Supplementary Information

Electrochemically driven [4 + 2] benzannulation: synthesis of polycyclic (hetero)aromatic compounds

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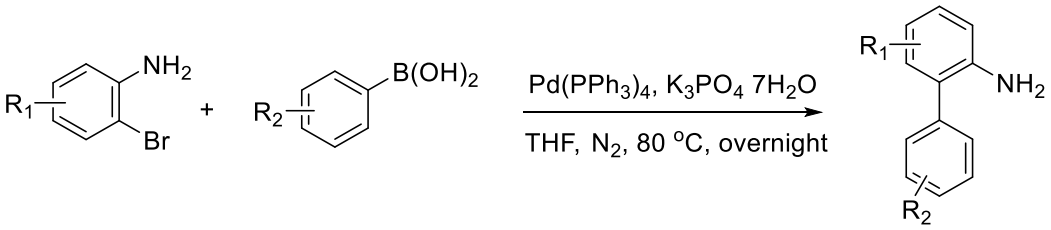
1. General Information

The electrodes were purchased from Fanyuedianzi, Shanghai. Potentiostats (A-BF， SS-L303SPD) were purchased on JD.COM. Solvents were purchased from Sinopharm (China), in GR (or CCER). Purification of products was conducted by column chromatography on silica gel (200-300 mesh, for some cases 300-400 mesh were used, from Qingdao, China). NMR spectra were measured on a Bruker ARX400 (^{1}H at 400 MHz, ^{13}C at 100 MHz) magnetic resonance spectrometer. Chemical shifts (δ) are reported in ppm using tetramethylsilane as internal standard (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet), and coupling constants (J) were reported in Hertz (Hz). LC-MS spectra were recorded on SHIMADZU LC-MS 2020. Mass spectra were recorded on an Agilent 6540 Q-Tof, a Bruker Daltonics Micro Tof, a Waters-Micromass Quatro LCZ (ESI) or Orbitrap LTQ XL (APCI); peaks are given in m/z (% of basis peak).

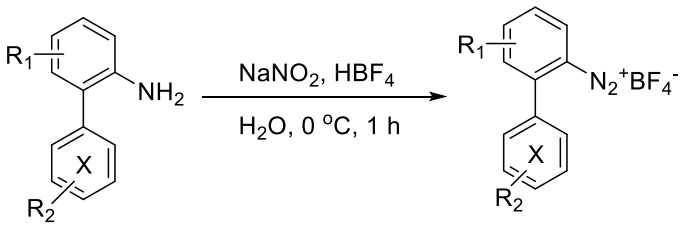
2. General Procedures

General Procedure A for the Synthesis of 2-Aminobiaryls.



Compounds were prepared according to literature.[1] $Pd(PPh_3)_4$ (461 mg, 5mol%), 2-bromobenzenamine (8 mmol), boronic acid (1.8 equiv.)and $K_3PO_4{\cdot}7H_2O$ (3 equiv., 8.12 g) were suspended in THF (20 mL) in a 50 mL schlenk tube under nitrogen. The reaction mixture was stirred at 80 °C and monitored by TLC. Upon completion of the reaction, the resulting mixture was cooled to room temperature and filtered through a short path of silica gel, eluting with CH_2Cl_2. The volatile compounds were removed in vacuo and the residue was purified by column chromatography (SiO_2, 2%-5% ethyl acetate in hexane).

General Procedure B for the Biaryldiazonium Tetrafluoroborates.



Compounds were prepared according to literature.[1] In a mixture of 5 mL of distilled water and 5 mL of 40% hydrofluoroboric acid was dissolved 10 mmol of the appropriate biaryl-2-amine. After the reaction mixture was cooled to 0 °C using an ice bath, a solution of sodium nitrite in distilled water (0.69 g in 1.5 mL) was added dropwise. The suspension was stirred for an additional 40 min and then filtered, and the resulting solid was redissolved in a minimum amount of acetone. Diethyl ether was added until precipitation of diazonium tetrafluoroborate, which was filtered, washed several times with diethyl ether, and dried under vacuum. (*Important Safety Note: Handling of aryldiazonium salts should be done in a well-ventilated fume cupboard. No incidents occurred handling of these reagents during this study, yet the readers should be aware of carcinogenicity and explosiveness of the herein described aryldiazonium salts. The aryldiazonium salts are highly energetic compounds (shock sensitive), which can undergo violent decomposition and even explosion in the process of synthesis and separation.[2] The aryldiazonium salts tend to decompose at low temperatures with enthalpies of decomposition of -160 to -180 kJ/mol.[3] The detected onset temperature of* ***1a*** *was determined to be 65 °C by DSC in our lab. General safety precautions when working with diazo compounds and diazonium salts should be followed. Any reactions described in this manuscript should not be performed without strict risk assessment.*)

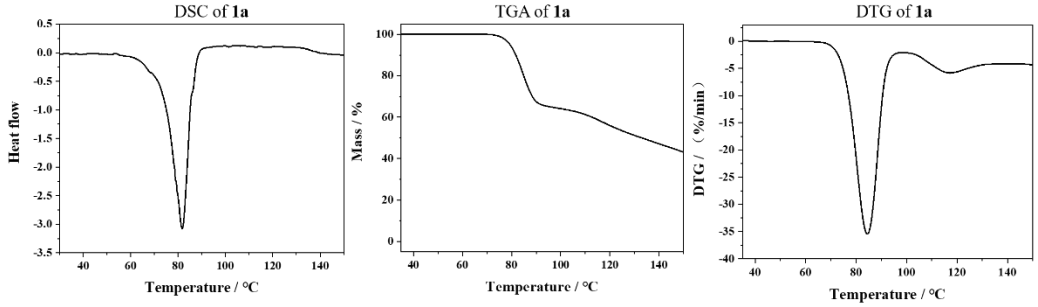


Fig. S1 DSC, TGA and DTG data of **1a**.

General Procedure C for the Electrocatalytic [4 + 2] benzannulation of biaryl diazonium salts and alkynes.

In a single-necked flask (10 mL) equipped with a stir bar, biaryl diazonium tetrafluoroborate (0.50 mmol), alkyne (2.5 mmol), *n*Bu_4NBF_4 (165 mg, 0.5 mmol), K_2CO_3 (69 mg, 0.5 mmol) and MeCN/H_2O (v/v=4:1, 5 mL) were combined and added. The bottle was equipped with NF (nickel foam) (10 ×10 mm^2) as the anode and graphite plate (10×10 mm^2) as the cathode. The reaction mixture was stirred and electrolyzed at a constant current of 5 mA under N_2 for 4 h. When the

reaction was finished, the solution was extracted with DCM and H_2O. The combined organic layer was dried with Na_2SO_4, filtered. The solvent was removed with a rotary evaporator. The pure product was obtained by flash chromatography on silica gel.

General Procedure D for the One Pot Diazotization/Electrochemical Reaction

In a single-necked flask (10 mL) equipped with a stir bar, biaryl-2-amine (0.50 mmol) was dissolved in the MeCN/H_2O (v/v=4:1, 5 mL). The reaction mixture was cooled to 0 ºC, then HBF_4 (50% in Et_2O, 0.525 mmol) and *t*BuONO (90%, 0.525 mmol) was added and the reaction mixture was stirred at 0 ºC for 30 min. After that, *n*Bu_4NBF_4 (165 mg, 0.5 mmol), K_2CO_3 (69 mg, 0.5 mmol) and alkyne (2.5 mmol) were added to the mixture. The bottle was equipped with NF (nickel foam) (10 ×10 mm^2) as the anode and graphite plate (10×10 mm^2) as the cathode. The reaction mixture was stirred and electrolyzed at a constant current of 5 mA under N_2 for 4 h. The current density was 5 mA/cm^2. When the reaction was finished, the solution was extracted with DCM and H_2O. The combined organic layer was dried with Na_2SO_4, filtered. The solvent was removed with a rotary evaporator. The pure product was obtained by flash chromatography on silica gel.

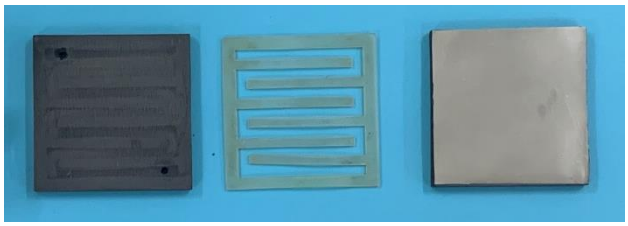
Procedure E for Gram Scale Synthesis:

Flow cell (single pass mode electrolysis): The flow cell was equipped with graphite plate (5.0 cm× 5.0 cm× 5 mm) as the anode and nickel plate (5.0 cm× 5.0 cm× 5 mm) as the cathode. The anode and cathode are held apart by a polytetrafluoroethylene (PTEF) board of 1.0 mm thick. A rectangular reaction channel (total length: 313 mm, width: 3.2 mm) is cut in the PTEF foil to give an overall channel volume of 1.0 mL, and the surface area for the electrode in contact with the reaction solution is 10 cm^{-2}. The whole device is held together by steel screws. The pump used in the experiment is a QHZS-001A model syringe pump manufactured by Yuanhang (Figure S1).

In a 50 mL syringe, [1,1'-biphenyl]-2-diazonium tetrafluoroborate **1a** (1.34 g, 5 mmol), phenylacetylene **2a** (2.55g, 25 mmol), K_2CO_3 (0.69 g, 5 mmol) and MeCN/H_2O (v/v=4:1, 50 mL) were added, then dissolved uniformly by shaking. The reaction mixture used for the flow experiments was a homogenous solution. The reaction mixture was pumped into the electrochemical reactor in a flow rate of 2.0 mL/h (Figure S1). A constant current of 20 mA was employed during the electrolysis under room temperature for 25 h. The current density was 2 mA/cm^2 and the total charge was 3.73 F/mol. When the reaction finished, the solution was extracted with DCM and H_2O.

The combined organic layer was dried with Na_2SO_4, filtered. The solvent was removed with a rotary evaporator. The pure product was obtained by flash chromatography on silica gel using petroleum ether and ethyl acetate as the eluent (25:1).

A



B

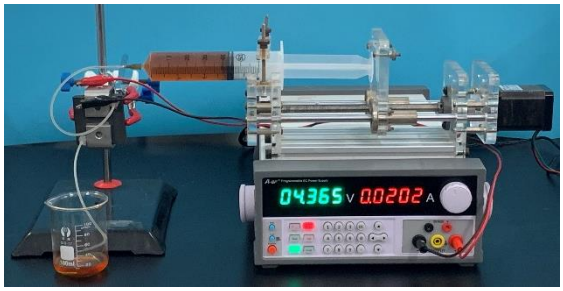


Fig. S2 Scale-up experiment equipment.

3. Preliminary mechanistic studies.

In a single-necked flask (10 mL) equipped with a stir bar, [1,1'-biphenyl]-2-diazonium tetrafluoroborate (0.50 mmol) **1a**, phenylacetylene (2.5 mmol) **2a**, *n*Bu_4NBF_4 (165 mg, 0.5 mmol), K_2CO_3 (69 mg, 0.5 mmol), radical scavengers (BHT or TEMPO, 1 mmol) and MeCN/H_2O (v/v=4:1, 5 mL) were combined and added. The bottle was equipped with NF (nickel foam) (10 ×10 mm^2) as the anode and graphite plate (10×10 mm^2) as the cathode. The reaction mixture was stirred and electrolyzed at a constant current of 5 mA under N_2 for 4 h. When the reaction was finished, the solution was extracted with DCM and H_2O. The combined organic layer was dried with Na_2SO_4, filtered. The reaction mixture was analyzed by LC-MS. The solvent was removed with a rotary evaporator. The pure product was obtained by flash chromatography on silica gel using petroleum ether and ethyl acetate as the eluent (25:1).

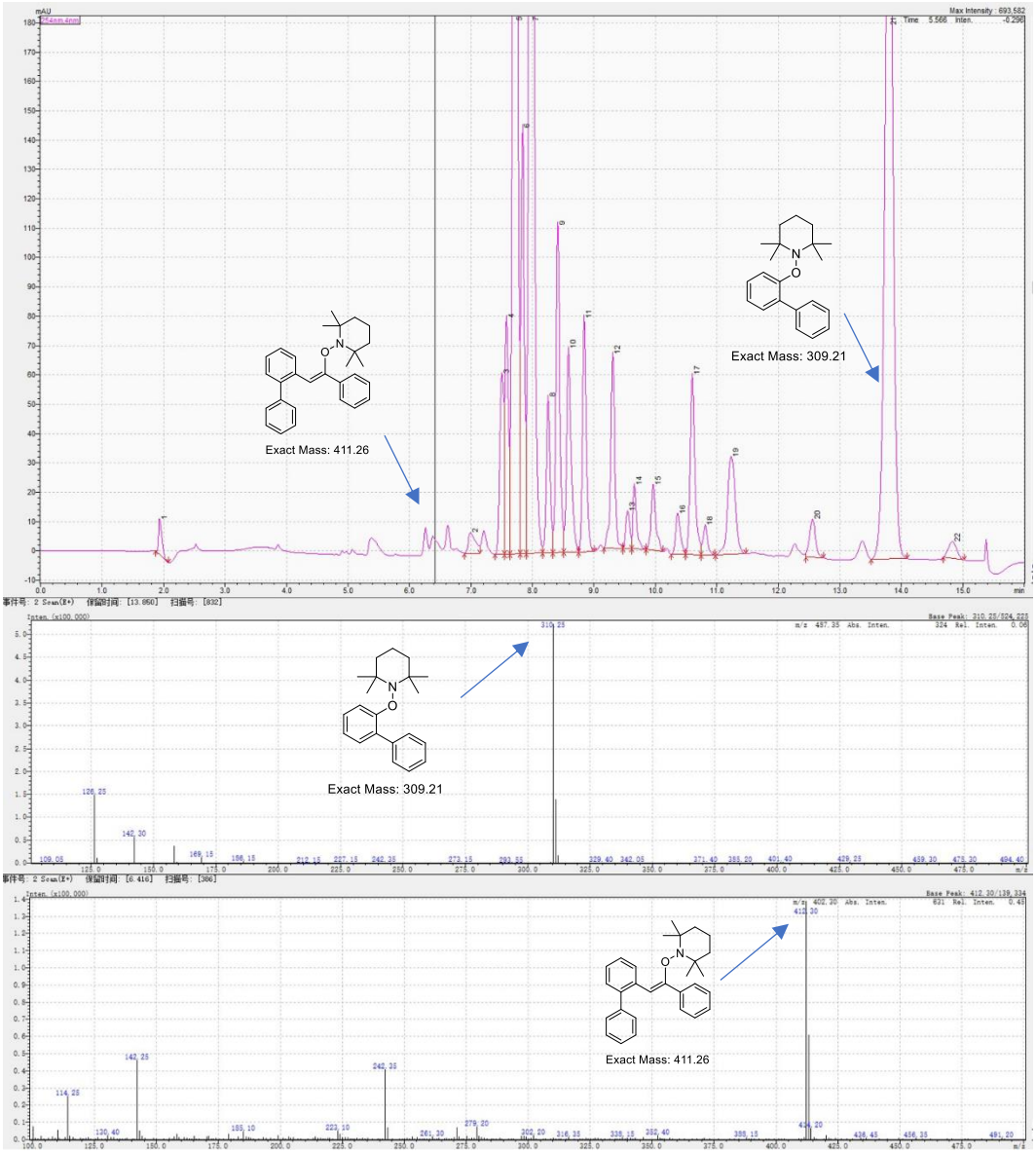
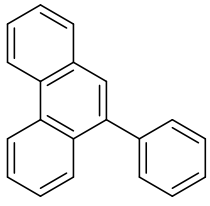


Fig. S3 LC-MS chart

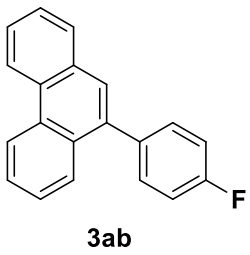
4. Characterization Data of Products.



3aa

9-phenylphenanthrene (3aa):

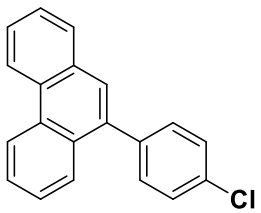
Synthesis carried out according to the General Procedure C, compound **3aa** was obtained in 76% yield as a white solid (96.5 mg, 0.38 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (d, J = 8.3 Hz, 1H), 8.68 (d, J = 8.1 Hz, 1H), 7.90 (d, J = 8.2 Hz, 1H), 7.86 (d, J = 7.6 Hz, 1H), 7.69 – 7.55 (m, 4H), 7.47 (ddd, J = 22.7, 13.2, 6.7 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.8, 138.8, 131.6, 131.1, 130.6, 130.1, 129.9, 128.7, 128.3, 127.5, 127.4, 126.9, 126.8, 126.6, 126.5, 126.4, 122.9, 122.5. IR (KBr) ν_{max} 2922, 1489, 1445, 891, 746, 722, 700 cm^{-1}. HRMS (APCI) calculated for $C_{20}H_{14}$ $[M]^+$: 254.1096, found: 254.1098. These spectral data correspond to previously reported data.[4]



3ab

9-(4-fluorophenyl)phenanthrene (3ab):

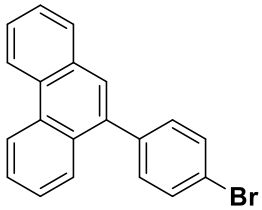
Synthesis carried out according to the General Procedure C, compound **3ab** was obtained in 63% yield as a white solid (85.7 mg, 0.315 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.76 (d, *J* = 8.3 Hz, 1H), 8.70 (d, *J* = 8.2 Hz, 1H), 7.86 (t, *J* = 8.4 Hz, 2H), 7.63 (td, *J* = 14.8, 7.2 Hz, 4H), 7.56 – 7.43 (m, 3H), 7.19 (t, *J* = 8.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4 (d, *J* = 246.1 Hz), 137.7, 136.7 (d, *J* = 3.2 Hz), 131.6 (d, *J* = 8.0 Hz), 131.5, 131.1, 130.7, 130.0, 128.7, 127.7, 127.0, 126.7, 126.7, 126.6, 126.6, 123.0, 122.6, 115.3 (d, *J* = 21.3 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ -115.1. IR (KBr) ν_{max} 2930, 1501, 1449, 1213, 1155, 834, 752, 727 cm^{-1}. HRMS (APCI) calculated for $C_{20}H_{13}F$ $[M]^+$: 272.1001, found: 272.1006. These spectral data correspond to previously reported data.[4]



3ac

9-(4-chlorophenyl)phenanthrene (3ac):

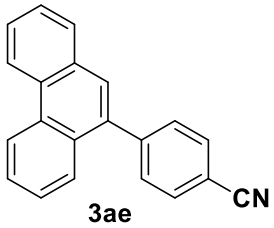
Synthesis carried out according to the General Procedure C, compound **3ac** was obtained in 62% yield as a white solid (89.28 mg, 0.31 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.77 (d, *J* = 8.3 Hz, 1H), 8.72 (d, *J* = 8.2 Hz, 1H), 7.87 (dd, *J* = 14.5, 7.9 Hz, 2H), 7.71 – 7.59 (m, 4H), 7.55 (dd, *J* = 15.8, 8.3 Hz, 1H), 7.51 – 7.40 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.2, 137.5, 133.5, 131.4, 130.9, 130.7, 130.1, 128.7, 128.6, 127.6, 127.0, 126.8, 126.7, 126.6, 123.0, 122.6. IR (KBr) ν_{max} 2921, 1487, 1450, 1090, 828, 748, 724, 584 cm^{-1}. HRMS (APCI) calculated for $C_{20}H_{13}Cl$ $[M]^+$: 288.0706, found: 288.0710. These spectral data correspond to previously reported data.[5]



3ad

9-(4-bromophenyl)phenanthrene (3ad):

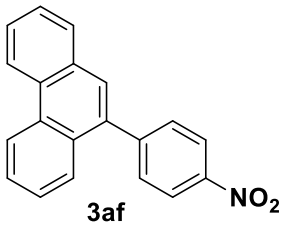
Synthesis carried out according to the General Procedure C, compound **3ad** was obtained in 70% yield as a white solid (116.2 mg, 0.35 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.78 (d, *J* = 8.3 Hz, 1H), 8.73 (d, *J* = 8.2 Hz, 1H), 7.90 (d, *J* = 7.3 Hz, 1H), 7.85 (d, *J* = 8.1 Hz, 1H), 7.73 – 7.60 (m, 6H), 7.55 (dd, *J* = 11.1, 4.0 Hz, 1H), 7.43 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.7, 137.5, 131.7, 131.5, 131.4, 130.8, 130.7, 130.1, 128.7, 127.6, 127.0, 126.8, 126.7, 126.6, 126.6, 123.0, 122.6, 121.6. IR (KBr) ν_{max} 2955, 2921, 1702, 1484, 1451, 1007, 824, 746, 722 cm^{-1}. HRMS (APCI) calculated for $C_{20}H_{13}Br$ $[M]^+$: 332.0201, found: 332.0207. These spectral data correspond to previously reported data.[5]



3ae

4-(phenanthren-9-yl)benzonitrile (3ae):

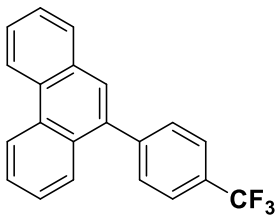
Synthesis carried out according to the General Procedure C, compound **3ae** was obtained in 79% yield as a white solid (110.2 mg, 0.395 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.80 (d, *J* = 8.3 Hz, 1H), 8.74 (d, *J* = 8.2 Hz, 1H), 7.91 (d, *J* = 7.8 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 2H), 7.78 (d, *J* = 8.2 Hz, 1H), 7.75 – 7.61 (m, 6H), 7.57 (t, *J* = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.8, 136.8, 132.2, 131.2, 131.2, 130.9, 130.7, 130.3, 130.2, 128.9, 128.0, 127.3, 127.2, 126.9, 126.2, 123.2, 122.6, 118.9, 111.3. IR (KBr) ν_{max} 3862, 2957, 2926, 2315, 2227, 1704, 1456, 1049, 751, 591 cm^{-1}. HRMS (APCI) calculated for $C_{21}H_{13}N$ $[M]^+$: 279.1048, found: 279.1052. These spectral data correspond to previously reported data.[6]



3af

9-(4-nitrophenyl)phenanthrene (3af):

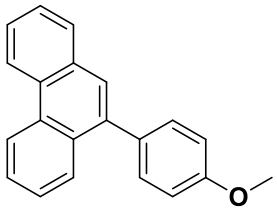
Synthesis carried out according to the General Procedure C, compound **3af** was obtained in 82% yield as a yellow solid (122.6 mg, 0.41 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.81 (d, *J* = 8.3 Hz, 1H), 8.74 (d, *J* = 8.3 Hz, 1H), 8.38 (d, *J* = 8.6 Hz, 2H), 7.92 (d, *J* = 7.6 Hz, 1H), 7.79 (d, *J* = 8.2 Hz, 1H), 7.75 – 7.68 (m, 5H), 7.66 (t, *J* = 7.5 Hz, 1H), 7.57 (t, *J* = 7.3 Hz, 1H).^{13}C NMR (100 MHz, $CDCl_3$) δ 147.8, 147.3, 136.5, 131.1, 130.9, 130.8, 130.4, 130.2, 128.9, 128.0, 127.4, 127.2, 126.9, 126.2, 123.6, 123.2, 122.6. IR (KBr) ν_{max} 2962, 1593, 1513, 1452, 1344, 1099, 847, 799, 747 cm^{-1}. HRMS (APCI) calculated for $C_{20}H_{13}NO_2$ $[M]^+$: 299.0946, found: 299.0952. These spectral data correspond to previously reported data.[1]



3ag

9-(4-(trifluoromethyl)phenyl)phenanthrene (3ag):

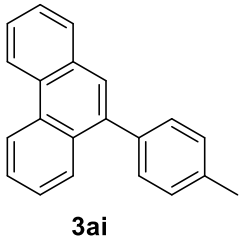
Synthesis carried out according to the General Procedure C, compound **3ag** was obtained in 71% yield as a white solid (114.3 mg, 0.355 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.78 (d, *J* = 8.2 Hz, 1H), 8.73 (d, *J* = 8.1 Hz, 1H), 7.89 (d, *J* = 7.6 Hz, 1H), 7.81 (d, *J* = 8.2 Hz, 1H), 7.77 (d, *J* = 7.8 Hz, 2H), 7.72 – 7.59 (m, 6H), 7.55 (t, *J* = 7.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.6, 137.3, 131.3, 130.7, 130.6, 130.4, 130.2, 129.6 (d, *J* = 32.2 Hz), 128.8, 127.8, 127.1, 127.0, 126.8, 126.5, 125.3 (q, *J* = 3.6 Hz), 124.3 (d, *J* = 270 Hz), 123.1, 122.6. ^{19}F NMR (377 MHz, $CDCl_3$) δ -62.34. IR (KBr) ν_{max} 2930, 1614, 1323, 1165, 1124, 1065, 893, 746 cm^{-1}. HRMS (APCI) calculated for $C_{21}H_{13}F_3$ $[M]^+$: 322.0969, found: 322.0973. These spectral data correspond to previously reported data.[5]



3ah

9-(4-methoxyphenyl)phenanthrene (3ah):

