Electronic Supplementary Information for

Deprotonation of isoazatruxene enables photocatalytic arylation and phosphorylation of (hetero)aryl halides

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1. General methods

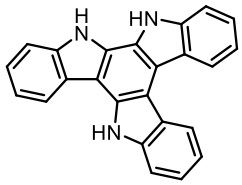
Unless stated otherwise, all reactions were carried out in flame-dried glassware under a dry nitrogen atmosphere. All solvents were purified and dried according to standard methods prior to use.

^{1}H and ^{13}C NMR spectra were recorded on a Bruker instrument (400 MHz and 100 MHz, respectively) and internally referenced to tetramethylsilane signal or residual protio solvent signals. Data for ^{1}H NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, coupling constant(s) in Hz, integration). Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm). High-resolution mass spectrometry (HRMS) was recorded on a Q-TOF (AB SCIEX X500R with ESI source, and Agilent 6545 LC with ESI source), which combines quadrupole precursor ion selection and a high-resolution accurate-mass (HR/AM) Time of Flight mass analyzer to deliver mass accuracy. Fourier Transform Infrared spectra were recorded on a Nicolet IS50 FT-IR spectrophotometer.

Substrates $^{n}Bu_4NPF_6$ were purchased from Energy-chemical, and solvents were purchased from Aladdin, and used without further purification.

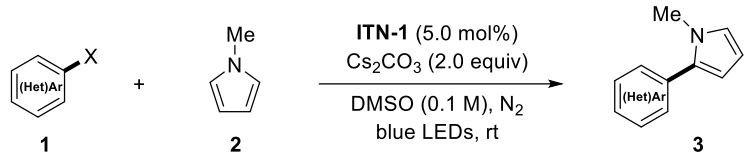
2. Synthesis of ITN-1

The substrate **ITN-1** was a known compound. The synthesis of **ITN-1** was accomplished following the reported procedure[1].

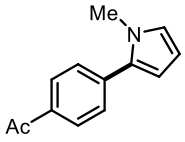


ITN-1[1], gray solid. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.87 (s, 1H), 11.48 (s, 1H), 11.38 (s, 1H), 8.87-8.78 (m, 3H), 7.82 (d, J = 8.4 Hz, 3H), 7.50-7.37 (m, 6H).

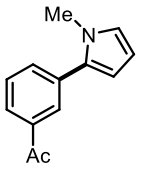
3. General procedure for photocatalytic dehalogenative arylation



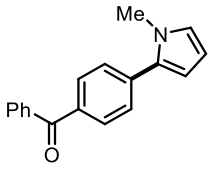
To a flame-dried sealed tube were added **ITN-1** (3.5 mg, 5.0 mol%), **1** (0.2 mmol), **2** (4.0 mmol) and Cs_2CO_3 (0.4 mmol) in DMSO (2.0 mL, 0.1 M). The reaction mixture was degassed via freeze-pump-thaw for 3 cycles. After the reaction mixture was thoroughly degassed, the vial was sealed and positioned approximately 2~3 cm from 30 W blue LEDs. Then the reaction mixture was stirred at room temperature for the indicated time (monitored by TLC) under a nitrogen atmosphere. Afterwards, the reaction mixture was concentrated by rotary evaporation. Then the residue was purified by silica gel column chromatography using ethyl acetate/petroleum ether as the eluent to afford the desired products **3**. The analytical data of the products **3a-3r** are summarized below.



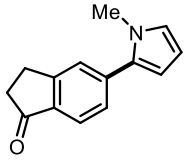
3a[2], 32.3 mg, brown liquid, 81% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 6.77 (t, *J* = 2.8 Hz, 1H), 6.35 (dd, *J* = 4.0, 2.0 Hz, 1H), 6.24-6.22 (m, 1H), 3.72 (s, 3H), 2.62 (s, 3H).



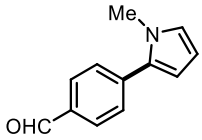
3b[2], 27.2 mg, brown liquid, 69% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (t, *J* = 1.6 Hz, 1H), 7.87 (dt, *J* = 7.6, 1.6 Hz, 1H), 7.60 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 1H), 6.74 (t, *J* = 2.0 Hz, 1H), 6.28 (dd, *J* = 3.6, 2.0 Hz, 1H), 6.22 (t, *J* = 2.8 Hz, 1H), 3.68 (s, 3H), 2.62 (s, 3H).



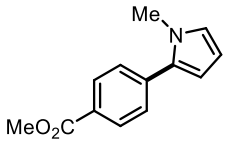
3c[2], 39.5 mg, yellow liquid, 76% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.81 (m, 4H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.52-7.47 (m, 4H), 6.77 (t, *J* = 2.0 Hz, 1H), 6.36 (dd, *J* = 4.0, 2.0 Hz, 1H), 6.23 (t, *J* = 3.2 Hz, 1H), 3.73 (s, 3H).



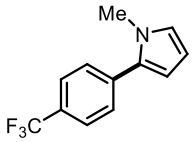
3d[2], 31.6 mg, yellow solid, 75% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.0 Hz, 1H), 7.49 (s, 1H), 7.41 (d, *J* = 8.0 Hz, 1H), 6.78 (t, *J* = 2.4 Hz, 1H), 6.37-6.35 (m, 1H), 6.24-6.22 (m, 1H), 3.73 (s, 3H), 3.17 (t, *J* = 5.6 Hz, 2H), 2.74-2.71 (m, 2H).



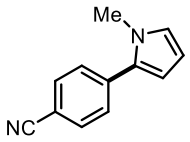
3e[2], for aryl bromide, 24.8 mg, brown liquid, 67% yield; for aryl chloride, 16.3 mg, brown liquid, 44% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.01 (s, 1H), 7.90 (dt, *J* = 8.4, 1.6 Hz, 2H), 7.56 (dt, *J* = 8.4, 1.6 Hz, 2H), 6.79-6.78 (m, 1H), 6.38 (dd, *J* = 3.6, 1.6 Hz, 1H), 6.24 (dd, *J* = 4.0, 2.8 Hz, 1H), 3.74 (s, 3H).



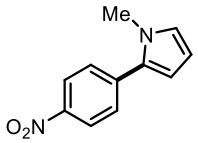
3f[3], 28.2 mg, white solid, 66% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08-8.04 (m, 2H), 7.50-7.46 (m, 2H), 6.78-7.75 (m, 1H), 6.36-6.33 (m, 1H), 6.24-6.21 (m, 1H), 3.93 (s, 3H), 3.71 (s, 3H).



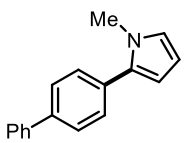
3g[3], 28.1 mg, white solid, 62% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 6.76 (t, *J* = 2.0 Hz, 1H), 6.31 (dd, *J* = 3.6, 1.6 Hz, 1H), 6.23-6.22 (m, 1H), 3.70 (s, 3H).



3h[3], 28.2 mg, yellow liquid, 77% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 6.78 (t, *J* = 2.4 Hz, 1H), 6.34 (dd, *J* = 3.6, 1.6 Hz, 1H), 6.24-6.22 (m, 1H), 3.71 (s, 3H).

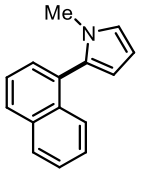


3i[2], 33.8 mg, yellow solid, 84% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (dt, *J* = 8.8, 2.8 Hz, 2H), 7.54 (dt, *J* = 8.8, 2.4 Hz, 2H), 6.82-6.80 (m, 1H), 6.41 (dd, *J* = 3.6, 2.0 Hz, 1H), 6.24 (dd, *J* = 3.6, 2.4 Hz, 1H), 3.75 (s, 3H).

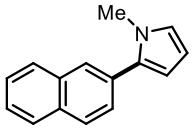


3j[4], 23.8 mg, white solid, 51% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66-7.61 (m,

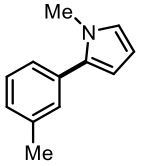
4H), 7.46 (d, *J* = 8.4 Hz, 2H), 7.45 (t, *J* = 7.6 Hz, 2H) 7.35 (t, *J* = 7.2 Hz, 1H), 6.74 (t, *J* = 2.0 Hz, 1H), 6.29-6.28 (m, 1H), 6.23-6.22 (m, 1H), 3.72 (s, 3H).



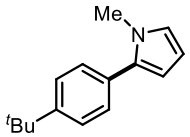
3k[4], 28.1 mg, yellow liquid, 68% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (t, *J* = 6.4 Hz, 2H), 7.71 (d, *J* = 8.4 Hz, 1H), 7.53-7.42 (m, 4H), 6.81 (t, *J* = 2.8 Hz, 1H), 6.31 (t, *J* = 3.2 Hz, 1H), 6.26 (dd, *J* = 3.6, 2.0 Hz, 1H), 3.39 (s, 3H).



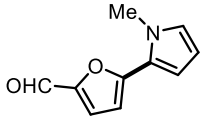
3l[4], 27.6 mg, white solid, 67% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.82 (m, 4H), 7.55 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.50-7.43 (m, 2H), 6.75 (t, *J* = 1.6 Hz, 1H), 6.34 (dd, *J* = 3.6, 1.6 Hz, 1H), 6.25 (dd, *J* = 3.6, 2.4 Hz, 1H), 3.72 (s, 3H).



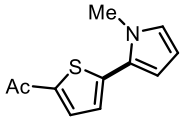
3m[5], 11.6 mg, brown liquid, 34% yield. δ 7.28 (t, *J* = 7.6 Hz, 1H), 7.23-7.18 (m, 2H), 7.11 (d, *J* = 7.6 Hz, 1H), 6.70 (t, *J* = 2.4 Hz, 1H), 6.22-6.18 (m, 2H), 3.65 (s, 3H), 2.38 (s, 3H).



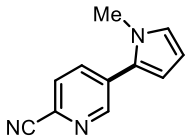
3n[6], 26.7 mg, brown liquid, 63% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.8 Hz, 2H), 7.33 (d, *J* = 8.8 Hz, 2H), 6.69 (t, *J* = 2.4 Hz, 1H), 6.21-6.18 (m, 2H), 3.66 (s, 3H), 1.35 (s, 9H).



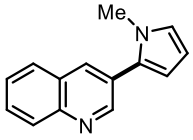
3o[4], 18.4 mg, brown liquid, 53% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.55 (s, 1H), 7.29 (d, *J* = 4.0 Hz, 1H), 6.75 (t, *J* = 2.0 Hz, 1H), 6.70 (dd, *J* = 4.0, 2.0 Hz, 1H), 6.54 (d, *J* = 3.6 Hz, 1H), 6.19 (dd, *J* = 4.0, 2.8 Hz, 1H), 3.91 (s, 3H).



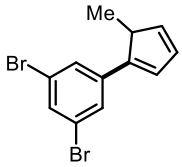
3p[2], 28.3 mg, brown liquid, 69% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 4.0 Hz, 1H), 7.05 (d, *J* = 4.0 Hz, 1H), 6.75 (t, *J* = 2.4 Hz, 1H), 6.51-6.49 (m, 1H), 6.19-6.16 (m, 1H), 3.80 (s, 3H), 2.54 (s, 3H).



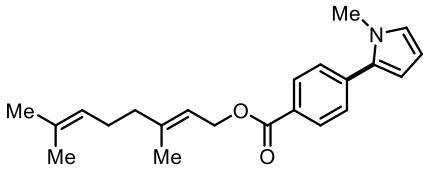
3q[4], 28.2 mg, yellow liquid, 77% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.77 (dd, *J* = 2.4, 0.8 Hz, 1H), 8.30 (dd, *J* = 8.0, 2.4 Hz, 1H), 7.71 (dd, *J* = 8.4, 0.8 Hz, 1H), 6.85-6.84 (m, 1H), 6.44 (dd, *J* = 4.0, 2.0 Hz, 1H), 6.26 (dd, *J* = 4.0, 2.8 Hz, 1H), 3.75 (s, 3H).



3r[4], 34.3 mg, yellow liquid, 82% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.02 (d, *J* = 2.4 Hz, 1H), 8.13-8.09 (m, 2H), 7.82 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.72-7.68 (m, 1H), 7.58-7.54 (m, 1H), 6.82-6.81 (m, 1H), 6.42 (dd, *J* = 3.6, 2.0 Hz, 1H), 6.28 (dd, *J* = 3.6, 2.4 Hz, 1H), 3.75 (s, 3H).



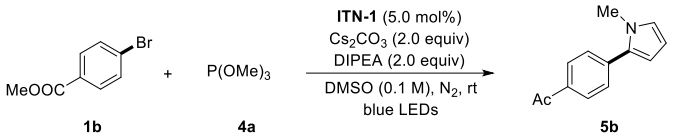
3s[18]**,** 27.2 mg, colorless oil, 79% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 1.6 Hz, 1H), 7.47 (d, *J* = 1.6 Hz, 2H), 6.73-6.72 (m, 1H), 6.26-6.25 (m, 1H), 6.20-6.18 (m, 1H), 3.67 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.8, 131.9, 131.4, 129.9, 125.1, 122.8, 110.2, 108.3, 35.2.



3t, 34.6 mg, brown oil, 55% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 2H), 6.75 (t, *J* = 2.0 Hz, 1H), 6.33-6.32 (m, 1H), 6.21 (t, *J* = 2.8 Hz, 1H), 5.48 (t, *J* = 6.8 Hz, 1H), 5.10 (t, *J* = 6.8 Hz, 1H), 4.85 (d, *J* = 6.8 Hz, 2H), 3.70 (s, 3H), 2.14-2.06 (m, 4H), 1.77 (s, 3H), 1.70 (s, 3H), 1.61 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 142.4, 137.6, 133.6, 131.9, 129.8, 128.4, 127.9, 125.1, 123.8, 118.5, 110.0, 108.3, 61.9, 39.6, 35.4, 26.3, 25.7, 17.7, 16.6. IR (thin film): νmax (cm^{-1}) = 2921, 1710, 1606, 1474, 1375, 1266, 1177, 1096, 1058, 768, 705, 682, 452. HRMS (ESI) calcd for $C_{22}H_{27}NO_2$ $[M+H]^+$: 338.2115. Found: 338.2123.

4. General procedure for photocatalytic dehalogenative phosphorylation

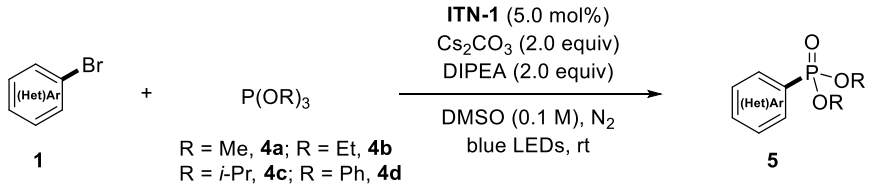
4.1 Table S1 Optimization of the reaction conditions



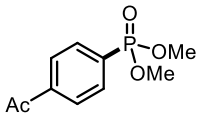
entry	variations from standard conditions	yield (%)[b]
1	none	71 (69)[c]
2	no Cs_2CO_3	11
3	K_2CO_3 instead of Cs_2CO_3	41
4	^{t}BuOK instead of Cs_2CO_3	29
5	DBU instead of Cs_2CO_3	30
6	no DIPEA	46
7	no **ITN-1**	0
8	no light	0

[a] Reaction conditions: a solution of **ITN-1** (1.7 mg, 5.0 mol%), **1b** (0.1 mmol, 1.0 equiv), **4a** (1.0 mmol, 10.0 equiv), DIPEA (0.2 mmol, 2.0 equiv), and Cs_2CO_3 (0.2 mmol, 2.0 equiv) in DMSO (1.0 mL, 0.1 M) was irradiated by blue LEDs (30 W) at room temperature under nitrogen atmosphere for 24 h. [b] Determined by ^{1}H NMR yield using CH_2Br_2 as an internal standard. [c] Isolated yield. DBU = 1,8-diazabicyclo[5.4.0]undec-7-ene.

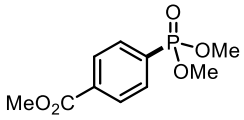
4.2 General procedure for photocatalytic dehalogenative phosphorylation



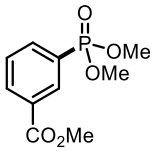
To a flame-dried sealed tube were added **ITN-1** (3.5 mg, 5.0 mol%), **1** (0.2 mmol), $P(OR)_3$ (2.0 mmol), DIPEA (0.4 mmol), and Cs_2CO_3 (0.4 mmol) in DMSO (2.0 mL, 0.1 M). The reaction mixture was degassed via freeze-pump-thaw for 3 cycles. After the reaction mixture was thoroughly degassed, the vial was sealed and positioned approximately 2~3 cm from 30 W blue LEDs. Then the reaction mixture was stirred at room temperature for the indicated time (monitored by TLC) under a nitrogen atmosphere. Afterwards, the reaction mixture was concentrated by rotary evaporation. Then the residue was purified by silica gel column chromatography using ethyl acetate/petroleum ether as the eluent to afford the desired products **5**. The analytical data of the product **5a-5t** are summarized below.



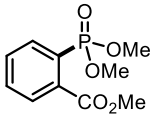
5a[7], 25.4 mg, colorless liquid, 56% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 8.4, 4.0 Hz, 2H), 7.91 (dd, *J* = 12.8, 8.4 Hz, 2H), 3.81 (s, 3H), 3.78 (s, 3H), 2.65 (s, 3H).



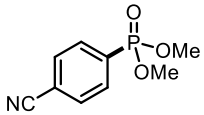
5b[8], 16.9 mg, yellow liquid, 69% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, *J* = 8.0, 3.6 Hz, 2H), 7.89 (dd, *J* = 12.8, 8.0 Hz, 2H), 3.96 (s, 3H), 3.81 (s, 3H), 3.78 (s, 3H).



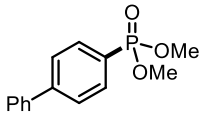
5c[8], 43.8 mg, yellow liquid, 90% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (dt, *J* = 14.0, 1.6 Hz, 1H), 8.26-8.23 (m, 1H), 8.04-7.98 (m, 1H), 7.62-7.56 (m, 1H), 3.95 (s, 3H), 3.81 (s, 3H), 3.78 (s, 3H).



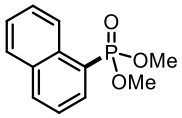
5d[9], 32.0 mg, yellow liquid, 74% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.93 (m, 1H), 7.79-7.75 (m, 1H), 7.64-7.56 (m, 2H), 3.95 (s, 3H), 3.83 (s, 3H), 3.80 (s, 3H).



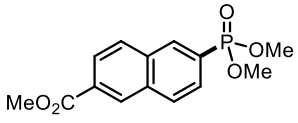
5e[7], 32.5 mg, brown liquid, 72% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95-7.89 (m, 2H), 7.80-7.76 (m, 2H), 3.82 (s, 3H), 3.79 (s, 3H).



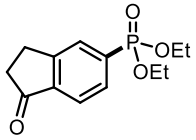
5f[6], 46.4 mg, brown solid, 89% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 8.8, 4.0 Hz, 2H), 7.71-7.68 (m, 2H), 7.62-7.59 (m, 2H), 7.46 (t, *J* = 7.2 Hz, 2H), 7.39 (t, *J* = 6.8 Hz, 1H), 3.80 (s, 3H), 3.78 (s, 3H).



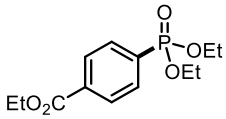
5g[7], 27.1 mg, colorless liquid, 57% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (d, *J* = 8.4 Hz, 1H), 8.24 (dd, *J* = 16.4, 7.2 Hz, 1H), 8.06 (d, *J* = 8.4 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.58-7.52 (m, 2H), 3.81 (s, 3H), 3.78 (s, 3H).



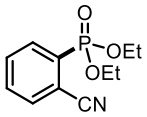
5h[7], 48.6 mg, colorless liquid, 83% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (s, 1H), 8.46 (d, *J* = 15.6 Hz, 1H), 8.15 (dd, *J* = 8.4, 1.6 Hz, 1H), 8.04 (dd, *J* = 8.4, 4.0 Hz, 1H), 7.99 (d, *J* = 7.6 Hz, 1H), 7.84-7.79 (m, 1H), 4.00 (s, 3H), 3.84 (s, 3H), 3.81 (s, 3H).



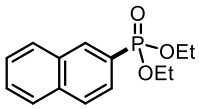
5i[10], 28.4 mg, yellow liquid, 53% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 13.6 Hz, 1H), 7.85-7.77 (m, 2H), 4.21-4.12 (m, 4H), 3.21 (t, *J* = 5.6 Hz, 2H), 2.77-2.74 (m, 2H), 1.35 (t, *J* = 7.2 Hz, 6H).



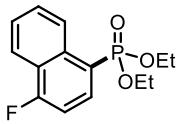
5j[8], 40.3 mg, orange liquid, 70% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, *J* = 8.0, 3.6 Hz, 2H), 7.89 (dd, *J* = 13.2, 8.0 Hz, 2H), 4.41 (q, *J* = 7.2 Hz, 2H), 4.21-4.07 (m, 4H), 1.41 (t, *J* = 6.8 Hz, 3H), 1.33 (t, *J* = 7.2 Hz, 6H).



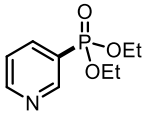
5k[8], 38.8 mg, orange liquid, 81% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, *J* = 14.0, 7.2 Hz, 1H), 7.82 (t, *J* = 6.8 Hz, 1H), 7.73-7.65 (m, 2H), 4.32-4.17 (m, 4H), 1.39 (t, *J* = 6.8 Hz, 6H).



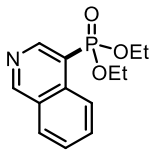
5l[9], 32.5 mg, brown liquid, 62% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, *J* = 14.0 Hz, 1H), 7.95-7.87 (m, 3H), 7.79-7.74 (m, 1H), 7.63-7.54 (m, 2H), 4.23-4.08 (m, 4H), 1.34 (t, *J* = 6.8 Hz, 6H).



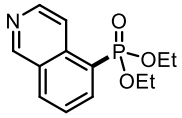
5m[10], 52.9 mg, yellow liquid, 94% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (d, *J* = 8.4 Hz, 1H), 8.27-8.21 (m, 1H), 8.18 (d, *J* = 9.2 Hz, 1H), 7.68 (t, *J* = 8.4 Hz, 1H), 7.62 (t, *J* = 8.4 Hz, 1H), 7.23-7.18 (m, 1H), 4.26-4.16 (m, 2H), 4.13-4.05 (m, 2H), 1.31 (t, *J* = 7.2 Hz, 6H).



