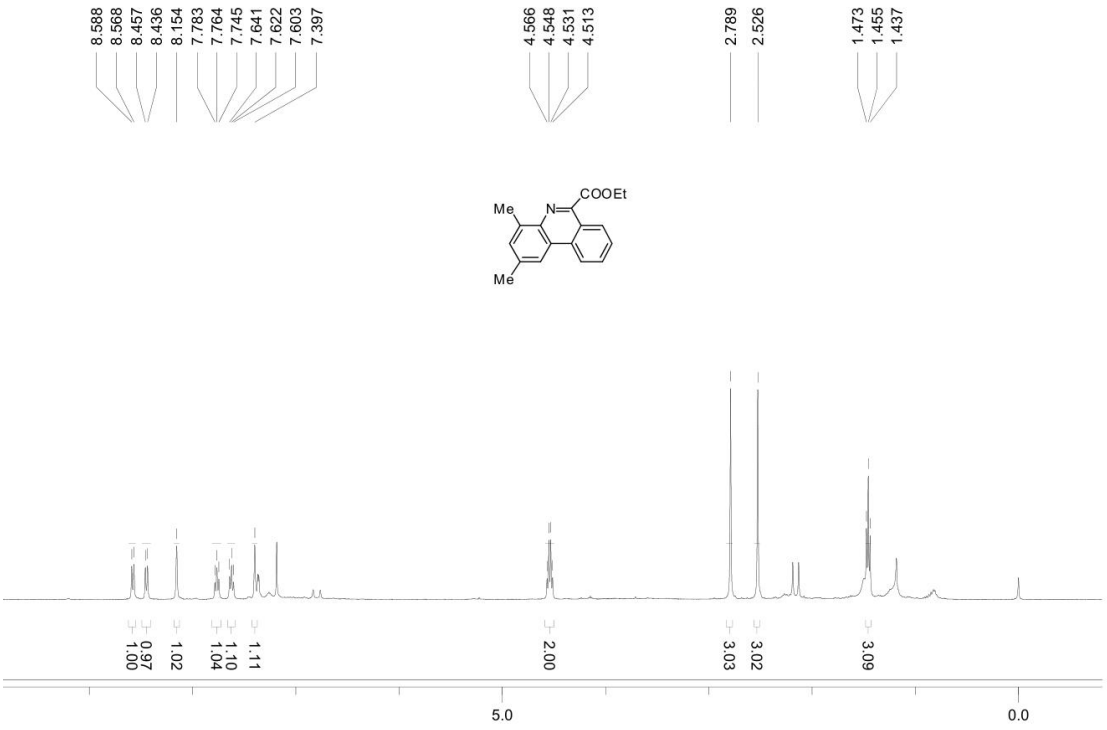


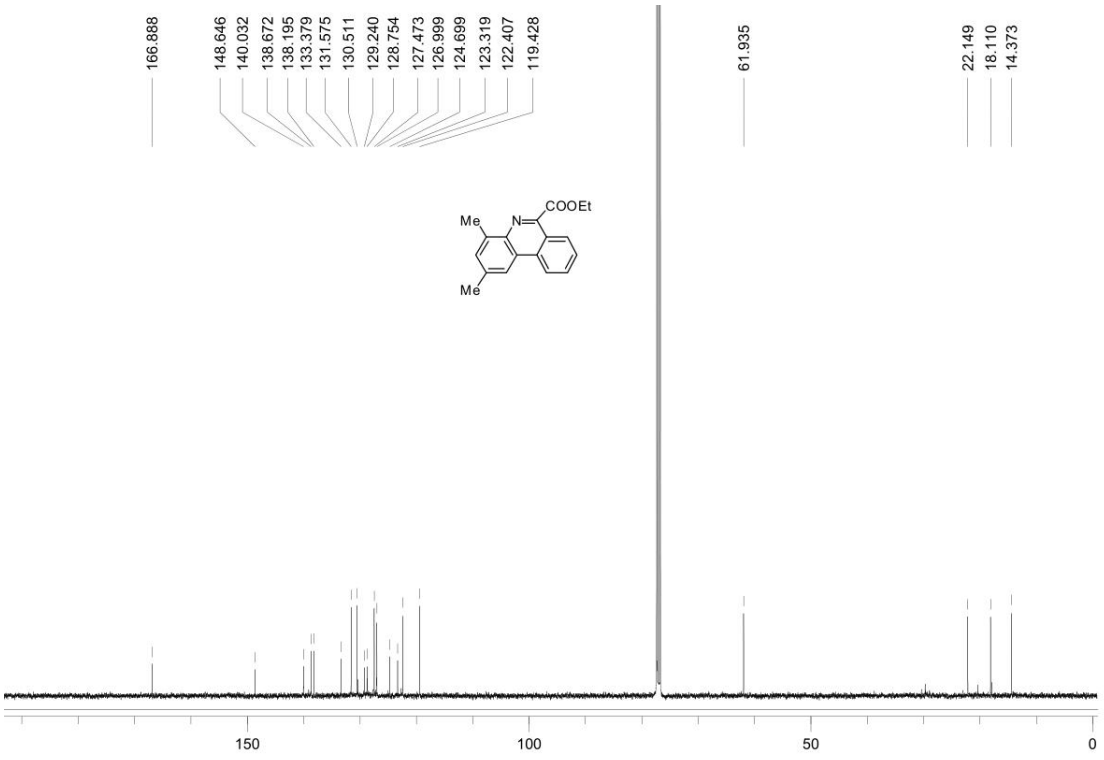
^{1}H NMR and ^{13}C NMR spectrum of **3q**



