

Supporting Information

C–N bond cleavage of primary amine for the synthesis of multi-substituted pyridine and pyrrole derivatives

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Table S1 Optimization of reaction conditions by screening the additives^a

Entry	Additive (1 equiv.)	Yield (%) ^b
1	CF ₃ SO ₃ H	33
2	CH ₃ COOH	45
3	C ₅ H ₅ N	70
4	Et ₃ N	34
5	NaOH	14
6	Na ₂ CO ₃	19
7	KH ₂ PO ₄	52
8	KHCO ₃	6
9	CH ₃ NH ₂	33
10	CH ₃ CH ₂ NH ₂	64
11	DEA	42
12	K ₂ CO ₃	0
13	Li ₂ CO ₃	58
14	NaOAc	47

^aReaction condition: **1a** (1 mmol), **2** (2 equiv.), I₂ (0.1 equiv.) and additive (1 equiv.) in PhNO₂ (1 mL) at 140 °C for 24 h. ^bHPLC yield.

Table S2 Optimization of reaction conditions by screening the amount of catalyst^a

Entry	NiCl ₂ (equiv.)	Yield (%) ^b
1	0.1	76
2	0.2	57
3	0.3	55
4	0.5	55

^aReaction condition: **1a** (1 mmol), **2** (2 equiv.), I₂ (0.1 equiv.) and NiCl₂ at the indicated amount in PhNO₂ (1 mL) at 140 °C for 24 h. ^bHPLC yield.

Table S3 Optimization of reaction conditions by screening the solvent volumes^a

Entry	PhNO₂ (mL)	Yield (%)^b
1	1	46
2	2	38
3	3	36
4	4	26

^aReaction condition: **1a** (1 mmol), **4** (1 equiv.) and I₂ (0.1 equiv.) in PhNO₂ (x mL) at 140 °C for 24 h. ^bHPLC yield.

Table S4 Optimization of reaction conditions by screening the temperatures^a

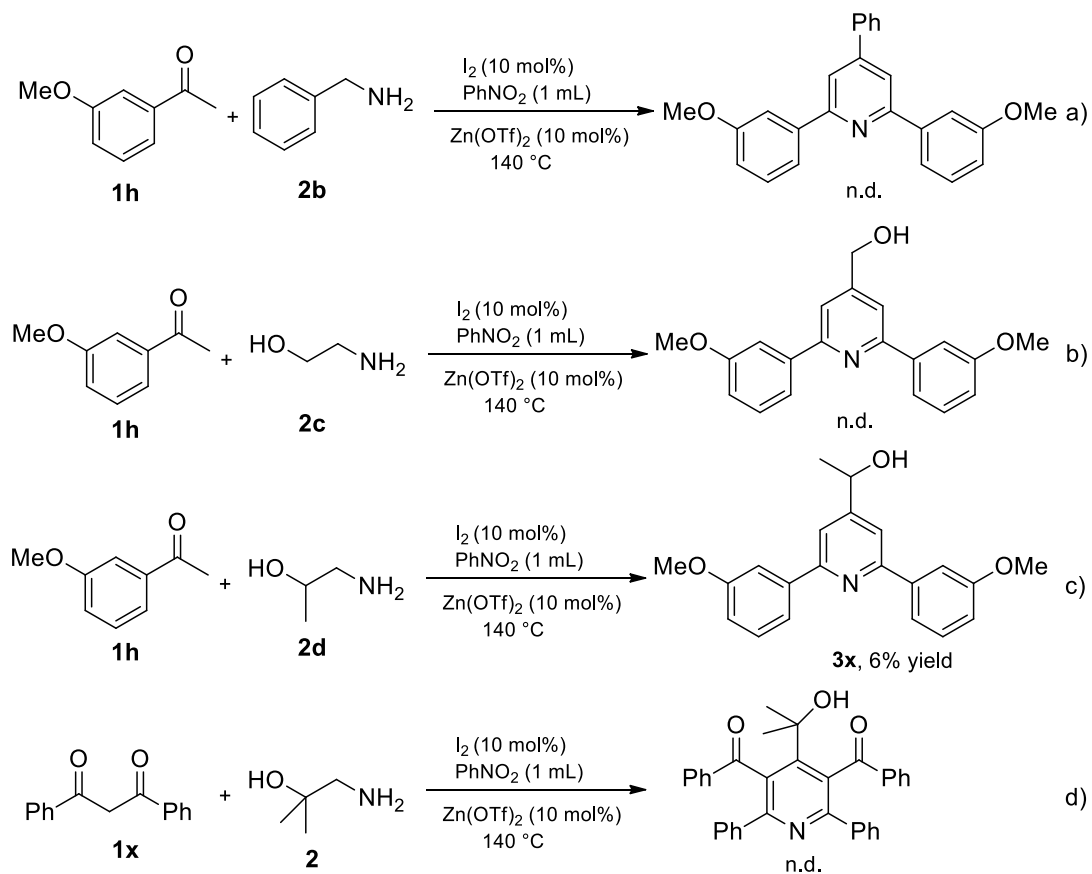
Entry	Temperature (°C)	Yield (%)^b
1	100	<5
2	110	22
3	120	36
4	130	38
5	140	46

^aReaction condition: **1a** (1 mmol), **4** (1 equiv.) and I₂ (0.1 equiv.) in PhNO₂ (1 mL) at x °C for 24 h. ^bHPLC yield.

Table S5 Optimization of reaction conditions by screening the ratio of 1a and 4^a

Entry	Ratio of 1a:4	Yield (%)^b
1	1:1	46
2	1:1.5	41
3	1:2	40
4	1:2.5	36

^aReaction condition: **1a** (1 mmol), **4** (x equiv.) and I₂ (0.1 equiv.) in PhNO₂ (1 mL) at 140 °C for 24 h. ^bHPLC yield.



Scheme S1 Additional attempts for the synthesis of pyridines

General methods All reagents and solvents were used from commercial sources unless otherwise stated. All raw materials are obtained from commercial sources. All experiments were conducted in the air. All the reactions were monitored by thin-layer chromatography (TLC). TLC was performed on pre-coated silica gel plates (Qingdao Haiyang Chemical Co., Ltd, China). Column chromatography was performed on silica gel (240-400 mesh) with petroleum ether and ethyl acetate as eluent. ¹H and ¹³C NMR (400 and 101 MHz) spectra were recorded on a Bruker Avance 400 MHz using CDCl₃ as solvent with tetramethylsilane as the internal standard. Melting points are determined using an WRS-1B apparatus and are uncorrected. High resolution mass spectra were obtained with the Q-TOF-Premier mass spectrometer. Reactions were monitored by TLC and visualized with ultraviolet light.

General Procedure for the synthesis of pyridine derivatives (3a as an example)

Acetophenone (120 mg, 1 mmol), 1-amino-2-methylpropan-2-ol (178 mg, 2 mmol), I₂ (12.5 mg, 0.05 mmol), Zn(OTf)₂ (18 mg, 0.05 mmol) and nitrobenzene (1 mL) were added to a tube, the reaction mixture was stirred in a sealed tube at 140 °C for 24 h. The reaction was monitored by TLC. Once the reaction was completed, the reaction mixture was treated with H₂O (15.0 mL) and EtOAc (8.0 mL). The organic and aqueous layers were then separated, and the aqueous layer was extracted with EtOAc (3 x 8 mL). The combined organic extracts were dried (Na₂SO₄), then the solvent was removed under reduced pressure and the remaining residue was purified by column chromatography. Compound **3a** (111 mg, 77% yield) was obtained as a white solid.

General Procedure for the synthesis of pyrrole derivatives (5a as an example)

Acetophenone (120 mg, 1 mmol), 1,2-diamino-2-methylpropane (88 mg, 1 mmol), I₂ (12.5 mg, 0.05 mmol), FeCl₃ (8 mg, 0.05 mmol) and nitrobenzene (1 mL) were added to a tube, the reaction mixture was stirred in a sealed tube at 140 °C for 24 h. The reaction was monitored by TLC. Once the reaction was completed, the reaction mixture was treated with H₂O (15.0 mL) and EtOAc (8.0 mL). The organic and aqueous layers were then separated, and the aqueous layer was extracted with EtOAc (3 x 8 mL). The combined organic extracts were dried (Na₂SO₄), then the solvent was removed under reduced pressure and the remaining residue was purified by column chromatography. Compound **5a** (112 mg, 77% yield) was obtained as a yellow oil.

2-(2,6-Diphenylpyridin-4-yl)propan-2-ol (3a): Petroleum ether/ethyl acetate = 4:1; white solid (111 mg, 77%); ¹H NMR (400 MHz, CDCl₃) δ 8.16 (d, *J* = 7.0 Hz, 4H), 7.79 (s, 2H), 7.52 – 7.41 (m, 6H), 1.66 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.3, 157.2, 139.7, 129.0, 128.7, 127.1, 114.8, 72.4, 31.5. HRMS(ESI): calcd. for C₂₀H₂₀NO⁺ [M+H]⁺ 290.1539; found: 290.1546.

2-(2,6-Di-p-tolylpyridin-4-yl)propan-2-ol (3b): Petroleum ether/ethyl acetate = 4:1; white solid (129 mg, 81%); ¹H NMR (400 MHz, CDCl₃) δ 8.05 (d, *J* = 7.9 Hz, 4H), 7.73 (s, 2H), 7.29 (d, *J* = 7.9 Hz, 4H), 2.42 (s, 6H), 1.64 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.1, 157.1, 138.8, 137.0, 129.4, 127.0, 114.2, 72.4, 31.4, 21.3. HRMS(ESI): calcd. for C₂₂H₂₄NO⁺ [M+H]⁺ 318.1852; found: 318.1853.

2-(2,6-Bis(4-methoxyphenyl)pyridin-4-yl)propan-2-ol (3c): Petroleum ether/ethyl acetate = 4:1; white solid (141 mg, 81%); ¹H NMR (400 MHz, CDCl₃) δ 8.11 (d, *J* = 8.4 Hz, 4H), 7.67 (s, 2H), 7.01 (d, *J* = 8.4 Hz, 4H), 3.88 (s, 6H), 2.00 (brs, 1H), 1.65 (s, 6H). ¹³C NMR (101 MHz,

CDCl₃) δ 160.4, 159.1, 156.7, 132.5, 128.4, 114.0, 113.3, 72.4, 55.4, 31.4. HRMS(ESI): calcd. for C₂₂H₂₄NO₃⁺ [M+H]⁺ 350.1751; found: 350.1756.

2-(2,6-Bis(4-fluorophenyl)pyridin-4-yl)propan-2-ol (3d): Petroleum ether/ethyl acetate = 4:1; white solid (104 mg, 64%); ¹H NMR (400 MHz, CDCl₃) δ 8.13(dd, *J* = 8.8, 5.6 Hz, 4H), 7.74 (s, 2H), 7.18 (t, *J* = 8.8 Hz, 4H), 1.67 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 163.5 (d, *J* = 247 Hz), 159.6, 156.2, 135.7 (d, *J* = 3.1 Hz), 128.9 (d, *J* = 8.5 Hz), 115.6 (d, *J* = 21.6 Hz), 114.4, 72.4, 31.5. HRMS(ESI): calcd. for C₂₀H₁₈F₂NO⁺ [M+H]⁺ 326.1351; found: 326.1355.

2-(2,6-Bis(4-chlorophenyl)pyridin-4-yl)propan-2-ol (3e): Petroleum ether/ethyl acetate = 4:1; white solid (111 mg, 62%); ¹H NMR (400 MHz, CDCl₃) δ 8.08 (d, *J* = 8.6 Hz, 4H), 7.77 (s, 2H), 7.46 (d, *J* = 8.6 Hz, 4H), 1.66 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.8, 156.0, 137.9, 135.2, 128.8, 128.4, 114.8, 72.3, 31.5. HRMS(ESI): calcd. for C₂₀H₁₈Cl₂NO⁺ [M+H]⁺ 358.0760; found: 358.0765.

2-(2,6-Bis(4-bromophenyl)pyridin-4-yl)propan-2-ol (3f): Petroleum ether/ethyl acetate = 4:1; white solid (140 mg, 63%); ¹H NMR (400 MHz, CDCl₃) δ 8.02 (d, *J* = 8.4 Hz, 4H), 7.78 (s, 2H), 7.62 (d, *J* = 8.4 Hz, 4H), 1.66 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.8, 156.1, 138.4, 131.8, 128.7, 123.5, 114.8, 72.4, 31.5. HRMS(ESI): calcd. for C₂₀H₁₈Br₂NO⁺ [M+H]⁺ 445.9750; found: 445.9747.

2-(2,6-Di-*m*-tolylpyridin-4-yl)propan-2-ol (3g): Petroleum ether/ethyl acetate = 4:1; white solid (119 mg, 75%); ¹H NMR (400 MHz, CDCl₃) δ 7.95 (s, 2H), 7.92 (d, *J* = 7.9 Hz, 2H), 7.75 (s, 2H), 7.38 (t, *J* = 7.7 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 2.46 (s, 6H), 2.00 (brs, 1H), 1.65 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.2, 157.4, 139.8, 138.3, 129.7, 128.6, 127.9, 124.3, 114.9, 72.5, 31.5, 21.7. HRMS(ESI): calcd. for C₂₂H₂₄NO⁺ [M+H]⁺ 318.1852; found: 318.1849.

2-(2,6-Bis(3-methoxyphenyl)pyridin-4-yl)propan-2-ol (3h): Petroleum ether/ethyl acetate = 4:1; white solid (127 mg, 73%); ¹H NMR (400 MHz, CDCl₃) δ 7.77 (s, 2H), 7.74 (t, *J* = 2.1 Hz, 2H), 7.70 (dd, *J* = 7.7, 1.6 Hz, 2H), 7.40 (t, *J* = 7.9 Hz, 2H), 6.98 (dd, *J* = 8.1, 2.6 Hz, 2H), 3.91 (s, 6H), 2.03 (brs, 1H), 1.65 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 160.0, 159.4, 156.8, 141.2, 129.7, 119.6, 115.1, 114.6, 112.7, 72.4, 55.4, 31.5. HRMS(ESI): calcd. for C₂₂H₂₄NO₃⁺ [M+H]⁺ 350.1751; found: 350.1756.

2-(2,6-Bis(3-fluorophenyl)pyridin-4-yl)propan-2-ol (3i): Petroleum ether/ethyl acetate = 4:1; white solid (107 mg, 66%); ¹H NMR (400 MHz, CDCl₃) δ 7.91 – 7.87 (m, 4H), 7.79 (s, 2H), 7.48 – 7.42 (m, 2H), 7.12 (td, *J* = 8.4, 2.0 Hz, 2H), 2.01 (brs, 1H), 1.66 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 163.3 (d, *J* = 244 Hz), 159.8, 155.9 (d, *J* = 2.6 Hz), 141.8 (d, *J* = 7.4 Hz), 130.2 (d, *J* = 8.1 Hz), 122.6 (d, *J* = 2.7 Hz), 115.9 (d, *J* = 21.2 Hz), 115.4, 114.0 (d, *J* = 22.6 Hz), 72.4, 31.5. HRMS(ESI): calcd. for C₂₀H₁₈F₂NO⁺ [M+H]⁺ 326.1351; found: 326.1348.

2-(2,6-Bis(3-chlorophenyl)pyridin-4-yl)propan-2-ol (3j): Petroleum ether/ethyl acetate = 4:1; yellow solid (114 mg, 64%); ¹H NMR (400 MHz, CDCl₃) δ 8.13 (s, 2H), 8.03 (d, *J* = 7.8 Hz, 2H), 7.80 (s, 2H), 7.46 (d, *J* = 7.9 Hz, 2H), 7.41 (t, *J* = 7.9 Hz, 2H), 1.68 (s, 6H). ¹³C NMR

(101 MHz, CDCl₃) δ 159.9, 155.9, 141.2, 134.8, 130.0, 129.1, 127.2, 125.2, 115.5, 72.4, 31.6. HRMS(ESI): calcd. for C₂₀H₁₈Cl₂NO⁺ [M+H]⁺ 358.0760; found: 358.0753.

2-(2,6-Bis(3-bromophenyl)pyridin-4-yl)propan-2-ol (3k): Petroleum ether/ethyl acetate = 4:1; white solid (131 mg, 59%); ¹H NMR (400 MHz, CDCl₃) δ 8.27 (s, 2H), 8.07 (d, *J* = 7.8 Hz, 2H), 7.78 (s, 2H), 7.57 (d, *J* = 7.9 Hz, 2H), 7.37 (t, *J* = 7.9 Hz, 2H), 1.68 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.9, 155.8, 141.5, 132.0, 130.3, 130.1, 125.7, 123.0, 115.5, 72.4, 31.6. HRMS(ESI): calcd. for C₂₀H₁₈Br₂NO⁺ [M+H]⁺ 445.9750; found: 445.9756.

2-(2,6-Di-*o*-tolylpyridin-4-yl)propan-2-ol (3l): Petroleum ether/ethyl acetate = 4:1; White solid (103 mg, 65%); ¹H NMR (400 MHz, CDCl₃) δ 7.45 - 7.42 (m, 4H), 7.27 - 7.23 (m, 6H), 2.11 (brs, 1H), 1.65 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.6, 158.3, 140.9, 135.9, 130.7, 129.9, 128.2, 125.8, 118.2, 72.1, 31.4, 20.6. HRMS(ESI): calcd. for C₂₂H₂₄NO⁺ [M+H]⁺ 318.1852; found: 318.1856.

2-(2,6-Bis(2-chlorophenyl)pyridin-4-yl)propan-2-ol (3m): Petroleum ether/ethyl acetate = 4:1; white solid (102 mg, 57%); ¹H NMR (400 MHz, CDCl₃) δ 7.74 (s, 2H), 7.69 (d, *J* = 7.4 Hz, 2H), 7.49 (d, *J* = 7.6 Hz, 2H), 7.37 - 7.30 (m, 4H), 2.00 (brs, 1H), 1.65 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 157.8, 156.7, 139.3, 132.3, 131.9, 130.1, 129.6, 127.1, 119.6, 72.3, 31.3. HRMS(ESI): calcd. for C₂₀H₁₈Cl₂NO⁺ [M+H]⁺ 358.0760; found: 358.0757.

2-(2,6-Bis(2-bromophenyl)pyridin-4-yl)propan-2-ol (3n): Petroleum ether/ethyl acetate = 4:1; yellow solid (116 mg, 52%); ¹H NMR (400 MHz, CDCl₃) δ 7.71 (s, 2H), 7.67 (t, *J* = 7.6 Hz, 4H), 7.40 (t, *J* = 7.7 Hz, 2H), 7.24 (d, *J* = 7.9 Hz, 2H), 1.66 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 158.0, 157.7, 141.3, 133.3, 131.8, 129.8, 127.6, 121.9, 119.6, 72.3, 31.3. HRMS(ESI): calcd. for C₂₀H₁₈Br₂NO⁺ [M+H]⁺ 445.9750; found: 445.9757.

2-(2,6-Bis(2,4-dimethylphenyl)pyridin-4-yl)propan-2-ol (3o): Petroleum ether/ethyl acetate = 4:1; White solid (116 mg, 67%); ¹H NMR (400 MHz, CDCl₃) δ 7.41 (s, 2H), 7.36 (d, *J* = 7.9 Hz, 2H), 7.09 (s, 2H), 7.07 (d, *J* = 7.9 Hz, 2H), 2.40 (s, 6H), 2.35 (s, 6H), 1.80 (brs, 1H), 1.62 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.6, 157.9, 138.1, 137.9, 135.7, 131.5, 129.9, 126.5, 117.9, 72.2, 31.4, 21.2, 20.6. HRMS(ESI): calcd. for C₂₄H₂₈NO⁺ [M+H]⁺ 346.2165; found: 346.2171.

2-(2,6-Bis(3,4-dimethylphenyl)pyridin-4-yl)propan-2-ol (3p): Petroleum ether/ethyl acetate = 4:1; White solid (128 mg, 74%); ¹H NMR (400 MHz, CDCl₃) δ 7.92 (s, 2H), 7.86 (d, *J* = 7.8 Hz, 2H), 7.72 (s, 2H), 7.25 (d, *J* = 7.8 Hz, 2H), 2.38 (s, 6H), 2.33 (s, 6H), 1.79 (brs, 1H), 1.66 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.0, 157.3, 137.52, 136.8, 129.9, 128.3, 124.6, 114.3, 72.5, 31.4, 20.0, 19.7. HRMS(ESI): calcd. for C₂₄H₂₈NO⁺ [M+H]⁺ 346.2165; found: 346.2170.

2-(2,6-Bis(3,4-dichlorophenyl)pyridin-4-yl)propan-2-ol (3q): Petroleum ether/ethyl acetate = 4:1; White solid (134 mg, 63%); ¹H NMR (400 MHz, CDCl₃) δ 8.23 (d, *J* = 2.0 Hz, 2H), 7.98 (dd, *J* = 8.4, 2.0 Hz, 2H), 7.78 (s, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 1.98 (brs, 1H), 1.68 (s, 6H). ¹³C

NMR (101 MHz, CDCl₃) δ 160.2, 155.0, 139.2, 133.3, 133.0, 130.7, 128.9, 126.2, 115.4, 72.4, 31.6. HRMS(ESI): calcd. for C₂₀H₁₆Cl₄NO⁺ [M+H]⁺ 425.9981; found: 425.9975.

2-(2,6-Bis(3,5-dimethoxyphenyl)pyridin-4-yl)propan-2-ol (3r): Petroleum ether/ethyl acetate = 4:1; White solid (157 mg, 77%); ¹H NMR (400 MHz, CDCl₃) δ 7.74 (s, 2H), 7.30 (d, *J* = 2.0 Hz, 4H), 6.54 (t, *J* = 2.0 Hz, 2H), 3.87 (s, 12H), 2.19 (brs, 1H), 1.63 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 161.0, 159.5, 156.6, 141.8, 115.4, 105.4, 101.0, 72.3, 55.5, 31.4. HRMS(ESI): calcd. for C₂₄H₂₈NO₅⁺ [M+H]⁺ 410.1962; found: 410.1957.

2-(2,6-Bis(3-chloro-4-methoxyphenyl)pyridin-4-yl)propan-2-ol (3s): Petroleum ether/ethyl acetate = 4:1; White solid (138 mg, 66%); ¹H NMR (400 MHz, CDCl₃) δ 8.17 (d, *J* = 2.0 Hz, 2H), 8.03 (dd, *J* = 8.8, 2.0 Hz, 2H), 7.67 (s, 2H), 7.03 (d, *J* = 8.6 Hz, 2H), 3.97 (s, 6H), 1.99 (brs, 1H), 1.66 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.6, 155.7, 155.5, 133.0, 128.8, 126.5, 122.8, 113.9, 111.9, 72.4, 56.3, 31.5. HRMS(ESI): calcd. for C₂₂H₂₂Cl₂NO₃⁺ [M+H]⁺ 418.0971; found: 418.0978.

2-(2,6-Di(naphthalen-2-yl)pyridin-4-yl)propan-2-ol (3t): Petroleum ether/ethyl acetate = 4:1; White solid (150 mg, 77%); ¹H NMR (400 MHz, CDCl₃) δ 8.64 (s, 2H), 8.38 (dd, *J* = 8.6, 1.8 Hz, 2H), 8.00 – 7.97 (m, 6H), 7.91 – 7.87 (m, 2H), 7.54 – 7.50 (m, 4H), 2.04 (brs, 1H), 1.73 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.5, 157.2, 137.1, 133.7, 133.5, 128.8, 128.4, 127.7, 126.5, 126.5, 126.3, 125.0, 115.2, 72.6, 31.6. HRMS(ESI): calcd. for C₂₈H₂₄NO⁺ [M+H]⁺ 390.1852; found: 390.1846.

2-(2,6-Di(naphthalen-1-yl)pyridin-4-yl)propan-2-ol (3u): Petroleum ether/ethyl acetate = 4:1; White solid (123 mg, 63%); ¹H NMR (400 MHz, CDCl₃) δ 8.23 – 8.18 (m, 2H), 7.91 – 7.85 (m, 4H), 7.73 – 7.71 (m, 4H), 7.55 (t, *J* = 7.6 Hz, 2H), 7.51 – 7.48 (m, 4H), 2.04 (brs, 1H), 1.67 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.2, 158.7, 138.9, 134.0, 131.4, 128.9, 128.4, 127.8, 126.4, 125.8, 125.7, 125.4, 119.6, 72.3, 31.5. HRMS(ESI): calcd. for C₂₈H₂₄NO⁺ [M+H]⁺ 390.1852; found: 390.1856.

2-(2,6-Di(thiophen-3-yl)pyridin-4-yl)propan-2-ol (3v): Petroleum ether/ethyl acetate = 4:1; yellow solid (62 mg, 41%); ¹H NMR (400 MHz, CDCl₃) δ 8.01 (dd, *J* = 3.2, 1.2 Hz, 2H), 7.78 (dd, *J* = 5.2, 1.2 Hz, 2H), 7.61 (s, 2H), 7.41 (dd, *J* = 5.2, 3.2 Hz, 2H), 1.65 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 159.3, 153.4, 142.6, 126.5, 126.1, 123.7, 114.3, 72.3, 31.4. HRMS(ESI): calcd. for C₁₆H₁₆NOS₂⁺ [M+H]⁺ 302.0668; found: 302.0673.

1-(2,5-Di(pyridin-2-yl)-1H-pyrrol-1-yl)-2-methylpropan-2-ol (3w): Petroleum ether/ethyl acetate = 4:1; white solid (66 mg, 45%); ¹H NMR (400 MHz, CDCl₃) δ 8.56 (d, *J* = 4.4 Hz, 2H), 7.74 (t, *J* = 7.6 Hz, 2H), 7.59 (d, *J* = 8.0 Hz, 2H), 7.19 (t, *J* = 6.40 Hz, 2H), 6.59 (s, 2H), 5.05 (s, 2H), 0.98 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 152.8, 147.8, 137.3, 135.5, 124.1, 121.3, 112.2, 70.7, 52.9, 28.2. HRMS(ESI): calcd. for C₁₈H₂₀N₃O⁺ [M+H]⁺ 294.1601; found: 294.1597.

2-(2,2-Dimethyl-5-phenyl-3,4-dihydro-2H-pyrrol-3-yl)-1-phenylethanone (5a): Petroleum

ether/ethyl acetate = 4:1; yellow oil (112 mg, 77%); ¹H NMR (400 MHz, CDCl₃) δ 8.00 (d, *J* = 7.2 Hz, 2H), 7.81 (d, *J* = 7.6 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.45 – 7.34 (m, 3H), 3.48 – 3.35 (m, 1H), 3.27 (dd, *J* = 16.0, 3.2 Hz, 1H), 3.00 (dd, *J* = 16.4, 10.4 Hz, 1H), 2.72 – 2.57 (m, 2H), 1.46 (s, 3H), 1.20 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) 199.6, 169.8, 136.9, 134.6, 133.3, 130.4, 128.7, 128.4, 128.1, 127.6, 73.5, 42.9, 41.2, 39.6, 28.8, 22.8. HRMS (ESI): calcd. for C₂₀H₂₂NO⁺ [M+H]⁺ 292.1696; found: 292.1702.

2-(2,2-Dimethyl-5-(*p*-tolyl)-3,4-dihydro-2*H*-pyrrol-3-yl)-1-(*p*-tolyl)ethanone (5b): Petroleum ether/ethyl acetate = 4:1; white solid (125 mg, 78%); ¹H NMR (400 MHz, CDCl₃) δ 7.89 (d, *J* = 8.0 Hz, 2H), 7.69 (d, *J* = 7.9 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 7.9 Hz, 2H), 3.44 – 3.30 (m, 1H), 3.23 (dd, *J* = 16.4, 2.8 Hz, 1H), 2.95 (dd, *J* = 16.0, 3.2 Hz, 1H), 2.72 – 2.55 (m, 2H), 2.42 (s, 3H), 2.36 (s, 3H), 1.44 (s, 3H), 1.19 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) 199.3, 169.7, 144.1, 140.6, 134.5, 131.9, 129.4, 129.1, 128.2, 127.6, 73.3, 43.0, 41.1, 39.5, 28.8, 22.8, 21.7, 21.4. HRMS (ESI): calcd. for C₂₂H₂₆NO⁺ [M+H]⁺ 320.2009; found: 320.2007.

1-(4-Methoxyphenyl)-2-(5-(4-methoxyphenyl)-2,2-dimethyl-3,4-dihydro-2*H*-pyrrol-3-yl)ethanone (5c): Petroleum ether/ethyl acetate = 2:1; yellow oil (132 mg, 75%); ¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, *J* = 8.8 Hz, 2H), 7.75 (d, *J* = 8.8 Hz, 2H), 6.96 (d, *J* = 8.9 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 3.88 (s, 3H), 3.82 (s, 3H), 3.40 – 3.28 (m, 1H), 3.19 (dd, *J* = 16.2, 3.3 Hz, 1H), 2.93 (dd, *J* = 16.2, 10.4 Hz, 1H), 2.66 – 2.53 (m, 2H), 1.43 (s, 3H), 1.18 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) 198.2, 169.1, 163.6, 161.4, 130.4, 130.0, 129.2, 127.5, 113.8, 113.7, 73.2, 55.5, 55.3, 43.2, 41.1, 39.3, 28.8, 22.8. HRMS (ESI): calcd. for C₂₂H₂₆NO₃⁺ [M+H]⁺ 352.1907; found: 352.1912.

1-(4-Fluorophenyl)-2-(5-(4-fluorophenyl)-2,2-dimethyl-3,4-dihydro-2*H*-pyrrol-3-yl)ethanone (5d): Petroleum ether/ethyl acetate = 4:1; yellow oil (116 mg, 71%); ¹H NMR (400 MHz, CDCl₃) δ 8.02 (dd, *J* = 8.8, 5.4 Hz, 2H), 7.80 (dd, *J* = 8.8, 5.6 Hz, 2H), 7.16 (t, *J* = 8.4 Hz, 2H), 7.06 (t, *J* = 8.4 Hz, 2H), 3.39 (dd, *J* = 15.6, 7.2 Hz, 1H), 3.23 (dd, *J* = 16.7, 3.3 Hz, 1H), 2.96 (dd, *J* = 16.8, 10.4 Hz, 1H), 2.67 – 2.54 (m, 2H), 1.44 (s, 3H), 1.19 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) 197.8, 168.6, 165.8 (d, *J* = 255 Hz), 164.7 (d, *J* = 249 Hz), 133.3 (d, *J* = 2.9 Hz), 130.9 (d, *J* = 2.2 Hz), 130.7 (d, *J* = 10.3 Hz), 129.6 (d, *J* = 8.6 Hz), 115.8 (d, *J* = 21.8 Hz), 115.4 (d, *J* = 21.6 Hz), 73.5, 42.9, 41.2, 39.5, 28.8, 22.8. HRMS (ESI): calcd. for C₂₀H₂₀F₂NO⁺ [M+H]⁺ 328.1507; found: 328.1511.