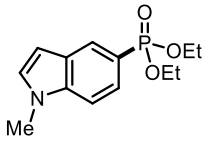
5n[11], 22.4 mg, orange liquid, 52% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.98 (d, *J* = 4.2 Hz, 1H), 8.78 (t, *J* = 2.4 Hz, 1H), 8.15-8.06 (m, 1H), 7.44-7.40 (m, 1H), 4.23-4.10 (m, 4H), 1.35 (t, *J* = 7.2 Hz, 6H).



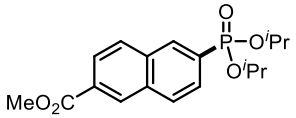
5o[9], 47.0 mg, brown liquid, 89% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.40 (d, *J* = 2.8 Hz, 1H), 9.07 (d, *J* = 9.6 Hz, 1H), 8.50 (d, *J* = 8.4 Hz, 1H), 8.05 (d, *J* = 8.0 Hz, 1H), 7.87-7.83 (m, 1H), 7.71 (t, *J* = 6.8 Hz, 1H), 4.29-4.12 (m, 4H), 1.34 (t, *J* = 6.8 Hz, 6H).



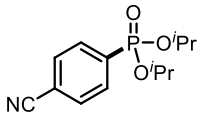
5p[9], 47.4 mg, brown liquid, 89% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.33 (s, 1H), 8.66 (d, *J* = 6.0 Hz, 1H), 8.44 (dd, *J* = 16.0, 7.2 Hz, 1H), 8.31 (d, *J* = 6.0 Hz, 1H), 8.18 (d, *J* = 6.0 Hz, 1H), 7.70 (td, *J* = 7.2, 3.2 Hz, 1H), 4.27-4.10 (m, 4H), 1.33 (t, *J* = 7.2 Hz, 6H).



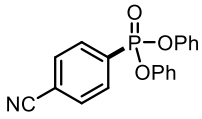
5q[9], 32.8 mg, yellow liquid, 61% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, *J* = 15.6 Hz, 1H), 7.66-7.61 (m, 1H), 7.40 (dd, *J* = 8.4, 2.8 Hz, 1H), 7.13 (d, *J* = 2.8 Hz, 1H), 6.58 (dd, *J* = 3.2, 1.2 Hz, 1H), 4.17-4.02 (m, 4H), 3.83 (s, 3H), 1.32 (t, *J* = 7.2 Hz, 6H).



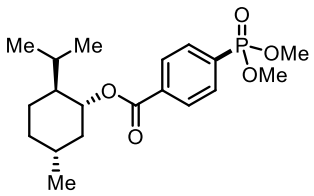
5r[10], 42.2 mg, white solid, 60% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (s, 1H), 8.48 (d, *J* = 15.6 Hz, 1H), 8.14 (d, *J* = 8.8 Hz, 1H), 8.04-7.98 (m, 2H), 7.86-7.81 (m, 1H), 4.80-4.72 (m, 2H), 4.00 (s, 3H), 1.41(d, *J* = 6.4 Hz, 6H), 1.24 (d, *J* = 6.4 Hz, 6H).



5s[9], 39.1 mg, colorless liquid, 73% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96-7.90 (m, 2H), 7.77-7.74 (m, 2H), 4.80-4.68 (m, 2H), 1.39 (d, *J* = 6.4 Hz, 6H), 1.24 (d, *J* = 6.0 Hz, 6H).

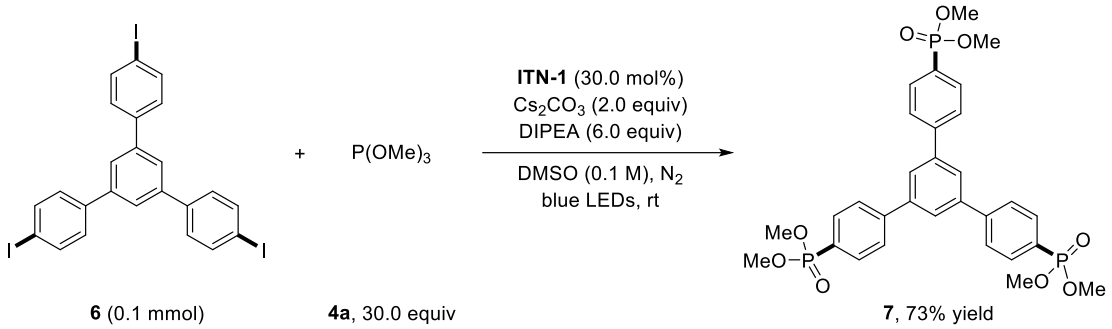


5t[9], 35.1 mg, colorless liquid, 52% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (dd, *J* = 13.6, 8.0 Hz, 2H), 7.79 (dd, *J* = 8.0, 3.6 Hz, 2H), 7.32 (t, *J* = 8.4 Hz, 4H), 7.21-7.15 (m, 6H).



5u, 40 mg, brown oil, 59% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15-8.12 (m, 2H), 7.91-7.86 (m, 2H), 4.96 (td, *J* = 10.8, 4.4 Hz, 1H), 3.79 (s, 3H), 3.77 (s, 3H), 2.15-2.10 (m, 1H), 1.97-1.90 (m, 1H), 1.76-1.72 (m, 2H), 1.61-1.54 (m, 2H), 1.16-1.01 (m, 2H), 0.95-0.91 (m, 7H), 0.79 (d, *J* = 10.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2, 134.5, 131.9 (d, *J* = 10.0 Hz), 131.5 (d, *J* = 186.0 Hz), 129.4 (d, *J* = 15.0 Hz), 75.5, 52.8 (d, *J* = 6.0 Hz), 47.2, 40.9, 34.3, 31.5, 26.6, 23.6, 22.0, 20.7, 16.5. IR (thin film): νmax (cm^{-1}) = 2953, 1715, 1455, 1286, 1265, 1180, 1103, 1054, 1018, 959, 830, 788, 762, 731, 696, 587, 521. HRMS (ESI) calcd for $C_{22}H_{27}NO_2$ $[M+Na]^+$: 391.1645. Found: 391.1652.

5. Application to photocatalytic multi-phosphorylation



To a flame-dried sealed tube were added **ITN-1** (10.2 mg, 30.0 mol%), **6** (0.1 mmol), **4a** (3.0 mmol), DIPEA (0.6 mmol), and Cs_2CO_3 (0.2 mmol) in DMSO (1.0 mL, 0.1 M). The reaction mixture was degassed via freeze-pump-thaw for 3 cycles. After the reaction mixture was thoroughly degassed, the vial was sealed and positioned approximately 2~3 cm from 30 W blue LEDs. Then the reaction mixture was stirred at room temperature for the indicated time (monitored by TLC) under nitrogen atmosphere. Afterwards, the reaction mixture was concentrated by rotary evaporation. Then the residue was purified by silica gel column chromatography (PE/EtOAc = 10/1) to afford the desired product **7** (46.2 mg, 73% yield) as an orange liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97-7.91 (m, 6H), 7.85 (s, 3H), 7.82-7.79 (m, 6H), 3.84 (s, 9H), 3.81 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.7 (d, J = 3.0 Hz), 141.6, 132.6 (d, J = 11.0 Hz), 127.5 (d, J = 15.0 Hz), 126.2, 126.1 (d, J = 190.0 Hz), 52.8 (d, J = 6.0 Hz). IR (thin film): νmax (cm^{-1}) = 3448, 2954, 2851, 1596, 1384, 1240, 1184, 1133, 1054, 1027, 821, 779, 689, 570, 543, 461. HRMS (ESI) calcd for $C_{30}H_{33}O_9P_3$ $[M+Na]^+$: 653.1229. Found: 653.1230.

6. Mechanistic studies

1) UV/vis absorption spectroscopic measurements

UV/vis absorption spectra of **ITN-1** and **ITN-1** with Cs_2CO_3 in DMSO were recorded in 1 cm path quartz cuvettes using Pgeneral TU-1901 UV/vis spectrometer.

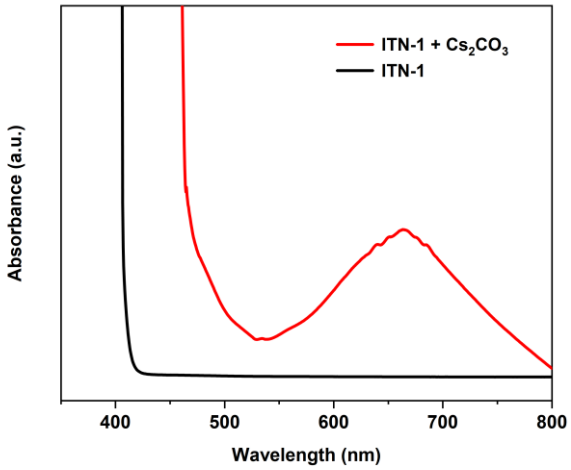


Fig. S1 UV/vis absorption spectra of **ITN-1** and **ITN-1** with 40 equiv Cs_2CO_3.

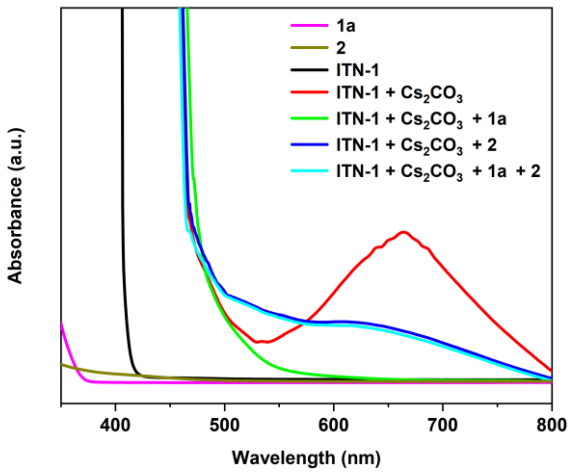


Fig. S2 UV/vis absorption spectra of the reaction mixture.

2) UV/vis absorption and emission spectra of ITN-1 with Cs_2CO_3

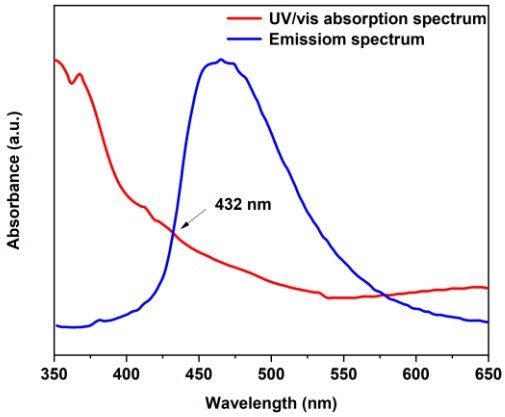


Fig. S3 UV/vis absorption and emission spectra of **ITN-1** with 40 equiv Cs_2CO_3 in DMSO (0.01 mM). Cross point λ: 432 nm. $E_{0\text{-}0}$: 2.87 eV.

3) Electrochemical measurements

Voltammetric experiments were conducted with a computer-controlled Shanghai Chen Hua CHI660E containing glassy carbon electrode serving as the working electrode, saturated calomel reference electrode, Pt wire auxiliary electrode.

Tetrabutylammonium hexafluorophosphate (775 mg, 2.0 mmol) was added to a 0.01 M solution of the **ITN-1** with 40.0 equiv Cs_2CO_3 in 20 mL DMSO, and the solution was vigorously bubbled with high purity nitrogen for 15 minutes before the measurement.

Excited state oxidation and reduction potentials were calculated by the following approximating formulas: $E_{1/2}$ ($PC^*/PC^{\bullet-}$) = $E_{1/2}(PC/PC^{\bullet-})$ + $E_{0,0}$ and $E_{1/2}$ ($PC^{\bullet+}/PC^*$) = $E_{1/2}$ ($PC^{\bullet+}/PC$) – $E_{0,0}$.

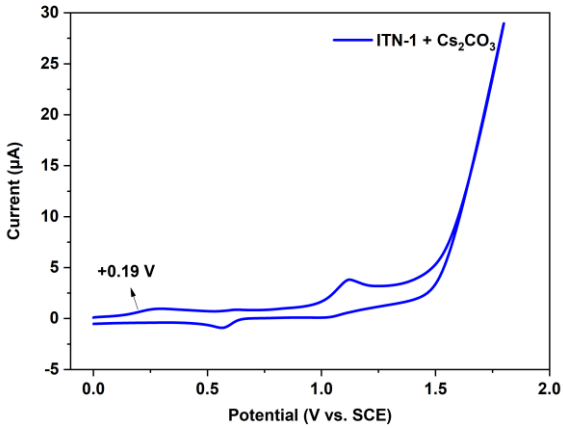


Fig. S4 Cyclic voltammogram of **ITN-1** with 40 equiv Cs_2CO_3 in DMSO (1.0 mM) containing 0.1 M ^{n}Bu_4NPF_6. Scan rate: 0.1 V/s. $E_{1/2}(PC^{\bullet+}/PC)$ = +0.19 V, $E_{1/2}(PC^{\bullet+}/PC^*)$ = -2.68 V.

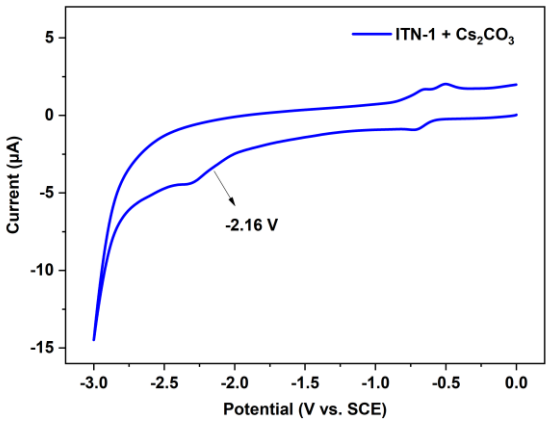


Fig. S5 Cyclic voltammogram of **ITN-1** with 40 equiv Cs_2CO_3 in DMSO (1.0 mM) containing 0.1 M $^{n}Bu_4NPF_6$. Scan rate: 0.1 V/s. $E_{1/2}(PC/PC^{\bullet-})$ =-2.16 V, $E_{1/2}(PC^{*}/PC^{\bullet-})$ = +0.71 V.

4) ^{1}H NMR analysis

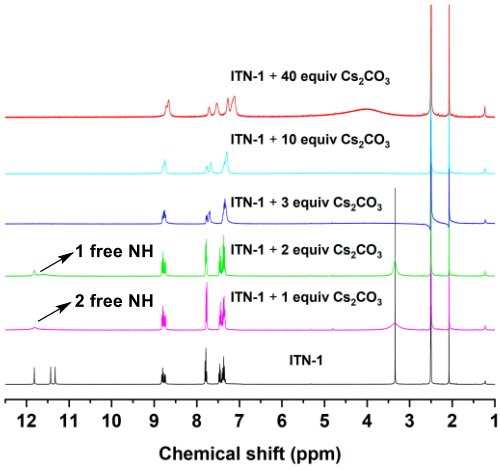


Fig. S6 Systematic ^{1}H NMR studies of **ITN-1** with Cs_2CO_3.

5) UV/vis absorption of ITN-1 with different equivalents of Cs_2CO_3

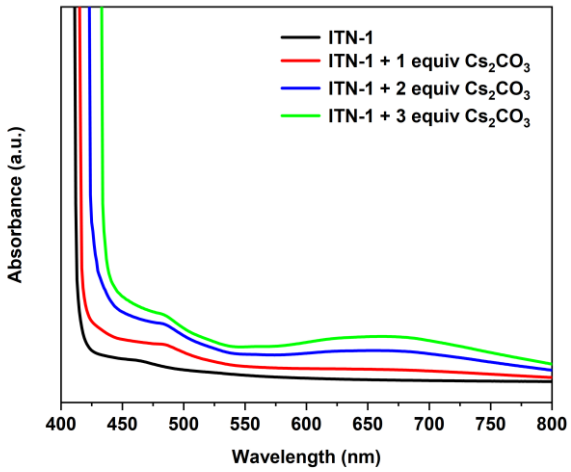


Fig. S7 UV/vis absorption spectra of **ITN-1** with 1, 2, 3 equivalents of Cs_2CO_3.

6) Fluorescence quenching experiments

The concentration of **ITN-1** was 0.01 mM with 40 equiv Cs_2CO_3 in DMSO. The concentration of the quencher (**1a**) was 0.1 M in DMSO. For each quenching experiment, the quencher was titrated to a solution (10 mL) of **ITN-1** with 40 equiv Cs_2CO_3 in a quartz glass bottle, respectively. The addition of the quencher refers to an increase of the quencher concentration of 5×10^{-4} M, 1×10^{-3} M, 3×10^{-3} M, 5×10^{-3} M, 8×10^{-3} M, 1×10^{-2} M. Then the emission intensity of ITN-1 with 40 equiv Cs_2CO_3 was collected respectively.

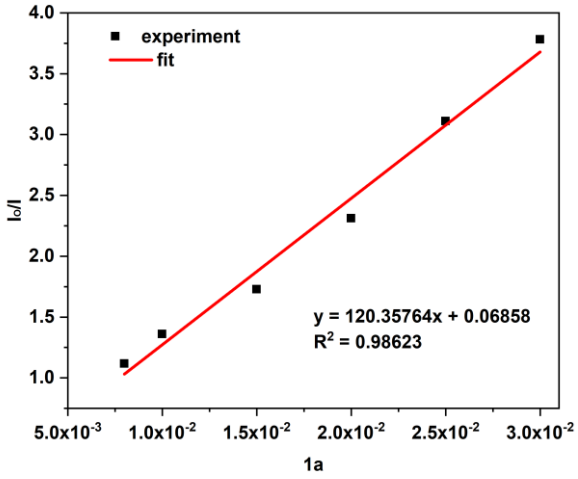


Fig. S8 Stern-Volmer quenching experiments.

7. The apparent quantum yield (AQY) measurement

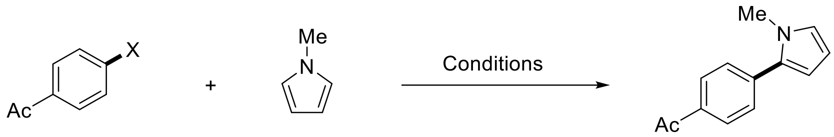
The AQY for reductive arylation and reductive phosphorylation were measured using monochromatic LED lamps with band passfilter of 455 nm. The irradiation area was controlled as 1 x 1 cm^2. The intensity was 17.4 mW cm^{-2}(ILT 950spectroradiometer). The AOY was calculated as AOY=$Ne/Np*100\% = MN_Ahc/SPt\lambda*100\%$, where *Ne* is the amount of reactionelectrons, *Np* is the amount of incident photons, *M* is the amount of reductive arylation or reductive phosphorylation products, N_A is Avogadroconstant, *h* is the Planck constant, *c* is the speed of light, *S* is the irradiation area, *P* is the intensity of the irradiation, *t* is the photo-irradiation time, and λ is the wavelengthof the monochromatic light[12].

Table S2. AQY for reductive arylation and reductive phosphorylation.

	reductive arylation	reductive phosphorylation
t (s)	86400	86400
M (mol)	$8.3x10^{-6}$	$4.96x10^{-5}$
AQY (%)	0.14	0.86

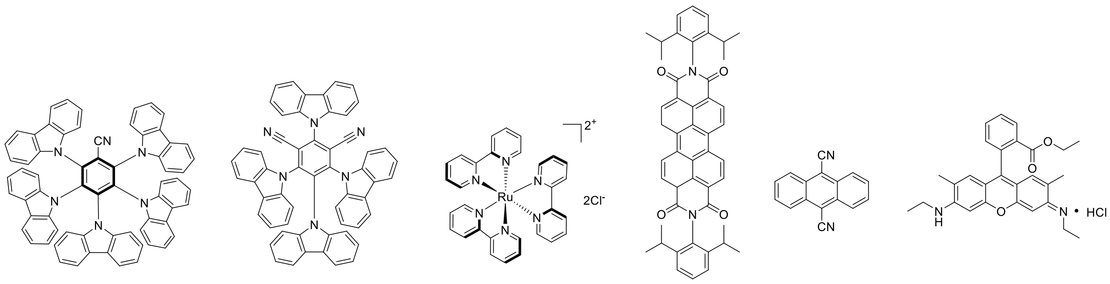
8. Comparison of photocatalytic arylation between our work and other reported synthetic methodologies

Table S3. The yield comparison of photocatalytic arylation between our work and other reports.