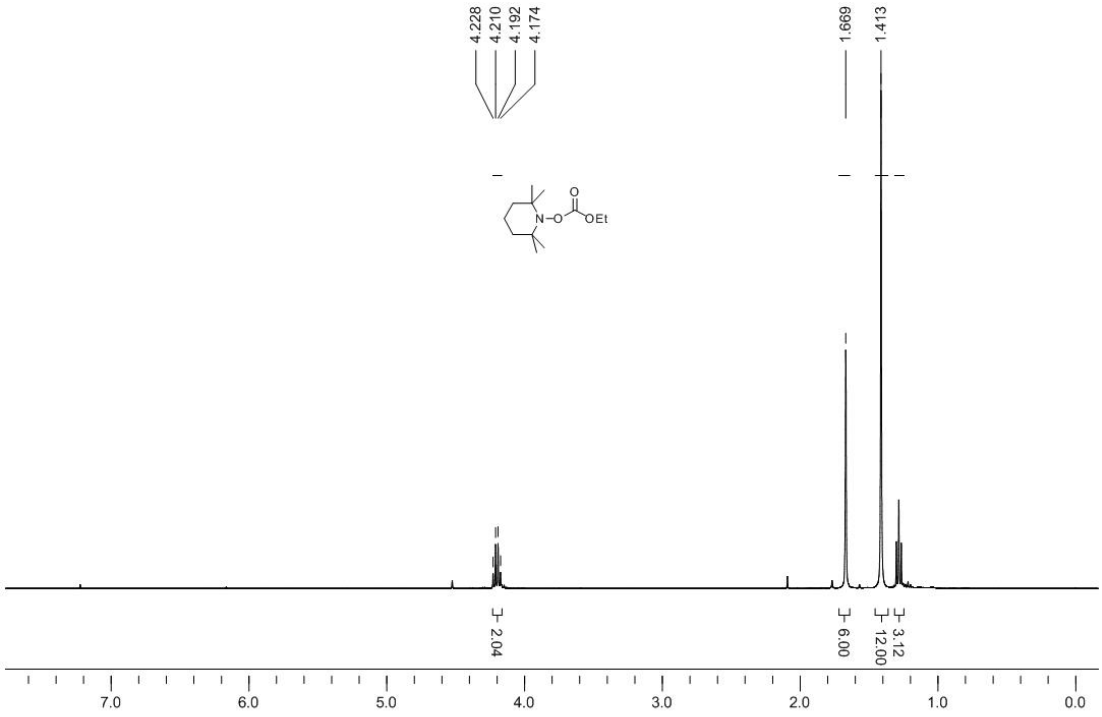


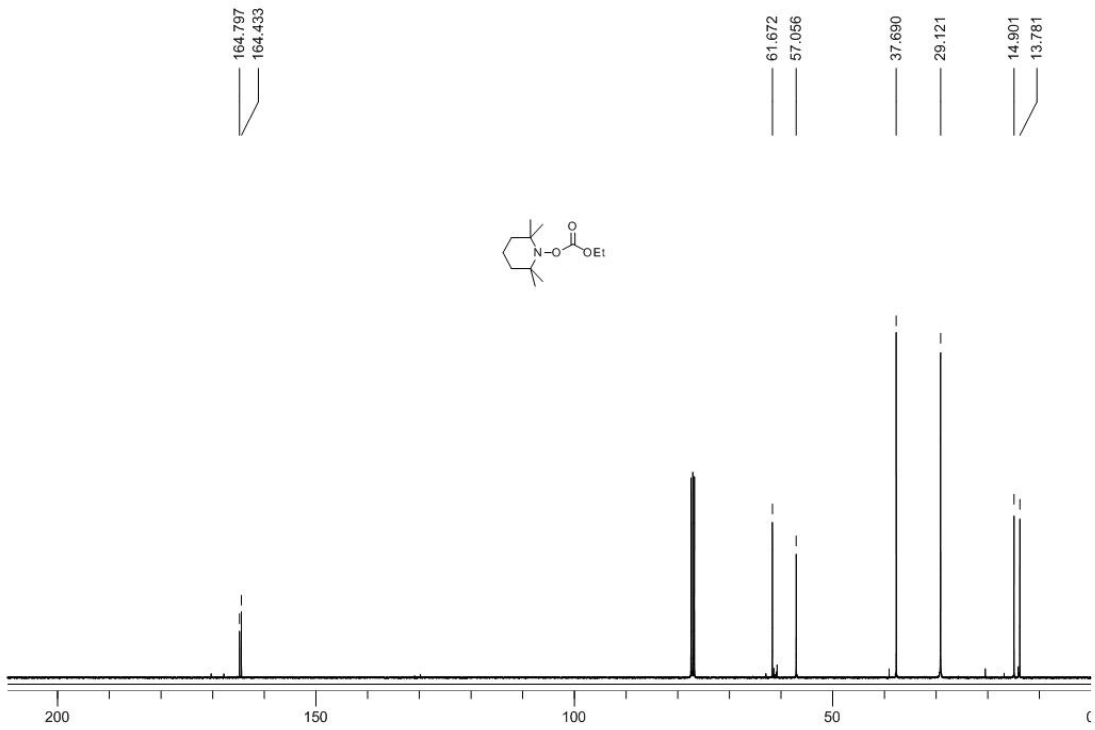
^{1}H NMR and ^{13}C NMR spectrum of **3r**



