Application of a 1-(2-aryl)dicyclohexylphosphonium tetrafluoroborate ligand in the conjugated synthesis of heteroaryl biphenyls

Yue Hu,[b] Wenjing Fu,[b] Mengyu Zhou,[b] Haoyue Xing,[b] Sixuan Meng,[b] Jian Chen,[b] Qiong Chen,[b] Rui Guo*[a] and Guang-Ao Yu*[b]

[a] Hubei Key Laboratory of Environmental and Health Effects of Persistent Toxic Substances, School of Environment and Health, Jianghan University, Wuhan 430056, China.

[b] State Key Laboratory of Green Pesticide, Key Laboratory of Pesticide & Chemical Biology of Ministry of Education, International Joint Research Center for Intelligent Biosensor Technology and Health, College of Chemistry, Central China Normal University, Wuhan 430079, P.R. China.

* Email: guorui2010@jhun.edu.cn (R. Guo), yuguang@mail.ccnu.edu.cn (G. Yu)

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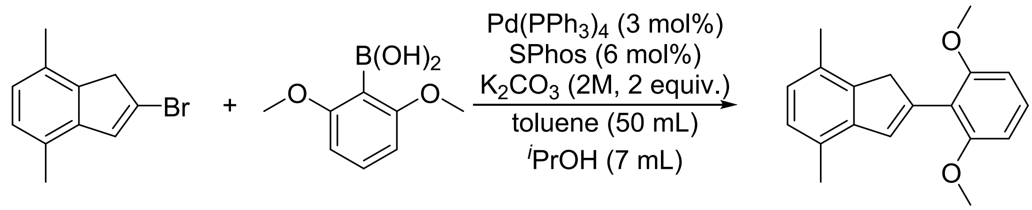
Experimental Section

General Considerations. All manipulations of air-sensitive materials were carried out under an atmosphere of dry argon by using modified Schlenk line and glovebox techniques. Aryl and heteroaryl halides, boronic acids, bases and catalysts were purchased from Alfa-Aesar and J&K Scientific Ltd. All solvents were distilled from appropriate drying agents under argon before use. The ^{1}H, ^{13}C, ^{11}B and ^{31}P NMR spectroscopic data were recorded on Bruker Mercury Plus 400 MHz NMR spectrometers. Chemical shifts (δ) for ^{1}H and ^{13}C are referenced to internal solvent resonances and reported relative to $SiMe_4$. Chemical shifts (δ) for ^{11}B NMR spectra data are referenced to external BF_3•Et_2O standard. Chemical shifts for ^{31}P are reported relative to an external 85% H_3PO_4 standard. HRMS were obtained on an IonSpec FT-ICR mass spectrometer with ESI resource. Compounds described in the literature were characterized by comparison of their ^{1}H NMR spectra to the previously reported data. 2-bromo-4,7-dimethyl-1H-indene was synthesized according to the published procedures. [1]

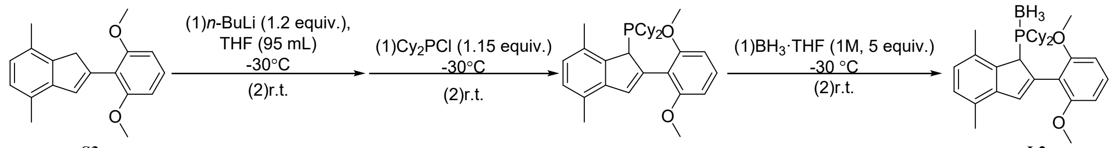
Synthesis of 2-(2,6-dimethoxyphenyl)-4,7-dimethyl-1H-indene



S1 **S2** **S3**

In a 250 mL three-necked round-bottom flask, 2-bromo-4,7-dimethyl-1H-indene (5 g, 22.4 mmol), 2,6-dimethoxyphenylboronic acid (4.48 g, 24.6 mmol), $Pd(PPh_3)_4$(777 mg, 3 mol%), and SPhos (551.8 mg, 6 mol%) were added under a nitrogen atmosphere. The flask was evacuated and refilled with nitrogen three times. Subsequently, a 2 M aqueous K_2CO_3 solution (22.4 mL), toluene (50 mL), and isopropanol (7 mL) were added sequentially. The reaction mixture was heated to 90 ºC and stirred for 8 h. After the reaction was completed, the mixture was cooled to ambient temperature. The organic phase was separated, and the aqueous phase was extracted with dichloromethane (DCM) three times. The combined organic phases were dried over anhydrous sodium sulfate, concentrated under reduced pressure, and purified by flash column chromatography to afford the target product as a yellow solid powder (5.4 g, 86%).

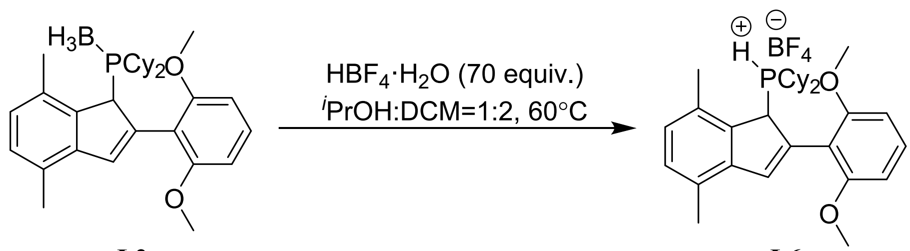
Synthesis of 2-(Dicyclohexylphosphino)-1-methyl-3-(2,6-dimethoxyphenyl)-1H-inden-2-yl)borane



S3 L2

In a 250 mL two-necked flask, a clean one taken out from the oven and dried with a heat gun for 15 minutes, 2-(2,6-dimethoxyphenyl)-4,7-dimethyl-1H-indene (4.5 g, 16 mmol) (Remove residual moisture by azeotropic drying with toluene under ambient atmospheric conditions) was weighed in a glovebox. Under nitrogen atmosphere, the flask was evacuated and refilled with nitrogen three times. Then THF (95 mL) was added dropwise over 20 minutes. After stirring to form a homogeneous solution (the reaction solution was pale yellow), the reaction setup was cooled to -30 °C. After cooling for several minutes, ^{n}BuLi (2.3 M solution in hexane, 8.4 mL, 19.2 mmol) was added under nitrogen over 15 minutes (the reaction solution darkened in color). The solution was stirred for 20 minutes at -30 °C and then for 8 hours and 50 minutes at ambient temperature. Then the mixture was cooled to -30 ºC. After cooling for several minutes, Cy_2PCl (weighed in the glovebox and added directly, 4.1 mL, 18.4 mmol) was added under nitrogen over 15 minutes. The mixture was warmed to room temperature and stirred for additional overnight. The reaction setup was cooled to -30 ºC, and BH_3·THF (1 M solution, 80 mL, 80 mmol, 5 equiv.) was added. The mixture was then warmed to room temperature and stirred until the reaction was complete as monitored by TLC. The reaction was quenched with water under ice bath. The organic layer was separated from the aqueous layer. The aqueous layer was extracted with DCM. The combined organic phase was dried over anhydrous Na_2SO_4, concentrated, and purified by recrystallization from acetone to provide the desired product as a white solid powder (5.1 g, 51%).

Synthesis of L6



L2 L6

In a 50 mL three-necked flask, solid A (500 mg, 1.02 mmol) was added. The flask was evacuated and refilled with nitrogen three times. Under a nitrogen atmosphere, dichloromethane (10 mL), isopropanol (5 mL), and $HBF_4·H_2O$ (11.36 mL, 71.4 mmol) were added sequentially. The reaction mixture was heated to 60 ° C and stirred for 24 hours. The reaction mixture was transferred to a separatory funnel, and the organic phase was separated from the aqueous phase. The aqueous phase was extracted with dichloromethane. The combined organic phases were dried over anhydrous sodium sulfate, concentrated under reduced pressure, and purified by recrystallization from dichloromethane/hexane to provide the desired product as a light yellow solid powder (410 mg, 71%).

General procedure for Suzuki－Miyaura cross-coupling reaction

A disposable tube with a screw cap, Teflon septum and stir bar was charged with $Pd(dba)_2$, ligand **6**, aryl halide (1.0 mmol), arylboronic acid (2.0 mmol), potassium phosphate tribasic trihydrate (3.0 mmol) and Xylene (2.0 mL). The tube was evacuated and flushed with nitrogen three times, and then placed in a preheated oil bath at 130 ° C for 24 hours. After completion of the reaction, the reaction tube was allowed to cool to room temperature, partioned with water (10 mL) and ethyl acetate (10 mL). The organic layer was separated, dried over sodium sulfate, concentrated, and purified by silica gel column chromatography to provide biaryl compounds.

Gram-scale upscaling experiment

4-Chlorobenzophenone (1.73 g, 8 mmol), phenylboronic acid (1.95 g, 8 mmol), $Pd(dba)_2$ (46 mg, 0.008 mmol), $K_3PO_4{\cdot}3H_2O$ (6.39 g, 24 mmol), and L6 (90.3 mmol, 0.16 mmol) were added to a 50 mL two-necked flask, evacuated and filled with nitrogen three times, and then 2 mL of xylene was added under nitrogen protection, followed by heating at 130 °C for 24 h. The was extracted with EA and purified by column chromatography (PE/EA=50:1) to give 2.04 g of a white solid, with a yield of 99%.

X-ray structural determination

The X-ray date was collected on a Rigaku Saturn CCDC diffractometer using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The structure was solved by direct methods (SHELXS-97)[2] and refined by full-matrix least squares on F^2. All non-hydrogen atoms were refined anisotropically and hydrogen atoms by a riding model (SHELXL-97).[3] The crystal data and structural refinements details are listed in Table S1. CCDC 2494488 (**L2**) contain the supplementary crystallographic data for this paper. This data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Table S1. Crystal Data and Summary of X-ray Data Collection for compounds **L2**

	L2
formula	$C_{31}H_{44}BO_2P$
fw	490.44
T (K)	296.15 K

space group	P-1
crystal system	Triclinic
a (Å)	11.684(5)
b (Å)	17.226(8)
c (Å)	18.637(9)
α (deg.)	115.124(6)
β (deg.)	105.514(7)
γ (deg.)	92.142(7)
V (Å3)	3223(3)
Z	4
*d*calcd. (mg/m^3)	1.011
F(000)	1064
GOF	1.001
R1 ($I > 2\sigma(I)$)	0.0820
*w*R2 (all data)	0.2659

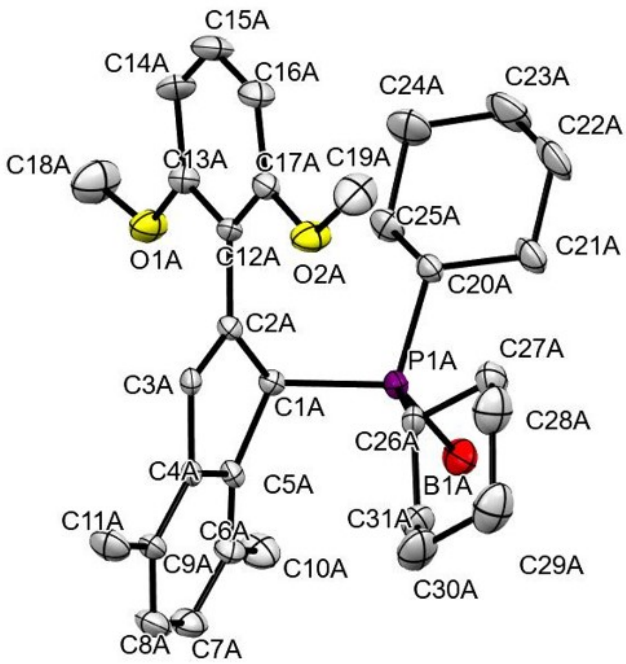
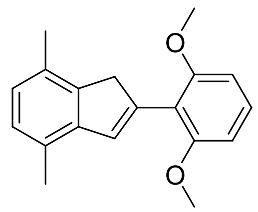
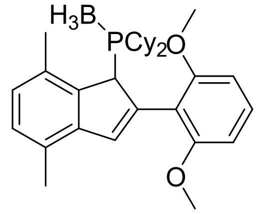


Figure S1. Molecular structure of **L2**. Thermal ellipsoids are set at 30% probability. H atoms have been omitted for clarity.

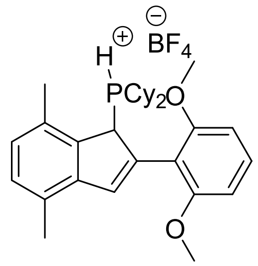
^{1}H, ^{13}C{^{1}H}, ^{11}B, ^{19}F and ^{31}P NMR spectra for all products



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.21 (t, *J* = 8.3 Hz, 1H), 7.08 (s, 1H), 6.98 (d, *J* = 7.6 Hz, 1H), 6.88 (d, *J* = 7.6 Hz, 1H), 6.61 (d, *J* = 8.4 Hz, 2H), 3.78 (s, 6H), 3.71 (s, 3H), 2.43 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.31, 143.94, 142.65, 139.39, 130.82, 129.87, 128.44, 127.59, 127.51, 125.55, 116.09, 104.35, 77.48, 77.16, 76.84, 56.02, 41.52, 18.58, 18.49. HRMS (ESI) calcd for [M+H, $C_{19}H_{21}O_2$]$^+$: 281.1536, found: 281.1537.

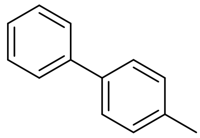


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.18 (t, *J* = 4.2 Hz, 1H), 7.01 – 6.98 (m, 1 H), 6.91 (d, *J* = 7.6 Hz, 1 H), 6.82 (d, *J* = 7.7 Hz, 1 H), 6.61 (d, *J* = 8.3 Hz, 1H), 6.48 (d, *J* = 8.3 Hz, 1 H), 5.01 (d, *J* = 10.7 Hz, 1 H), 3.81 (s, 3 H), 3.67 (s, 3 H), 2.46 (s, 3 H), 2.32 (s, 3 H), 1.67 – 1.25 (m, 13 H), 1.20 – 0.69 (m, 9 H), 0.18 (q, *J* = 12.4 Hz, 3 H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 158.68 (s), 156.97 (s), 143.02 (s), 142.23 (s), 139.06 (d, *J* = 8.0 Hz), 134.37 (d, *J* = 4.8 Hz), 132.10 (s), 129.38 (s), 128.37 (s), 127.68 (s), 127.28 (s), 115.60 (s), 104.24 (s), 103.90 (s), 55.74 (s), 47.35 (d, *J* = 16.0 Hz), 32.24 (s), 31.95 (s), 31.67 (s), 29.12 (s), 28.42 (s), 27.95 (s), 27.79 (s), 27.41 (s), 27.27 (d, *J* = 3.8 Hz), 27.15 (s), 26.78 (d, *J* = 11.2 Hz), 26.12 (d, *J* = 12.2 Hz), 21.61 (s), 18.29 (s). ^{31}P NMR (162 MHz, Chloroform-*d*) δ 37.46 (s) . HRMS (ESI) calcd for [M+Na, $C_{31}H_{44}BO_2NaP$]$^+$: 512.3101, found: 512.3102.

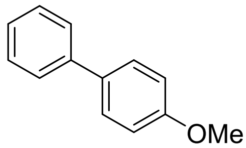


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39 (t, *J* = 8.4 Hz, 1 H), 7.29 – 7.25 (m, 1 H), 7.16 (d, *J* = 7.7 Hz, 1 H), 7.04 (d, *J* = 7.8 Hz, 1 H), 6.92 (t, *J* = 6.5 Hz, 0.5 H), 6.71 (d, *J* = 8.5 Hz, 2 H), 5.73 (t, *J* = 6.4 Hz, 0.5 H), 5.45 (d, *J* = 18.2 Hz, 1 H), 3.87 (s, 6H), 2.54 (s, 3 H), 2.44 (s, 3 H), 2.19 – 2.05 (m, 2 H), 1.87 – 1.54 (m, 10 H), 1.31 – 0.91 (m, 10 H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.82 (s), 143.41 (d, *J* = 2.3 Hz), 136.85 (d, *J* = 8.0 Hz), 136.52 (d, *J* = 5.1 Hz), 132.91 (d, *J* = 6.2 Hz), 131.32 (d, *J* = 3.1 Hz), 131.24 (s), 130.79 (s), 129.68 (s), 128.86 (s), 111.49 (s), 104.51 (s), 56.03 (s), 40.76 (d, *J* = 35.1 Hz), 29.38 (s), 29.02 (s), 28.64 (s), 28.31 (d, *J* = 4.1 Hz), 27.25 (d, *J* = 3.8 Hz), 27.05 (d, *J* = 3.5 Hz), 26.94 (d, *J* = 3.3 Hz), 26.59 (d, *J* = 1.5 Hz), 26.47 (d, *J* = 3.0 Hz), 26.34 (d, *J* = 1.0 Hz), 26.20 (s), 24.99 (d, *J* = 10.0 Hz), 19.63 (s), 18.31 (s). ^{31}P NMR (162

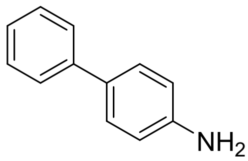
MHz, Chloroform-*d*) δ 18.86 (s). HRMS(ESI): m/z: $[M-BF_4]^+$ calculated for $C_{31}H_{42}O_2P$: 477.2917, found 477.2906.



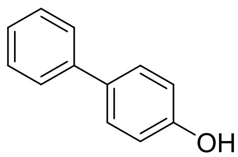
3a:^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 7.5 Hz, 2 H), 7.49 (d, *J* = 8.0 Hz, 2 H), 7.42 (t, *J* = 7.6 Hz, 2 H), 7.31 (t, *J* = 7.3 Hz, 1 H), 7.23 (d, *J* = 4.3 Hz, 2 H), 2.39 (s, 3 H). Data is consistent with that reported in the literature.[4]



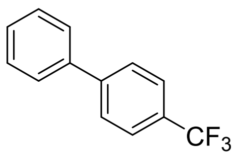
3b: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 (t, *J* = 8.3 Hz, 4 H), 7.43 (t, *J* = 7.7 Hz, 2 H), 7.31 (t, *J* = 7.3 Hz, 1 H), 6.99 (d, *J* = 8.7 Hz, 2 H), 3.86 (s, 3 H). Data is consistent with that reported in the literature.[5]



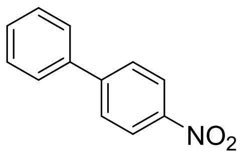
3c:^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 (dd, *J* = 8.1, 1.4 Hz, 2 H), 7.45 – 7.35 (m, 4 H), 7.27 (d, *J* = 7.5 Hz, 1 H), 6.78 – 6.70 (m, 2 H), 3.69 (s, 2 H). Data is consistent with that reported in the literature.[6]



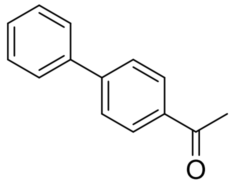
3d: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.54 (d, *J* = 7.4 Hz, 2 H), 7.48 (d, *J* = 8.5 Hz, 2 H), 7.41 (t, *J* = 7.6 Hz, 2 H), 7.30 (t, *J* = 7.3 Hz, 1 H), 6.90 (d, *J* = 8.6 Hz, 2 H), 4.86 (s, 1 H). Data is consistent with that reported in the literature.[7]



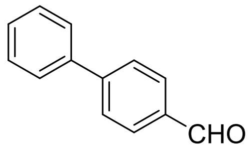
3e: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.69 (s, 4 H), 7.60 (d, *J* = 7.6 Hz, 2 H), 7.47 (t, *J* = 7.5 Hz, 2 H), 7.40 (t, *J* = 7.3 Hz, 1 H). Data is consistent with that reported in the literature.[8]



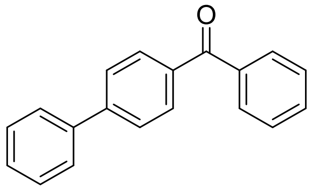
3f: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.33 – 8.25 (m, 2 H), 7.77 – 7.70 (m, 2 H), 7.62 (dt, *J* = 6.0, 1.4 Hz, 2 H), 7.55 – 7.40 (m, 3 H).[5]



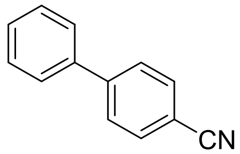
3g: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 8.4 Hz, 2 H), 7.69 (d, *J* = 8.4 Hz, 2 H), 7.63 (d, *J* = 7.1 Hz,2 H), 7.47 (t, *J* = 7.4 Hz, 2 H), 7.40 (t, *J* = 7.3 Hz, 1 H), 2.64 (s, 3 H).[9]



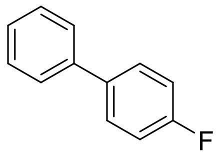
3h: 1H NMR (400 MHz, Chloroform-d) δ 10.05 (s, 1 H), 7.95 (d, J = 8.3 Hz, 2 H), 7.75 (d, J = 8.2 Hz, 2 H), 7.68 – 7.59 (m, 2 H), 7.51 – 7.45 (m, 2 H), 7.44 – 7.39 (m, 1H).[10]



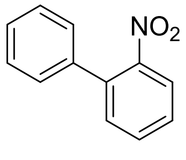
3i: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.2 Hz, 2 H), 7.83 (d, *J* = 7.2 Hz, 2 H), 7.70 (d, *J* = 8.2 Hz, 2 H), 7.64 (d, *J* = 7.3 Hz, 2 H), 7.59 (t, *J* = 7.4 Hz, 1 H), 7.48 (q, *J* = 7.7 Hz, 4 H), 7.40 (t, *J* = 7.3 Hz, 1 H).[11]



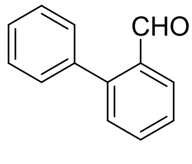
3j: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.70 (q, *J* = 8.4 Hz, 4 H), 7.59 (d, *J* = 7.2 Hz, 2 H), 7.48 (t, *J* = 7.4 Hz, 2 H), 7.42 (t, *J* = 7.2 Hz, 1 H).[5]



3k: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 (d, *J* = 7.2 Hz, 4 H), 7.43 (t, *J* = 7.5 Hz, 2 H), 7.34 (t, *J* = 7.3 Hz, 1 H), 7.12 (t, *J* = 8.7 Hz, 2 H).[12]

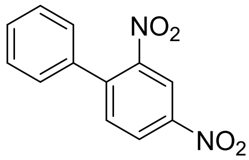


3l: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84 (dd, *J* = 8.1, 1.1 Hz, 1 H), 7.60 (td, *J* = 7.6, 1.2 Hz, 1 H), 7.50 – 7.38 (m, 5 H), 7.34 – 7.29 (m, 2 H).[10]

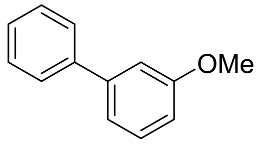


3m: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.98 (s, 1 H), 8.03 (d, *J* = 7.8 Hz, 1 H), 7.64 (t, *J* = 7.5 Hz, 1

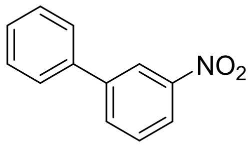
H), 7.47 (dt, J = 14.2, 7.4 Hz, 5 H), 7.38 (d, J = 7.8 Hz, 2 H). [13]



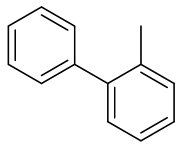
3n:[1]H NMR (400 MHz, Chloroform-*d*) δ 8.71 (d, J = 2.3 Hz, 1 H), 8.47 (d, J = 10.6 Hz, 1 H), 7.68 (d, J = 8.4 Hz, 1 H), 7.49 (d, J = 2.6 Hz,3 H), 7.34 (d, J = 3.8 Hz, 2 H).[12]



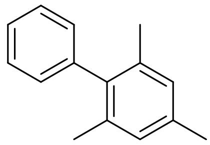
3o: [1]H NMR (400 MHz, Chloroform-*d*) δ 7.59 (d, J = 8.1 Hz, 2 H), 7.43 (t, J = 7.6 Hz, 2 H), 7.35 (dt, J = 7.8, 4.1 Hz, 2 H), 7.18 (d, J = 7.7 Hz, 1 H), 7.12 (s, 1 H), 6.90 (dd, J = 8.7, 2.0 Hz, 1 H), 3.86 (s, 3 H).[14]



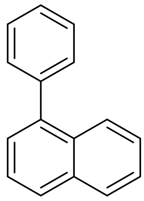
3p: [1]H NMR (400 MHz, Chloroform-*d*) δ 8.44 (s, 1 H), 8.24 – 8.15 (m, 1 H), 7.90 (d, J = 7.7 Hz, 1 H), 7.60 (t, J = 9.3 Hz, 3 H), 7.46 (dt, J = 26.0, 7.2 Hz, 3 H).[12]



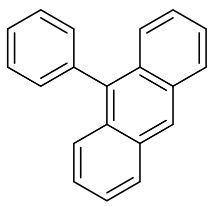
3q: [1]H NMR (400 MHz, Chloroform-*d*) δ 7.23 (d, J = 10.7 Hz, 8 H), 2.40 (s, 3 H), 2.27 (s, 3 H).[10]



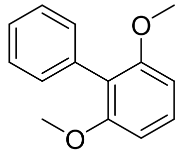
3s: [1]H NMR (400 MHz, Chloroform-*d*) δ 7.45 – 7.38 (m, 2 H), 7.36 – 7.29 (m, 1 H), 7.16 – 7.11 (m, 2 H), 6.94 (s, 2 H), 2.48 – 2.15 (m, 3 H), 2.00 (s, 6 H).[15]



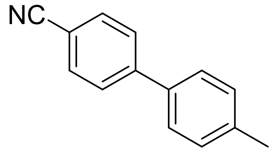
3t: [1]H NMR (400 MHz, Chloroform-*d*) δ 7.93 – 7.81 (m, 3 H), 7.54 – 7.37 (m, 9 H).[5]



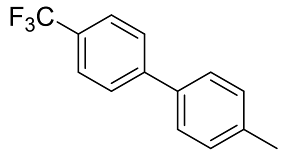
3u: [1]H NMR (400 MHz, Chloroform-*d*) δ 8.49 (s, 1 H), 8.04 (d, J = 8.4 Hz, 2 H), 7.67 (s, 2 H), 7.56 (dt, J = 13.6, 6.9 Hz, 3 H), 7.49 – 7.40 (m, 4 H), 7.38 – 7.31 (m, 2 H).[16]



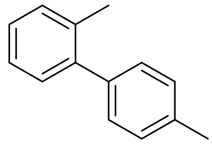
3v: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45 – 7.22 (m, 6 H), 6.72 – 6.60 (m, 2 H), 3.73 (s, 6 H).[17]