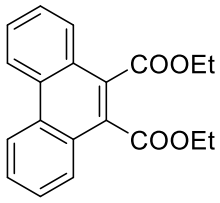
Synthesis carried out according to the General Procedure C, compound **3ah** was obtained in 56% yield as a white solid (79.6 mg, 0.28 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.77 (d, *J* = 8.3 Hz, 1H), 8.71 (d, *J* = 8.1 Hz, 1H), 7.94 (d, *J* = 8.2 Hz, 1H), 7.88 (d, *J* = 7.0 Hz, 1H), 7.69 – 7.58 (m, 4H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.47 (d, *J* = 8.6 Hz, 2H), 7.05 (d, *J* = 8.6 Hz, 2H), 3.91 (s, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 138.4, 133.2, 131.7, 131.4, 131.1, 130.7, 129.9, 128.6, 127.5, 127.0, 126.8, 126.5, 126.4, 122.9, 122.5, 113.7, 55.4. IR (KBr) ν_{max} 2956, 2924, 2316, 1509, 1456, 1245, 832, 749 cm^{-1}. HRMS (APCI) calculated for $C_{21}H_{16}O$ $[M]^+$: 284.1201, found: 284.1205. These spectral data correspond to previously reported data.[5]


3ai

9-(p-tolyl)phenanthrene (3ai):

Synthesis carried out according to the General Procedure C, compound **3ai** was obtained in 53% yield as a white solid (71 mg, 0.265 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.78 (d, *J* = 8.3 Hz, 1H), 8.73 (d, *J* = 8.2 Hz, 1H), 7.94 (d, *J* = 8.2 Hz, 1H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.67 (dd, *J* = 8.5, 6.2 Hz, 3H), 7.64 – 7.58 (m, 1H), 7.56 – 7.51 (m, 1H), 7.45 (d, *J* = 7.9 Hz, 2H), 7.33 (d, *J* = 7.8 Hz, 2H), 2.48 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.7, 137.8, 137.0, 131.6, 131.2, 130.6, 129.9, 129.9, 129.0, 128.6, 127.4, 127.0, 126.8, 126.5, 126.4, 126.4, 122.9, 122.5, 21.27. IR (KBr) ν_{max} 2923, 2855, 1505, 1450, 1241, 892, 818, 747, 723 cm^{-1}. HRMS (APCI) calculated

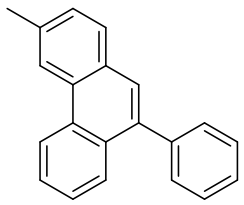
for $C_{21}H_{16}$ $[M]^+$: 268.1252, found: 268.1255. These spectral data correspond to previously reported data.[1]



3aj

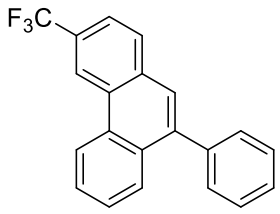
diethyl phenanthrene-9,10-dicarboxylate (3aj):

Synthesis carried out according to the General Procedure C, compound **3aj** was obtained in 43% yield as a yellow solid (69.2 mg, 0.215 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, J = 8.3 Hz, 1H), 8.17 (d, J = 8.2 Hz, 1H), 7.75 (t, J = 7.6 Hz, 1H), 7.67 (t, J = 7.6 Hz, 1H), 4.52 (q, J = 7.1 Hz, 2H), 1.46 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.0, 131.0, 129.9, 128.4, 127.6, 127.2, 126.8, 122.9, 62.0, 14.2. IR (KBr) ν_{max} 2958, 2921, 1723, 1523, 1476, 1373, 1250, 1189, 1020, 800, 757 cm^{-1}. HRMS (ESI) calculated for $C_{20}H_{19}O_4$ $[M+H]^+$: 323.1278 found: 323.1281. These spectral data correspond to previously reported data.[1]


3ba

3-methyl-9-phenylphenanthrene (3ba):

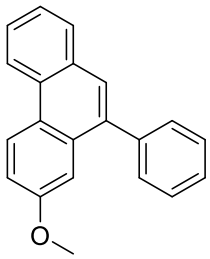
Synthesis carried out according to the General Procedure C, compound **3ba** was obtained in 54% yield as a yellow solid (72.4 mg, 0.27 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.76 (d, J = 8.3 Hz, 1H), 8.51 (s, 1H), 7.90 (d, J = 8.2 Hz, 1H), 7.78 (d, J = 8.1 Hz, 1H), 7.69 – 7.60 (m, 2H), 7.52 (dt, J = 14.9, 7.4 Hz, 5H), 7.46 – 7.41 (m, 2H), 2.64 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.9, 136.7, 135.3, 130.2, 129.3, 129.1, 128.9, 128.5, 127.6, 127.5, 127.2, 126.3, 126.2, 125.8, 125.3, 125.2, 121.8, 121.2, 21.2. IR (KBr) ν_{max} 2954, 2920, 1497, 1448, 890, 766, 701, 584 cm^{-1}. HRMS (APCI) calculated for $C_{21}H_{16}$ $[M]^+$: 268.1252, found: 268.1256. These spectral data correspond to previously reported data.[4]



3ca

9-phenyl-3-(trifluoromethyl)phenanthrene (3ca):

Synthesis carried out according to the General Procedure C, compound **3ca** was obtained in 71% yield as a yellow solid (114.3 mg, 0.355 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.00 (s, 1H), 8.80 (d, J = 8.3 Hz, 1H), 8.00 (d, J = 8.2 Hz, 1H), 7.95 (d, J = 8.2 Hz, 1H), 7.81 (d, J = 8.3 Hz, 1H), 7.78 – 7.72 (m, 2H), 7.61 (t, J = 7.6 Hz, 1H), 7.54 (d, J = 3.6 Hz, 4H), 7.40 – 7.33 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.3, 140.2, 133.5, 132.5, 131.5, 130.5, 129.9, 129.5, 129.4, 128.4, 128.0 (d, J = 32.6 Hz), 127.8, 127.4, 127.3, 127.2, 126.7, 124.7 (d, J = 272.1 Hz), 123.0, 122.8 (d, J = 3.2 Hz), 120.2 (q, J = 4.3 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ -61.72 (s). IR (KBr) ν_{max} 2956, 2920, 1706, 1458, 1321, 1127, 1074, 888, 764, 698 cm^{-1}. HRMS (APCI) calculated for $C_{21}H_{13}F_3$ $[M]^+$: 322.0969, found: 322.0973. These spectral data correspond to previously reported data.[7]

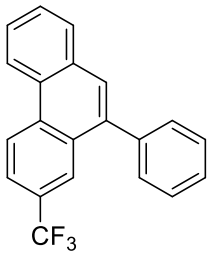


3da

2-methoxy-10-phenylphenanthrene (3da):

Synthesis carried out according to the General Procedure C, compound **3da** was obtained in 62% yield as a yellow solid (88 mg, 0.31 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 (d, J = 8.9 Hz, 1H), 8.62 (d, J = 8.3 Hz, 1H), 7.87 (d, J = 7.8 Hz, 1H), 7.72 – 7.62 (m, 2H), 7.55 (dd, J = 13.7, 7.2 Hz, 5H), 7.49 – 7.43 (m, 1H), 7.36 – 7.29 (m, 2H), 3.81 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.2, 140.9, 138.3, 132.6, 130.59, 129.9, 128.7, 128.4, 128.2, 127.4, 126.7, 125.9, 125.0, 124.5, 122.0, 116.4, 114.2, 107.9, 55.3. IR (KBr) ν_{max} 2925, 2853, 1613,

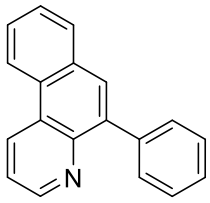
1529, 1460, 1218, 1154, 1041, 821, 773, 701 cm^{-1}. HRMS (APCI) calculated for $C_{21}H_{16}O$ $[M]^+$: 284.1201, found: 284.1205. These spectral data correspond to previously reported data.[5]



3ea

10-phenyl-2-(trifluoromethyl)phenanthrene (3ea):

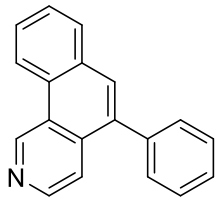
Synthesis carried out according to the General Procedure C, compound **3ea** was obtained in 73% yield as a yellow solid (117.5 mg, 0.365 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.85 (d, *J* = 8.7 Hz, 1H), 8.72 (d, *J* = 7.8 Hz, 1H), 8.21 (s, 1H), 7.92 (d, *J* = 7.6 Hz, 1H), 7.85 (d, *J* = 8.6 Hz, 1H), 7.77 (s, 1H), 7.73 – 7.64 (m, 2H), 7.60 – 7.43 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8, 138.7, 132.7, 132.2, 130.6, 123.0, 129.2, 128.9, 128.9, 128.6, 128.3 (d, *J* = 32.5 Hz), 128.0, 127.8, 127.1, 124.4 (d, *J* = 272.3 Hz), 124.2 (q, *J* = 4.2 Hz), 123.8, 122.9, 122.3 (q, *J* = 3.3 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ -61.96. IR (KBr) ν_{max} 2834, 1325, 1311, 1248, 1158, 1123, 748, 702 cm^{-1}. HRMS (APCI) calculated for $C_{21}H_{13}F_3$ $[M]^+$: 322.0969, found: 322.0976. These spectral data correspond to previously reported data.[8]



3fa

5-phenylbenzo[f]quinoline (3fa):

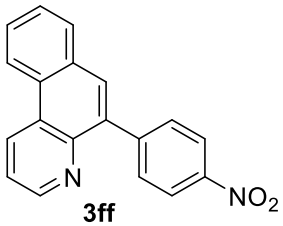
Synthesis carried out according to the General Procedure C, compound **3fa** was obtained in 29.3% yield as a yellow solid (37.56 mg, 0.147 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.04 (dd, *J* = 8.4, 1.5 Hz, 1H), 9.01 (dd, *J* = 4.3, 1.6 Hz, 1H), 8.67 (d, *J* = 8.0 Hz, 1H), 8.01 (s, 1H), 8.00 – 7.96 (m, 1H), 7.79 – 7.73 (m, 2H), 7.69 (ddd, *J* = 13.3, 7.4, 3.7 Hz, 2H), 7.59 (dd, *J* = 8.3, 4.3 Hz, 1H), 7.53 (t, *J* = 7.5 Hz, 2H), 7.44 (t, *J* = 7.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.4, 146.8, 139.8, 139.4, 131.5, 131.2, 130.8, 130.6, 129.6, 128.8, 128.0, 127.5, 127.4, 127.0, 125.8, 122.4, 121.1. IR (KBr) ν_{max} 2956, 2927, 1483, 1451, 1371, 896, 790, 750, 698 cm^{-1}. HRMS (ESI) calculated for $C_{19}H_{14}N$ $[M+H]^+$: 256.1121, found: 256.1123. These spectral data correspond to previously reported data.[4]



3fa'

5-phenylbenzo[h]isoquinoline (3fa'):

Synthesis carried out according to the General Procedure C, compound **3fa'** was obtained in 35.8% yield as a yellow solid (45.6 mg, 0.18 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.13 (s, 1H), 8.84 (d, *J* = 8.2 Hz, 1H), 8.67 (d, *J* = 5.3 Hz, 1H), 7.94 (d, *J* = 7.8 Hz, 1H), 7.90 (s, 1H), 7.76 (t, *J* = 5.7 Hz, 2H), 7.69 (t, *J* = 7.3 Hz, 1H), 7.58 – 7.46 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.0, 145.2, 139.0, 137.0, 135.1, 131.7, 131.7, 129.9, 129.0, 128.9, 128.6, 127.9, 127.8, 127.7, 125.2, 121.8, 119.4. IR (KBr) ν_{max} 2957, 2920, 1459, 1375, 1070, 888 cm^{-1}. HRMS (ESI) calculated for $C_{19}H_{14}N$ $[M+H]^+$: 256.1121, found: 256.1125. These spectral data correspond to previously reported data.[4]

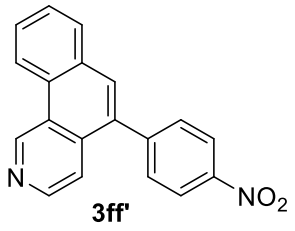


3ff

5-(4-nitrophenyl)benzo[f]quinoline (3ff):

Synthesis carried out according to the General Procedure C, compound **3ff** was obtained in 50% yield as a yellow solid (74.8 mg, 0.25 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.06 (d, *J* = 8.4 Hz, 1H), 8.98 (d, *J* = 3.1 Hz, 1H), 8.68 (d, *J* = 8.1 Hz, 1H), 8.37 (d, *J* = 8.7 Hz, 2H), 8.04 (s, 1H), 8.00 (d, *J* = 7.7 Hz, 1H), 7.93 (d, *J* = 8.7 Hz, 2H), 7.74 (dt, *J* = 14.6, 7.1 Hz, 2H), 7.63 (dd, *J* = 8.3, 4.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.5, 147.1, 146.6, 146.0,

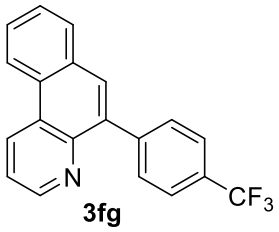
137.0, 131.7, 131.5, 131.1, 123.0, 129.1, 127.9, 127.8, 126.0, 123.2, 122.6, 121.7. IR (KBr) ν_{max} 2921, 1514, 1346, 850, 741, 697 cm^{-1}. HRMS (ESI) calculated for $C_{19}H_{13}N_2O_2$ $[M+H]^+$: 301.0972, found: 301.0977.



3ff'

5-(4-nitrophenyl)benzo[h]isoquinoline (3ff'):

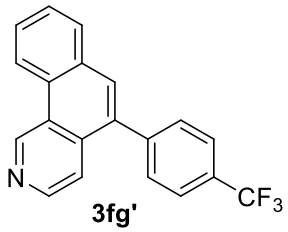
Synthesis carried out according to the General Procedure C, compound **3ff'** was obtained in 36% yield as a yellow solid (54.2 mg, 0.18 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.16 (s, 1H), 8.87 (d, *J* = 8.3 Hz, 1H), 8.71 (d, *J* = 5.6 Hz, 1H), 8.42 (d, *J* = 8.5 Hz, 2H), 7.98 (d, *J* = 7.8 Hz, 1H), 7.93 (s, 1H), 7.82 (t, *J* = 7.6 Hz, 1H), 7.73 (d, *J* = 8.4 Hz, 3H), 7.63 (d, *J* = 5.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 147.3, 145.9, 145.7, 134.7, 134.2, 132.3, 131.3, 130.9, 129.3, 128.7, 128.1, 125.3, 123.9, 122.0, 118.7. IR (KBr) ν_{max} 2957, 2318, 1511, 1348, 702 cm^{-1}. HRMS (ESI) calculated for $C_{19}H_{13}N_2O_2$ $[M+H]^+$: 301.0972, found: 301.0975.



3fg

5-(4-(trifluoromethyl)phenyl)benzo[f]quinoline (3fg):

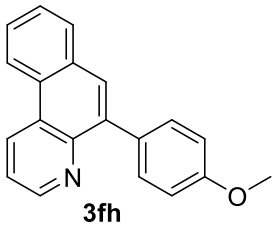
Synthesis carried out according to the General Procedure C, compound **3fg** was obtained in 39% yield as a yellow solid (63 mg, 0.195 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.04 (d, *J* = 8.3 Hz, 1H), 8.98 (d, *J* = 2.9 Hz, 1H), 8.67 (d, *J* = 7.9 Hz, 1H), 8.05 – 7.96 (m, 2H), 7.87 (d, *J* = 7.7 Hz, 2H), 7.80 – 7.67 (m, 4H), 7.61 (dd, *J* = 7.9, 4.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.5, 146.3, 143.4, 137.9, 131.6, 131.2, 131.0, 130.9, 129.8, 129.4 (d, *J* = 32.3 Hz), 129.0, 127.7, 127.5, 125.9, 124.9 (d, *J* = 3.7 Hz), 124.5 (d, *J* = 270 Hz), 122.5, 121.5.^{19}F NMR (377 MHz, $CDCl_3$) δ -62.41 (s). IR (KBr) ν_{max} 2919, 1324, 1162, 1111, 841, 745, 617 cm^{-1}. HRMS (ESI) calculated for $C_{20}H_{13}F_3N$ $[M+H]^+$: 324.0995, found: 324.0998. These spectral data correspond to previously reported data.[9]



3fg'

5-(4-(trifluoromethyl)phenyl)benzo[h]isoquinoline (3fg'):

Synthesis carried out according to the General Procedure C, compound **3fg'** was obtained in 28% yield as a yellow solid (45.2 mg, 0.14 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.15 (s, 1H), 8.86 (d, *J* = 8.3 Hz, 1H), 8.69 (d, *J* = 5.6 Hz, 1H), 7.97 (d, *J* = 7.8 Hz, 1H), 7.91 (s, 1H), 7.80 (dd, *J* = 12.1, 4.6 Hz, 3H), 7.73 (dd, *J* = 11.0, 3.9 Hz, 1H), 7.67 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 145.4, 142.8, 135.6, 134.6, 132.1, 131.5, 130.3, 130.2 (d, *J* = 32.5 Hz), 129.2, 128.4, 128.0, 125.6 (q, *J* = 3.7 Hz), 124.0 (d, *J* = 246.2 Hz), 121.9, 119.0. ^{19}F NMR (377 MHz, $CDCl_3$) δ -62.49. IR (KBr) ν_{max} 2957, 1325, 1110, 1020, 800, 747 cm^{-1}. HRMS (ESI) calculated for $C_{20}H_{13}F_3N$ $[M+H]^+$: 324.0995, found: 324.0996.

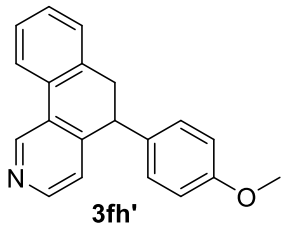


3fh

5-(4-methoxyphenyl)benzo[f]quinoline (3fh):

Synthesis carried out according to the General Procedure C, compound **3fh** was obtained in 36% yield as a yellow solid (51.3 mg, 0.18 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.02 (ddd, *J* = 6.4, 6.0, 1.5 Hz, 2H), 8.65 (d, *J* = 7.7 Hz, 1H), 7.99 (s, 1H), 7.98–7.93 (m, 1H), 7.75–7.63 (m, 4H), 7.58 (dd, *J* = 8.3, 4.3 Hz, 1H), 7.07 (d, *J* = 8.7 Hz, 2H), 3.90 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 149.3, 146.8, 138.8, 132.0, 131.7, 131.6, 130.9, 130.8, 129.4,

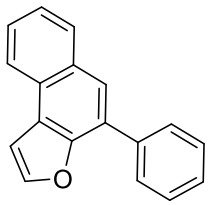
128.7, 127.5, 126.8, 125.9, 122.4, 121.1, 113.6, 55.4. IR (KBr) ν_{max} 2956, 1511, 1457, 1245, 1035, 829, 748 cm^{-1}. HRMS (ESI) calculated for $C_{20}H_{16}NO$ $[M+H]^+$: 286.1226, found: 286.1232. These spectral data correspond to previously reported data.[4]



3fh'

5-(4-methoxyphenyl)-5,6-dihydrobenzo[h]isoquinoline (3fh'):

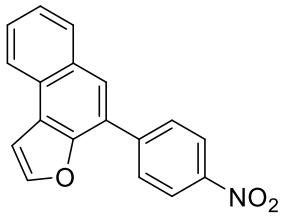
Synthesis carried out according to the General Procedure C, compound **3fh'** was obtained in 33% yield as a yellow solid (47 mg, 0.165 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.13 (s, 1H), 8.84 (d, J = 8.2 Hz, 1H), 8.67 (d, J = 5.5 Hz, 1H), 7.95 (d, J = 7.7 Hz, 1H), 7.89 (s, 1H), 7.83 – 7.73 (m, 2H), 7.69 (t, J = 7.1 Hz, 1H), 7.46 (d, J = 8.6 Hz, 2H), 7.08 (d, J = 8.6 Hz, 2H), 3.92 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4, 147.0, 145.1, 136.8, 135.5, 131.9, 131.6, 131.4, 131.0, 129.0, 128.9, 127.7, 125.4, 121.8, 119.5, 114.1, 55.4. IR (KBr) ν_{max} 2956, 2920, 1604, 1511, 1458, 1244, 1035, 832, 748 cm^{-1}. HRMS (ESI) calculated for $C_{20}H_{16}NO$ $[M+H]^+$: 286.1226, found: 286.1223. These spectral data correspond to previously reported data.[4]



3ga

4-phenylnaphtho[2,1-b]furan (3ga):

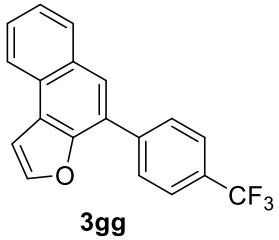
Synthesis carried out according to the General Procedure C, compound **3fh'** was obtained in 60% yield as a yellow solid (73.2 mg, 0.30 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, J = 8.1 Hz, 1H), 7.98 (dd, J = 16.3, 7.8 Hz, 3H), 7.90 – 7.82 (m, 2H), 7.56 (dq, J = 13.0, 7.4 Hz, 4H), 7.45 (t, J = 7.4 Hz, 1H), 7.34 (d, J = 1.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.5, 144.3, 136.5, 130.9, 128.8, 128.7, 128.0, 127.3, 126.7, 126.2, 124.9, 124.0, 123.5, 123.3, 105.8. IR (KBr) ν_{max} 2922, 1362, 1203, 1131, 1050, 878, 846, 743, 694 cm^{-1}. HRMS (ESI) calculated for $C_{18}H_{13}O$ $[M+H]^+$: 245.0961, found: 245.0958. These spectral data correspond to previously reported data.[4]



3gf

4-(4-nitrophenyl)naphtho[2,1-b]furan (3gf):

Synthesis carried out according to the General Procedure C, compound **3fh'** was obtained in 46% yield as a yellow solid (66.5 mg, 0.23 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (d, J = 8.8 Hz, 2H), 8.16 (t, J = 10.0 Hz, 3H), 8.02 (d, J = 8.1 Hz, 1H), 7.92 (s, 1H), 7.86 (s, 1H), 7.65 (t, J = 7.2 Hz, 1H), 7.54 (dd, J = 14.7, 7.7 Hz, 1H), 7.37 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.7, 146.3, 143.6, 142.0, 129.6, 128.4, 128.1, 126.9, 126.2, 124.3, 123.8, 123.1, 122.9, 122.9, 122.4, 105.0. IR (KBr) ν_{max} 2957, 2922, 1514, 1342, 851, 749 cm^{-1}. HRMS (ESI) calculated for $C_{18}H_{12}NO_3$ $[M+H]^+$: 290.0812, found: 290.0816.

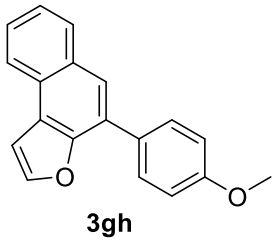


3gg

4-(4-(trifluoromethyl)phenyl)naphtho[2,1-b]furan (3gg):

Synthesis carried out according to the General Procedure C, compound **3fh'** was obtained in 70% yield as a yellow solid (109 mg, 0.35 mmol) after purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, J = 8.1 Hz, 1H), 8.08 (d, J = 8.1 Hz, 2H), 8.01 (d, J = 8.1 Hz, 1H), 7.88 (s, 1H), 7.84 (d, J = 1.5 Hz, 1H), 7.79 (d, J = 8.1 Hz, 2H), 7.63 (t, J = 7.5 Hz, 1H), 7.54 (t, J = 7.5 Hz, 1H), 7.36 (d, J = 1.7 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.0, 144.5, 140.1, 130.7, 129.9 (q, J = 32.4 Hz), 129.0, 129.0, 127.6, 126.8, 125.6 (q, J = 3.7 Hz), 125.2, 124.4, 124.3 (q, J = 270.0 Hz), 123.7, 123.3, 105.9. ^{19}F NMR (377 MHz, $CDCl_3$) δ -62.48. IR (KBr) ν_{max} 2957,

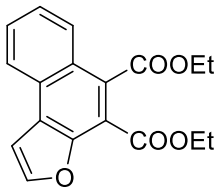
1324, 1166, 1124, 1067, 839, 747 cm^{-1}. HRMS (ESI) calculated for $C_{19}H_{12}F_3O$ $[M+H]^+$: 313.0835, found: 313.0839.