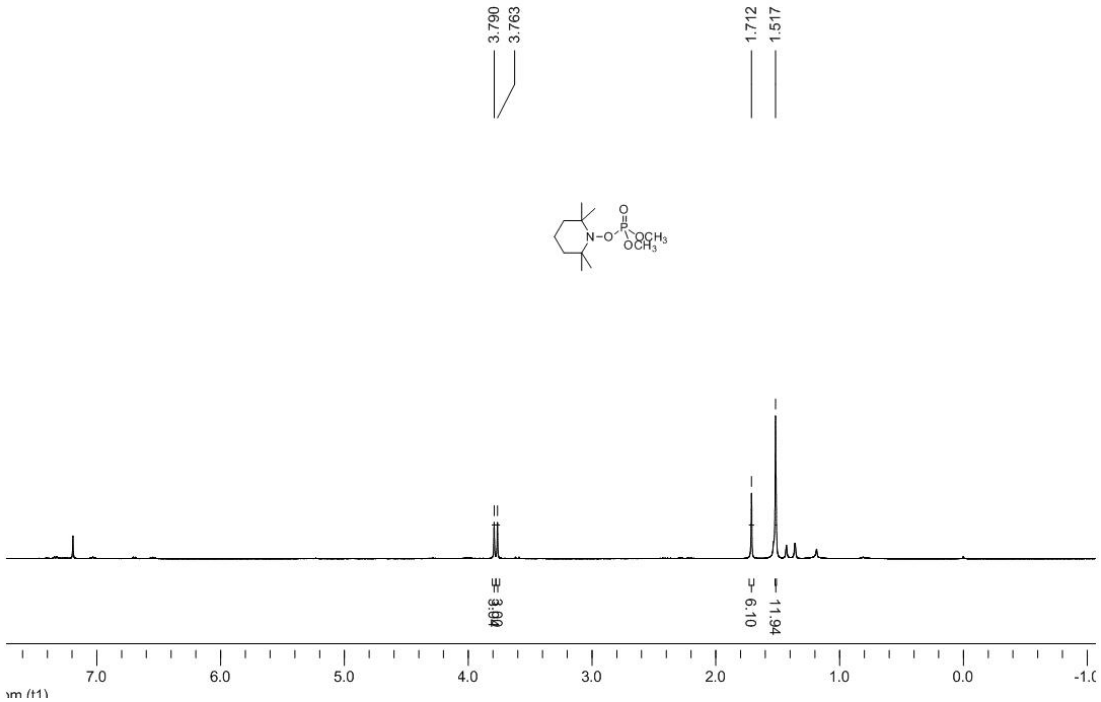


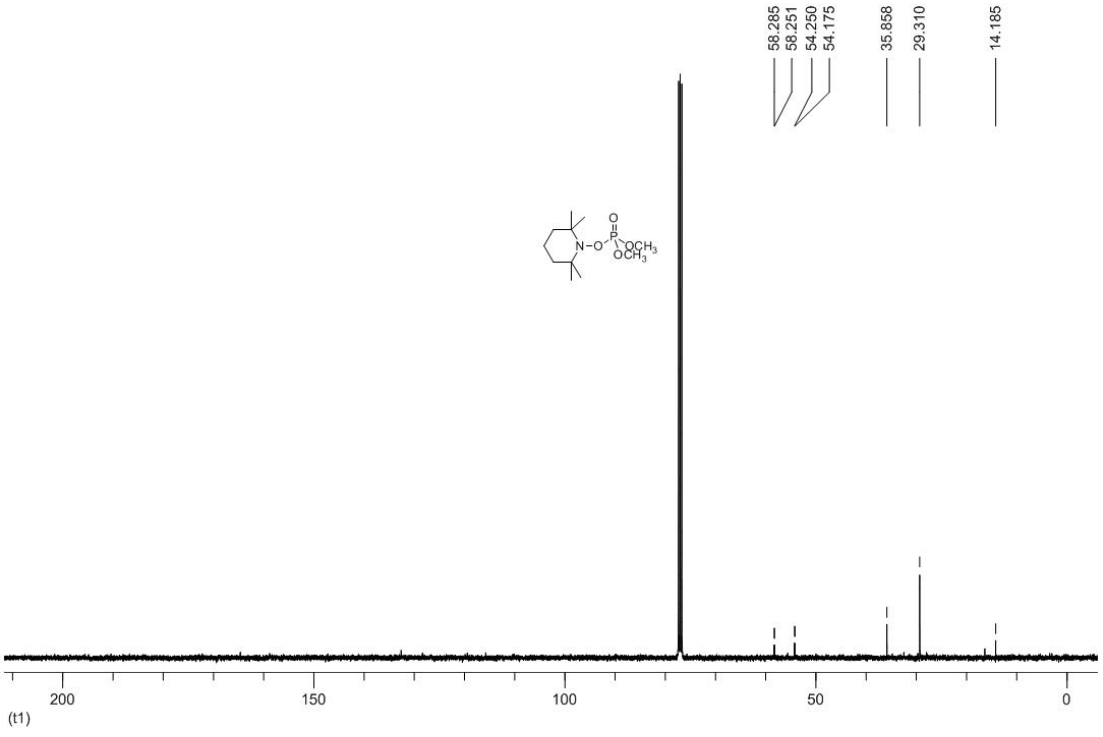
^{1}H NMR and ^{13}C NMR spectrum of **4**