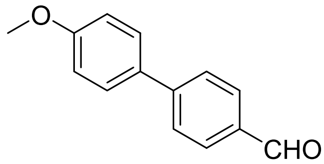
3aa: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.68 (q, *J* = 8.5 Hz, 4 H), 7.49 (d, *J* = 8.1 Hz, 2 H), 7.28 (d, *J* = 8.0 Hz, 2 H), 2.41 (s, 3 H).[18]



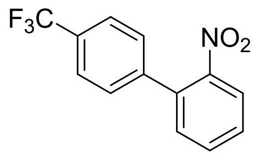
3ab: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.67 (s, 4 H), 7.50 (d, *J* = 8.0 Hz, 2 H), 7.31 – 7.24 (m, 2 H), 2.41 (s, 3 H). [18]



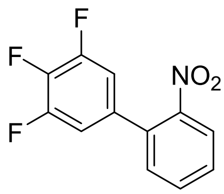
3ac: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.27 – 7.20 (m, *J* = 10.7 Hz, 8 H), 2.40 (s, 3 H), 2.27 (s, 3 H).[19]



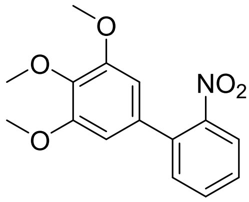
3ae: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.03 (s, 1 H), 7.92 (d, *J* = 8.2 Hz, 2 H), 7.71 (d, *J* = 8.2 Hz, 2 H), 7.59 (d, *J* = 8.7 Hz, 2 H), 7.01 (d, *J* = 8.7 Hz, 2 H), 3.86 (s, 3 H). Data is consistent with that reported in the literature.[20]



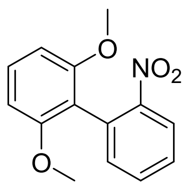
3af: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 (dd, *J* = 8.1, 1.3 Hz, 1 H), 7.71 – 7.64 (m, 3 H), 7.56 (td, *J* = 7.8, 1.5 Hz, 1 H), 7.46 – 7.40 (m, 3 H). Data is consistent with that reported in the literature.[21]



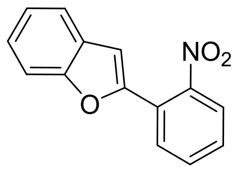
3ag: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 8.0 Hz, 1 H), 7.66 (td, *J* = 7.6, 1.2 Hz, 1 H), 7.57 (td, *J* = 8.0, 1.3 Hz, 1 H), 7.39 (dd, *J* = 7.6, 1.2 Hz, 1 H), 6.95 (t, *J* = 7.7 Hz, 2 H). Data is consistent with that reported in the literature.[22]



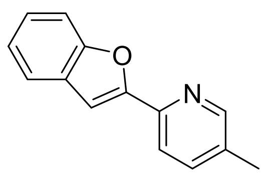
3ah: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.80 (d, *J* = 7.3 Hz, 1 H), 7.61 (t, *J* = 16.4 Hz, 1 H), 7.48 (t, *J* = 7.8 Hz, 2 H), 6.52 (s, 2 H), 3.90 (s, 3 H), 3.86 (s, 6 H). Data is consistent with that reported in the literature.[22]



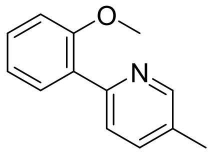
3ai:^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 8.0 Hz, 1 H), 7.63 – 7.58 (m, 1 H), 7.48 – 7.40 (m, 2 H), 7.31 (t, *J* = 8.4 Hz, 1 H), 6.64 (d, *J* = 8.4 Hz, 2 H), 3.71 (s, 6 H). Data is consistent with that reported in the literature.[23]



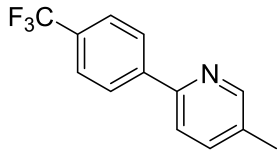
3a′:^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84 (dd, *J* = 7.8, 1.1 Hz, 1H), 7.76 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.65 – 7.58 (m, 2H), 7.53 – 7.44 (m, 2 H), 7.36 – 7.30 (m, 1H), 7.28 – 7.23 (m, 1H), 7.00 (d, *J* = 0.9 Hz, 1H). Data is consistent with that reported in the literature.[24]



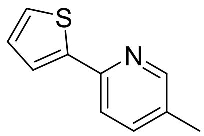
3b′: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.50 (s, 1 H), 7.78 (d, *J* = 8.1 Hz, 1 H), 7.63 (d, *J* = 7.5 Hz, 1 H), 7.56 (dd, J = 8.2, 2.8 Hz, 2 H), 7.35 (s, 1 H), 7.34 – 7.29 (m, 1 H), 7.27 – 7.22 (m, 1 H), 2.36 (s, 3 H). ^{13}C NMR (101 MHz, Chloroform-d) δ 155.36, 155.24, 150.42, 146.74, 137.30, 132.81, 129.03, 125.01, 123.20, 121.61, 119.53, 111.55, 104.04, 77.48, 77.16, 76.84, 18.54. HRMS (ESI) calcd for [M+H, $C_{14}H_{12}NO]^+$: 210.0913, found: 210.0915.



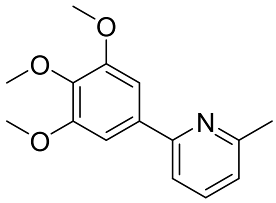
3c′: ^{1}H NMR (400 MHz, Chloroform-d) δ 8.52 (d, J = 2.2 Hz, 1 H), 7.71 (ddd, J = 15.8, 7.8, 1.4 Hz, 2 H), 7.49 (ddd, J = 8.0, 2.3, 0.8 Hz, 1 H), 7.34 (ddd, J = 8.3, 7.4, 1.8 Hz, 1 H), 7.06 (td, J = 7.5, 1.1 Hz, 1 H), 6.98 (dd, J = 8.3, 1.0 Hz, 1 H), 3.83 (s, 3 H), 2.35 (s, 3 H). Data is consistent with that reported in the literature.[25]



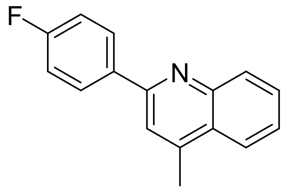
3d′: ^{1}H NMR (400 MHz, Chloroform-d) δ 8.54 (d, J = 1.4 Hz, 1 H), 8.07 (d, J = 8.1 Hz, 2 H), 7.70 (d, J = 8.2 Hz, 2 H), 7.64 (d, J = 8.0 Hz, 1 H), 7.57 (dd, J = 8.1, 2.2 Hz,1 H), 2.38 (s, 3 H). Data is consistent with that reported in the literature.[26]



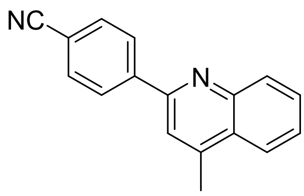
3e′: ^{1}H NMR (400 MHz, Chloroform-d) δ 8.39 (d, J = 1.8 Hz, 1 H), 7.58 – 7.46 (m, 3 H), 7.36 (dd, J = 5.1, 1.0 Hz, 1 H), 7.10 (dd, J = 5.0, 3.7 Hz, 1 H), 2.34 (s, 3 H). Data is consistent with that reported in the literature.[27]



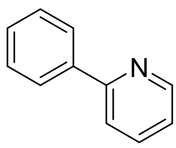
3f′: ^{1}H NMR (400 MHz, Chloroform-d) δ 7.61 (t, J = 7.7 Hz, 1 H), 7.46 (d, J = 7.8 Hz, 1 H), 7.21 (s, 1 H), 7.07 (d, J = 7.6 Hz, 1 H), 3.95 (s, 6 H), 3.89 (s, 3 H), 2.62 (s, 3 H). Data is consistent with that reported in the literature.[28]



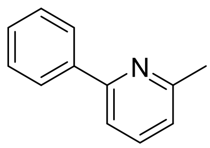
3g′: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.17 (d, *J* = 8.4 Hz, 1 H), 8.05 (td, *J* = 7.8, 1.8 Hz, 1 H), 8.00 (d, *J* = 8.3 Hz, 1 H), 7.75 – 7.68 (m, 2 H), 7.56 (t, *J* = 8.1 Hz, 1 H), 7.41 (qd, *J* = 7.3, 1.8 Hz, 1 H), 7.30 (td, *J* = 7.6, 1.0 Hz, 1 H), 7.19 (dd, *J* = 10.7, 8.7 Hz, 1 H), 2.74 (s, 3 H). Data is consistent with that reported in the literature.[29]



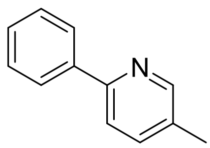
3h′:^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.27 (d, *J* = 8.6 Hz, 1H), 8.17 (d, *J* = 8.4 Hz, 1H), 8.02 (d, *J* = 9.2 Hz, 1H), 7.83 – 7.71 (m, 2H), 7.60 (ddd, *J* = 8.2, 6.9, 1.2 Hz, 1H), 2.79 (s, 1H). Data is consistent with that reported in the literature.[29]



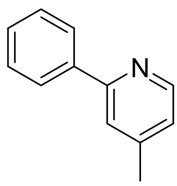
3i′: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.69 (d, *J* = 4.8 Hz, 1 H), 8.02 – 7.96 (m, 2 H), 7.72 (dd, *J* = 6.1, 1.4 Hz, 2 H), 7.47 (t, *J* = 7.4 Hz, 2 H), 7.41 (t, *J* = 7.2 Hz, 1 H), 7.25 – 7.19 (m, 1H). Data is consistent with that reported in the literature. [12]



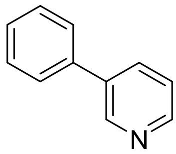
3j′:^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 7.2 Hz, 2 H), 7.62 (t, *J* = 7.7 Hz, 1 H), 7.53 – 7.43 (m, 3 H), 7.39 (t, *J* = 7.3 Hz, 1 H), 7.08 (d, *J* = 7.6 Hz, 1 H), 2.62 (s, 3 H). Data is consistent with that reported in the literature. [30]



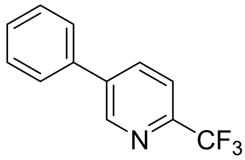
3k′: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.52 (d, *J* = 2.0 Hz, 1 H), 7.98 – 7.94 (m, 2 H), 7.62 (d, *J* = 8.0 Hz, 1 H), 7.55 (dd, *J* = 8.1, 1.8 Hz, 1 H), 7.50 – 7.43 (m, 2 H), 7.41 – 7.36 (m, 1 H), 2.36 (s, 3 H). Data is consistent with that reported in the literature. [31]



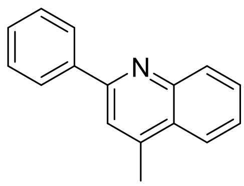
3l′: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.55 (d, *J* = 5.0 Hz, 1 H), 7.97 (d, *J* = 7.3 Hz, 2 H), 7.54 (s, 1 H), 7.43 (dt, *J* = 25.8, 7.2 Hz, 3 H), 7.05 (d, *J* = 4.8 Hz, 1 H), 2.41 (s, 3 H). Data is consistent with that reported in the literature. [30]



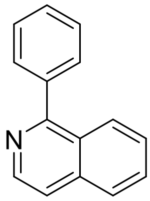
3m′: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.86 (s, 1 H), 8.60 (s, 1 H), 7.87 (dt, *J* = 7.9, 1.9 Hz, 1 H), 7.58 (d, *J* = 7.3 Hz, 2 H), 7.48 (t, *J* = 7.5 Hz, 2 H), 7.44 – 7.35 (m, 2 H). Data is consistent with that reported in the literature. [10]



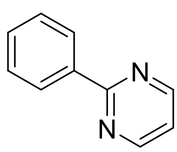
3n′: 1H NMR (400 MHz, Chloroform-*d*) δ 8.95 (d, *J* = 1.8 Hz, 1 H), 8.04 (dd, *J* = 8.1, 1.7 Hz, 1 H), 7.76 (d, *J* = 8.1 Hz, 1H), 7.64 – 7.57 (m, 2 H), 7.55 – 7.45 (m, 3 H). Data is consistent with that reported in the literature. [32]



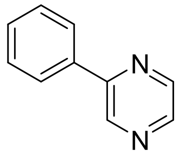
3o′: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 – 8.10 (m, 3 H), 7.95 (d, *J* = 8.3 Hz, 1 H), 7.73 – 7.65 (m, 2 H), 7.50 (t, *J* = 8.1 Hz, 1 H), 7.43 (t, *J* = 7.2 Hz, 1 H), 2.71 (s, 3 H). Data is consistent with that reported in the literature. [33]



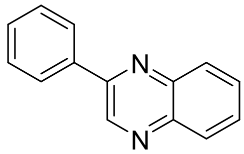
3p′(3p″): ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.61 (d, *J* = 5.7 Hz, 1 H), 8.09 (d, *J* = 8.5 Hz, 1 H), 7.86 (d, *J* = 8.2 Hz, 1 H), 7.73 – 7.59 (m, 4 H), 7.56 – 7.45 (m, 4 H). Data is consistent with that reported in the literature. [30]



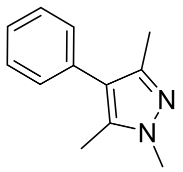
3q′: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.81 (d, *J* = 4.8 Hz, 2 H), 8.44 (dd, *J* = 6.8, 3.0 Hz, 2 H), 7.50 (dd, *J* = 5.0, 1.7 Hz, 3 H), 7.18 (t, *J* = 4.8 Hz, 1 H). Data is consistent with that reported in the literature. [34]



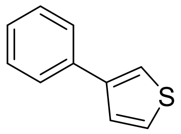
3r′: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.03 (s, 1 H), 8.63 (s, 1 H), 8.50 (s, 1 H), 8.01 (d, *J* = 6.9 Hz, 2 H), 7.55 – 7.44 (m, 3 H). Data is consistent with that reported in the literature. [35]



3s′:^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.34 (s, 1 H), 8.20 (dd, *J* = 8.1, 1.4 Hz, 2 H), 8.15 (ddd, *J* = 15.2, 8.4, 1.6 Hz, 2 H), 7.82 – 7.73 (m, 2 H), 7.61 – 7.51 (m, 3 H). Data is consistent with that reported in the literature. [36]



3t′: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40 (t, *J* = 7.5 Hz, 2 H), 7.30 – 7.22 (m, 3 H), 3.78 (s, 3 H), 2.25 (s, 3 H), 2.24 (s, 3 H). ^{13}C NMR (101 MHz, Chloroform-d) δ 145.05, 136.28, 134.25, 129.47, 128.44, 126.18, 119.24, 36.02, 12.51, 10.30. HRMS (ESI) calcd for [M+H, $C_{12}H_{15}N_2$]$^+$: 187.1229, found: 187.1227.



3u′: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.62 – 7.57 (m, 2 H), 7.44 (dd, *J* = 2.5, 1.7 Hz, 1 H), 7.42 – 7.36 (m, 4 H), 7.28 (t, *J* = 7.4 Hz, 1 H). Data is consistent with that reported in the literature. [37]

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

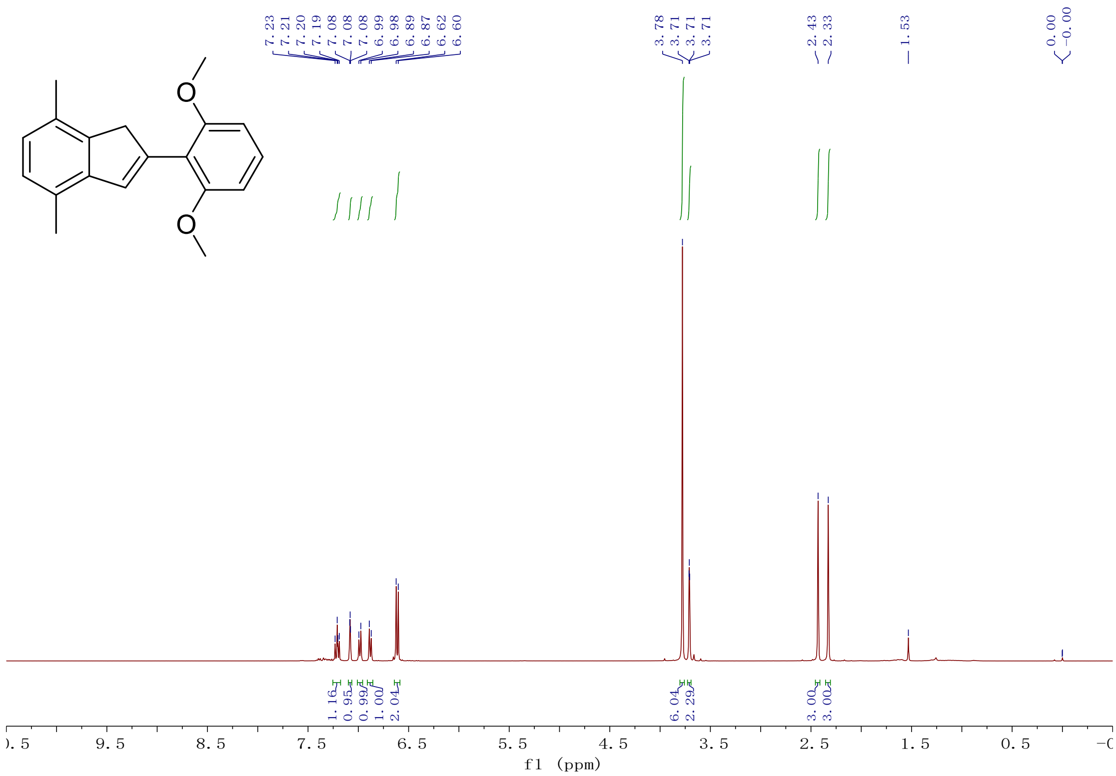


Figure S2. ^{1}H NMR spectrum of **S3** in $CDCl_3$

^{13}C NMR (101 MHz, $CDCl_3$)

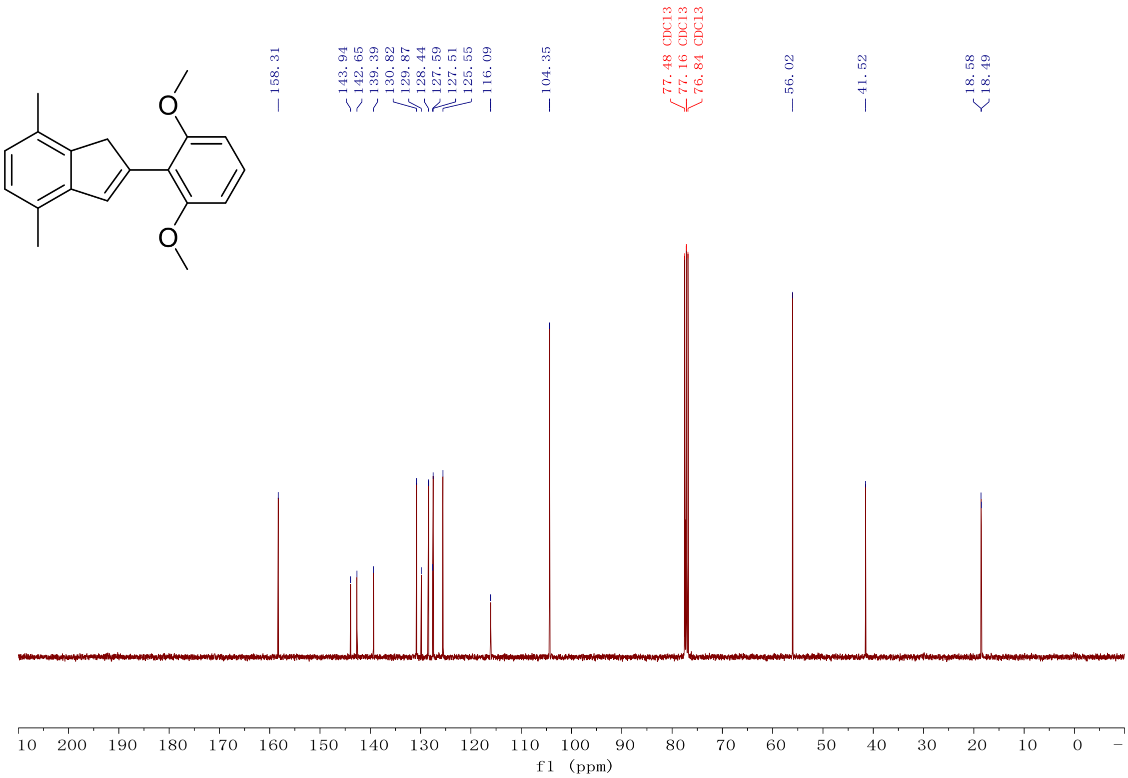


Figure S3. ^{13}C NMR spectrum of **S3** in $CDCl_3$

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

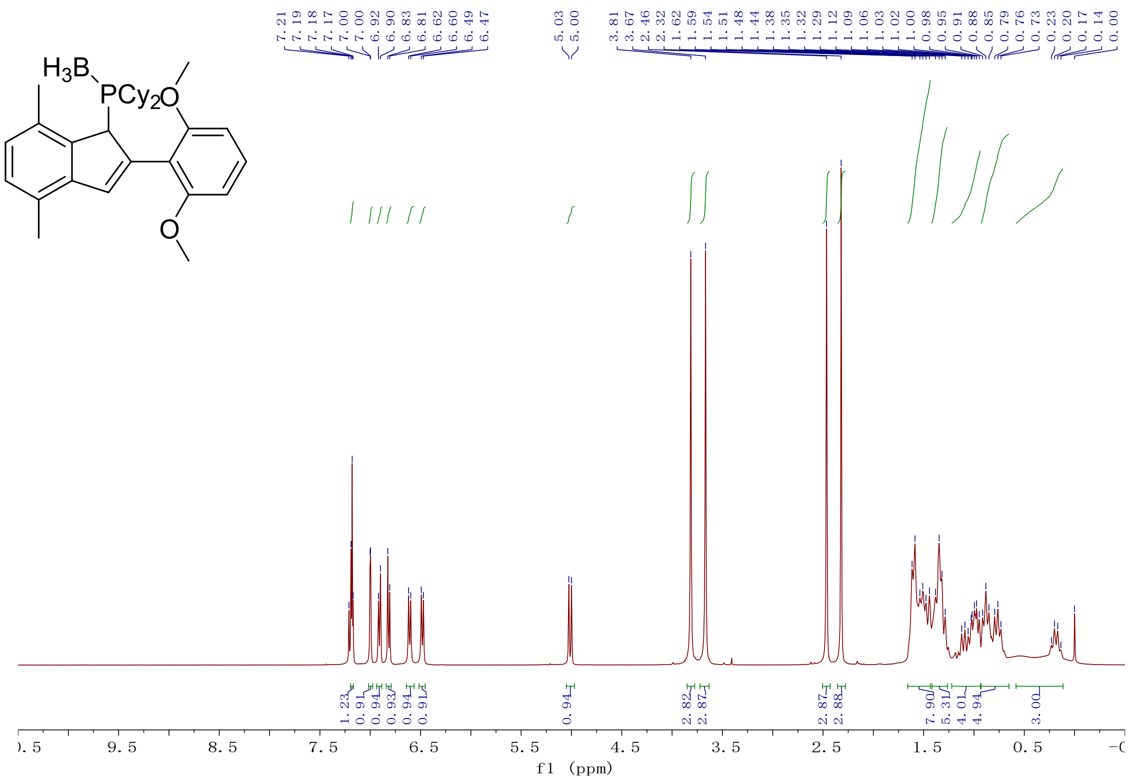


Figure S4. ^{1}H NMR spectrum of **L2** in $CDCl_3$

^{13}C NMR (101 MHz, $CDCl_3$)

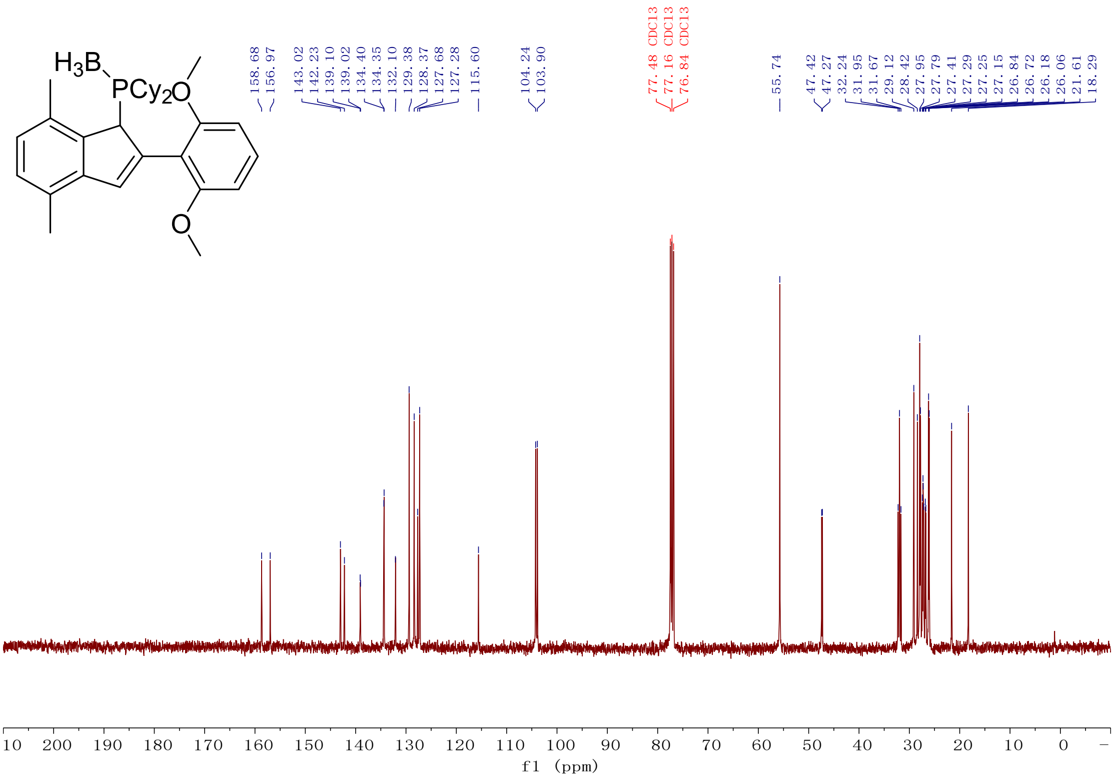


Figure S5. ^{13}C NMR spectrum of **L2** in $CDCl_3$

^{31}P NMR (162 MHz, $CDCl_3$)