1-(4-Chlorophenyl)-2-(5-(4-chlorophenyl)-2,2-dimethyl-3,4-dihydro-2*H*-pyrrol-3-yl)ethanone (5e): Petroleum ether/ethyl acetate = 4:1; yellow oil (130 mg, 72%); ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.73 (d, *J* = 8.3 Hz, 2H), 7.46 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.2 Hz, 2H), 3.37 (dd, *J* = 15.6, 7.3 Hz, 1H), 3.22 (dd, *J* = 16.8, 3.4 Hz, 1H), 2.95 (dd, *J* = 16.8, 10.1 Hz, 1H), 2.66 – 2.51 (m, 2H), 1.44 (s, 3H), 1.18 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) 198.2, 168.7, 139.8, 136.5, 135.1, 133.1, 129.5, 129.1, 128.9, 128.6, 73.6, 42.9, 41.1, 39.6, 28.7, 22.7. HRMS (ESI): calcd. for C₂₀H₂₀Cl₂NO⁺ [M+H]⁺ 360.0916; found: 360.0921.

1-(4-Bromophenyl)-2-(5-(4-bromophenyl)-2,2-dimethyl-3,4-dihydro-2*H*-pyrrol-3-

yl)ethanone (5f): Petroleum ether/ethyl acetate = 4:1; yellow oil (152 mg, 68%); ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, *J* = 8.6 Hz, 2H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.63 (d, *J* = 8.8 Hz, 2H), 7.51 (d, *J* = 8.4 Hz, 2H), 3.37 (dd, *J* = 15.7, 7.2 Hz, 1H), 3.22 (dd, *J* = 16.8, 3.4 Hz, 1H), 2.94 (dd, *J* = 16.8, 10.1 Hz, 1H), 2.69 – 2.53 (m, 2H), 1.44 (s, 3H), 1.18 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) 198.3, 168.8, 135.5, 133.5, 132.1, 131.6, 129.6, 129.1, 128.5, 124.9, 73.6, 42.8, 41.0, 39.5, 28.7, 22.7. HRMS(ESI): calcd. for C₂₀H₂₀Br₂NO⁺ [M+H]⁺ 447.9906; found: 447.9912.

1-(4-(Trifluoromethyl)phenyl)-2-(5-(4-(trifluoromethyl)phenyl)-2,2-dimethyl-3,4-dihydro-2H-pyrrol-3-yl)ethanone (5g): Petroleum ether/ethyl acetate = 4:1; yellow oil (122 mg, 57%); ¹H NMR (400 MHz, CDCl₃) δ 8.10 (d, *J* = 7.9 Hz, 2H), 7.92 (d, *J* = 7.9 Hz, 2H), 7.77 (d, *J* = 8.0 Hz, 2H), 7.65 (d, *J* = 8.0 Hz, 2H), 3.45 (dd, *J* = 15.4, 7.1 Hz, 1H), 3.30 (dd, *J* = 17.2, 3.4 Hz, 1H), 3.03 (dd, *J* = 17.0, 9.9 Hz, 1H), 2.77 – 2.58 (m, 2H), 1.47 (s, 3H), 1.21 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) 198.3, 168.6, 139.4, 137.8, 134.7 (d, *J* = 32.7 Hz), 132.1 (d, *J* = 32.5 Hz), 128.4, 127.9, 125.9 (q, *J* = 3.7 Hz), 125.4 (d, *J* = 3.8 Hz), 123.5 (q, *J* = 271 Hz), 121.5 (q, *J* = 223 Hz), 73.9, 42.7, 41.2, 39.9, 28.7, 22.7. HRMS(ESI): calcd. for C₂₂H₂₀F₆NO⁺ [M+H]⁺ 428.1444; found: 428.1447.

2-(2,2-Dimethyl-5-(*m*-tolyl)-3,4-dihydro-2H-pyrrol-3-yl)-1-(*m*-tolyl)ethanone (5h): Petroleum ether/ethyl acetate = 4:1; yellow oil (120 mg, 75%); ¹H NMR (400 MHz, CDCl₃) δ 7.80 (s, 1H), 7.79 (d, *J* = 7.8 Hz, 1H), 7.68 (s, 1H), 7.55 (d, *J* = 7.5 Hz, 1H), 7.41 – 7.35 (m, 2H), 7.29 – 7.20 (m, 2H), 3.45 – 3.35 (m, 1H), 3.25 (dd, *J* = 16.6, 3.0 Hz, 1H), 2.97 (dd, *J* = 16.4, 10.4 Hz, 1H), 2.67 – 2.56 (m, 2H), 2.43 (s, 3H), 2.37 (s, 3H), 1.45 (s, 3H), 1.20 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) 199.8, 170.0, 138.5, 138.1, 137.0, 134.6, 134.0, 131.2, 128.6, 128.6, 128.3, 128.0, 125.3, 124.9, 73.4, 42.9, 41.2, 39.7, 28.8, 22.8, 21.4, 21.3. HRMS(ESI): calcd. for C₂₂H₂₆NO⁺ [M+H]⁺ 320.2009; found: 320.2006.

1-(3-Methoxyphenyl)-2-(5-(3-methoxyphenyl)-2,2-dimethyl-3,4-dihydro-2H-pyrrol-3-yl)ethanone (5i): Petroleum ether/ethyl acetate = 2:1; yellow oil (127 mg, 72%); ¹H NMR (400 MHz, CDCl₃) δ 7.56 (d, *J* = 7.2 Hz, 1H), 7.51 (s, 1H), 7.44 – 7.38 (m, 2H), 7.34 (d, *J* = 7.2 Hz, 1H), 7.28 (t, *J* = 7.2 Hz, 1H), 7.13 (dd, *J* = 8.0, 2.0 Hz, 1H), 6.96 (dd, *J* = 7.6, 2.0 Hz, 1H), 3.87 (s, 3H), 3.84 (s, 3H), 3.44 – 3.36 (m, 1H), 3.24 (dd, *J* = 16.8, 3.2 Hz, 1H), 2.98 (dd, *J* = 16.8, 5.2 Hz, 1H), 2.65 – 2.56 (m, 2H), 1.46 (s, 3H), 1.20 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) 199.4, 169.8, 159.9, 159.7, 138.2, 136.0, 129.7, 129.4, 120.7, 120.3, 119.6, 117.0, 112.4, 112.0, 73.5, 55.49, 55.46, 42.9, 41.3, 39.7, 28.7, 22.7. HRMS(ESI): calcd. for C₂₂H₂₆NO₃⁺ [M+H]⁺ 352.1907; found: 352.1913.

1-(3-Fluorophenyl)-2-(5-(3-fluorophenyl)-2,2-dimethyl-3,4-dihydro-2H-pyrrol-3-yl)ethanone (5j): Petroleum ether/ethyl acetate = 4:1; yellow oil (115 mg, 70%); ¹H NMR (400 MHz, CDCl₃) δ 7.77 (d, *J* = 7.8 Hz, 1H), 7.67 (d, *J* = 9.2 Hz, 1H), 7.56 (s, 1H), 7.53 (s, 1H), 7.51 – 7.45 (m, 1H), 7.36 (t, *J* = 7.2 Hz, 1H), 7.31 (d, *J* = 9.2 Hz, 1H), 7.11 (t, *J* = 8.2 Hz, 1H), 3.39 (dd, *J* = 15.9, 7.4 Hz, 1H), 3.24 (d, *J* = 16.9 Hz, 1H), 2.97 (dd, *J* = 17.0, 10.2 Hz, 1H), 2.70 – 2.55 (m, 2H), 1.46 (s, 3H), 1.19 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) 198.1 (d, *J* = 2.0 Hz), 168.7 (d, *J* = 2.7 Hz), 162.9 (d, *J* = 247 Hz), 162.8 (d, *J* = 245 Hz), 138.9 (d, *J* = 6.0 Hz), 136.9 (d, *J* = 7.1 Hz), 130.4 (d, *J* = 10.0 Hz), 128.9 (d, *J* = 8.1 Hz), 123.8 (d, *J* = 4.0 Hz), 123.4 (d, *J*

= 3.0 Hz), 120.3 (d, $J = 21.2$ Hz), 120.3 (d, $J = 21.2$ Hz), 114.8 (d, $J = 22.2$ Hz), 114.3 (d, $J = 22.2$ Hz), 73.6, 42.8, 41.2, 39.7, 28.7, 22.7. HRMS(ESI): calcd. for $C_{20}H_{20}F_2NO^+$ $[M+H]^+$ 328.1507; found: 328.1511.

1-(3-Chlorophenyl)-2-(5-(3-chlorophenyl)-2,2-dimethyl-3,4-dihydro-2H-pyrrol-3-yl)ethanone (5k): Petroleum ether/ethyl acetate = 4:1; yellow oil (122 mg, 68%); 1H NMR (400 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.86 (d, $J = 7.2$ Hz, 1H), 7.83 (s, 1H), 7.65 (d, $J = 7.7$ Hz, 1H), 7.57 (d, $J = 8.1$ Hz, 1H), 7.44 (t, $J = 7.8$ Hz, 1H), 7.39 (d, $J = 8.1$ Hz, 1H), 7.32 (t, $J = 8.3$ Hz, 1H), 3.39 (dd, $J = 16.2, 7.6$ Hz, 1H), 3.23 (d, $J = 17.1$ Hz, 1H), 2.97 (dd, $J = 17.0, 10.2$ Hz, 1H), 2.70 – 2.51 (m, 2H), 1.45 (s, 3H), 1.20 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) 198.0, 168.6, 138.4, 136.3, 135.1, 134.6, 133.2, 130.4, 130.1, 129.7, 128.2, 127.6, 126.1, 125.7, 73.7, 42.7, 41.1, 39.7, 28.7, 22.7. HRMS(ESI): calcd. for $C_{20}H_{20}Cl_2NO^+$ $[M+H]^+$ 360.0916; found: 360.0919.

1-(3-Bromophenyl)-2-(5-(3-bromophenyl)-2,2-dimethyl-3,4-dihydro-2H-pyrrol-3-yl)ethanone (5l): Petroleum ether/ethyl acetate = 4:1; yellow oil (154 mg, 69%); 1H NMR (400 MHz, $CDCl_3$) δ 8.11 (s, 1H), 7.99 (s, 1H), 7.91 (d, $J = 7.8$ Hz, 1H), 7.73 (d, $J = 9.2$ Hz, 1H), 7.70 (d, $J = 9.2$ Hz, 1H), 7.54 (d, $J = 8.0$ Hz, 1H), 7.38 (t, $J = 7.8$ Hz, 1H), 7.25 (t, $J = 7.6$ Hz, 1H), 3.39 (dd, $J = 16.3, 7.6$ Hz, 1H), 3.22 (d, $J = 17.2$ Hz, 1H), 2.96 (dd, $J = 17.1, 10.4$ Hz, 1H), 2.73 – 2.52 (m, 2H), 1.45 (s, 3H), 1.19 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) 197.9, 168.5, 138.6, 136.6, 136.2, 133.3, 131.1, 130.5, 130.3, 130.0, 126.5, 126.2, 123.1, 122.8, 73.7, 42.7, 41.1, 39.7, 28.7, 22.7. HRMS(ESI): calcd. for $C_{20}H_{20}Br_2NO^+$ $[M+H]^+$ 447.9906; found: 447.9910.

2-(2,2-Dimethyl-5-(*o*-tolyl)-3,4-dihydro-2H-pyrrol-3-yl)-1-(*o*-tolyl)ethan-1-one (5m): Petroleum ether/ethyl acetate = 4:1; yellow oil (45 mg, 28%); 1H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, $J = 7.6$ Hz, 1H), 7.40 (t, $J = 7.6$ Hz, 1H), 7.35 (d, $J = 7.6$ Hz, 1H), 7.33 – 7.18 (m, 5H), 3.28 – 3.22 (m, 1H), 3.18 (dd, $J = 16.8, 3.2$ Hz, 1H), 2.92 (dd, $J = 16.8, 10.4$ Hz, 1H), 2.66 – 2.58 (m, 2H), 2.51 (s, 3H), 2.46 (s, 3H), 1.44 (s, 3H), 1.18 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.7, 171.8, 138.0, 138.0, 136.5, 135.4, 132.1, 131.4, 131.0, 128.9, 128.3, 125.8, 125.6, 73.8, 44.6, 43.1, 42.6, 28.8, 22.7, 21.3, 20.9. HRMS(ESI): calcd. for $C_{22}H_{26}NO^+$ $[M+H]^+$ 320.2009; found: 320.2004.

1-(3,4-Dimethylphenyl)-2-(5-(3,4-dimethylphenyl)-2,2-dimethyl-3,4-dihydro-2H-pyrrol-3-yl)ethan-1-one (5n): Petroleum ether/ethyl acetate = 4:1; yellow oil (120 mg, 69%); 1H NMR (400 MHz, $CDCl_3$) δ 7.76 (s, 1H), 7.72 (d, $J = 7.6$ Hz, 1H), 7.63 (s, 1H), 7.48 (d, $J = 7.6$ Hz, 1H), 7.23 (d, $J = 8.0$ Hz, 1H), 7.14 (d, $J = 8.0$ Hz, 1H), 3.40 – 3.30 (m, 1H), 3.21 (dd, $J = 16.4, 3.2$ Hz, 1H), 2.94 (dd, $J = 16.4, 10.4$ Hz, 1H), 2.65 – 2.55 (m, 2H), 2.33 (s, 6H), 2.27 (s, 6H), 1.44 (s, 3H), 1.19 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.7, 169.9, 142.8, 139.4, 137.1, 136.7, 134.8, 132.3, 129.9, 129.6, 129.2, 128.5, 125.8, 125.3, 73.3, 43.0, 41.1, 39.5, 28.8, 22.8, 20.1, 19.9, 19.8, 19.7. HRMS(ESI): calcd. for $C_{24}H_{30}NO^+$ $[M+H]^+$ 348.2322; found: 348.2318.

1-(3,5-Dimethoxyphenyl)-2-(5-(3,5-dimethoxyphenyl)-2,2-dimethyl-3,4-dihydro-2H-pyrrol-3-yl)ethan-1-one (5o): Petroleum ether/ethyl acetate = 4:1; yellow oil (136 mg, 66%);

^1H NMR (400 MHz, CDCl_3) δ 7.12 (d, $J = 2.4$ Hz, 2H), 6.95 (d, $J = 2.4$ Hz, 2H), 6.67 (t, $J = 2.4$ Hz, 1H), 6.52 (t, $J = 2.4$ Hz, 1H), 3.85 (s, 6H), 3.82 (s, 6H), 3.35 (dd, $J = 15.2, 6.8$ Hz, 1H), 3.20 (dd, $J = 16.8, 3.2$ Hz, 1H), 2.94 (dd, $J = 16.8, 10.4$ Hz, 1H), 2.66 – 2.54 (m, 2H), 1.45 (s, 3H), 1.18 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 199.3, 169.7, 160.9, 160.7, 138.8, 136.7, 106.0, 105.4, 105.2, 103.1, 73.5, 55.6, 55.6, 43.0, 41.3, 39.7, 28.7, 22.7. HRMS(ESI): calcd. for $\text{C}_{24}\text{H}_{30}\text{NO}_5^+$ $[\text{M}+\text{H}]^+$ 412.2118; found: 412.2122.

1-(2,4-Dimethylphenyl)-2-(5-(2,4-dimethylphenyl)-2,2-dimethyl-3,4-dihydro-2H-pyrrol-3-yl)ethan-1-one (5p): Petroleum ether/ethyl acetate = 4:1; yellow oil (38 mg, 24%); ^1H NMR (400 MHz, CDCl_3) δ 7.61 (d, $J = 8.4$ Hz, 1H), 7.25 (d, $J = 8.0$ Hz, 1H), 7.10 – 7.05 (m, 2H), 7.01 (s, 1H), 6.99 (d, $J = 8.0$ Hz, 1H), 3.30 - 3.14 (m, 2H), 2.89 (dd, $J = 16.0, 10.4$ Hz, 1H), 2.63 – 2.56 (m, 2H), 2.49 (s, 3H), 2.43 (s, 3H), 2.36 (s, 3H), 2.31 (s, 3H), 1.43 (s, 3H), 1.16 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.0, 171.7, 142.1, 138.8, 138.6, 136.5, 134.9, 133.0, 131.9, 128.9, 128.5, 126.4, 126.2, 73.7, 44.5, 43.2, 42.2, 28.8, 22.7, 21.6, 21.4, 21.2, 21.0. HRMS(ESI): calcd. for $\text{C}_{24}\text{H}_{30}\text{NO}^+$ $[\text{M}+\text{H}]^+$ 348.2322; found: 348.2317.

1-(3,4-Dichlorophenyl)-2-(5-(3,4-dichlorophenyl)-2,2-dimethyl-3,4-dihydro-2H-pyrrol-3-yl)ethan-1-one (5q): Petroleum ether/ethyl acetate = 4:1; yellow oil (124 mg, 58%); ^1H NMR (400 MHz, CDCl_3) δ 8.06 (d, $J = 1.6$ Hz, 1H), 7.92 (d, $J = 1.6$ Hz, 1H), 7.81 (dd, $J = 8.4, 2.0$ Hz, 1H), 7.62 (dd, $J = 8.4, 1.6$ Hz, 1H), 7.58 (d, $J = 8.4$ Hz, 1H), 7.46 (d, $J = 8.4$ Hz, 1H), 3.38 (dd, $J = 16.0, 7.8$ Hz, 1H), 3.21 (dd, $J = 17.2, 3.6$ Hz, 1H), 2.95 (dd, $J = 17.2, 10.6$ Hz, 1H), 2.69 – 2.60 (m, 1H), 2.54 (dd, $J = 16.4, 9.6$ Hz, 1H), 1.45 (s, 3H), 1.19 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.0, 167.8, 138.0, 136.2, 134.7, 134.4, 133.5, 132.9, 130.9, 130.4, 130.1, 129.4, 127.0, 126.8, 73.8, 42.7, 41.0, 39.5, 28.6, 22.7. HRMS(ESI): calcd. for $\text{C}_{20}\text{H}_{18}\text{Cl}_4\text{NO}^+$ $[\text{M}+\text{H}]^+$ 428.0137; found: 428.0132.

2-(2,2-Dimethyl-5-(naphthalen-2-yl)-3,4-dihydro-2H-pyrrol-3-yl)-1-(naphthalen-2-yl)ethan-1-one (5r): Petroleum ether/ethyl acetate = 4:1; yellow oil (119 mg, 61%); ^1H NMR (400 MHz, CDCl_3) δ 8.52 (s, 1H), 8.15 (s, 1H), 8.08 (t, $J = 8.0$ Hz, 2H), 7.99 (d, $J = 8.0$ Hz, 1H), 7.93 (d, $J = 8.8$ Hz, 1H), 7.90 (d, $J = 8.0$ Hz, 1H), 7.87 – 7.82 (m, 2H), 7.64 – 7.46 (m, 4H), 3.64 – 3.55 (m, 1H), 3.43 (dd, $J = 16.8, 3.2$ Hz, 1H), 3.17 (dd, $J = 16.8, 10.0$ Hz, 1H), 2.84 – 2.71 (m, 2H), 1.53 (s, 3H), 1.29 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 199.6, 169.8, 135.7, 134.4, 134.2, 133.0, 132.6, 132.1, 129.8, 129.6, 128.7, 128.7, 128.6, 128.2, 128.1, 127.8, 127.7, 127.0, 126.9, 126.4, 124.5, 123.8, 73.7, 43.1, 41.2, 39.7, 28.9, 23.0. HRMS(ESI): calcd. for $\text{C}_{28}\text{H}_{26}\text{NO}^+$ $[\text{M}+\text{H}]^+$ 392.2009; found: 392.2014.

Crystal Data

The crystal of **3b** was obtained from CH₂Cl₂/petroleum ether at room temperature. The structure of compound **3b** was assigned by single crystal X-ray analysis. Diffraction data were collected on a Bruker Smart Apex II CCD diffractometer with graphite-monochromated Mo K α ($\lambda = 0.71073$ Å). The crystal data of compound **3b** have been deposited in CCDC with number 2211998.

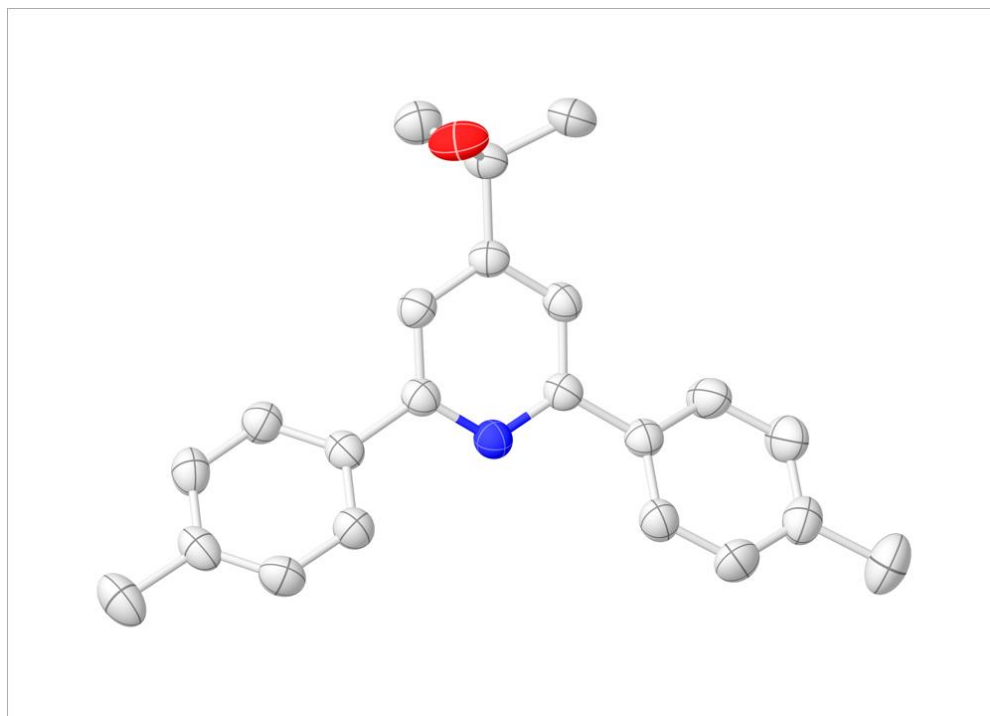


Figure S1 X-ray structure of compound **3b** (CCDC 2211998) (Thermal ellipsoids are drawn at the 50% probability level).

Table C1 Crystal data and structure refinement for **3b**

Identification code	3b
Empirical formula	C ₂₂ H ₂₃ NO
Formula weight	317.41
Temperature/K	296.15
Crystal system	orthorhombic
Space group	P2 ₁ 2 ₁ 2 ₁
a/Å	7.408(4)
b/Å	12.962(7)
c/Å	18.561(11)
α /°	90
β /°	90
γ /°	90

Volume/Å ³	1782.2(17)
Z	4
ρ _{calc} /cm ³	1.183
μ/mm ⁻¹	0.072
F(000)	680
Crystal size/mm ³	0.15 × 0.15 × 0.1
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	3.832 to 61.03
Index ranges	-10 ≤ h ≤ 10, -18 ≤ k ≤ 16, -19 ≤ l ≤ 25
Reflections collected	12522
Independent reflections	4748 [R _{int} = 0.0428, R _{sigma} = 0.0529]
Data/restraints/parameters	4748/0/222
Goodness-of-fit on F ²	1.035
Final R indexes [I >= 2σ (I)]	R ₁ = 0.0495, wR ₂ = 0.1181
Final R indexes [all data]	R ₁ = 0.0752, wR ₂ = 0.1321
Largest diff. peak/hole / e Å ⁻³	0.17/-0.19
Flack parameter	0.4(10)

Table C2 Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for 3b. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij} tensor.

Atom	x	y	z	U(eq)
O2	-7282(2)	-400.9(14)	-6174.4(10)	61.0(5)
N1	-4732(2)	-3135.9(14)	-4631.3(9)	40.0(4)
C1	-4836(3)	-2234.8(17)	-4271.8(12)	38.4(5)
C2	-5085(3)	-1296.3(17)	-4622.2(11)	41.1(5)
C3	-5264(3)	-1258.6(16)	-5358.5(11)	38.4(5)
C4	-5167(3)	-2195.1(17)	-5726.3(12)	42.5(5)
C5	-4892(3)	-3105.4(17)	-5352.9(11)	39.0(5)
C6	-4753(3)	-2269.9(17)	-3474.8(11)	39.6(5)
C7	-5267(3)	-3144.4(19)	-3098.3(13)	47.6(5)
C8	-5305(4)	-3148(2)	-2355.8(14)	55.5(6)
C10	-4317(4)	-1418(2)	-2337.4(14)	59.0(7)
C9	-4853(3)	-2284(2)	-1960.9(13)	53.3(6)
C11	-4240(4)	-1407.6(19)	-3078.6(13)	53.6(6)
C12	-4899(5)	-2278(3)	-1148.7(13)	81.7(10)
C13	-4820(3)	-4107.2(17)	-5743.6(12)	41.0(5)
C14	-4050(3)	-4180.6(19)	-6422.0(12)	48.1(6)
C15	-4028(4)	-5113.7(19)	-6791.6(13)	51.8(6)
C16	-4791(3)	-5993.5(19)	-6501.8(13)	50.4(6)
C17	-5539(4)	-5920(2)	-5822.1(14)	54.6(6)

C18	-5547(3)	-4996.3(18)	-5444.3(12)	48.9(6)
C19	-4811(4)	-6997(2)	-6918.0(17)	69.2(8)
C20	-5665(3)	-260.1(17)	-5770.3(12)	43.2(5)
C21	-5864(4)	669.6(18)	-5277.6(14)	59.0(7)
C22	-4194(4)	-52(2)	-6317.9(13)	56.8(7)

Table C3 Anisotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 3b. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^*U_{11}+2hka^*b^*U_{12}+\dots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O2	56.2(10)	56.8(11)	70.1(12)	22.5(9)	-20.3(9)	-7.8(9)
N1	41.9(9)	38.5(10)	39.8(10)	-1.6(8)	-2.6(8)	1.0(7)
C1	37.0(10)	37.7(12)	40.4(11)	-0.3(9)	-3.9(9)	0.3(9)
C2	43.5(11)	37.1(12)	42.6(12)	-4.5(9)	-3.8(9)	0.2(9)
C3	36.0(10)	37.8(12)	41.3(12)	2.7(9)	-3.5(9)	-0.2(8)
C4	47.0(12)	43.7(13)	36.7(11)	0.6(10)	-3.8(9)	0.0(10)
C5	38.5(10)	37.6(12)	41.1(12)	-1.1(9)	-2.4(9)	0.9(9)
C6	39.1(11)	40.0(12)	39.7(11)	-0.2(10)	-3.4(9)	3.7(9)
C7	53.5(12)	43.4(13)	45.9(13)	-4.2(11)	4.7(11)	-2.6(11)
C8	62.6(15)	53.6(16)	50.2(14)	7.9(12)	11.5(12)	-5.0(12)
C10	77.7(18)	54.4(17)	45.0(14)	-7.3(12)	-15.8(13)	-0.7(13)
C9	52.8(14)	65.5(17)	41.5(12)	-3.1(12)	0.0(11)	7.0(12)
C11	71.5(16)	44.1(14)	45.2(14)	1.5(12)	-12.7(12)	-3.9(12)
C12	95(2)	107(3)	43.6(15)	-1.7(16)	4.8(15)	-4(2)
C13	41.7(11)	39.1(12)	42.1(12)	-3.4(10)	-7.2(9)	3.5(9)
C14	54.7(13)	46.3(14)	43.4(13)	-0.4(10)	1.0(10)	-4.4(11)
C15	56.8(14)	55.8(16)	42.9(13)	-10.2(12)	0.0(11)	2.6(12)
C16	51.3(13)	41.8(14)	58.1(14)	-11.6(12)	-9.9(11)	6.9(10)
C17	65.4(16)	39.2(14)	59.2(16)	1.3(12)	1.2(12)	-5.4(12)
C18	57.4(14)	43.1(14)	46.3(13)	-1.7(11)	2.3(11)	-2.0(10)
C19	73.9(18)	52.3(17)	81(2)	-21.8(15)	-6.9(16)	5.8(14)
C20	45.7(12)	35.9(13)	48.0(12)	3.1(10)	-5.6(10)	-1.7(9)
C21	72.8(17)	39.4(14)	64.8(17)	5.4(12)	2.5(14)	5.8(11)
C22	61.7(15)	51.7(15)	56.9(15)	5.8(13)	5.1(13)	-6.2(12)

Table C4 Bond Lengths for 3b.