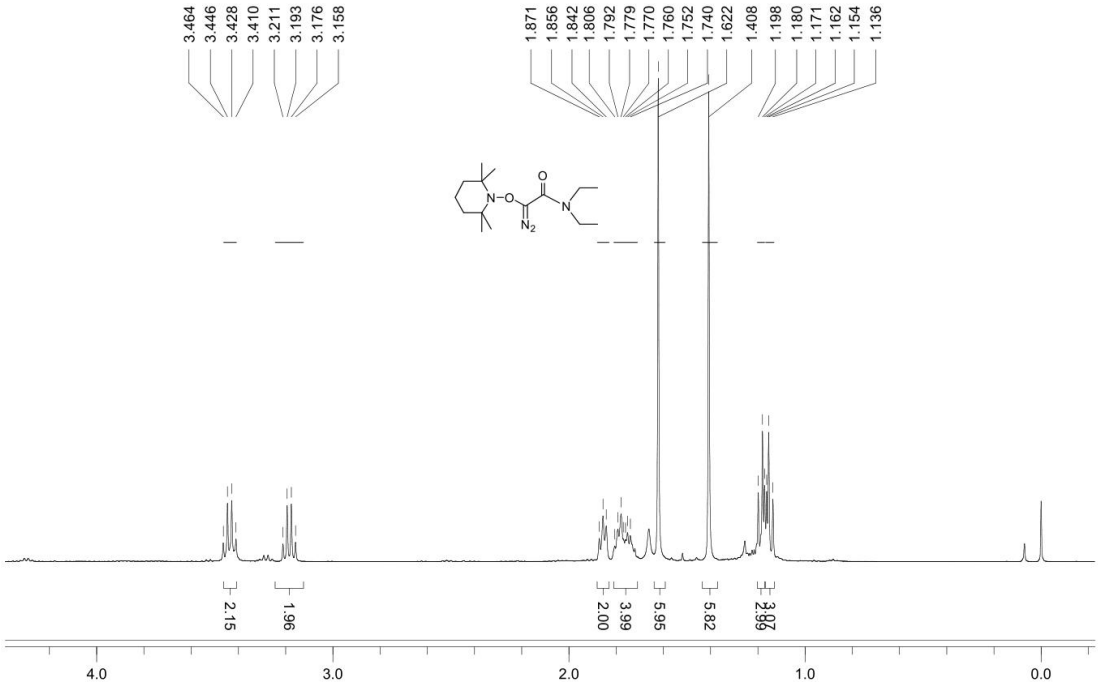


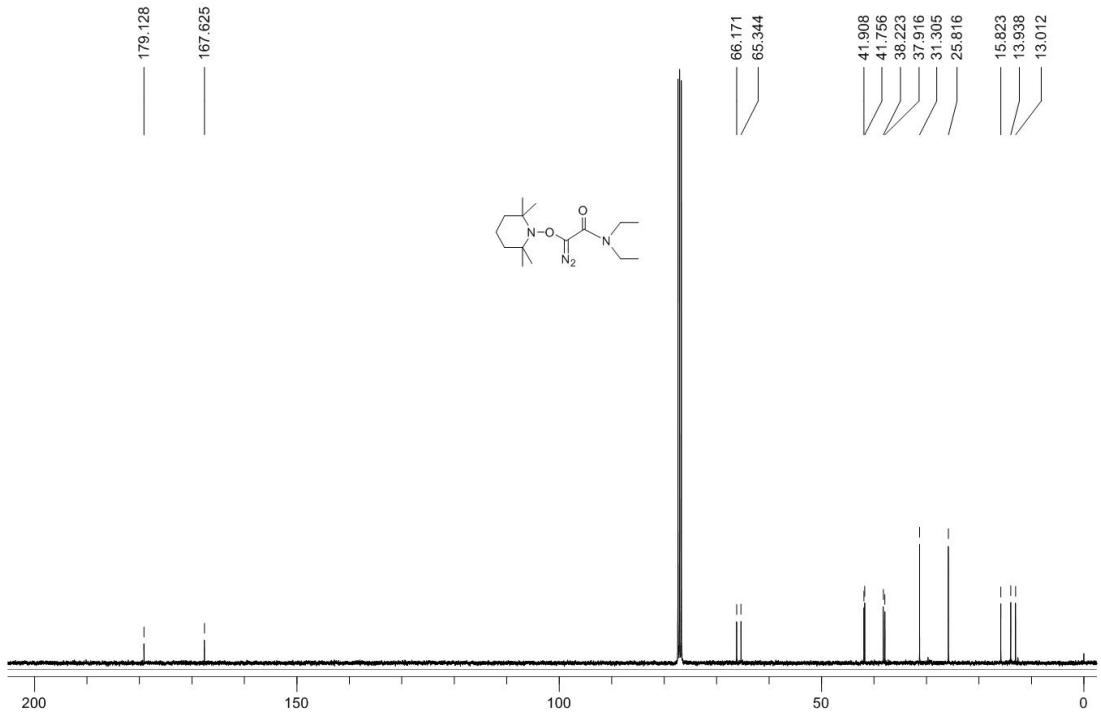
^{1}H NMR and ^{13}C NMR spectrum of **5**





^{1}H NMR and ^{13}C NMR spectrum of **6**





^{1}H NMR and ^{13}C NMR spectrum of **7**