3gh

4-(4-methoxyphenyl)naphtho[2,1-b]furan (3gh):

Synthesis carried out according to the General Procedure C, compound **3gh** was obtained in 69% yield as a yellow solid (94.5 mg, 0.345 mmol) after purification. ^1H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, J = 8.1 Hz, 1H), 7.98 (d, J = 8.0 Hz, 1H), 7.95 – 7.89 (m, 2H), 7.83 (d, J = 2.1 Hz, 1H), 7.81 (s, 1H), 7.61 – 7.55 (m, 1H), 7.54 – 7.47 (m, 1H), 7.33 (d, J = 2.1 Hz, 1H), 7.11 – 7.06 (m, 2H), 3.90 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 150.5, 144.2, 130.9, 129.9, 128.9, 128.7, 127.0, 126.3, 125.9, 124.8, 123.4, 123.3, 123.2, 114.1, 105.8, 55.4. IR (KBr) ν_{max} 2975, 1511, 1456, 1250, 1046, 881, 747 cm^{-1}. HRMS (ESI) calculated for $C_{19}H_{15}O_2$ $[M+H]^+$: 275.1067, found: 275.1071. These spectral data correspond to previously reported data.[4]



3gj

diethyl naphtho[2,1-b]furan-4,5-dicarboxylate (3gj):

Synthesis carried out according to the General Procedure C, compound **3fh'** was obtained in 41% yield as a yellow solid (64.0 mg, 0.205 mmol) after purification. ^1H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, J = 8.2 Hz, 1H), 8.11 (d, J = 8.5 Hz, 1H), 7.92 (d, J = 1.3 Hz, 1H), 7.69 (t, J = 7.5 Hz, 1H), 7.59 (t, J = 7.7 Hz, 1H), 7.31 (d, J = 1.3 Hz, 1H), 4.52 (dd, J = 15.0, 7.4 Hz, 4H), 1.47 – 1.43 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 163.6, 145.5, 129.4, 128.1, 127.4, 126.0, 125.7, 125.2, 124.6, 122.7, 104.5, 61.0, 13.2, 13.1. IR (KBr) ν_{max} 2957, 2922, 1726, 1458, 1279, 1237, 1202, 750 cm^{-1}. HRMS (ESI) calculated for $C_{18}H_{17}O_5$ $[M+H]^+$: 313.1071, found: 313.1068.

5. References

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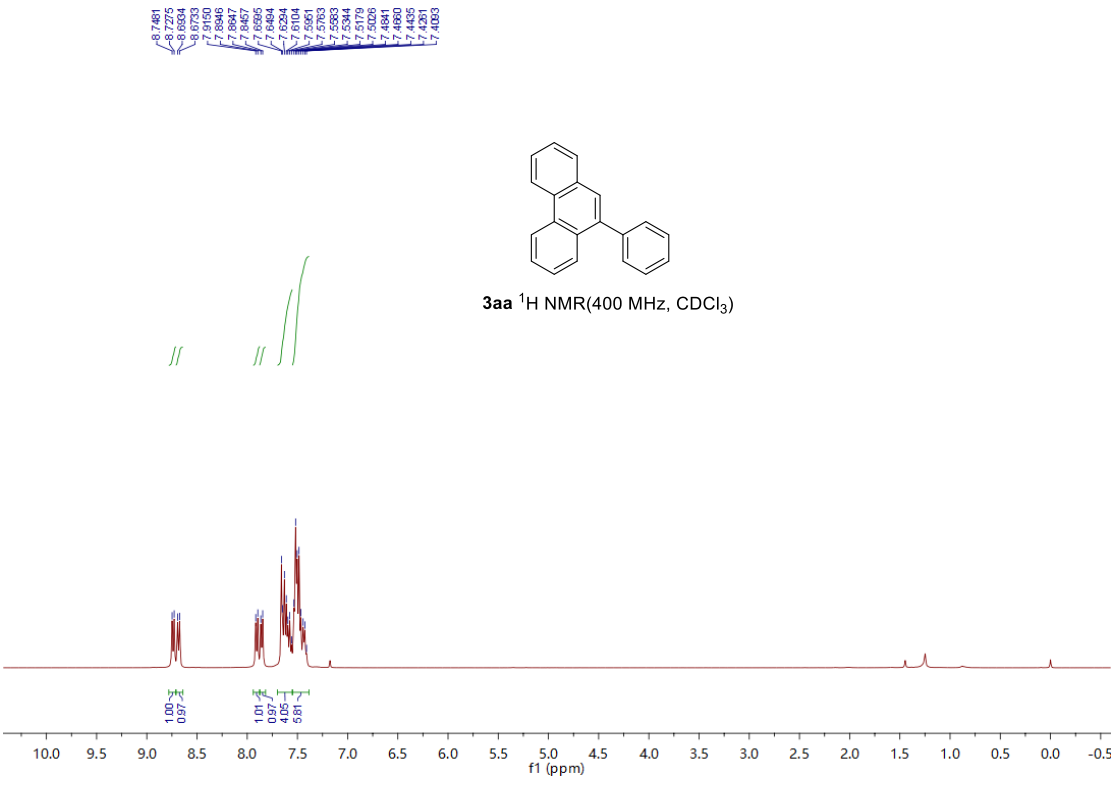
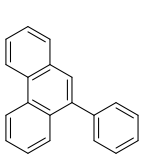
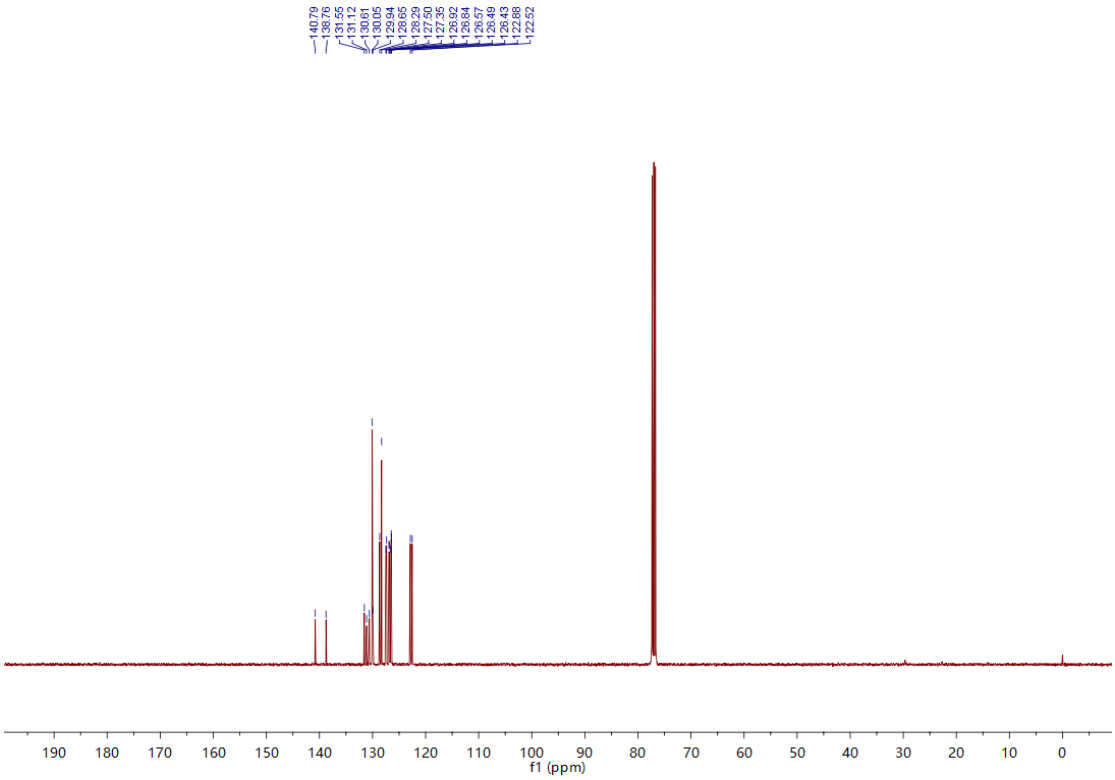
6. Copy of ^{1}H and ^{13}C NMR Spectra of Products

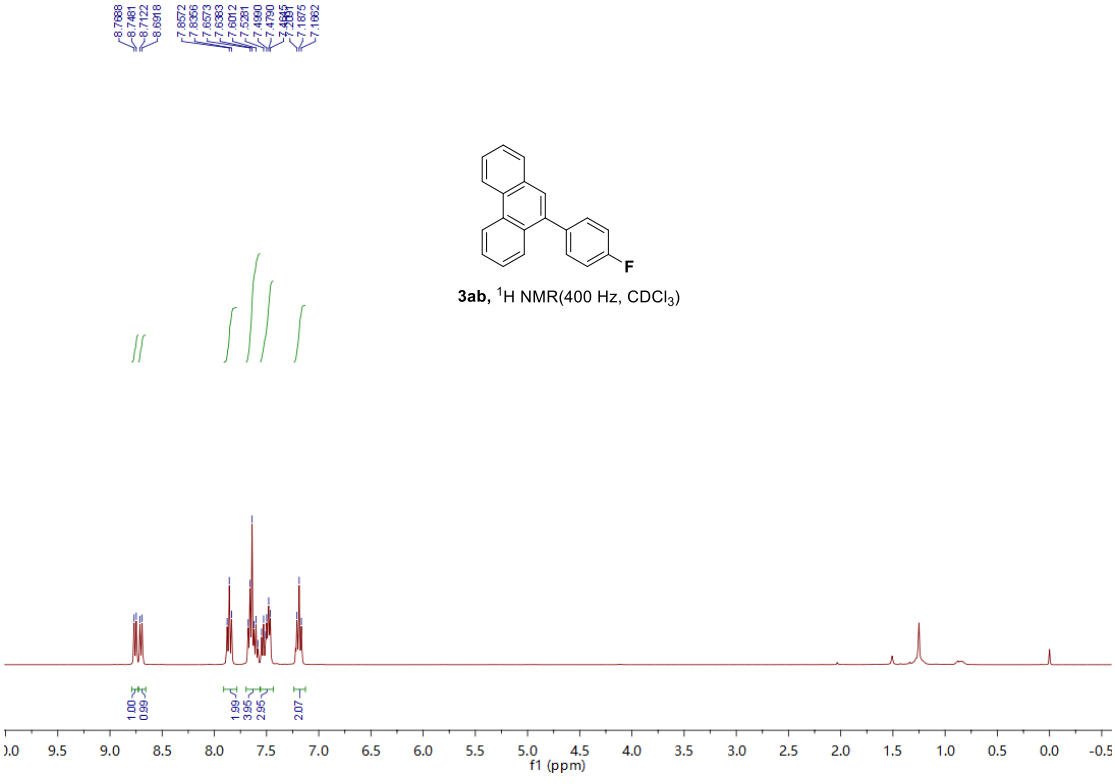


3aa ^{1}H NMR(400 MHz, $CDCl_3$)

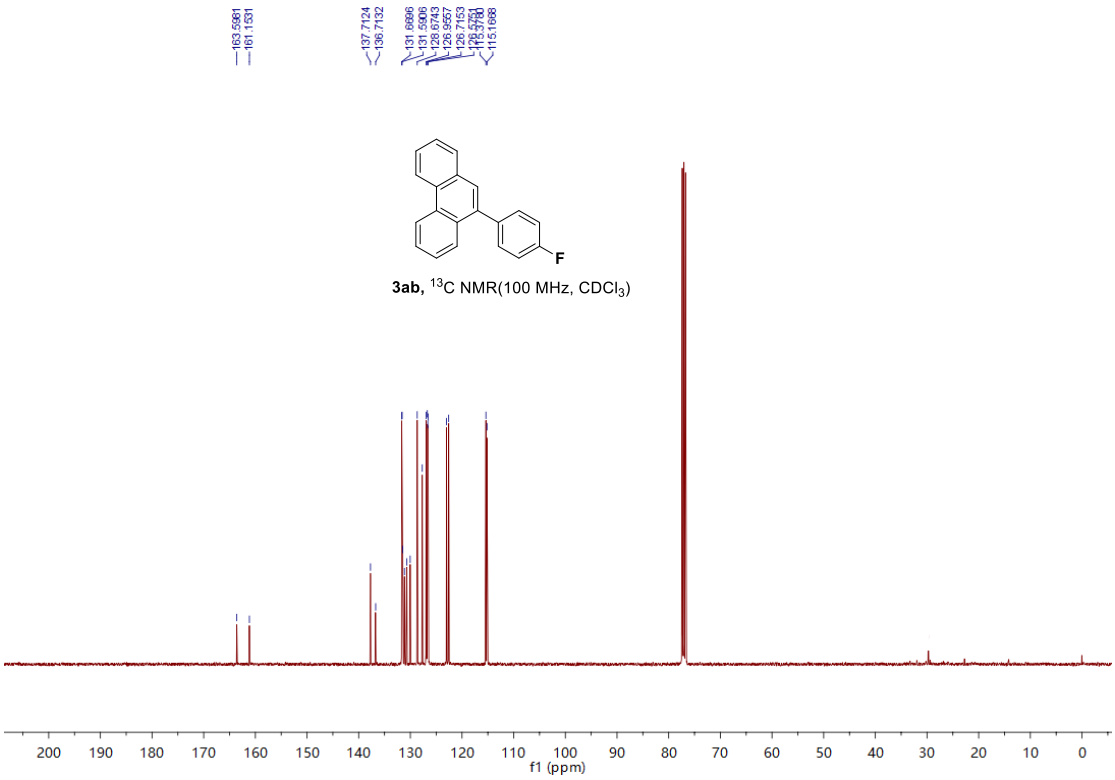


3aa ^{13}C NMR(100 MHz, $CDCl_3$)

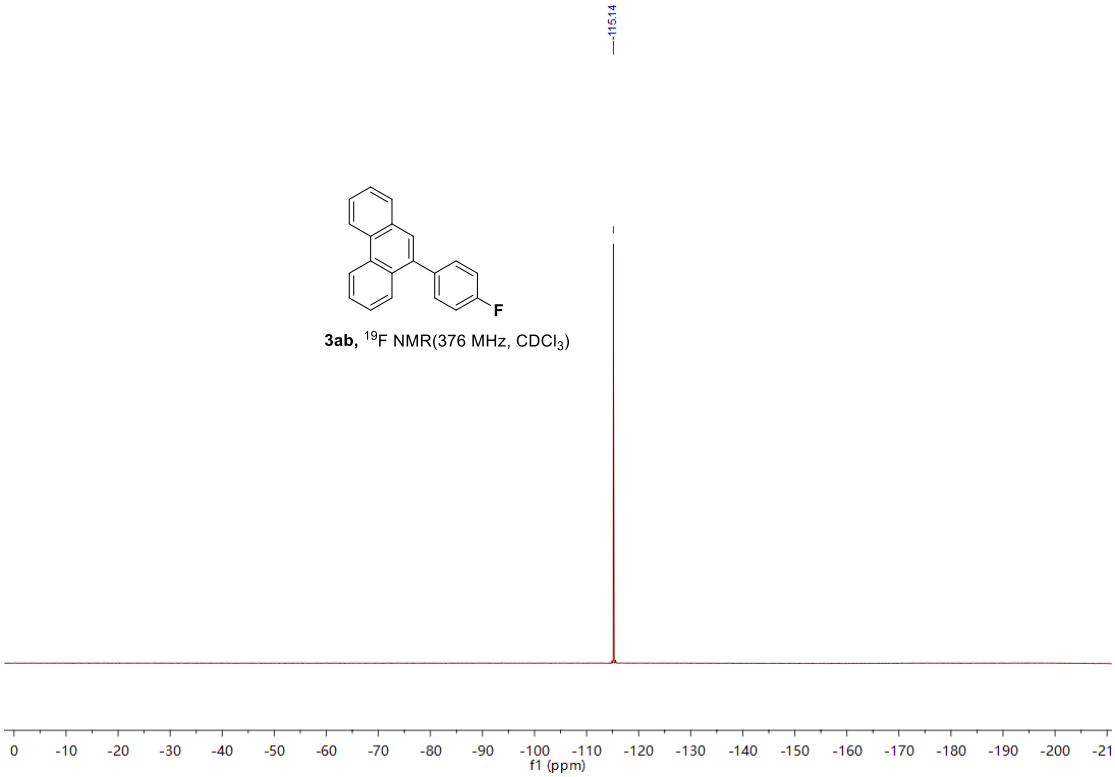




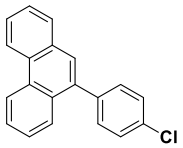
3ab, ^{1}H NMR(400 Hz, $CDCl_3$)



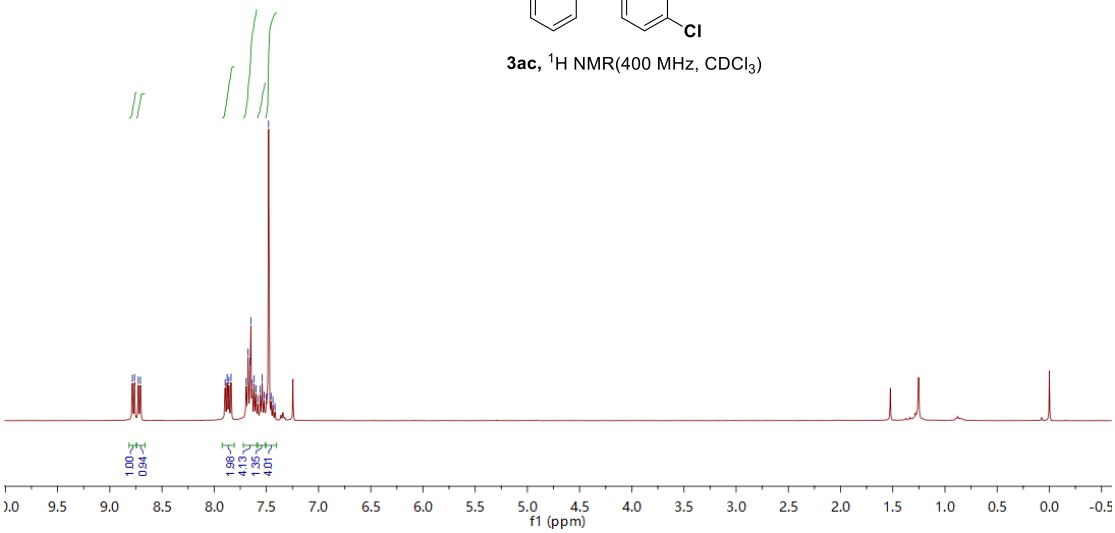
3ab, ^{13}C NMR(100 MHz, $CDCl_3$)

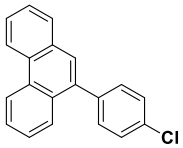


3ab, ^{19}F NMR(376 MHz, $CDCl_3$)

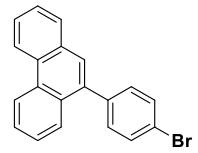


3ac, ^{1}H NMR(400 MHz, $CDCl_3$)

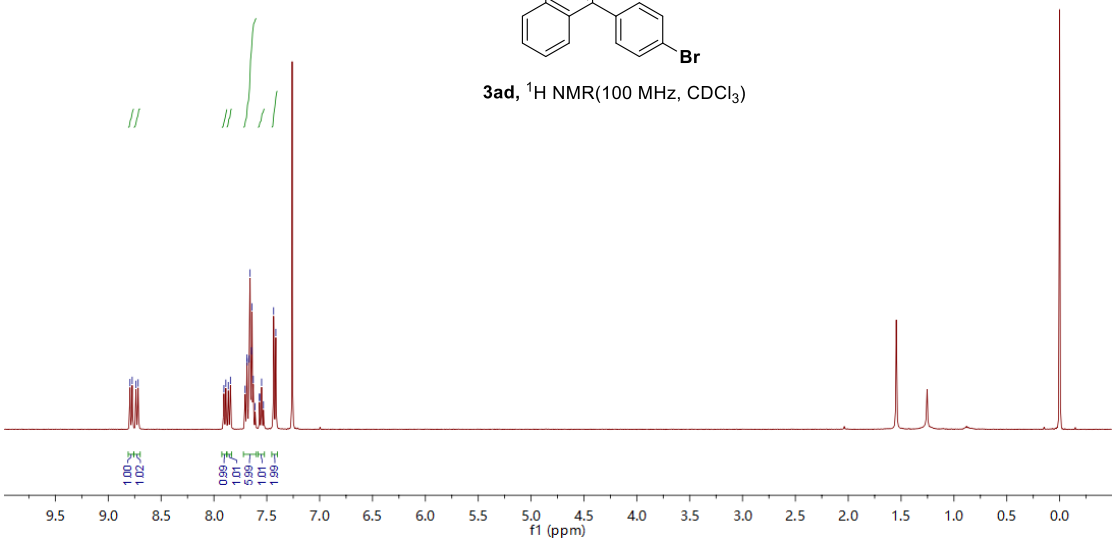


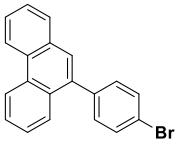


3ac, ^{13}C NMR(100 MHz, $CDCl_3$)

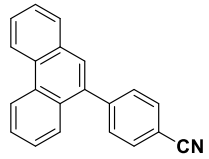


3ad, ^{1}H NMR(100 MHz, $CDCl_3$)

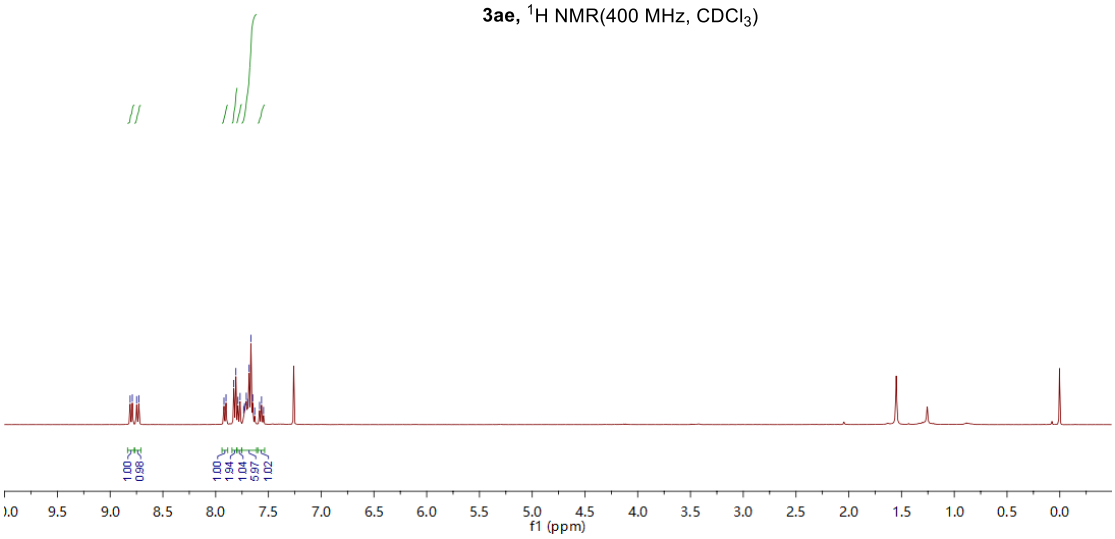


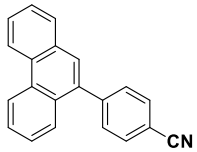


3ad, ^{13}C NMR(100 MHz, $CDCl_3$)

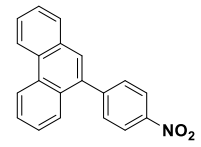


3ae, ^{1}H NMR(400 MHz, $CDCl_3$)

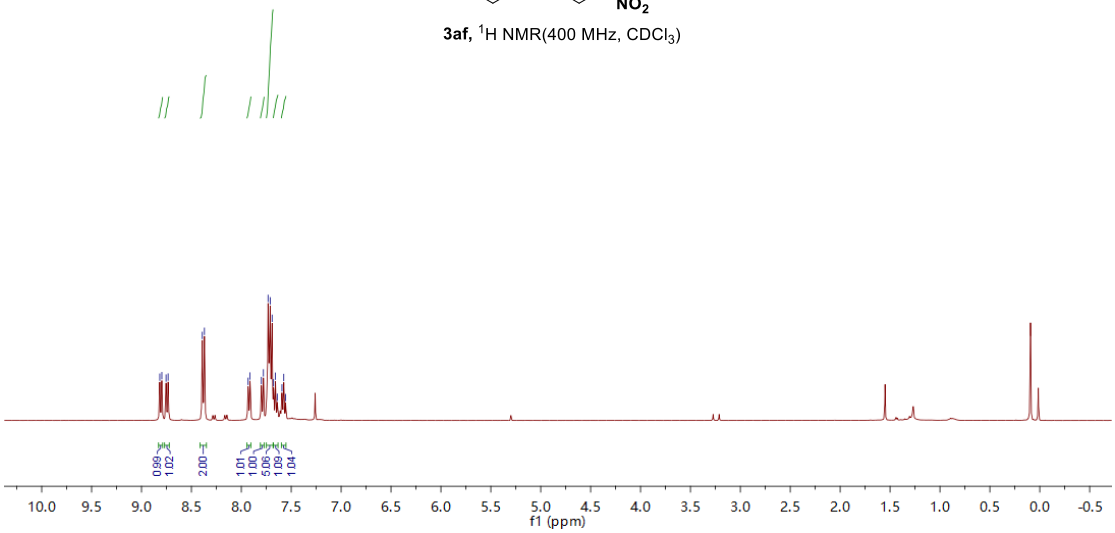


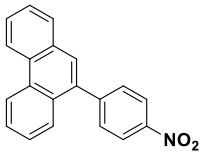


3ae, ^{13}C NMR(100 MHz, $CDCl_3$)

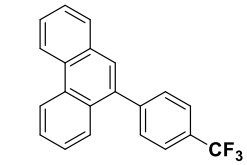


3af, ^{1}H NMR(400 MHz, $CDCl_3$)