Atom	Atom	Length/ \AA	Atom	Atom	Length/ \AA
O2	C20	1.425(3)	C8	C9	1.379(4)
N1	C1	1.347(3)	C10	C9	1.380(4)

N1	C5	1.345(3)		C10	C11	1.377(3)
C1	C2	1.392(3)		C9	C12	1.508(3)
C1	C6	1.481(3)		C13	C14	1.386(3)
C2	C3	1.374(3)		C13	C18	1.388(3)
C3	C4	1.394(3)		C14	C15	1.391(3)
C3	C20	1.532(3)		C15	C16	1.382(4)
C4	C5	1.383(3)		C16	C17	1.381(4)
C5	C13	1.488(3)		C16	C19	1.513(3)
C6	C7	1.385(3)		C17	C18	1.388(3)
C6	C11	1.391(3)		C20	C21	1.520(3)
C7	C8	1.378(3)		C20	C22	1.514(3)

Table C5 Bond Angles for 3b

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C5	N1	C1	117.56(19)	C8	C9	C12	122.0(3)
N1	C1	C2	122.26(19)	C10	C9	C12	120.6(3)
N1	C1	C6	117.75(19)	C10	C11	C6	120.6(2)
C2	C1	C6	119.94(19)	C14	C13	C5	121.2(2)
C3	C2	C1	120.6(2)	C14	C13	C18	117.8(2)
C2	C3	C4	116.8(2)	C18	C13	C5	121.0(2)
C2	C3	C20	123.0(2)	C13	C14	C15	120.9(2)
C4	C3	C20	120.08(19)	C16	C15	C14	121.4(2)
C5	C4	C3	120.3(2)	C15	C16	C19	121.0(2)
N1	C5	C4	122.5(2)	C17	C16	C15	117.6(2)
N1	C5	C13	117.1(2)	C17	C16	C19	121.4(2)
C4	C5	C13	120.36(19)	C16	C17	C18	121.5(2)
C7	C6	C1	121.2(2)	C17	C18	C13	120.8(2)
C7	C6	C11	117.8(2)	O2	C20	C3	108.49(17)
C11	C6	C1	121.0(2)	O2	C20	C21	109.7(2)
C8	C7	C6	120.8(2)	O2	C20	C22	105.93(19)
C7	C8	C9	121.6(2)	C21	C20	C3	112.85(19)
C11	C10	C9	121.7(2)	C22	C20	C3	110.26(19)
C8	C9	C10	117.4(2)	C22	C20	C21	109.4(2)

Table C6 Torsion Angles for 3b

A	B	C	D	Angle/°	A	B	C	D	Angle/°
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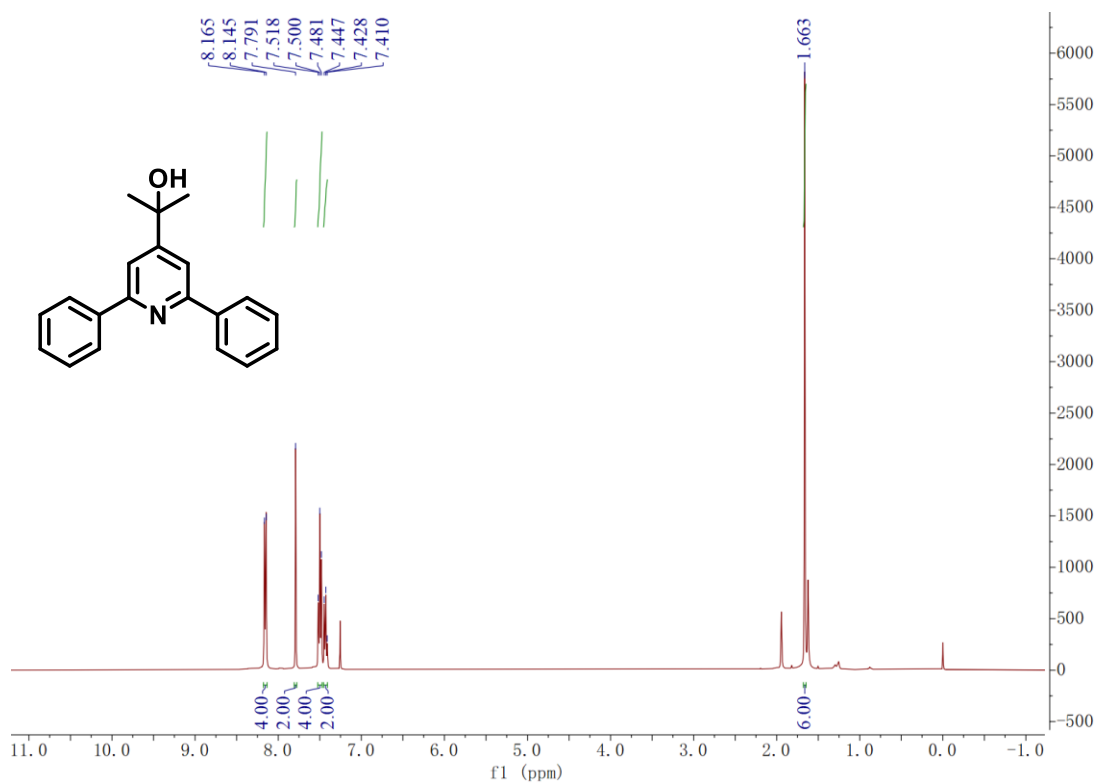
N1	C1	C2	C3	1.0(3)	C4	C5	C13	C18	-143.1(2)
N1	C1	C6	C7	-25.3(3)	C5	N1	C1	C2	-0.5(3)
N1	C1	C6	C11	158.0(2)	C5	N1	C1	C6	177.0(2)
N1	C5	C13	C14	-145.9(2)	C5	C13	C14	C15	-178.3(2)
N1	C5	C13	C18	35.1(3)	C5	C13	C18	C17	177.5(2)
C1	N1	C5	C4	-0.4(3)	C6	C1	C2	C3	-176.4(2)
C1	N1	C5	C13	-178.57(18)	C6	C7	C8	C9	0.7(4)
C1	C2	C3	C4	-0.5(3)	C7	C6	C11	C10	-2.6(4)
C1	C2	C3	C20	175.86(19)	C7	C8	C9	C10	-1.5(4)
C1	C6	C7	C8	-175.5(2)	C7	C8	C9	C12	179.7(3)
C1	C6	C11	C10	174.3(2)	C9	C10	C11	C6	1.7(4)
C2	C1	C6	C7	152.3(2)	C11	C6	C7	C8	1.4(4)
C2	C1	C6	C11	-24.5(3)	C11	C10	C9	C8	0.3(4)
C2	C3	C4	C5	-0.4(3)	C11	C10	C9	C12	179.1(3)
C2	C3	C20	O2	-122.5(2)	C13	C14	C15	C16	0.9(4)
C2	C3	C20	C21	-0.8(3)	C14	C13	C18	C17	-1.6(3)
C2	C3	C20	C22	121.9(2)	C14	C15	C16	C17	-1.6(4)
C3	C4	C5	N1	0.9(3)	C14	C15	C16	C19	178.1(2)
C3	C4	C5	C13	179.0(2)	C15	C16	C17	C18	0.7(4)
C4	C3	C20	O2	53.8(3)	C16	C17	C18	C13	0.8(4)
C4	C3	C20	C21	175.5(2)	C18	C13	C14	C15	0.7(3)
C4	C3	C20	C22	-61.8(3)	C19	C16	C17	C18	-179.0(2)
C4	C5	C13	C14	36.0(3)	C20	C3	C4	C5	-176.88(19)

Table C7 Hydrogen Atom Coordinates ($\text{\AA}\times 10^4$) and Isotropic Displacement Parameters ($\text{\AA}^2\times 10^3$) for 3b

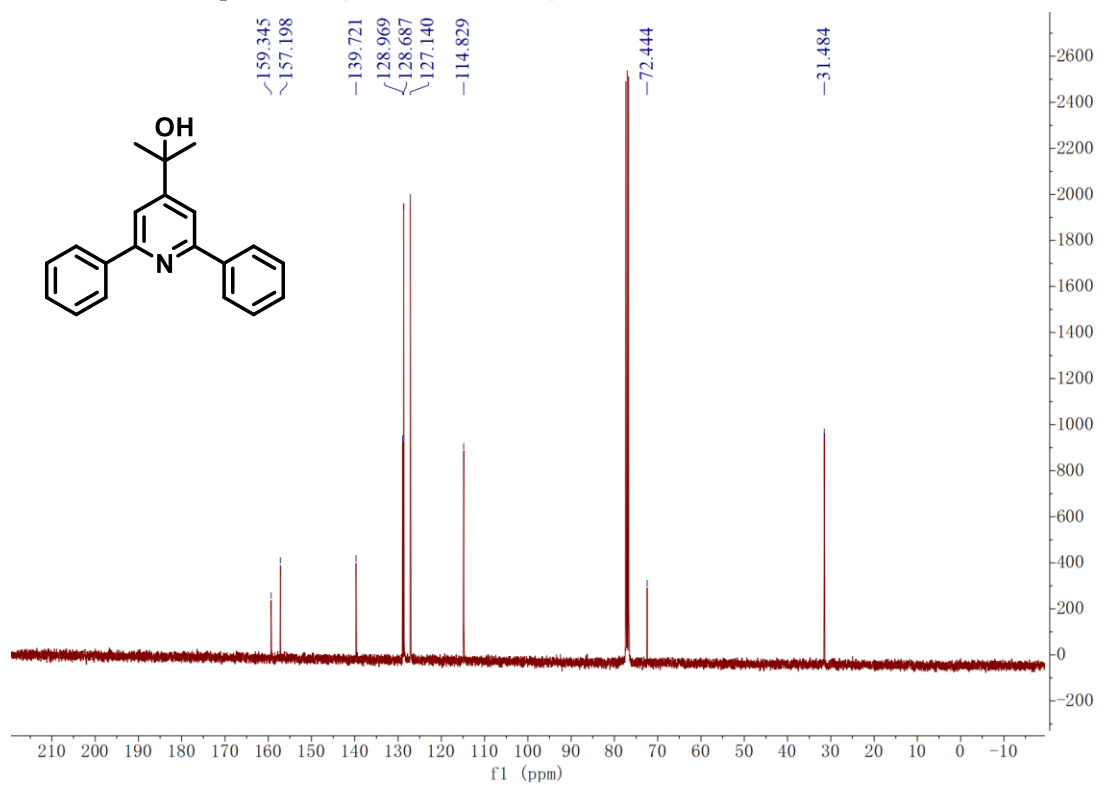
Atom	<i>x</i>	<i>y</i>	<i>z</i>	U(eq)
H2	-8037	-692	-5924	92
H2A	-5131	-689	-4356	49
H4	-5288	-2208	-6225	51
H7	-5591	-3737	-3350	57
H8	-5643	-3747	-2115	67
H10	-4000	-826	-2084	71
H11	-3843	-818	-3316	64
H12A	-3731	-2463	-965	123
H12B	-5779	-2767	-983	123
H12C	-5216	-1601	-981	123
H14	-3541	-3598	-6633	58
H15	-3488	-5146	-7243	62
H17	-6048	-6503	-5613	66

H18	-6046	-4973	-4985	59
H19A	-4044	-6937	-7333	104
H19B	-6021	-7147	-7070	104
H19C	-4380	-7545	-6615	104
H21A	-6786	535	-4926	89
H21B	-6194	1263	-5558	89
H21C	-4739	798	-5037	89
H22A	-4514	540	-6602	85
H22B	-4059	-641	-6627	85
H22C	-3076	79	-6072	85

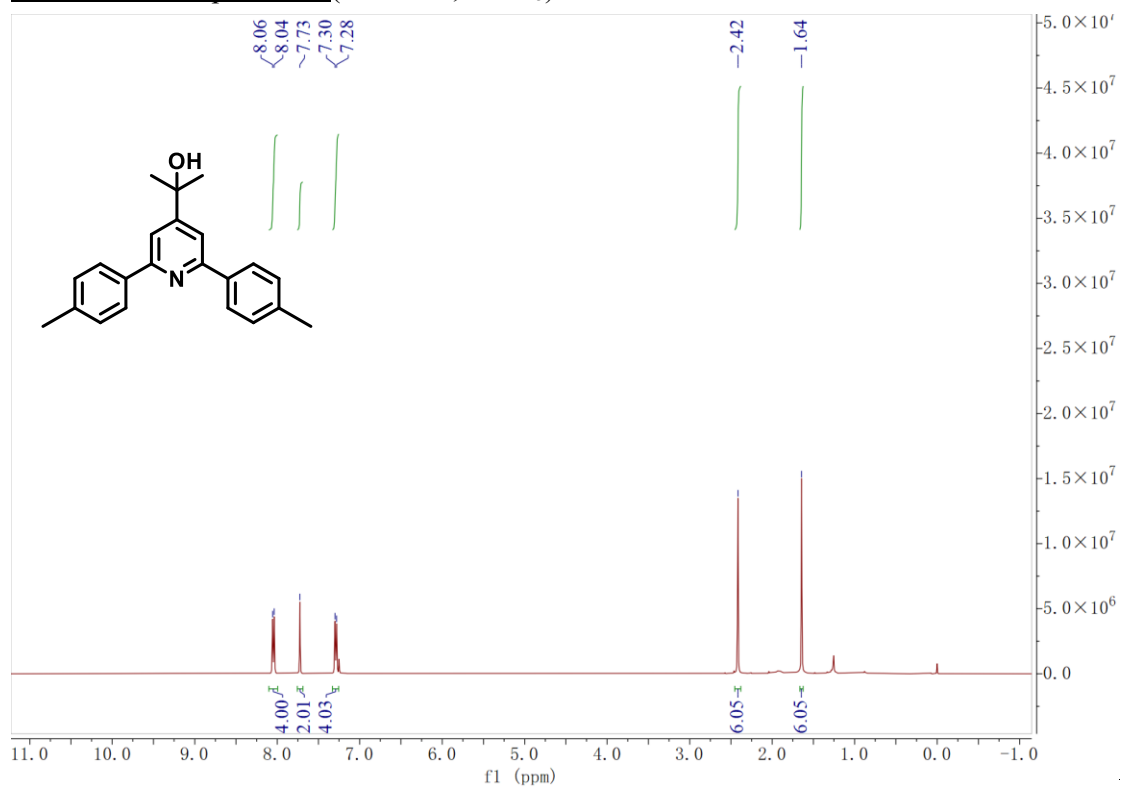
¹H NMR of Compound **3a** (400 MHz, CDCl₃)



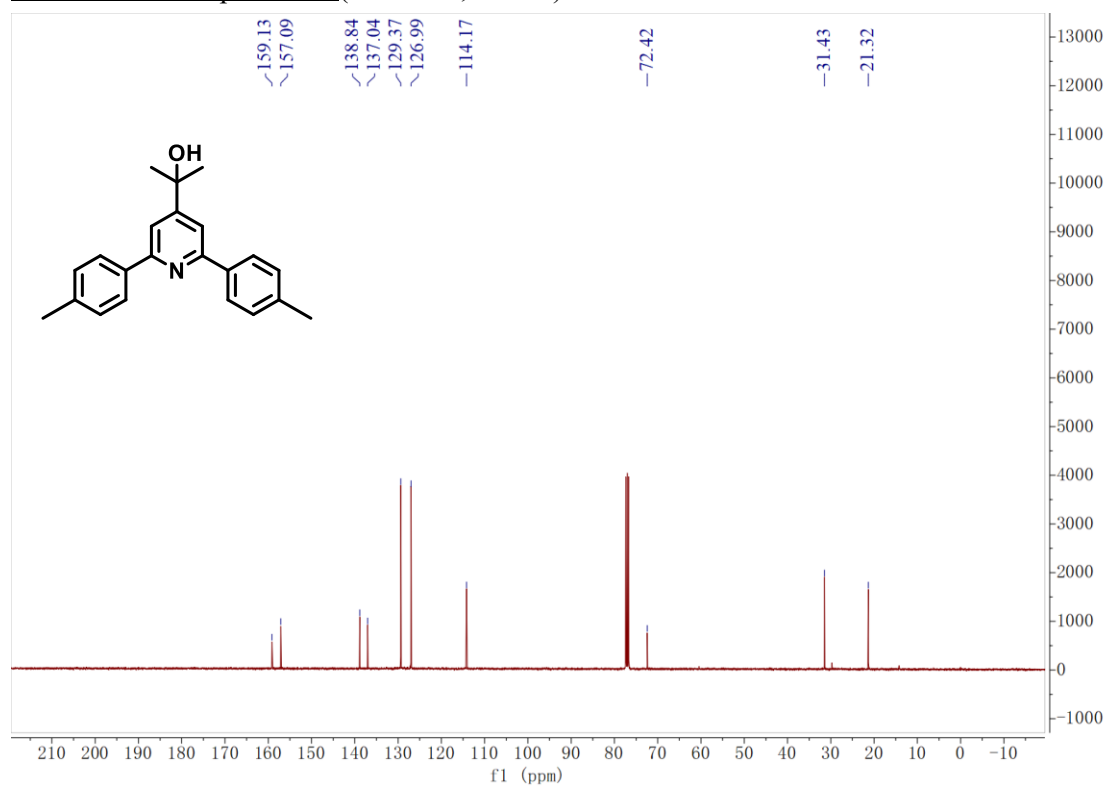
¹³C NMR of Compound **3a** (101 MHz, CDCl₃)



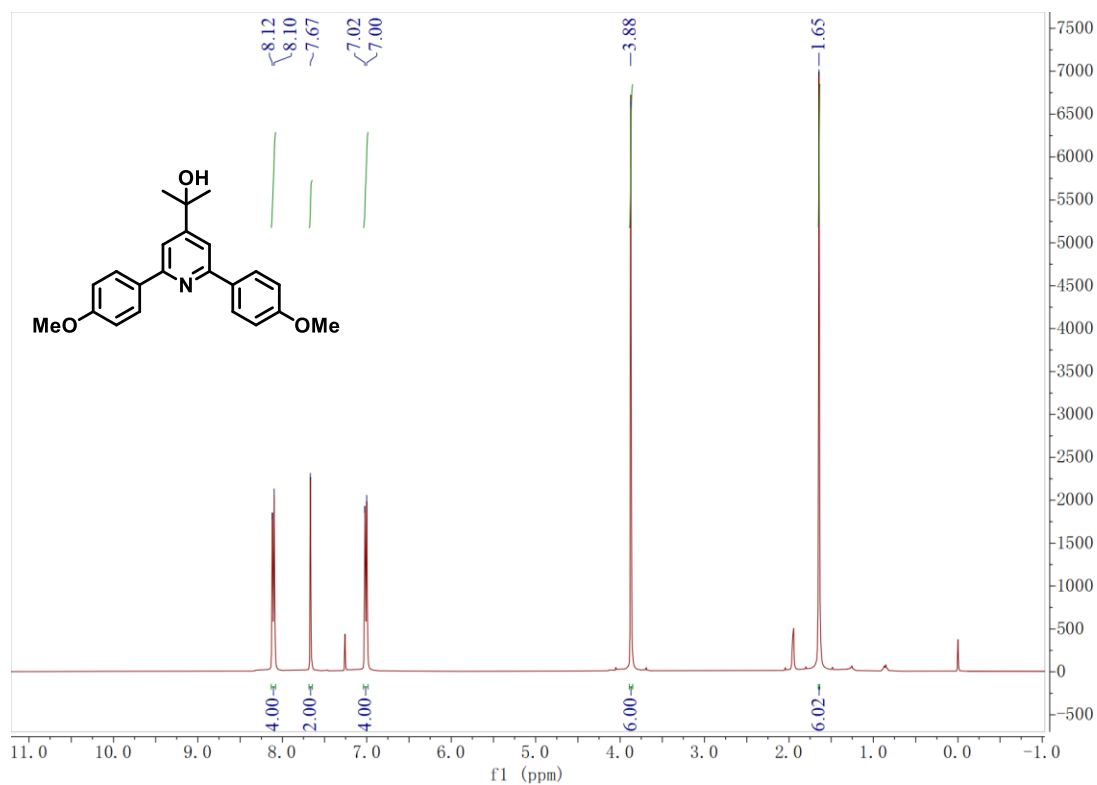
¹H NMR of Compound 3b (400 MHz, CDCl₃)



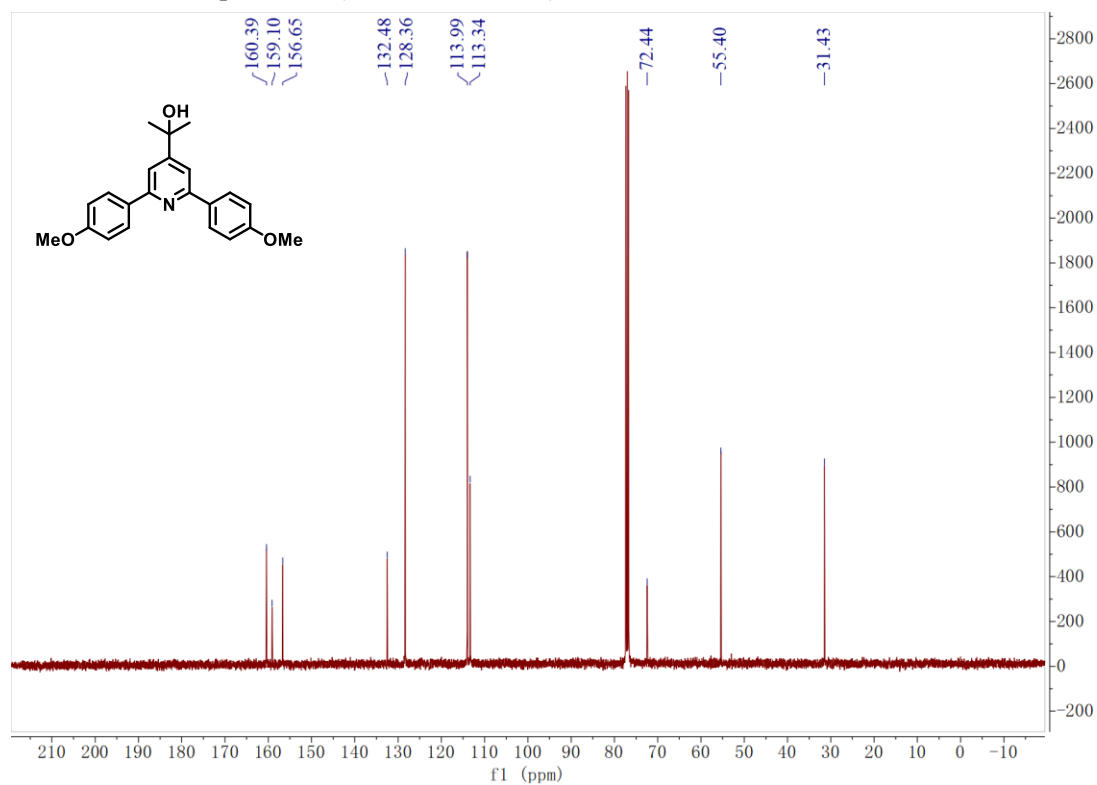
¹³C NMR of Compound 3b (101 MHz, CDCl₃)



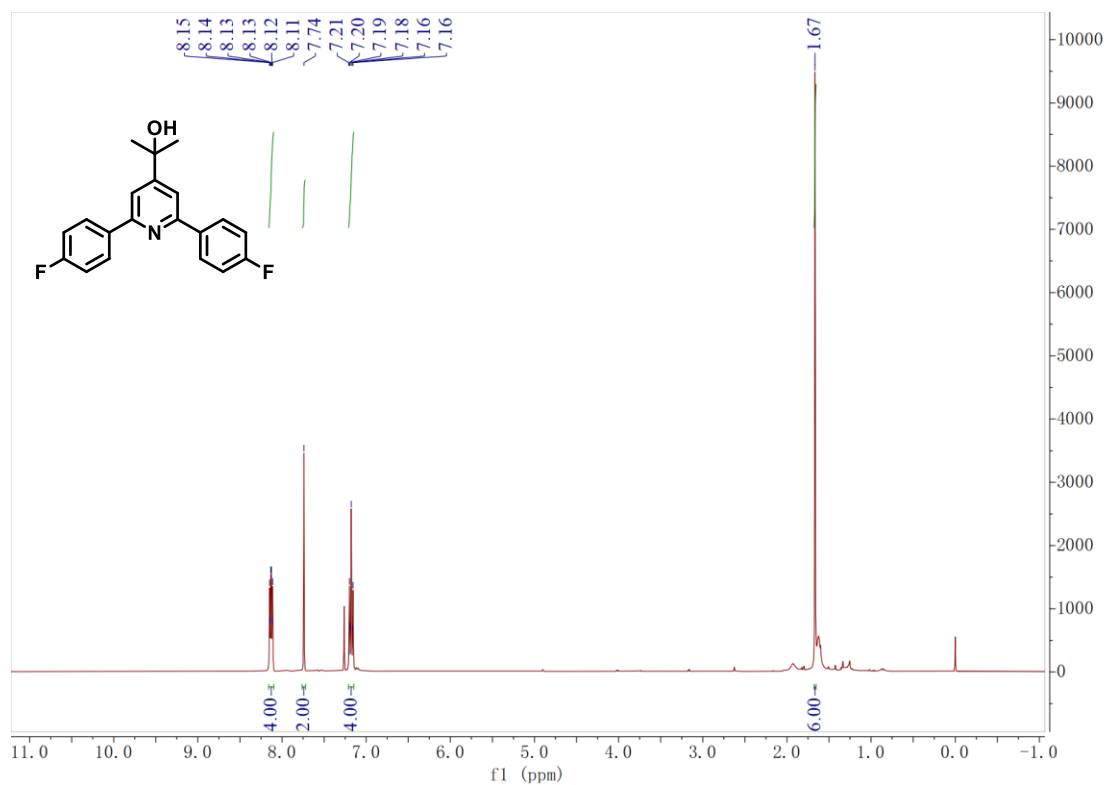
¹H NMR of Compound **3c** (400 MHz, CDCl₃)



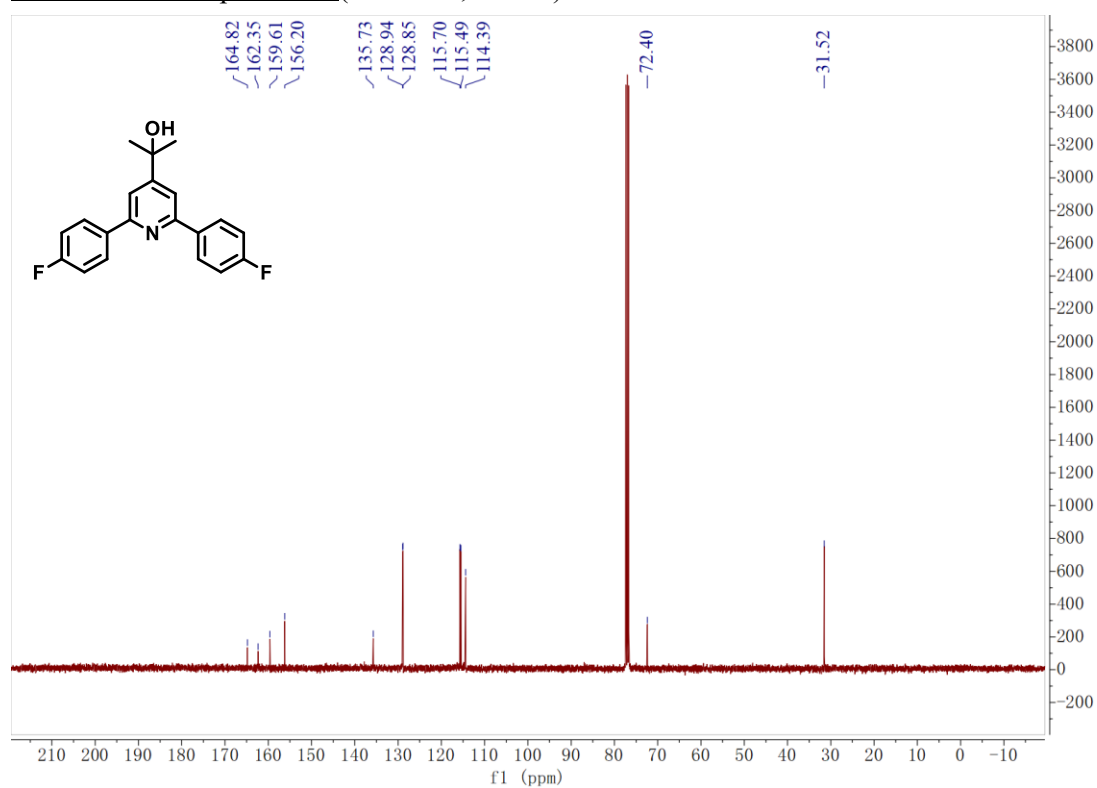
¹³C NMR of Compound **3c** (101 MHz, CDCl₃)



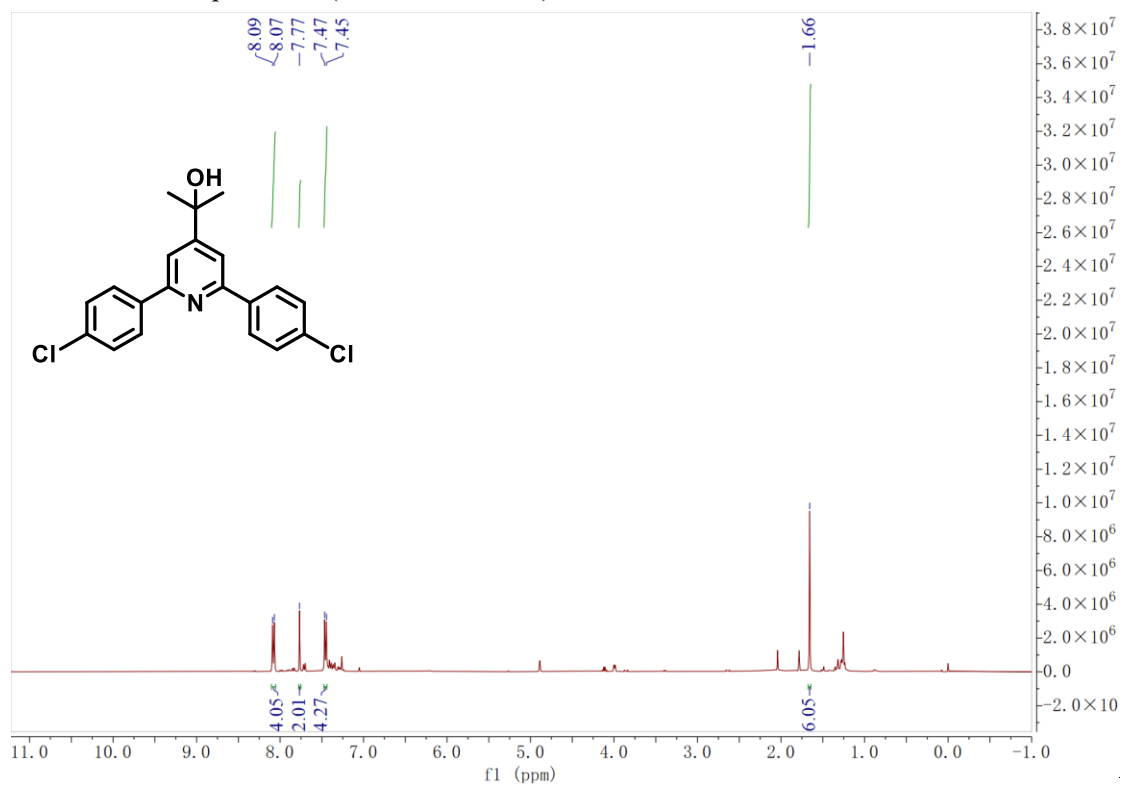
¹H NMR of Compound **3d** (400 MHz, CDCl₃)



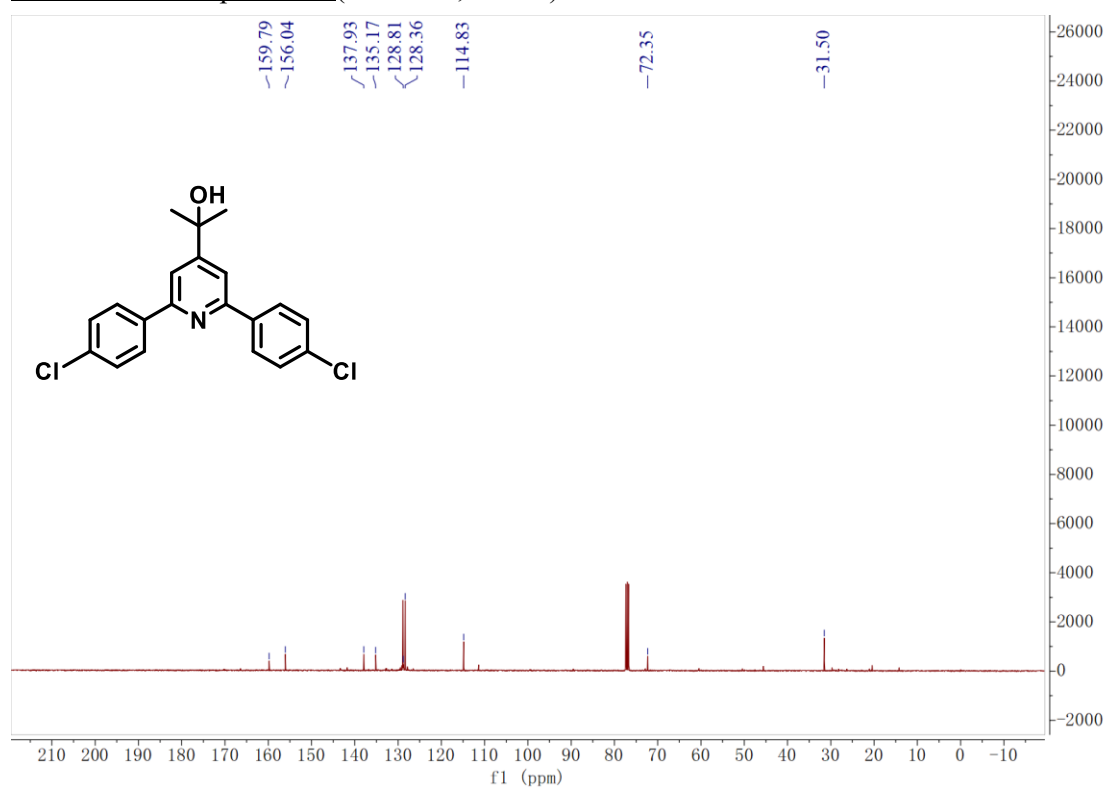
¹³C NMR of Compound **3d** (101 MHz, CDCl₃)