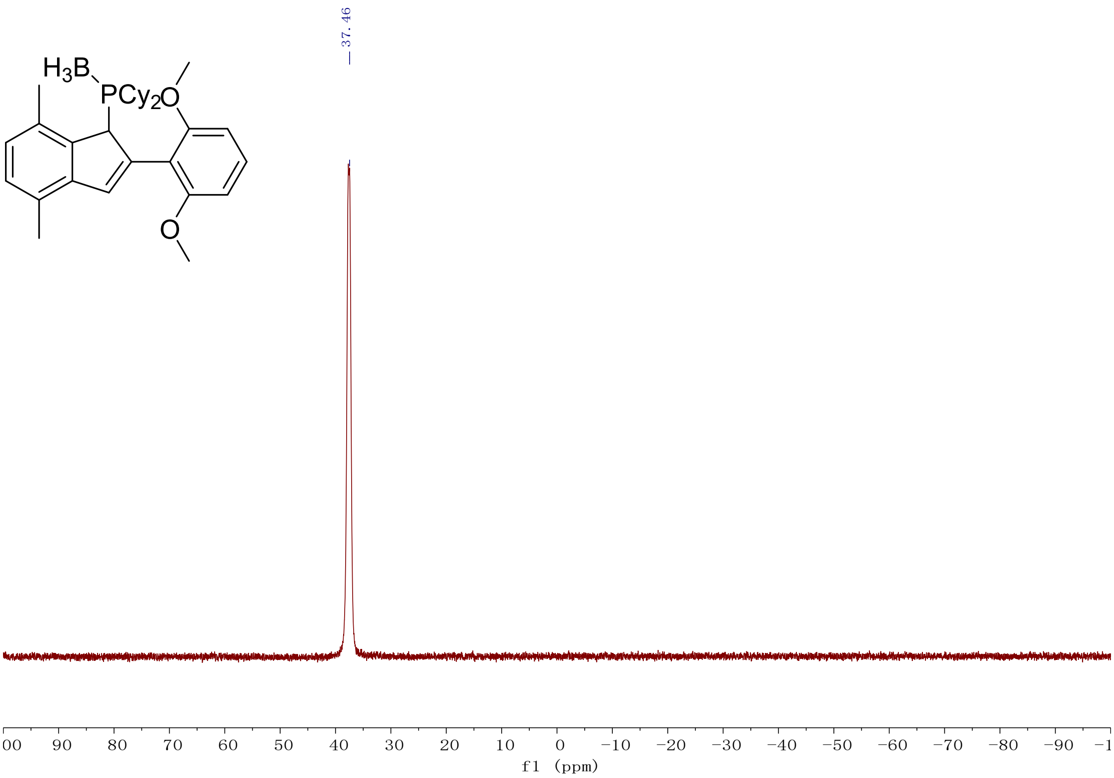


Figure S6. ^{31}P NMR spectrum of **L2** in $CDCl_3$

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

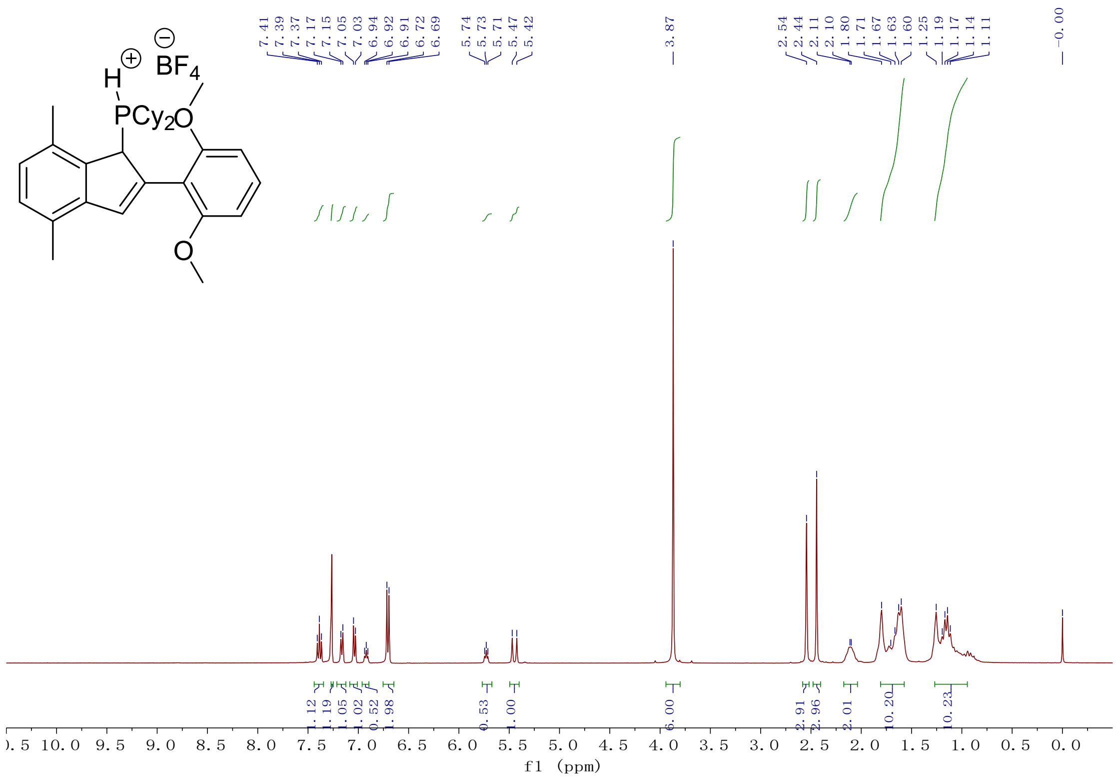


Figure S7. ^{1}H NMR spectrum of **L6** in $CDCl_3$

^{13}C NMR (101 MHz, $CDCl_3$)

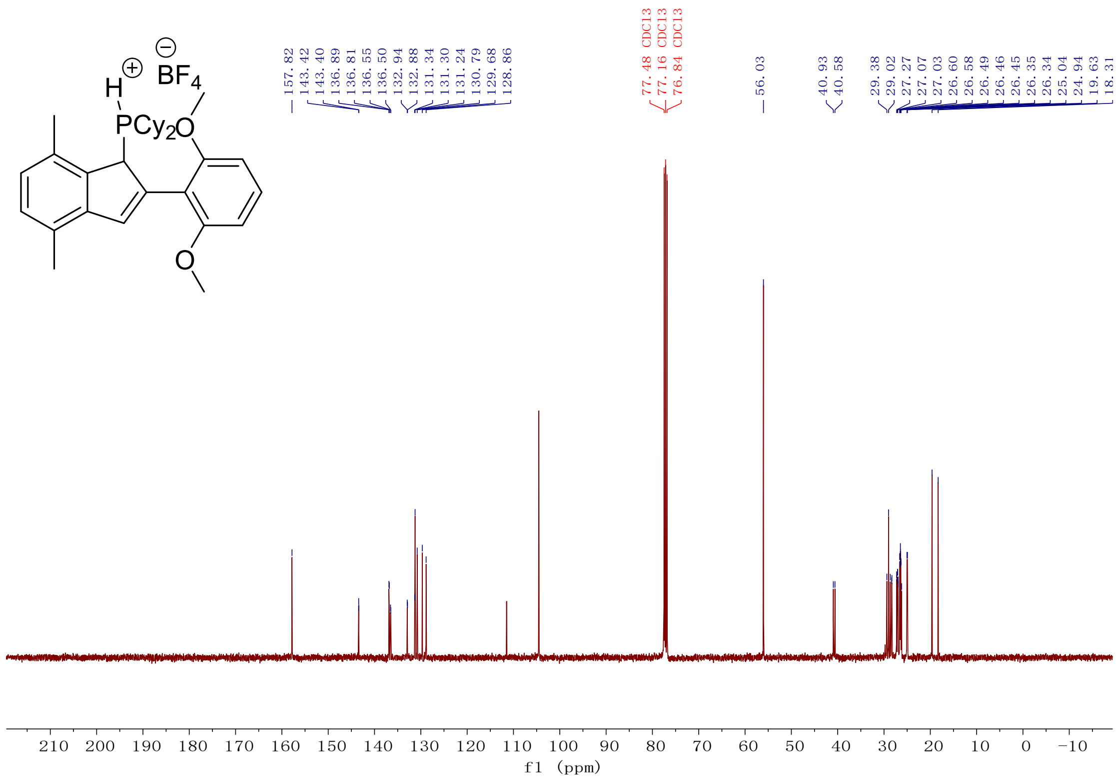


Figure S8. ^{13}C NMR spectrum of **L6** in $CDCl_3$

^{31}P NMR (162 MHz, $CDCl_3$)

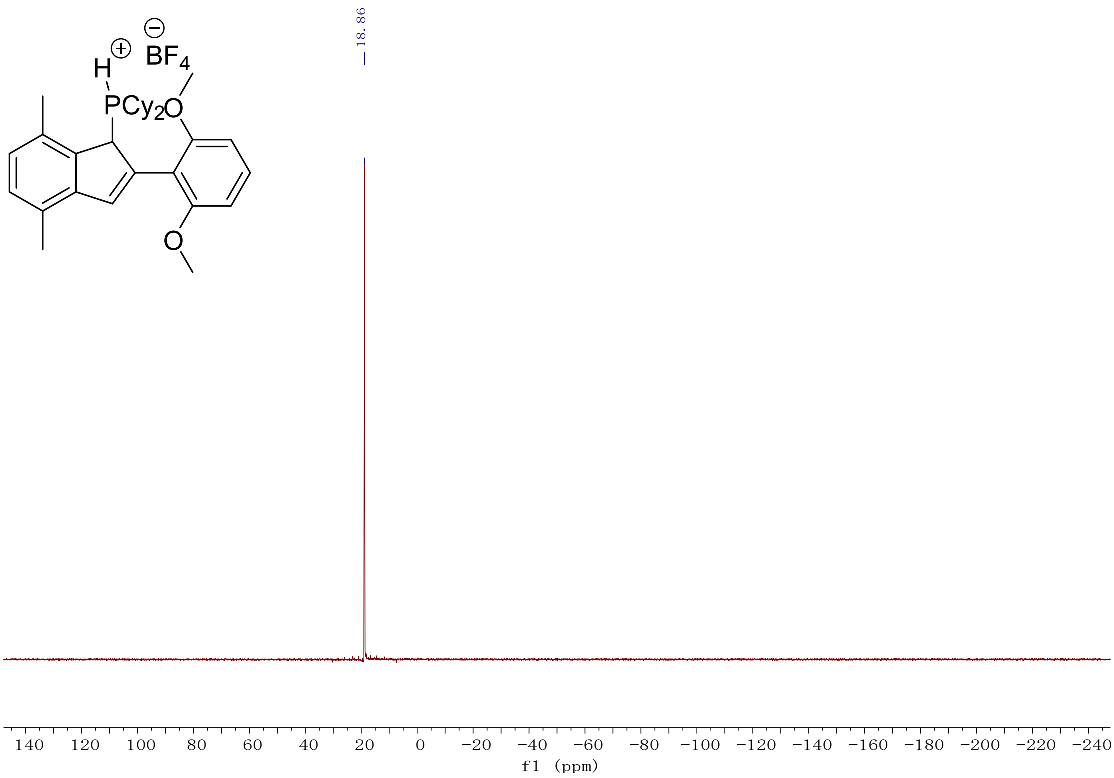


Figure S9. ^{31}P NMR spectrum of **L6** in $CDCl_3$

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

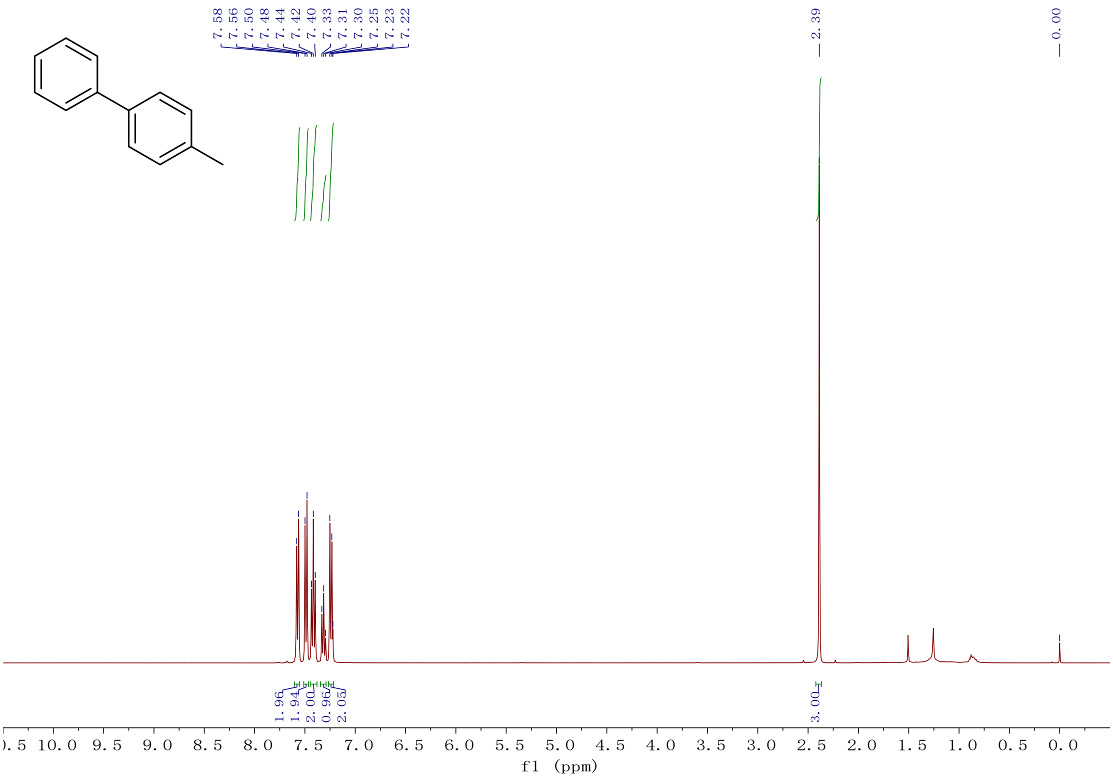


Figure S10. ^{1}H NMR spectrum of **3a** in $CDCl_3$

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

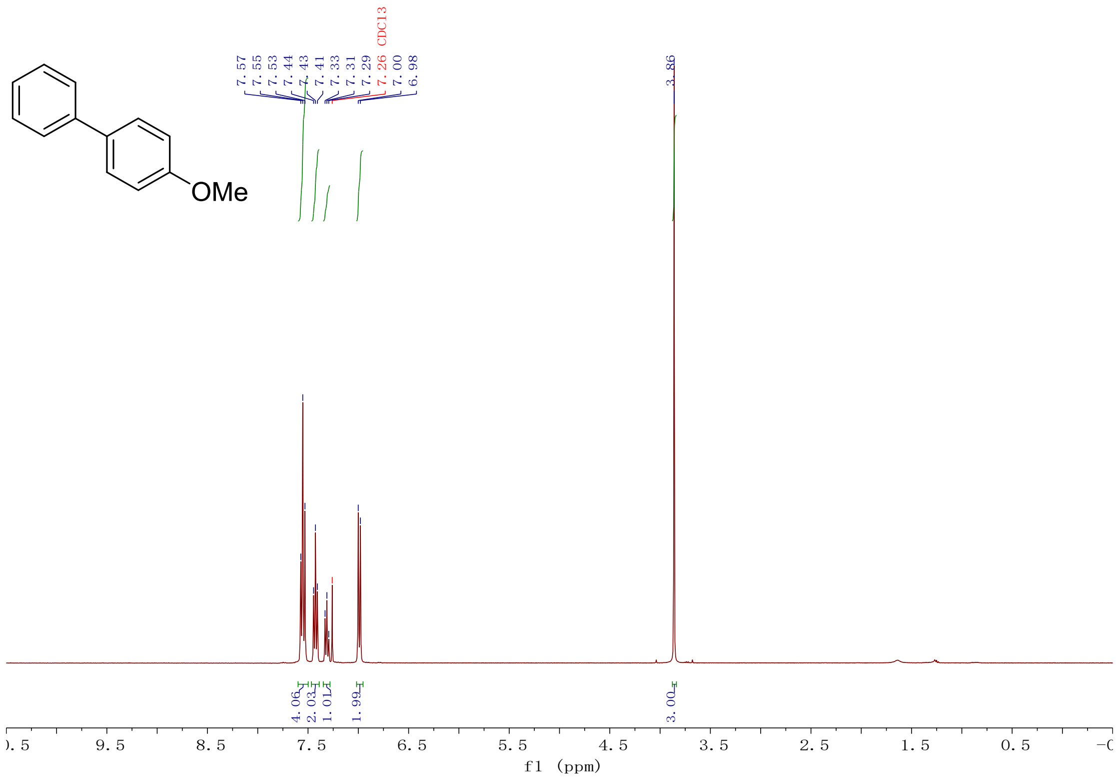


Figure S11. [1]H NMR spectrum of **3b** in $CDCl_3$

[1]H NMR (400 MHz, $CDCl_3$)

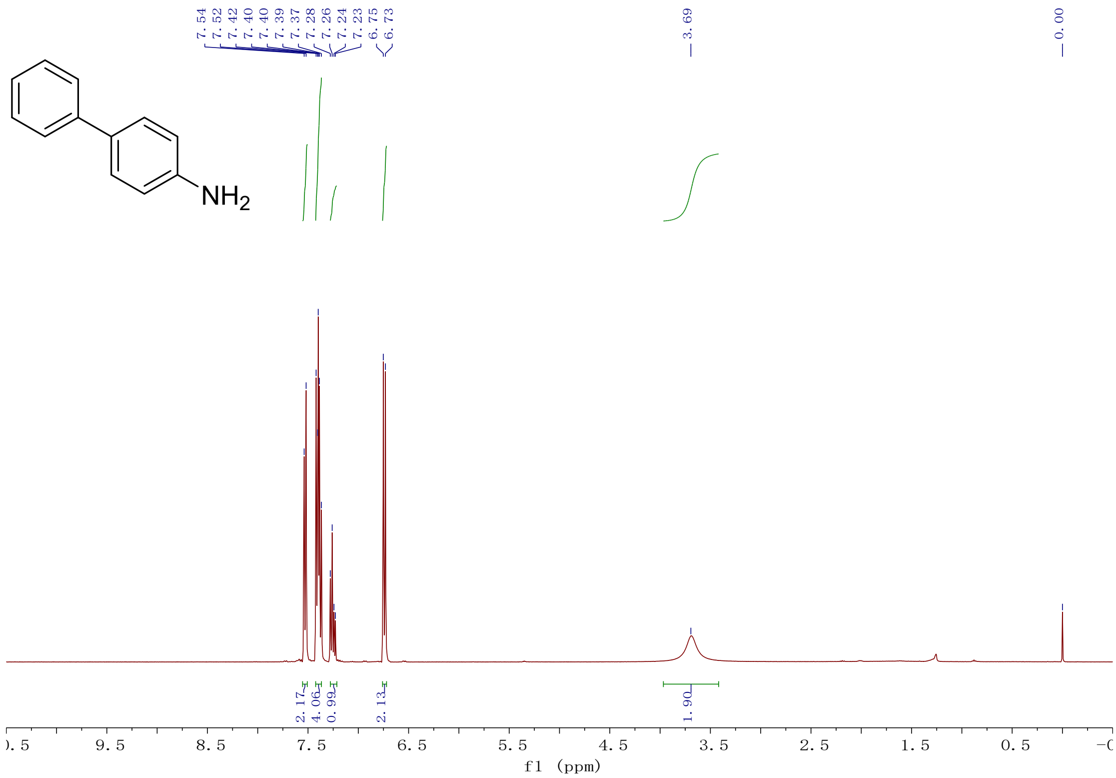


Figure S12. [1]H NMR spectrum of **3c** in $CDCl_3$

[1]H NMR (400 MHz, $CDCl_3$)