1a **2** **3a**

Entry	Conditions	Yield (%)	Reference
1	5 mol% **ITN-1**, 2.0 equiv Cs_2CO_3, DMSO, blue LEDs (X = Br)	**81**	**Our work**
2	5 mol% **5CzBN**, 1.6 equiv Et_3N, DMSO, 420 nm (X = I)	69	[13]
3	5 mol% **4CzIPN**, 2.0 equiv *n*-Bu_3N, 2.0 equiv TMG, DMSO/H_2O, blue LEDs (X = I)	73	[14]
4	1 mol% **Ru(bpy)$_3$Cl$_2$**, 5 mol% pyrene, 1.4 equiv DIPEA, DMSO, 455 nm (X =Br)	74	[15]
5	2 mol% **PDI**, 0.8 mmol *n*-Bu_4NOAc, 3.0 mL DMSO, E = -0.84 V, $Sb_2(S, Se)_3$(-), Pt(+), Ag/AgCl(RE), Xe lamp	84	[16]
6	5 mol% **DCA**, 1.2 equiv DIPEA, MeCN, white LEDs (X = Br)	64	[17]
7	10 mol% **Rh-6G**, 2.2 equiv DIPEA, DMSO, 455 nm (X = Br)	54	[18]
8	**K$_2$Sx** (12.5 mol% per S), 1.5 equiv K_2CO_3, 2.0 equiv H_2O, DMSO, 440 nm (X = Br)	86	[19]



5CzBN **4CzIPN** **Ru(bpy)$_3$Cl$_2$** **PDI** **DCA** **Rh-6G**

9. X-Ray crystal data of 3f

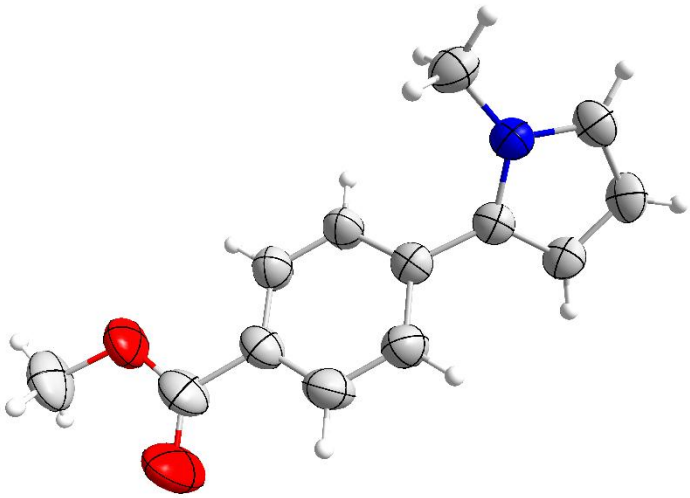


Fig. S9 X-Ray crystal structure of 3f (The crystal was obtained by slow evaporation of the solution of DCM and PE) (CCDC: 2450746)

Table S4 Crystal data and structure refinement for 3f

Identification code	**3f**
Empirical formula	$C_{13}H_{13}NO_2$
Formula weight	215.24
Temperature/K	298.81(10)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	11.5980(4)
b/Å	6.6697(2)
c/Å	14.8861(5)
α/°	90
β/°	100.902(3)
γ/°	90
Volume/Å^3	1130.74(7)
Z	4
ρ_{calc}g/cm^3	1.264
μ/mm^{-1}	0.693
F(000)	456.0
Crystal size/mm^3	0.3 × 0.25 × 0.2
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	7.762 to 150.968
Index ranges	$-14 \le h \le 14$, $-3 \le k \le 8$, $-18 \le l \le 18$
Reflections collected	6237

Independent reflections	2185 [R_{int} = 0.0373, R_{sigma} = 0.0271]
Data/restraints/parameters	2185/0/148
Goodness-of-fit on F^2	1.087
Final R indexes [I>=2σ (I)]	R_1 = 0.0593, wR_2 = 0.1713
Final R indexes [all data]	R_1 = 0.0621, wR_2 = 0.1755
Largest diff. peak/hole / e $Å^{-3}$	0.24/-0.21

Table S5 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 3f. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
N11	133.8(10)	5987.2(18)	6122.8(7)	45.9(4)
O14	5804.5(10)	10333(2)	6167.7(9)	72.7(4)
C7	1218.4(12)	5153(2)	6444.5(9)	44.1(4)
C8	1036.3(14)	3389(2)	6876.6(9)	50.2(4)
C9	-178.6(14)	3152(2)	6818.4(10)	53.2(4)
C1	2350.4(12)	6055(2)	6354.3(9)	46.2(4)
O16	6572.3(12)	7277(3)	6091.1(15)	104.1(6)
C12	-125.8(15)	7831(2)	5604.6(11)	58.6(4)
C10	-712.4(13)	4757(3)	6348.3(10)	51.9(4)
C6	2623.4(14)	8061(2)	6542.8(12)	56.1(4)
C4	4577.9(13)	7574(3)	6258.3(10)	55.1(4)
C13	5758.4(14)	8336(3)	6165.9(12)	64.8(5)
C5	3719.6(14)	8816(3)	6494.1(12)	58.0(4)
C3	4316.4(15)	5564(3)	6085.4(13)	65.3(5)
C2	3220.5(14)	4823(3)	6120.5(12)	59.8(5)
C15	6916.6(16)	11206(4)	6091.4(15)	82.9(7)

Table S6 Anisotropic Displacement Parameters (Å^2×10^3) for 3f. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N11	47.7(7)	50.3(7)	38.8(6)	0.5(5)	5.7(5)	0.1(5)
O14	46.1(7)	86.4(10)	84.4(9)	7.6(7)	9.5(6)	-13.8(6)
C7	46.7(8)	47.9(8)	36.8(7)	-2.5(5)	5.8(5)	-0.6(5)
C8	55.4(9)	49.2(8)	44.1(7)	2.1(6)	4.3(6)	-1.2(6)
C9	57.5(9)	57.7(9)	44.3(7)	0.5(6)	9.6(6)	-12.4(6)
C1	46.7(8)	51.0(8)	40.7(7)	0.4(5)	7.7(5)	0.3(6)
O16	46.3(8)	109.2(12)	158.8(16)	-19.8(11)	24.7(8)	1.9(7)
C12	64.2(10)	56.0(9)	52.5(8)	7.3(7)	3.0(7)	6.5(7)
C10	46.1(8)	64.2(9)	45.3(8)	-4.0(6)	8.4(6)	-6.1(6)

C6	51.4(9)	52.5(9)	68.1(10)	-6.8(7)	21.2(7)	-0.5(6)
C4	41.5(8)	68.6(10)	53.3(8)	-0.3(7)	4.4(6)	-0.8(6)
C13	39.5(8)	89.6(14)	62.2(10)	-4.6(9)	2.1(7)	-1.2(7)
C5	55.0(9)	53.0(9)	67.4(10)	-3.8(7)	15.0(7)	-5.6(7)
C3	47.3(9)	67.6(10)	80.7(12)	-10.1(9)	11.7(7)	9.2(7)
C2	53.6(9)	52.2(9)	72.9(11)	-9.1(7)	10.1(8)	3.1(7)
C15	47.7(10)	121.2(19)	76.6(12)	12.6(12)	3.7(8)	-25.0(10)

Table S7 Bond Lengths for 3f.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N11	C7	1.3760(19)	C1	C6	1.392(2)
N11	C12	1.4527(19)	C1	C2	1.395(2)
N11	C10	1.369(2)	O16	C13	1.201(2)
O14	C13	1.333(2)	C6	C5	1.382(2)
O14	C15	1.439(2)	C4	C13	1.491(2)
C7	C8	1.376(2)	C4	C5	1.390(2)
C7	C1	1.4730(19)	C4	C3	1.388(3)
C8	C9	1.404(2)	C3	C2	1.374(2)
C9	C10	1.362(2)			

Table S8 Bond Angles for 3f.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C7	N11	C12	127.66(13)	C9	C10	N11	108.65(14)
C10	N11	C7	108.85(12)	C5	C6	C1	121.09(14)
C10	N11	C12	123.47(13)	C5	C4	C13	122.27(16)
C13	O14	C15	116.14(16)	C3	C4	C13	118.98(15)
N11	C7	C8	107.29(13)	C3	C4	C5	118.74(15)
N11	C7	C1	125.12(13)	O14	C13	C4	112.19(15)
C8	C7	C1	127.57(13)	O16	C13	O14	123.78(18)
C7	C8	C9	107.94(13)	O16	C13	C4	124.0(2)
C10	C9	C8	107.26(13)	C6	C5	C4	120.38(15)
C6	C1	C7	123.16(13)	C2	C3	C4	120.78(15)
C6	C1	C2	117.95(14)	C3	C2	C1	121.03(15)
C2	C1	C7	118.78(14)				

Table S9 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 3f.

Atom	*x*	*y*	*z*	U(eq)
H8	1615.76	2508.56	7157.52	60
H9	-549.69	2096.21	7056.6	64

H12A	-45.3	8950.49	6017.24	88
H12B	-915.07	7781.75	5262.95	88
H12C	411.38	7979.89	5191.3	88
H10	-1517.81	4979.96	6204.39	62
H6	2058.94	8908.19	6704.16	67
H5	3884.09	10163.17	6619.7	70
H3	4889.7	4708.42	5944.09	78
H2	3055.94	3479.31	5986.39	72
H15A	7098.38	10877.72	5505.9	124
H15B	7518.49	10686.67	6567.01	124
H15C	6874.65	12636.13	6150.45	124

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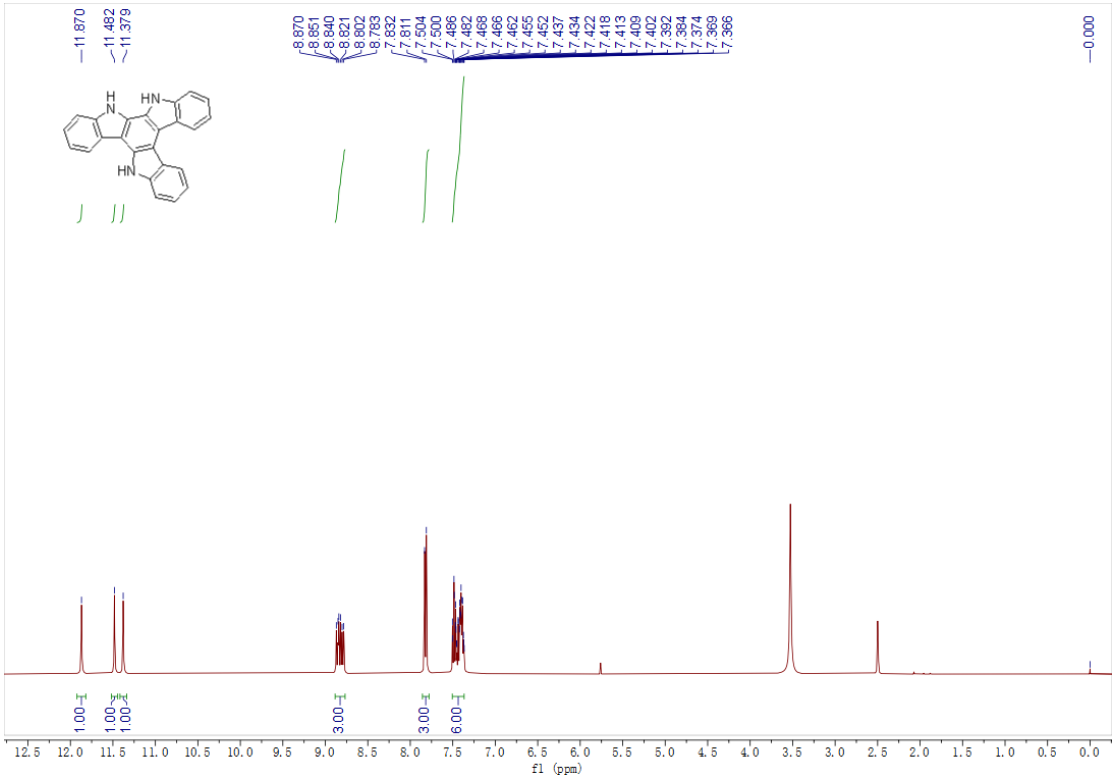
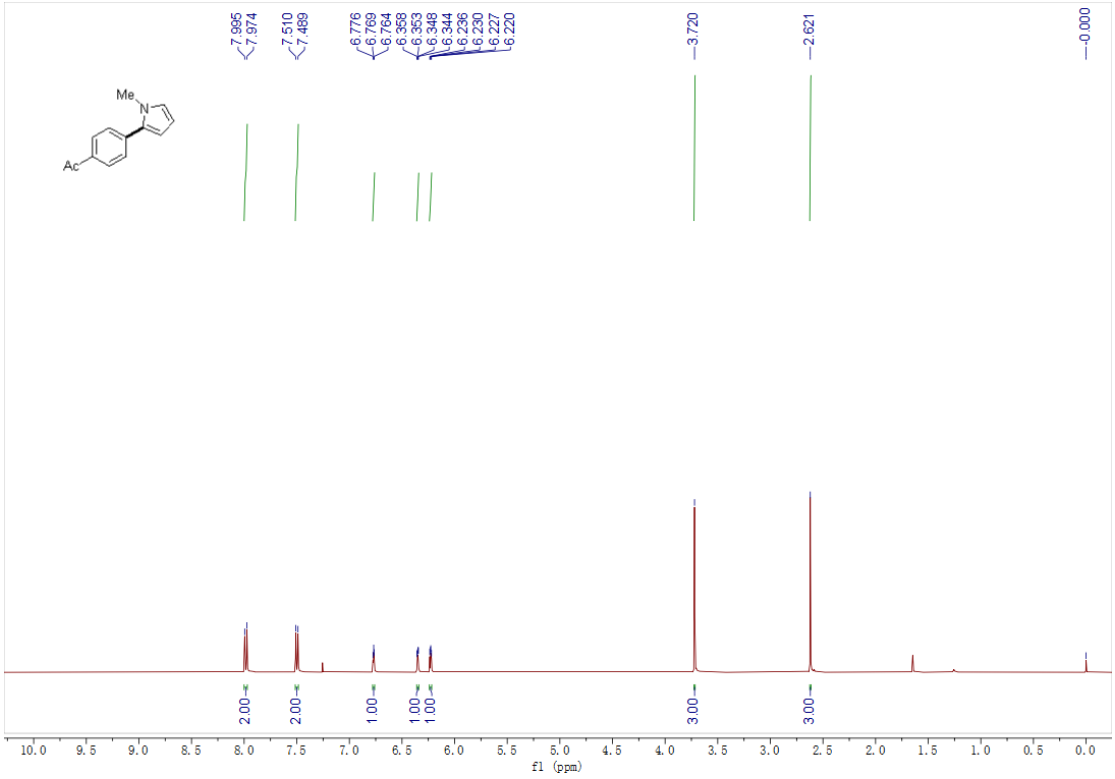
11. Copies of NMR spectra