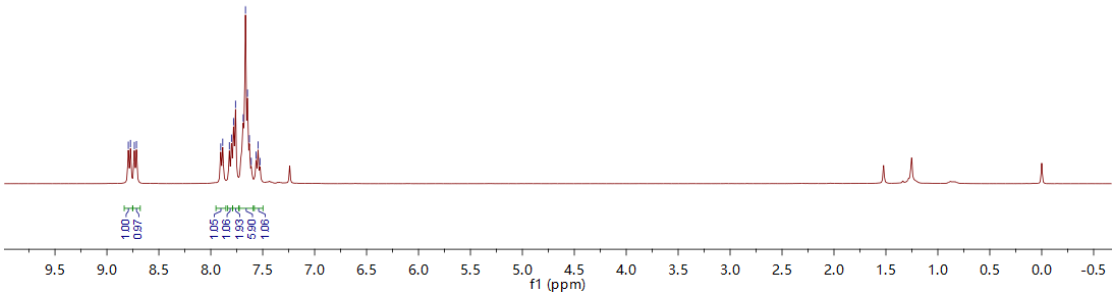


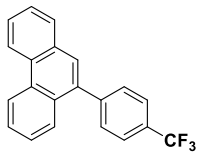


3af, ^{13}C NMR(100 MHz, $CDCl_3$)

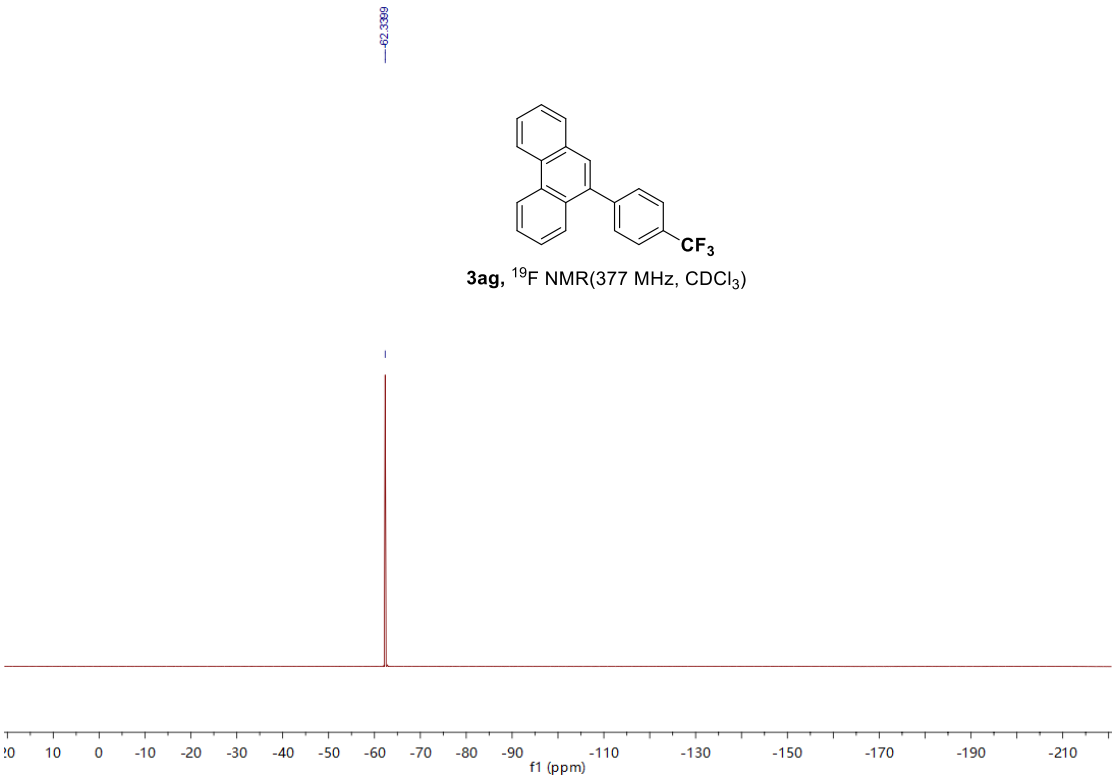


3ag, ^{1}H NMR(400 MHz, $CDCl_3$)

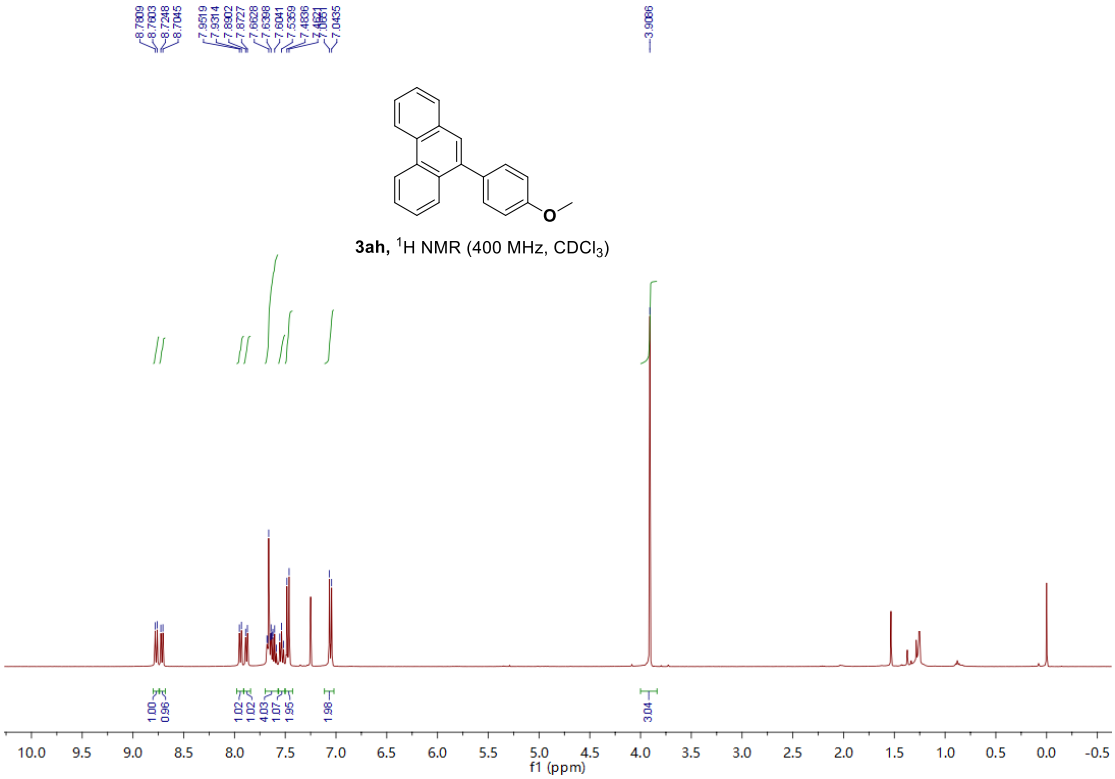




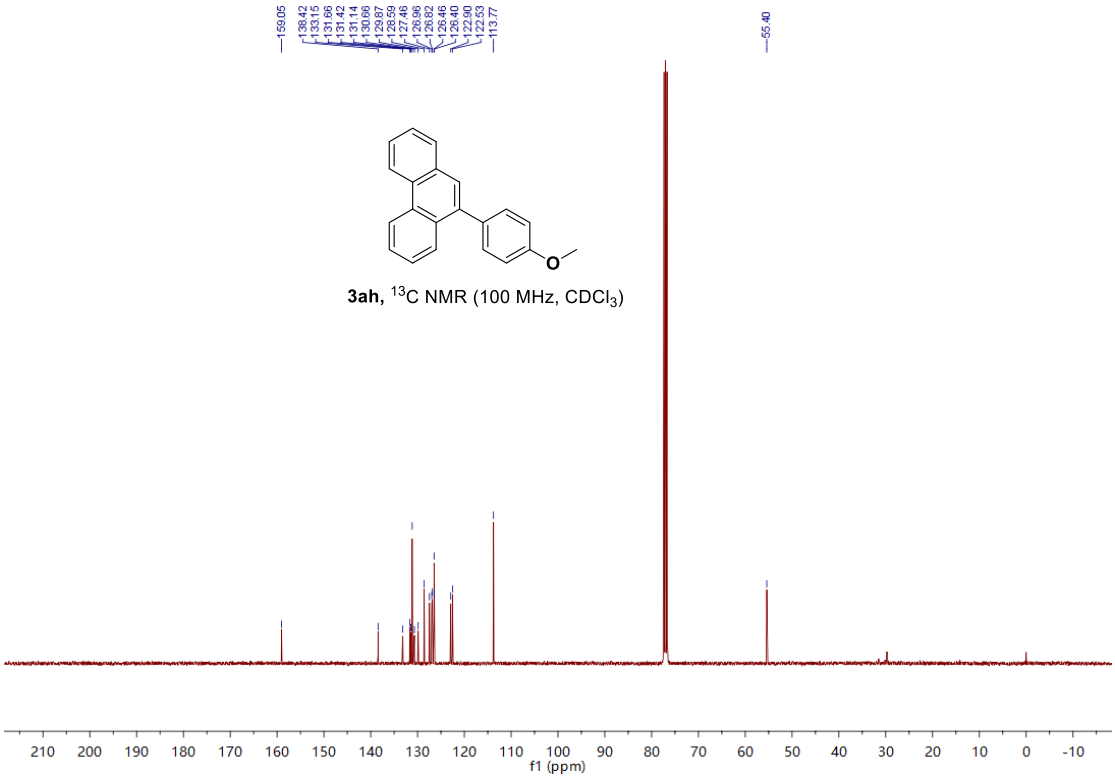
3ag, ^{13}C NMR(100 MHz, $CDCl_3$)



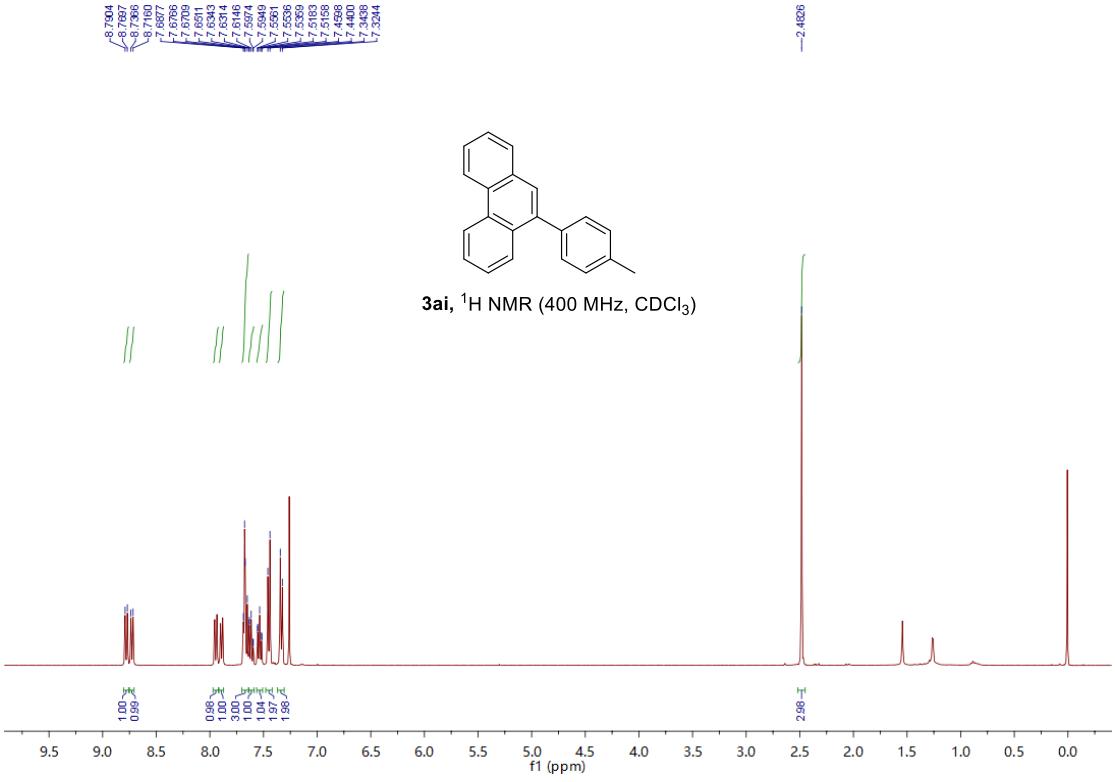
3ag, ^{19}F NMR(377 MHz, $CDCl_3$)



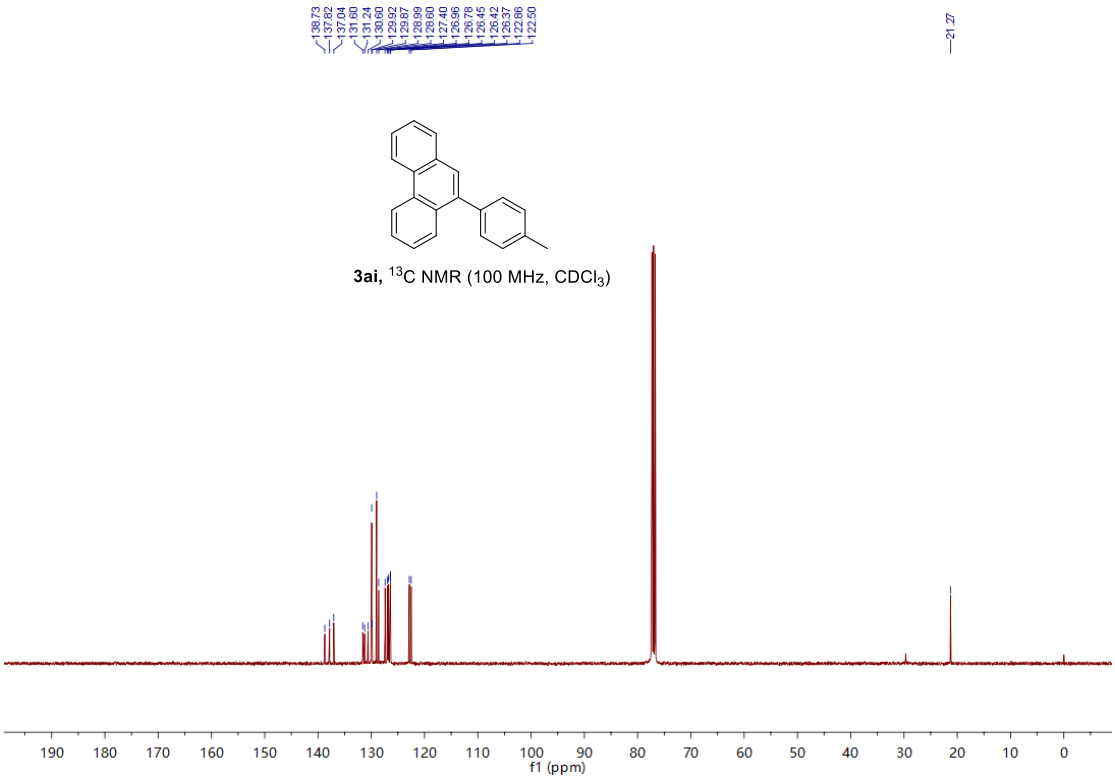
3ah, ^{1}H NMR (400 MHz, $CDCl_3$)



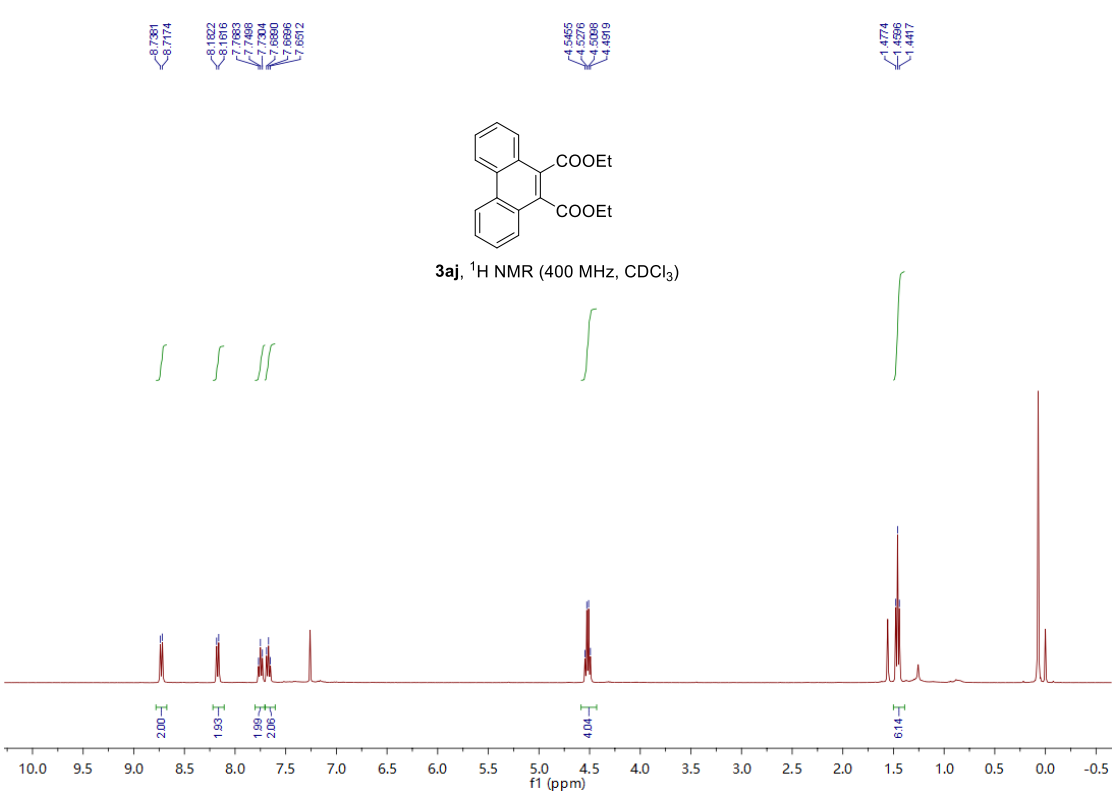
3ah, ^{13}C NMR (100 MHz, $CDCl_3$)



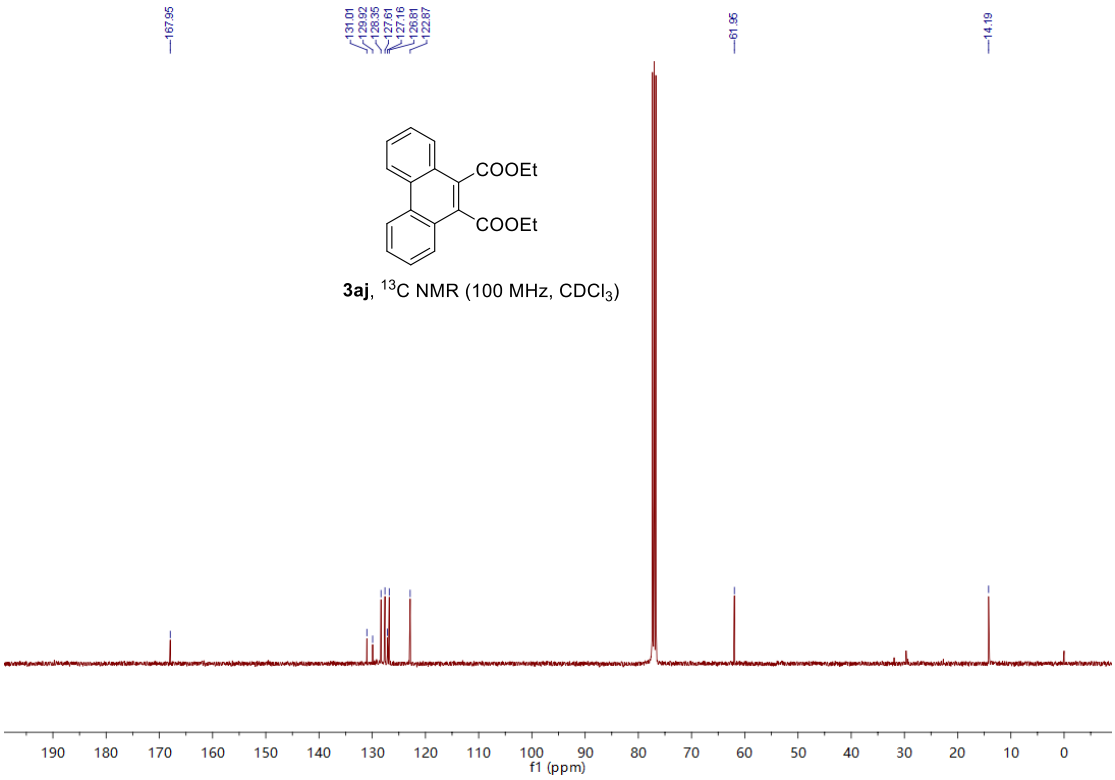
3ai, ^{1}H NMR (400 MHz, $CDCl_3$)



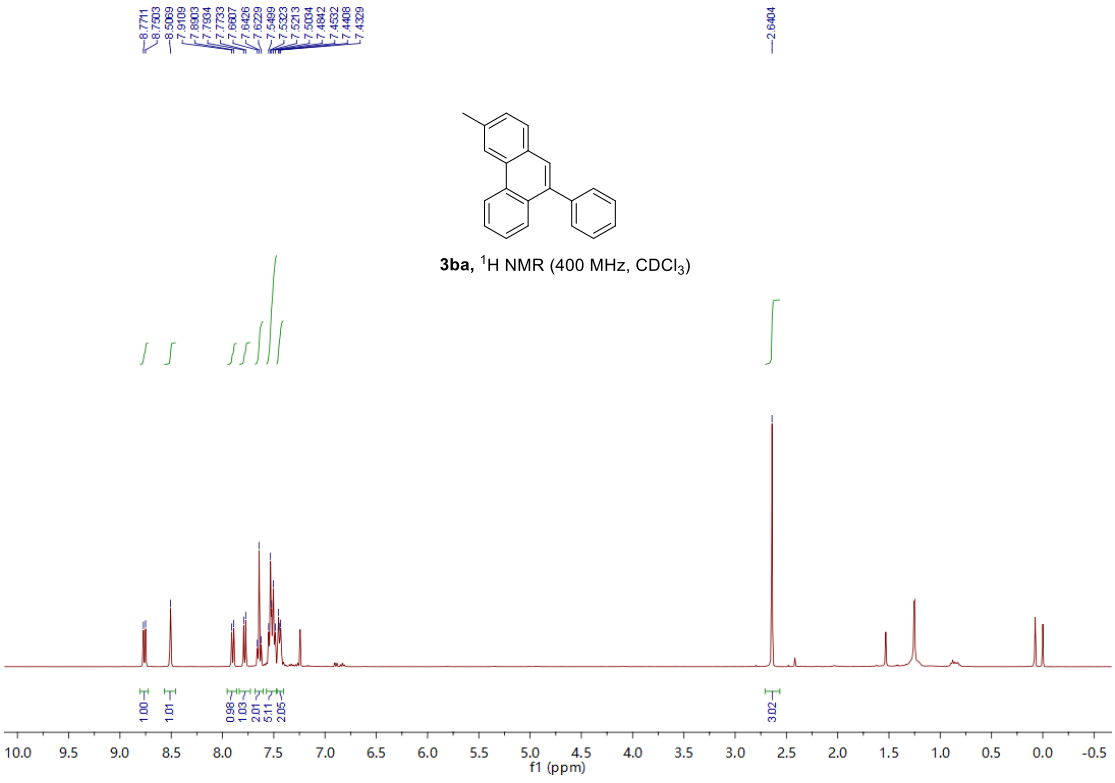
3ai, ^{13}C NMR (100 MHz, $CDCl_3$)



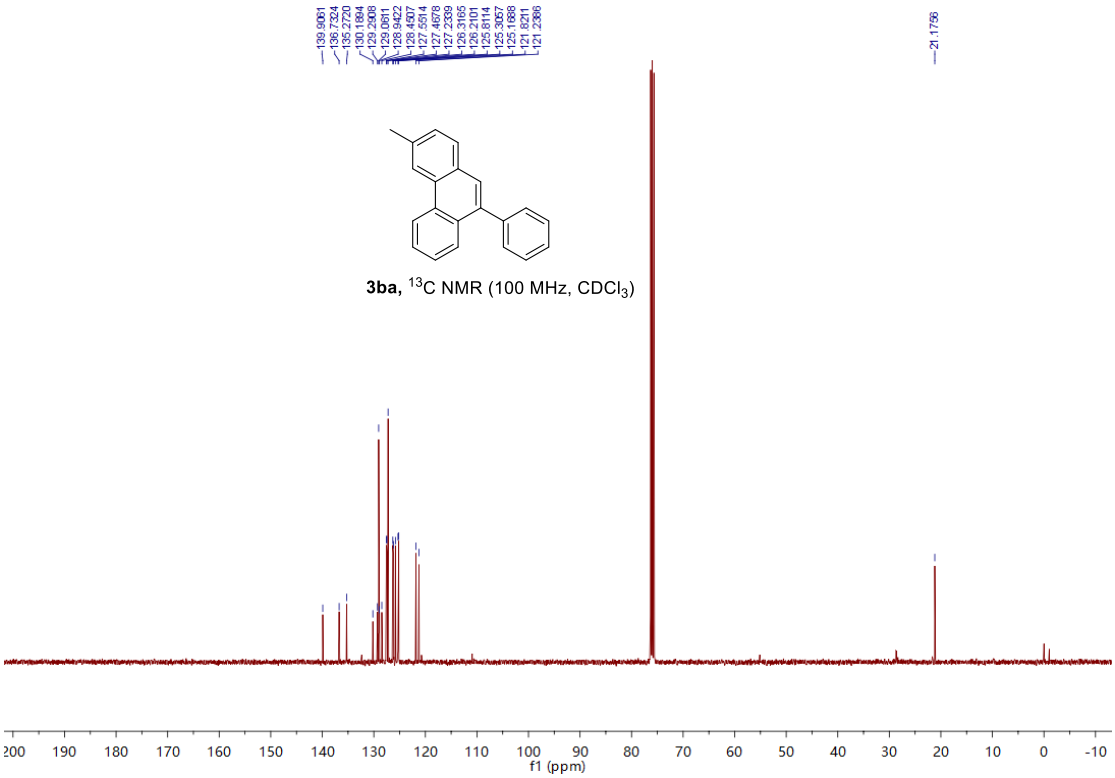
3aj, ^{1}H NMR (400 MHz, $CDCl_3$)