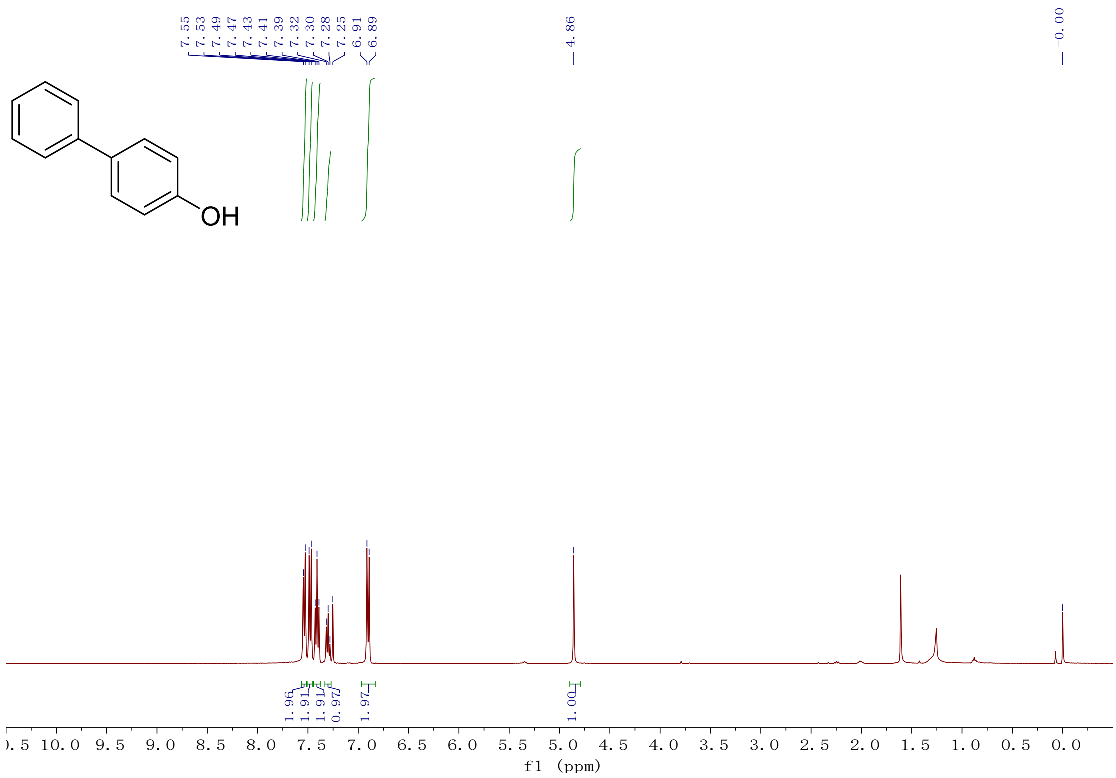


Figure S13. ^{1}H NMR spectrum of **3d** in $CDCl_3$

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

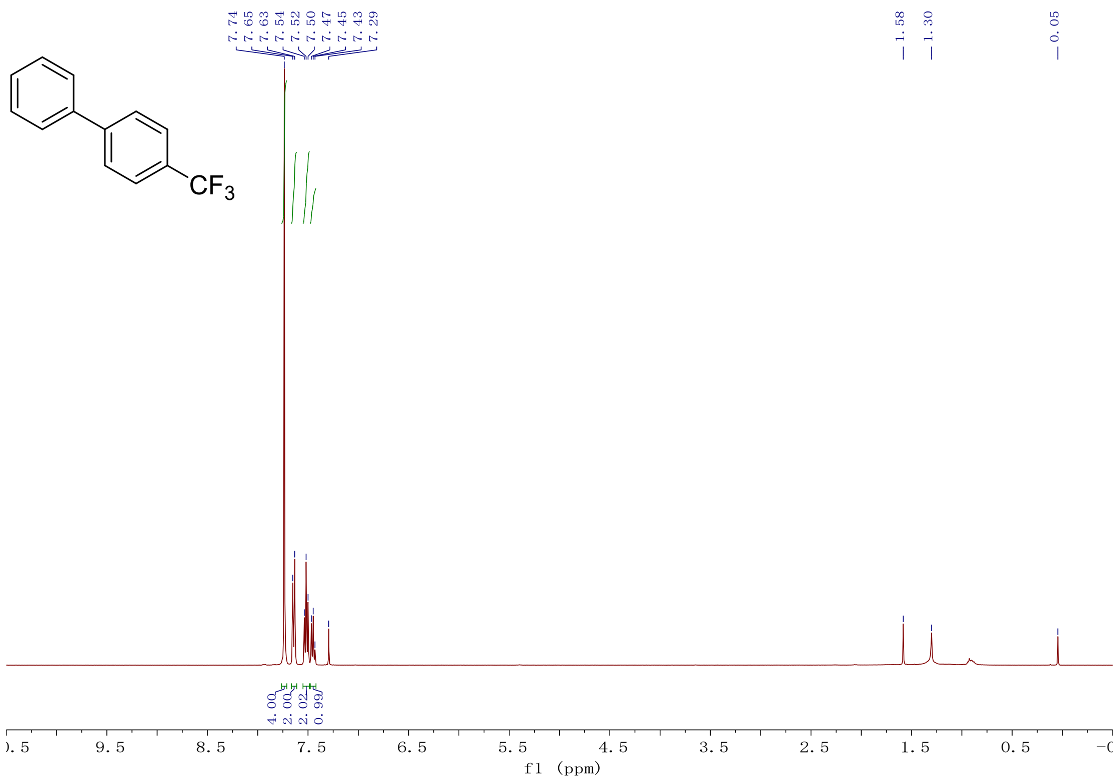


Figure S14. ^{1}H NMR spectrum of **3e** in $CDCl_3$

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

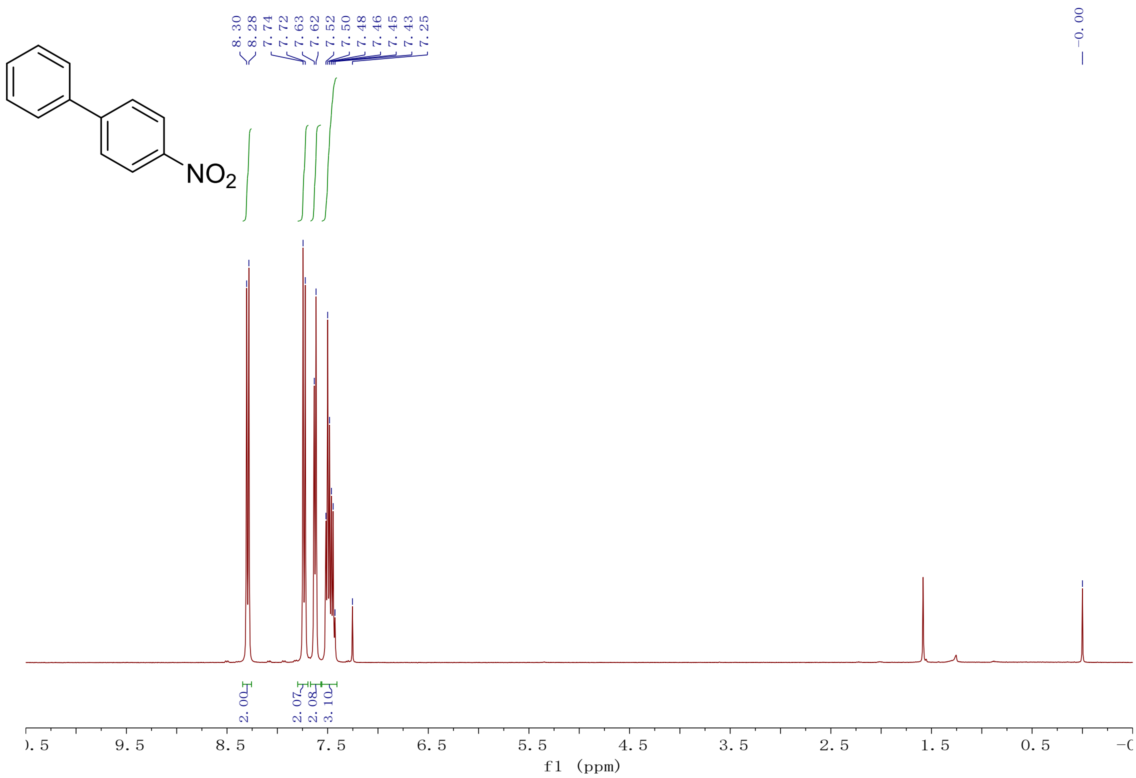


Figure S15. ^{1}H NMR spectrum of **3f** in $CDCl_3$

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

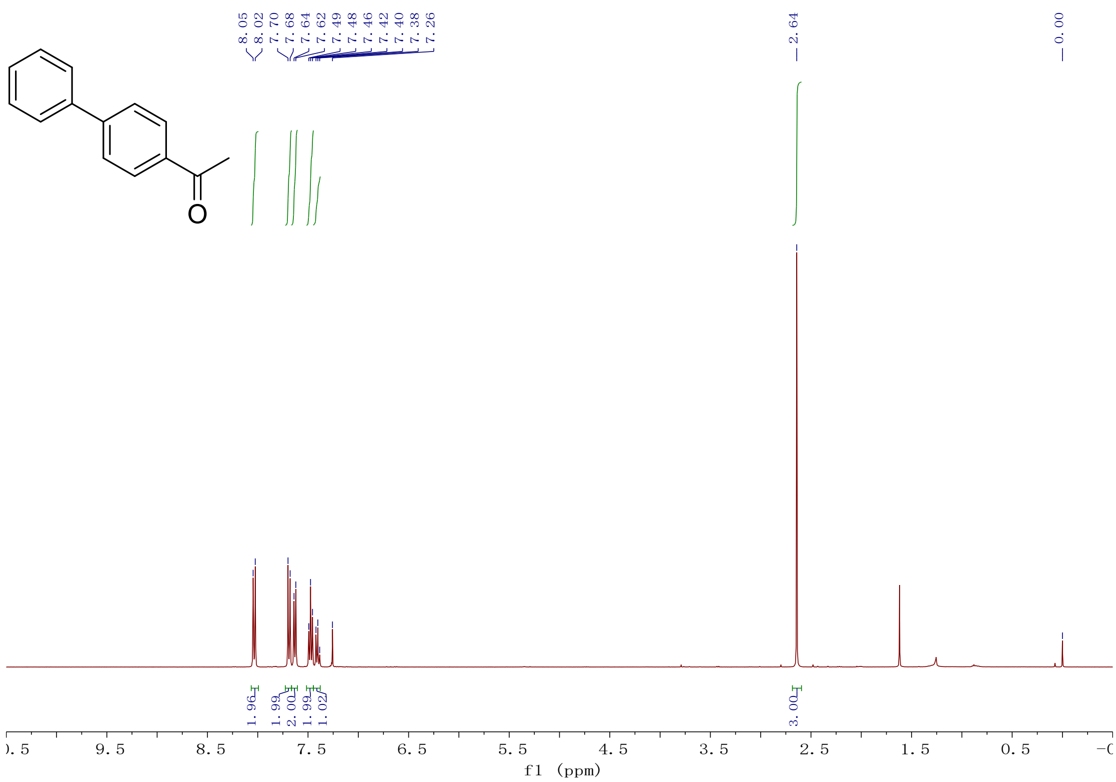


Figure S16. ^{1}H NMR spectrum of **3g** in $CDCl_3$

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

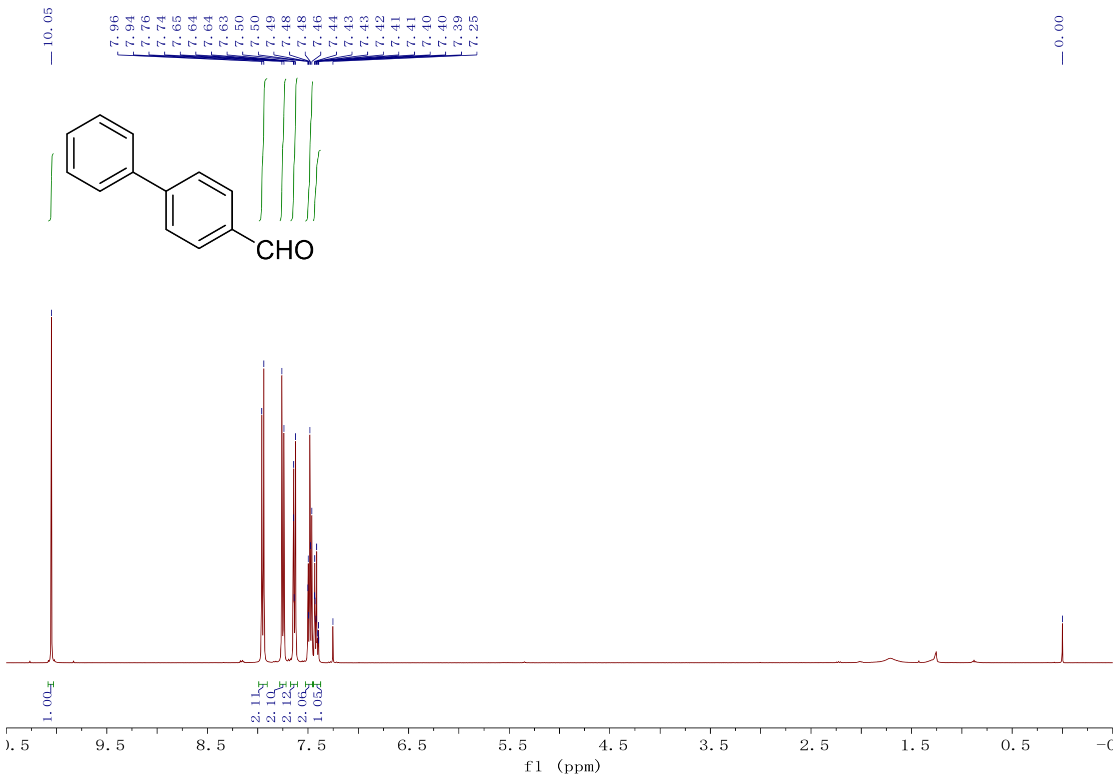


Figure S17. ^{1}H NMR spectrum of **3h** in $CDCl_3$

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

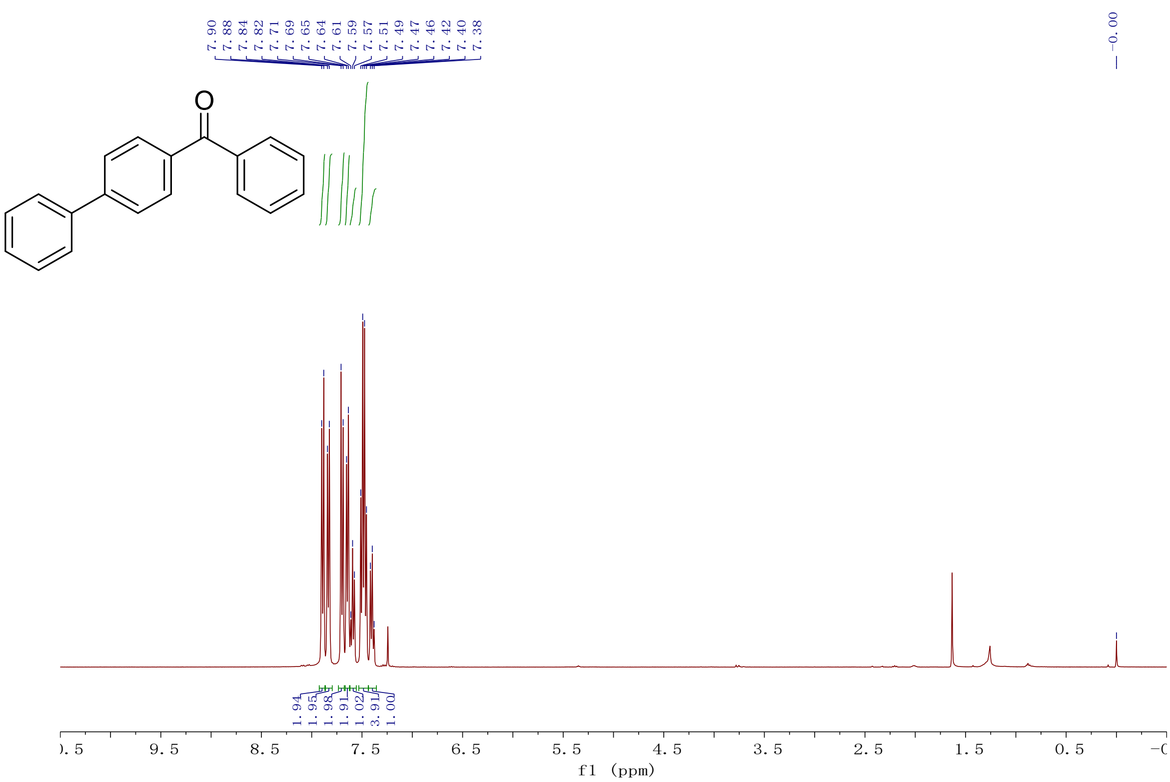


Figure S18. ^{1}H NMR spectrum of **3i** in $CDCl_3$

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

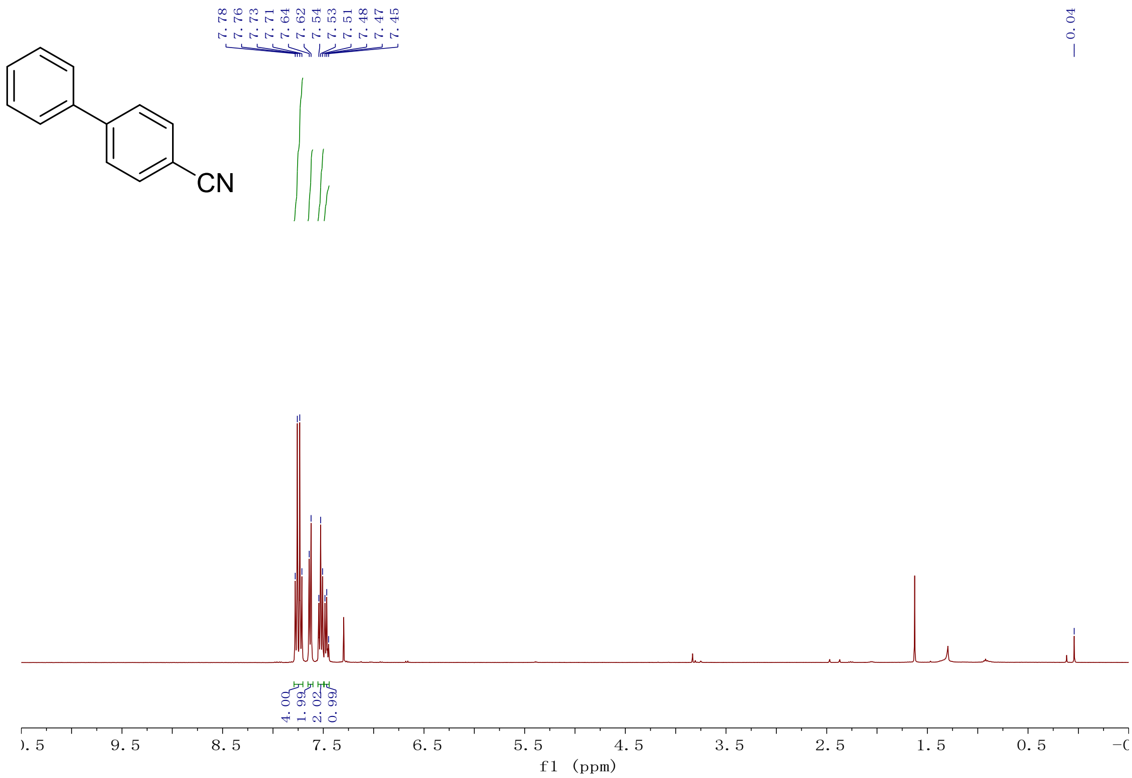


Figure S19. ^{1}H NMR spectrum of **3j** in $CDCl_3$

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

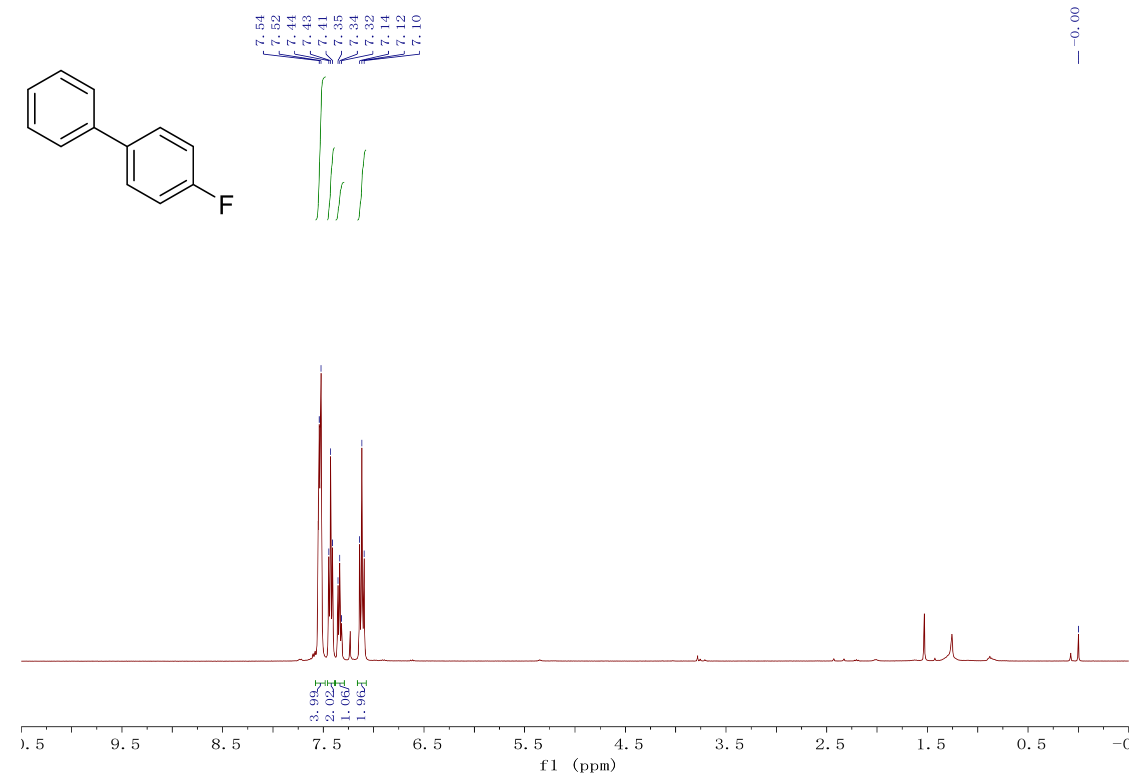


Figure S20. ^{1}H NMR spectrum of **3k** in $CDCl_3$

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

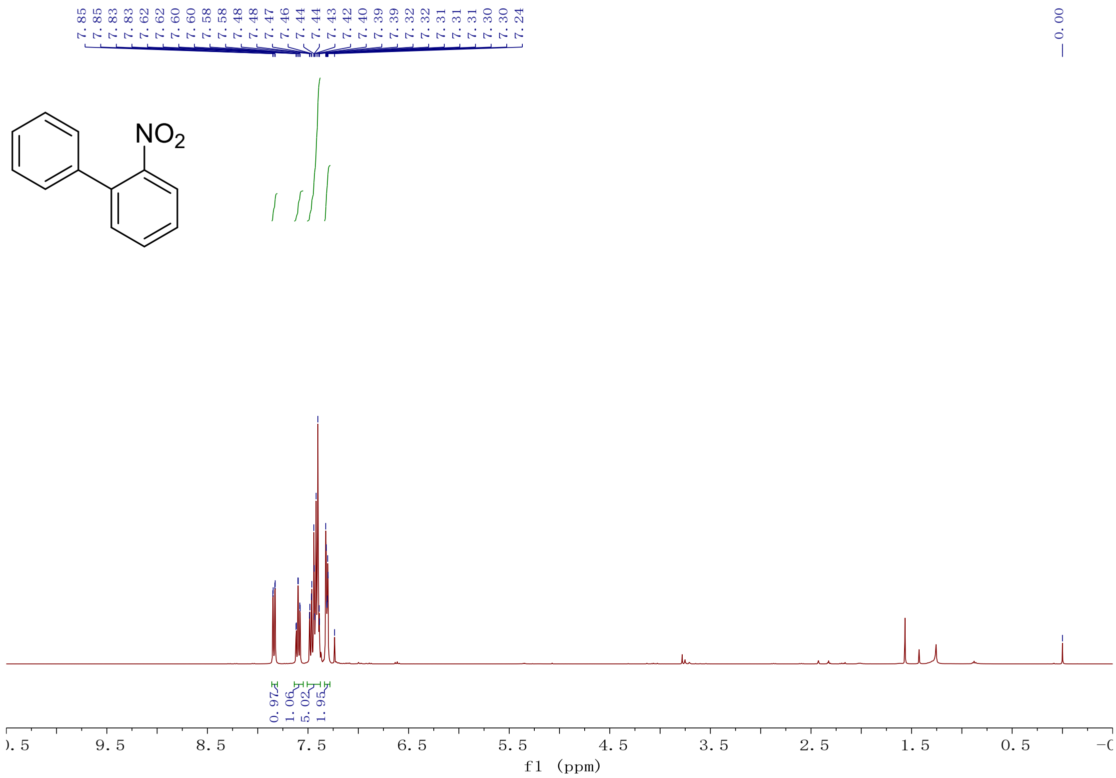


Figure S21. ^{1}H NMR spectrum of **3l** in $CDCl_3$

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

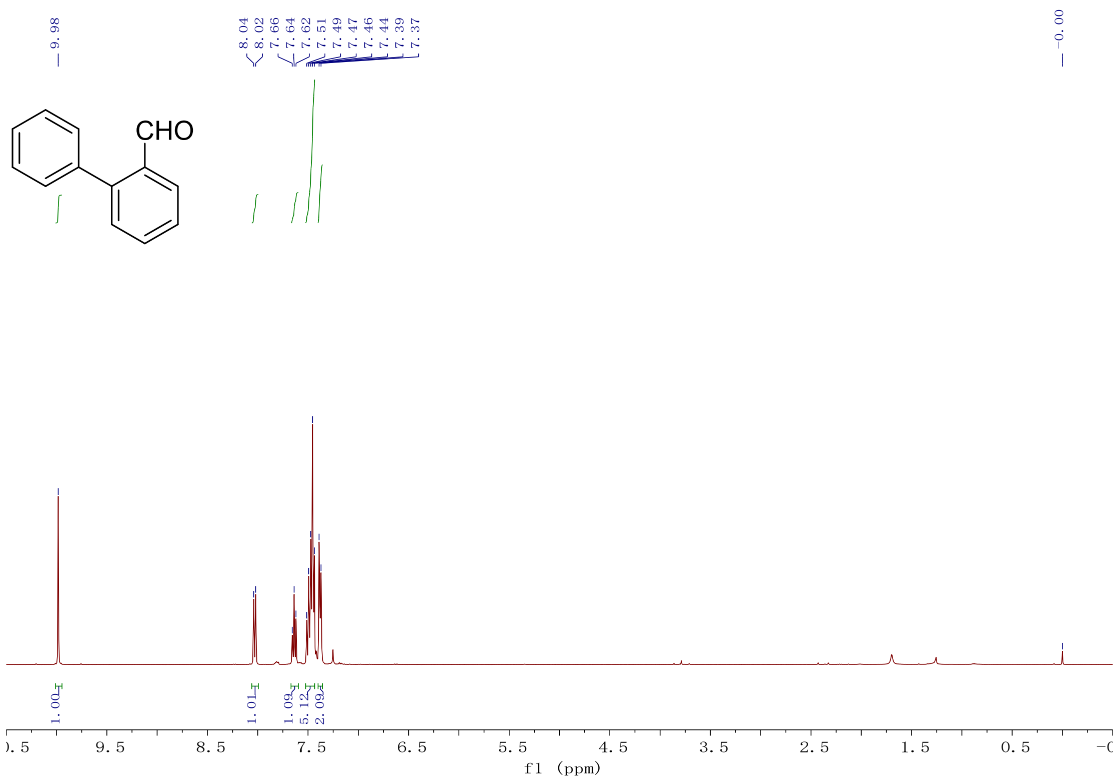


Figure S22. ^{1}H NMR spectrum of **3m** in $CDCl_3$

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