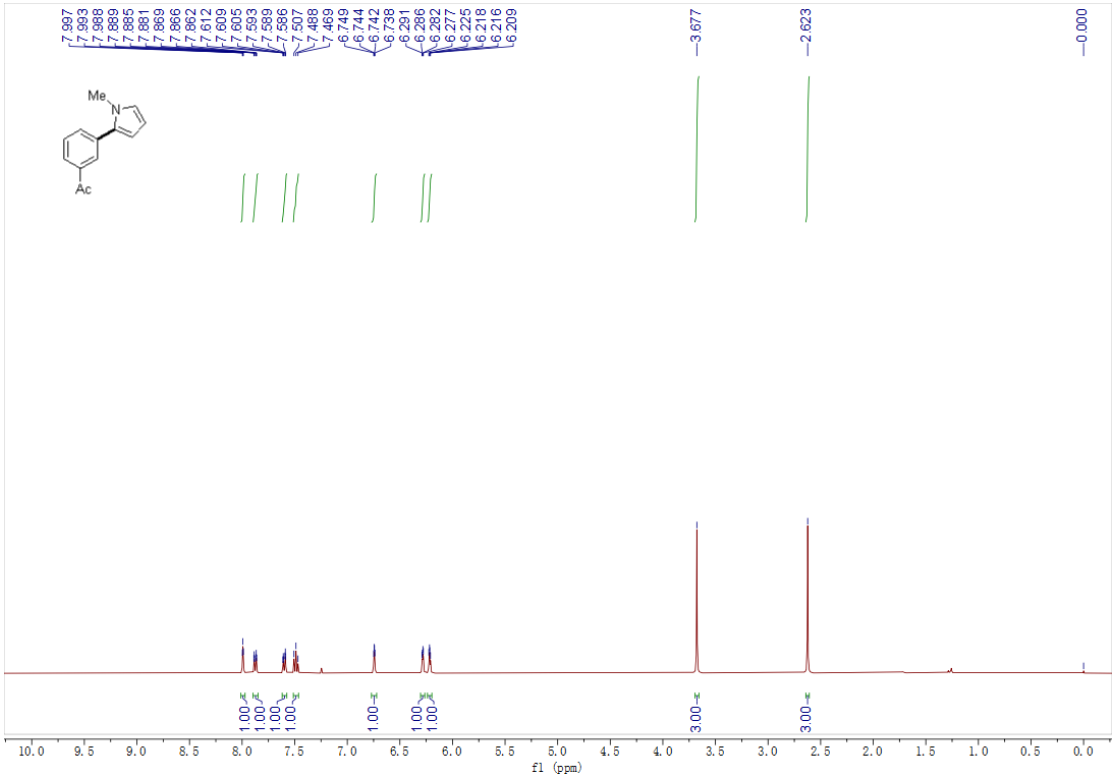
^{1}H NMR Spectrum of **ITN-1**



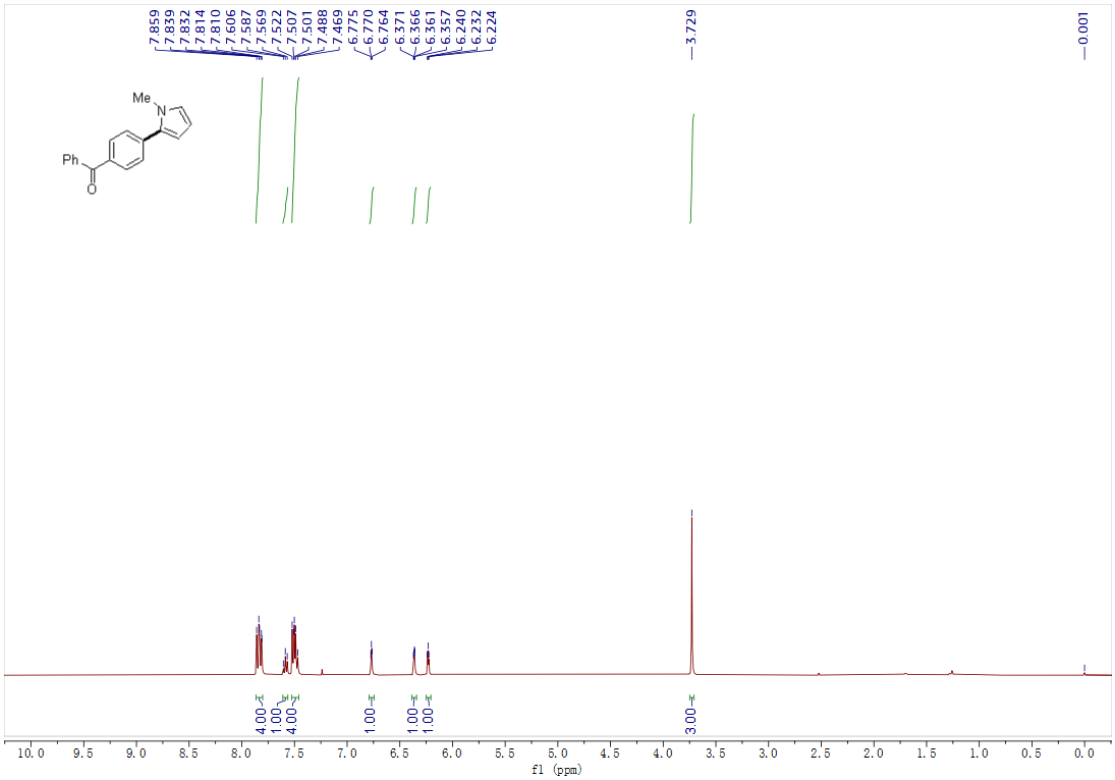
^{1}H NMR Spectrum of **3a**



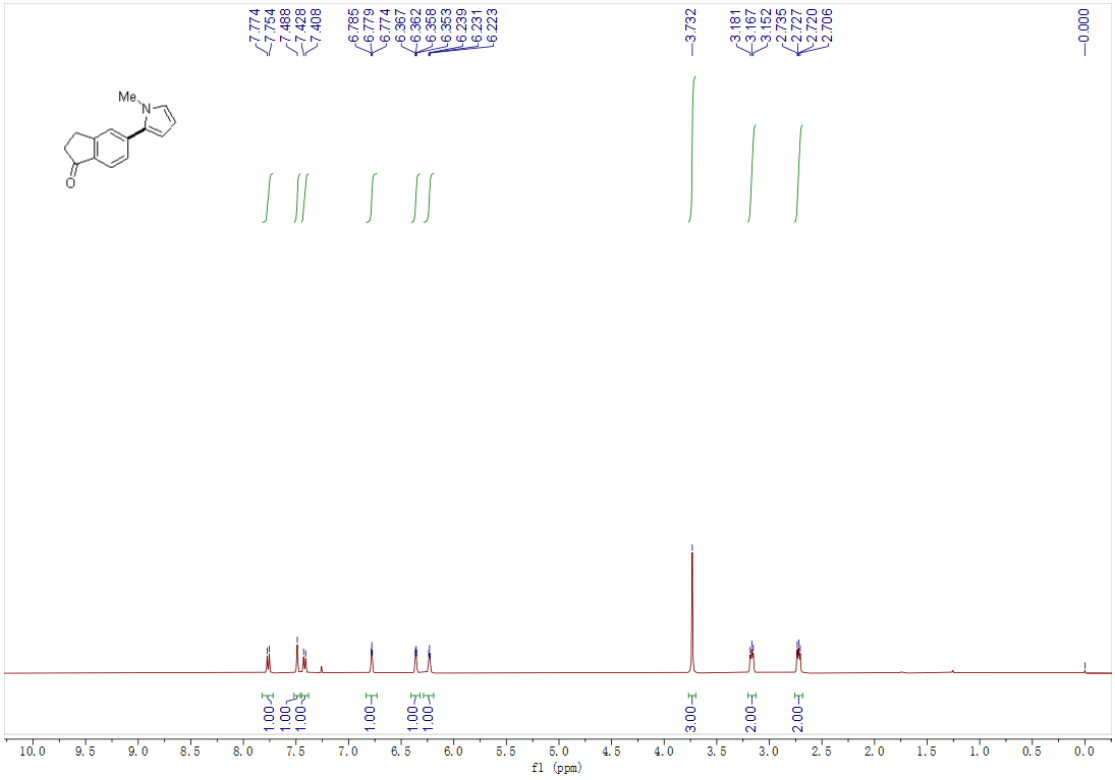
^{1}H NMR Spectrum of **3b**



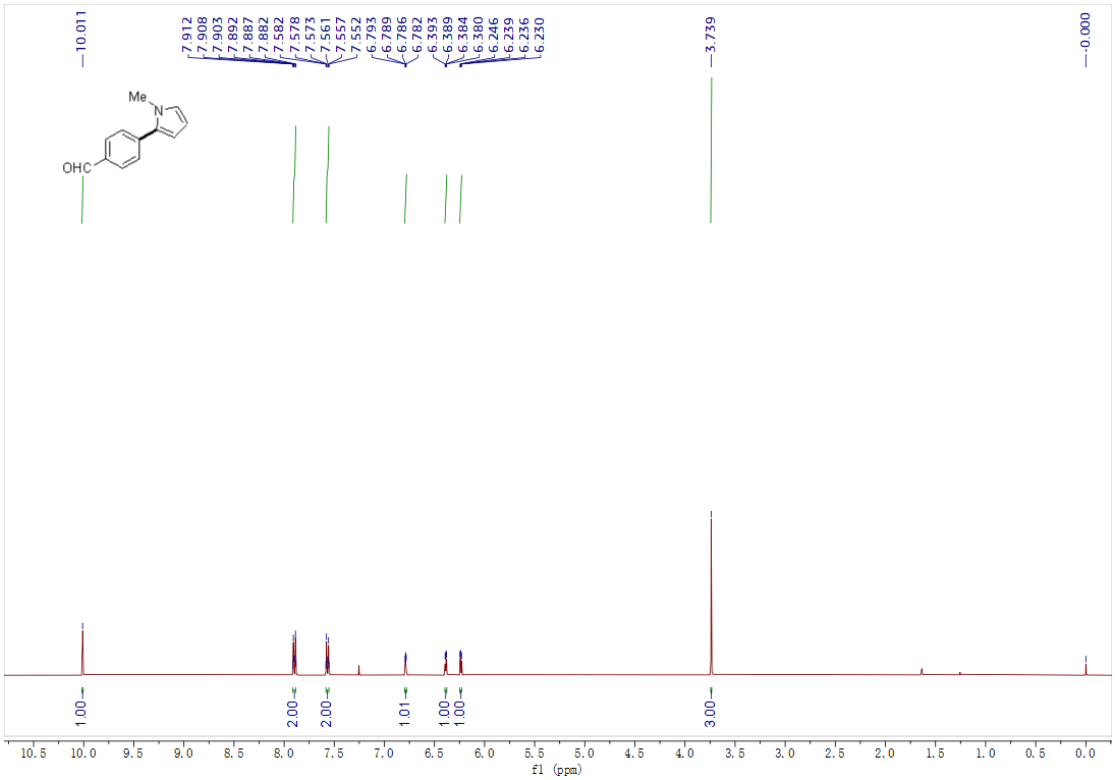
^{1}H NMR Spectrum of **3c**



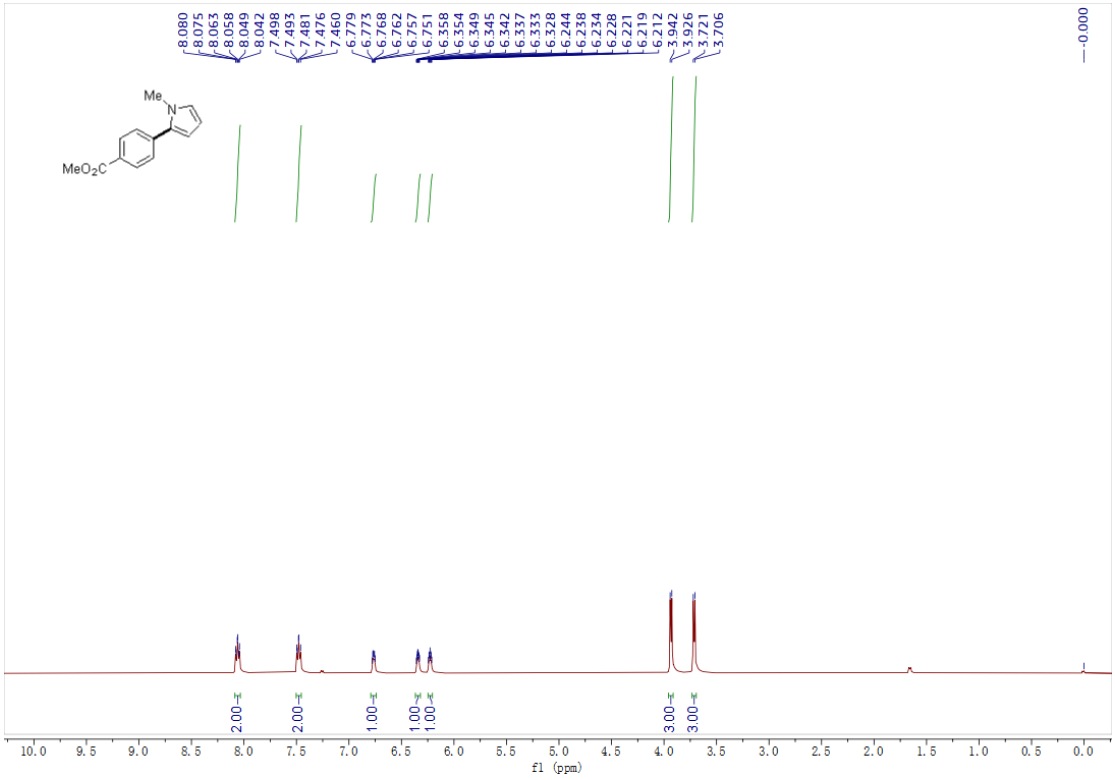
^{1}H NMR Spectrum of **3d**



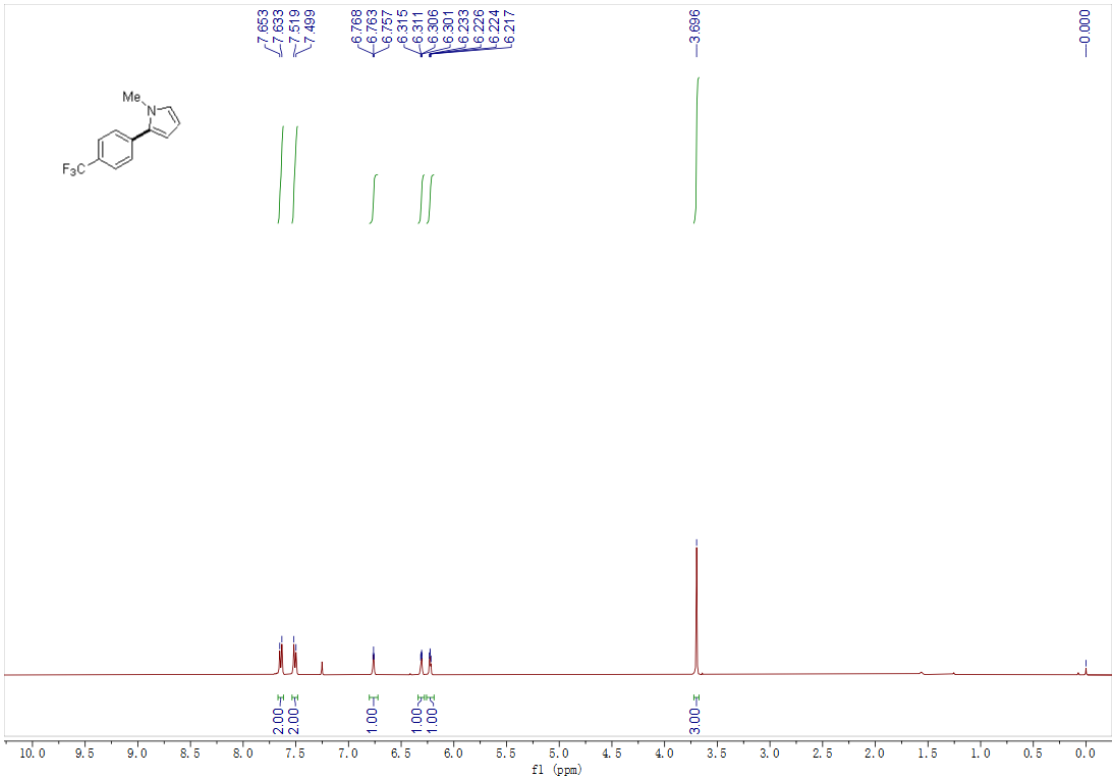
^{1}H NMR Spectrum of **3e**



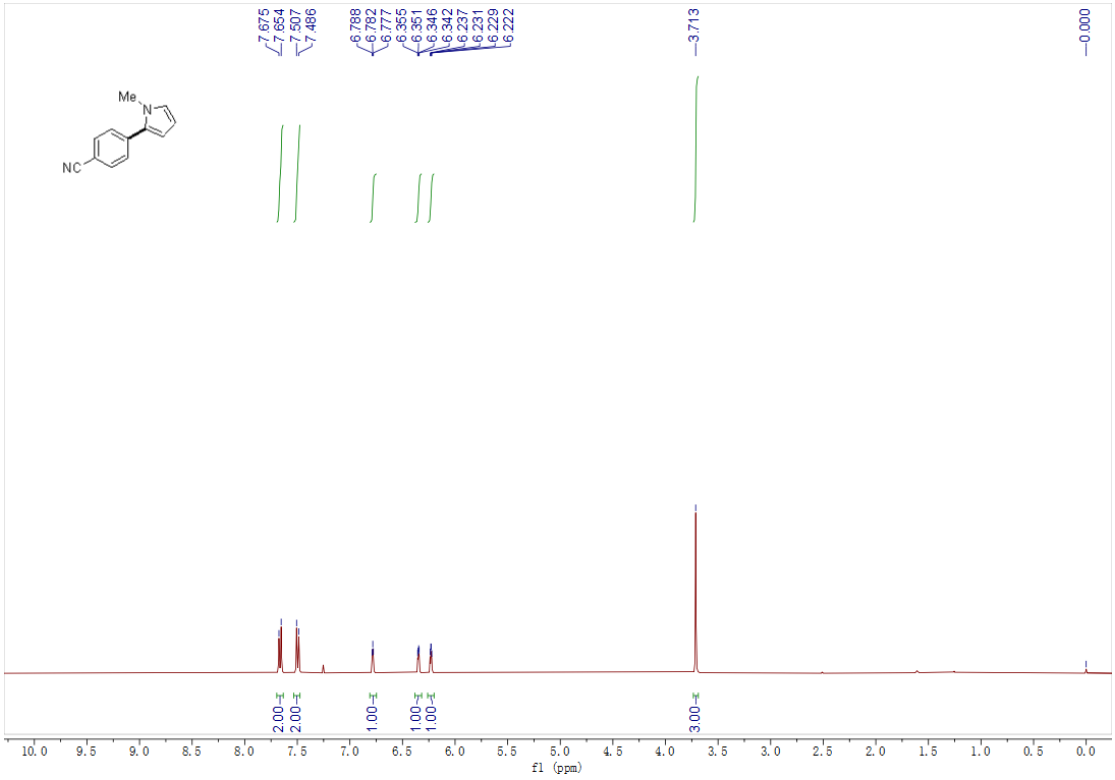
^{1}H NMR Spectrum of **3f**



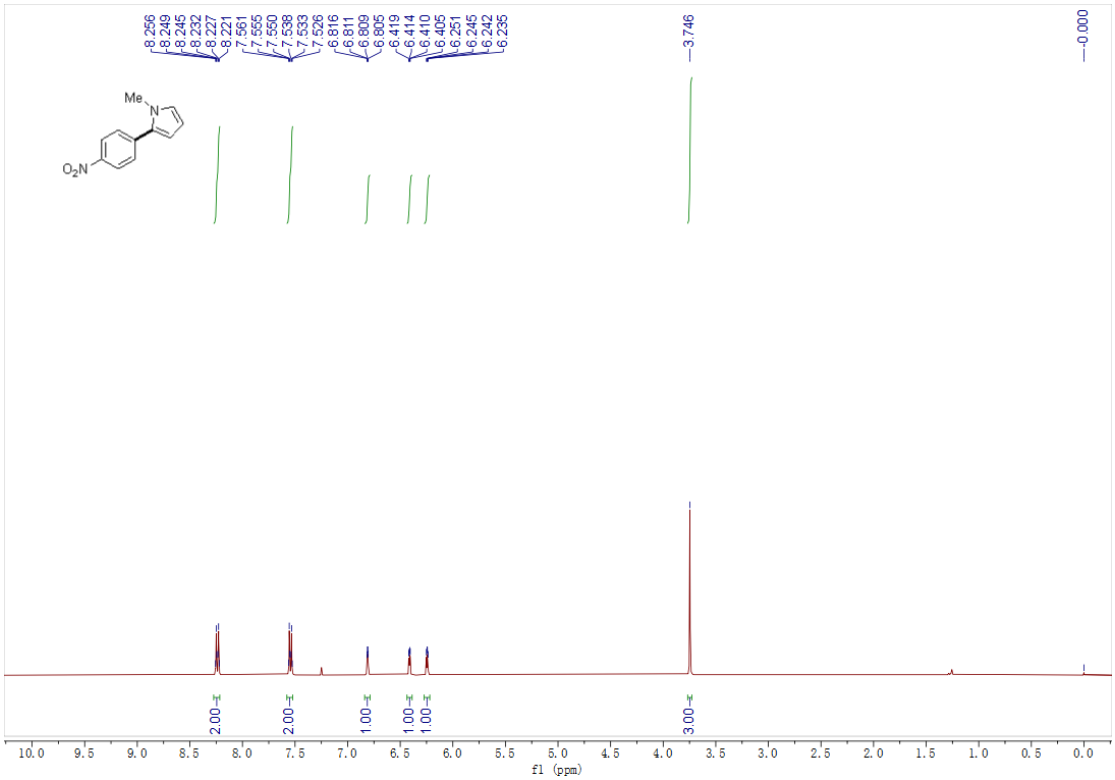
^{1}H NMR Spectrum of **3g**



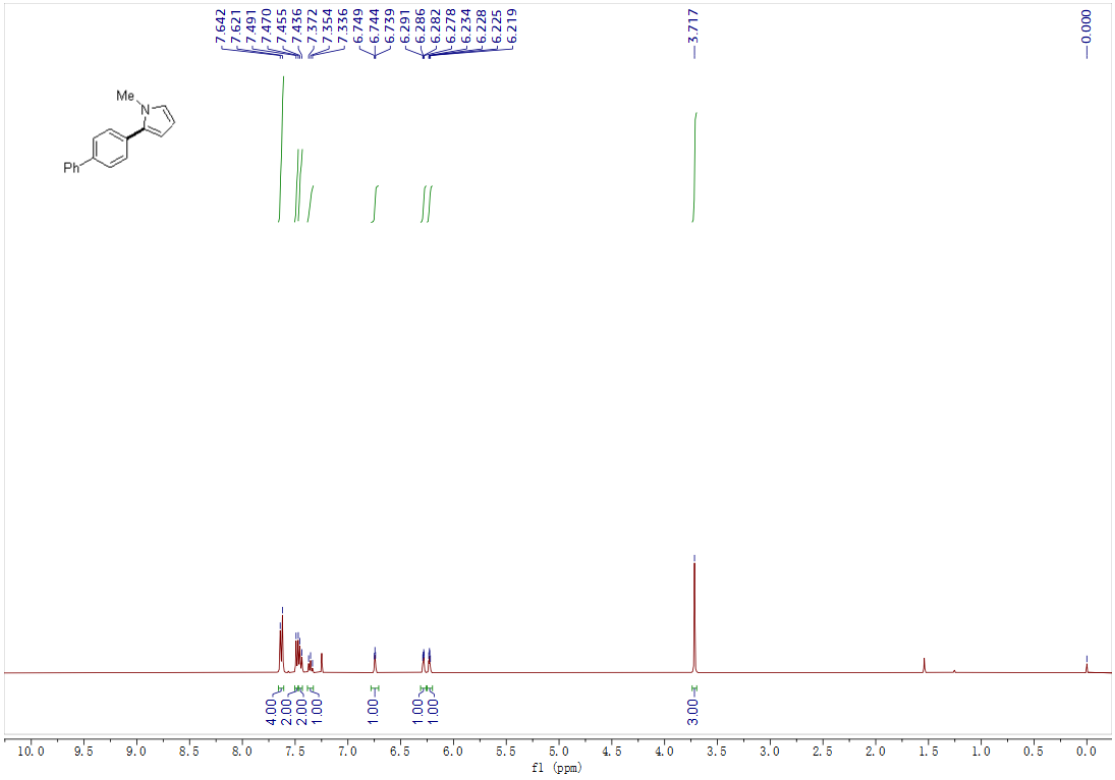
^{1}H NMR Spectrum of **3h**