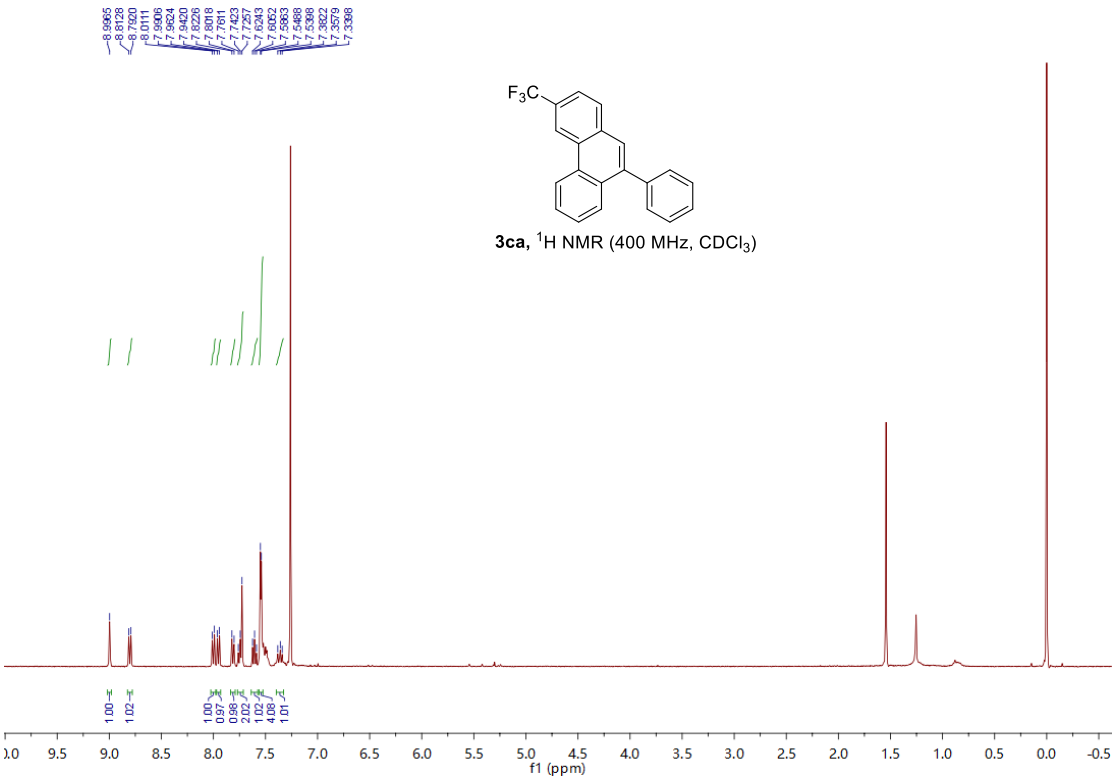
3aj, ^{13}C NMR (100 MHz, $CDCl_3$)



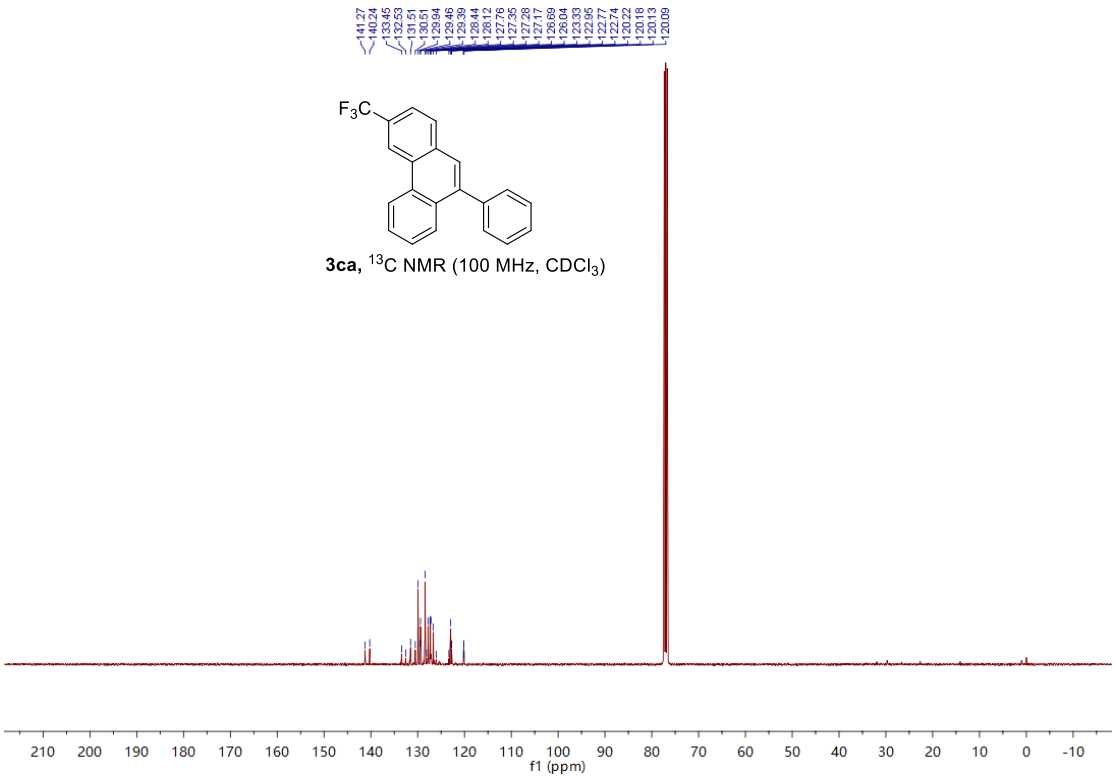
3ba, ^{1}H NMR (400 MHz, $CDCl_3$)



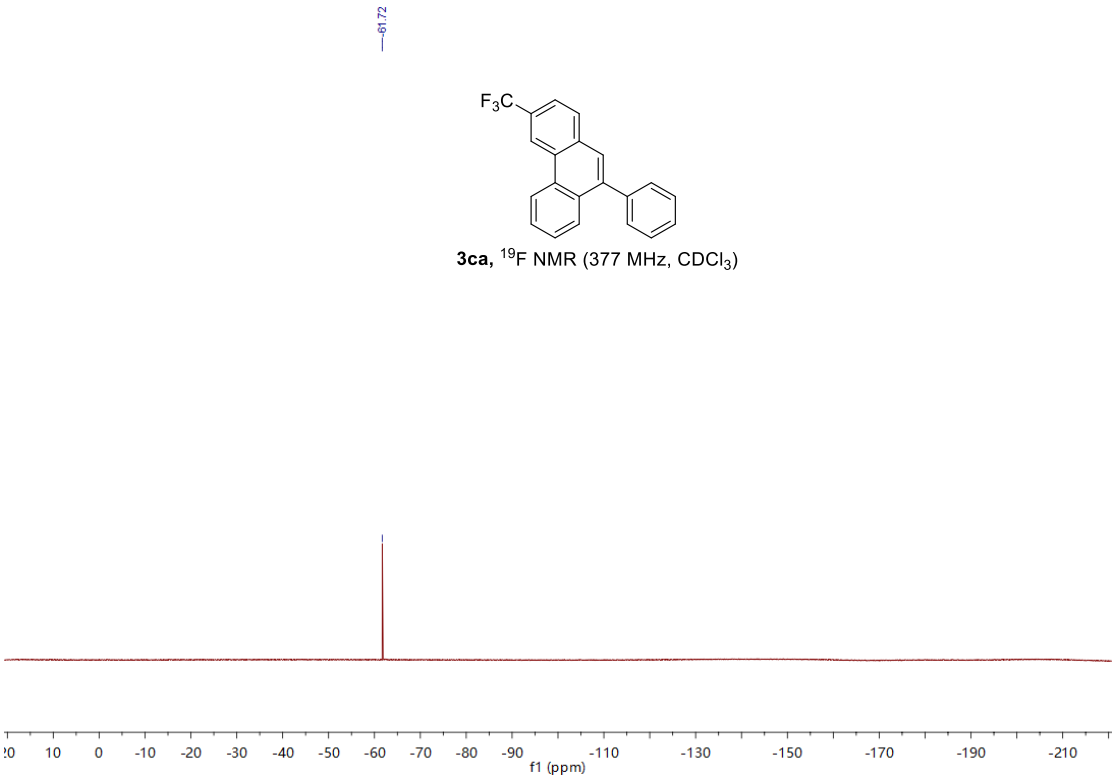
3ba, ^{13}C NMR (100 MHz, $CDCl_3$)



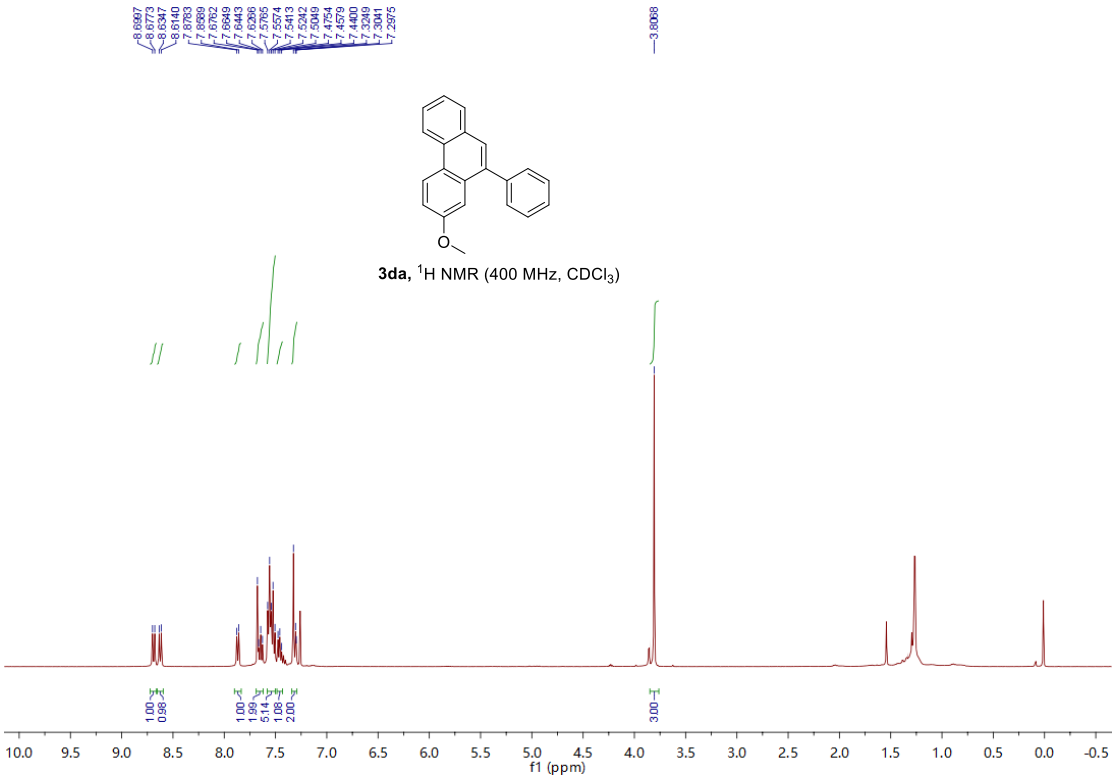
3ca, ^{1}H NMR (400 MHz, $CDCl_3$)



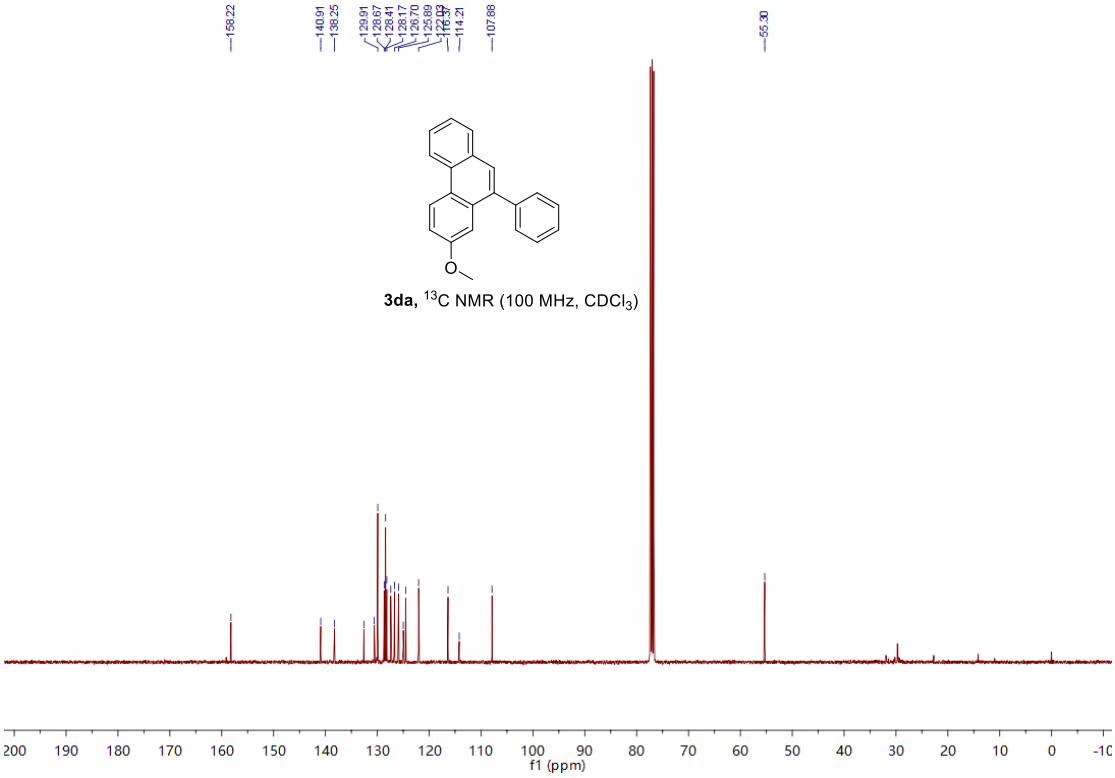
3ca, ^{13}C NMR (100 MHz, $CDCl_3$)



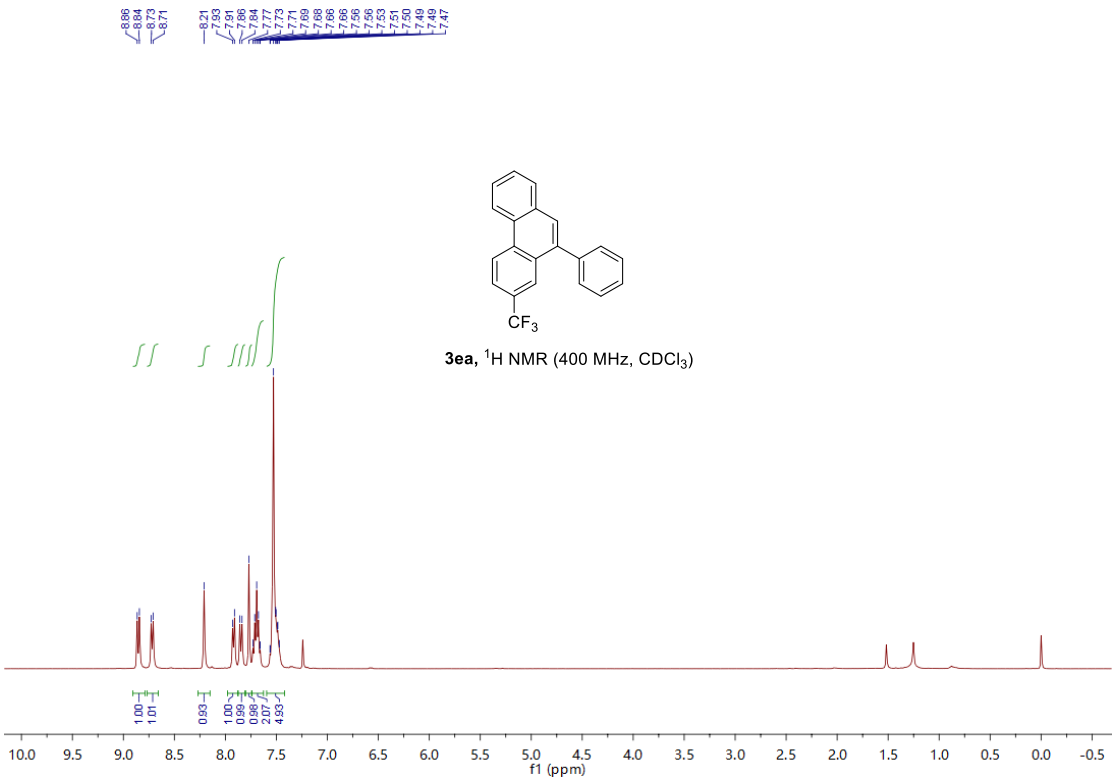
3ca, ^{19}F NMR (377 MHz, $CDCl_3$)



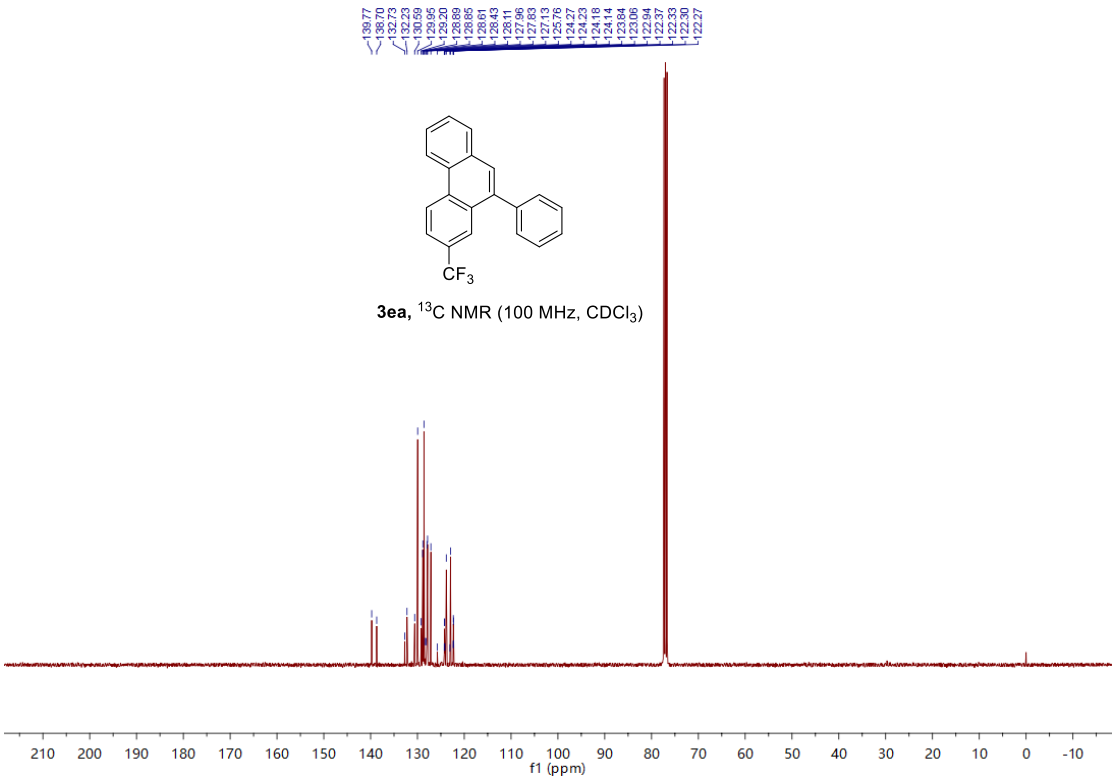
3da, ^{1}H NMR (400 MHz, $CDCl_3$)



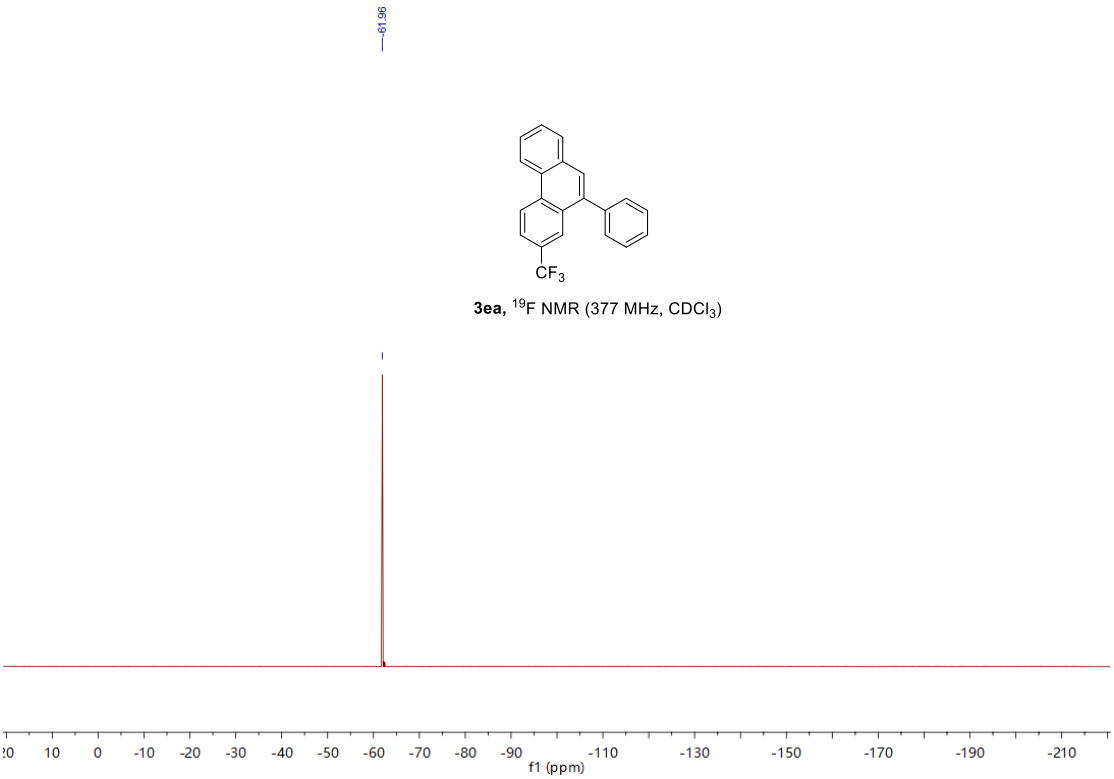
3da, ^{13}C NMR (100 MHz, $CDCl_3$)



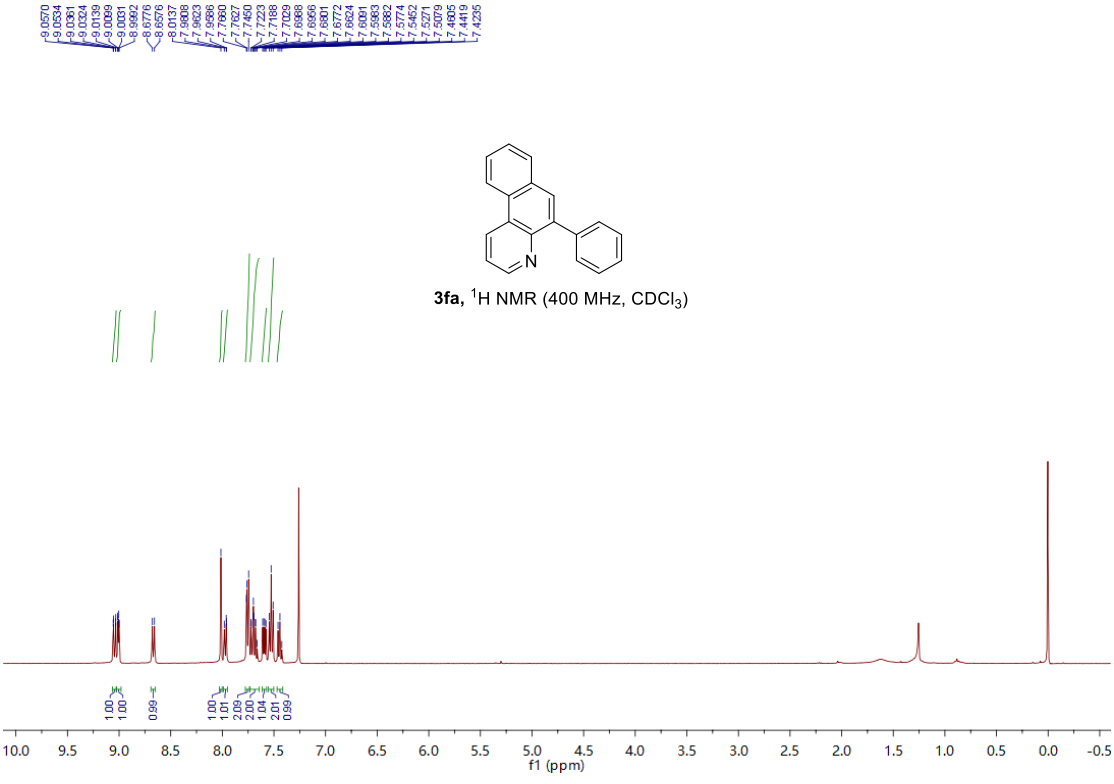
3ea, ^{1}H NMR (400 MHz, $CDCl_3$)