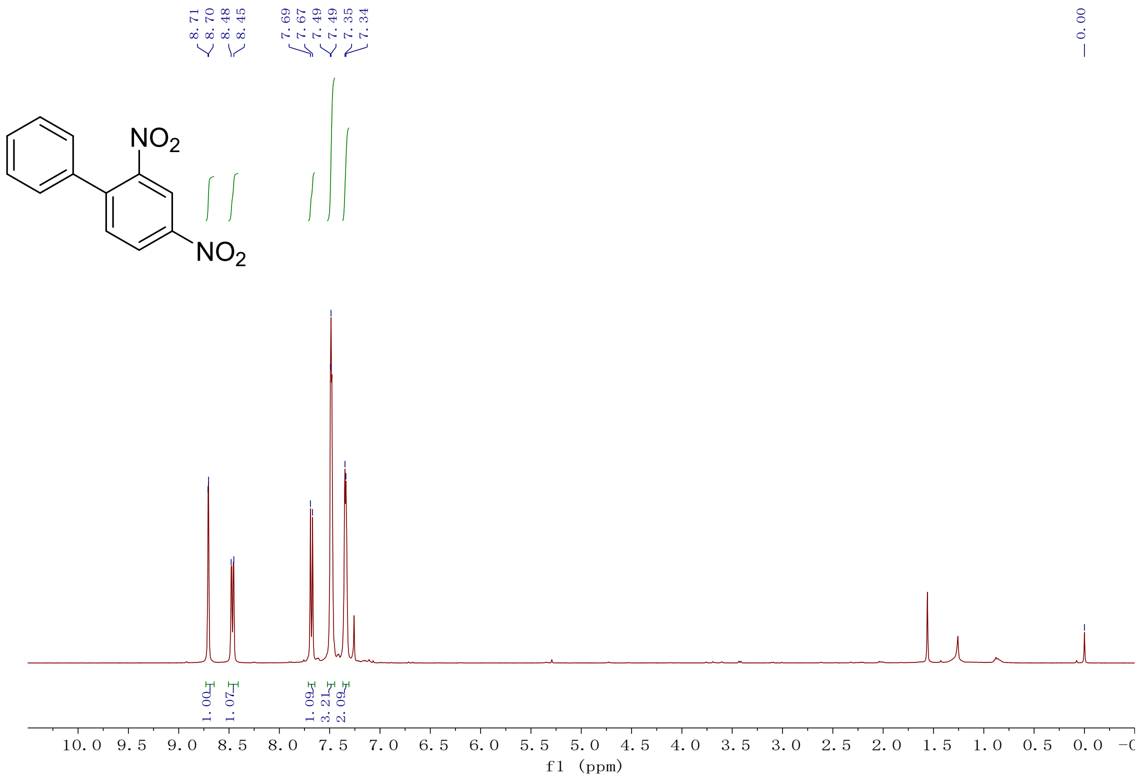


Figure S23. ^{1}H NMR spectrum of **3n** in $CDCl_3$

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

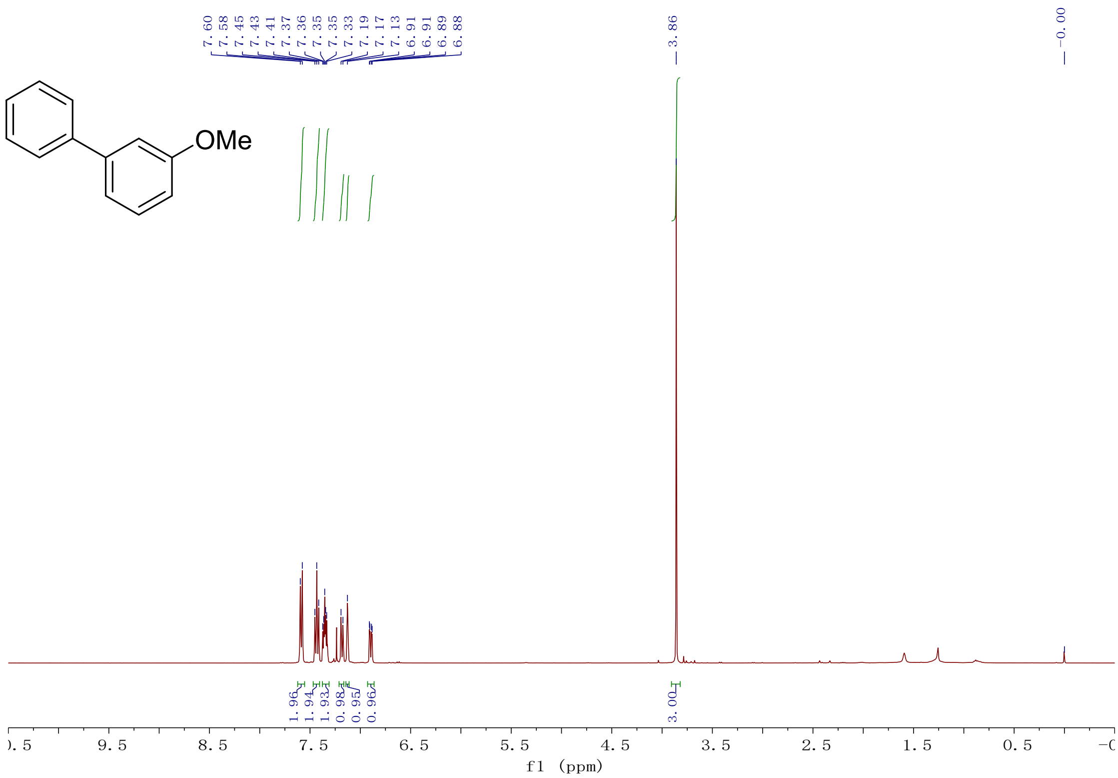


Figure S24. ^{1}H NMR spectrum of **3o** in $CDCl_3$

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

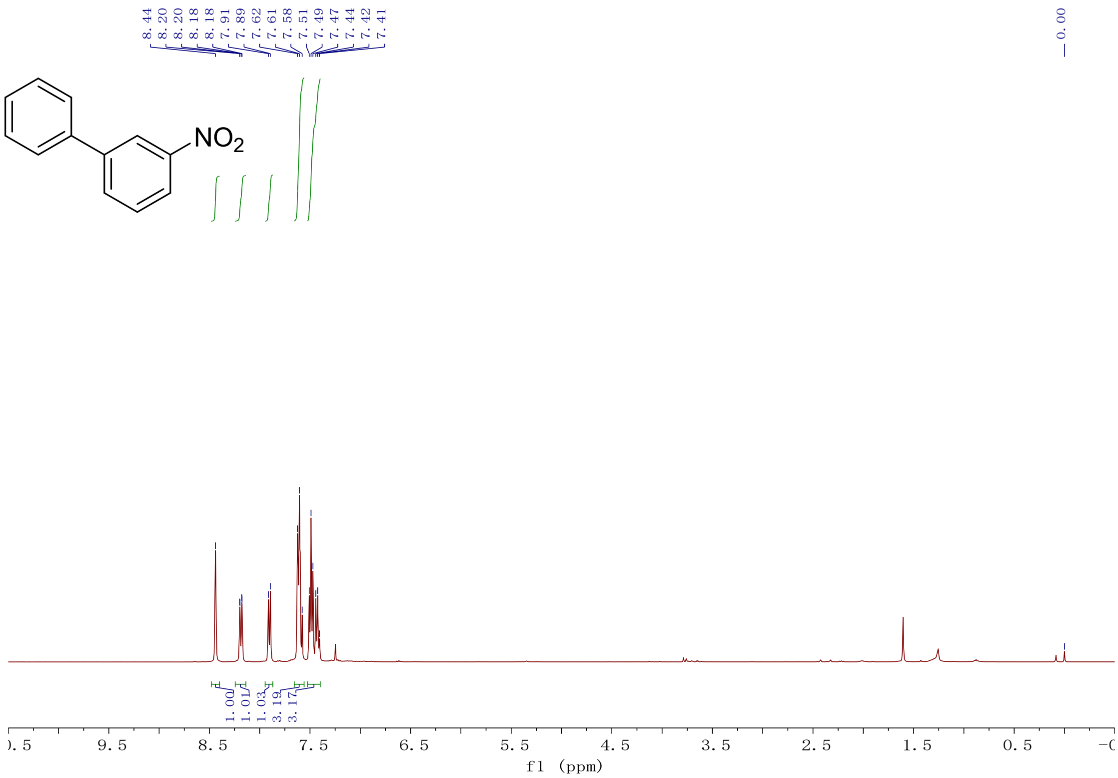


Figure S25. ^{1}H NMR spectrum of **3p** in $CDCl_3$

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

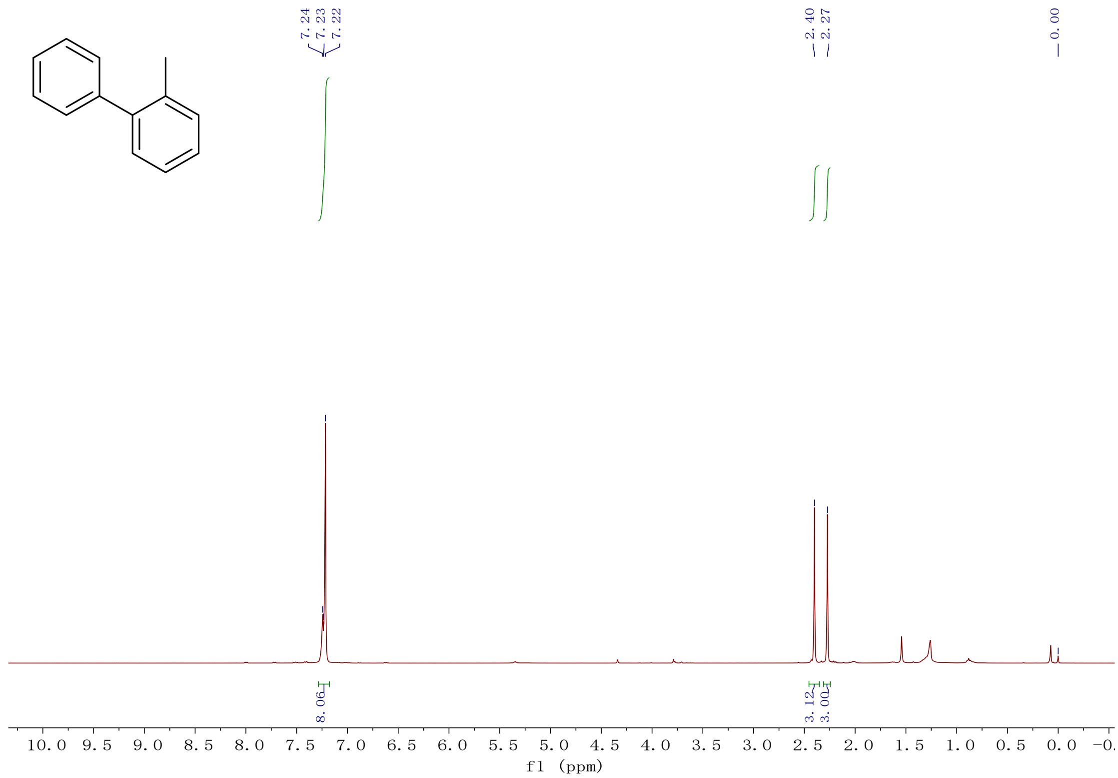


Figure S26. ^{1}H NMR spectrum of **3q** in $CDCl_3$

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

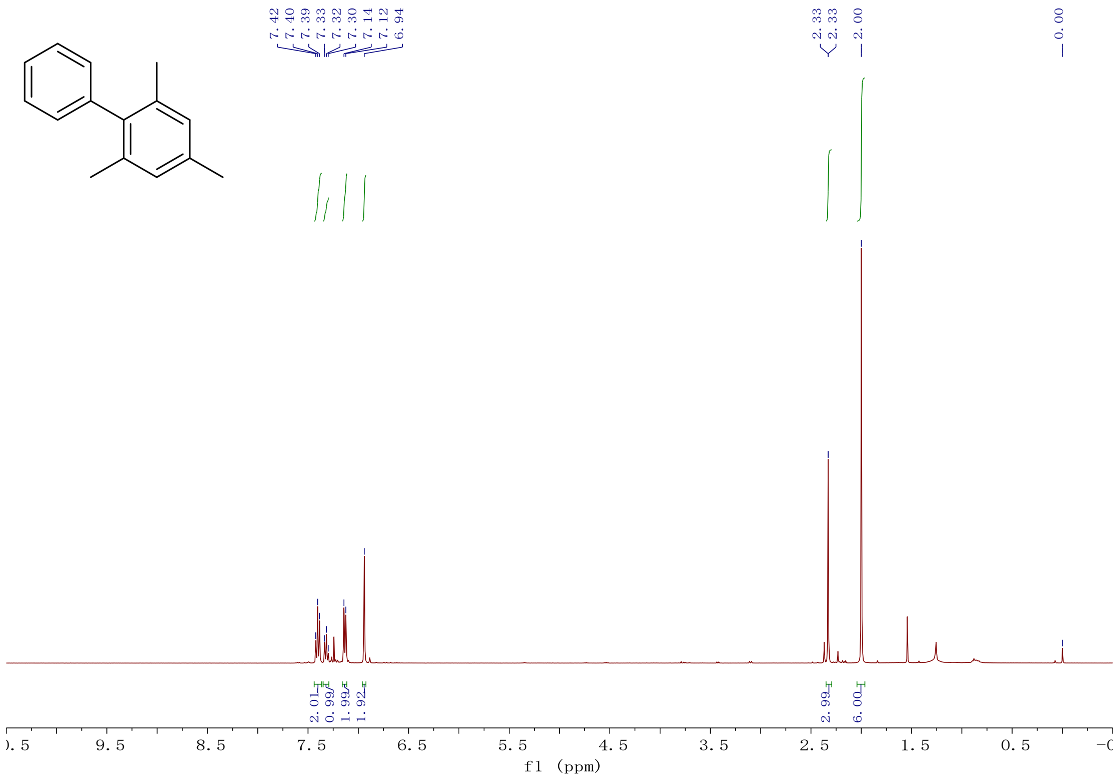


Figure S27. ^{1}H NMR spectrum of **3s** in $CDCl_3$

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

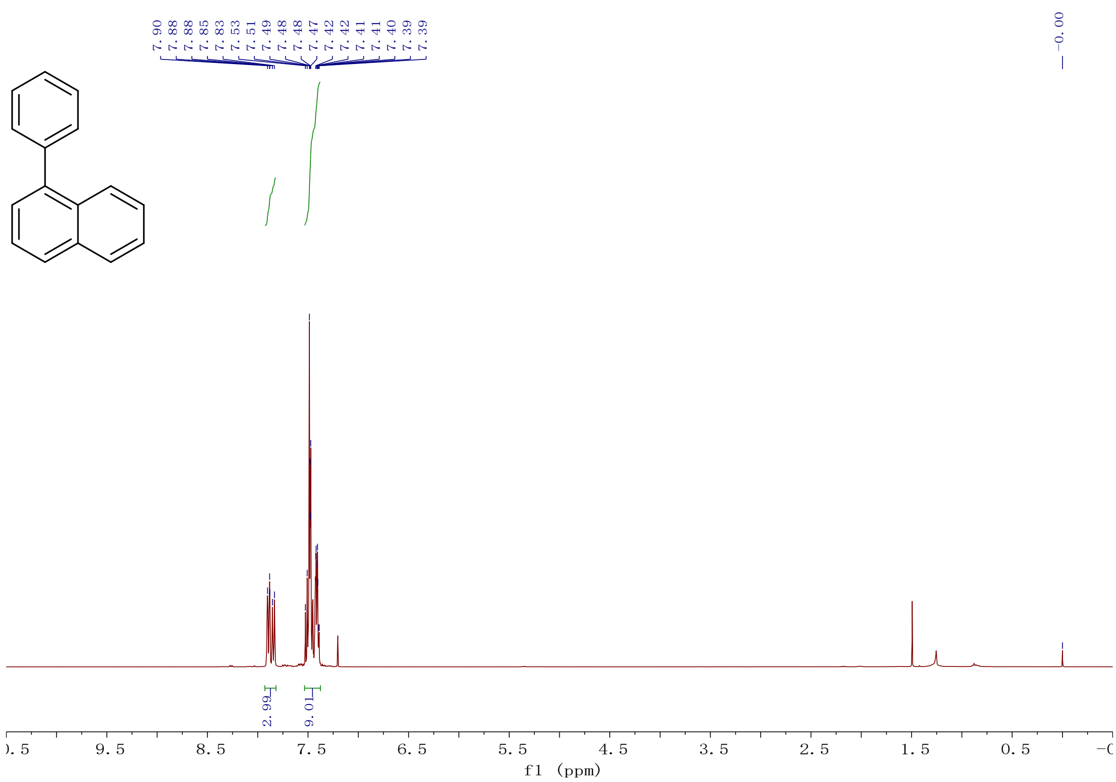


Figure S28. ^{1}H NMR spectrum of **3t** in $CDCl_3$

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

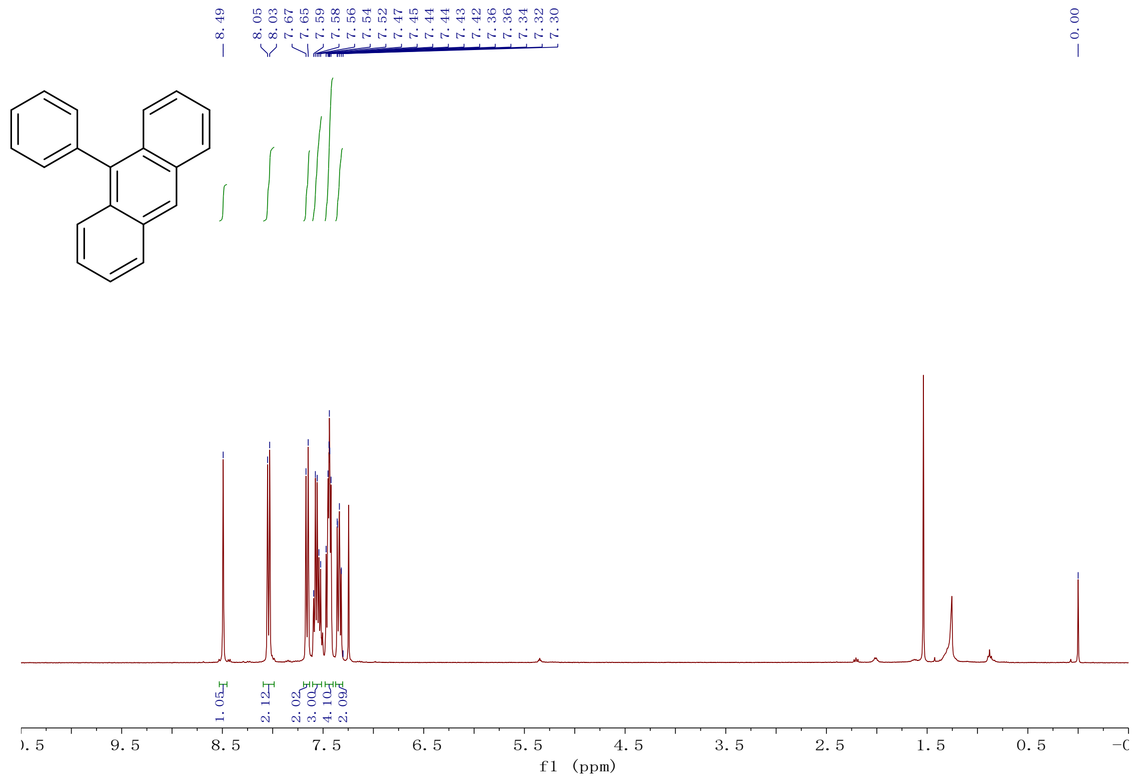


Figure S29. ^{1}H NMR spectrum of **3u** in $CDCl_3$

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

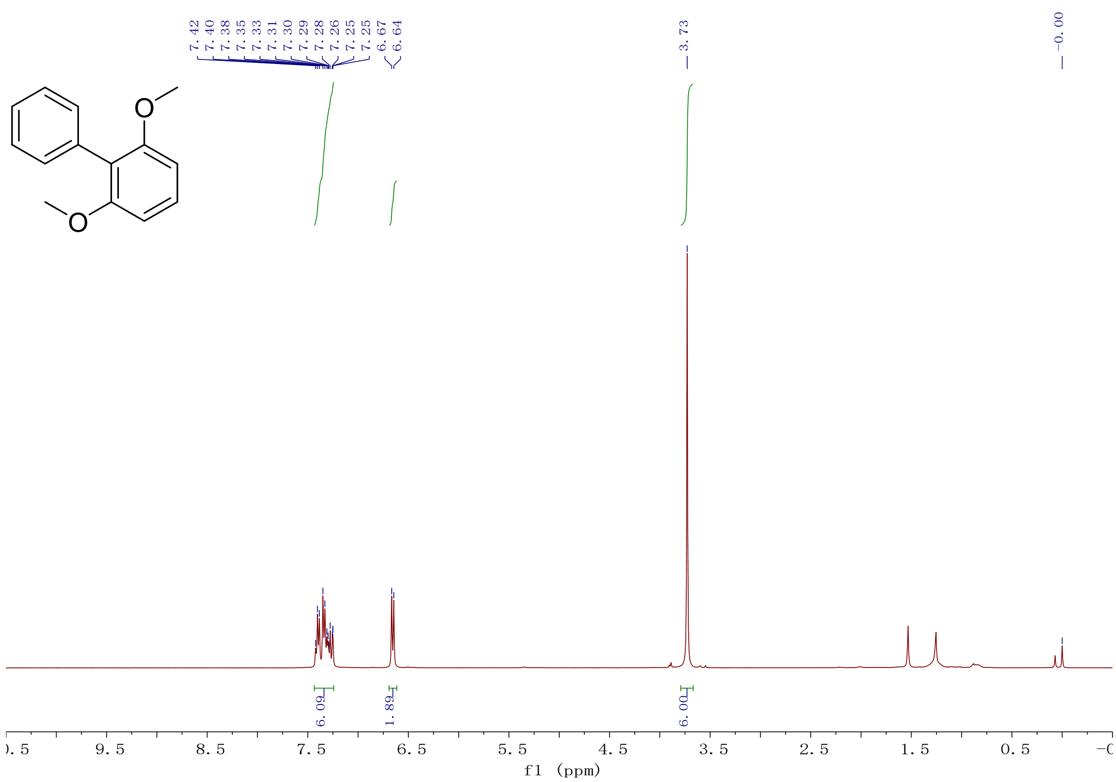


Figure S30. ^{1}H NMR spectrum of **3v** in $CDCl_3$

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

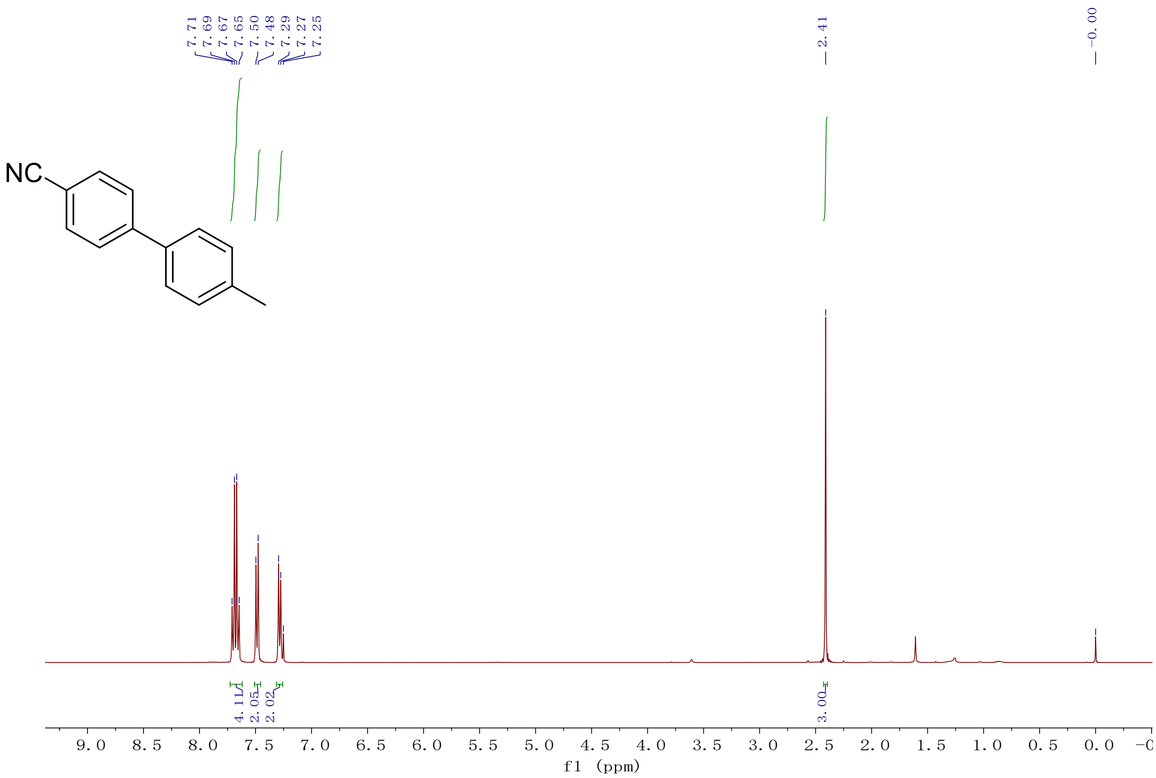


Figure S31. [1]H NMR spectrum of **3aa** in $CDCl_3$

[1]H NMR (400 MHz, $CDCl_3$)

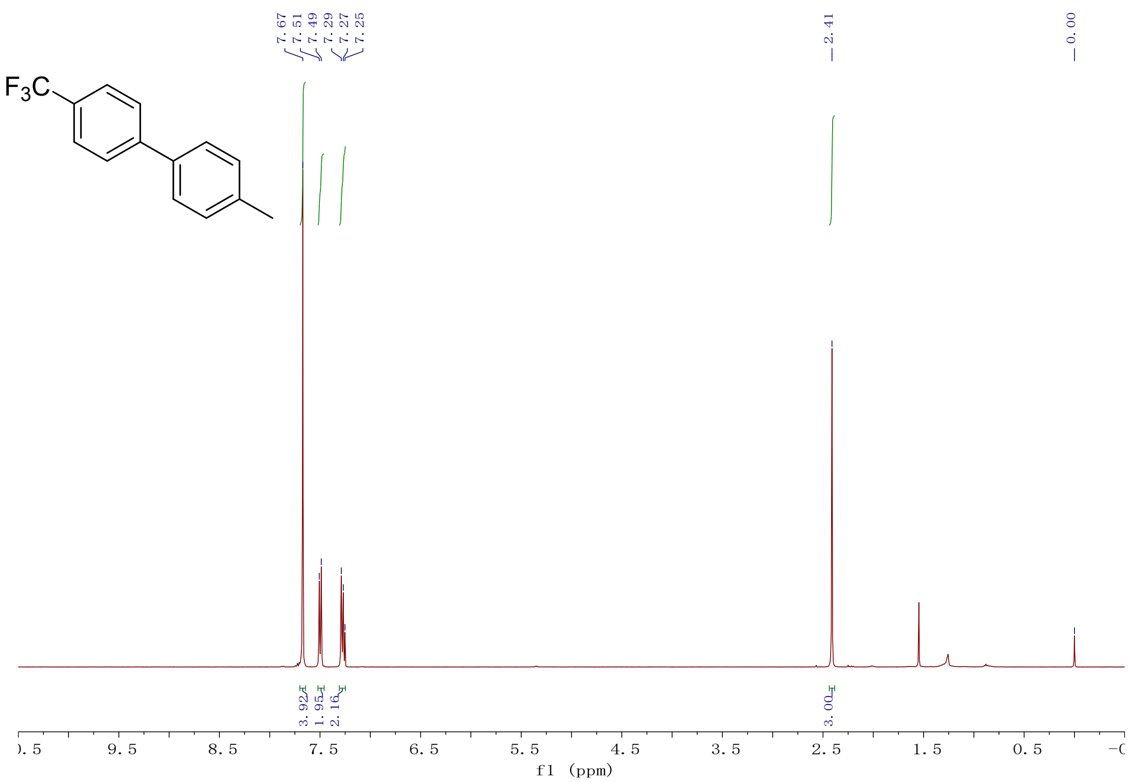


Figure S32 [1]H NMR spectrum of **3ab** in $CDCl_3$

[1]H NMR (400 MHz, $CDCl_3$)