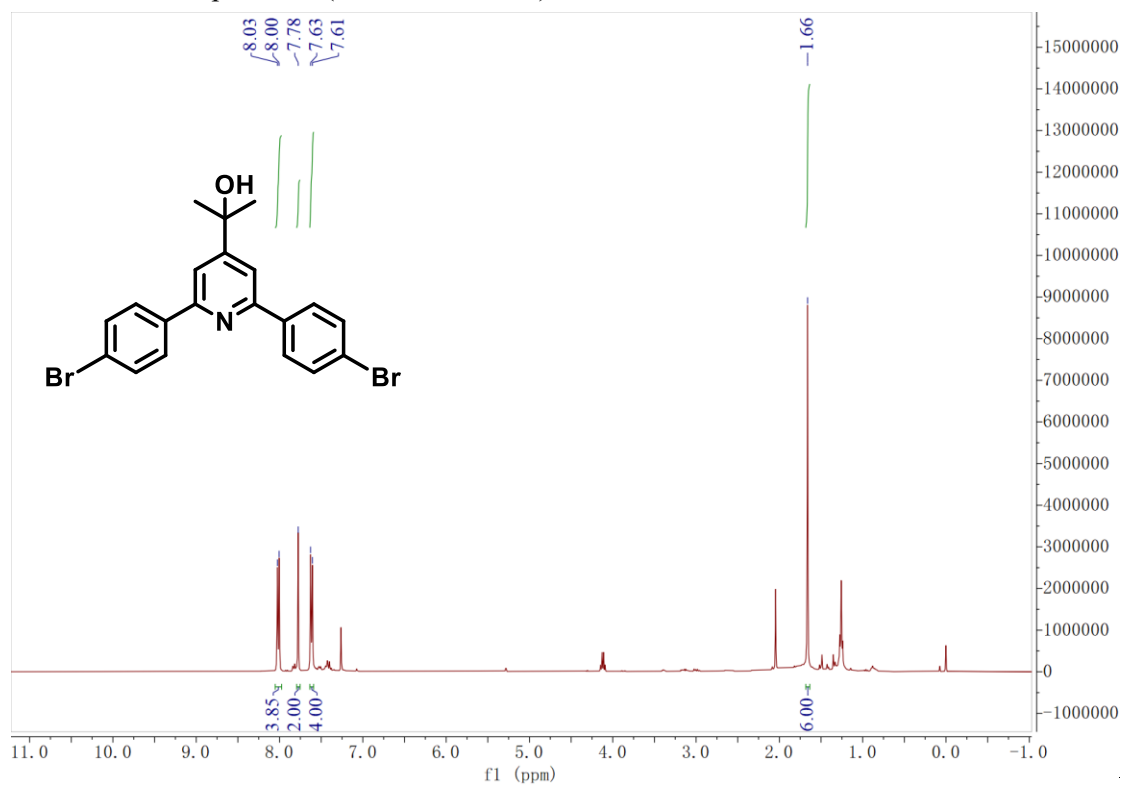
¹H NMR of Compound **3e** (400 MHz, CDCl₃)



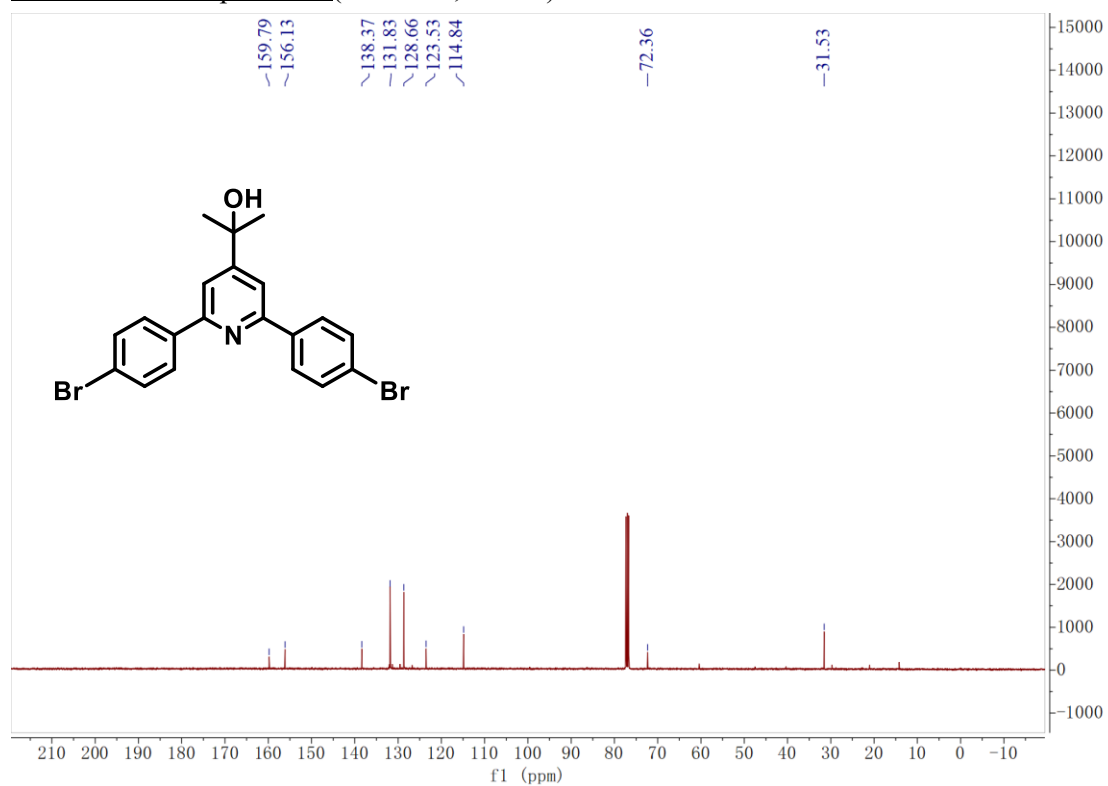
¹³C NMR of Compound **3e** (101 MHz, CDCl₃)



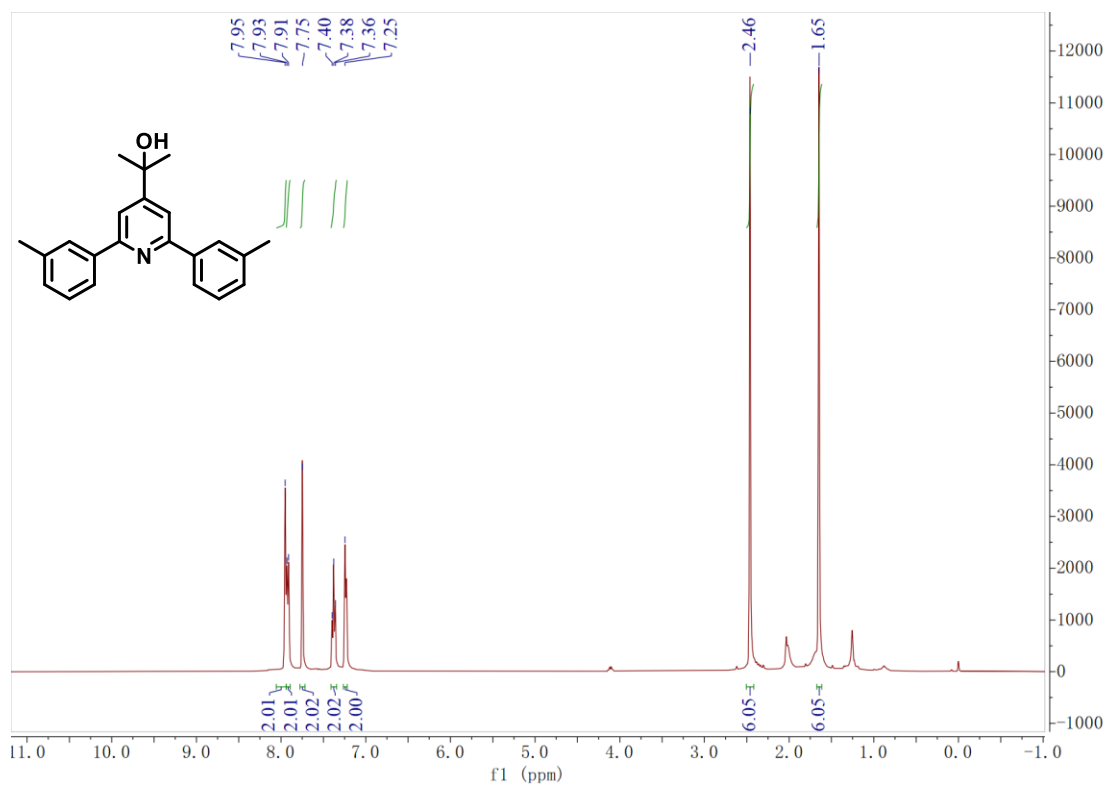
¹H NMR of Compound **3f** (400 MHz, CDCl₃)



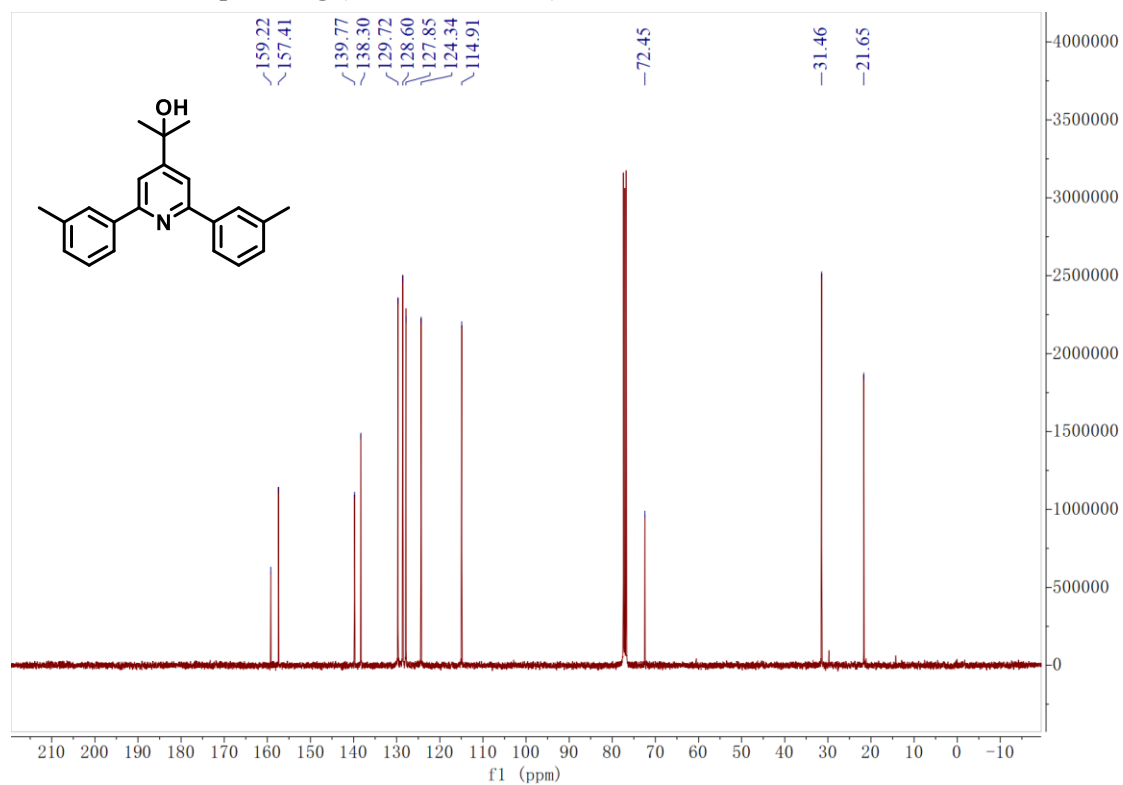
¹³C NMR of Compound **3f** (101 MHz, CDCl₃)



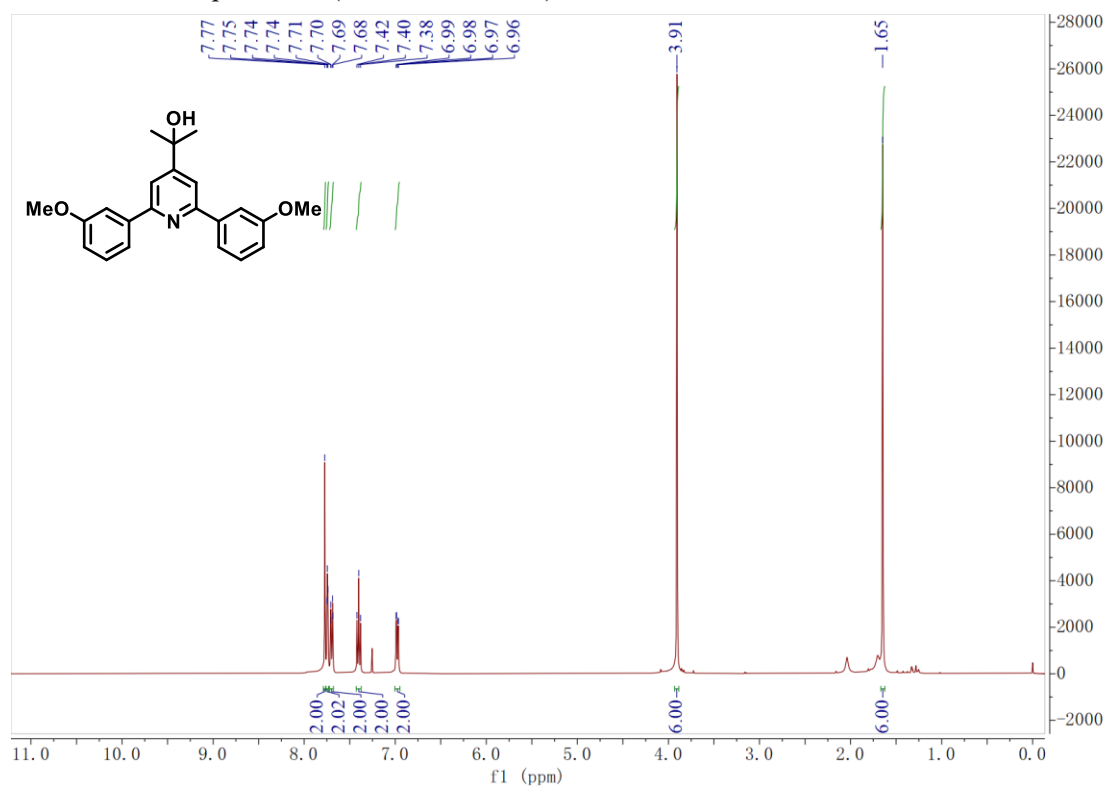
¹H NMR of Compound **3g** (400 MHz, CDCl₃)



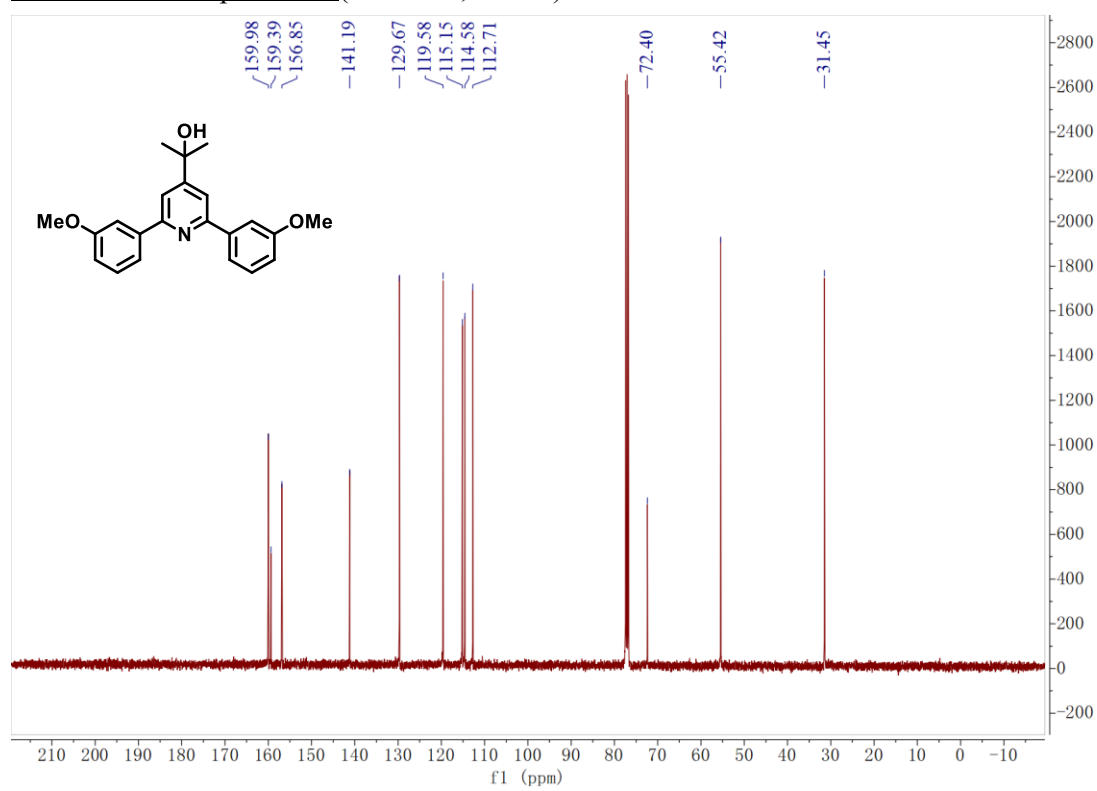
¹³C NMR of Compound **3g** (101 MHz, CDCl₃)



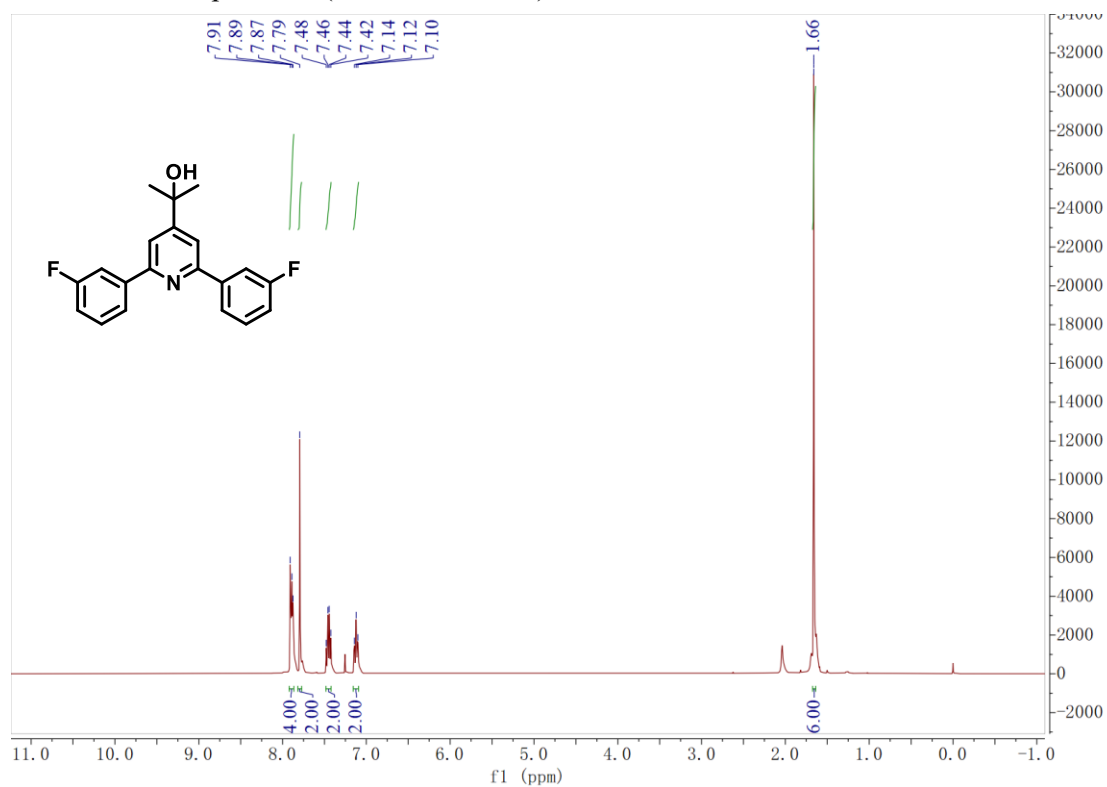
¹H NMR of Compound **3h** (400 MHz, CDCl₃)



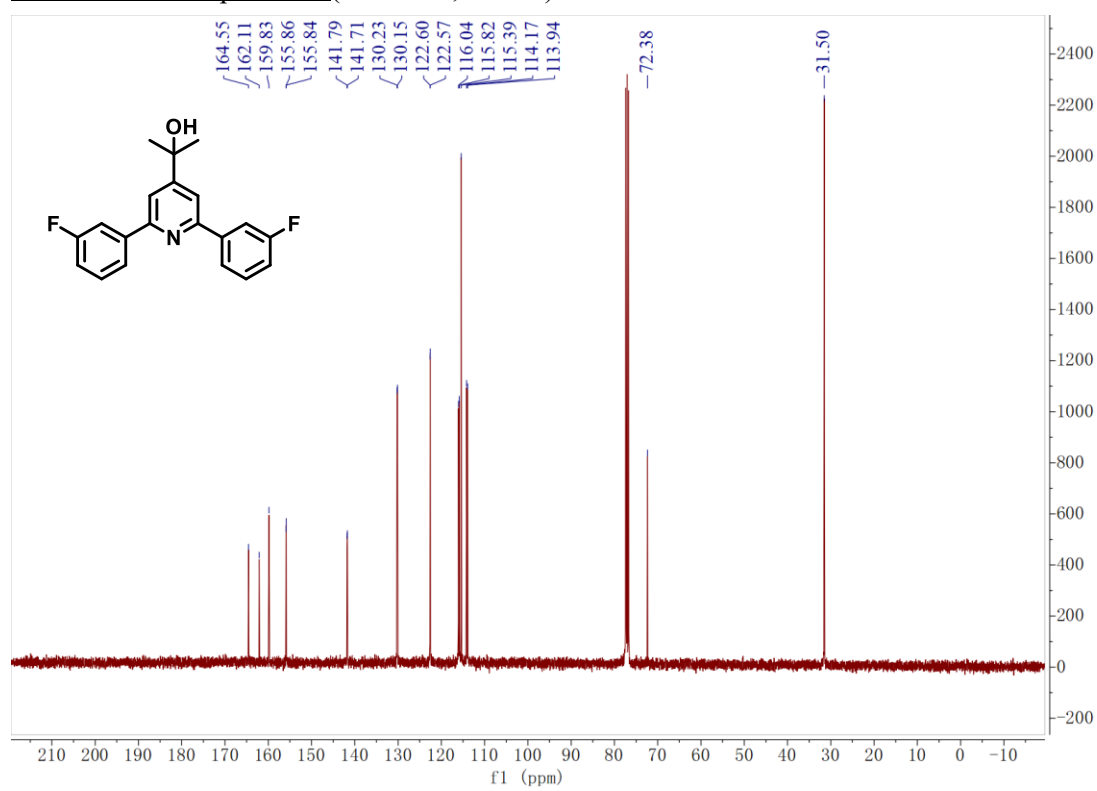
¹³C NMR of Compound **3h** (101 MHz, CDCl₃)



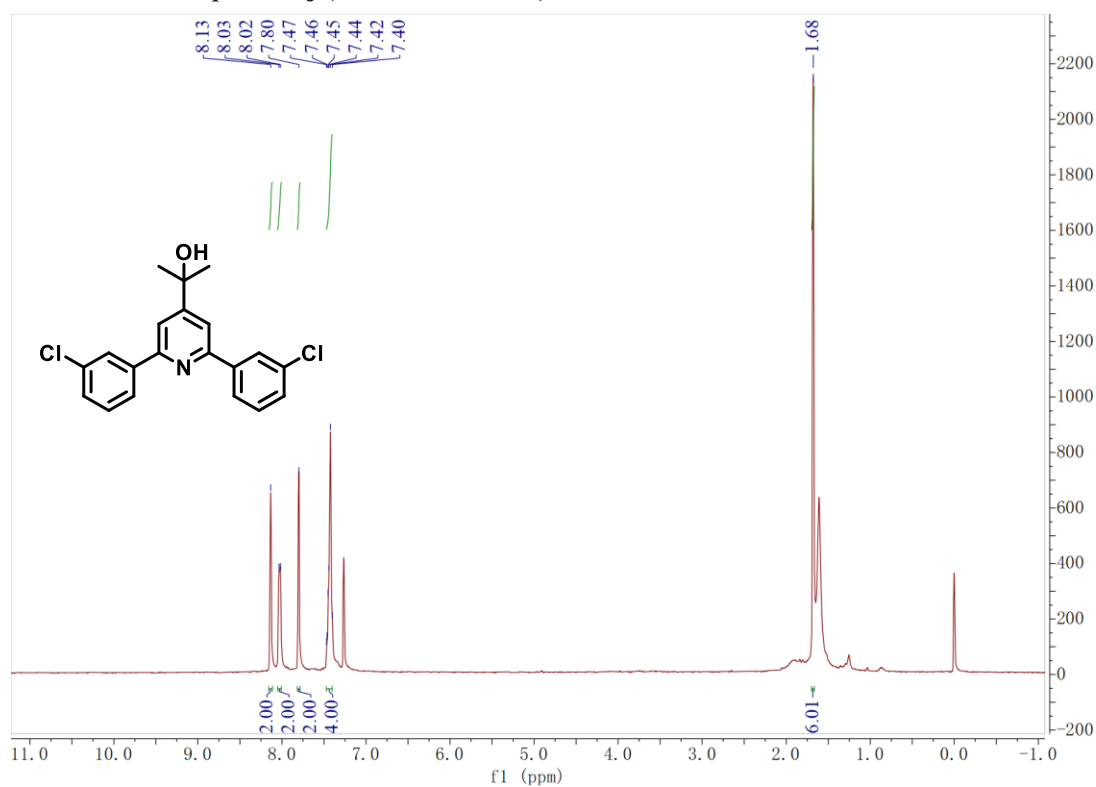
¹H NMR of Compound 3i (400 MHz, CDCl₃)



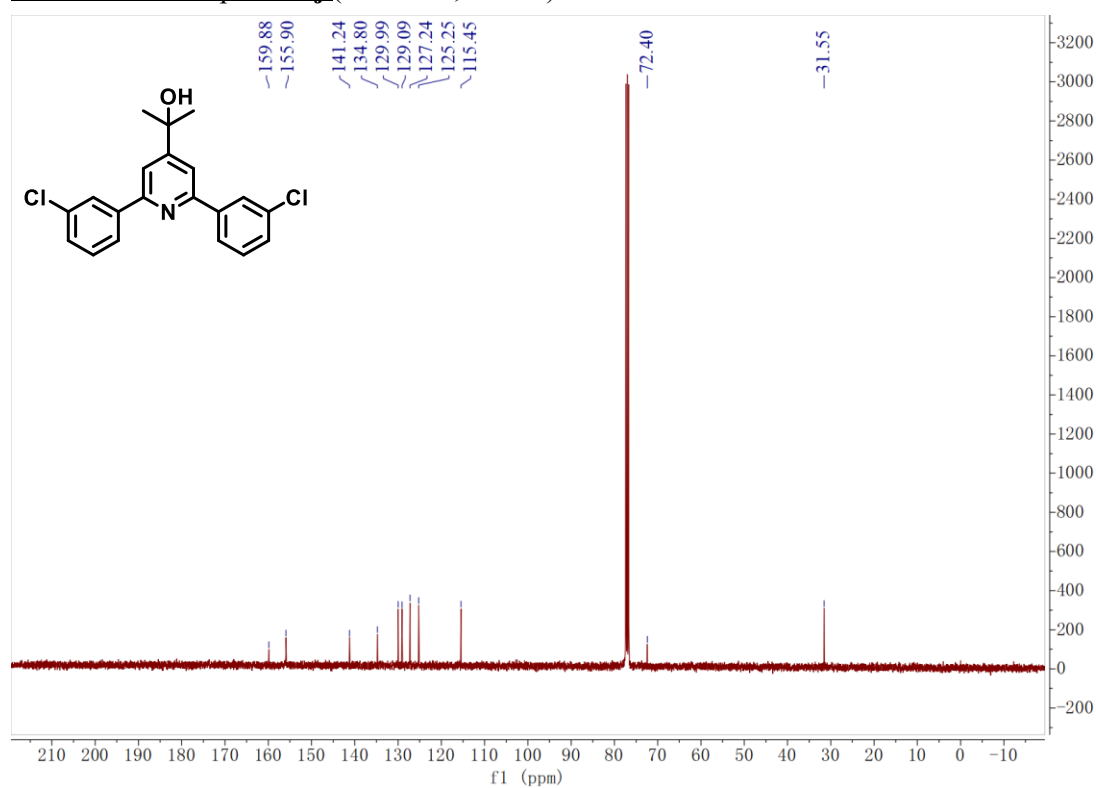
¹³C NMR of Compound 3i (101 MHz, CDCl₃)