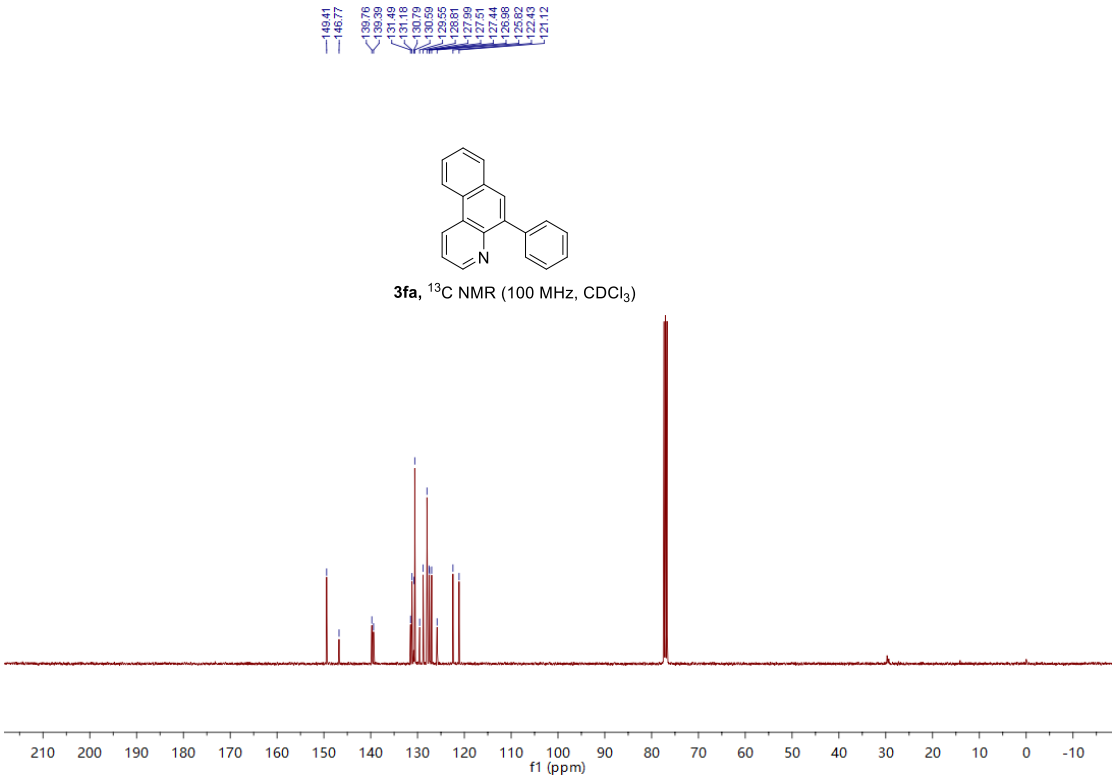
3ea, ^{13}C NMR (100 MHz, $CDCl_3$)



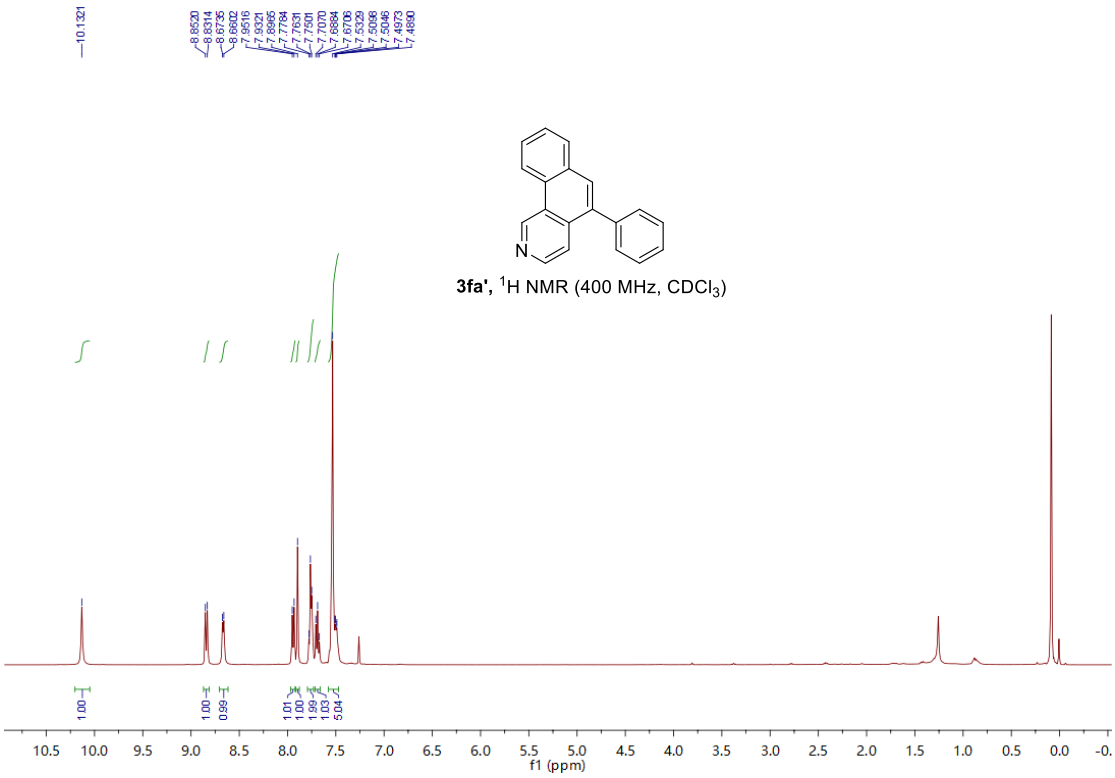
3ea, ^{19}F NMR (377 MHz, $CDCl_3$)



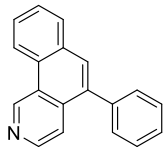
3fa, ^{1}H NMR (400 MHz, $CDCl_3$)



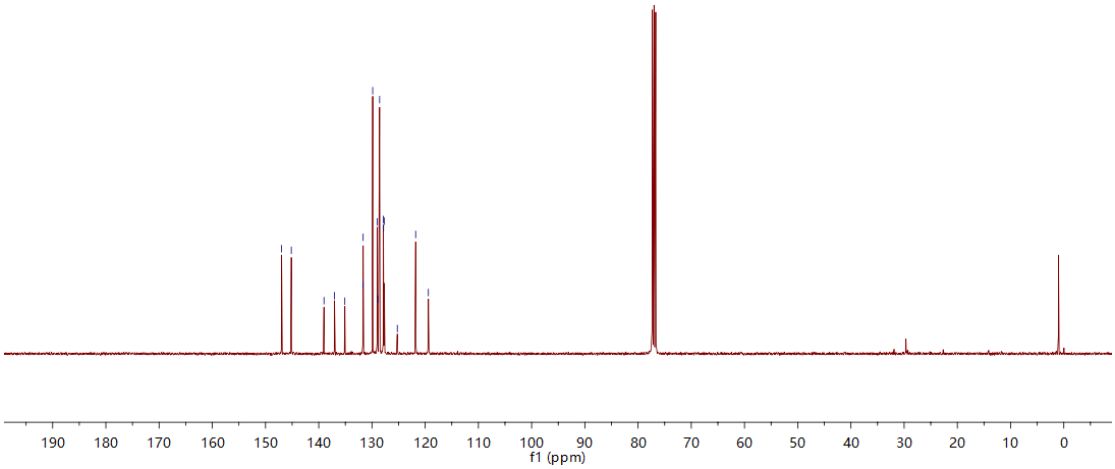
3fa, ^{13}C NMR (100 MHz, $CDCl_3$)

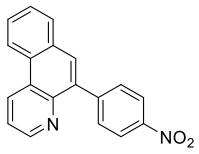


3fa', ^{1}H NMR (400 MHz, $CDCl_3$)

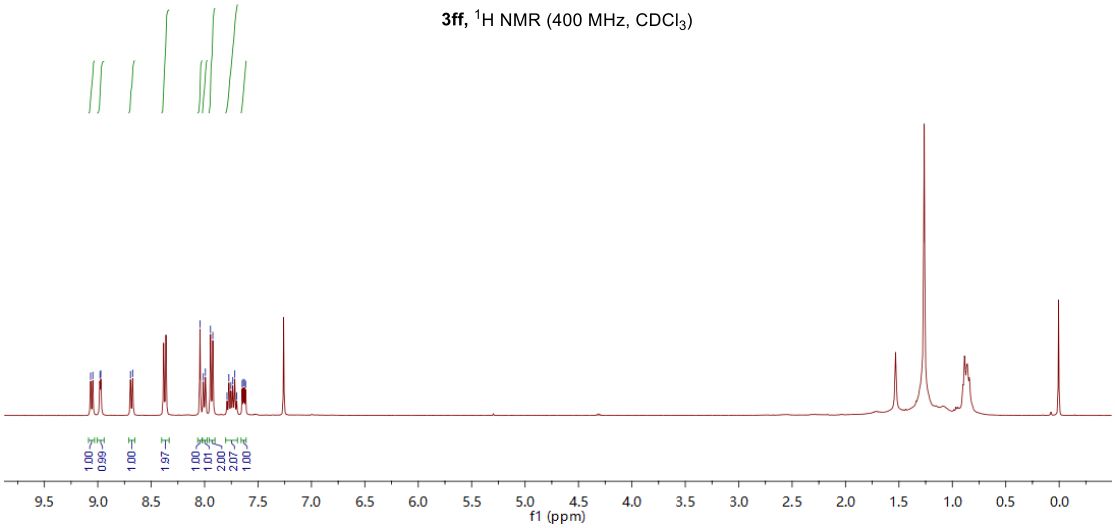


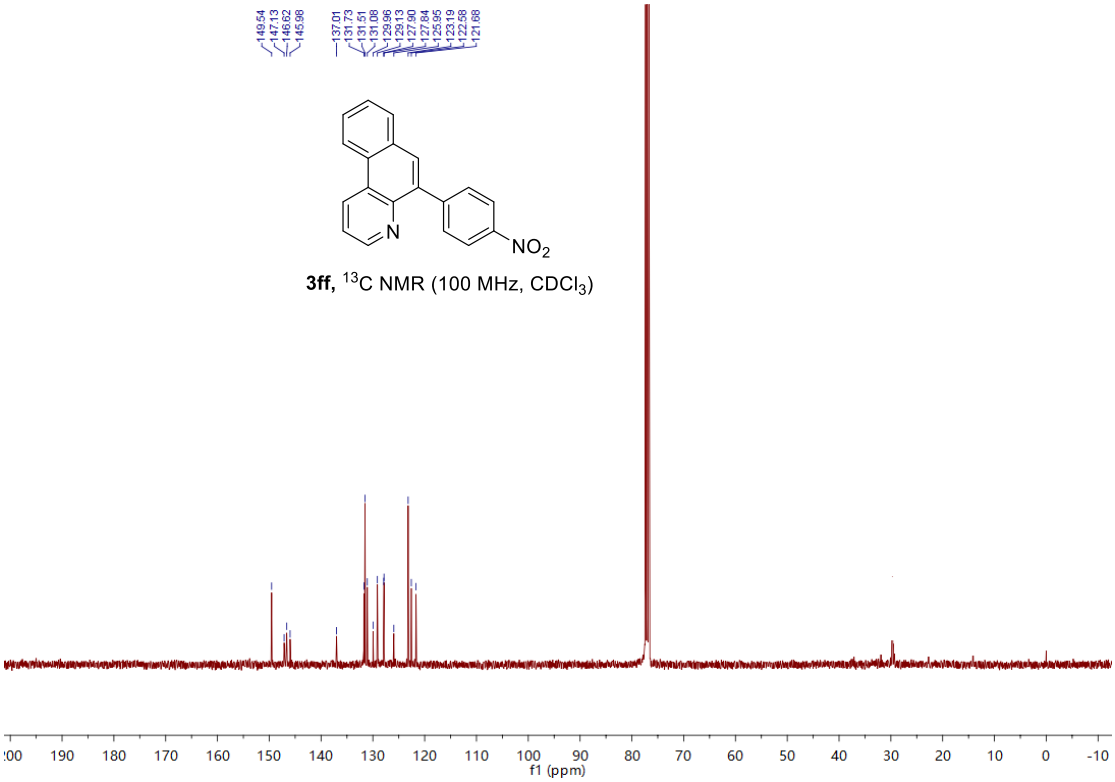
3fa', ^{13}C NMR (100 MHz, $CDCl_3$)



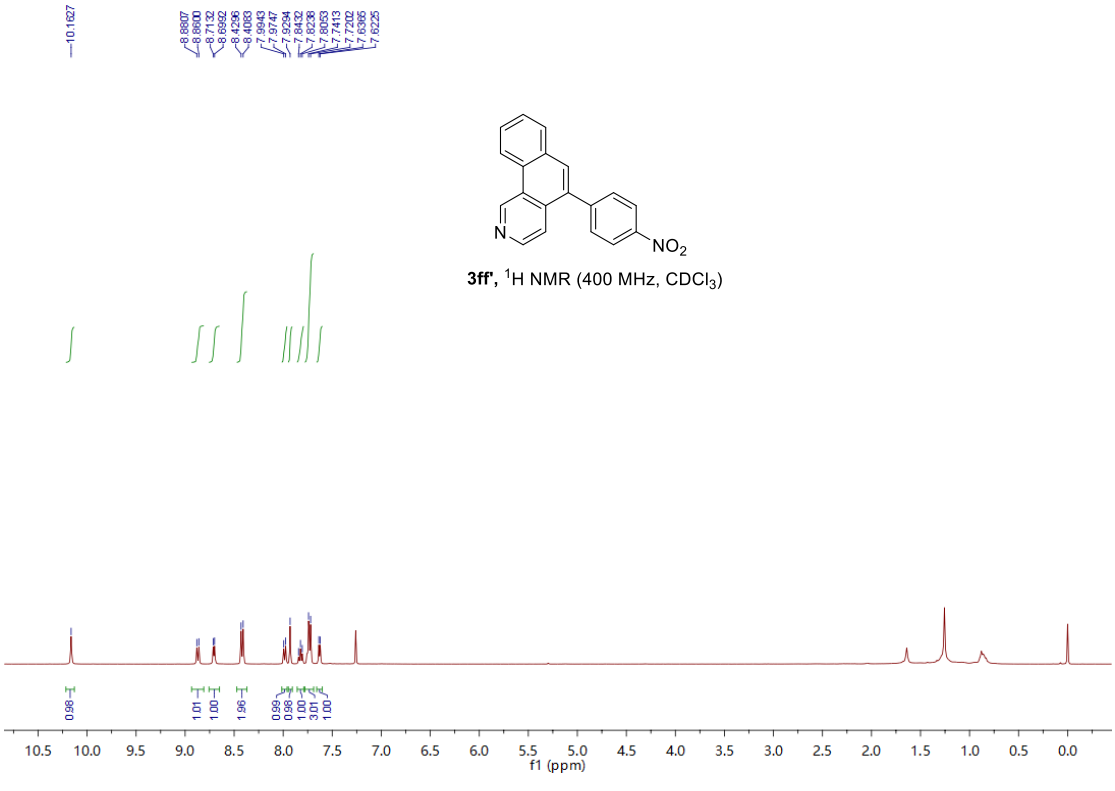


3ff, ^{1}H NMR (400 MHz, $CDCl_3$)

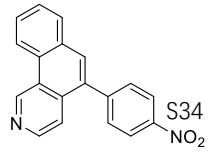




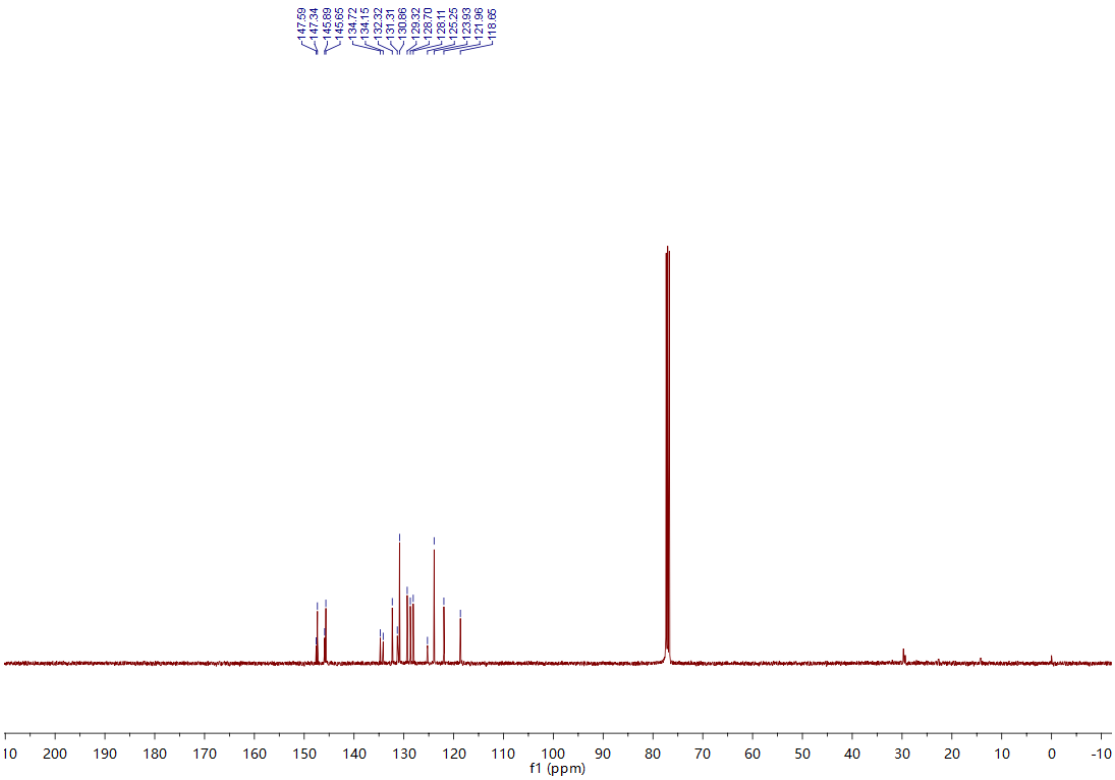
3ff, ^{13}C NMR (100 MHz, $CDCl_3$)

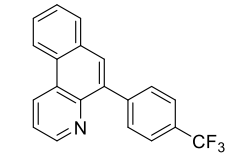


3ff', ^{1}H NMR (400 MHz, $CDCl_3$)

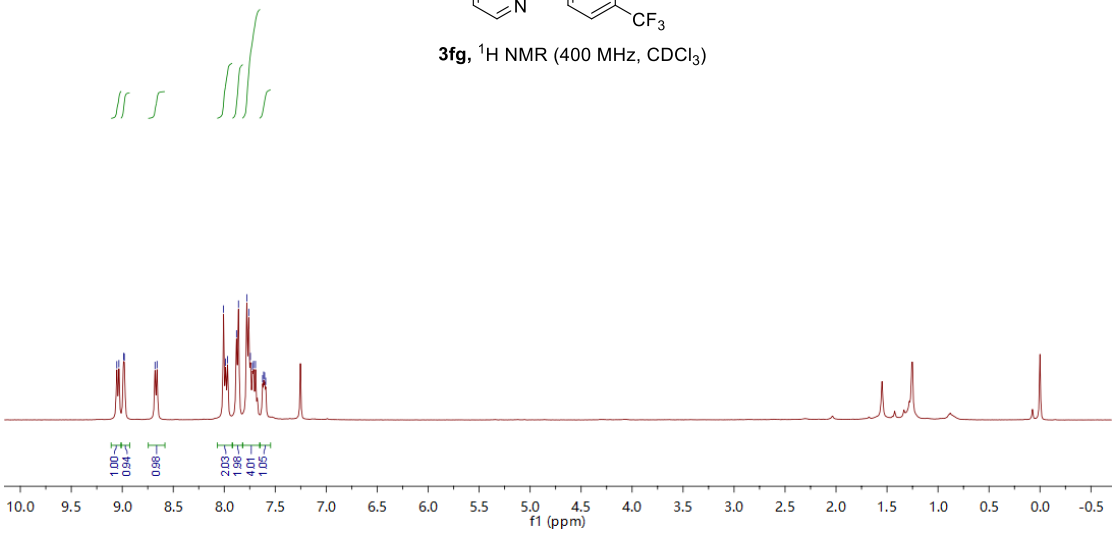


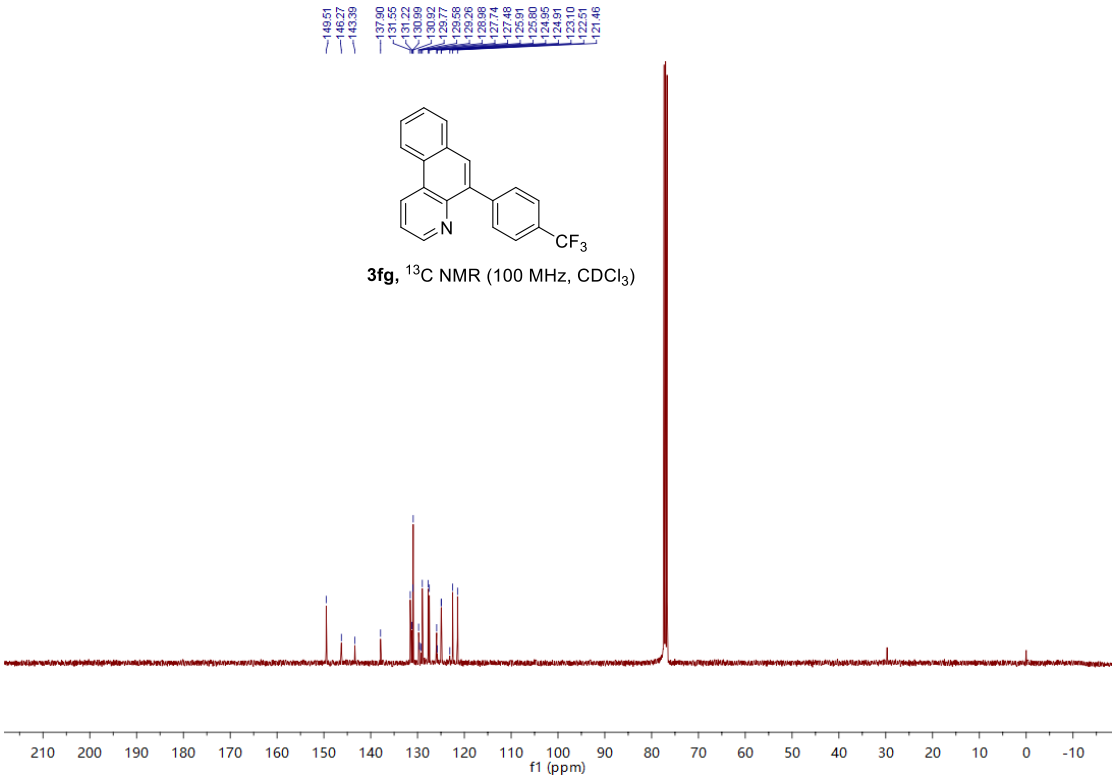
3ff', ^{13}C NMR (100 MHz, $CDCl_3$)



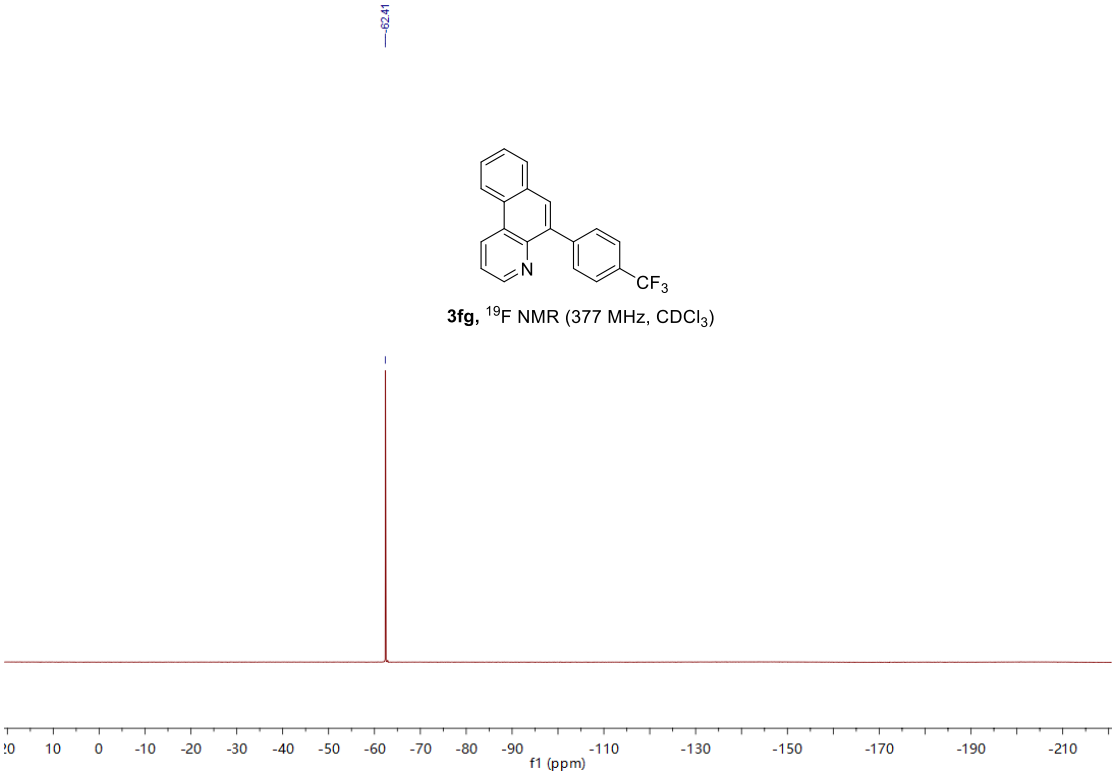


3fg, ^{1}H NMR (400 MHz, $CDCl_3$)