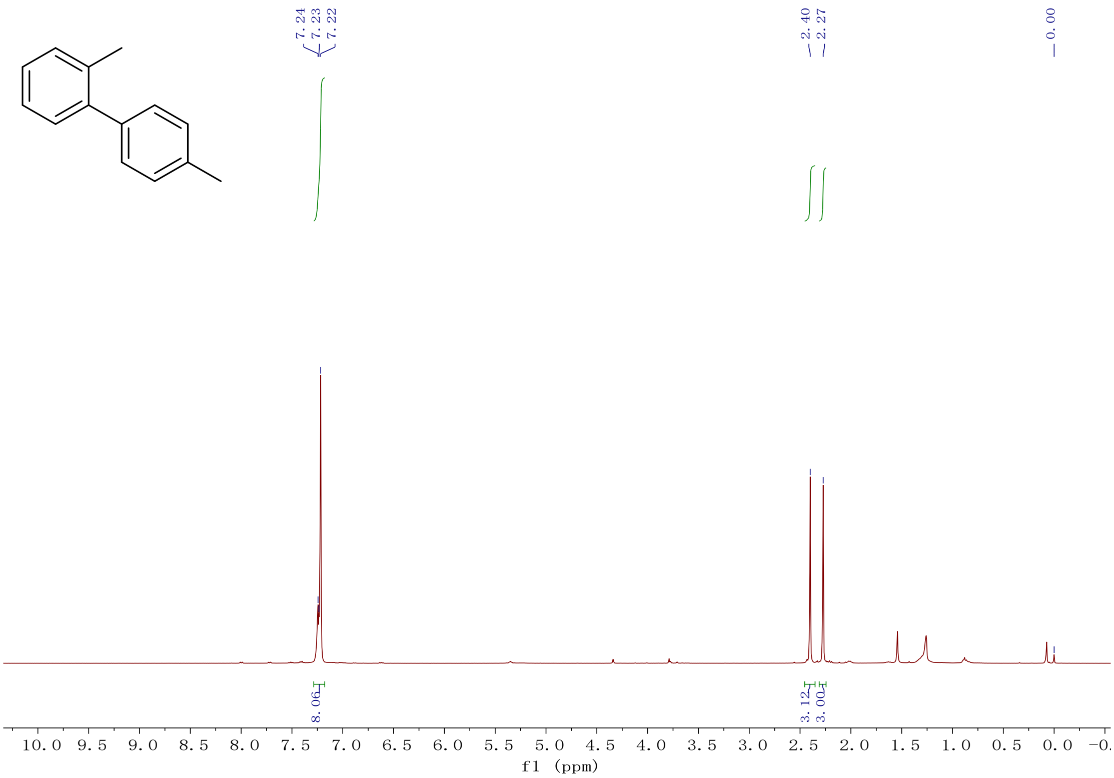


Figure S33. ^{1}H NMR spectrum of **3ac** in $CDCl_3$

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

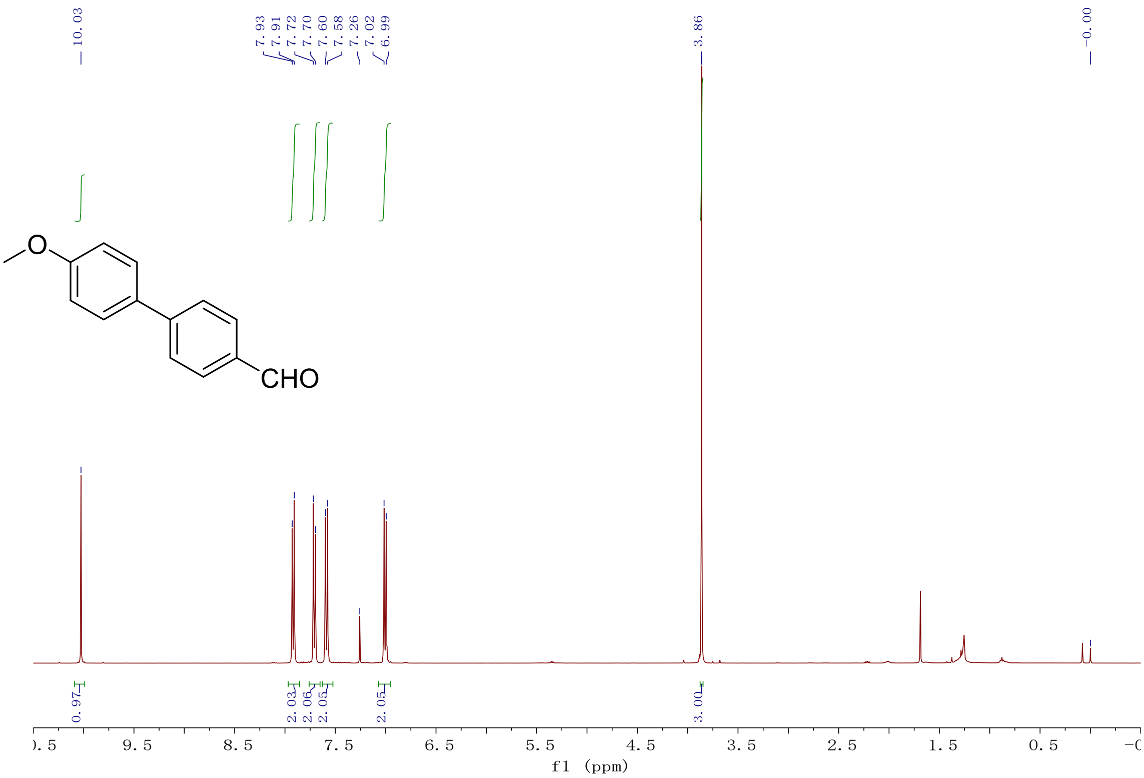


Figure S34. ^{1}H NMR spectrum of **3ae** in $CDCl_3$

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

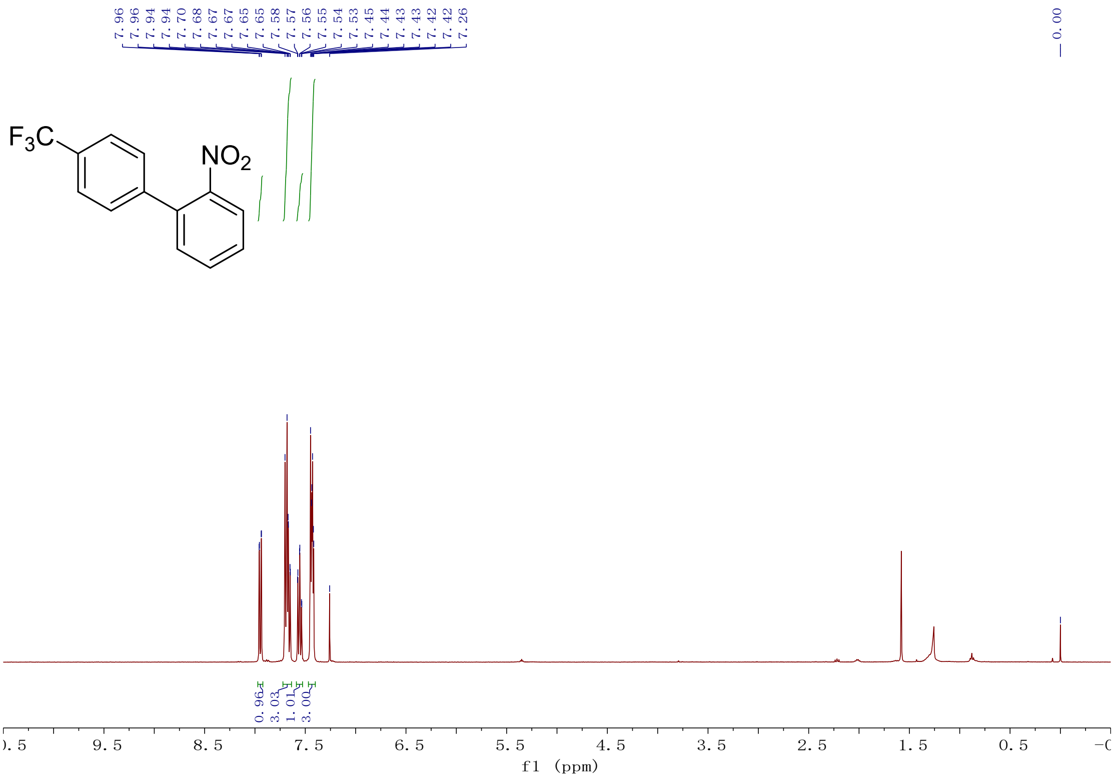


Figure S35. ^{1}H NMR spectrum of **3af** in $CDCl_3$

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

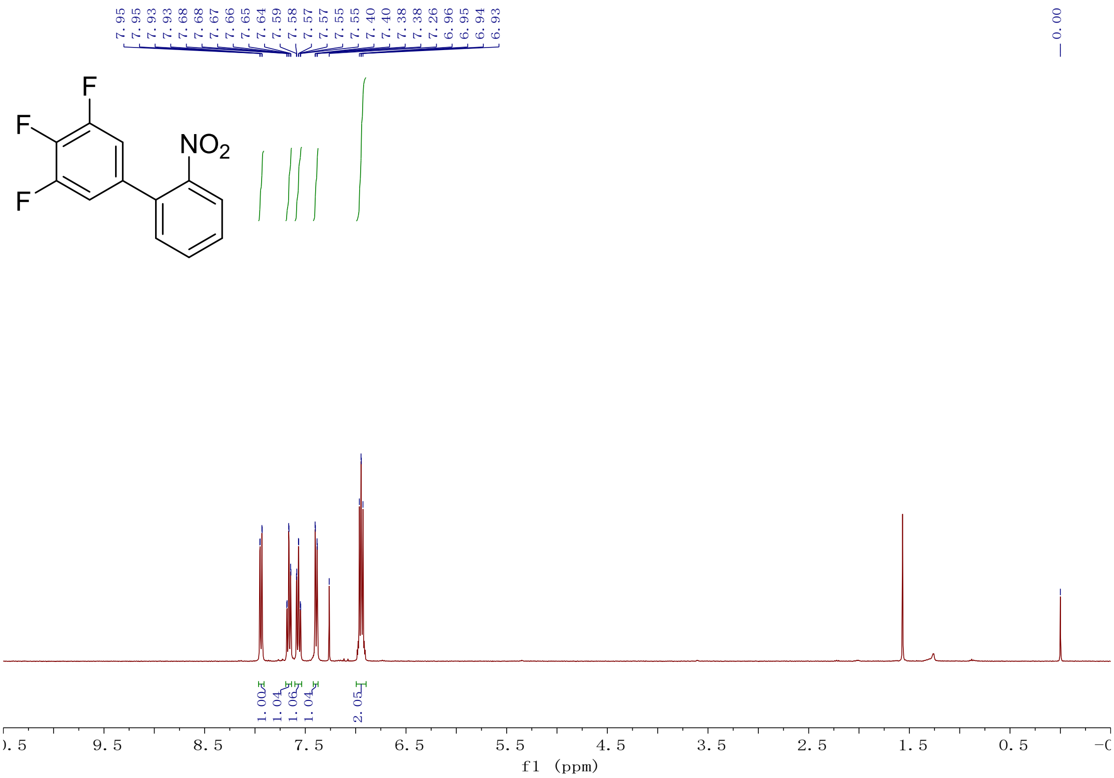


Figure S36. ^{1}H NMR spectrum of **3ag** in $CDCl_3$

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

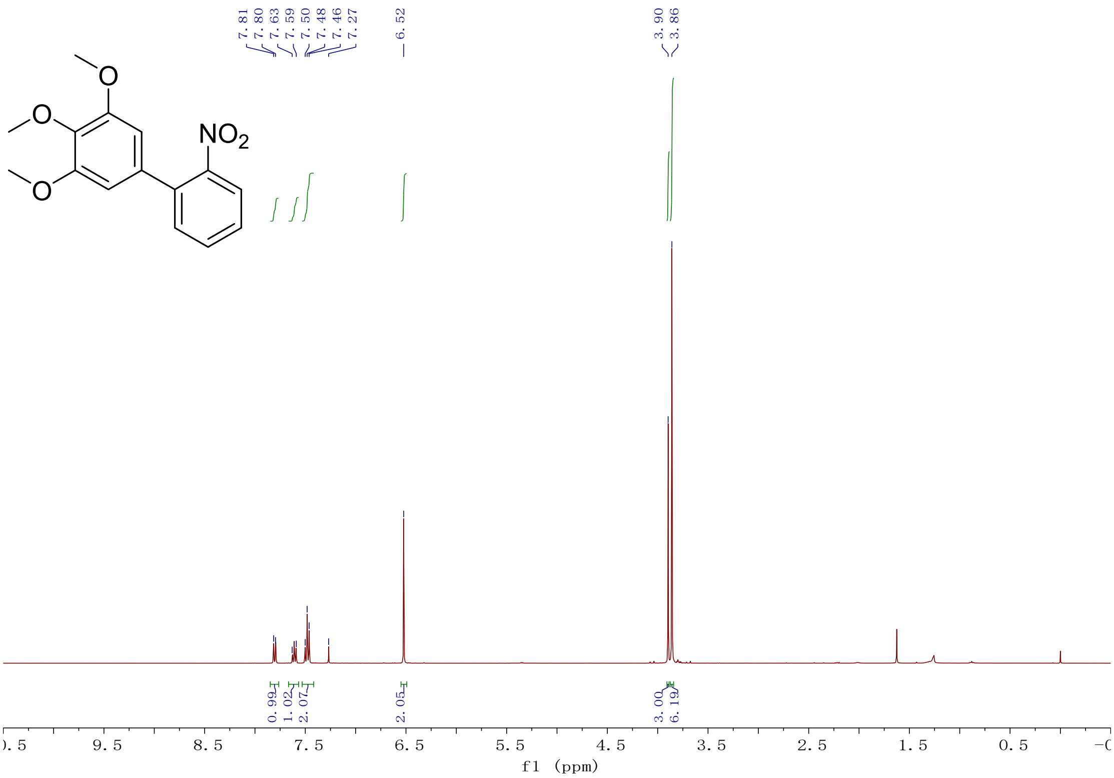


Figure S37. ^{1}H NMR spectrum of **3ah** in $CDCl_3$

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

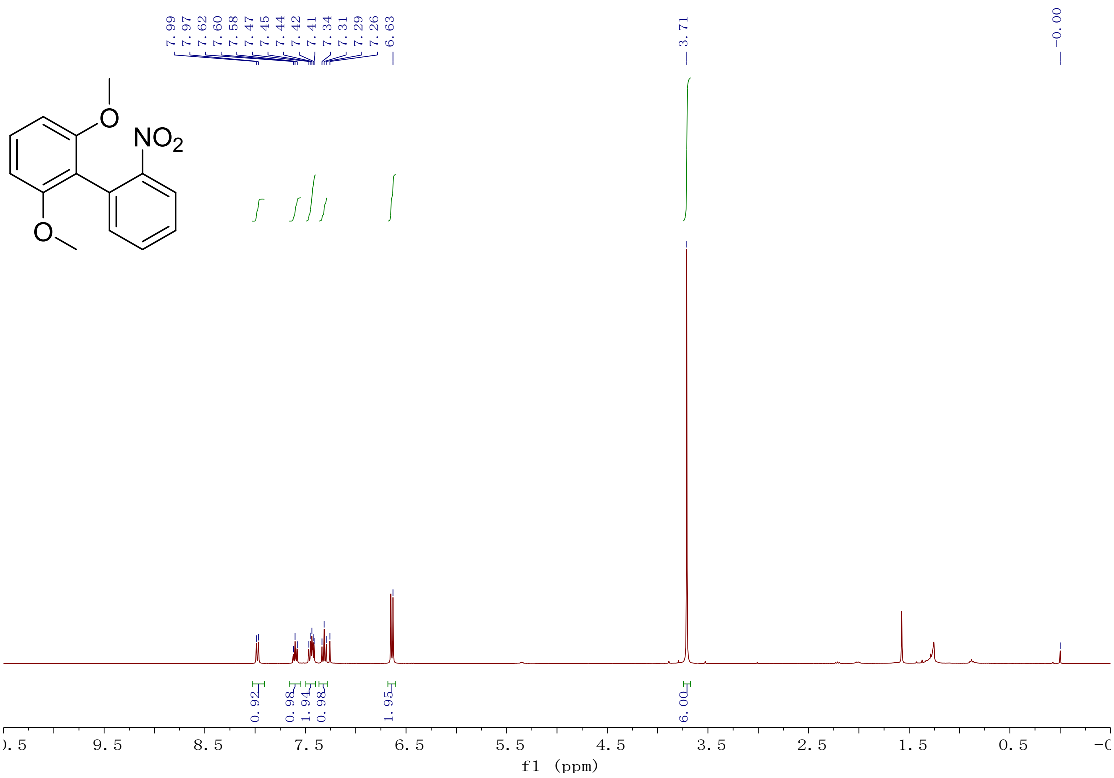


Figure S38. ^{1}H NMR spectrum of **3ai** in $CDCl_3$

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

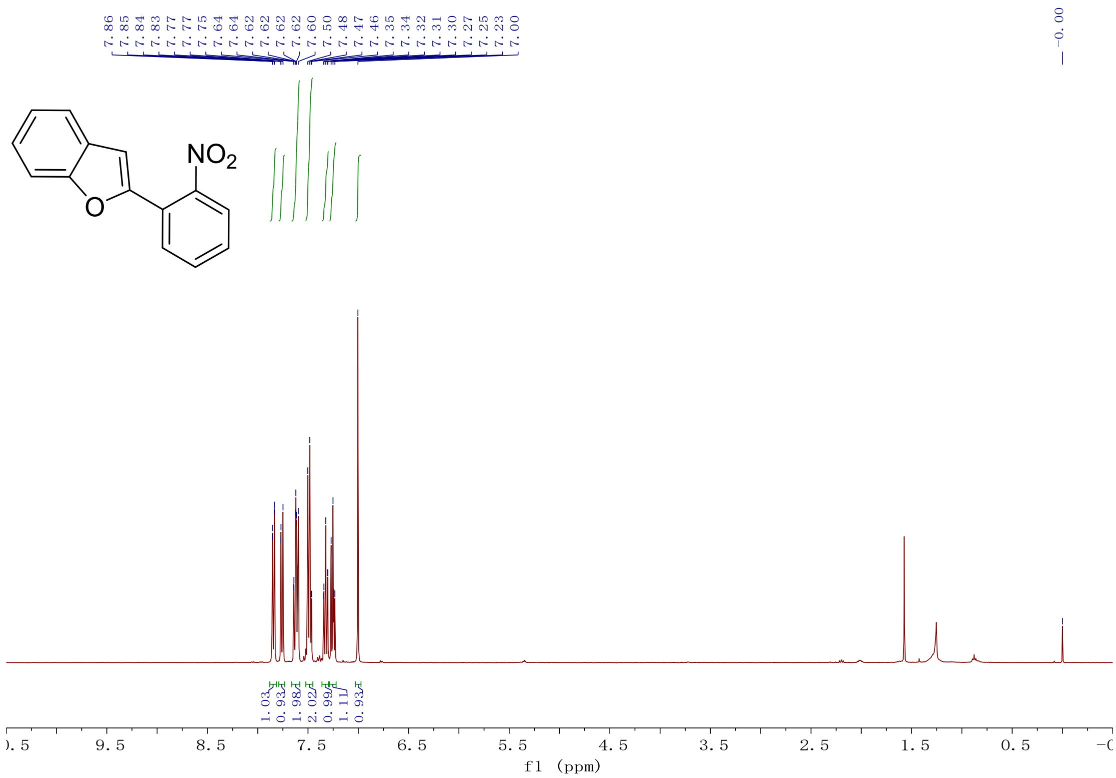


Figure S39. ^{1}H NMR spectrum of **3a′** in $CDCl_3$

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

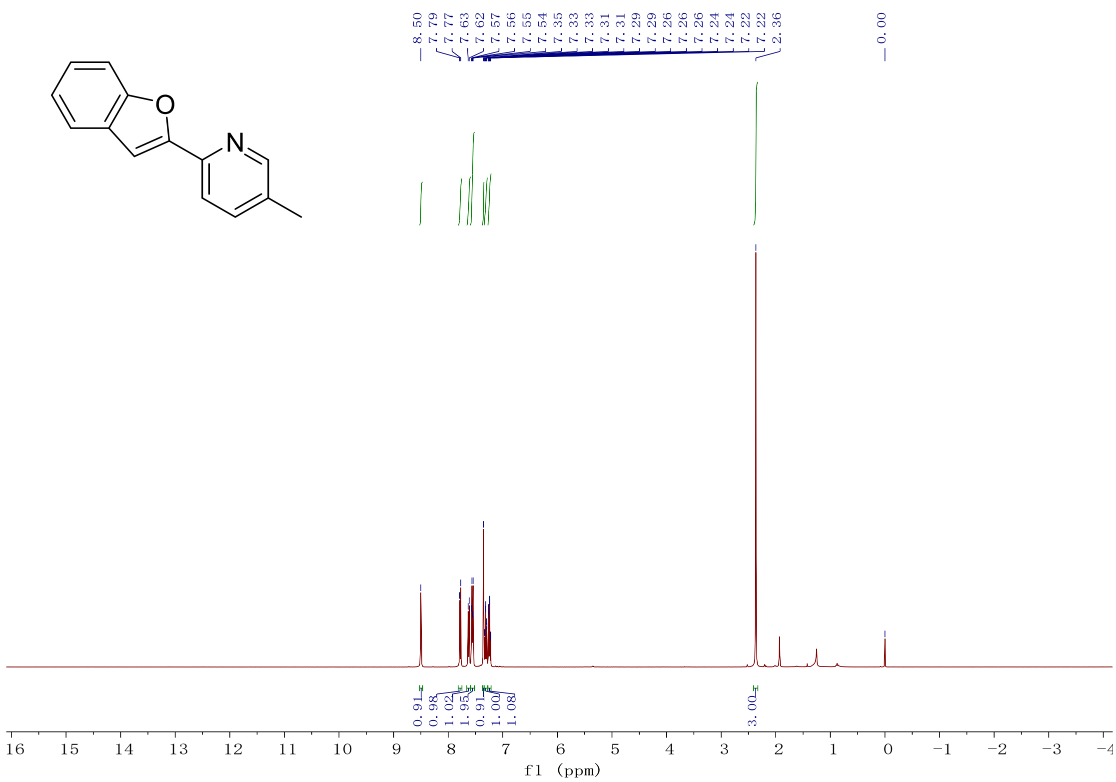


Figure S40. ^{1}H NMR spectrum of **3b′** in $CDCl_3$

^{13}C NMR (101 MHz, $CDCl_3$)