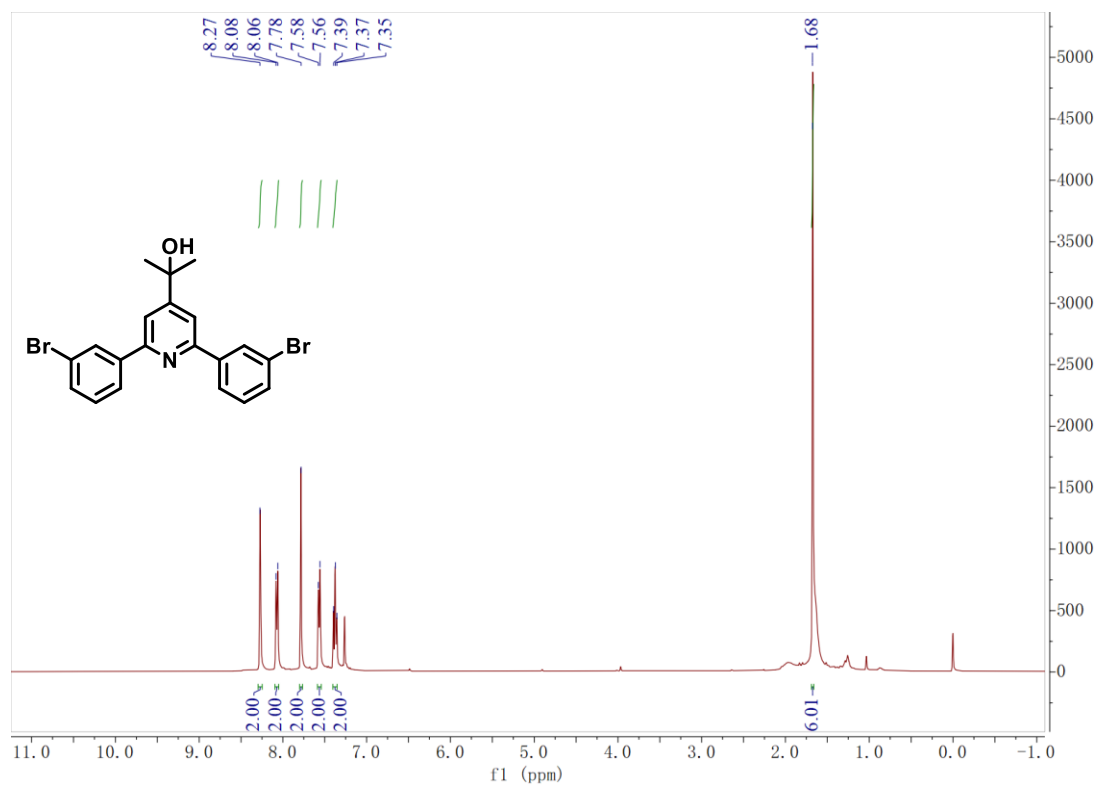
¹H NMR of Compound **3j** (400 MHz, CDCl₃)



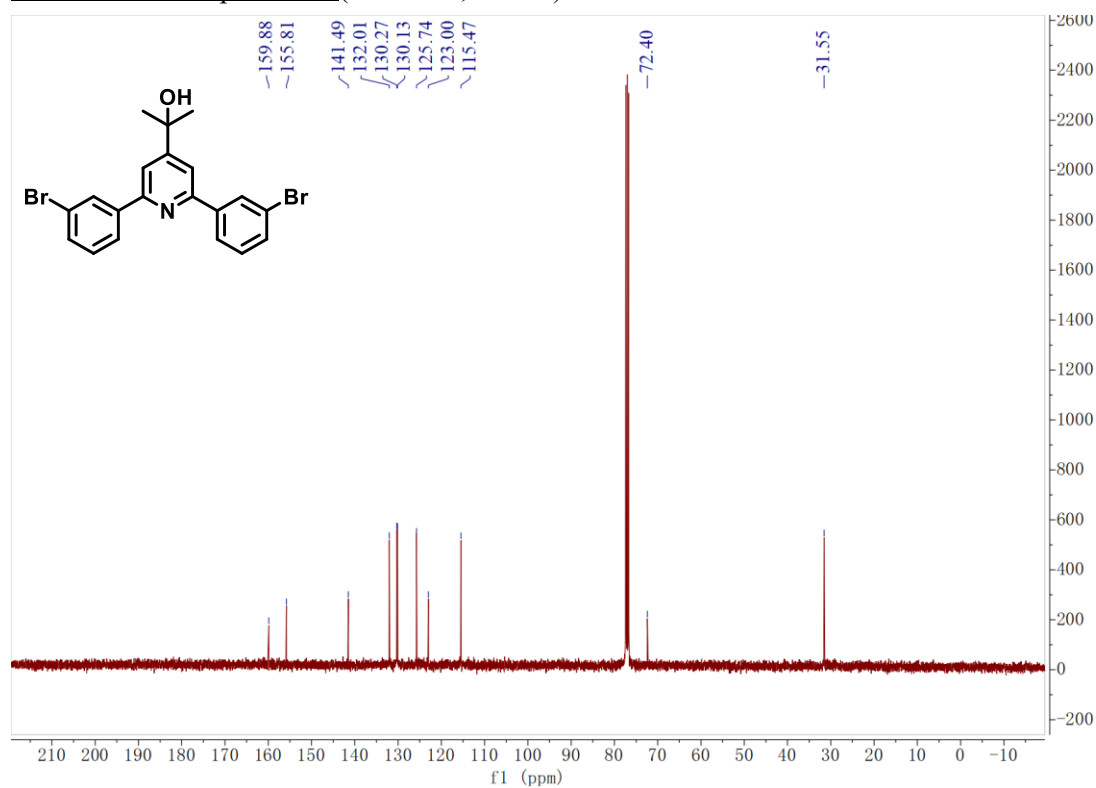
¹³C NMR of Compound **3j** (101 MHz, CDCl₃)



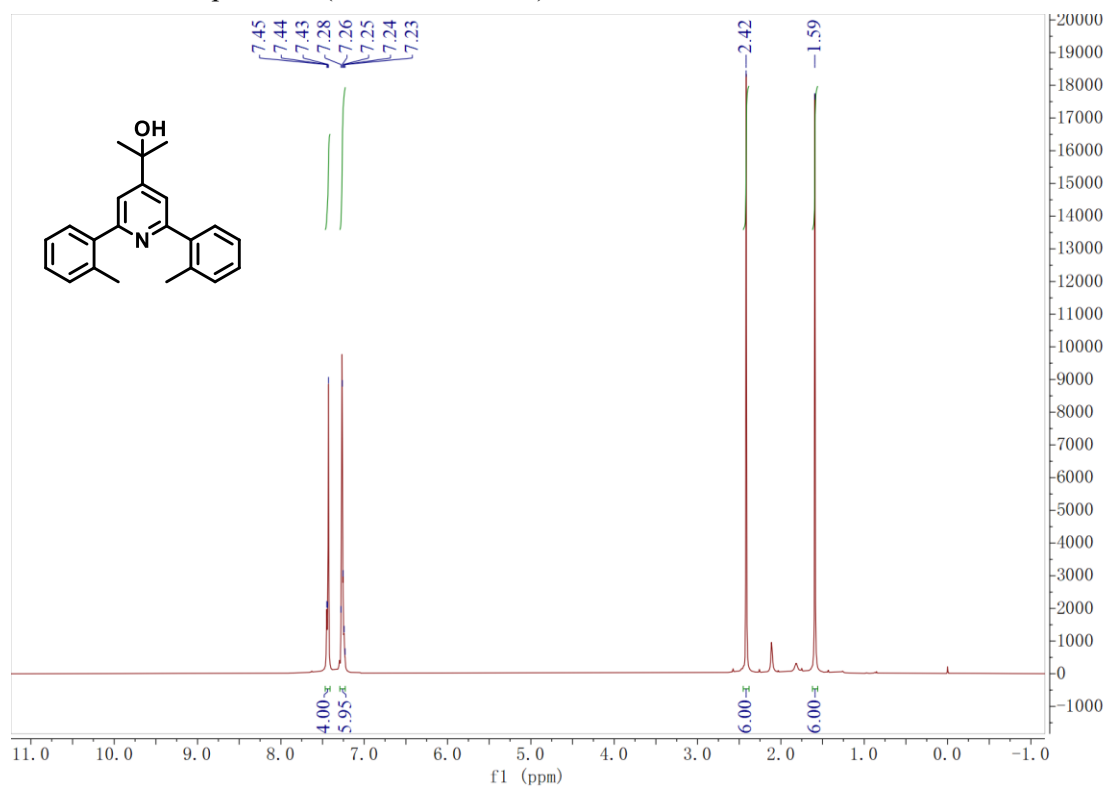
¹H NMR of Compound **3k** (400 MHz, CDCl₃)



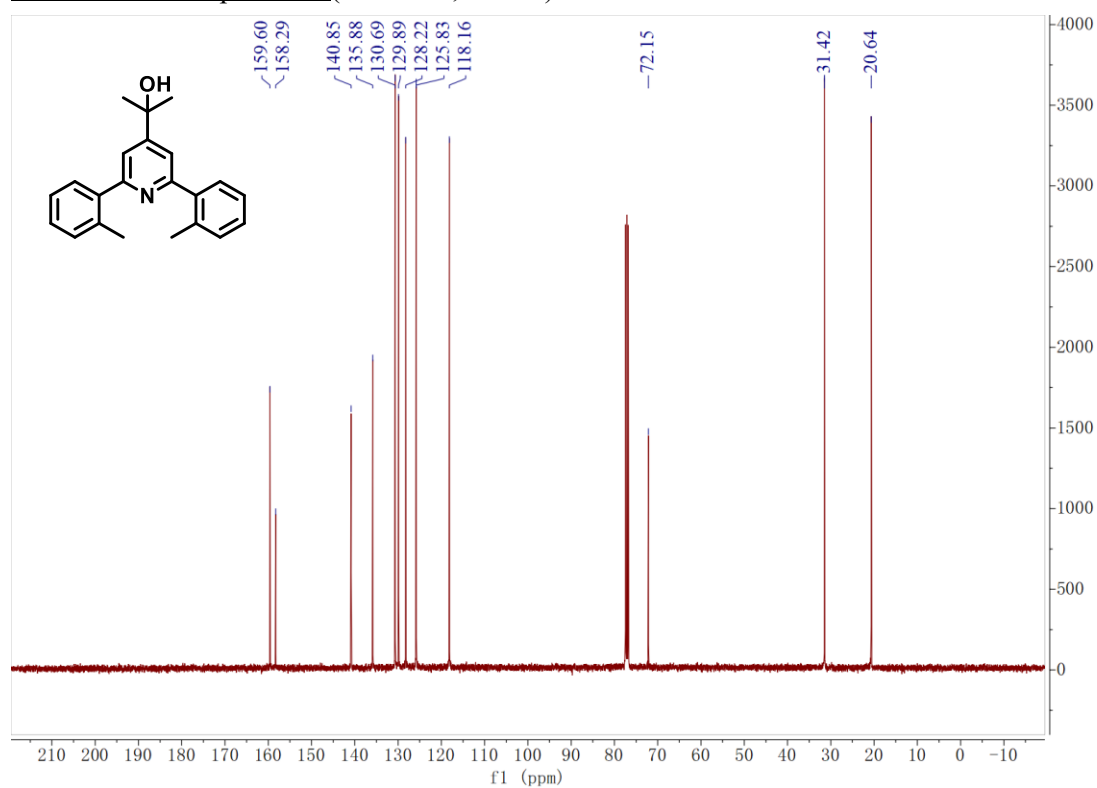
¹³C NMR of Compound **3k** (101 MHz, CDCl₃)



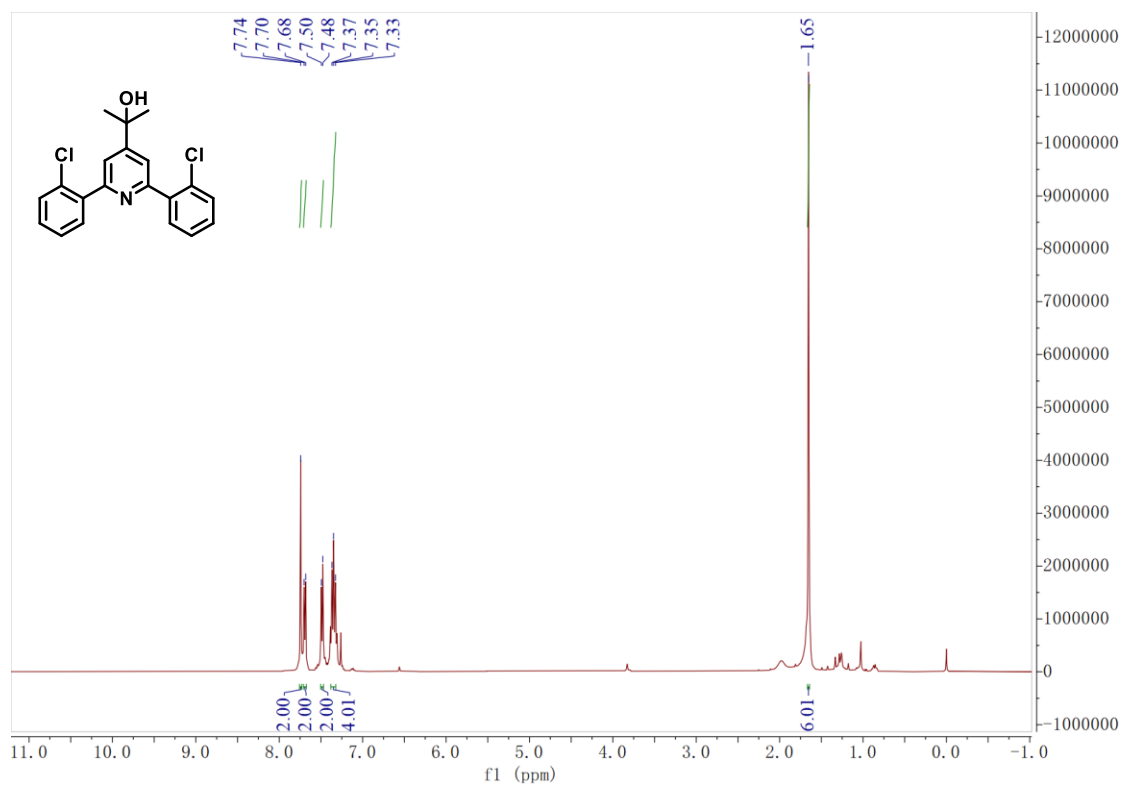
¹H NMR of Compound **31** (400 MHz, CDCl₃)



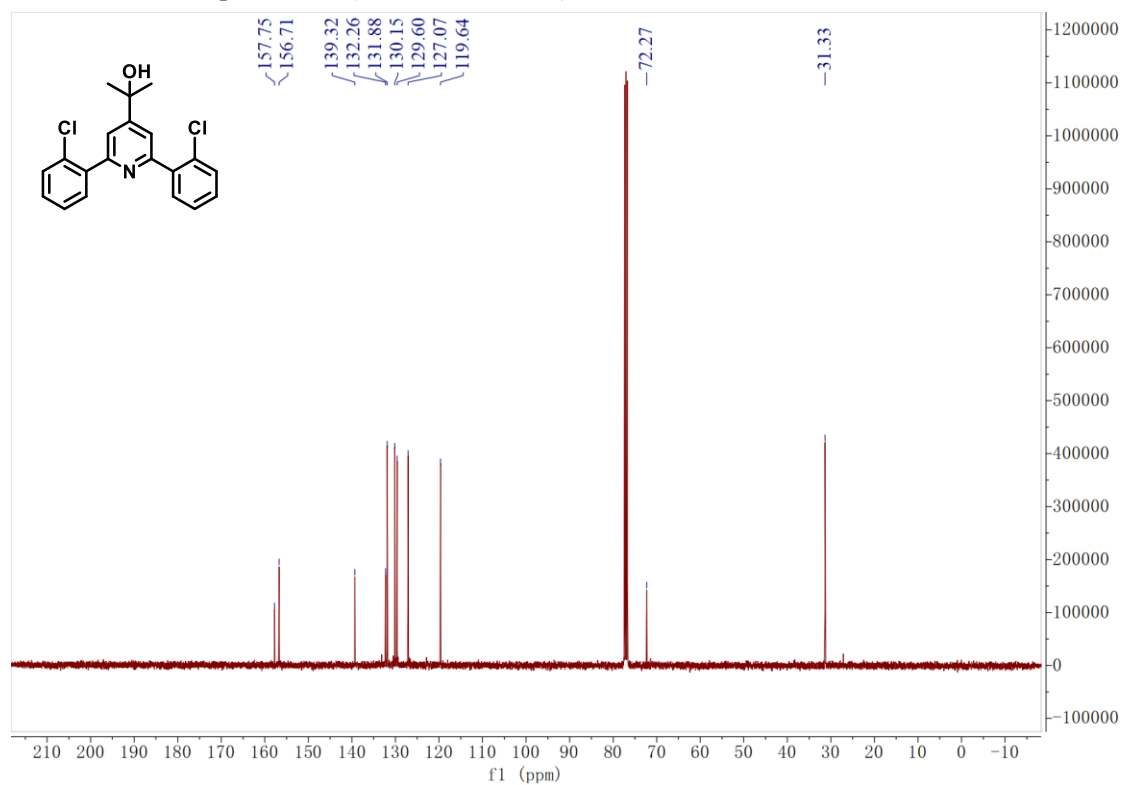
¹³C NMR of Compound **31** (101 MHz, CDCl₃)



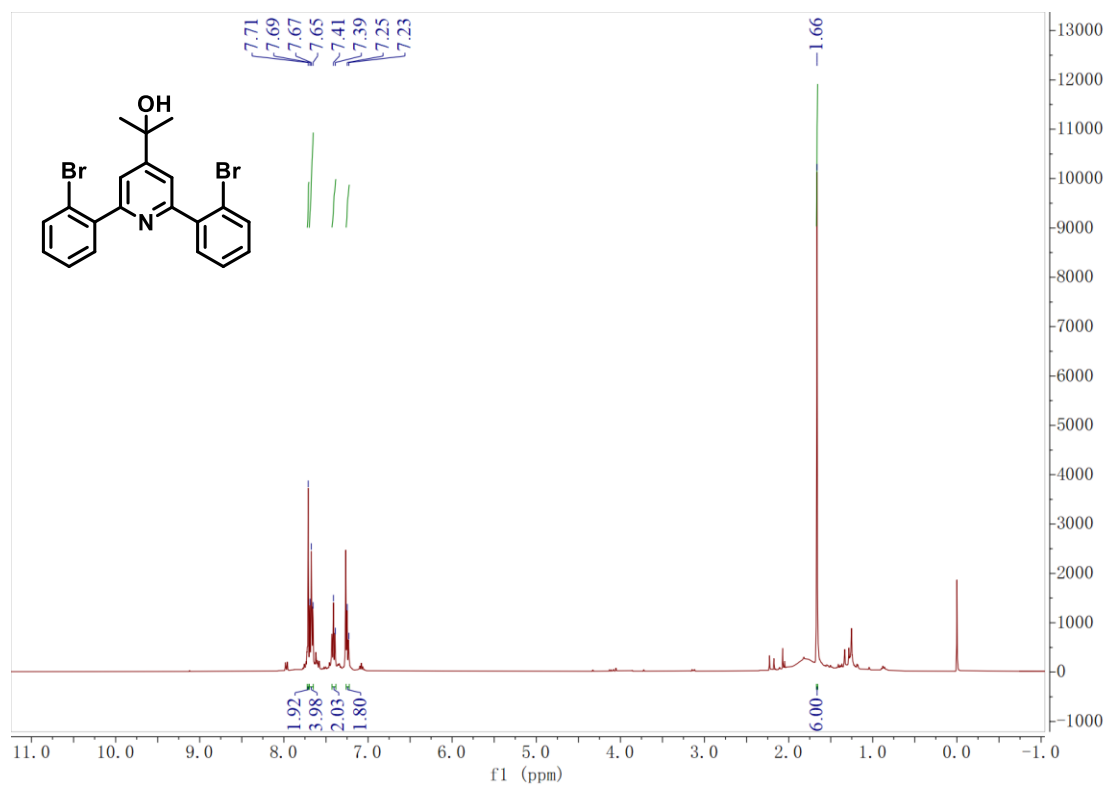
¹H NMR of Compound **3m** (400 MHz, CDCl₃)



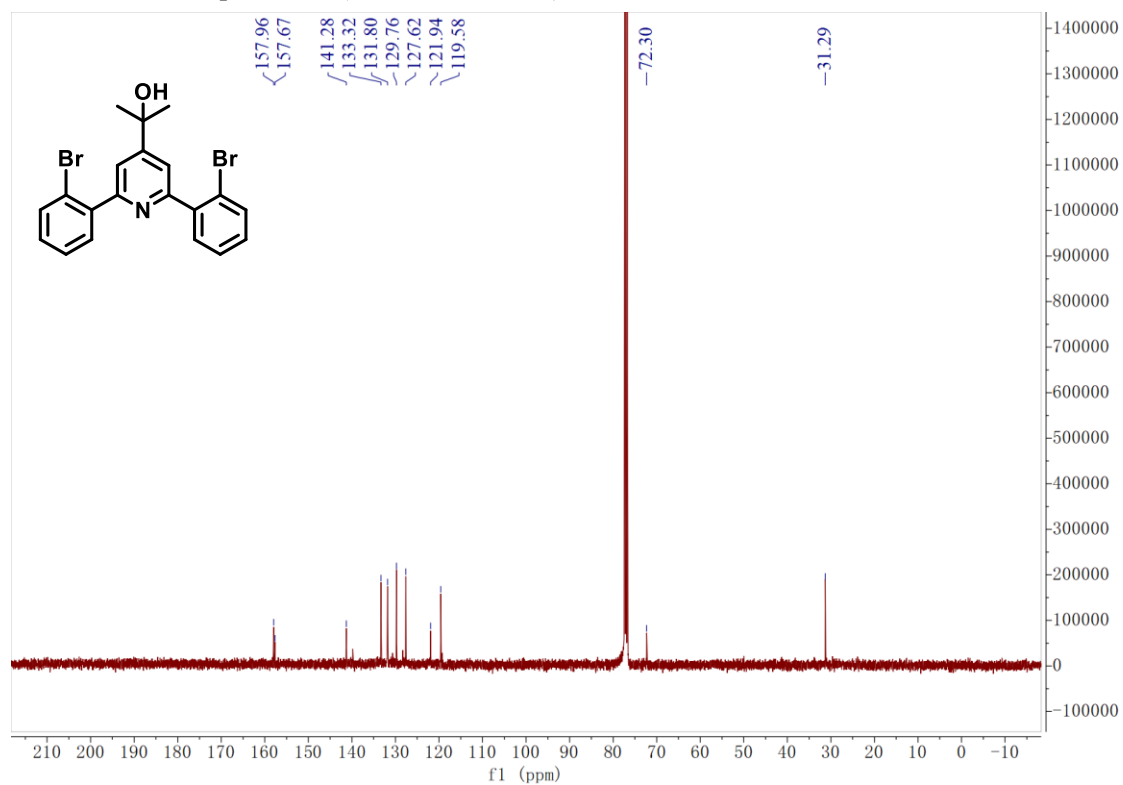
¹³C NMR of Compound **3m** (101 MHz, CDCl₃)



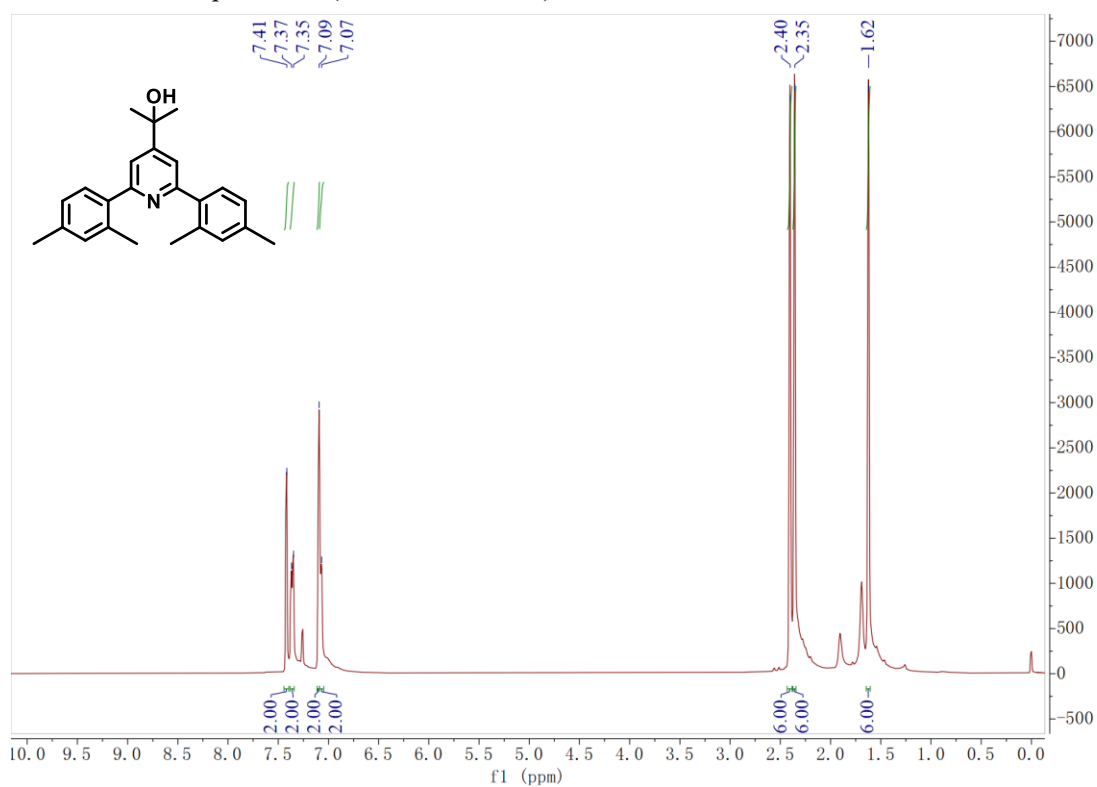
¹H NMR of Compound **3n** (400 MHz, CDCl₃)



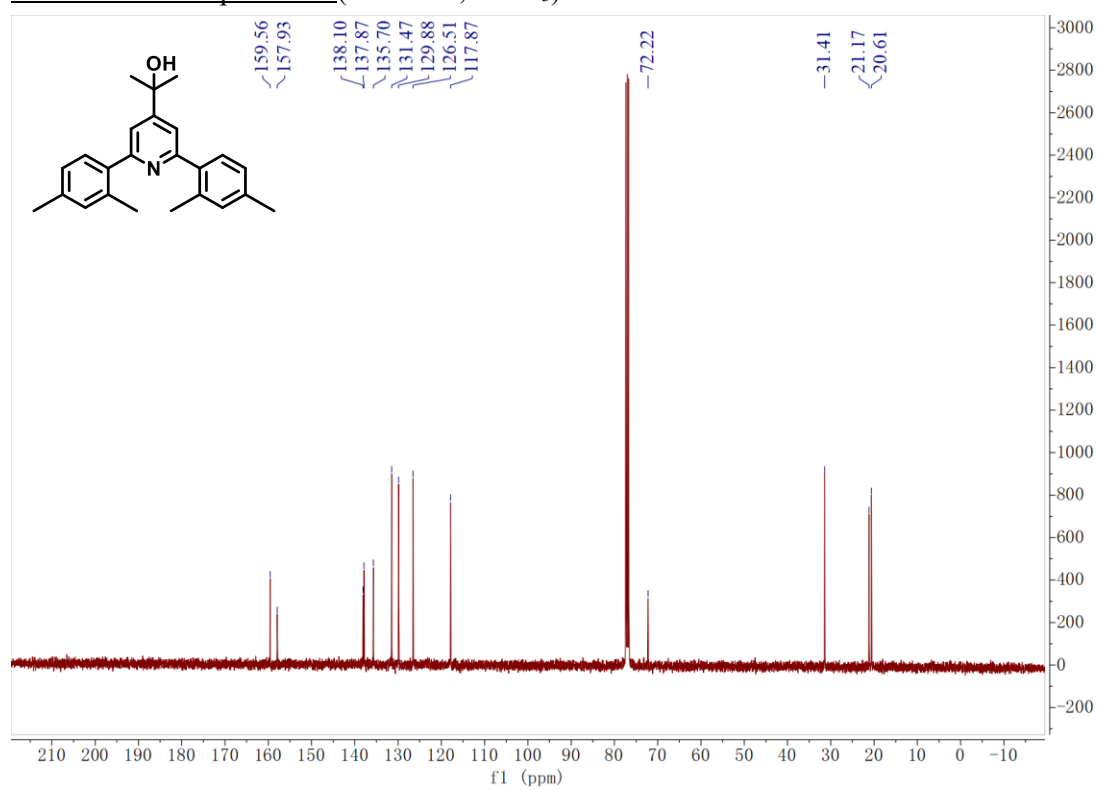
¹³C NMR of Compound **3n** (101 MHz, CDCl₃)



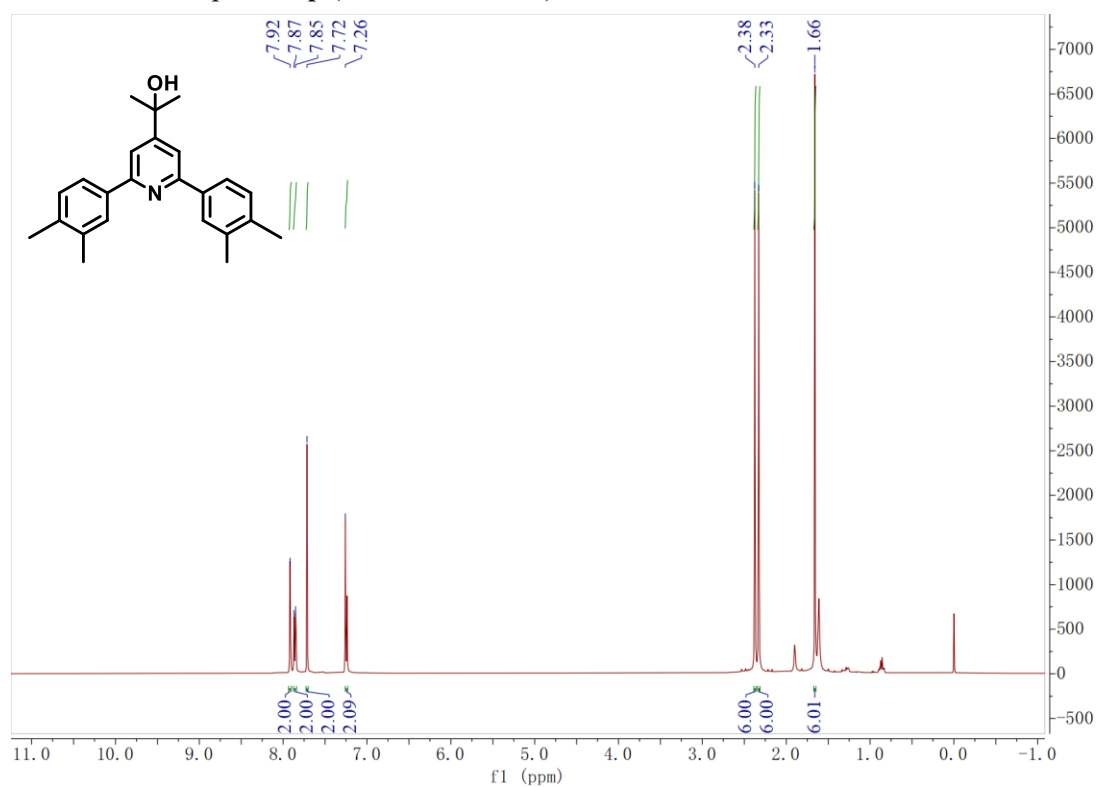
¹H NMR of Compound **3o** (400 MHz, CDCl₃)