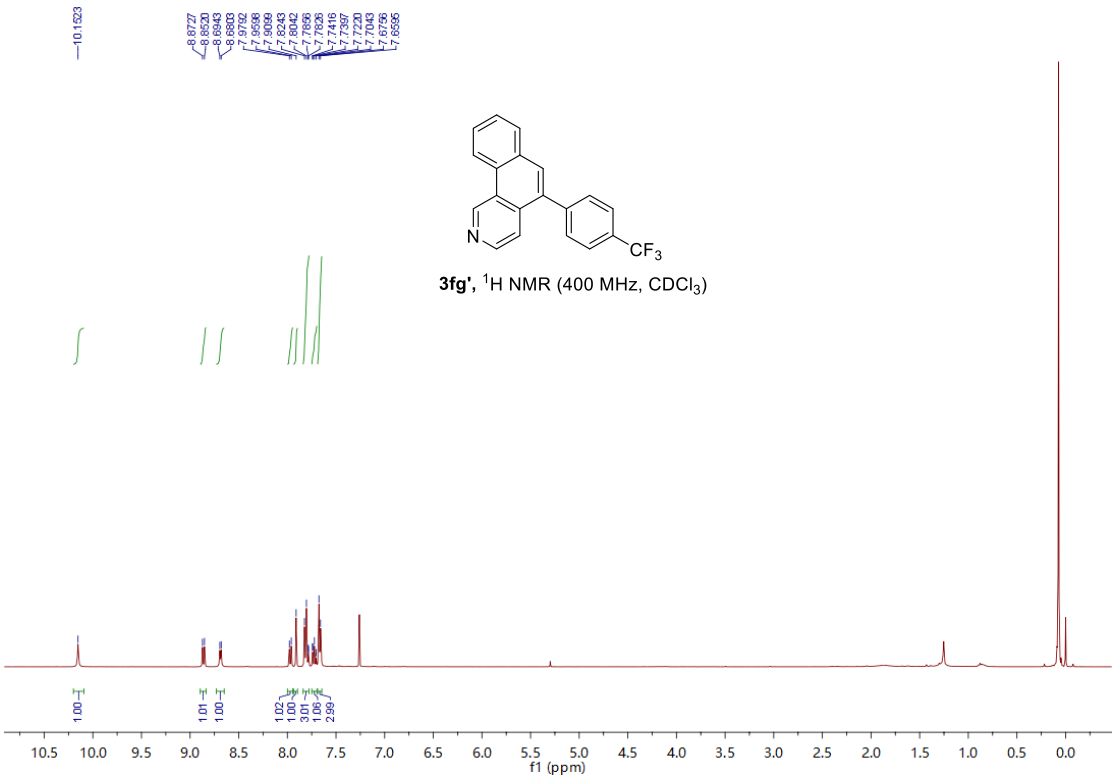




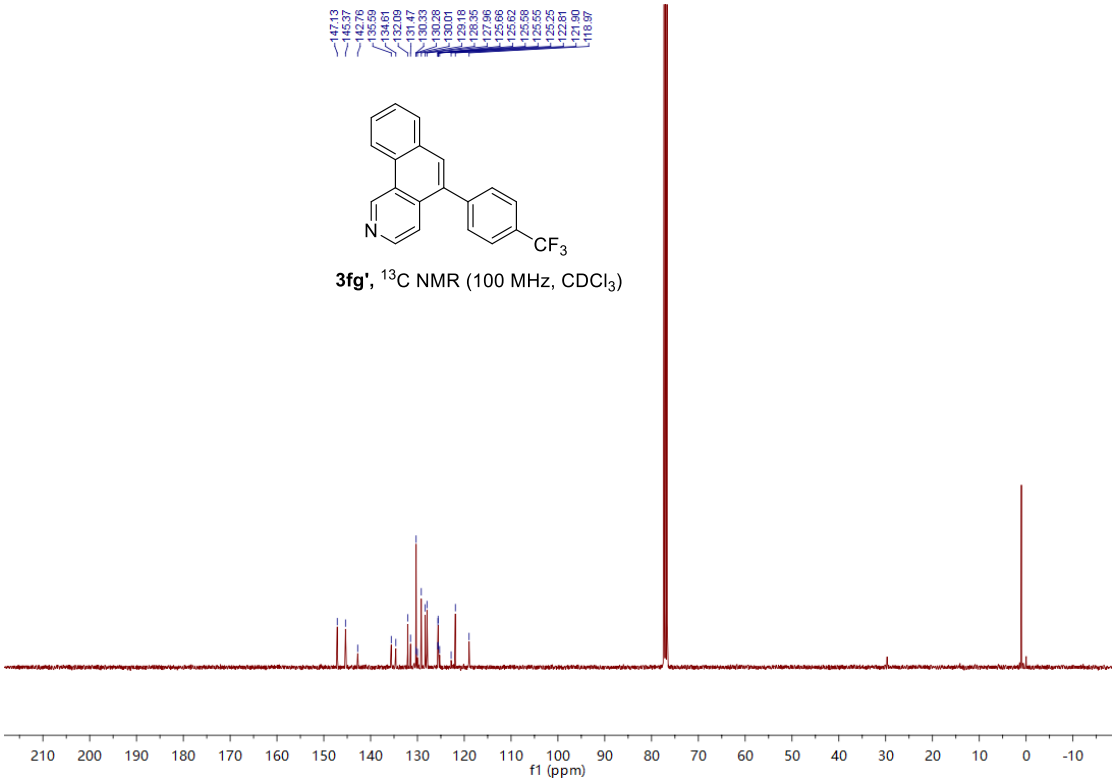
3fg, ^{13}C NMR (100 MHz, $CDCl_3$)



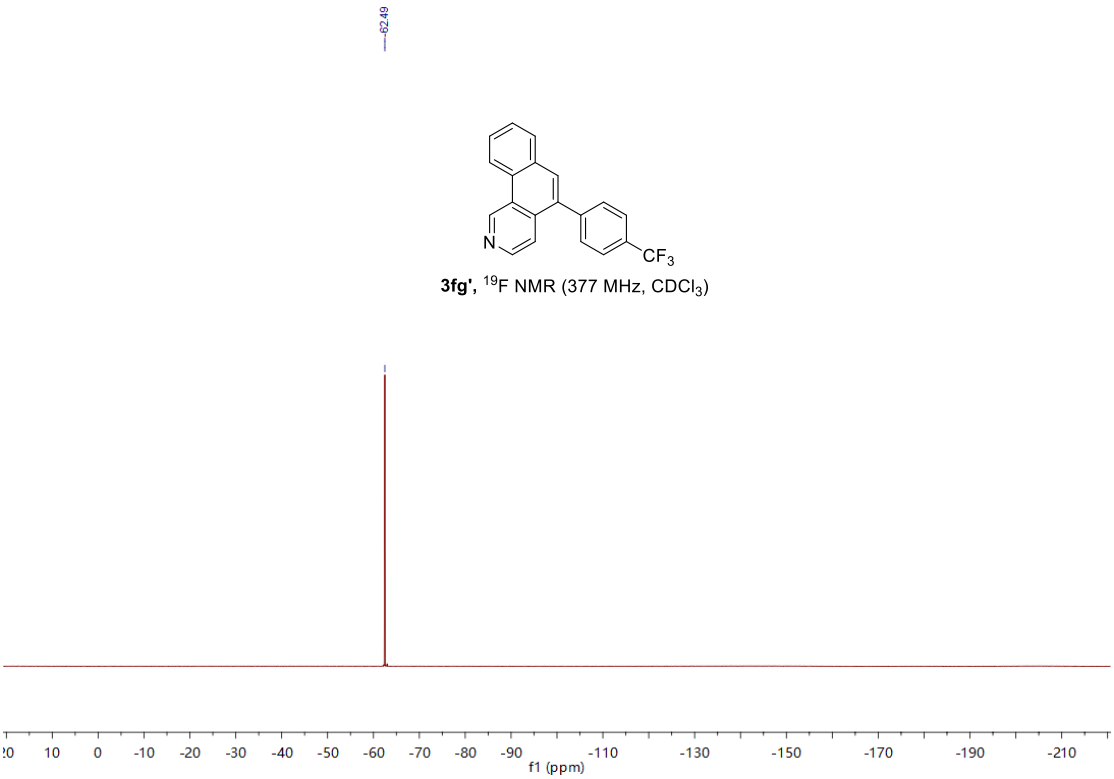
3fg, ^{19}F NMR (377 MHz, $CDCl_3$)



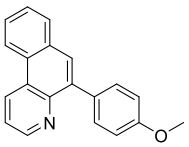
3fg', ^{1}H NMR (400 MHz, $CDCl_3$)



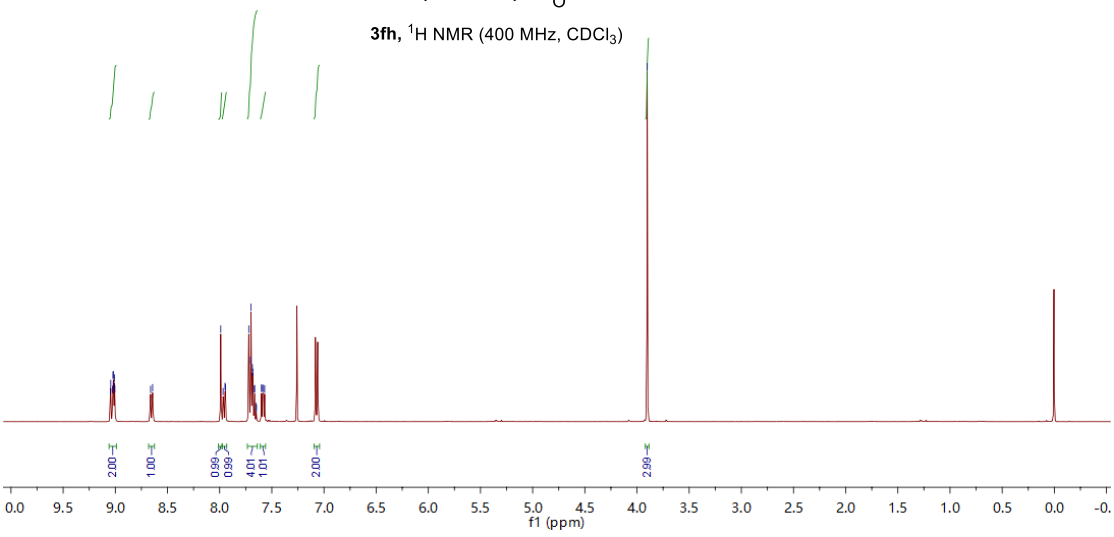
3fg', ^{13}C NMR (100 MHz, $CDCl_3$)

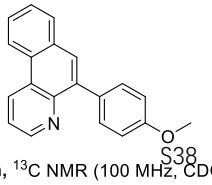


3fg', ^{19}F NMR (377 MHz, $CDCl_3$)

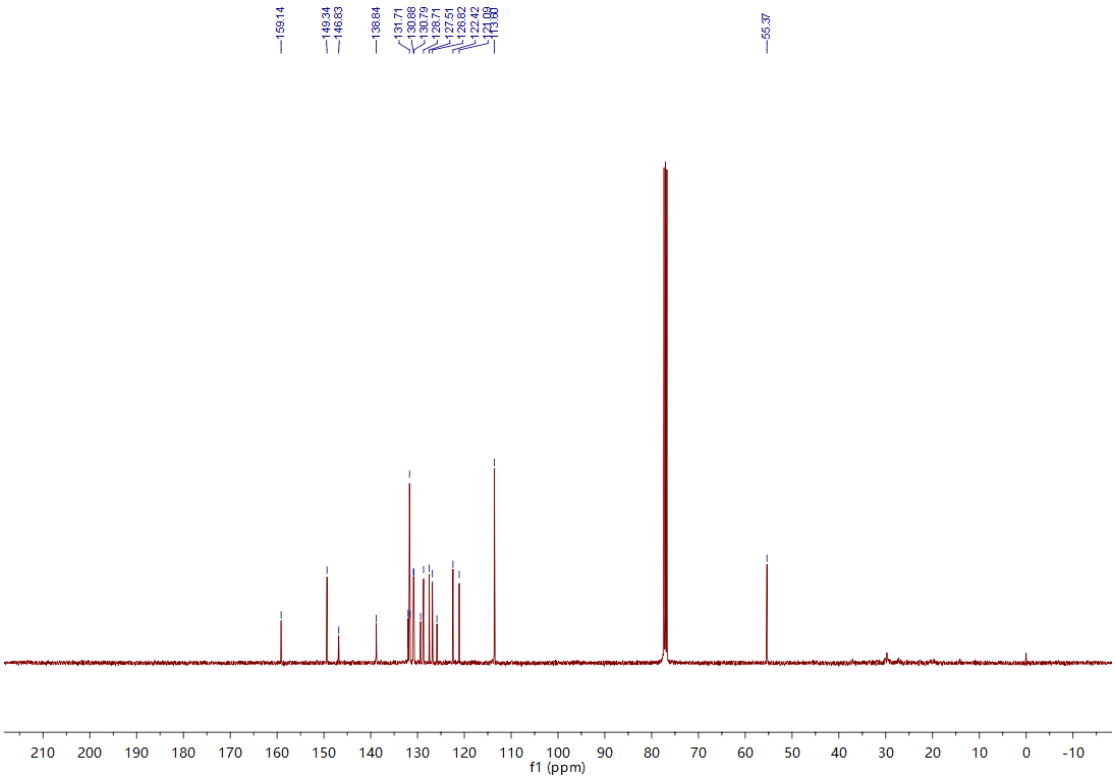


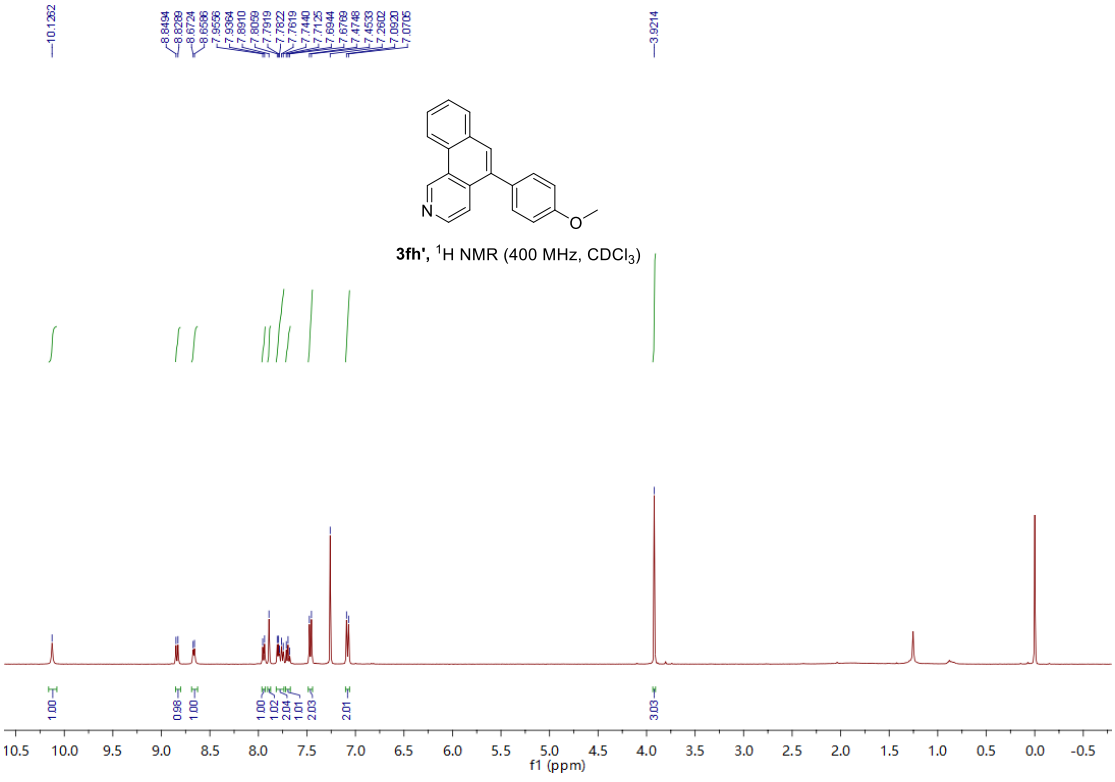
3fh, ^{1}H NMR (400 MHz, $CDCl_3$)



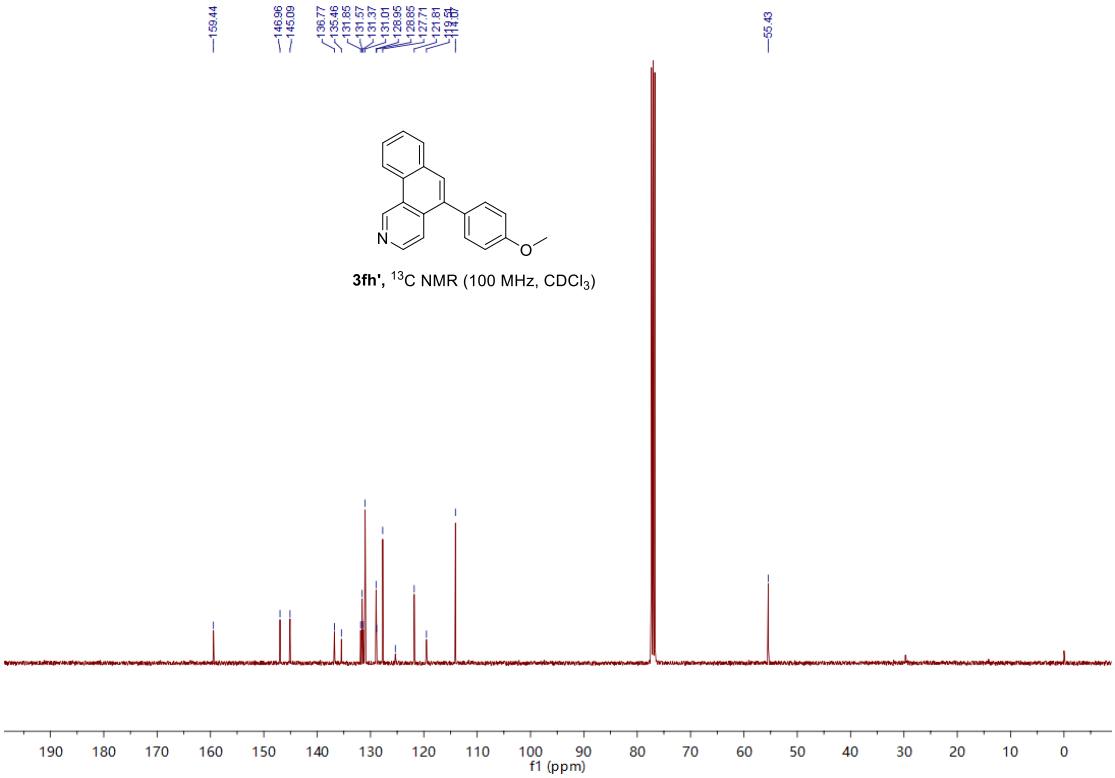


3fh, ^{13}C NMR (100 MHz, $CDCl_3$)

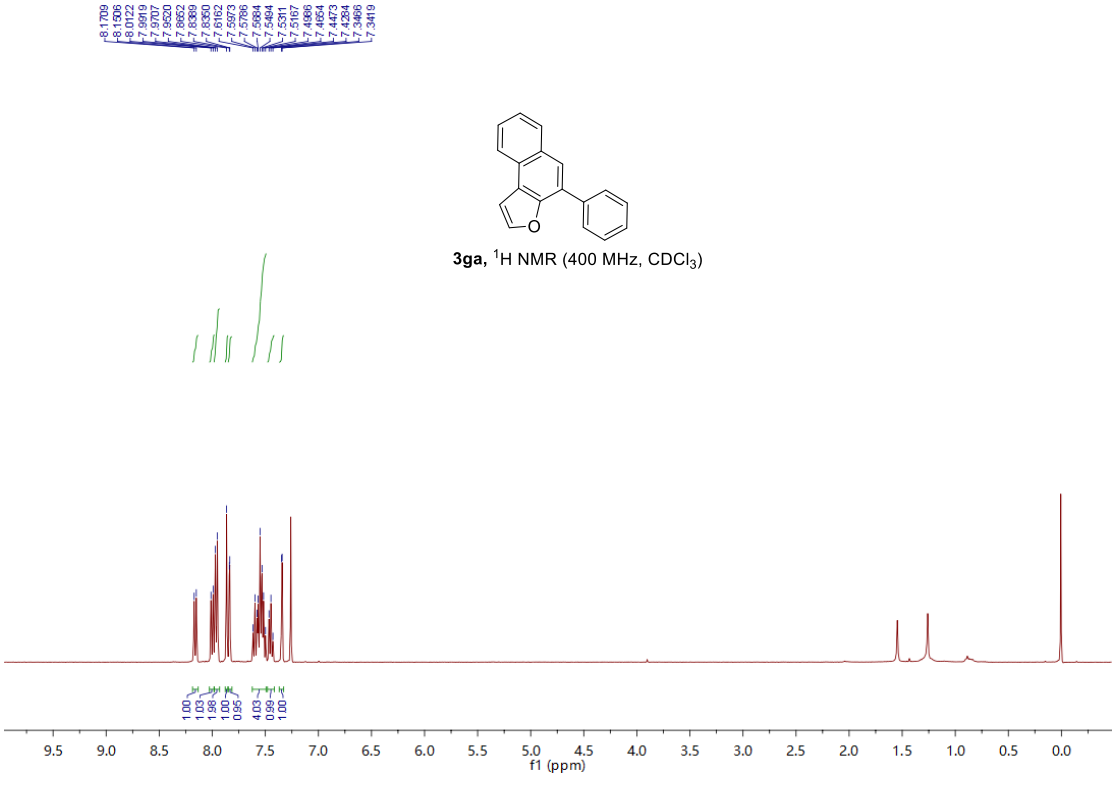




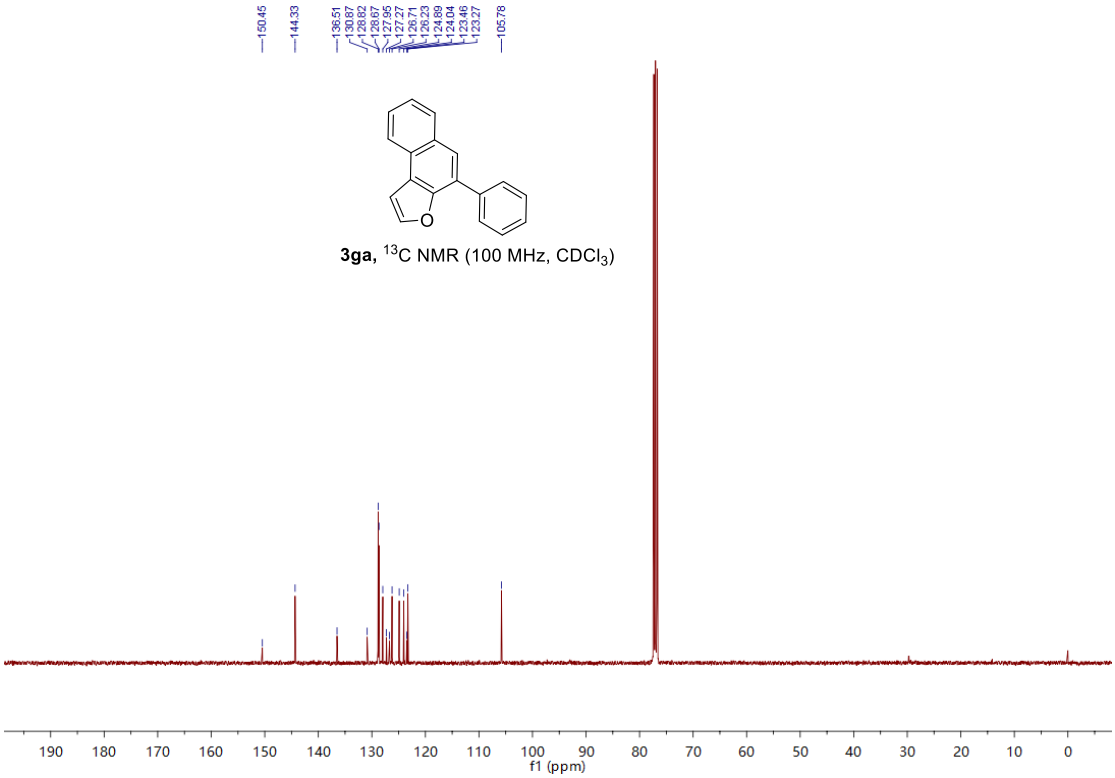
3fh', ^{1}H NMR (400 MHz, $CDCl_3$)