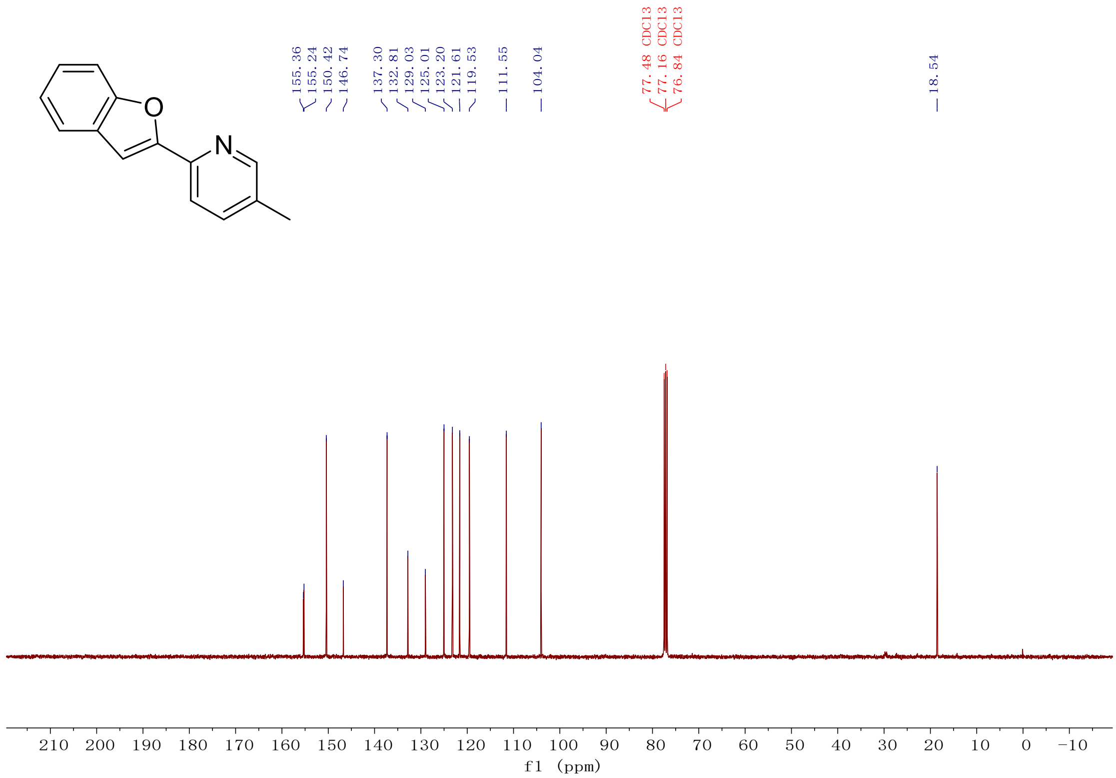


Figure S41. ^{13}C NMR spectrum of **3b′** in $CDCl_3$

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

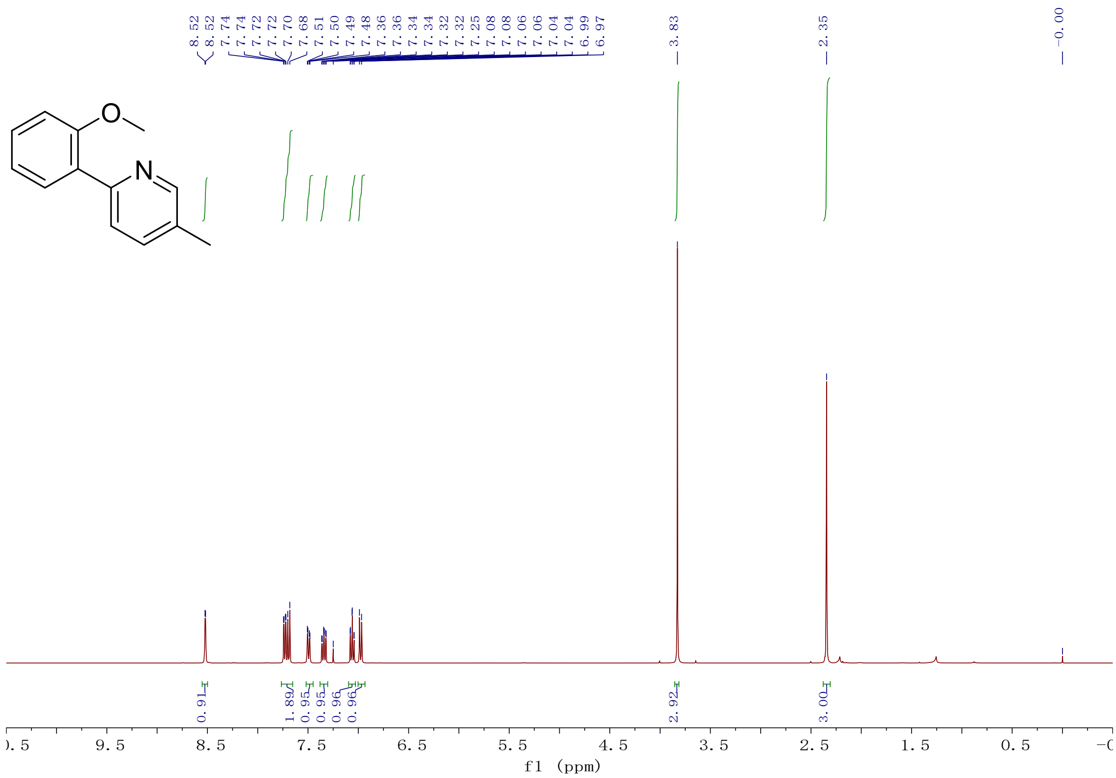


Figure S42. ^{1}H NMR spectrum of **3c′** in $CDCl_3$

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

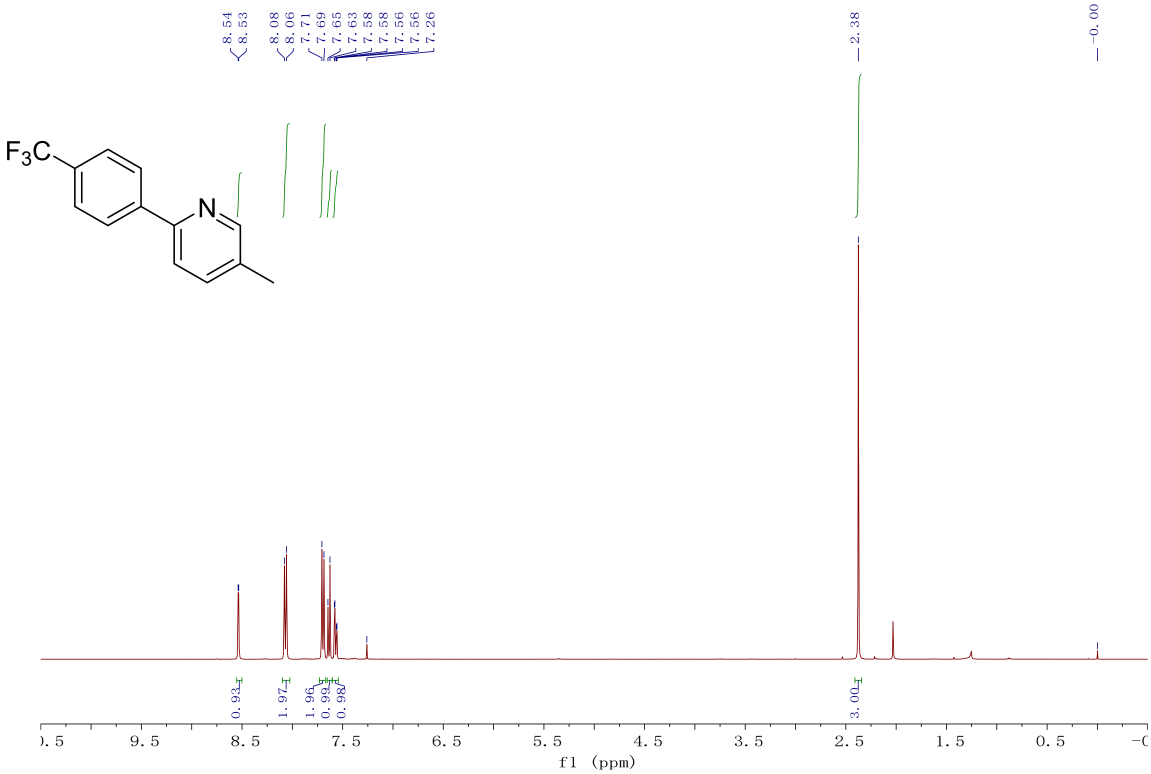


Figure S43. ^{1}H NMR spectrum of **3d′** in $CDCl_3$

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

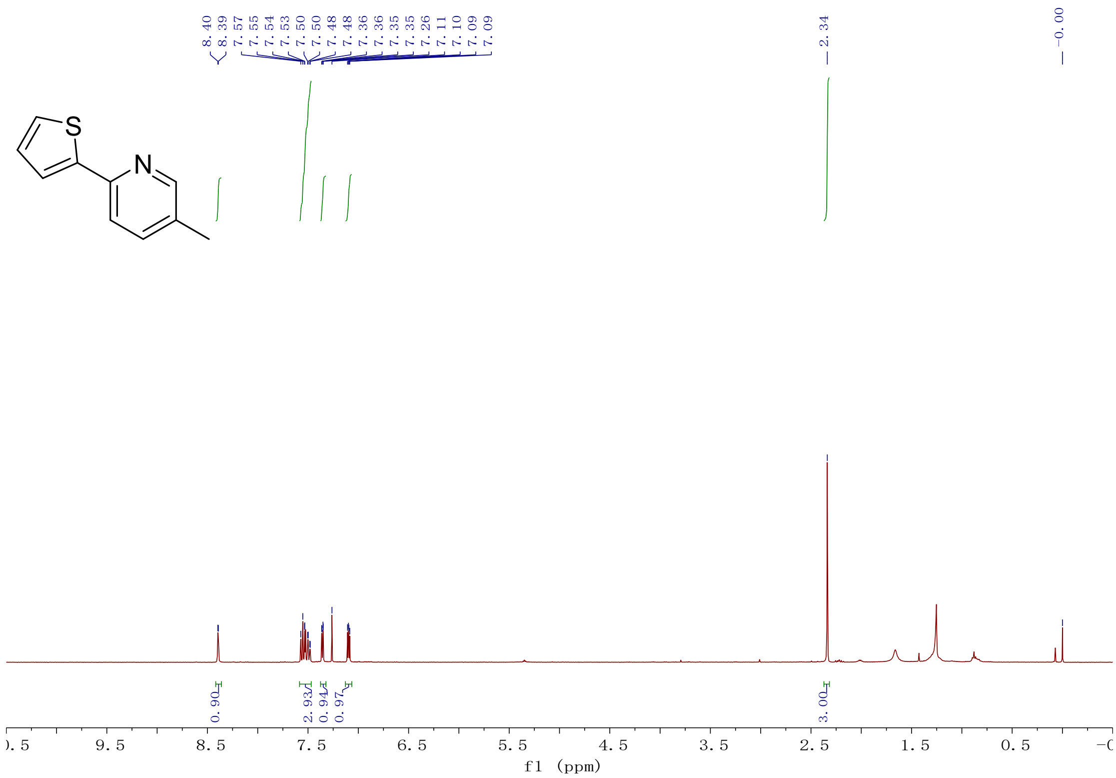


Figure S44. ^{1}H NMR spectrum of **3e′** in $CDCl_3$

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

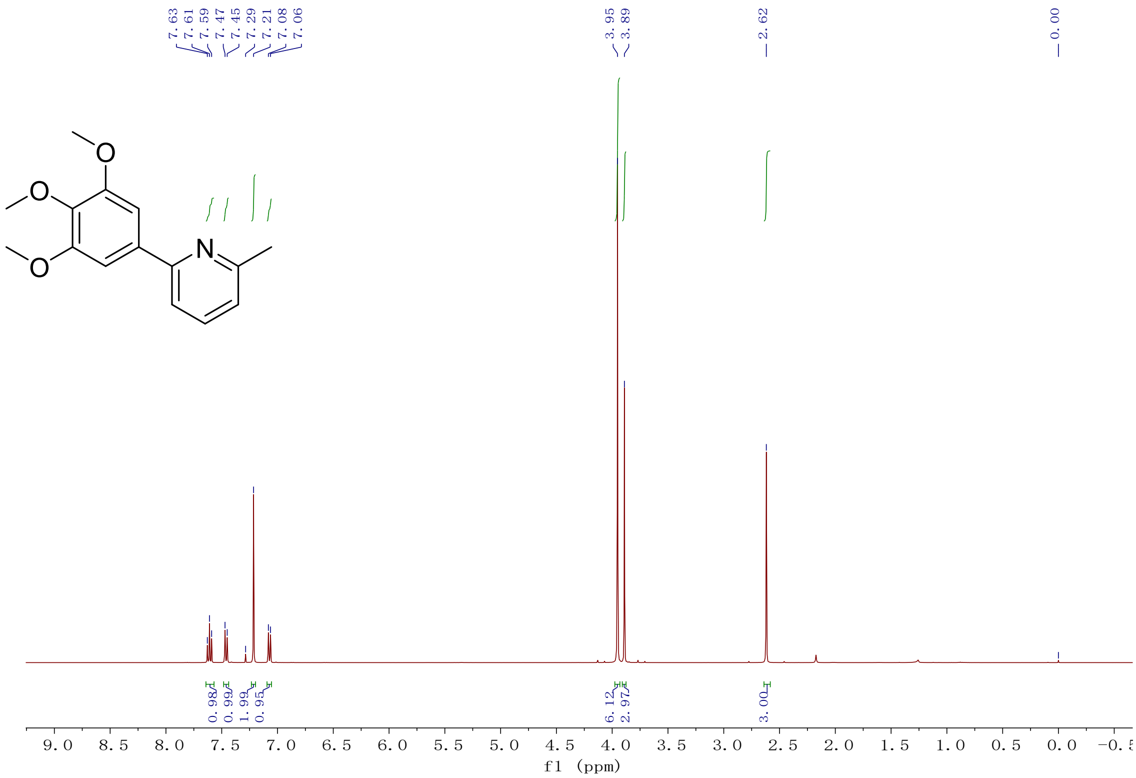


Figure S45. ^{1}H NMR spectrum of **3f′** in $CDCl_3$

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

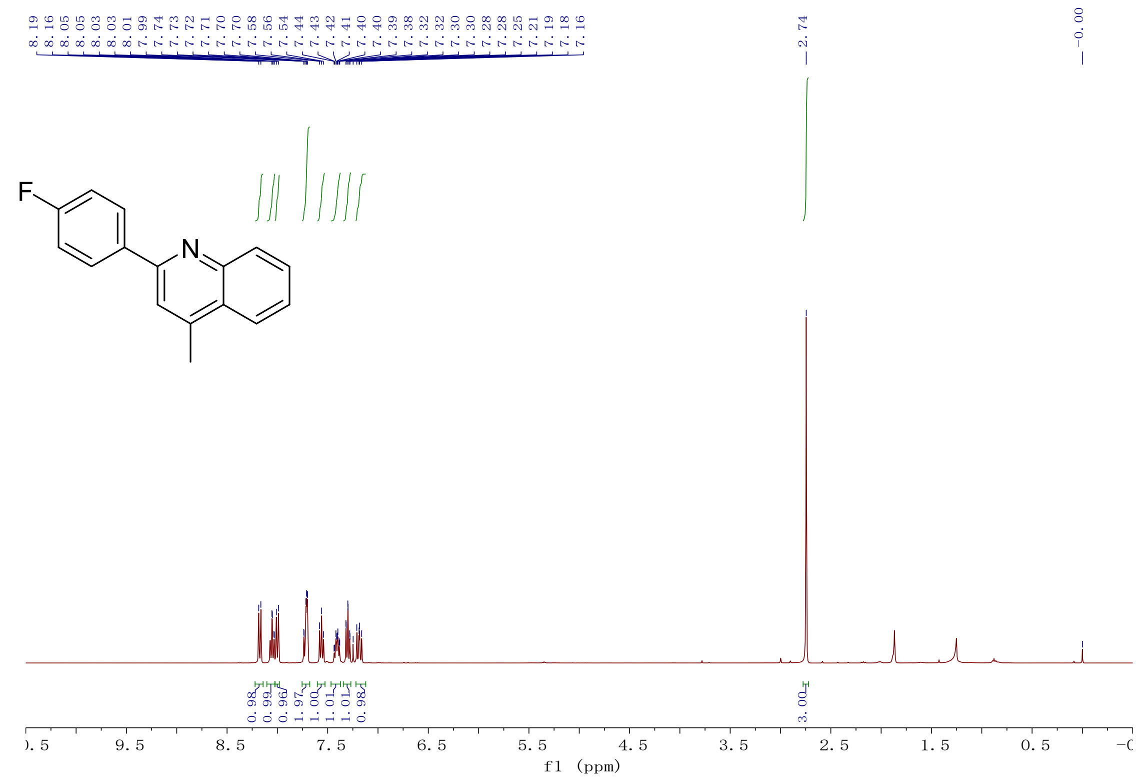


Figure S46. ^{1}H NMR spectrum of **3g′** in $CDCl_3$

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

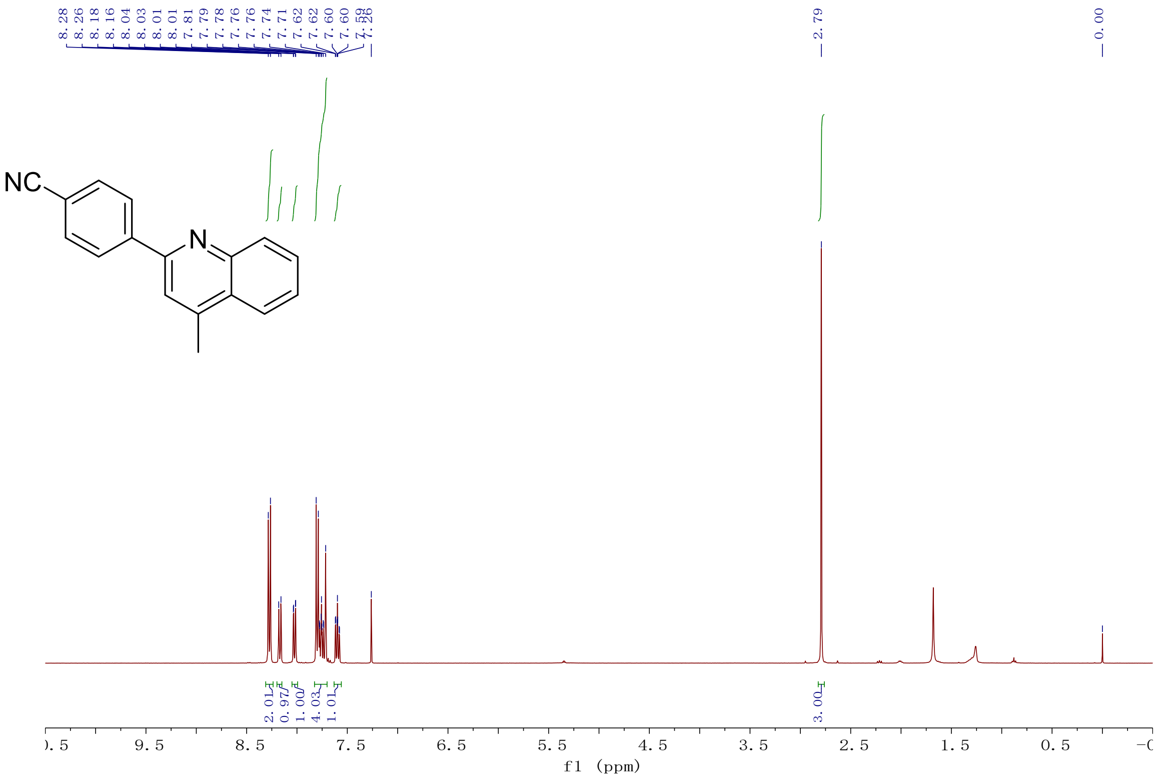


Figure S47. [1]H NMR spectrum of **3h′** in $CDCl_3$

[1]H NMR (400 MHz, $CDCl_3$)

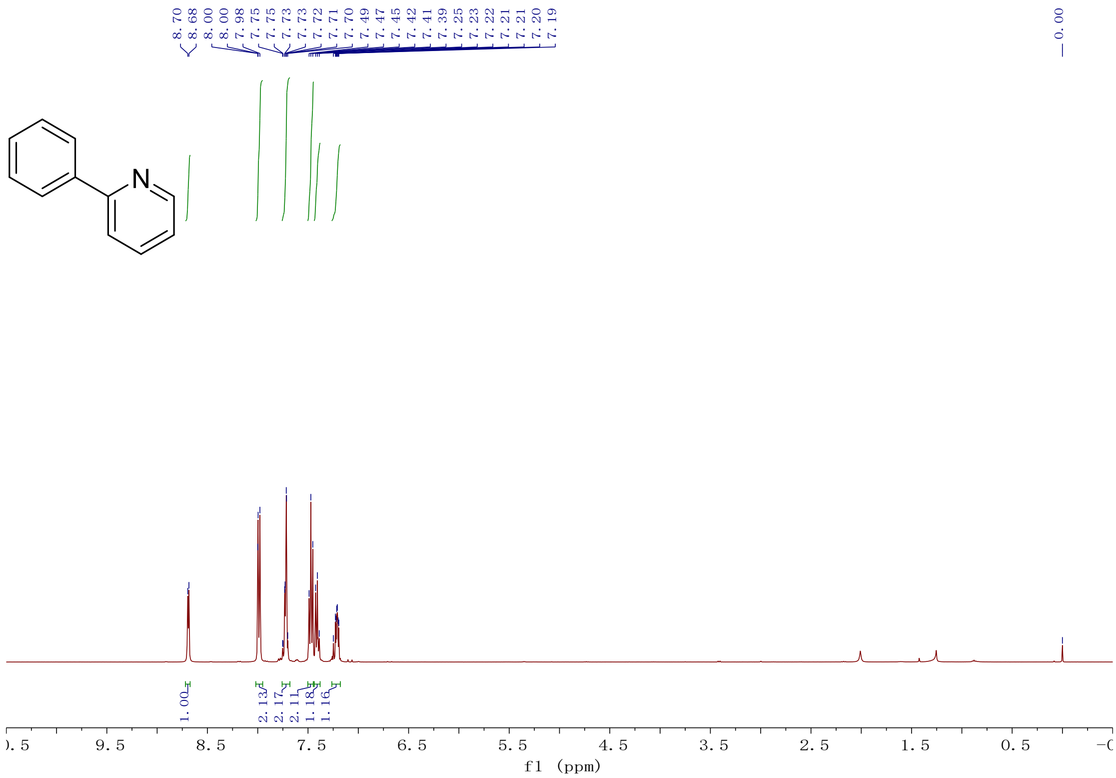


Figure S48. [1]H NMR spectrum of **3i′** in $CDCl_3$

[1]H NMR (400 MHz, $CDCl_3$)