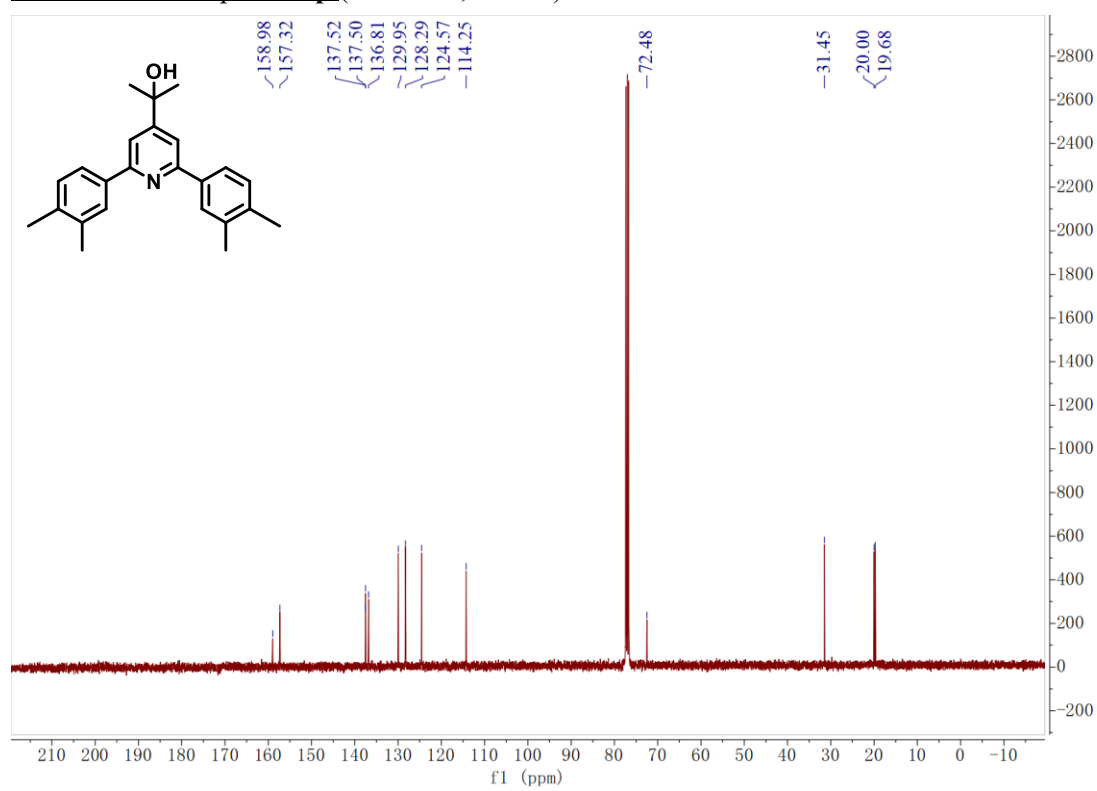
¹³C NMR of Compound **3o** (101 MHz, CDCl₃)



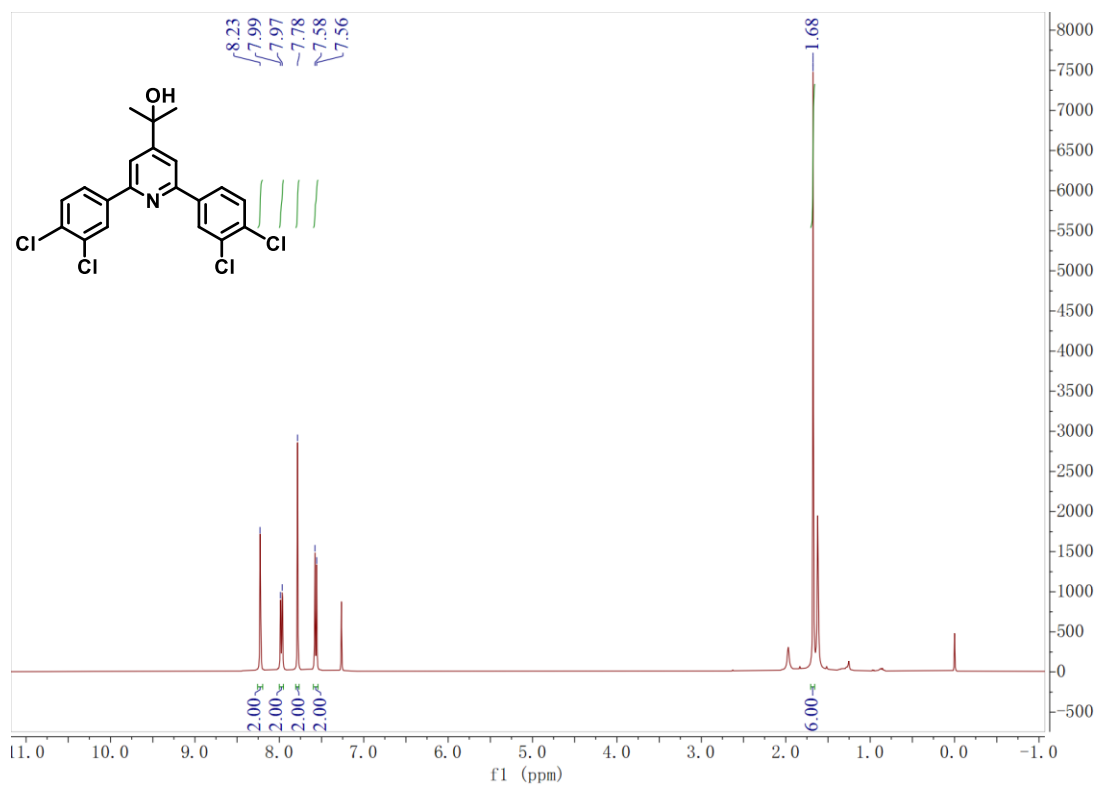
¹H NMR of Compound **3p** (400 MHz, CDCl₃)



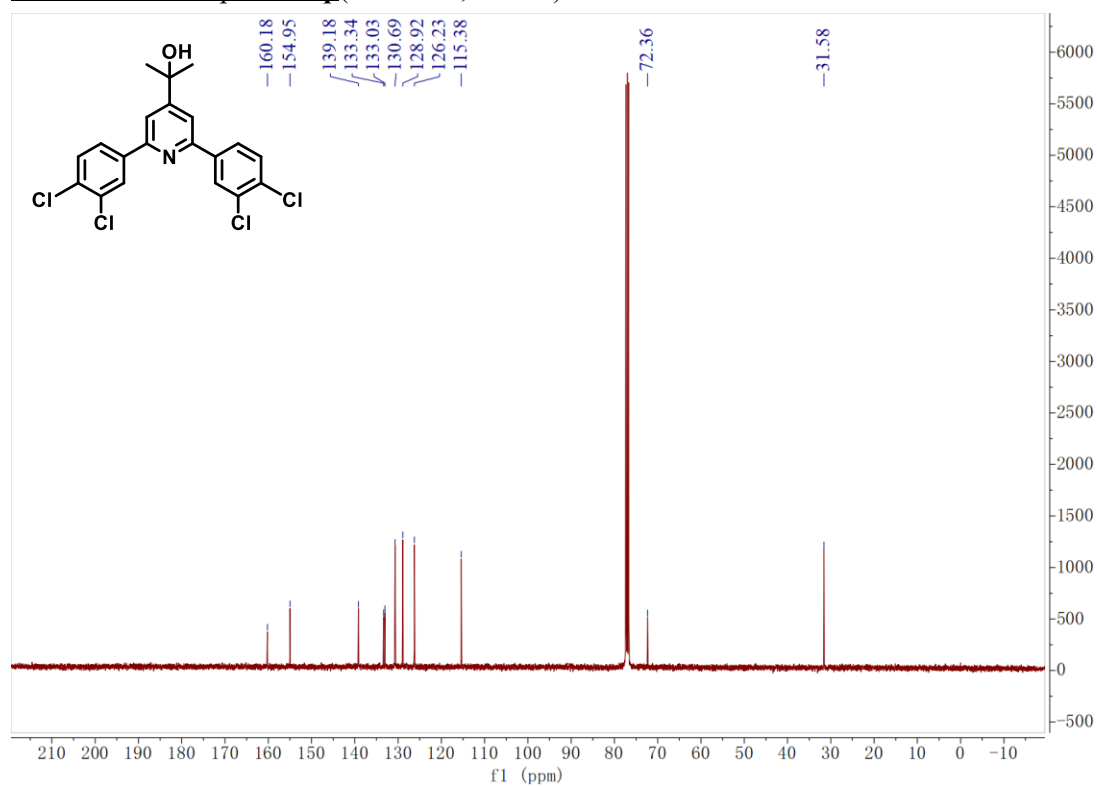
¹³C NMR of Compound **3p** (101 MHz, CDCl₃)



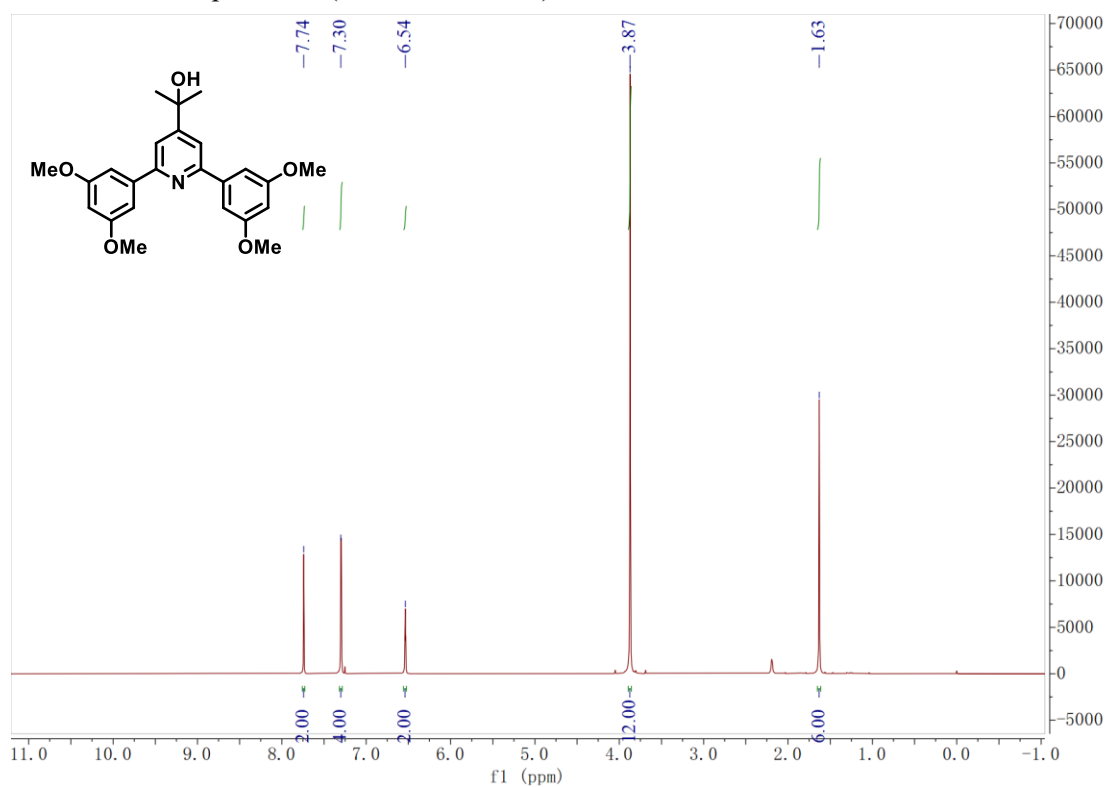
¹H NMR of Compound **3q** (400 MHz, CDCl₃)



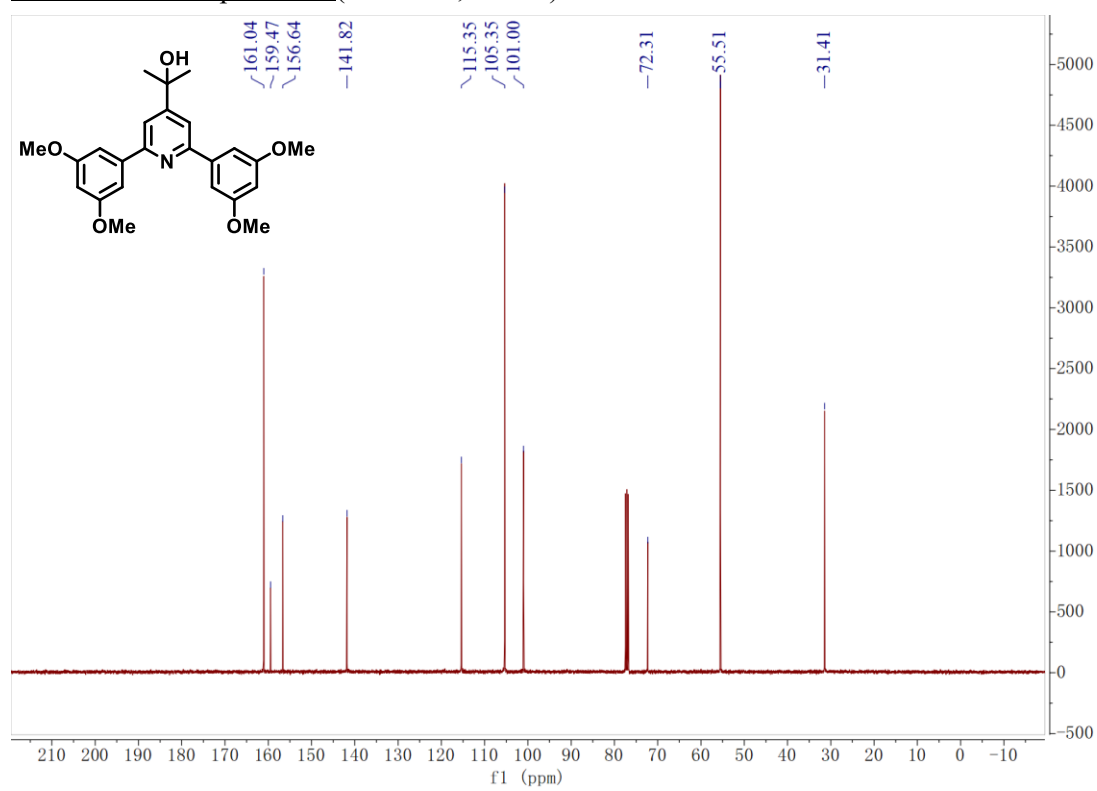
¹³C NMR of Compound **3q** (101 MHz, CDCl₃)



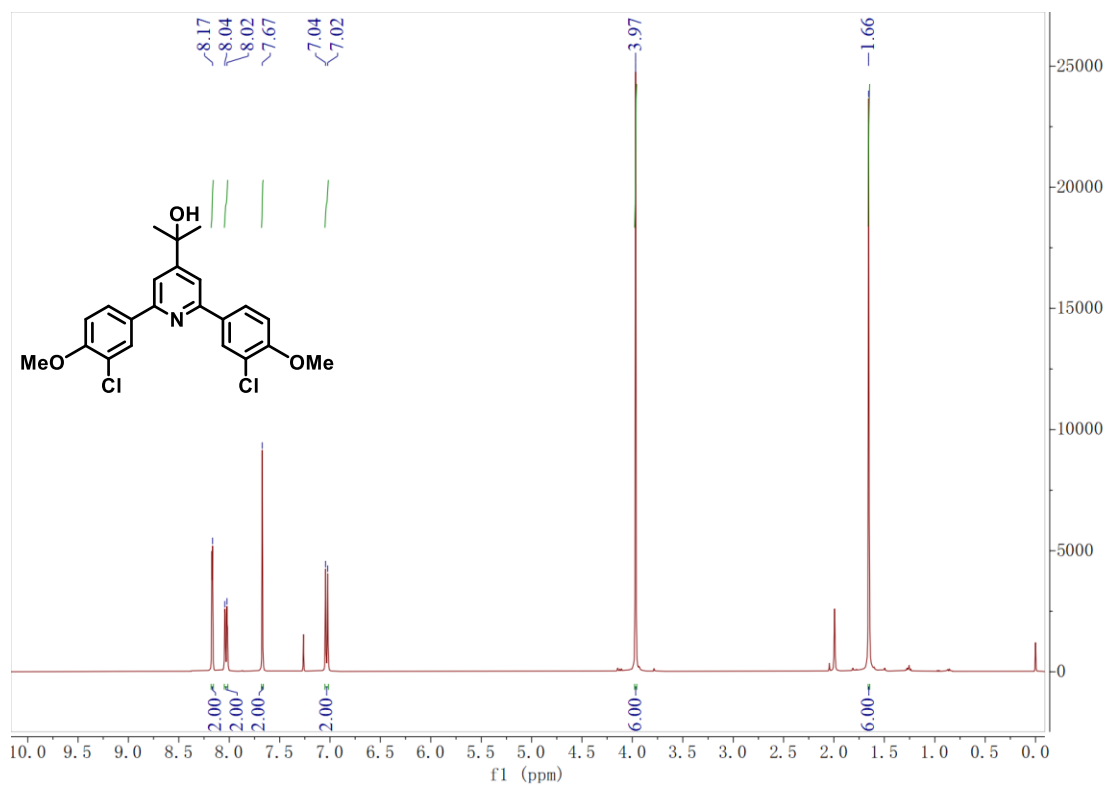
¹H NMR of Compound 3r (400 MHz, CDCl₃)



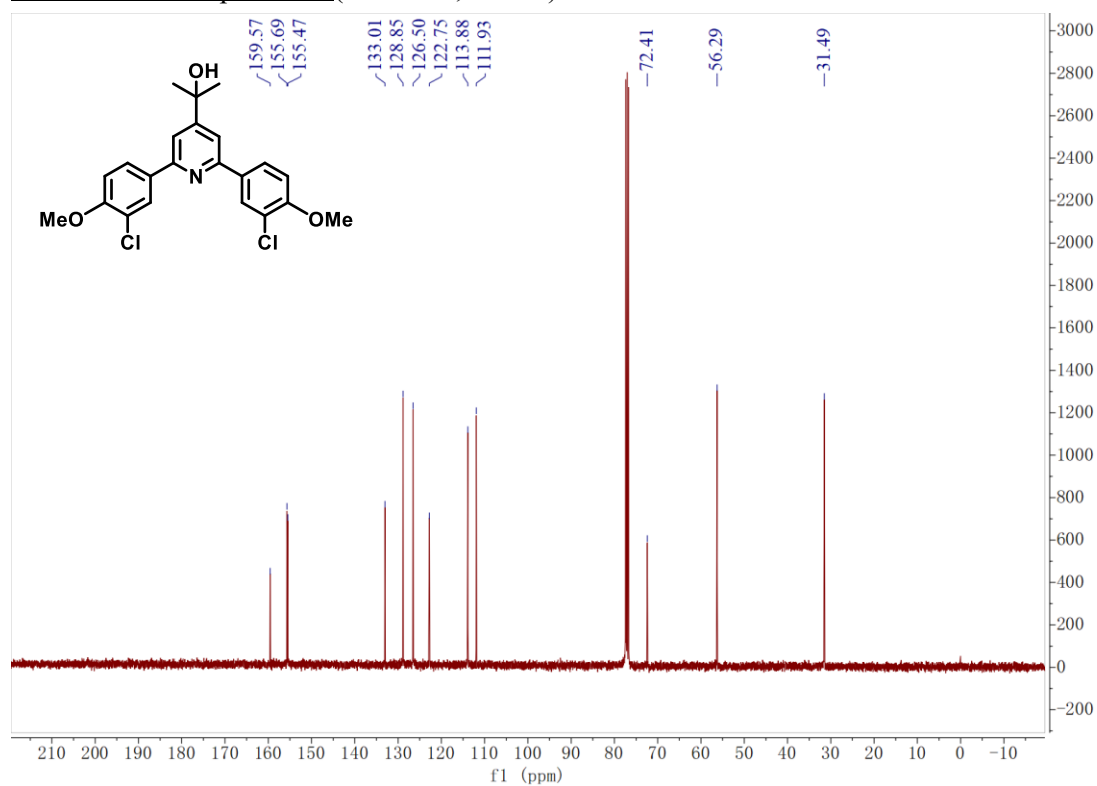
¹³C NMR of Compound 3r (101 MHz, CDCl₃)



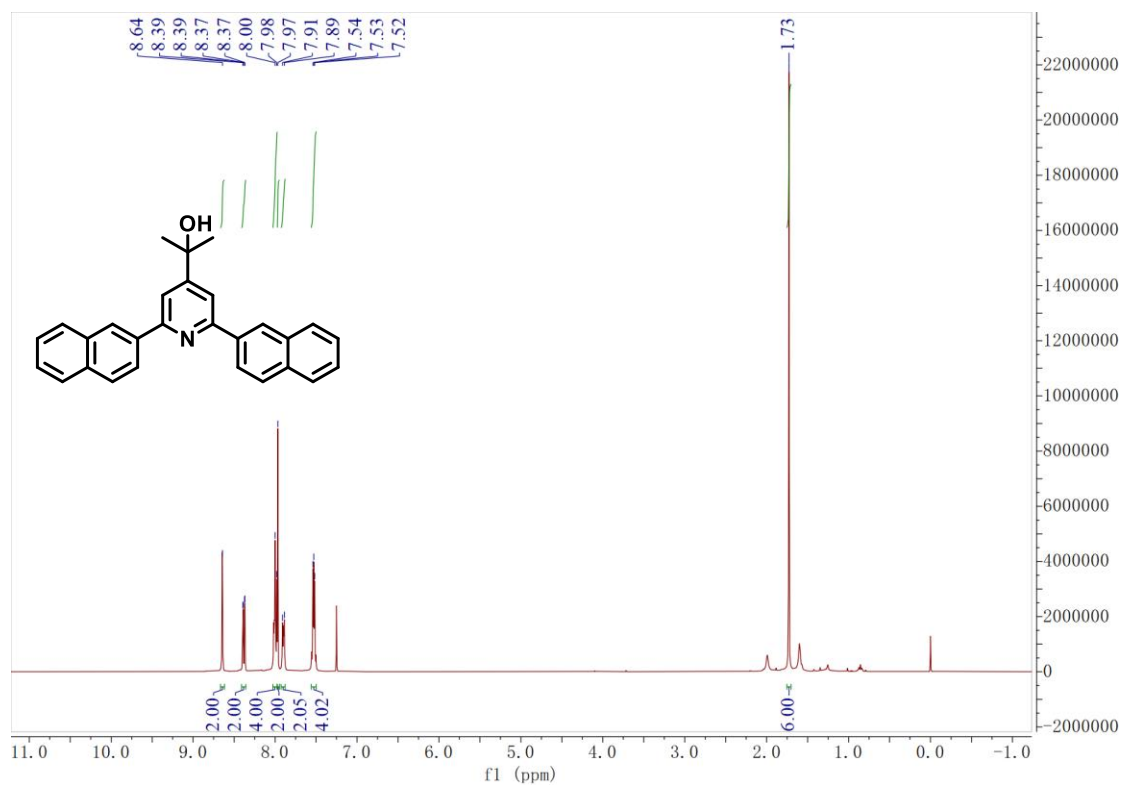
¹H NMR of Compound 3s (400 MHz, CDCl₃)



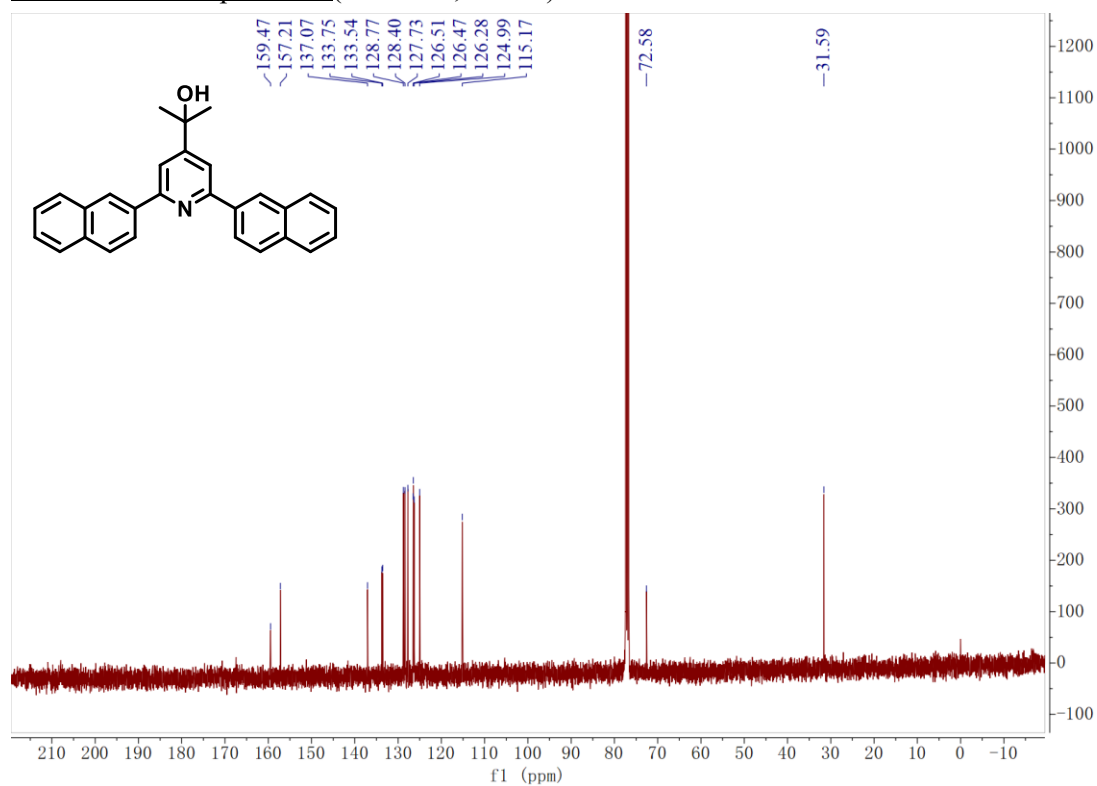
¹³C NMR of Compound 3s (101 MHz, CDCl₃)



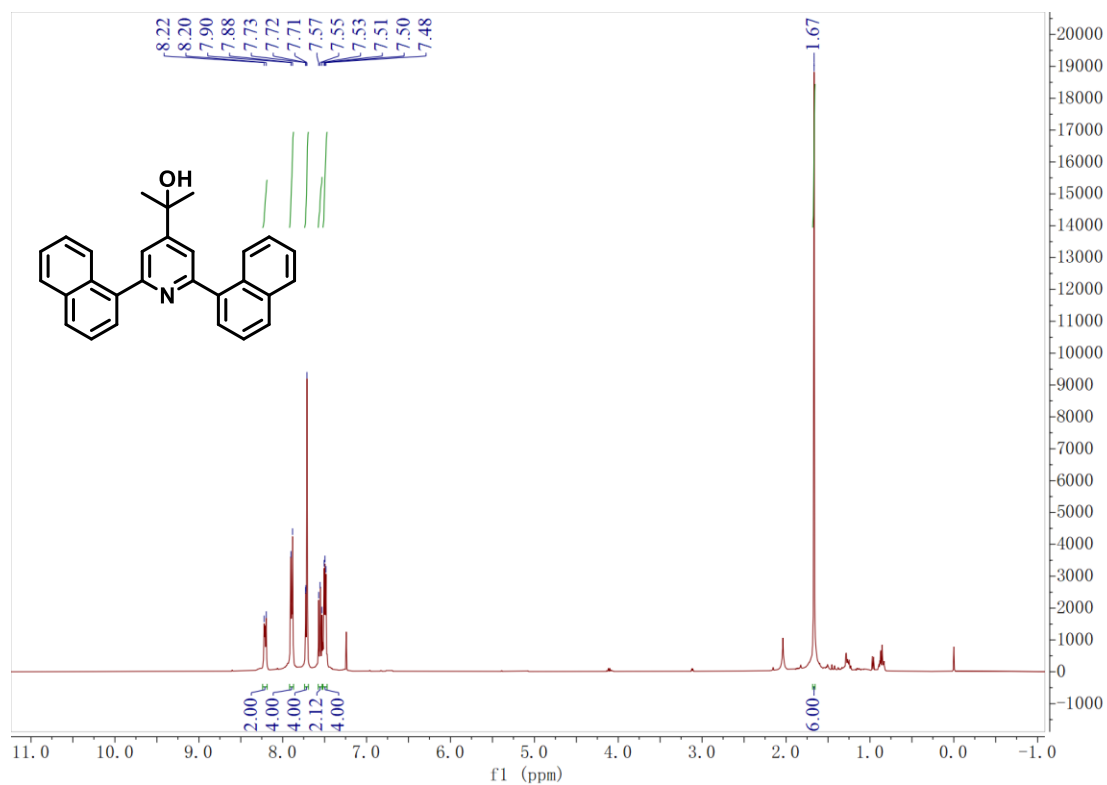
¹H NMR of Compound **3t** (400 MHz, CDCl₃)



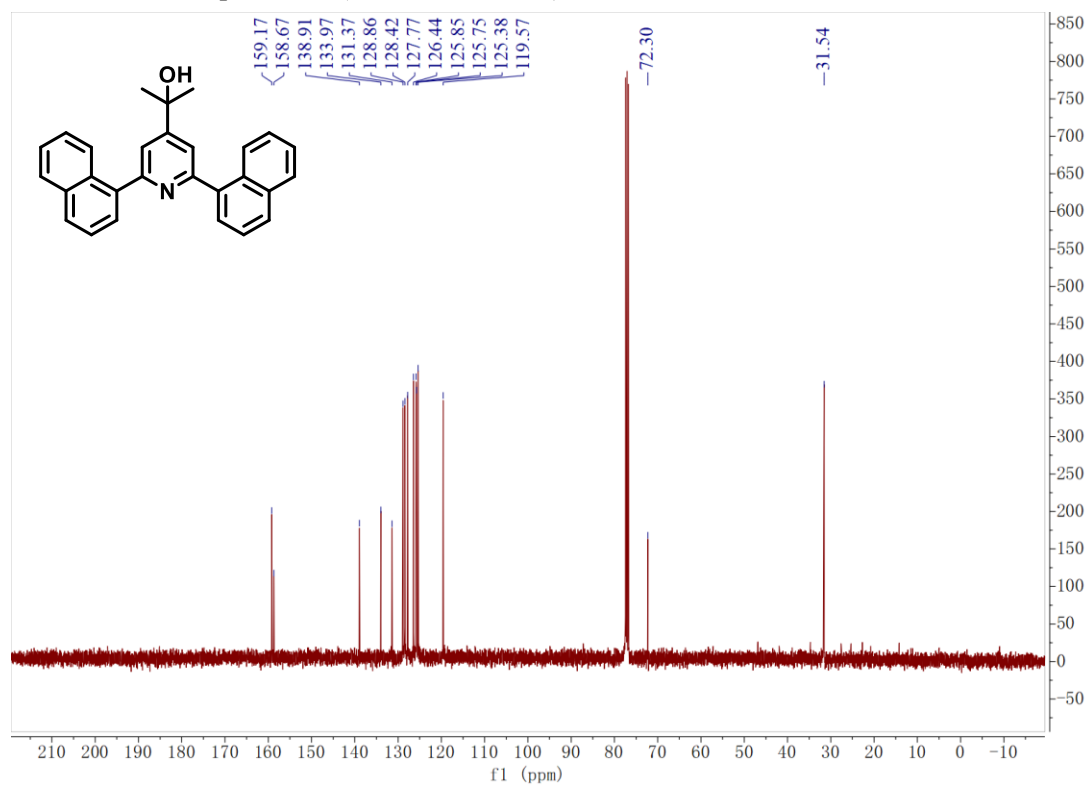
¹³C NMR of Compound **3t** (101 MHz, CDCl₃)