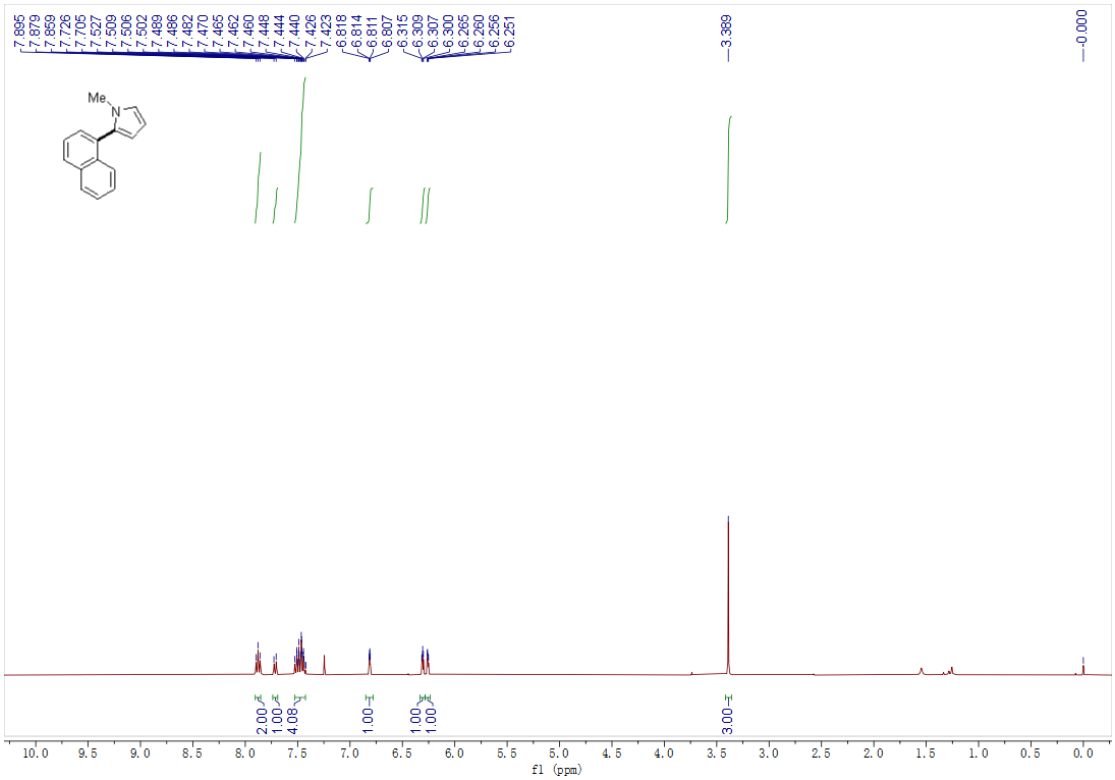
^{1}H NMR Spectrum of **3i**



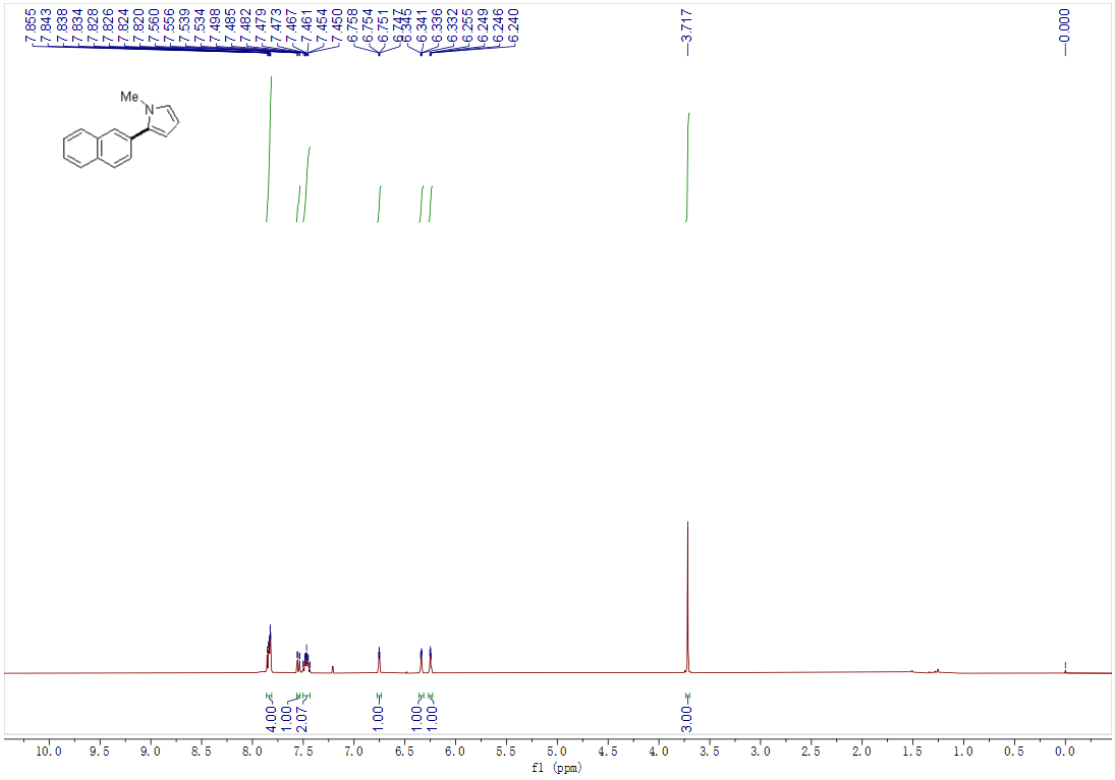
^{1}H NMR Spectrum of **3j**



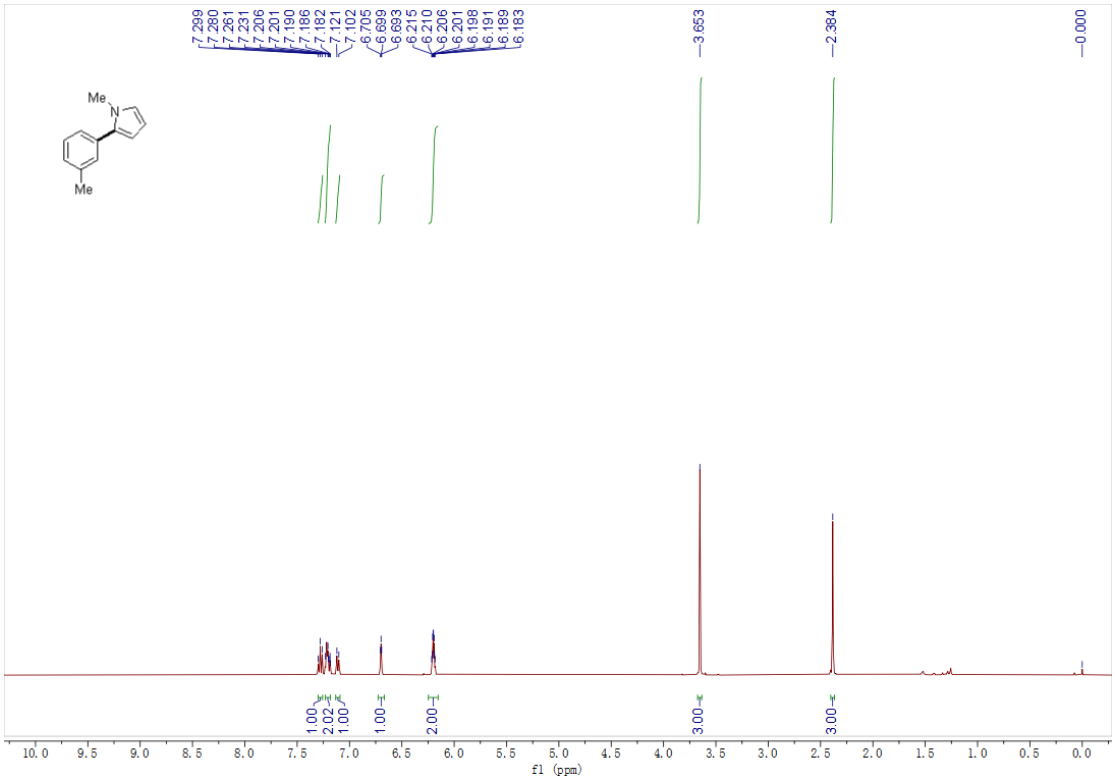
^{1}H NMR Spectrum of **3k**



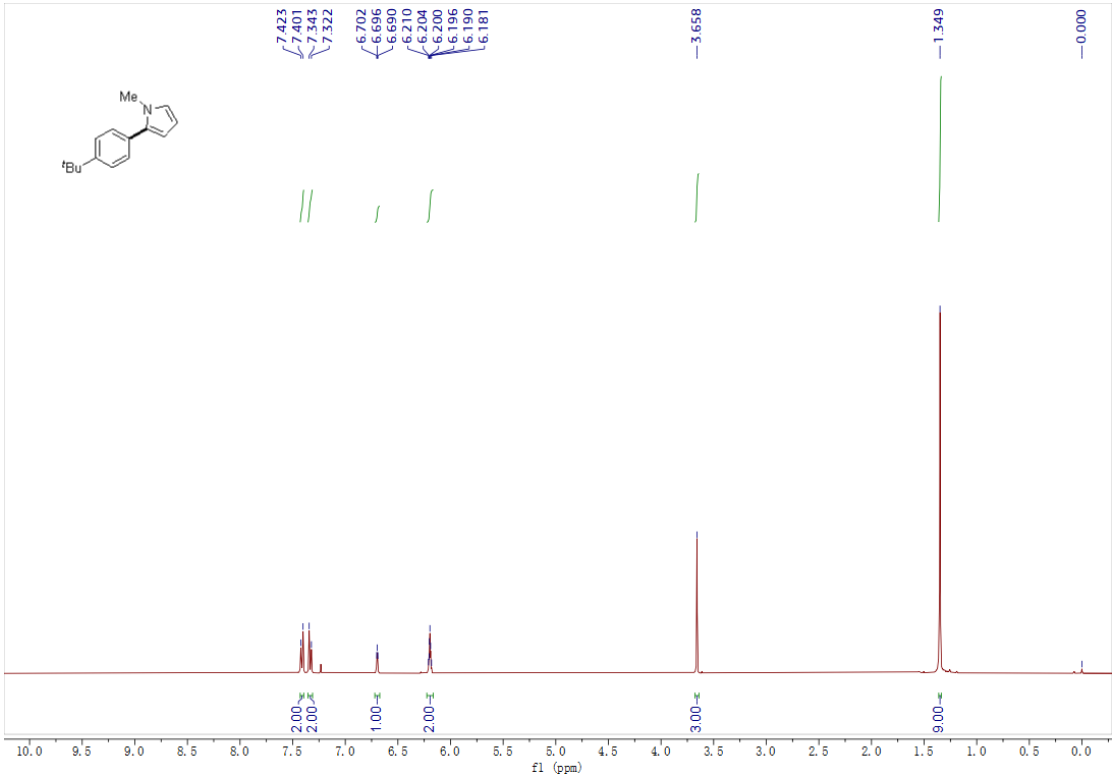
^{1}H NMR Spectrum of **3l**



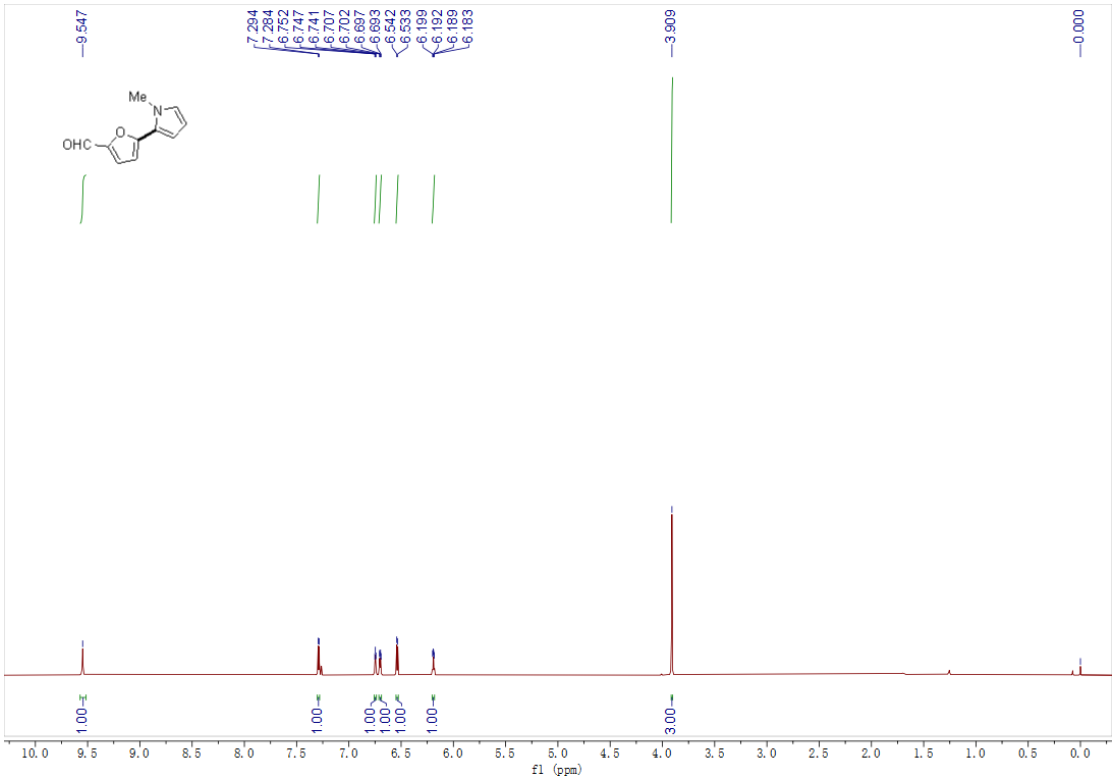
^{1}H NMR Spectrum of **3m**



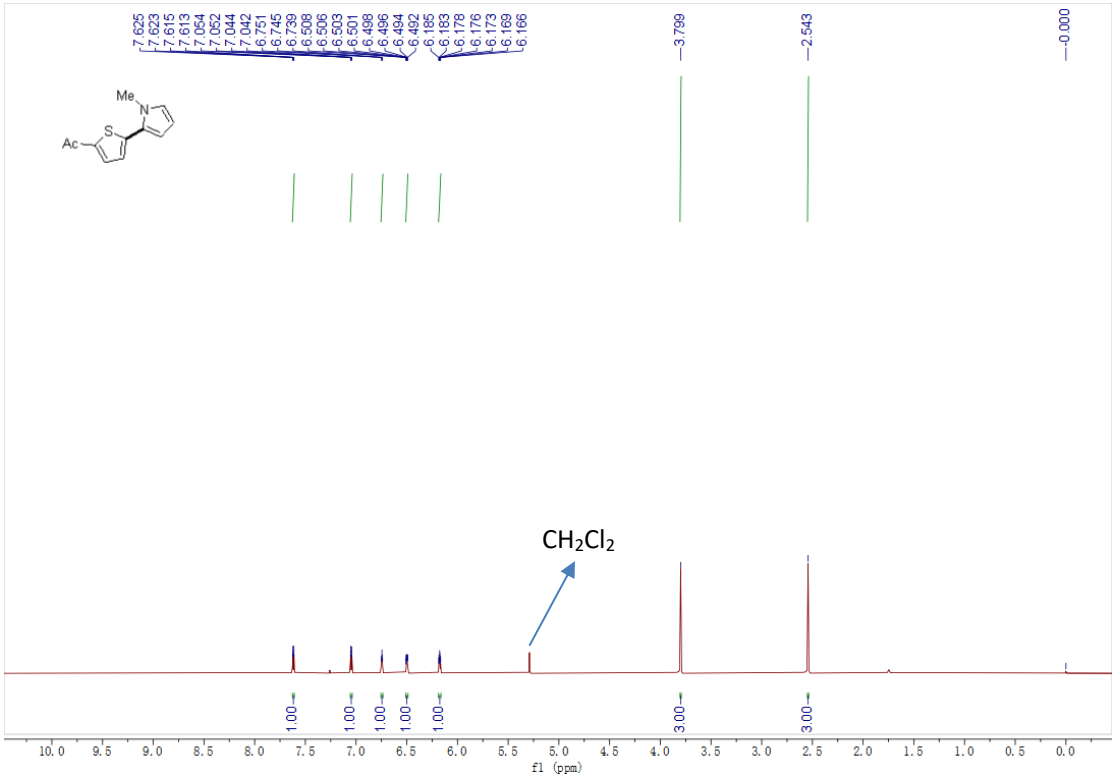
^{1}H NMR Spectrum of **3n**



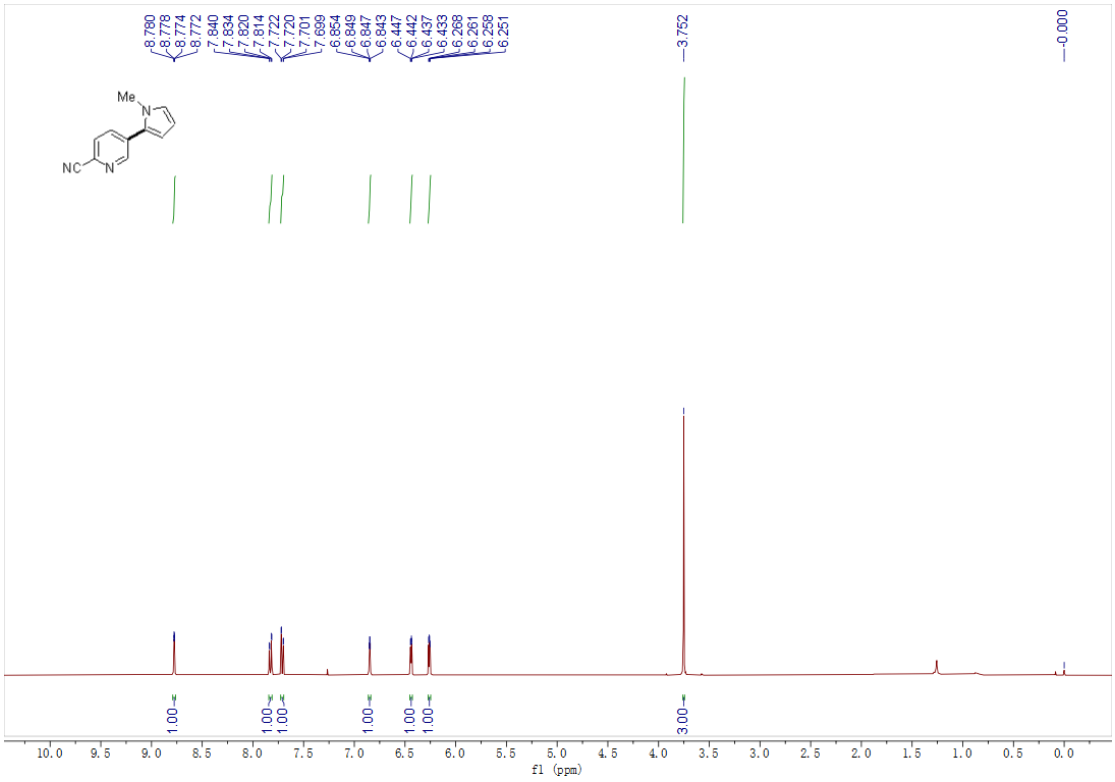
^{1}H NMR Spectrum of **3o**



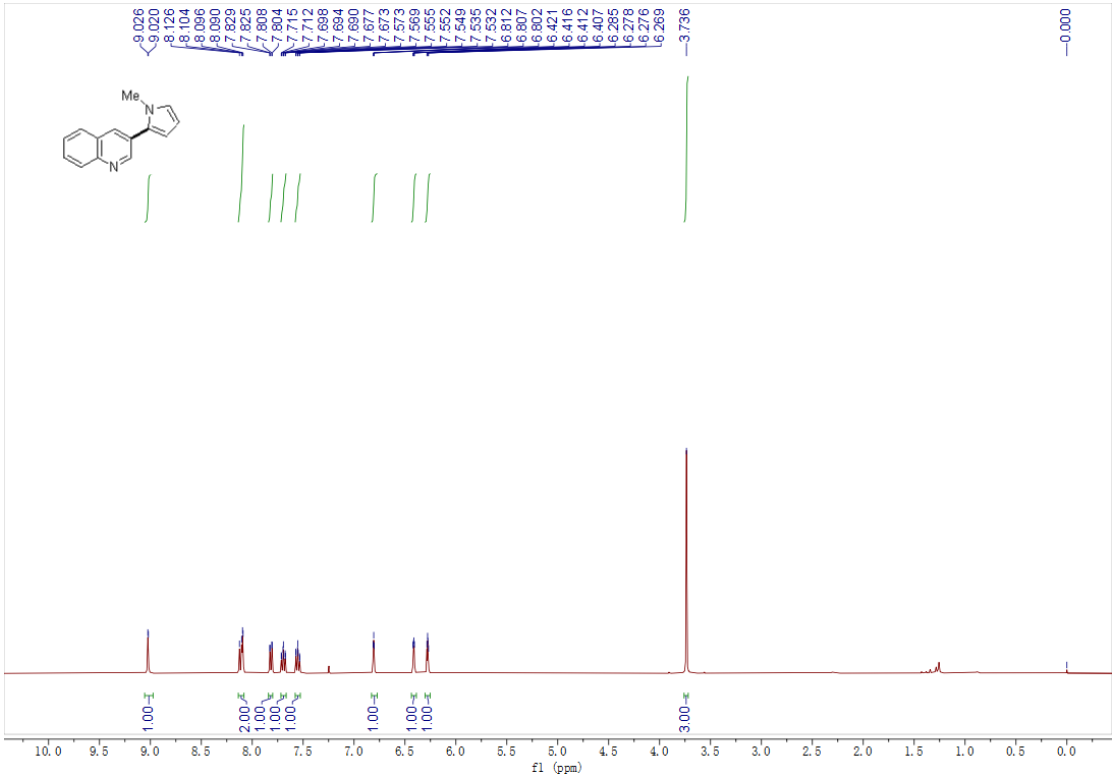
^{1}H NMR Spectrum of **3p**



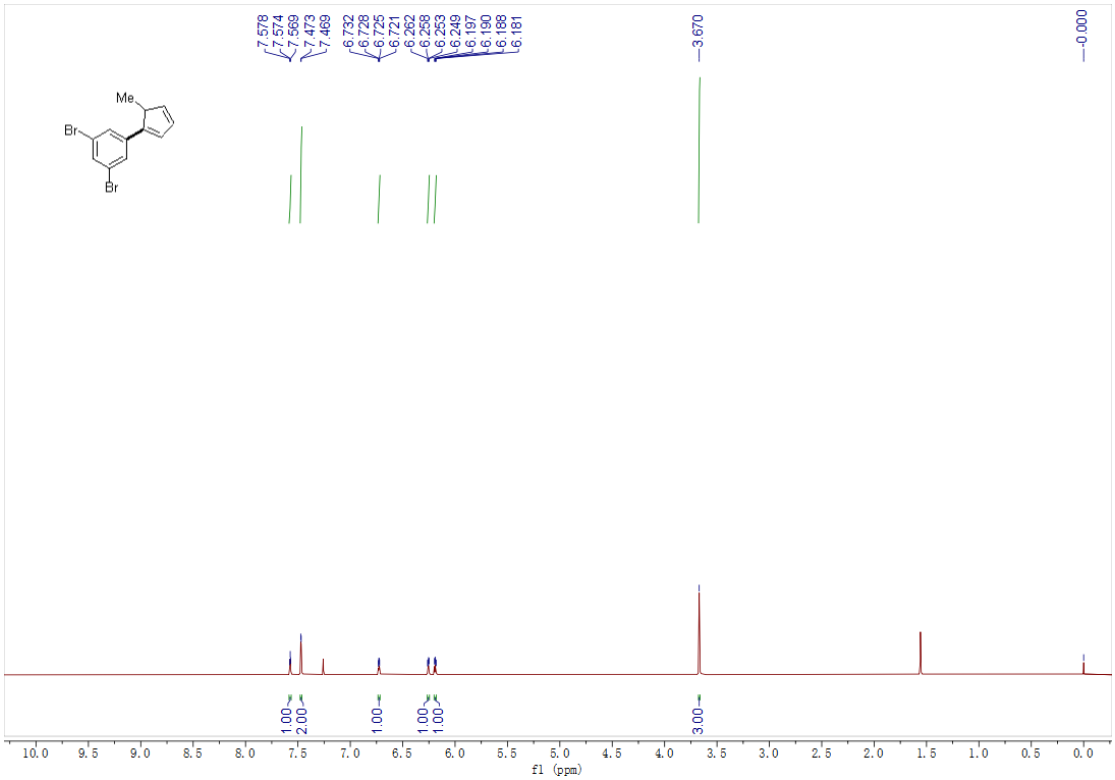
^{1}H NMR Spectrum of **3q**