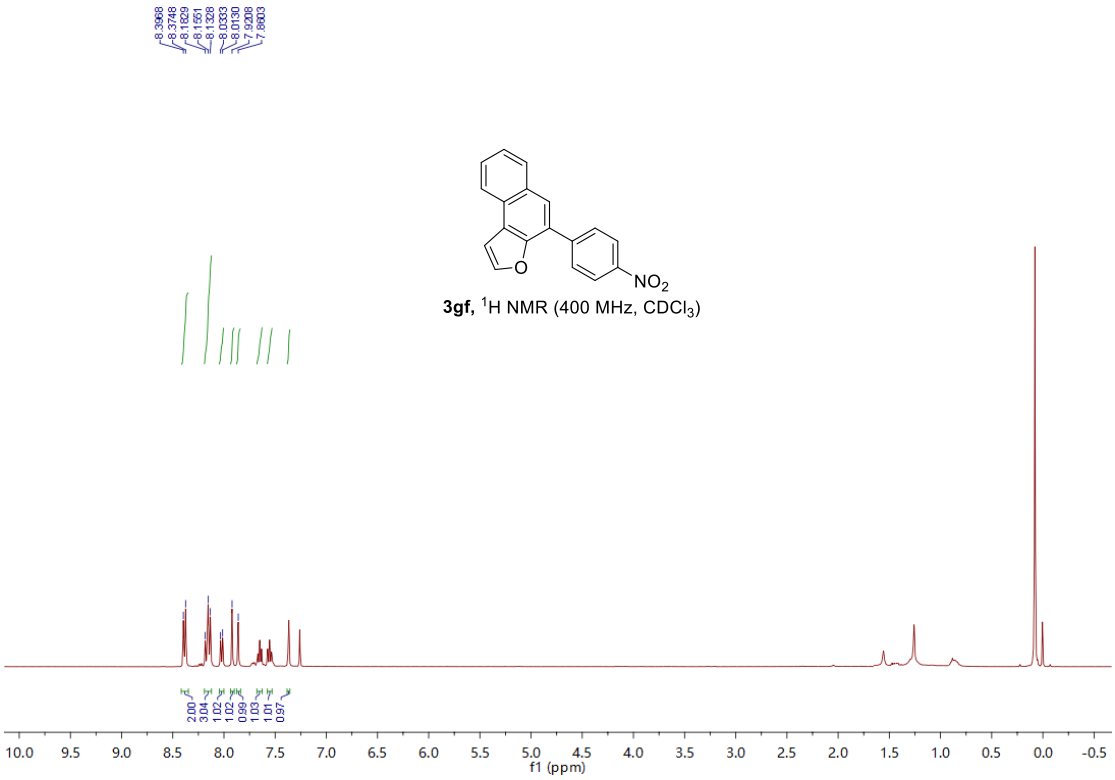
3fh', ^{13}C NMR (100 MHz, $CDCl_3$)



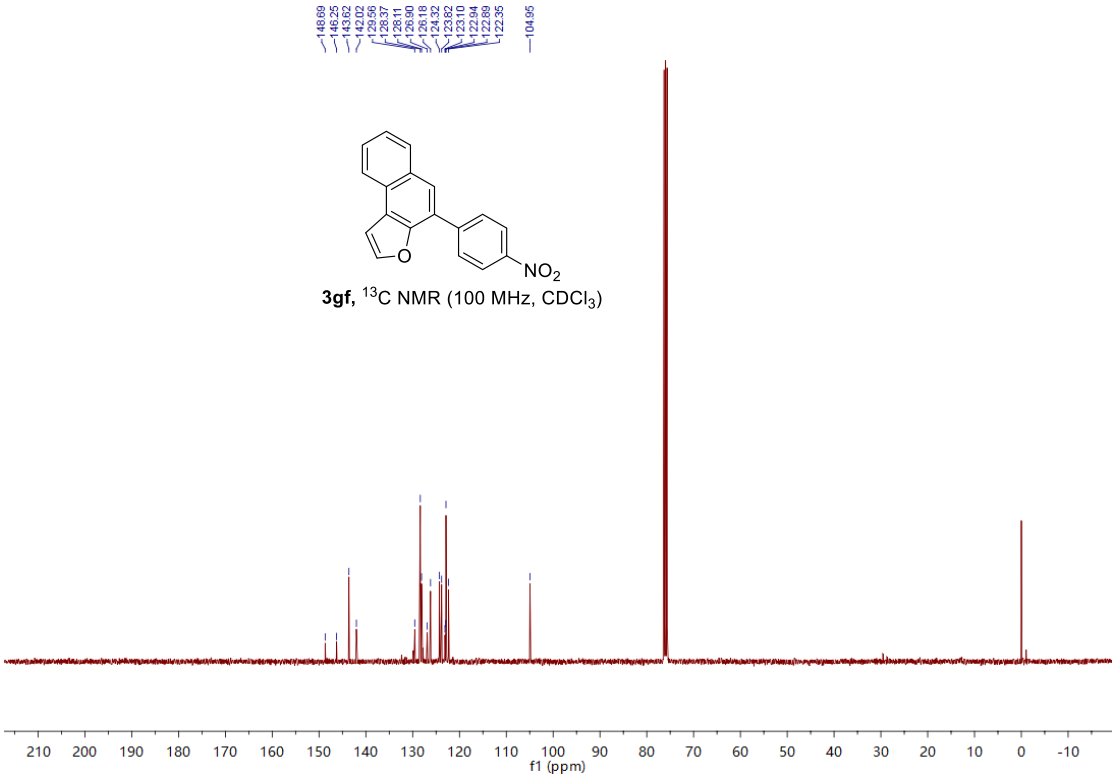
3ga, ^{1}H NMR (400 MHz, $CDCl_3$)



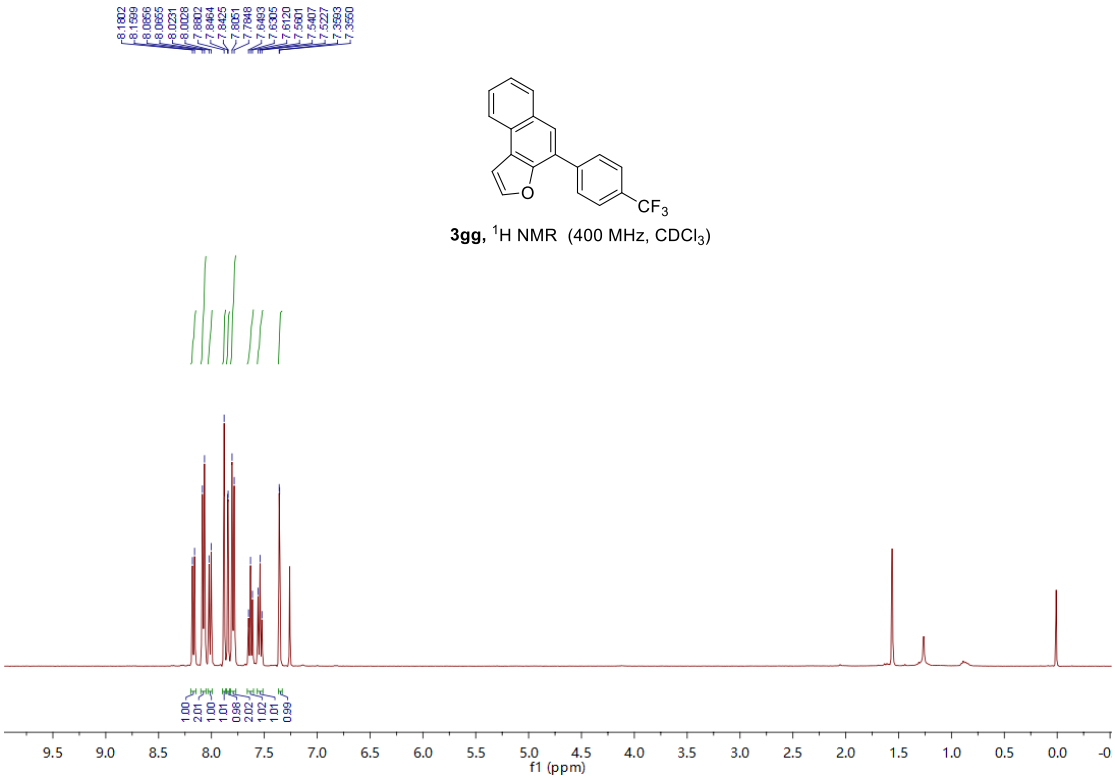
3ga, ^{13}C NMR (100 MHz, $CDCl_3$)



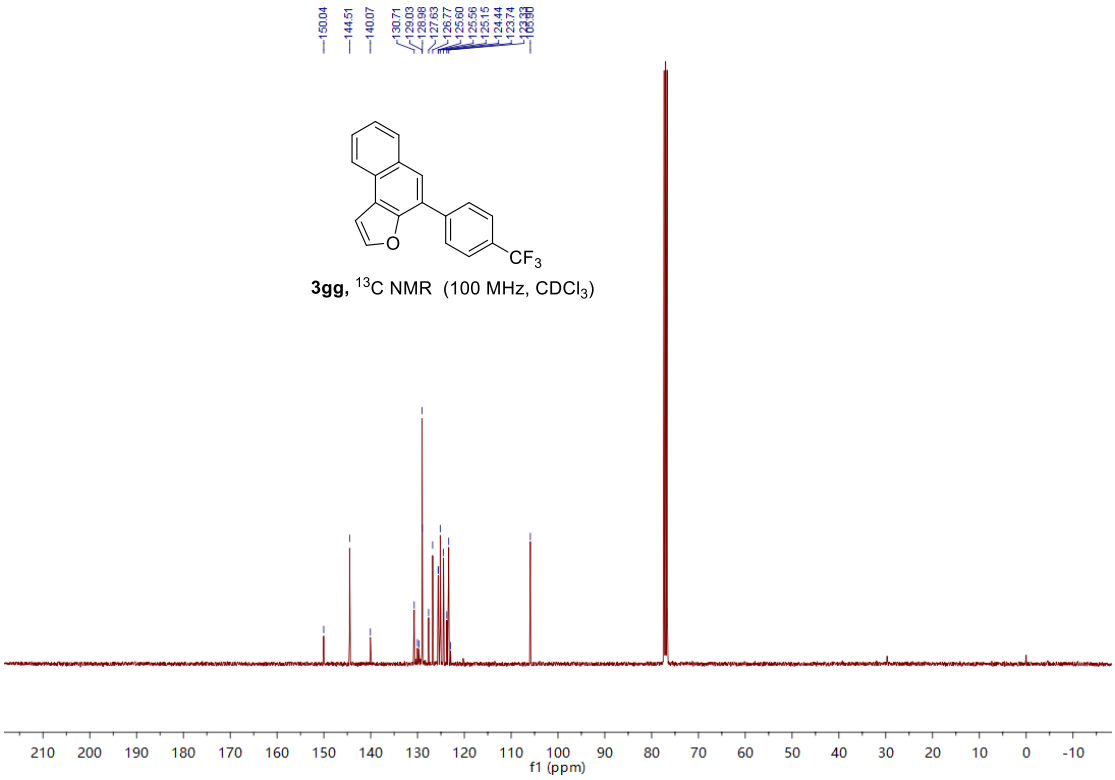
3gf, ^{1}H NMR (400 MHz, $CDCl_3$)



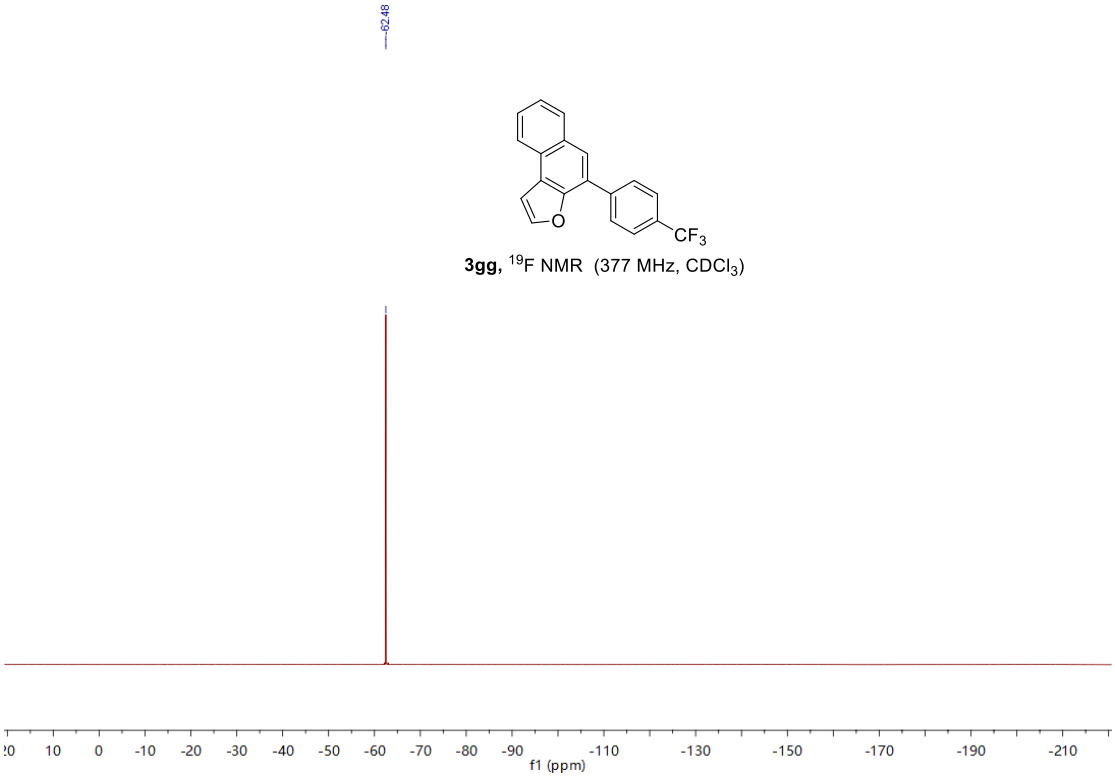
3gf, ^{13}C NMR (100 MHz, $CDCl_3$)



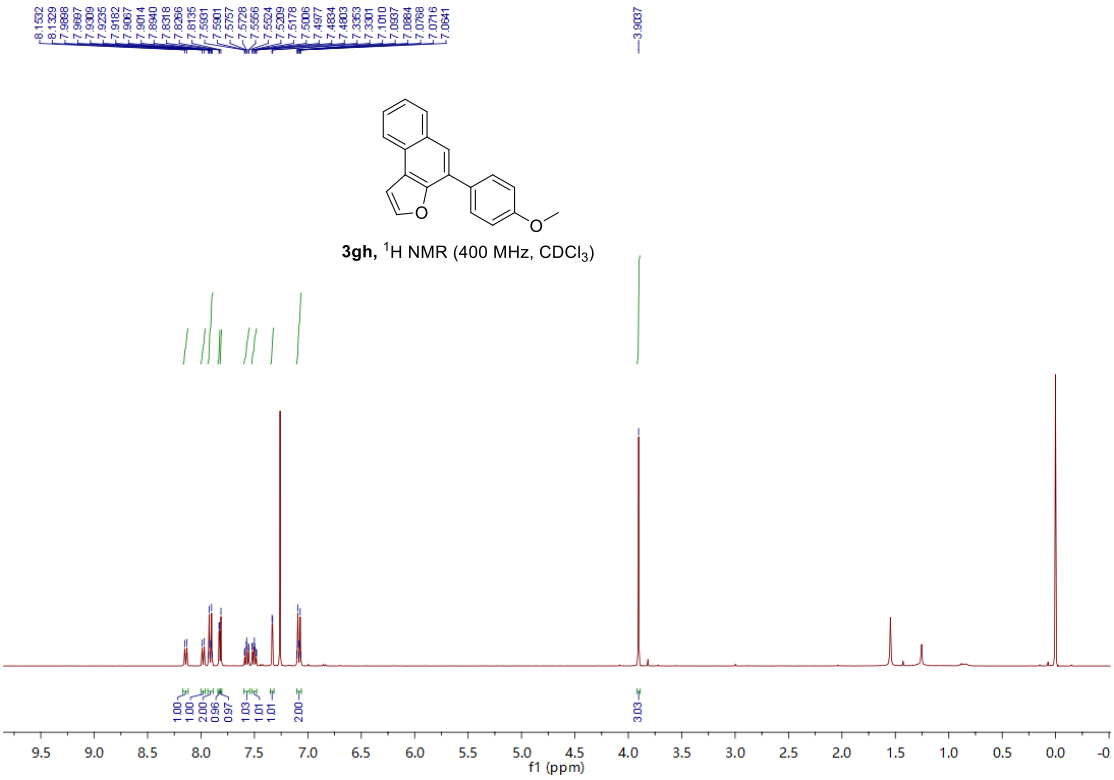
3gg, ^{1}H NMR (400 MHz, $CDCl_3$)



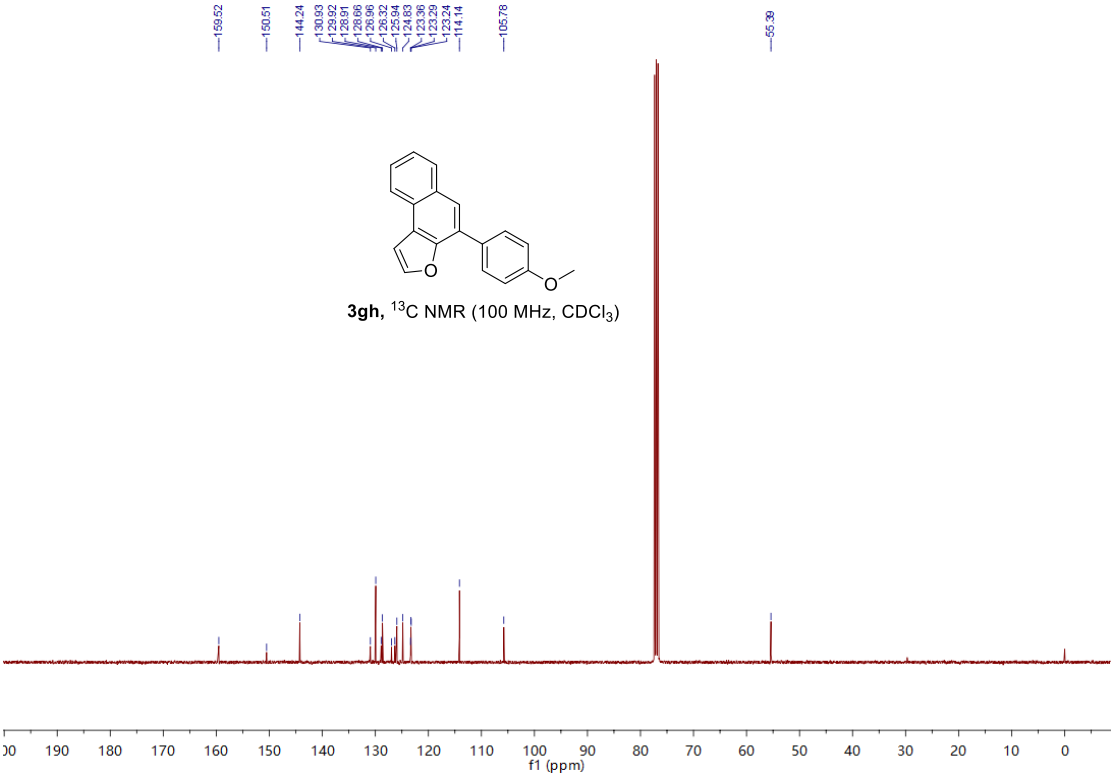
3gg, ^{13}C NMR (100 MHz, $CDCl_3$)



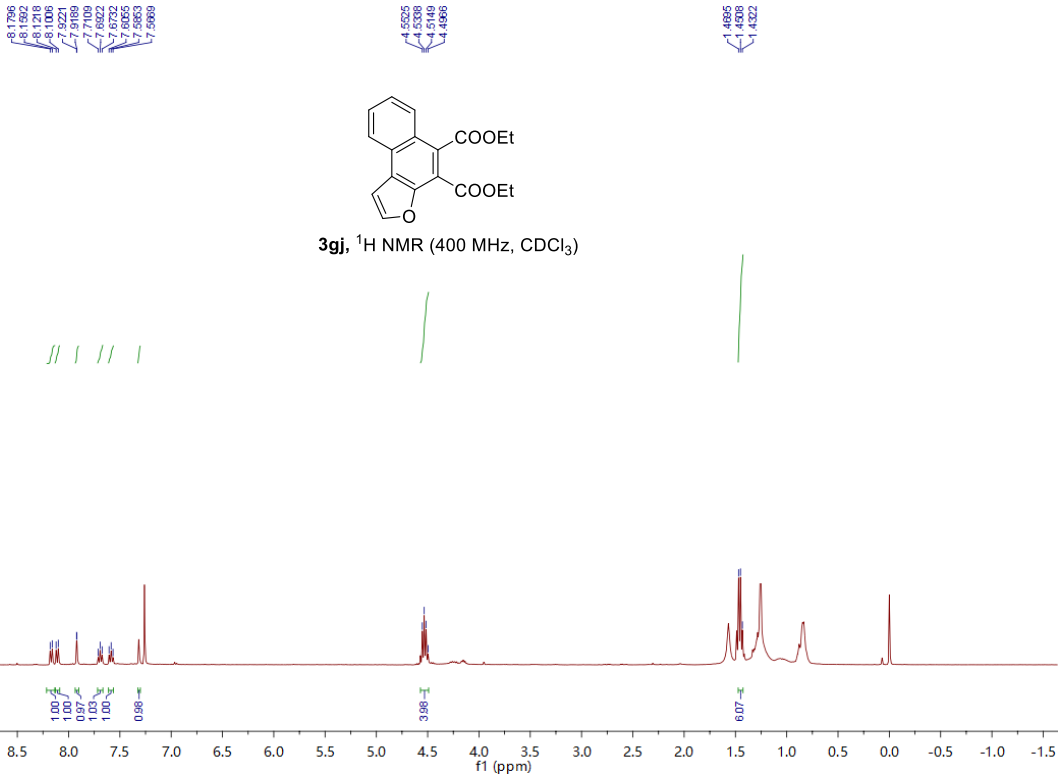
3gg, ^{19}F NMR (377 MHz, $CDCl_3$)



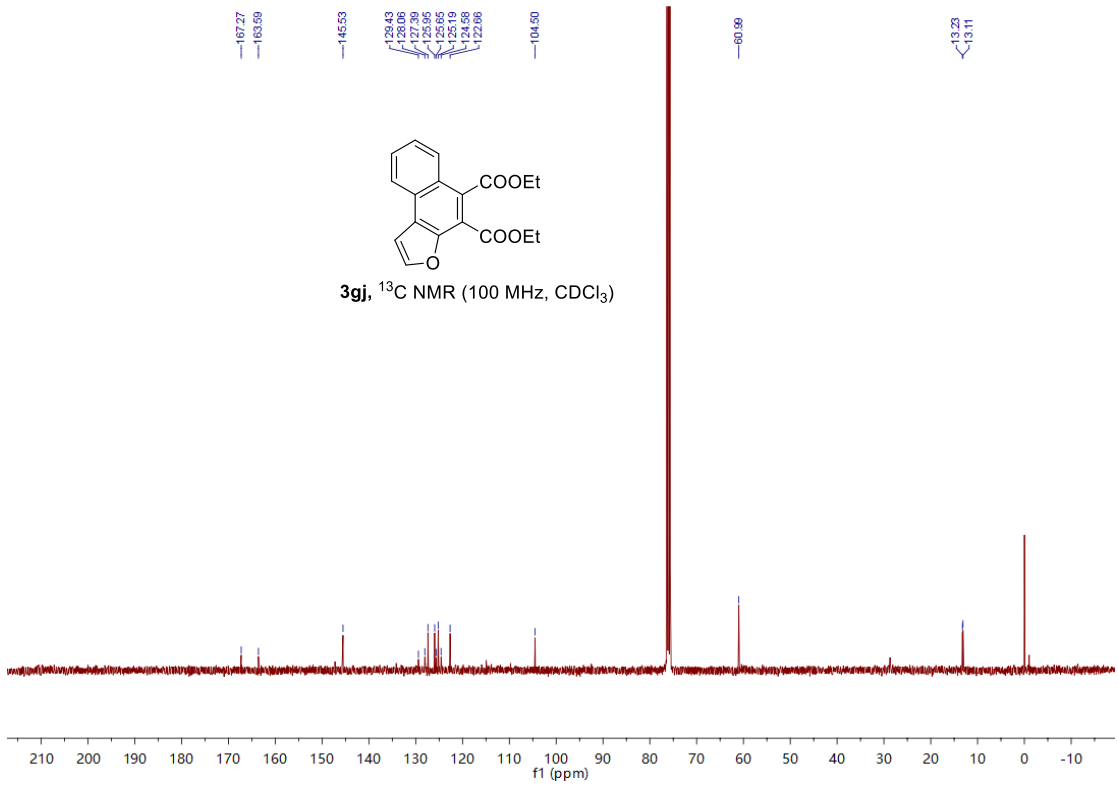
3gh, ^{1}H NMR (400 MHz, $CDCl_3$)



3gh, ^{13}C NMR (100 MHz, $CDCl_3$)



3gj, ^{1}H NMR (400 MHz, $CDCl_3$)



3gj, ^{13}C NMR (100 MHz, $CDCl_3$)