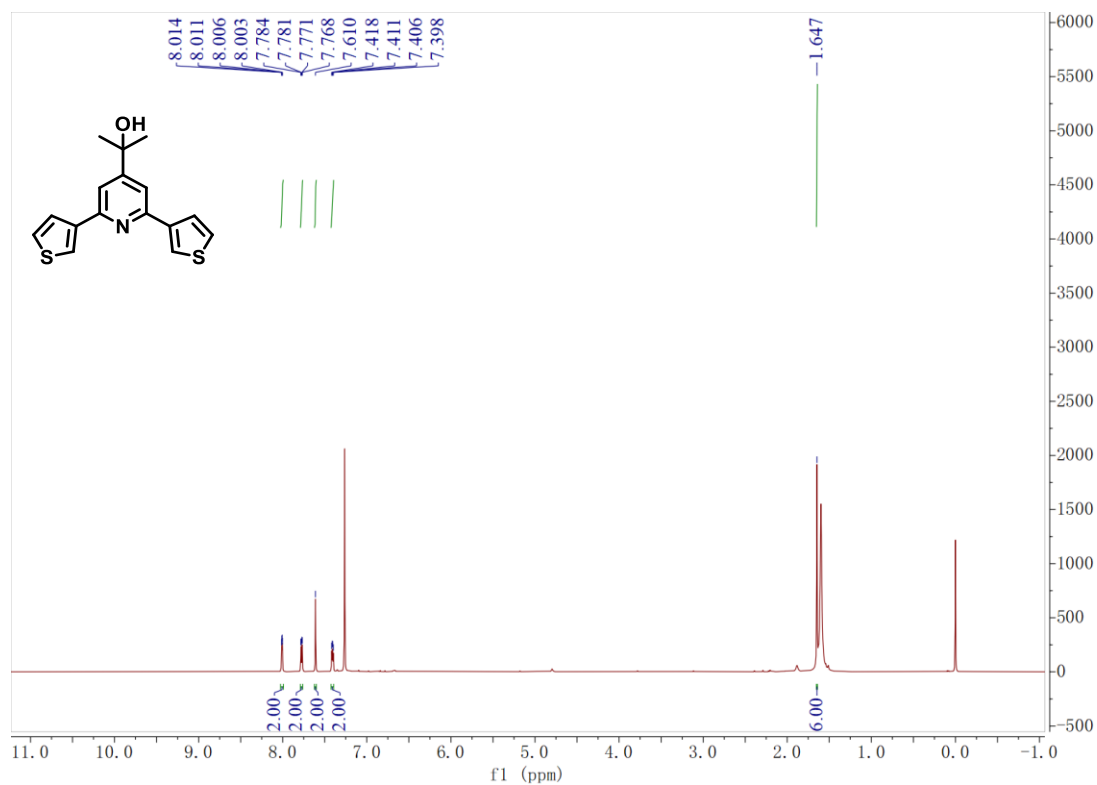
¹H NMR of Compound **3u** (400 MHz, CDCl₃)



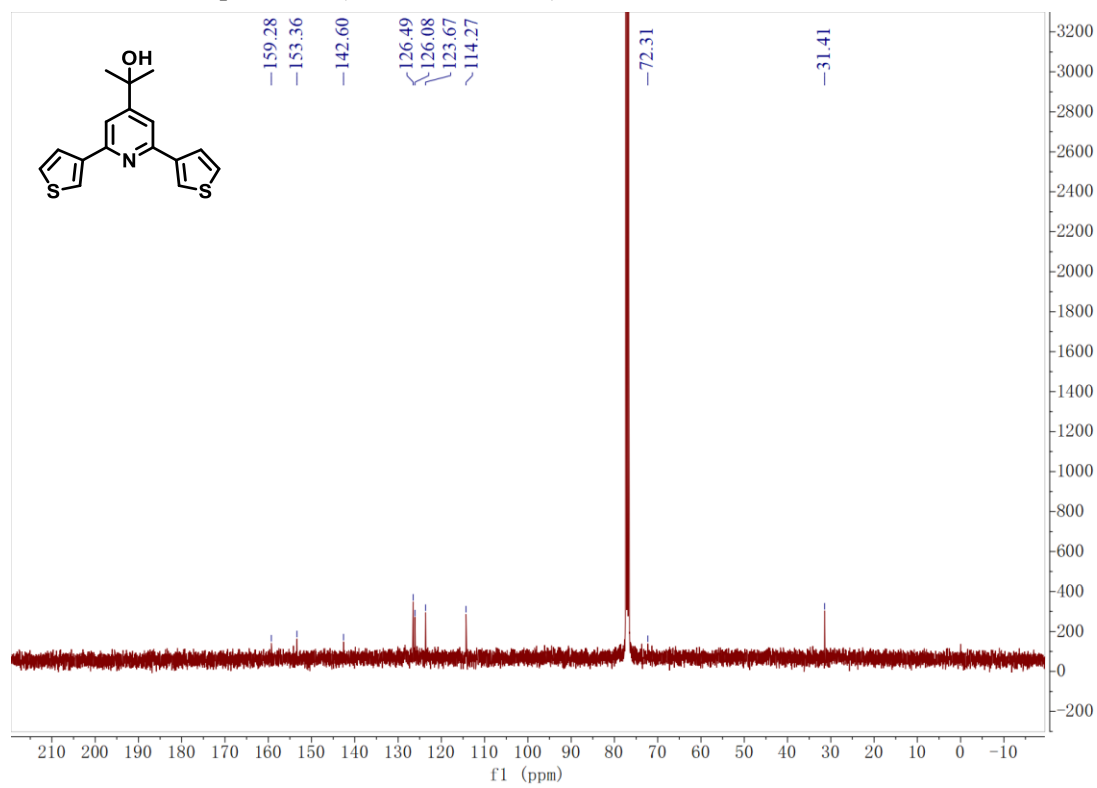
¹³C NMR of Compound **3u** (101 MHz, CDCl₃)



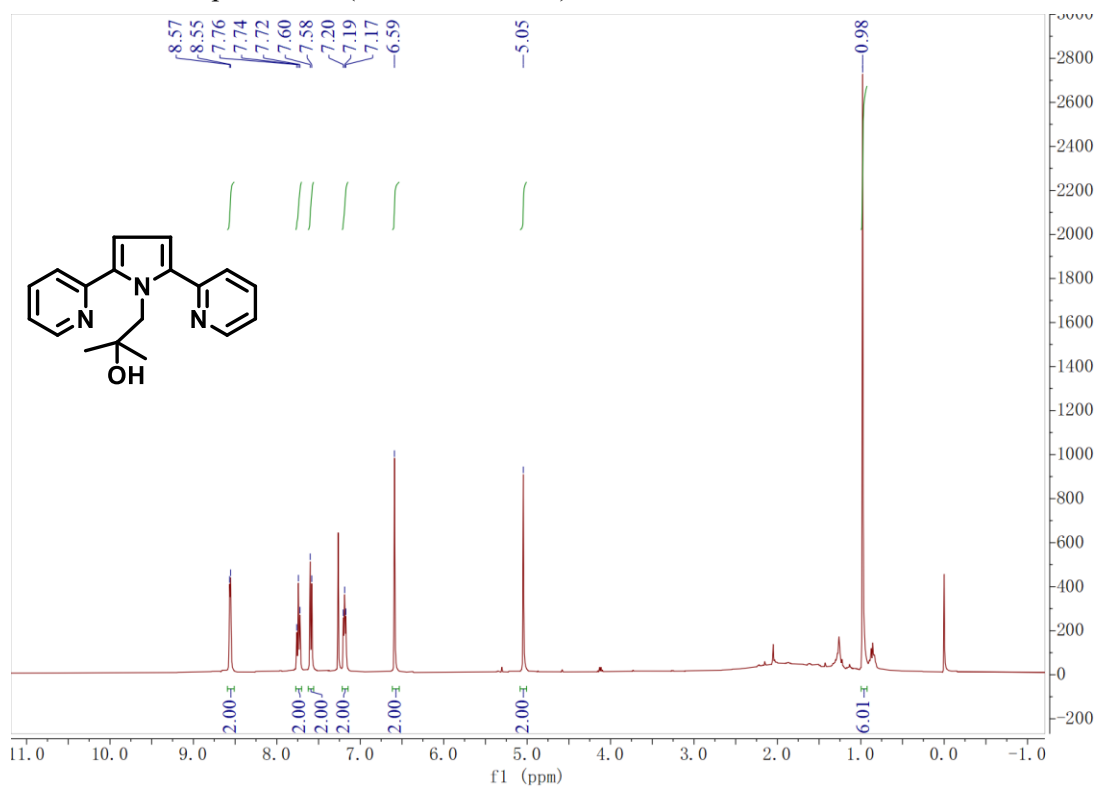
¹H NMR of Compound **3v** (400 MHz, CDCl₃)



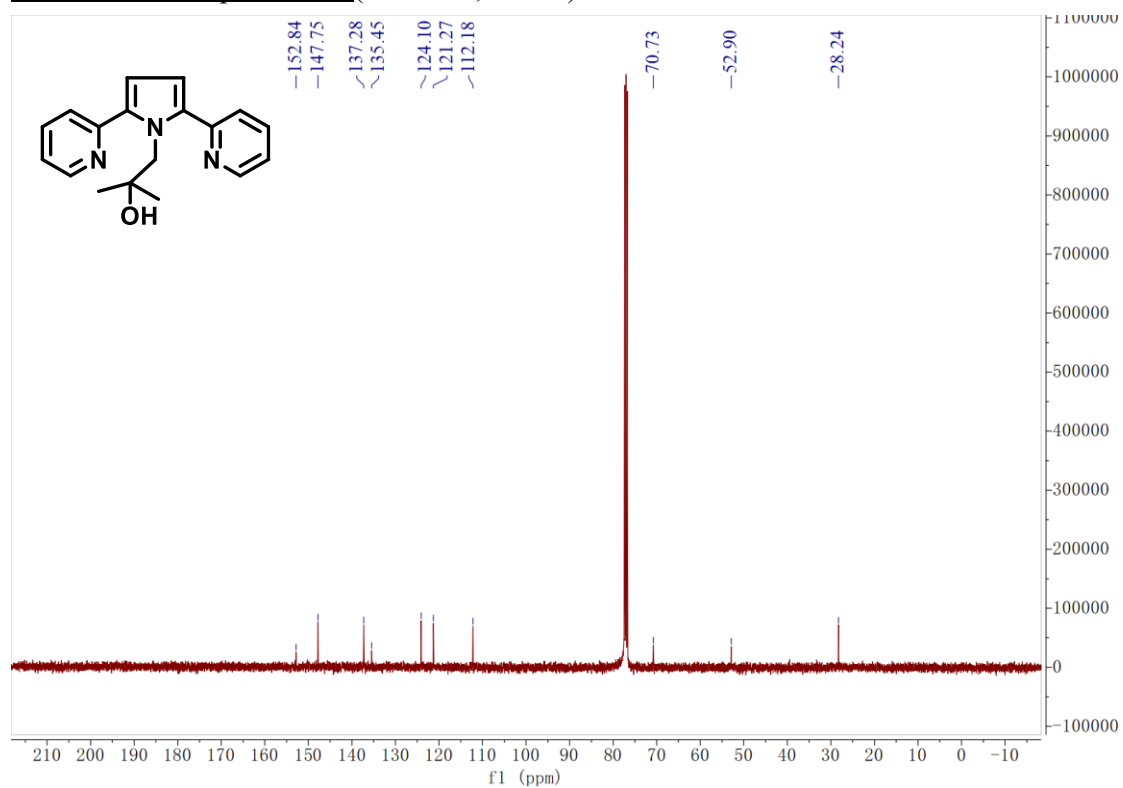
¹³C NMR of Compound **3v** (101 MHz, CDCl₃)



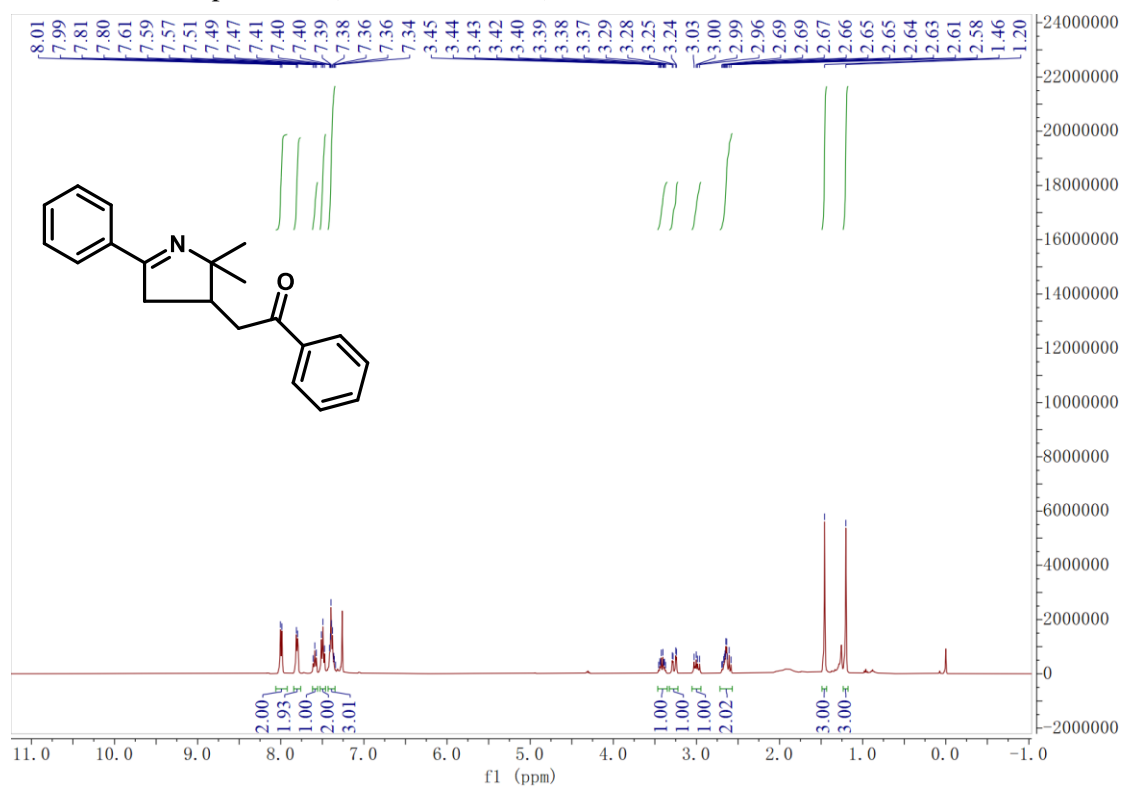
¹H NMR of Compound 3w' (400 MHz, CDCl₃)



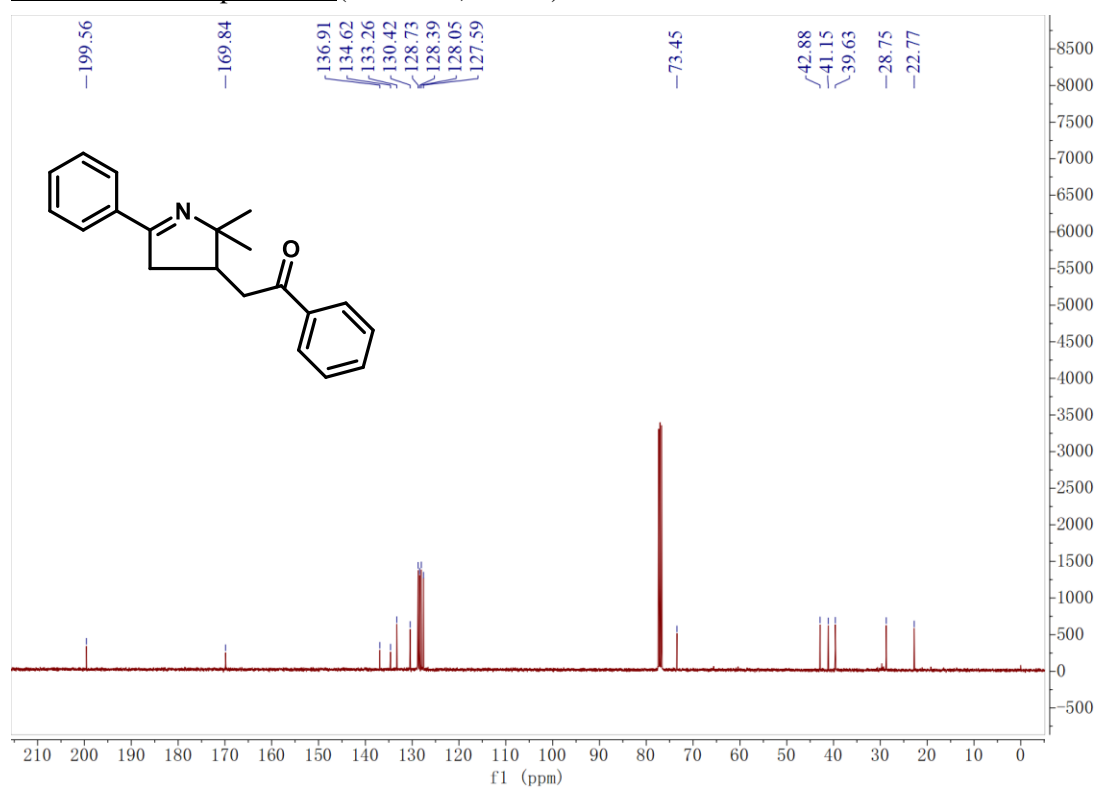
¹³C NMR of Compound 3w' (101 MHz, CDCl₃)



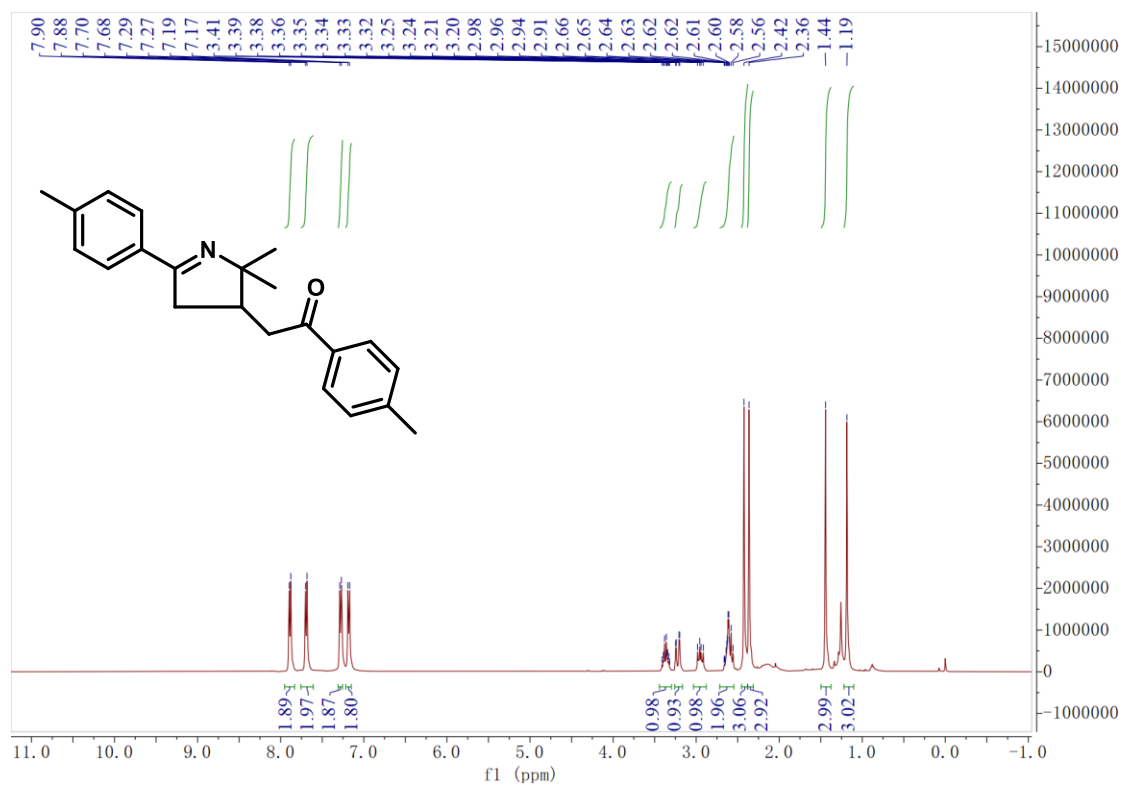
¹H NMR of Compound **5a** (400 MHz, CDCl₃)



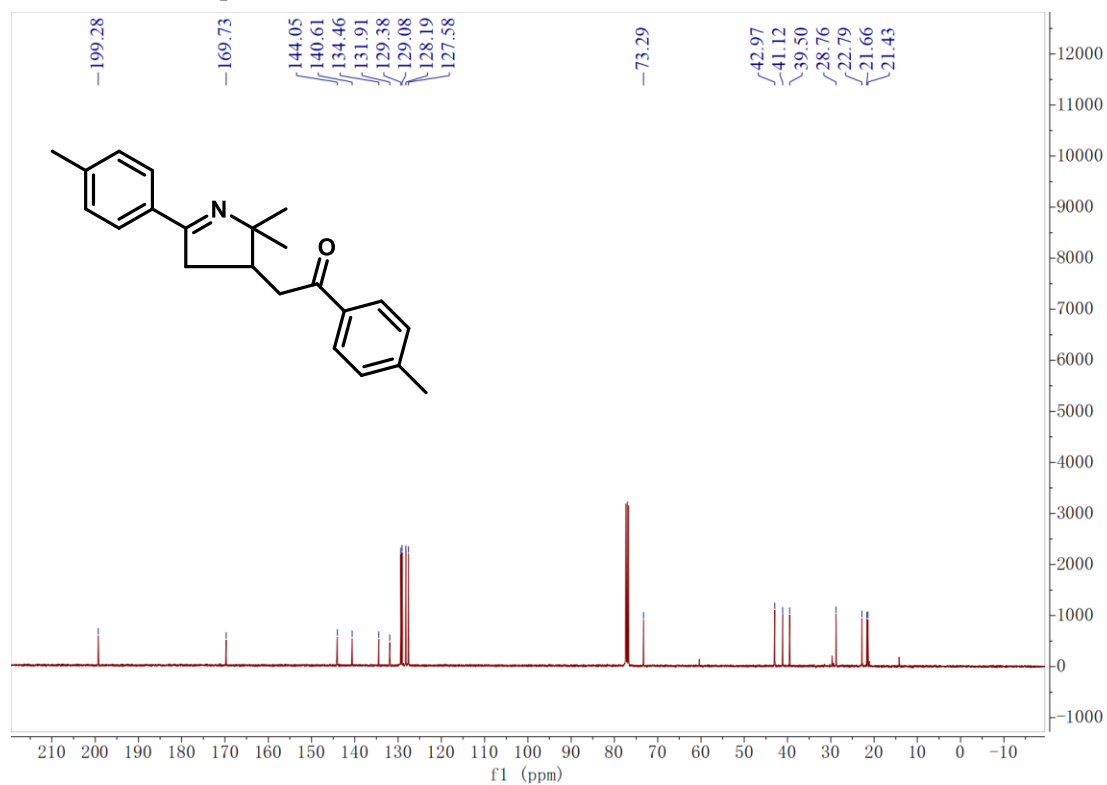
¹³C NMR of Compound **5a** (101 MHz, CDCl₃)



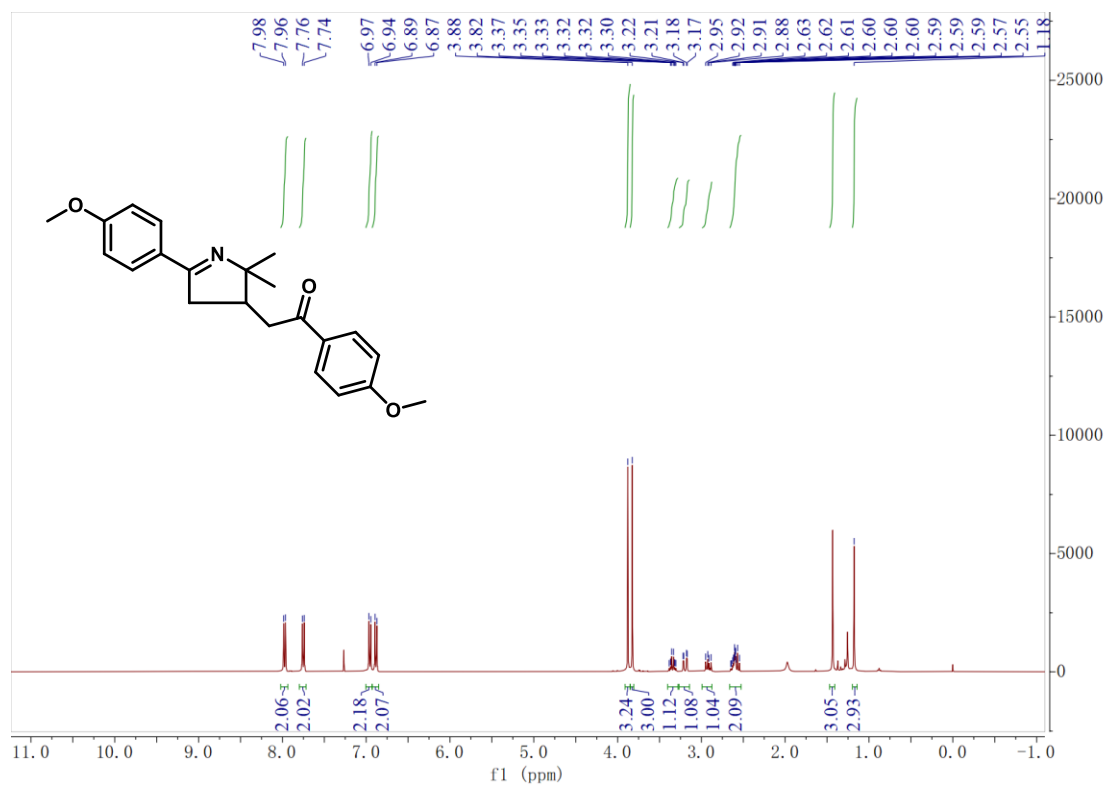
¹H NMR of Compound 5b (400 MHz, CDCl₃)



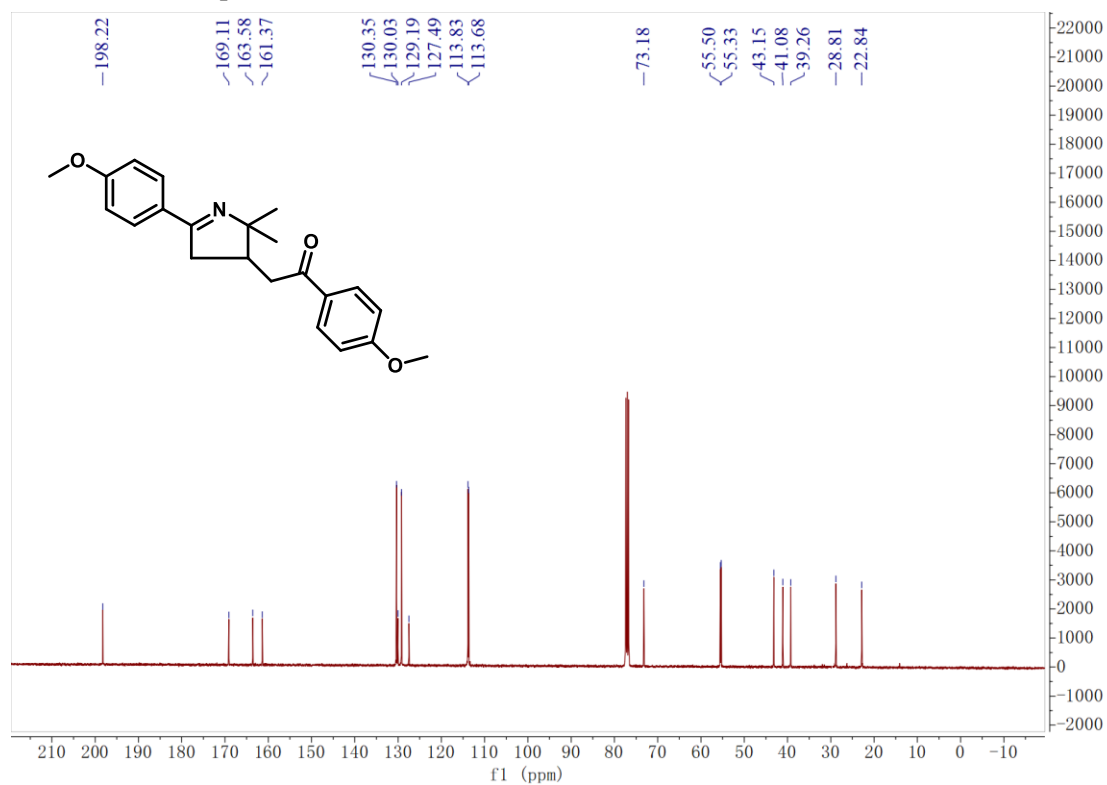
¹³C NMR of Compound 5b (101 MHz, CDCl₃)