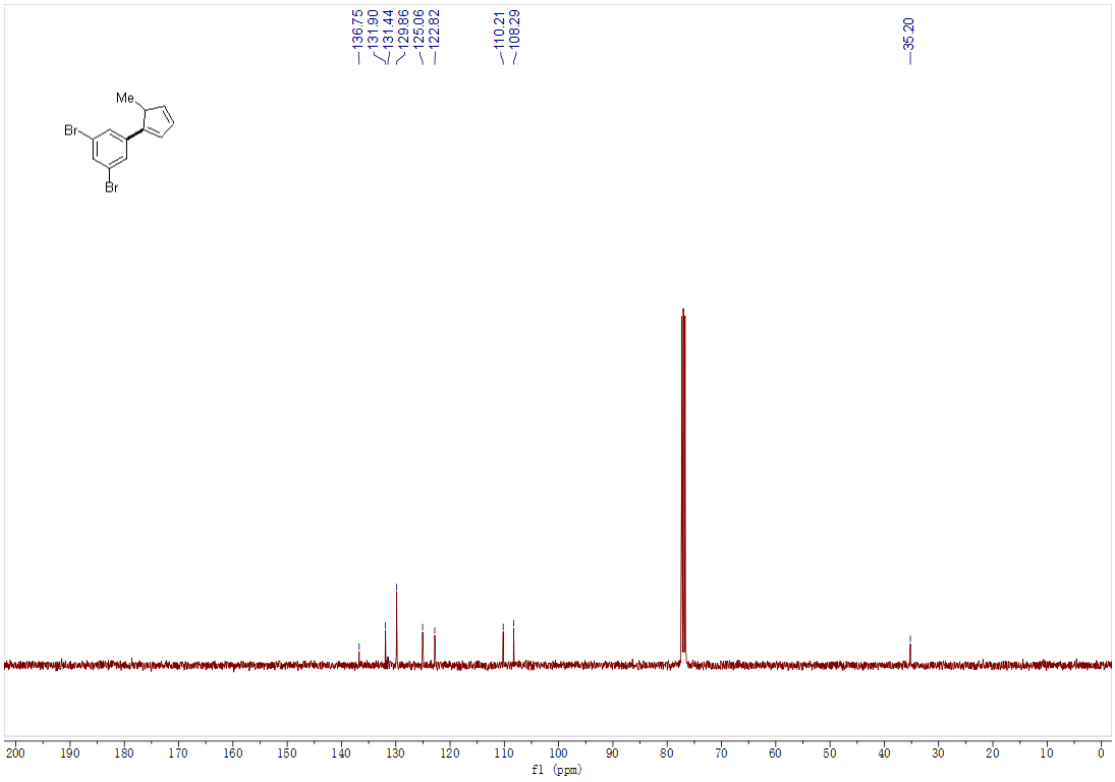
^{1}H NMR Spectrum of **3r**



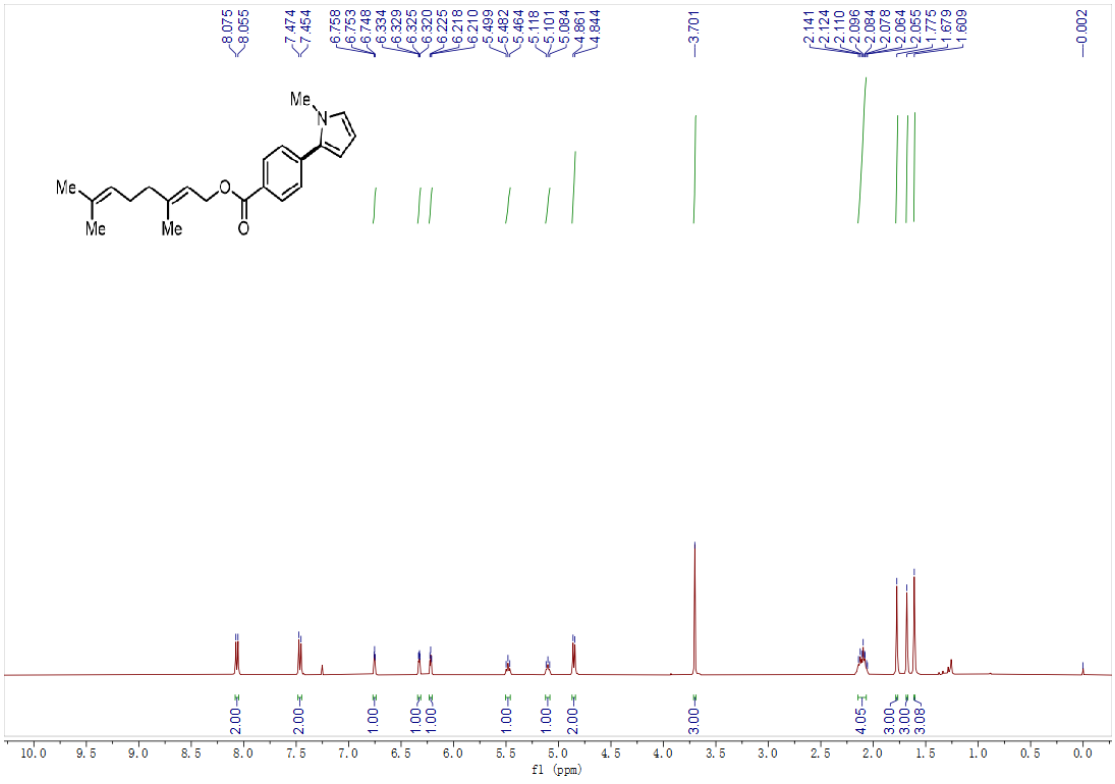
^{1}H NMR Spectrum of **3s**



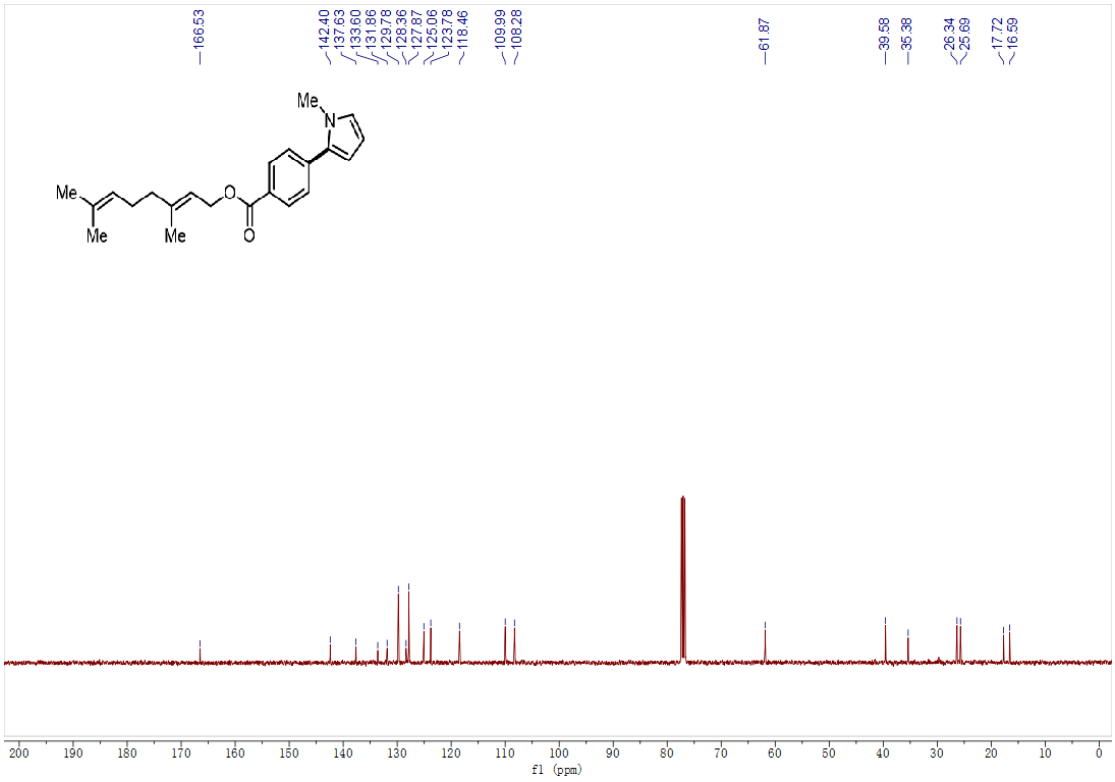
^{13}C NMR Spectrum of **3s**



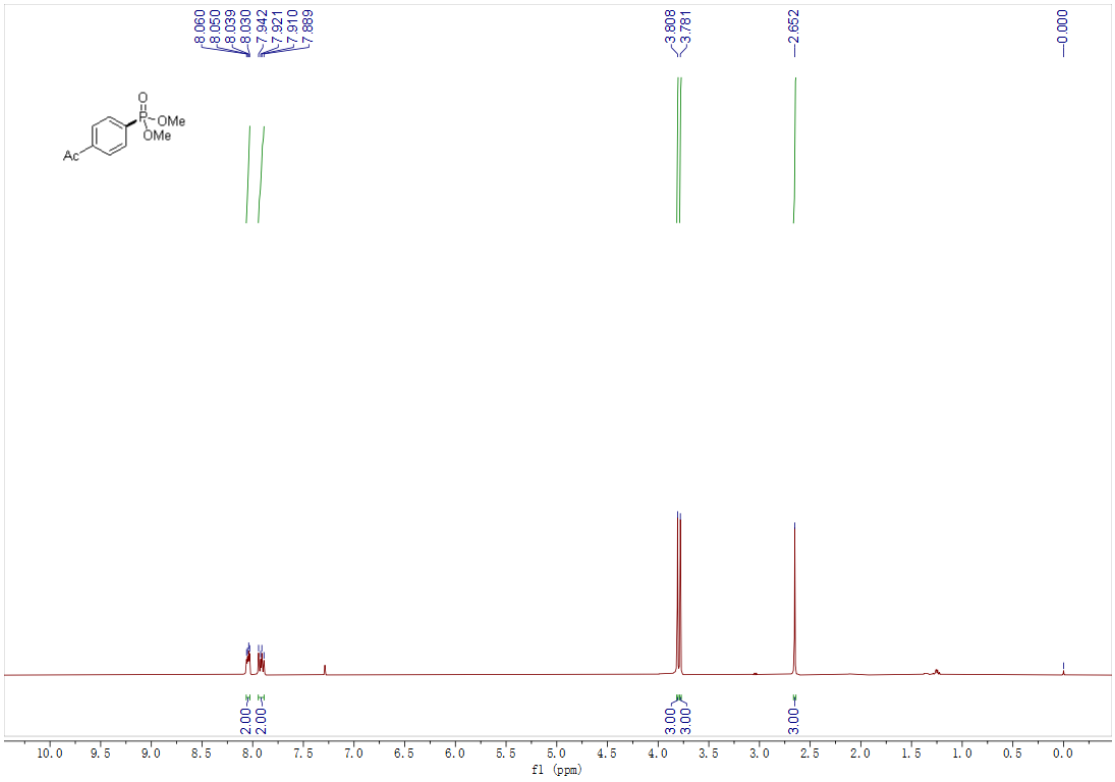
^{1}H NMR Spectrum of **3t**



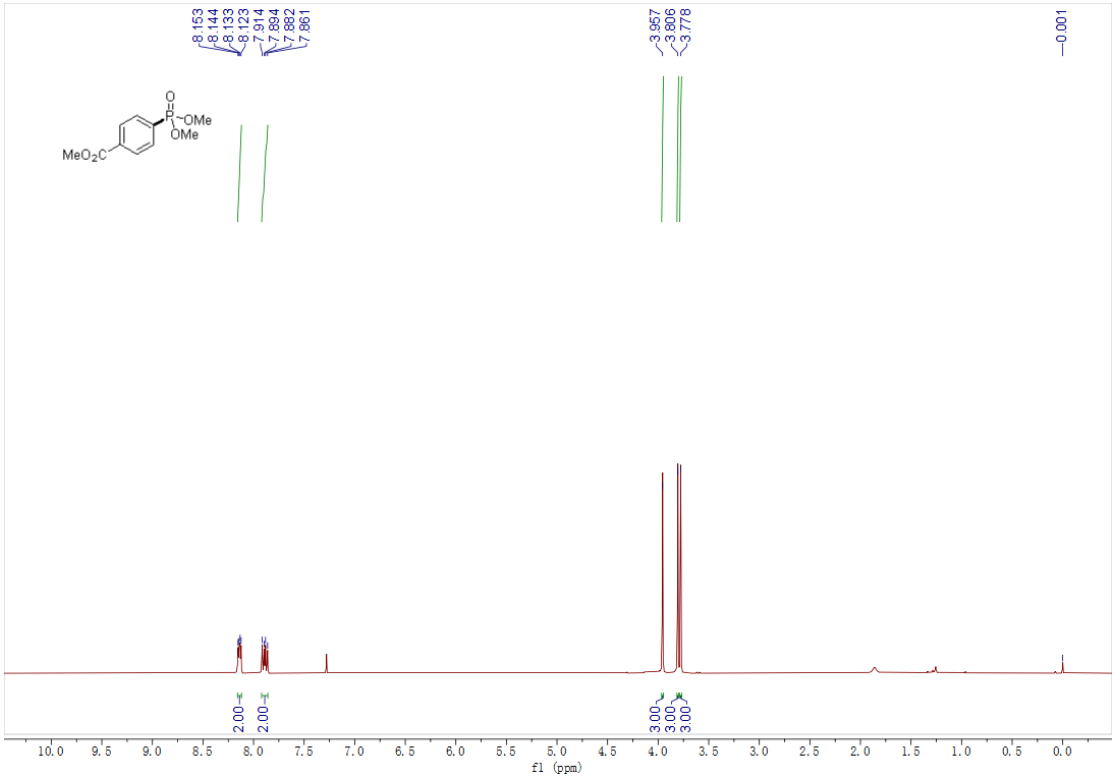
^{13}C NMR Spectrum of **3t**



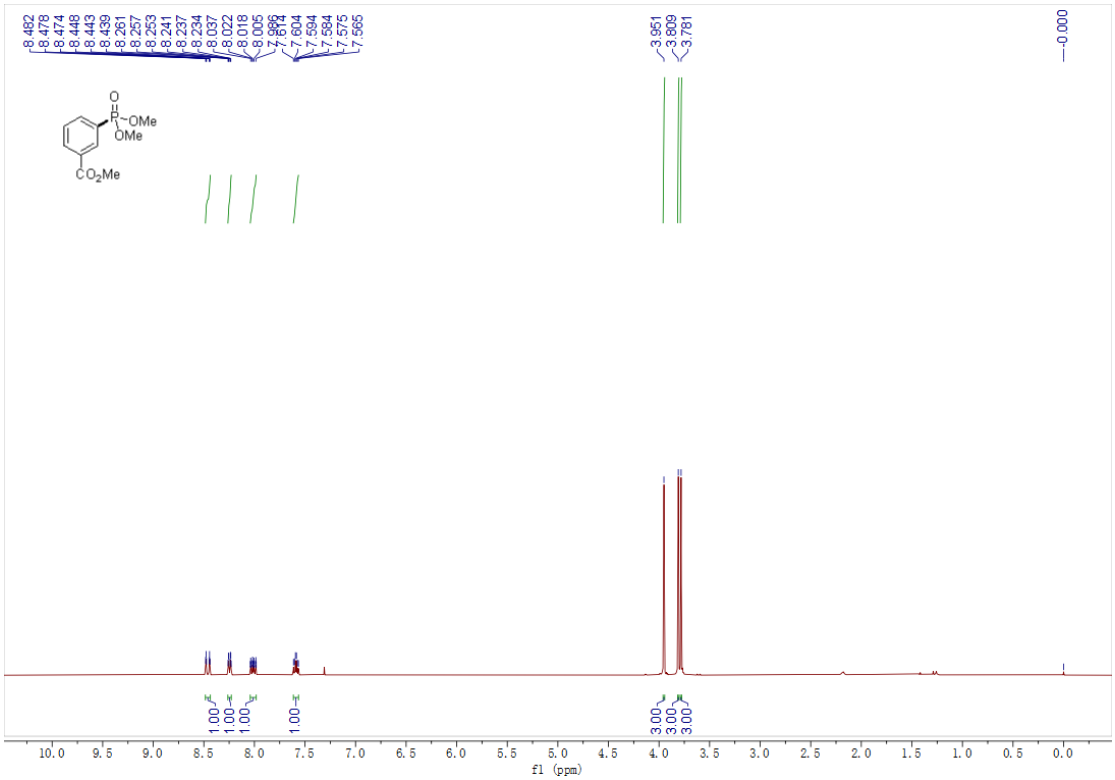
^{1}H NMR Spectrum of **5a**



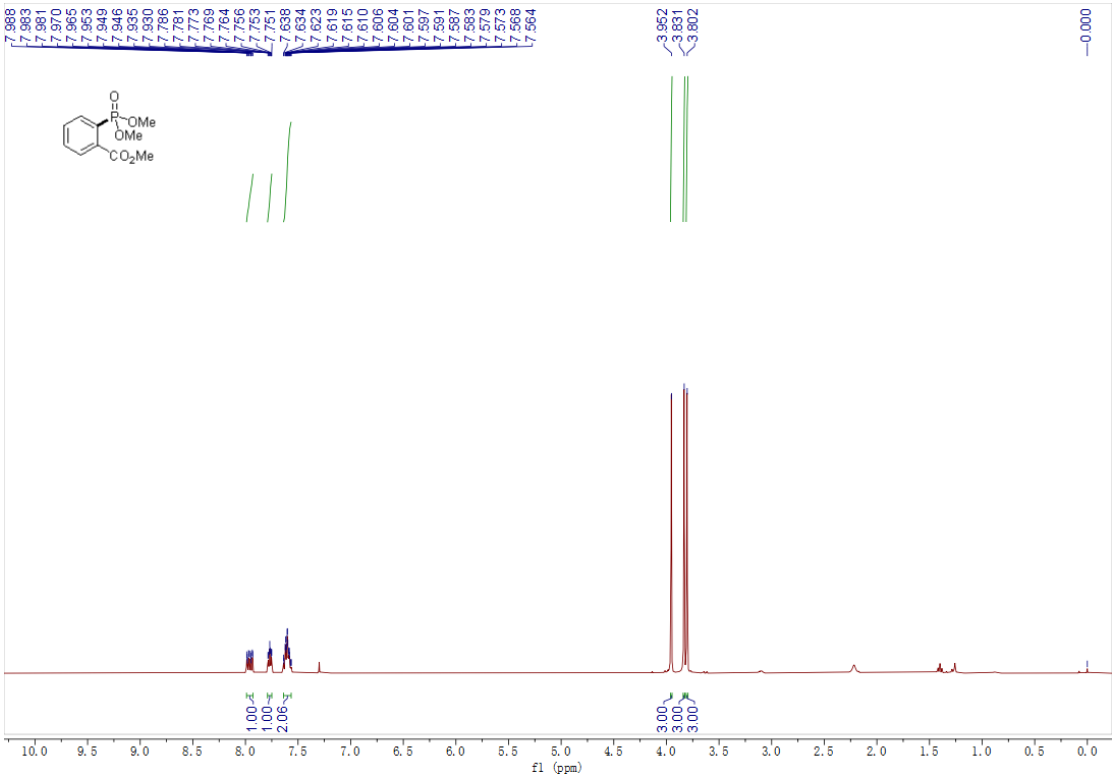
^{1}H NMR Spectrum of **5b**



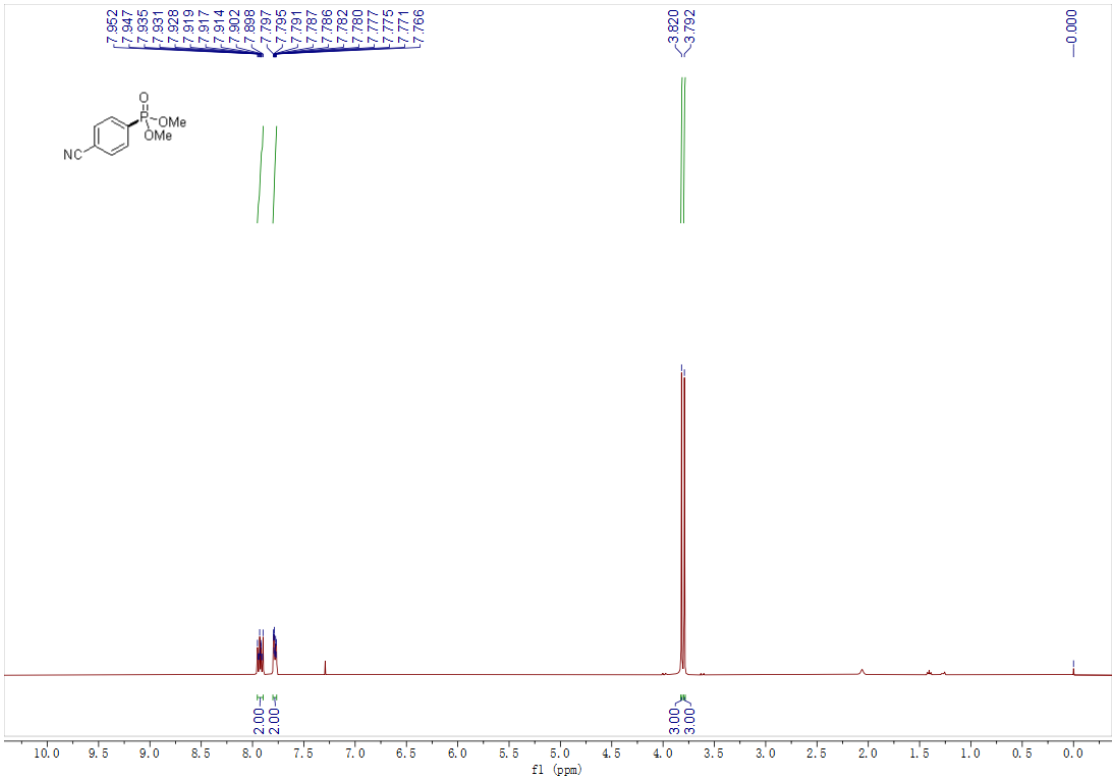
^{1}H NMR Spectrum of **5c**



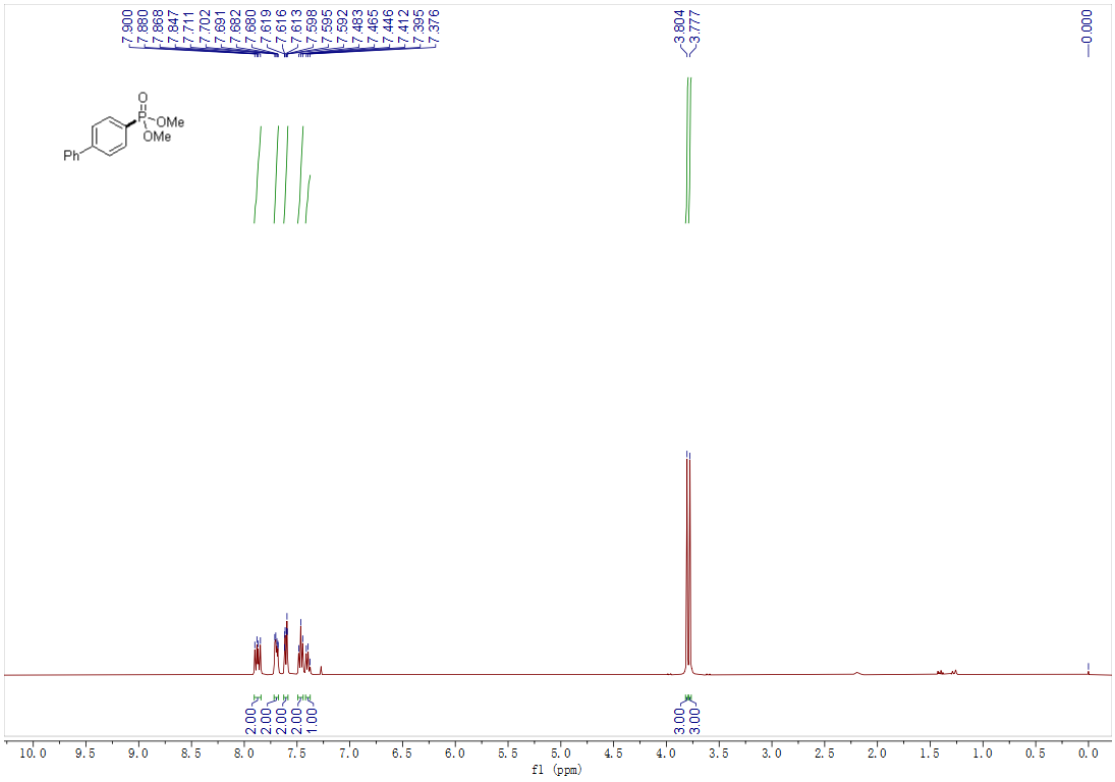
^{1}H NMR Spectrum of **5d**



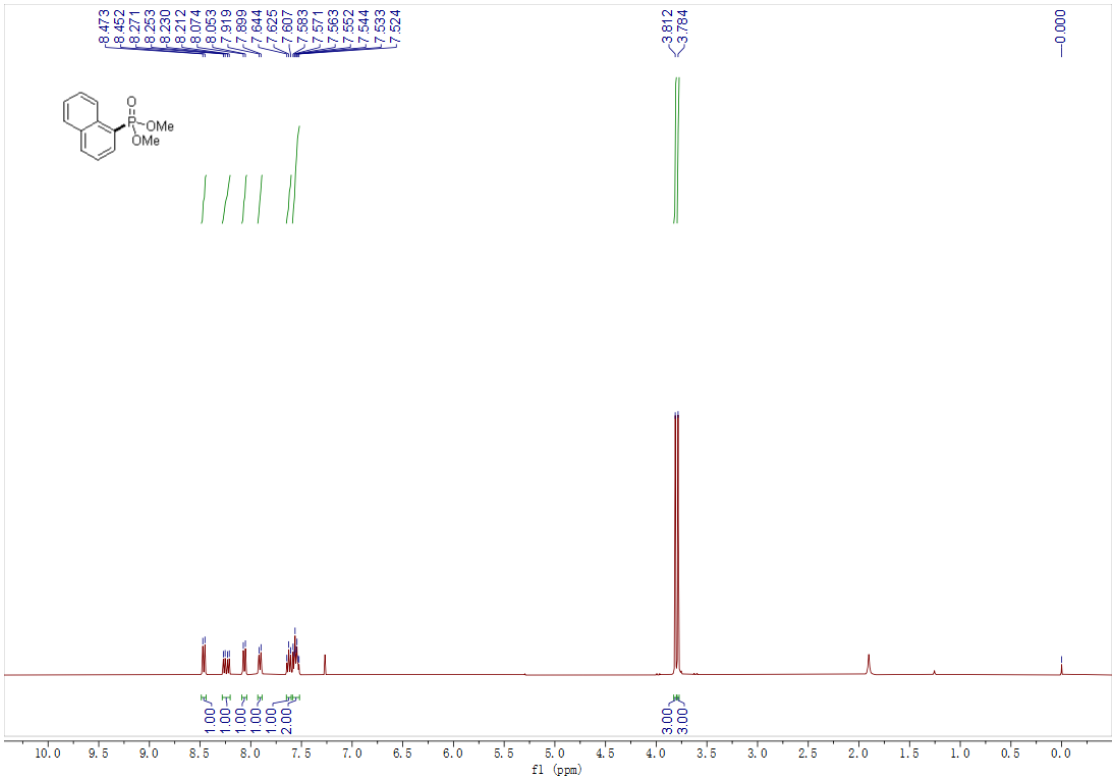