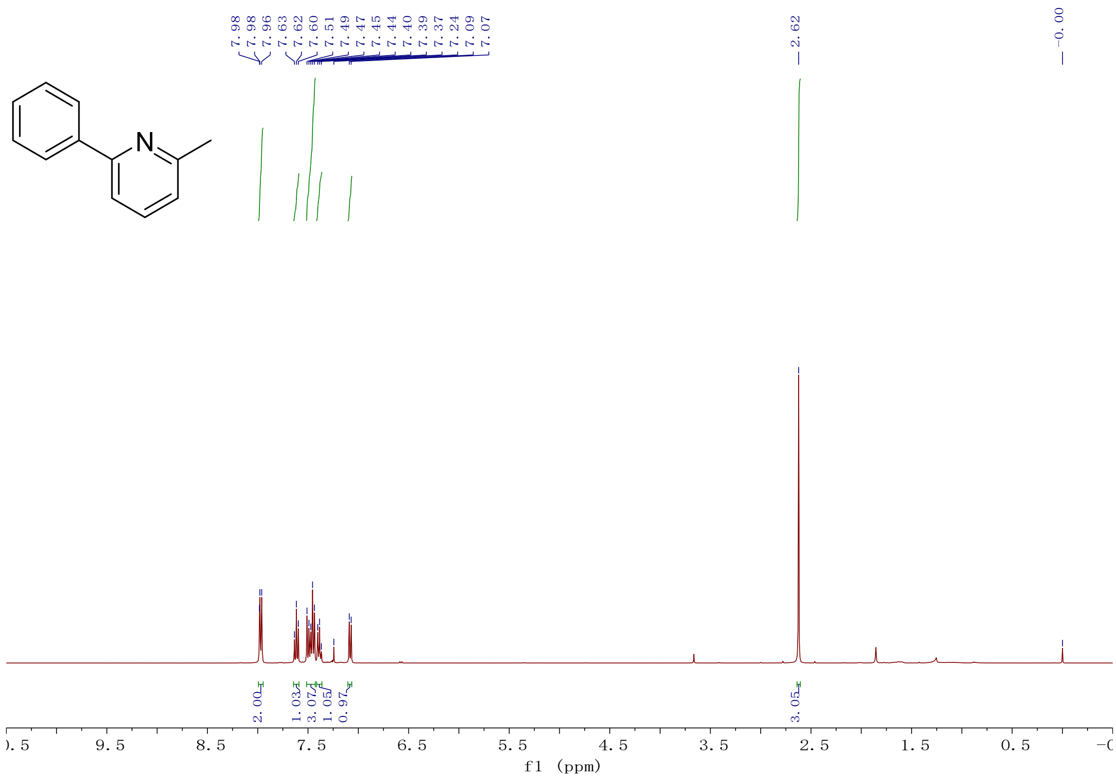


Figure S49. ^{1}H NMR spectrum of **3j′** in $CDCl_3$

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

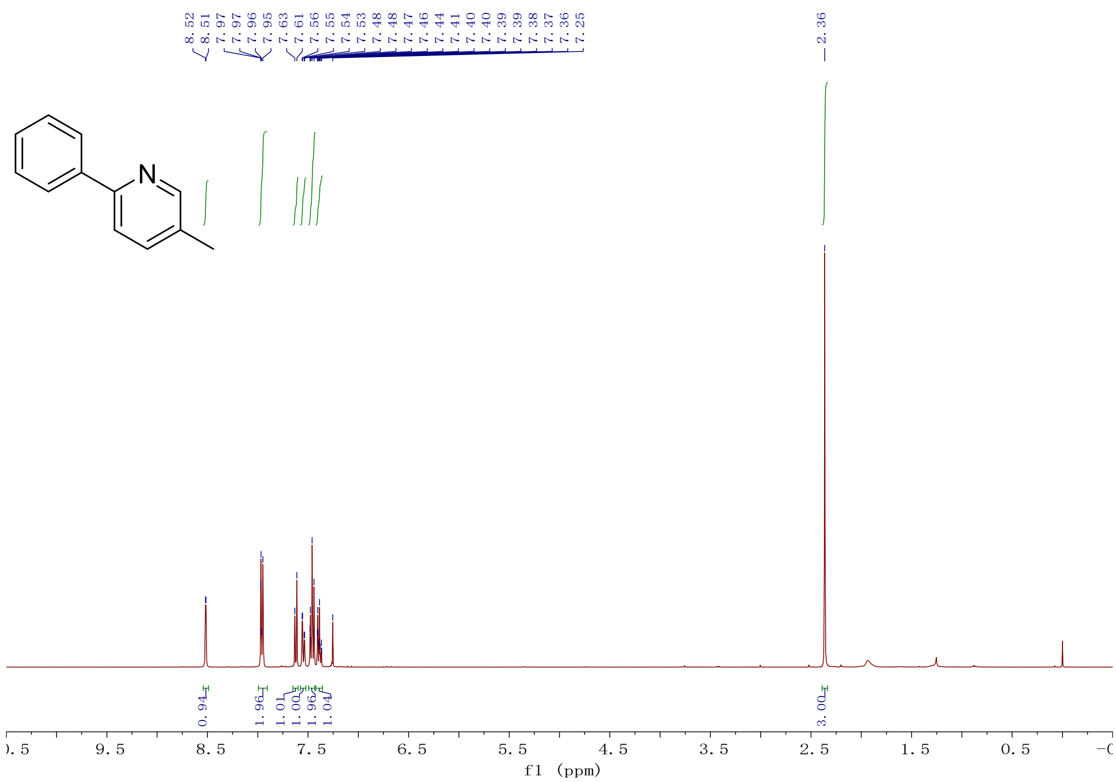


Figure S50. ^{1}H NMR spectrum of **3k′** in $CDCl_3$

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

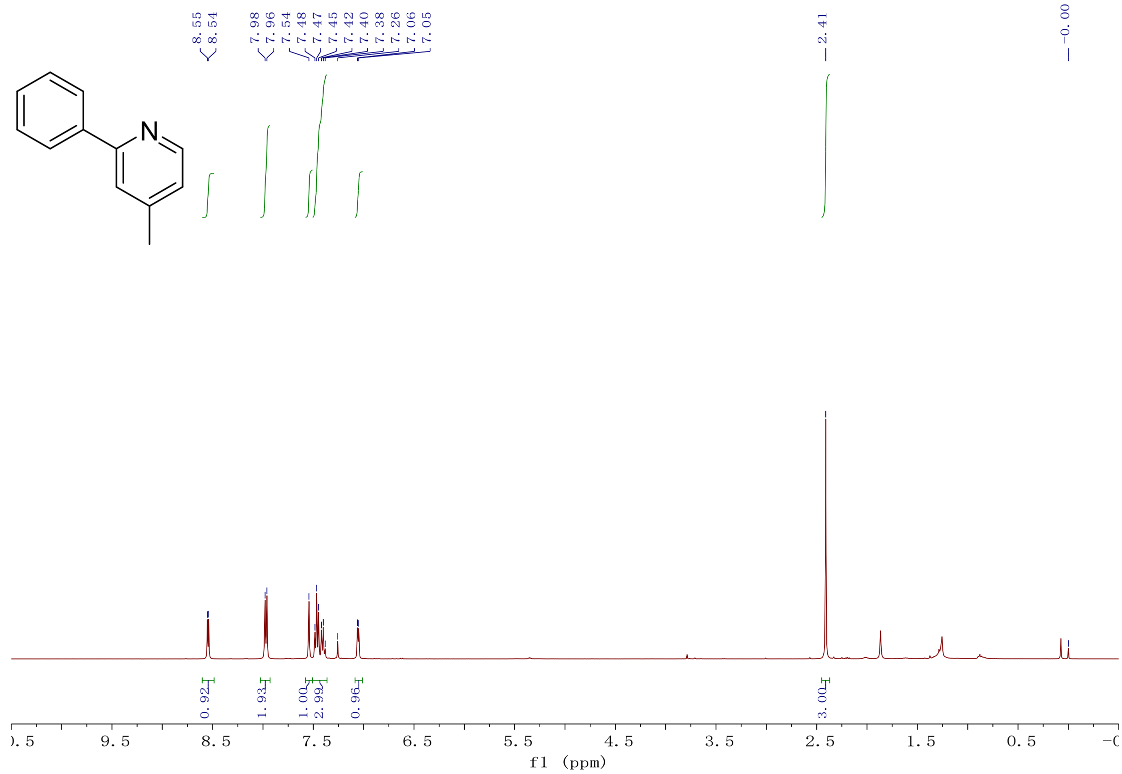


Figure S51. ^{1}H NMR spectrum of **3l′** in $CDCl_3$

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

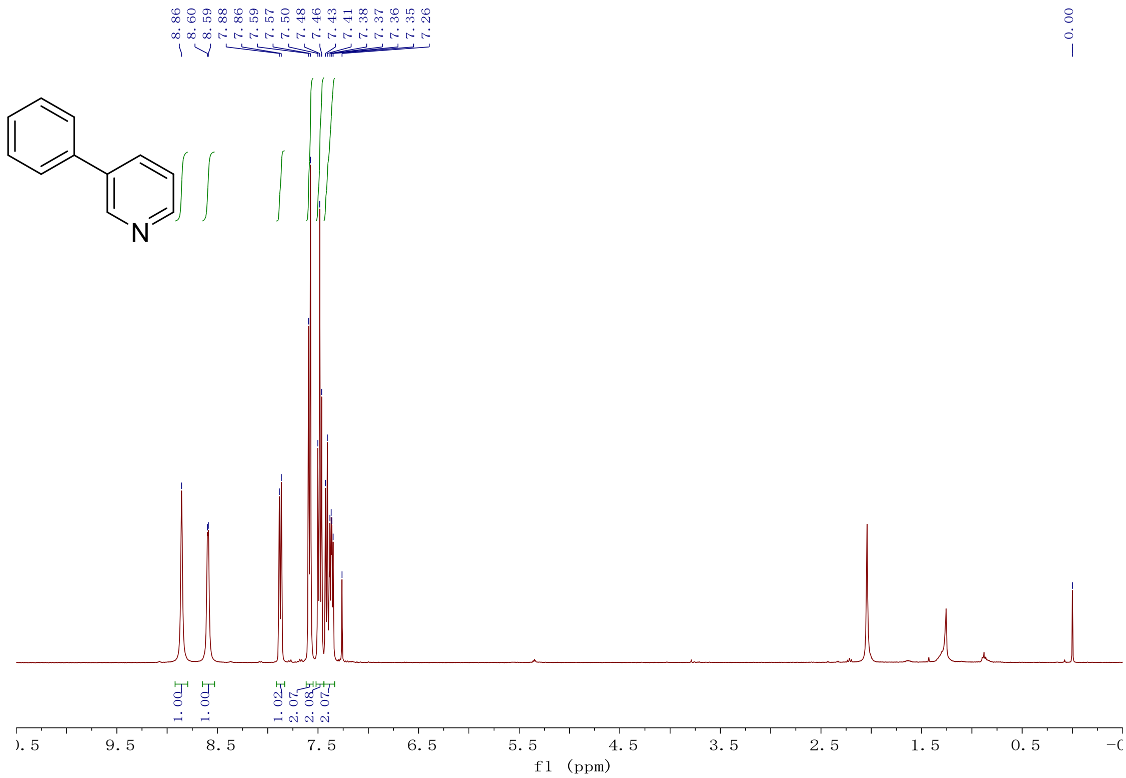


Figure S52. ^{1}H NMR spectrum of **3m′** in $CDCl_3$

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

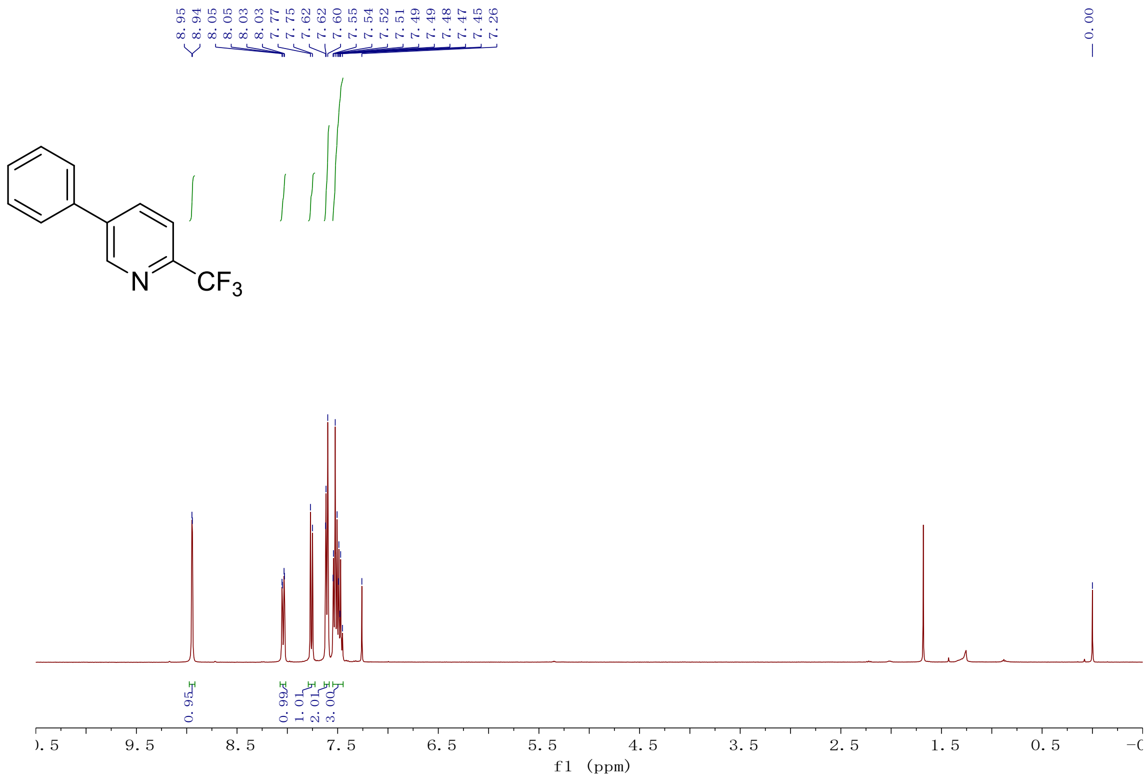


Figure S53. ^{1}H NMR spectrum of **3n′** in $CDCl_3$

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

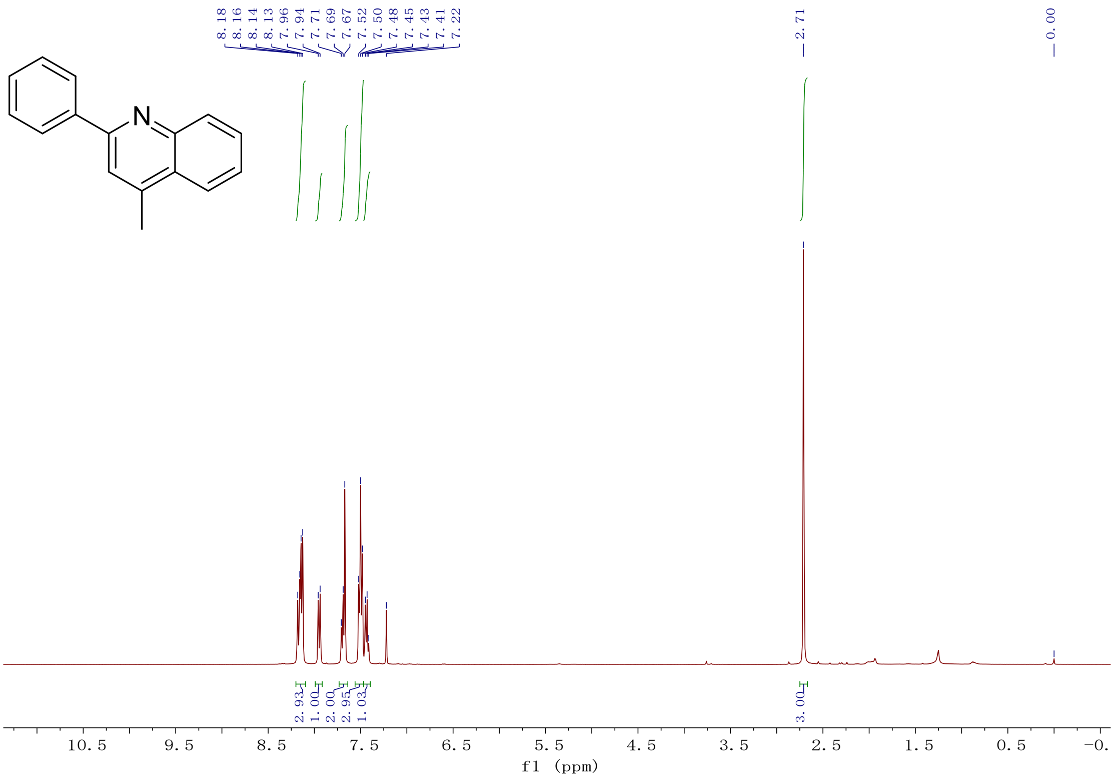


Figure S54. ^{1}H NMR spectrum of **3o′** in $CDCl_3$

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

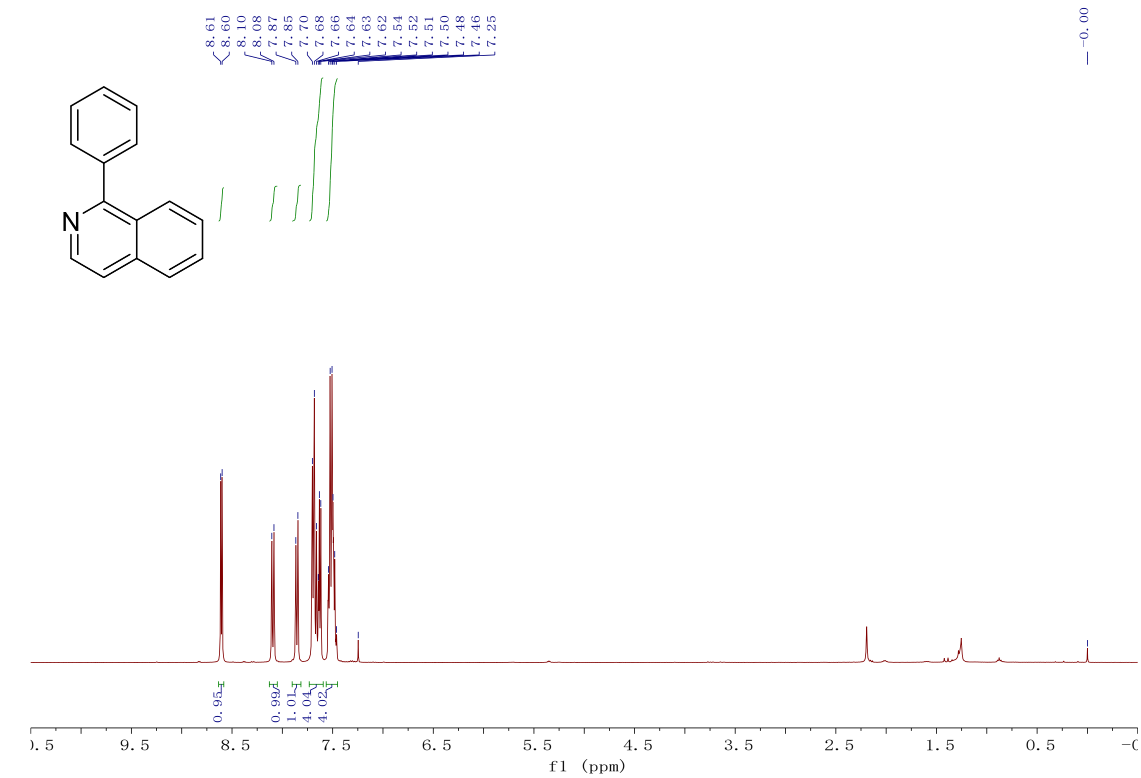


Figure S55. ^{1}H NMR spectrum of **3p′** in $CDCl_3$

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

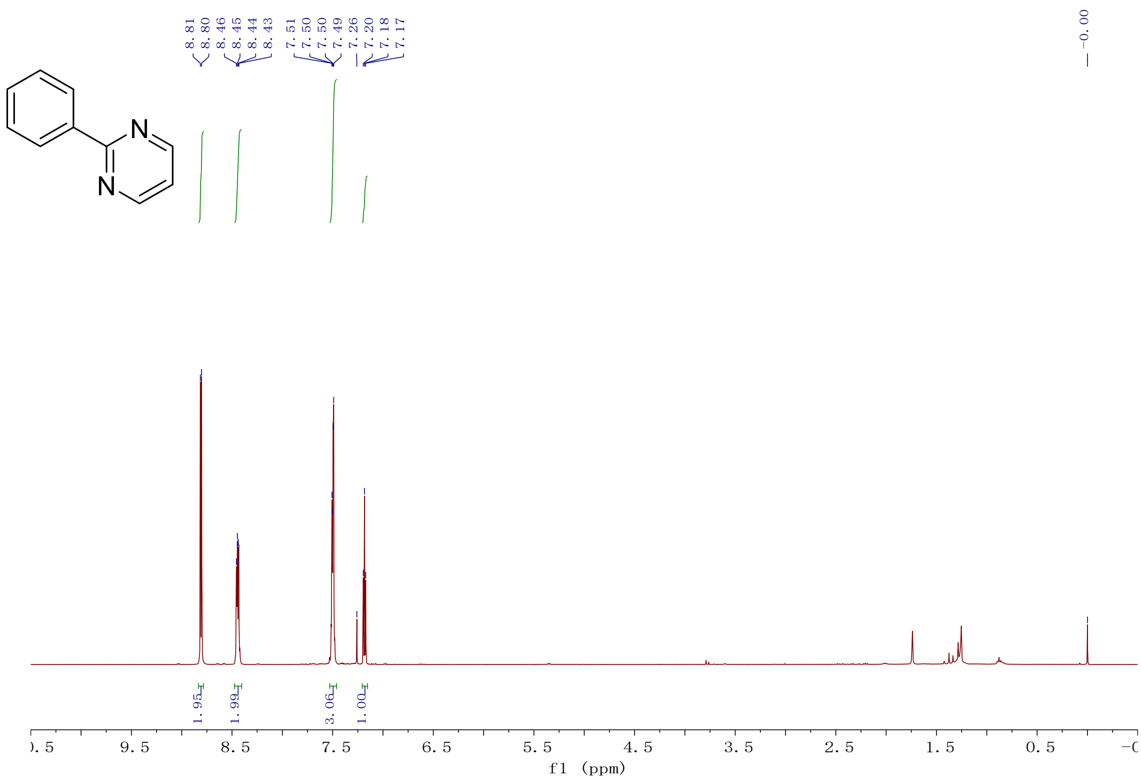


Figure S56. ^{1}H NMR spectrum of **3q′** in $CDCl_3$

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

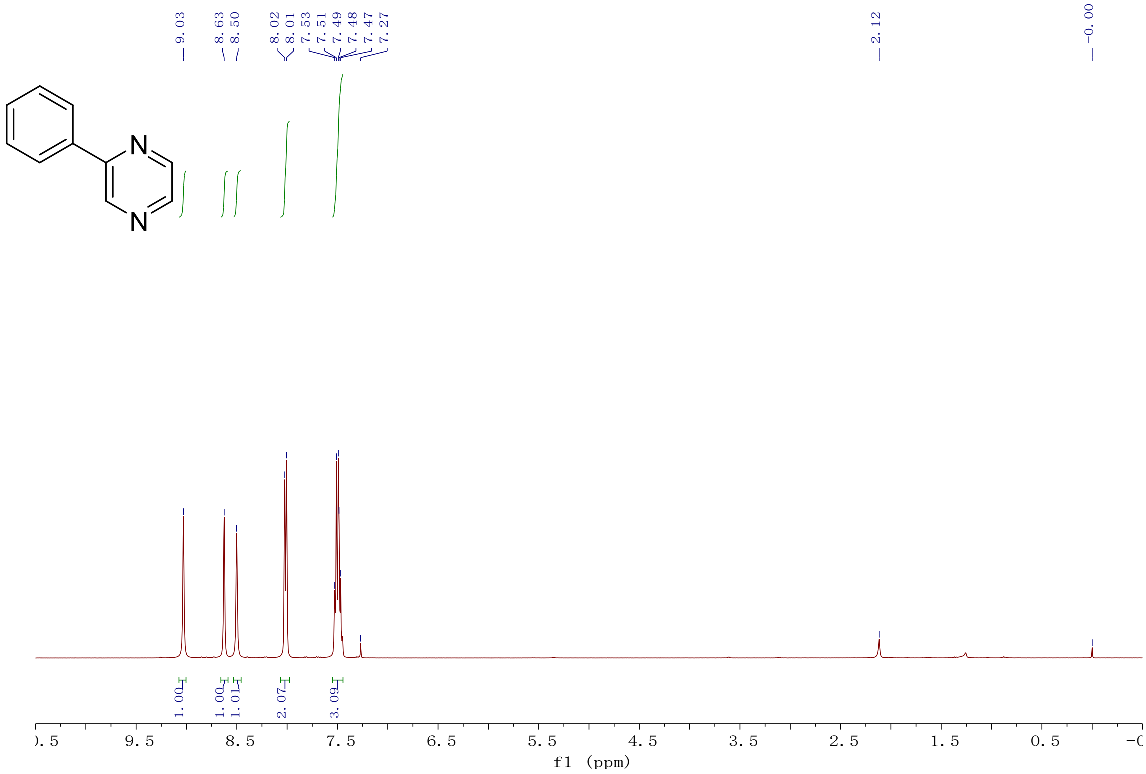


Figure S57. ^{1}H NMR spectrum of **3r′** in $CDCl_3$

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

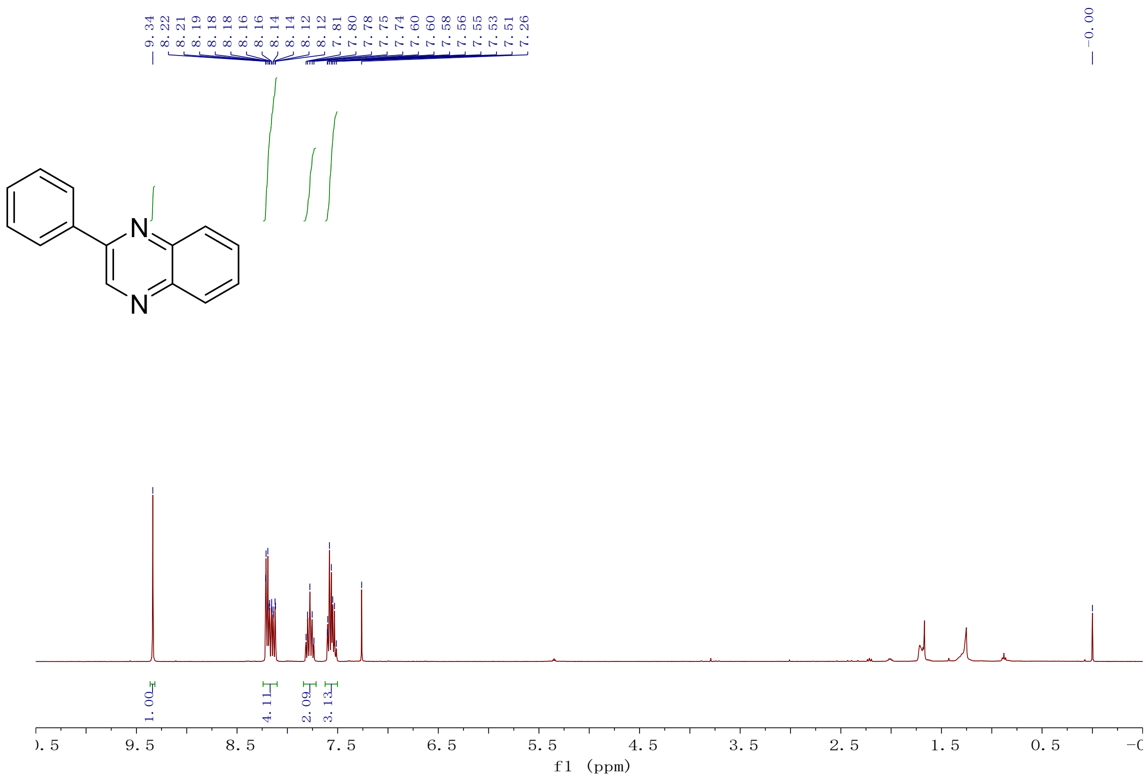


Figure S58. ^{1}H NMR spectrum of **3s′** in $CDCl_3$

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

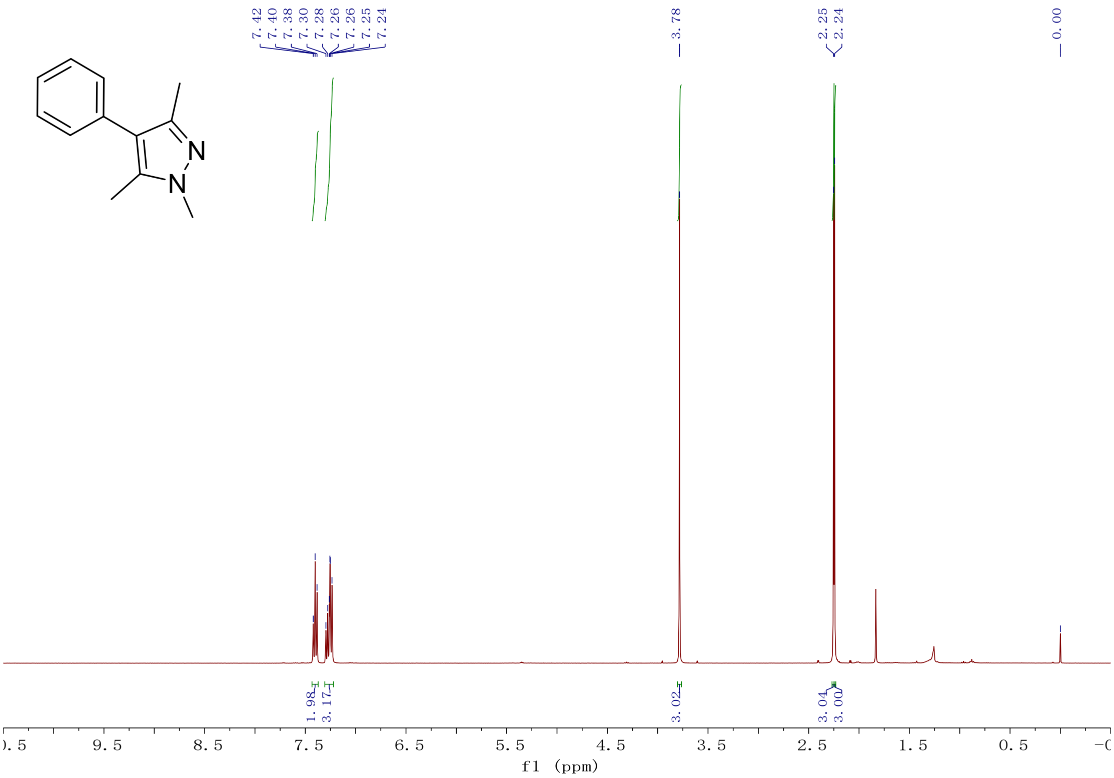


Figure S59. ^{1}H NMR spectrum of **3t′** in $CDCl_3$

^{13}C NMR (101 MHz, $CDCl_3$)

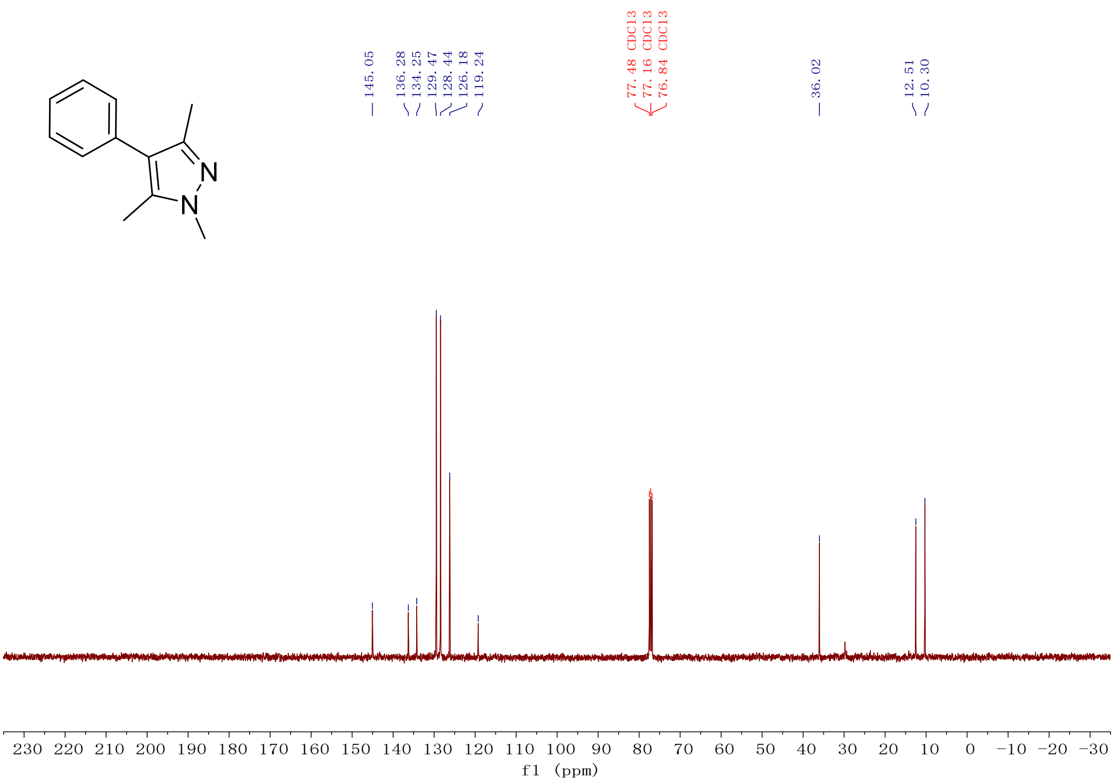


Figure S60 ^{13}C NMR spectrum of **3t′** in $CDCl_3$

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

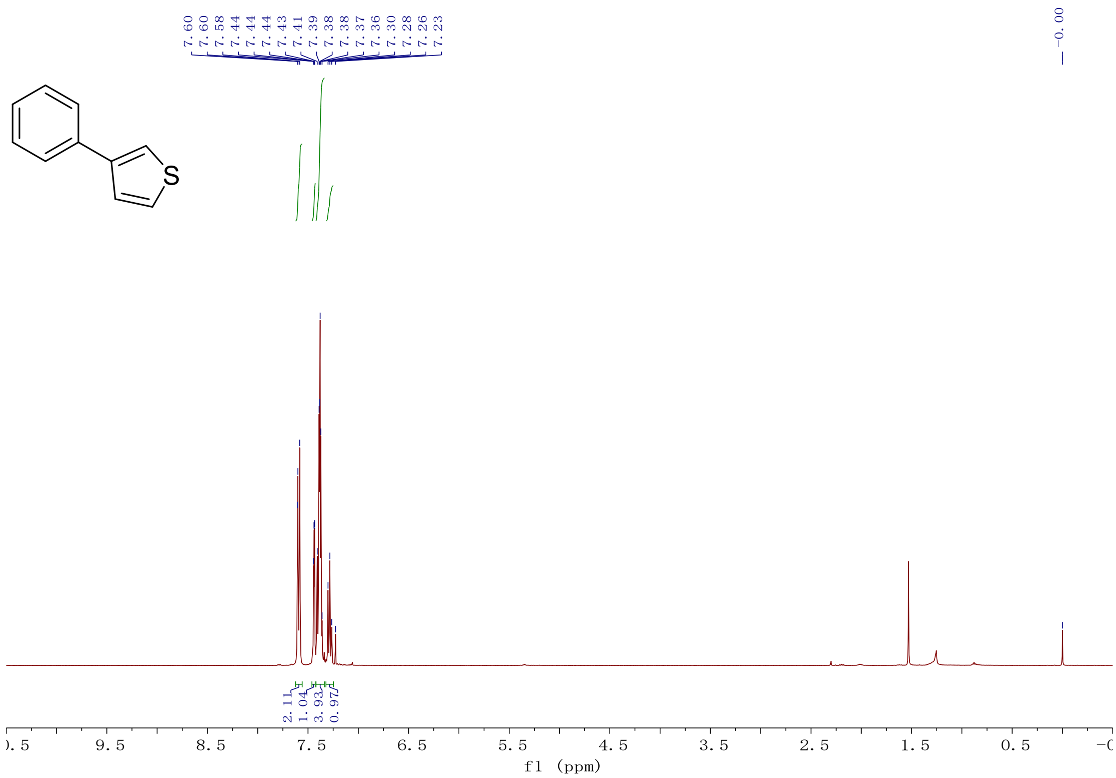


Figure S61. ^{1}H NMR spectrum of **3u′** in $CDCl_3$

References

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