^{1}H NMR Spectrum of **5e**



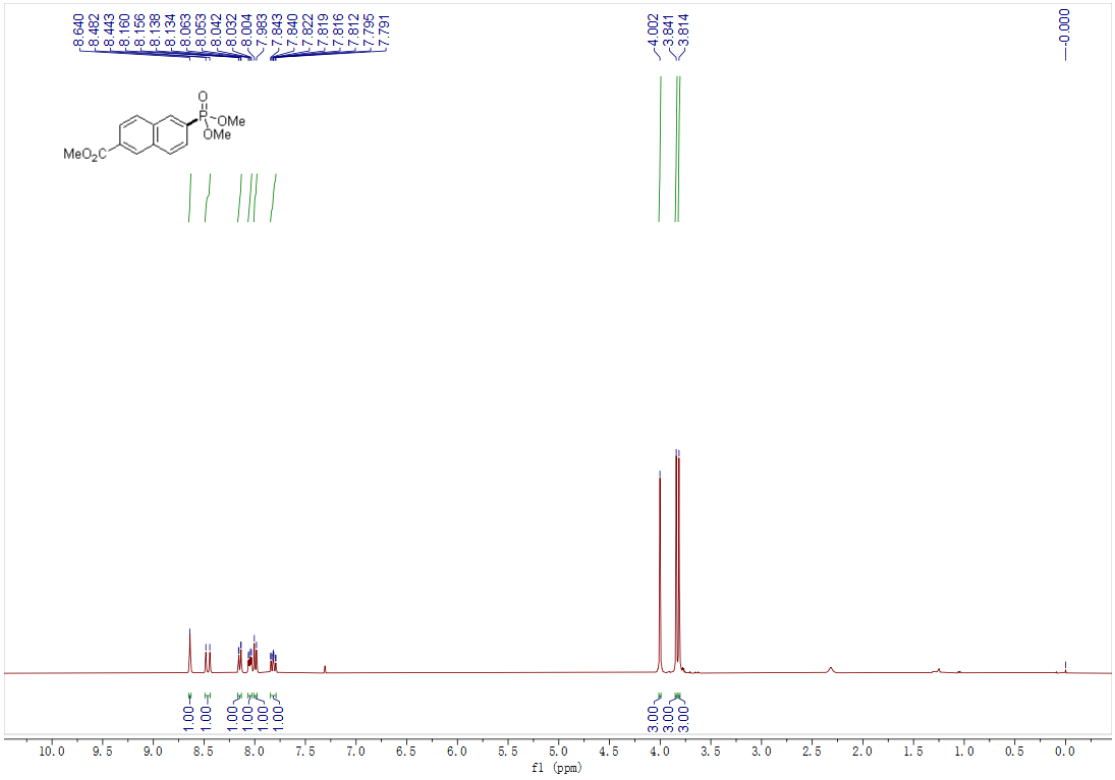
^{1}H NMR Spectrum of **5f**



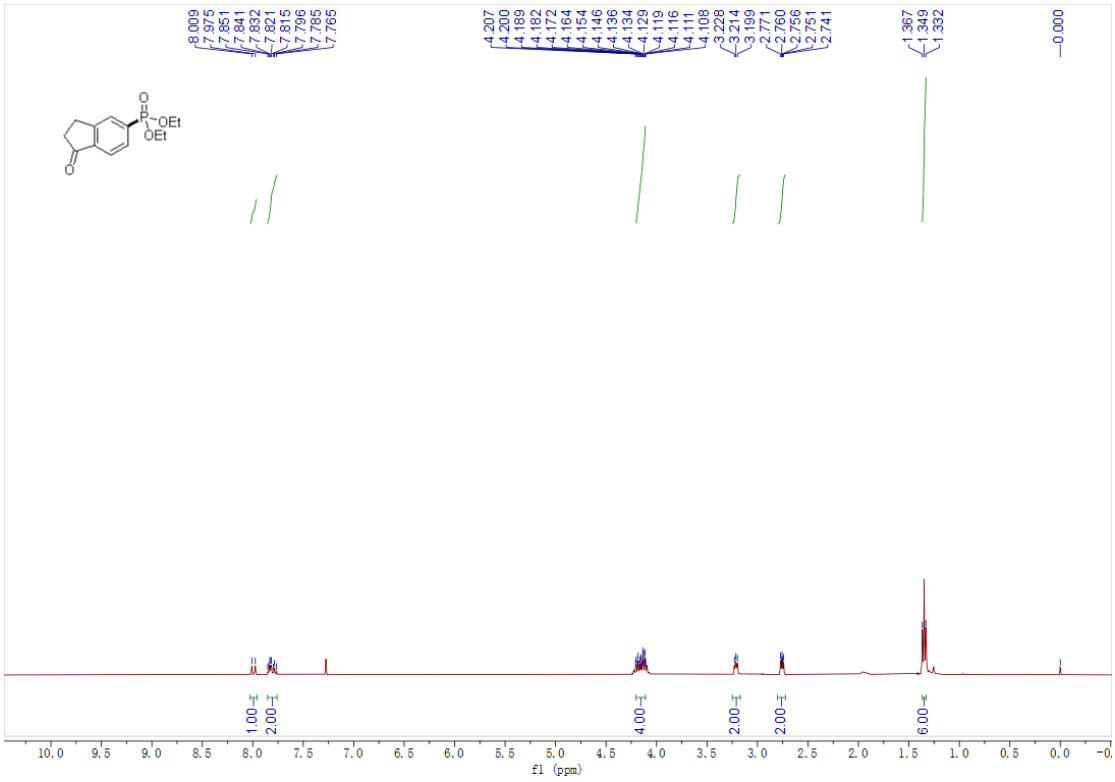
^{1}H NMR Spectrum of **5g**



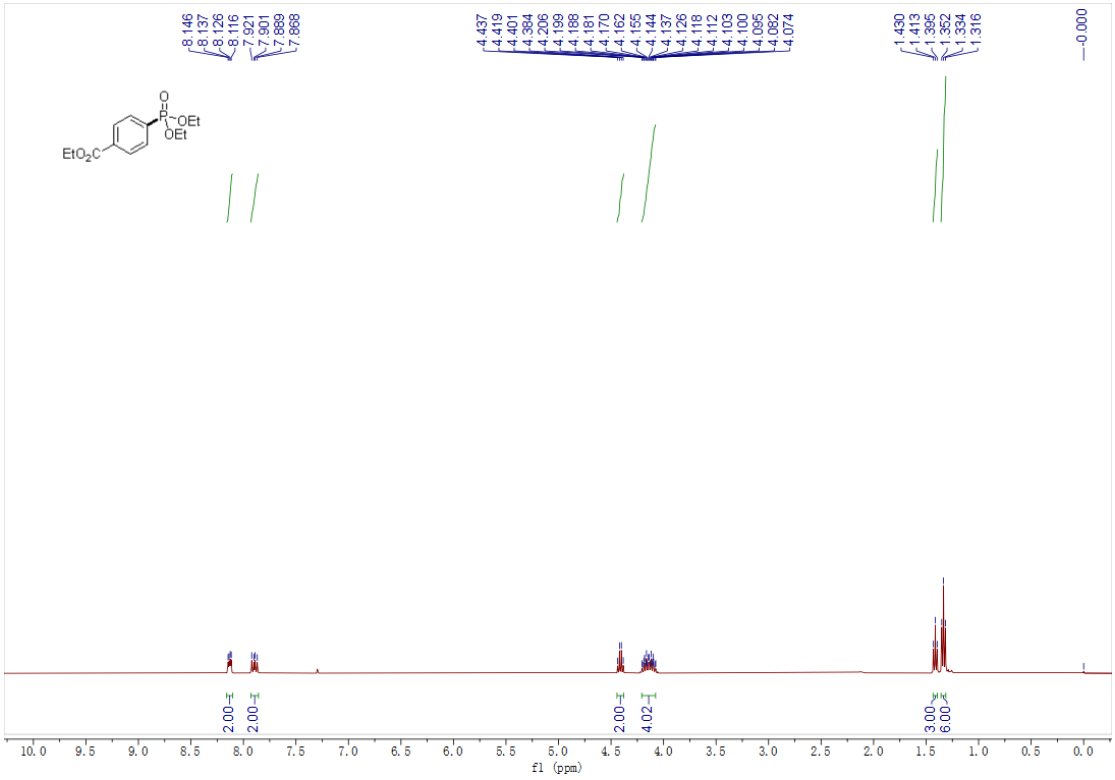
^{1}H NMR Spectrum of **5h**



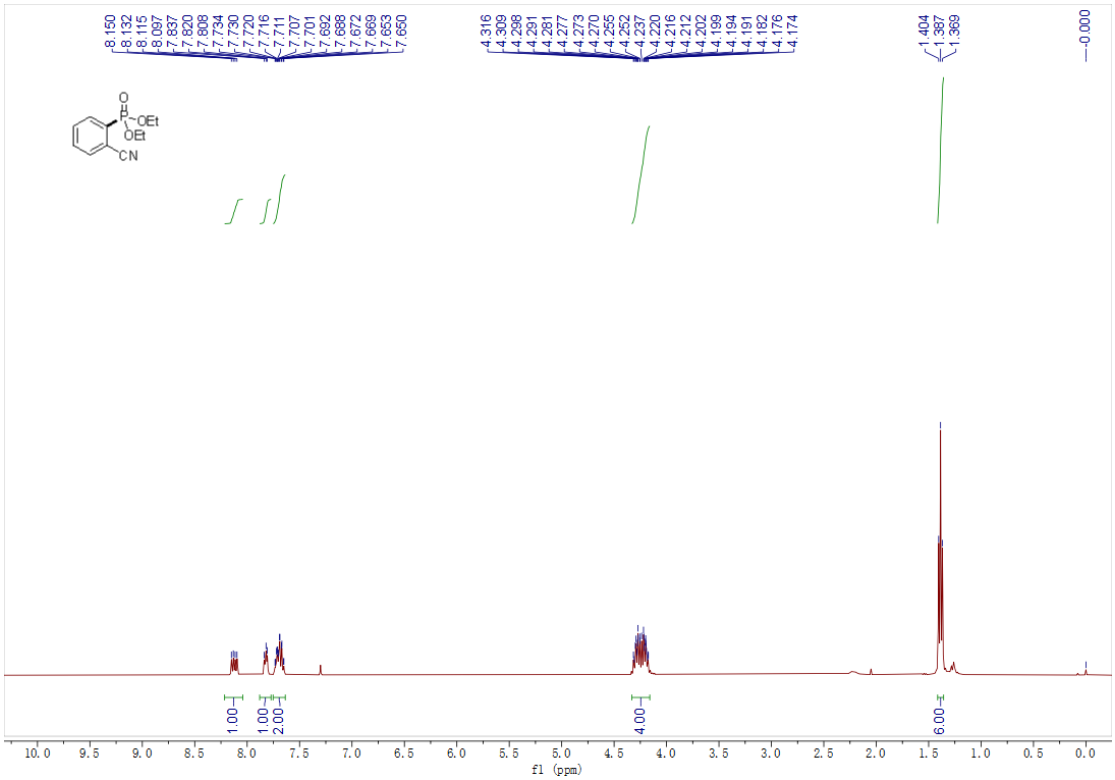
^{1}H NMR Spectrum of **5i**



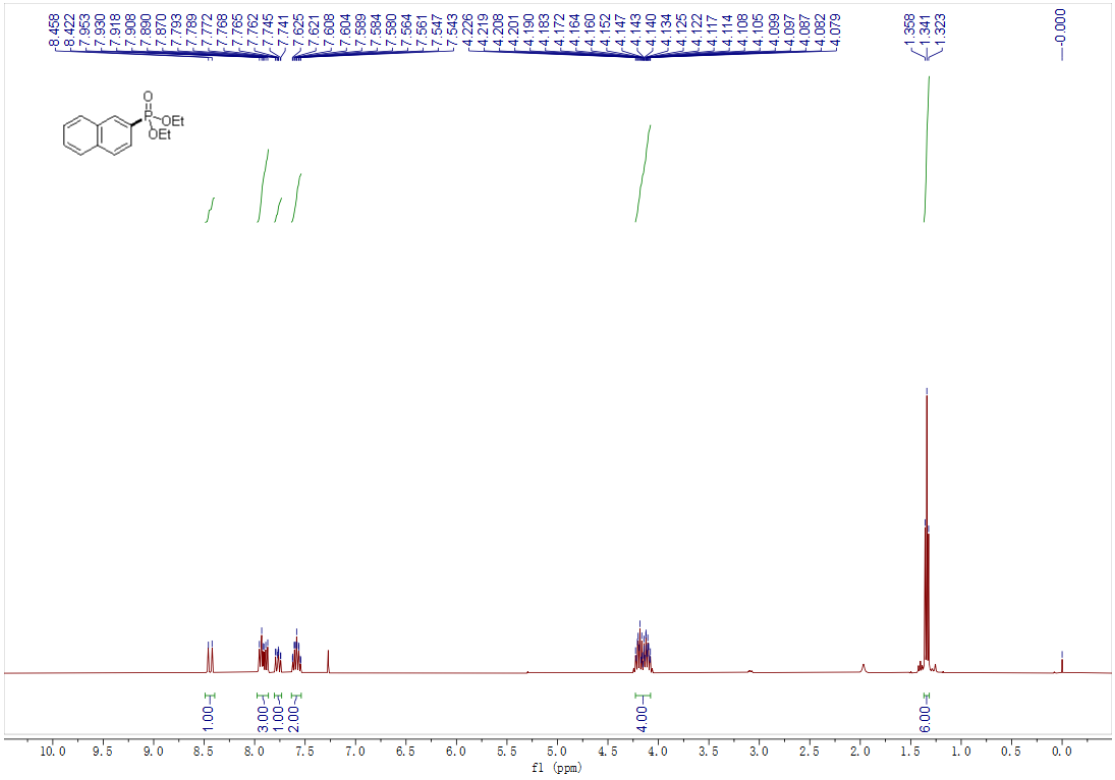
^{1}H NMR Spectrum of **5j**



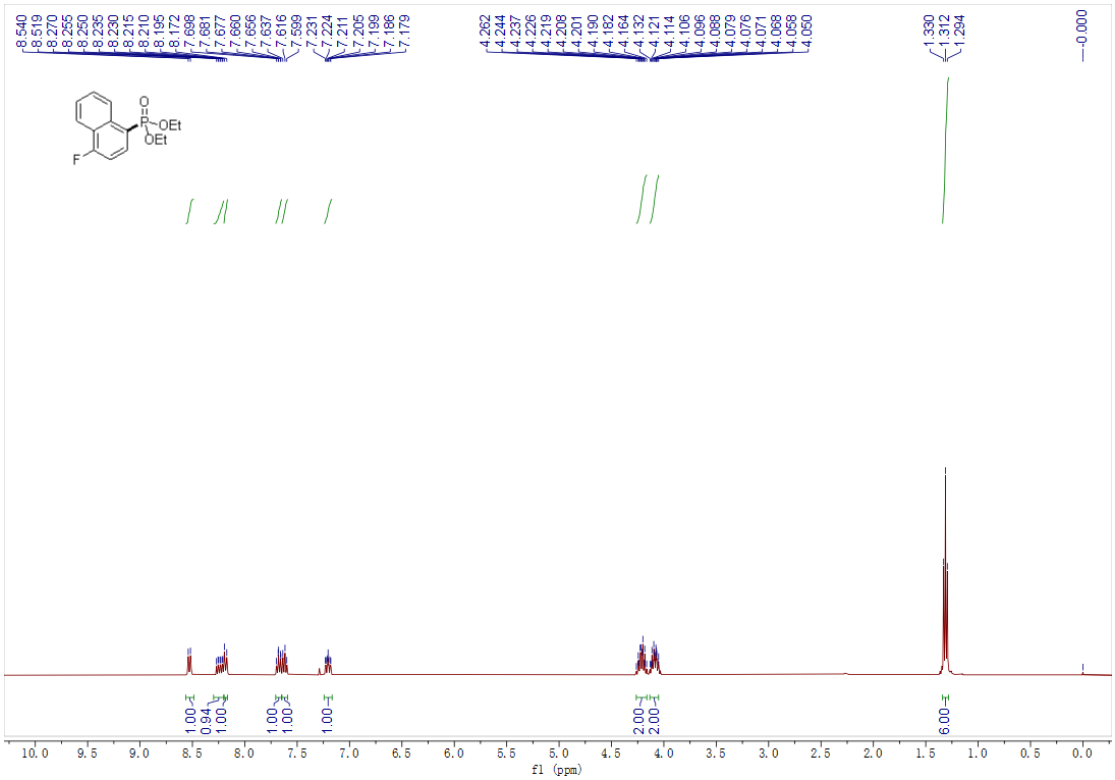
^{1}H NMR Spectrum of **5k**



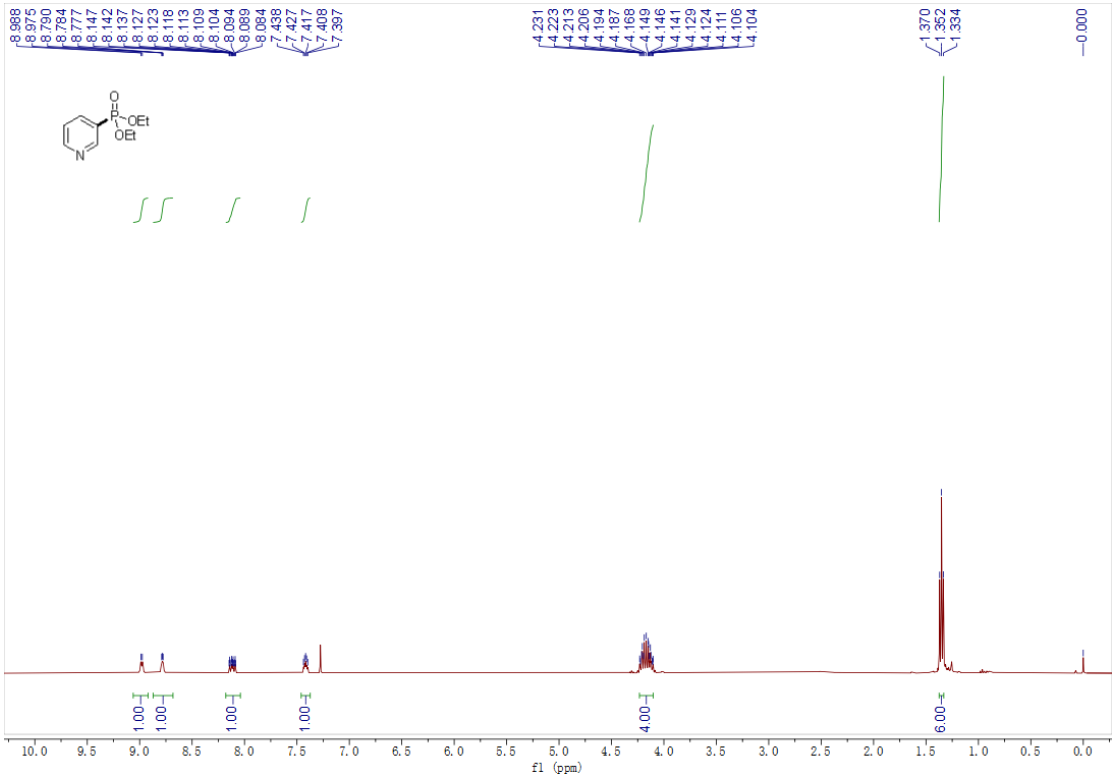
^{1}H NMR Spectrum of **5l**



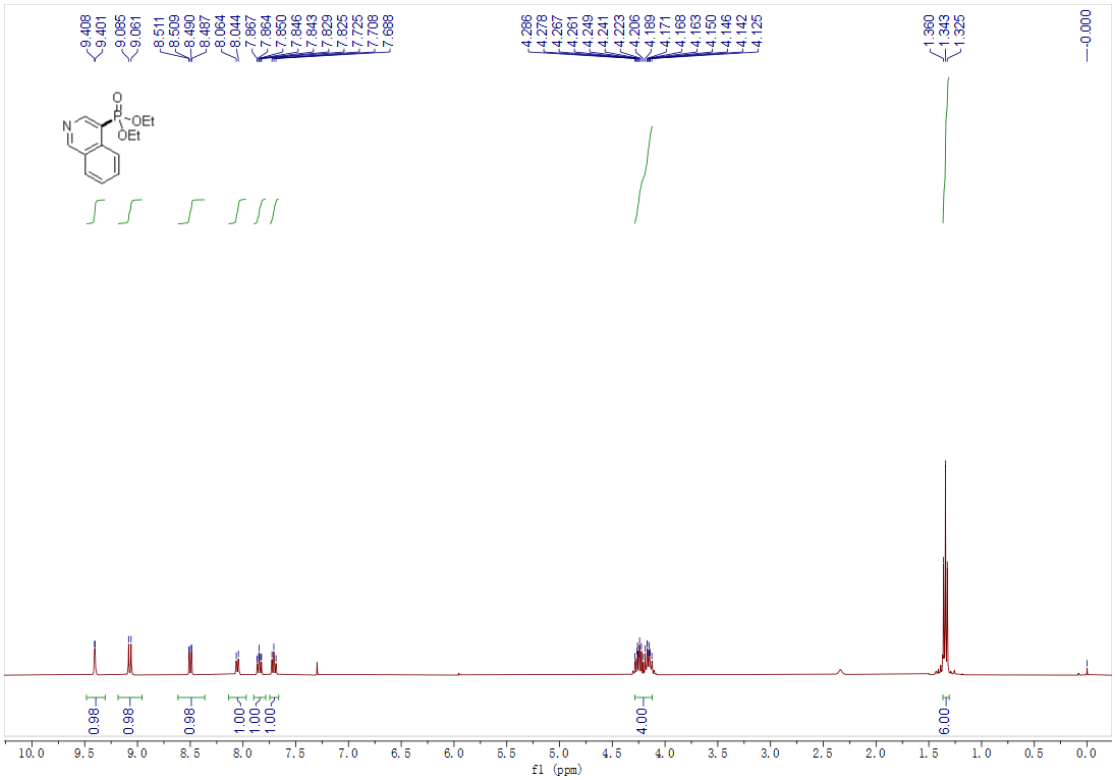
^{1}H NMR Spectrum of **5m**



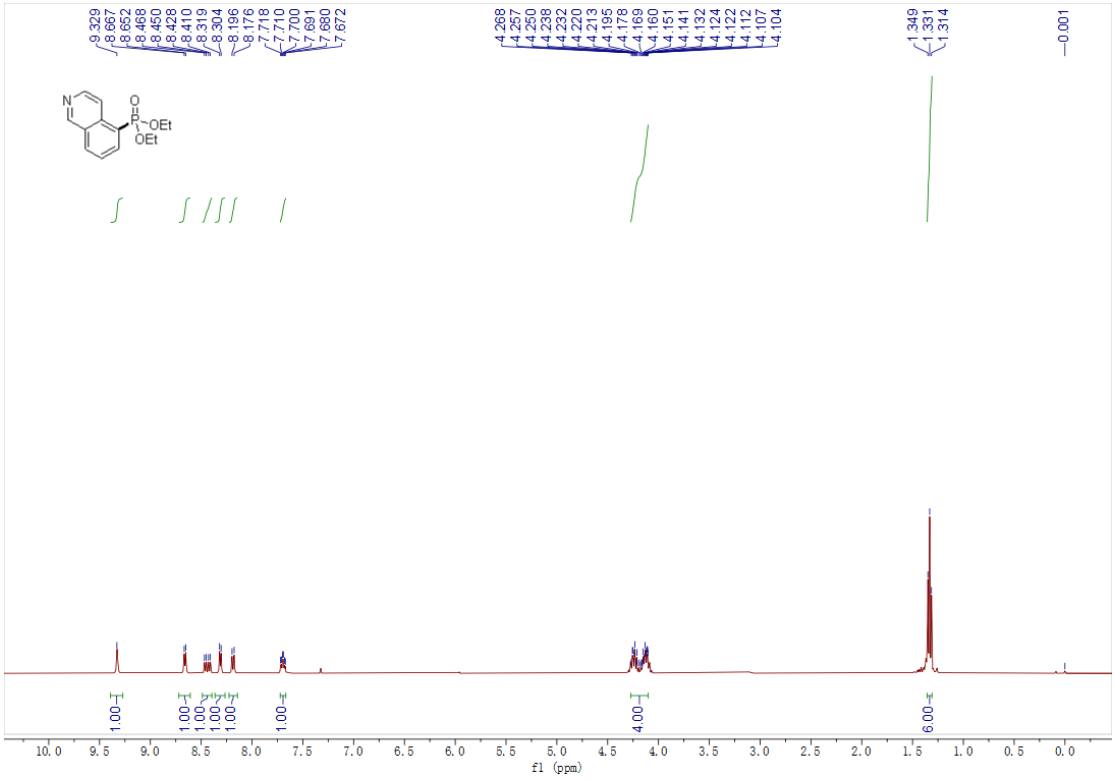
^{1}H NMR Spectrum of **5n**



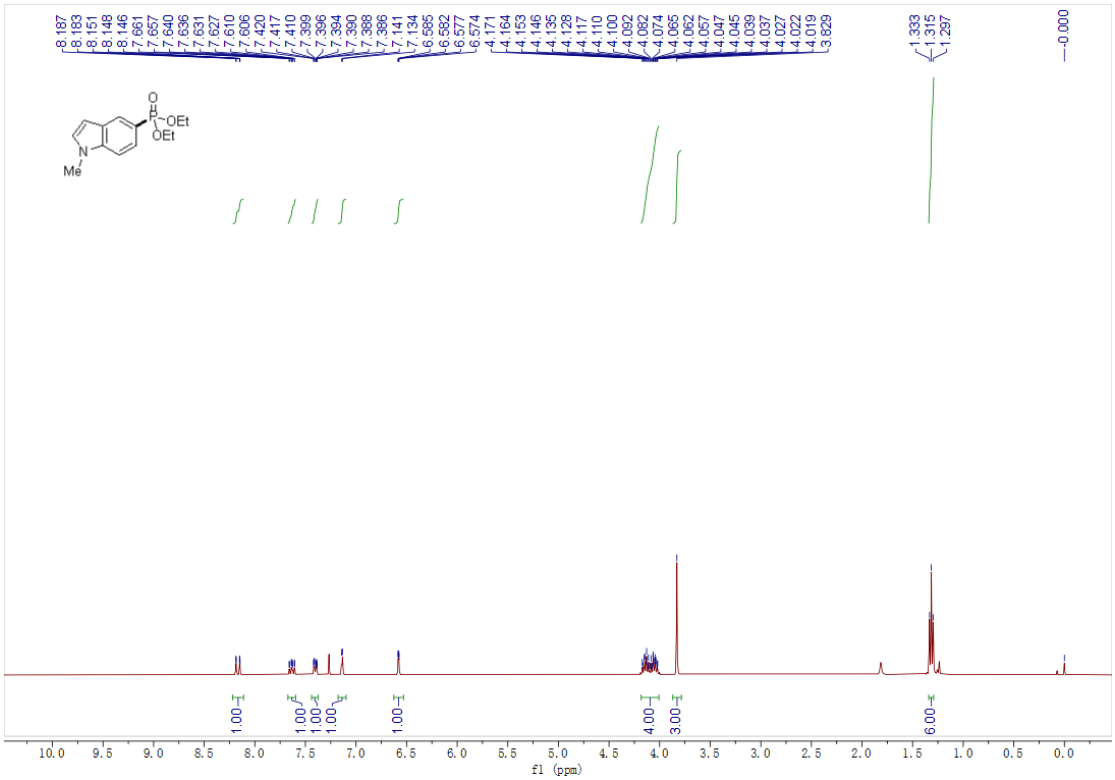
^{1}H NMR Spectrum of **5o**



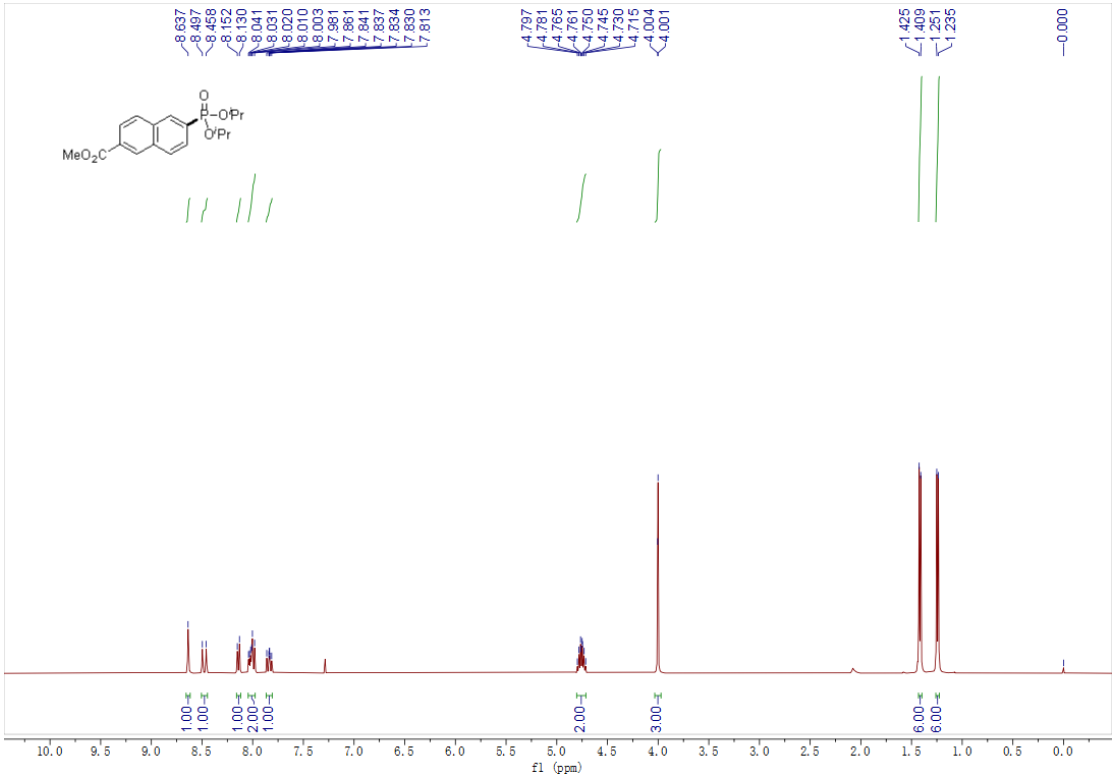
^{1}H NMR Spectrum of **5p**



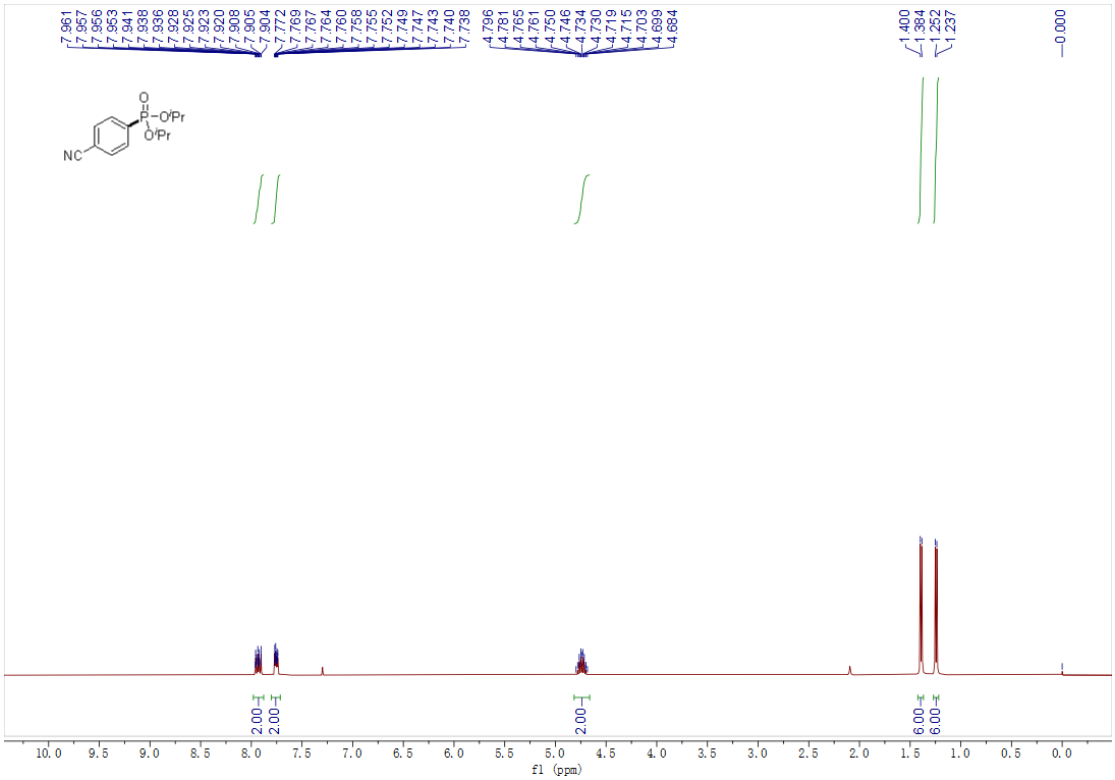
^{1}H NMR Spectrum of **5q**



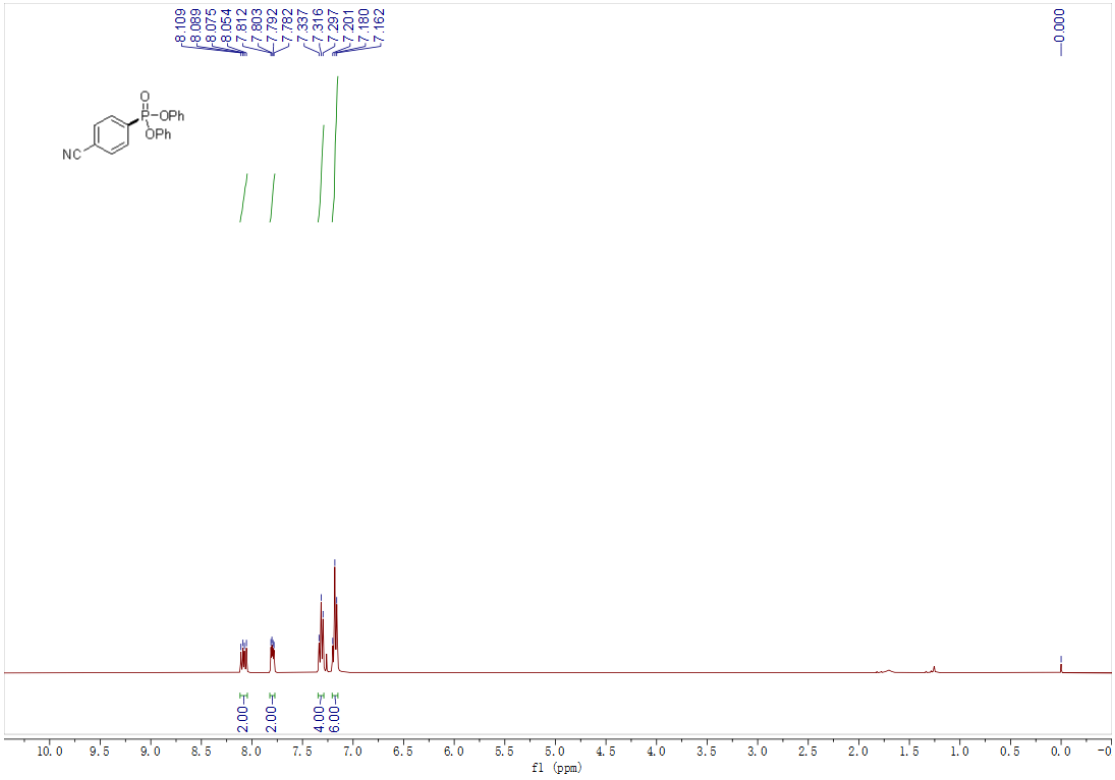
^{1}H NMR Spectrum of **5r**



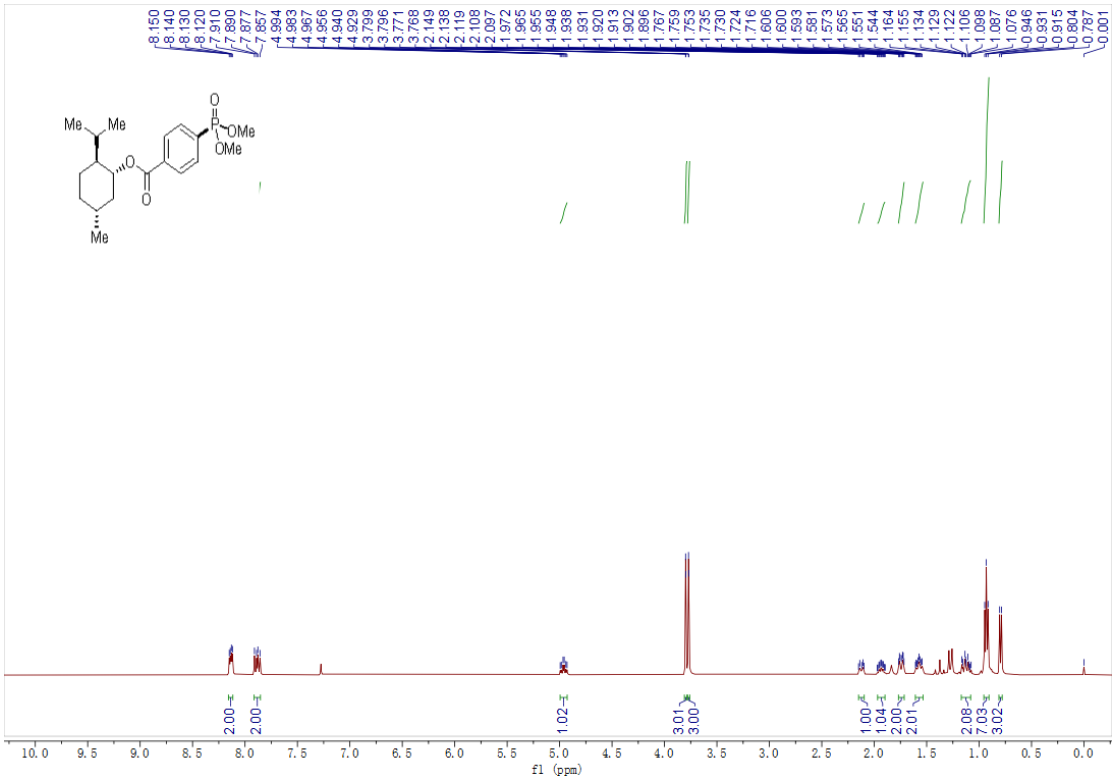
^{1}H NMR Spectrum of **5s**



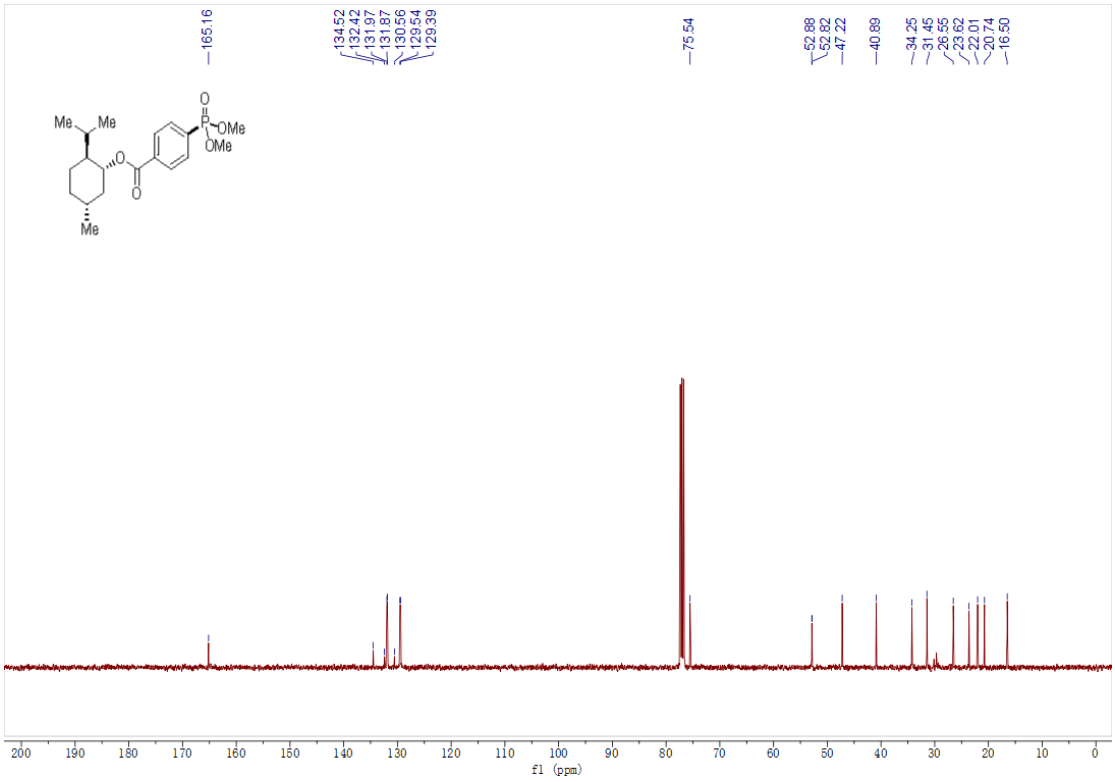
^{1}H NMR Spectrum of **5t**



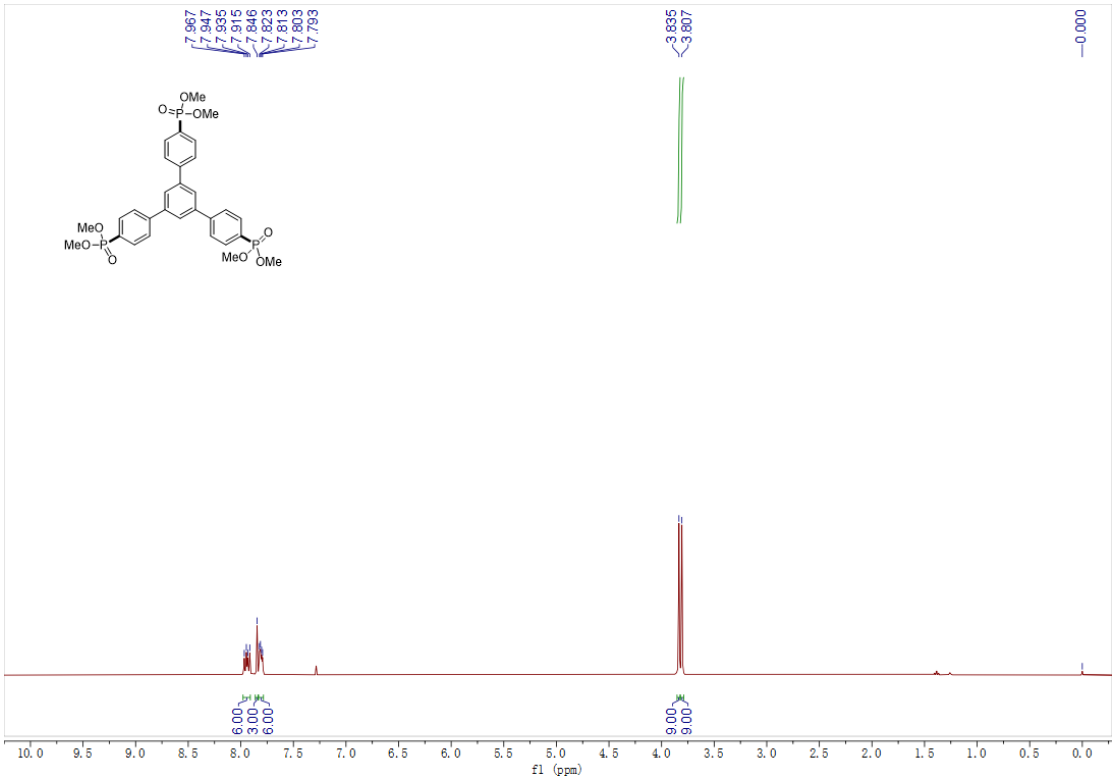
^{1}H NMR Spectrum of **5u**



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



^{1}H NMR Spectrum of **7**



^{13}C NMR Spectrum of **7**