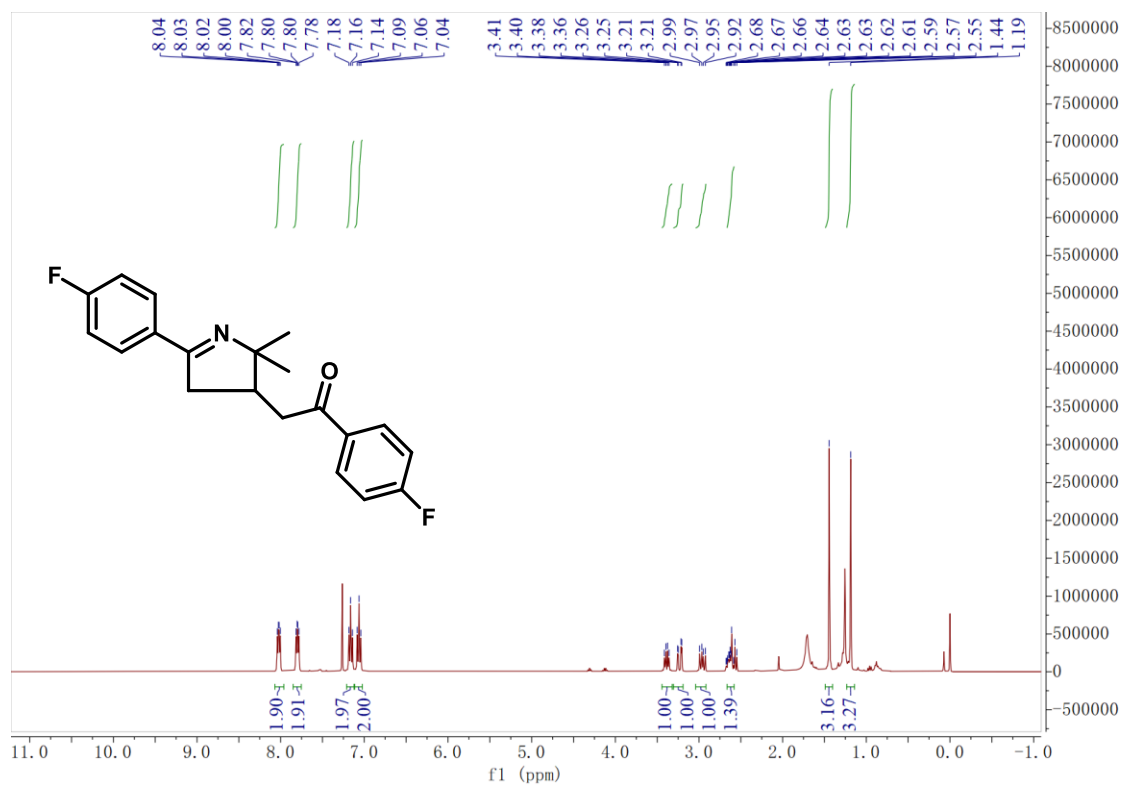
¹H NMR of Compound 5c (400 MHz, CDCl₃)



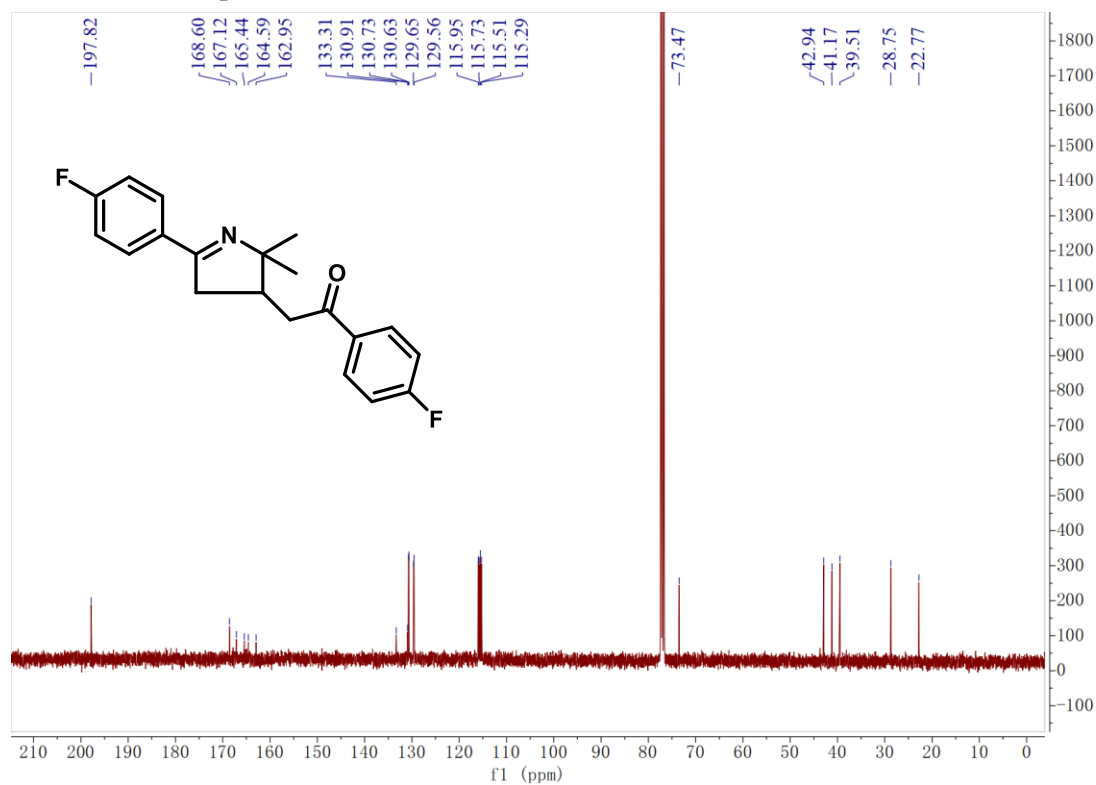
¹³C NMR of Compound 5c (101 MHz, CDCl₃)



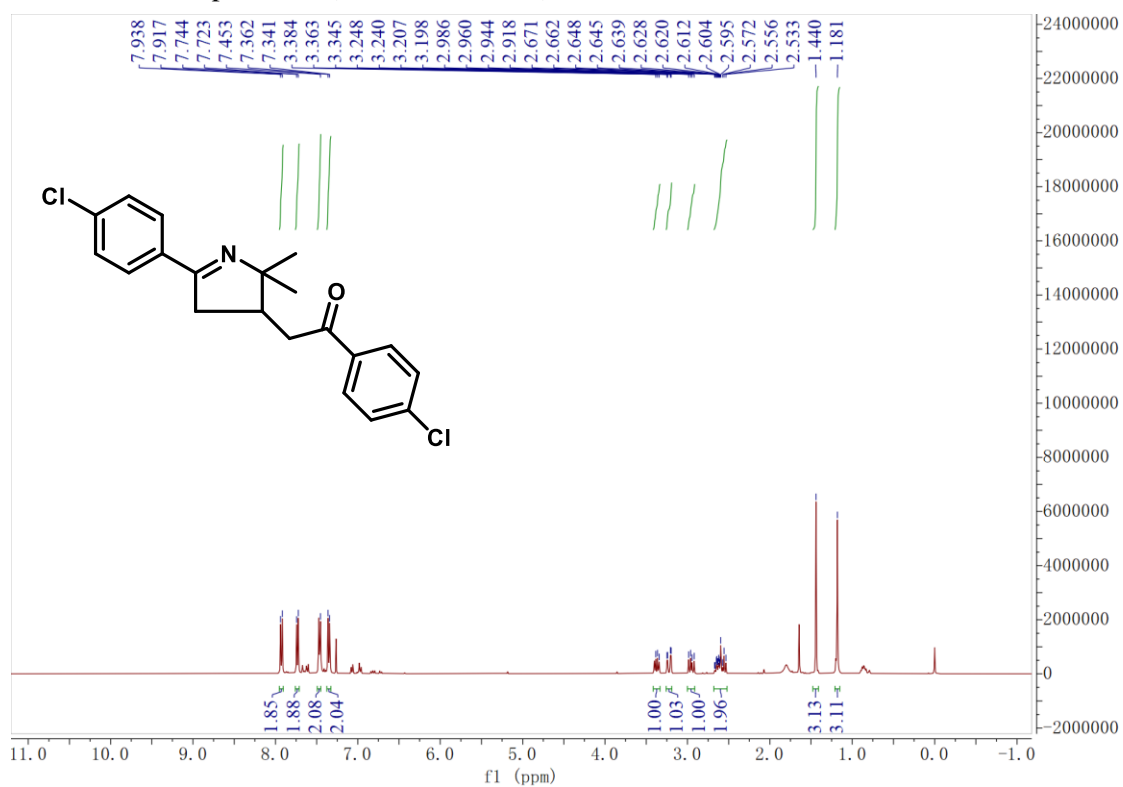
¹H NMR of Compound **5d** (400 MHz, CDCl₃)



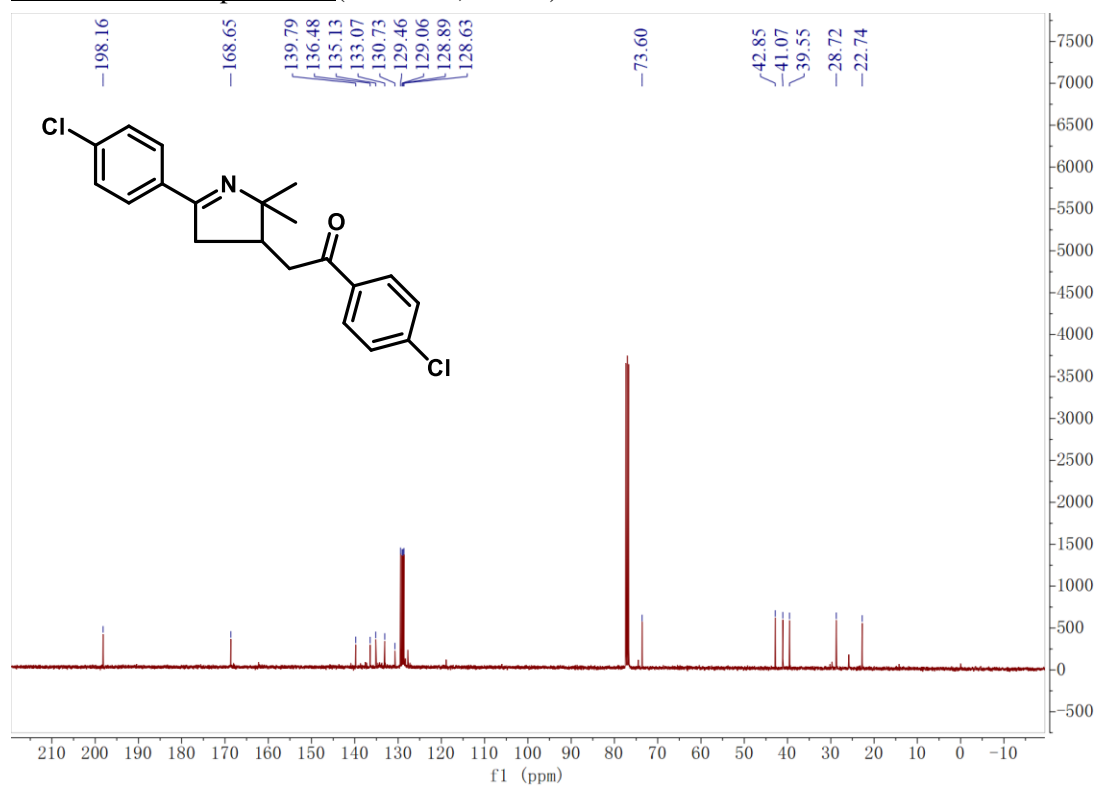
¹³C NMR of Compound **5d** (101 MHz, CDCl₃)



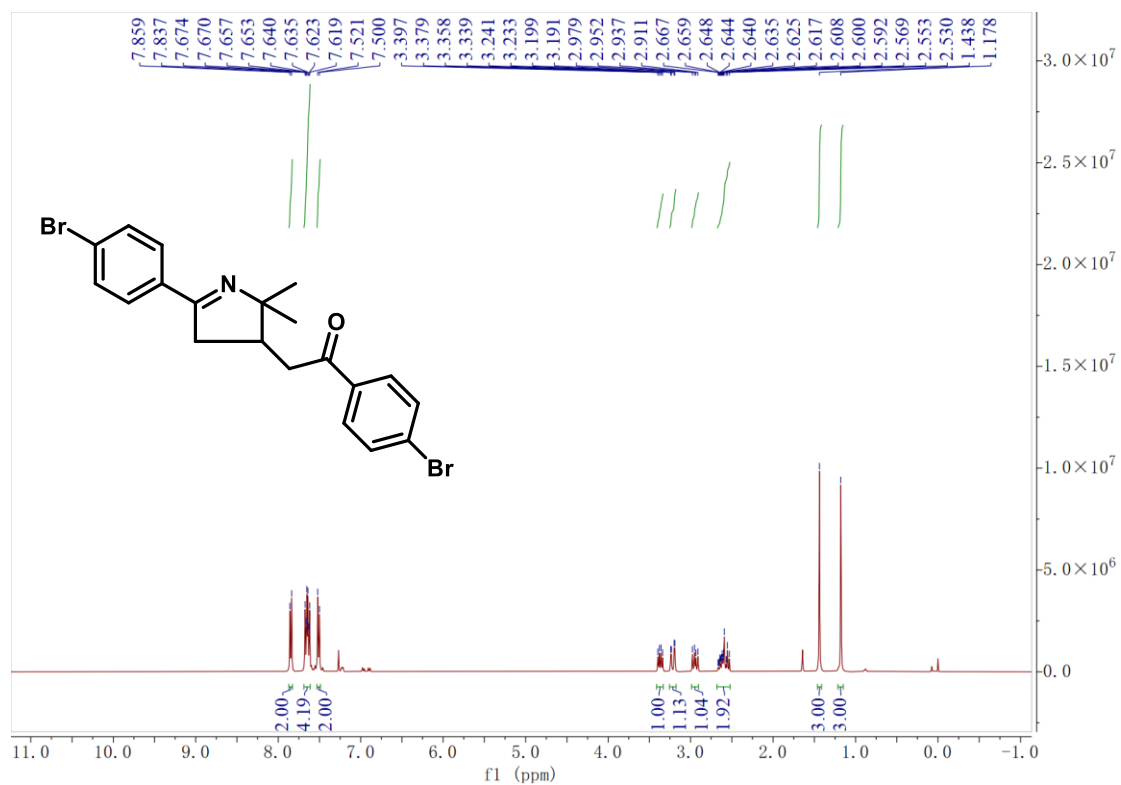
¹H NMR of Compound **5e** (400 MHz, CDCl₃)



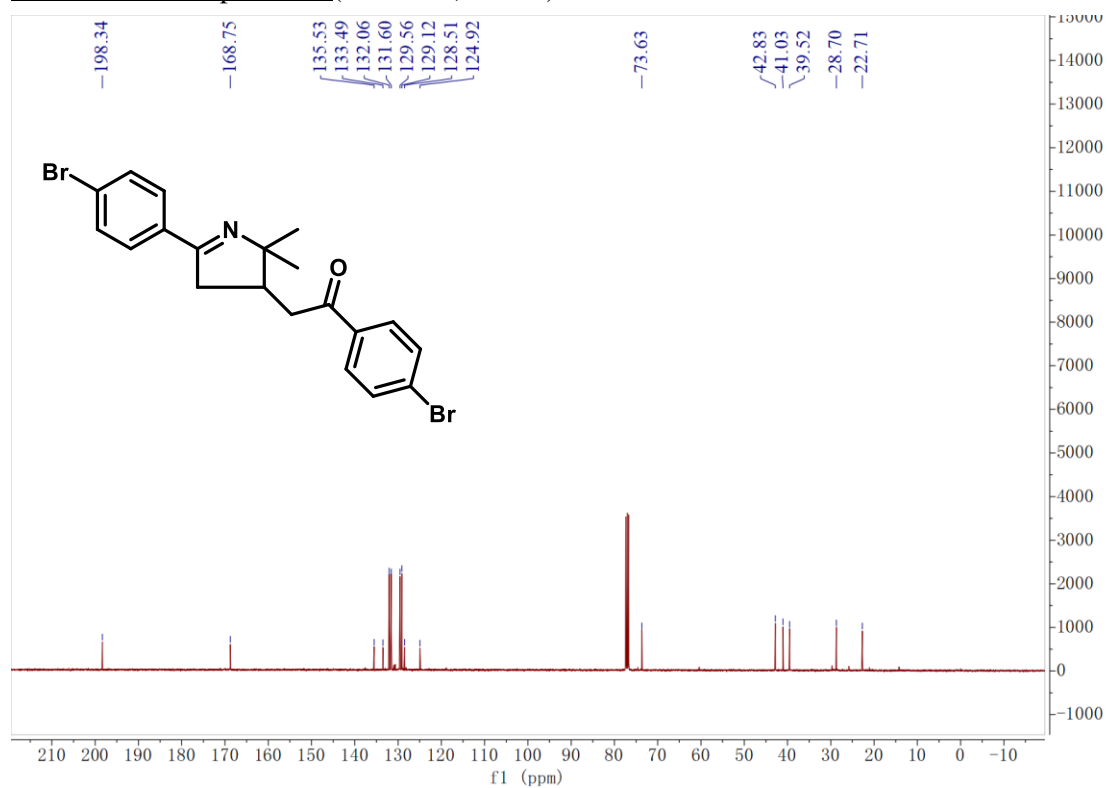
¹³C NMR of Compound **5e** (101 MHz, CDCl₃)



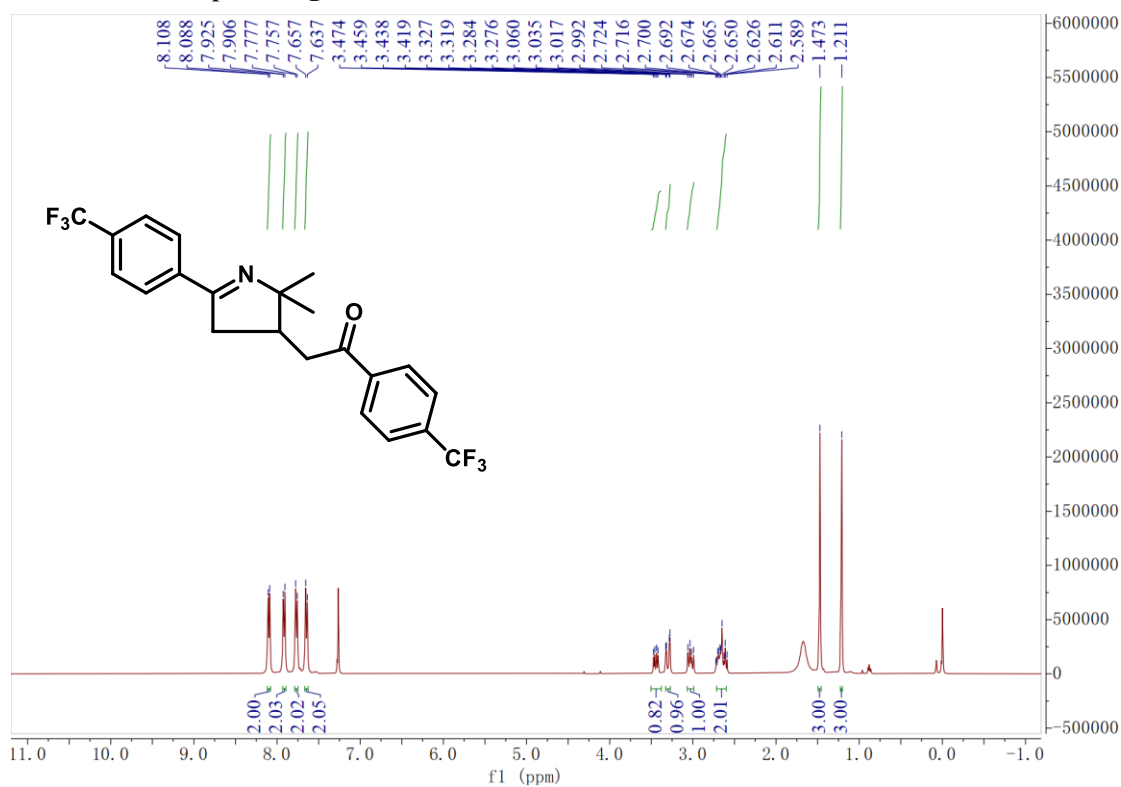
¹H NMR of Compound **5f** (400 MHz, CDCl₃)



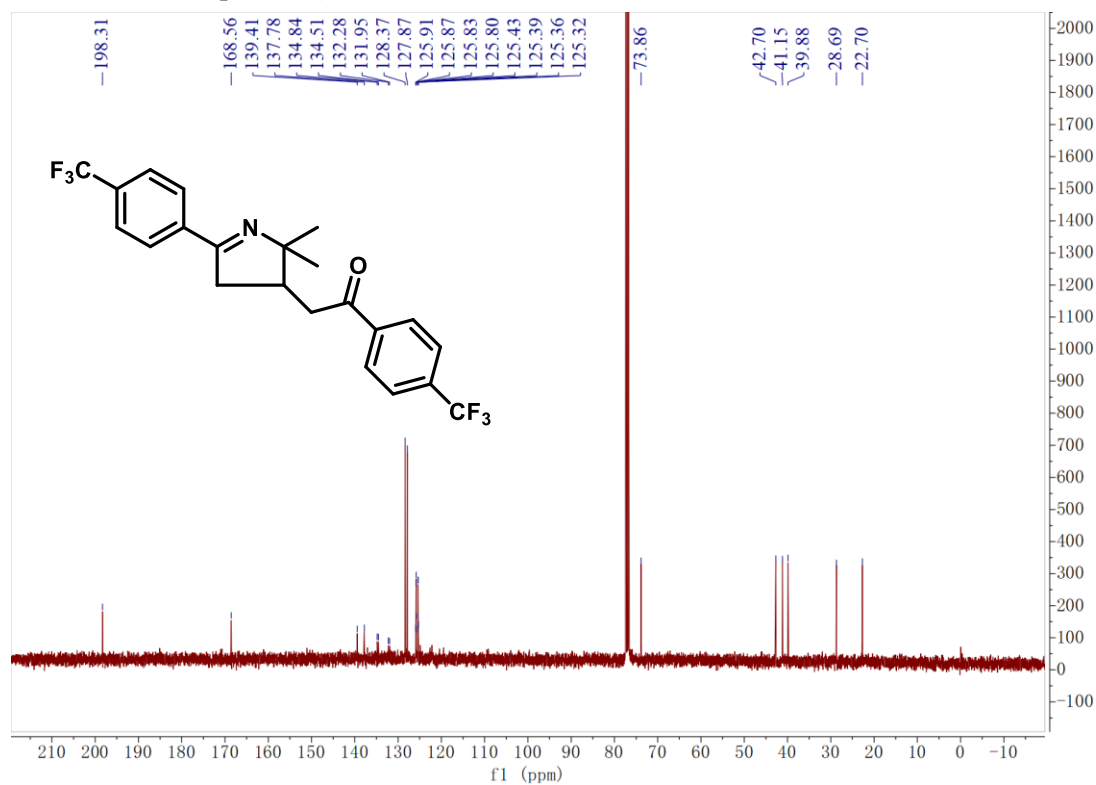
¹³C NMR of Compound **5f** (101 MHz, CDCl₃)



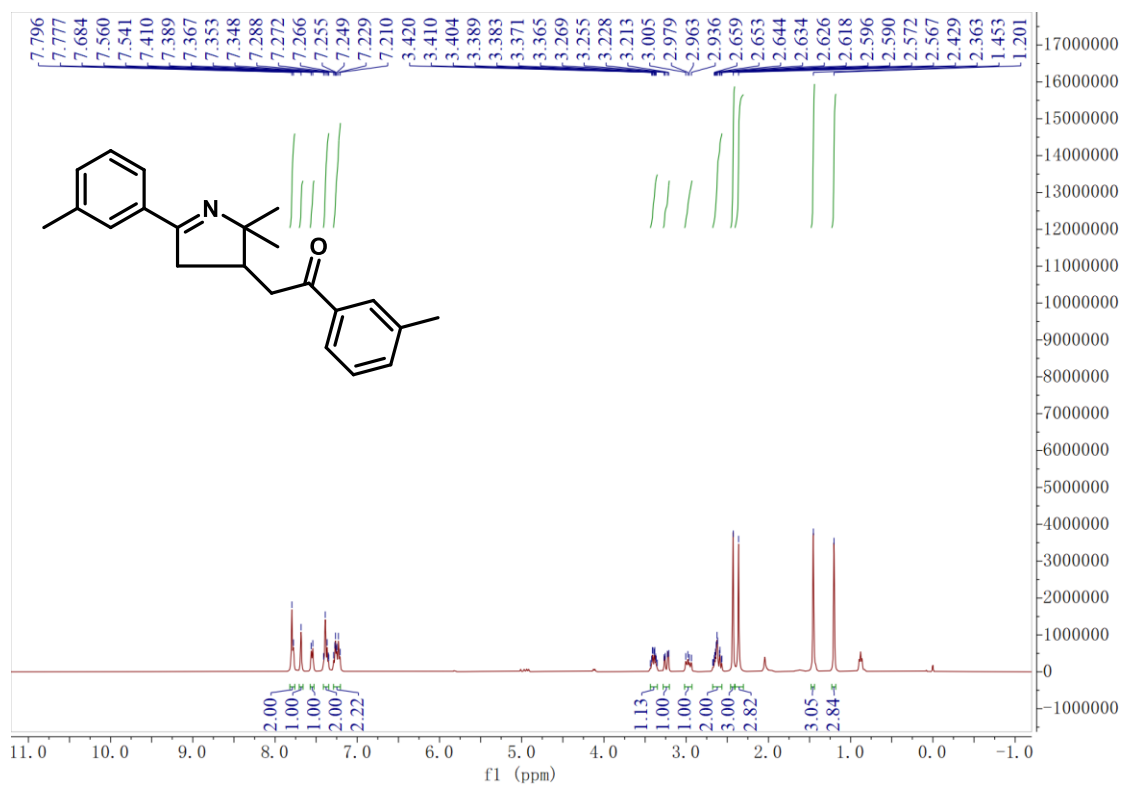
¹H NMR of Compound **5g** (400 MHz, CDCl₃)



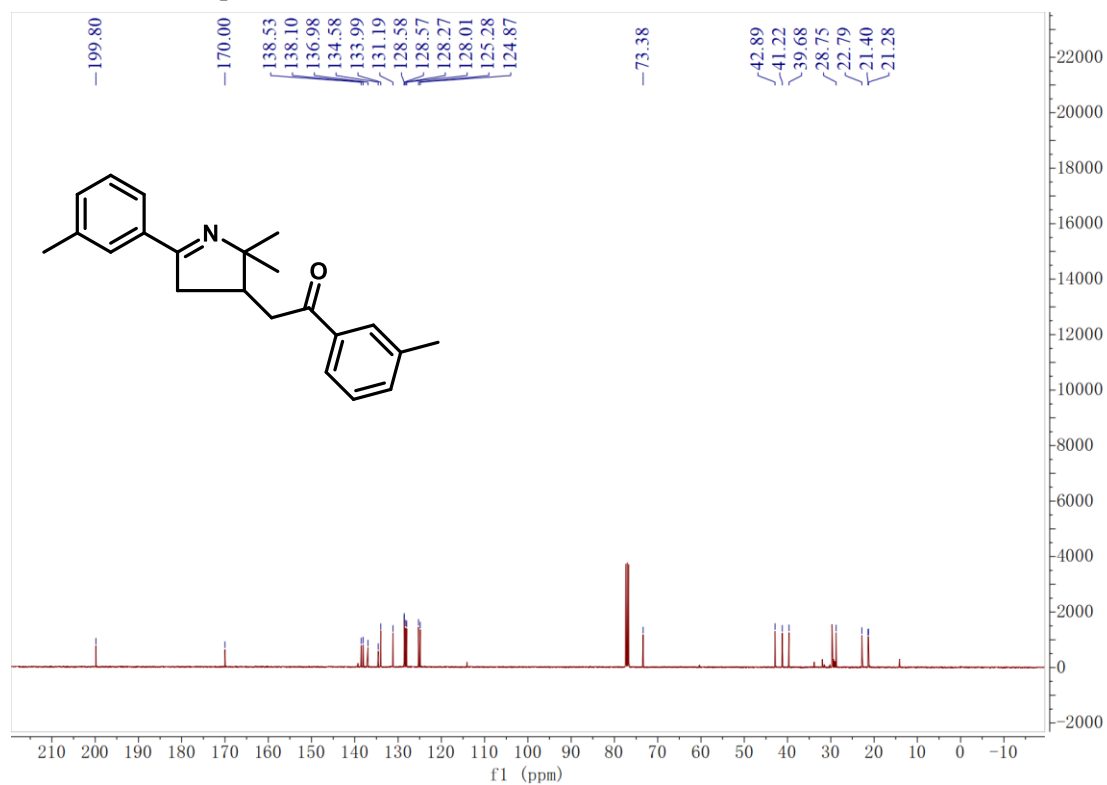
¹³C NMR of Compound **5g** (101 MHz, CDCl₃)



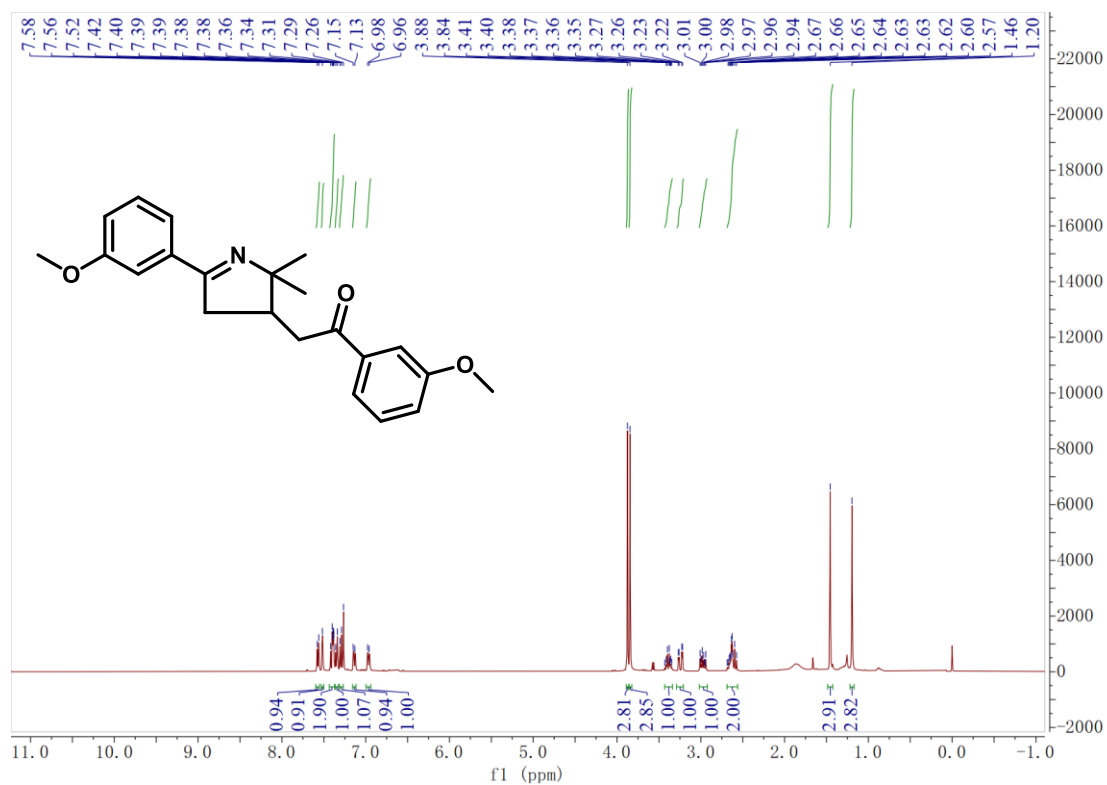
¹H NMR of Compound 5h (400 MHz, CDCl₃)



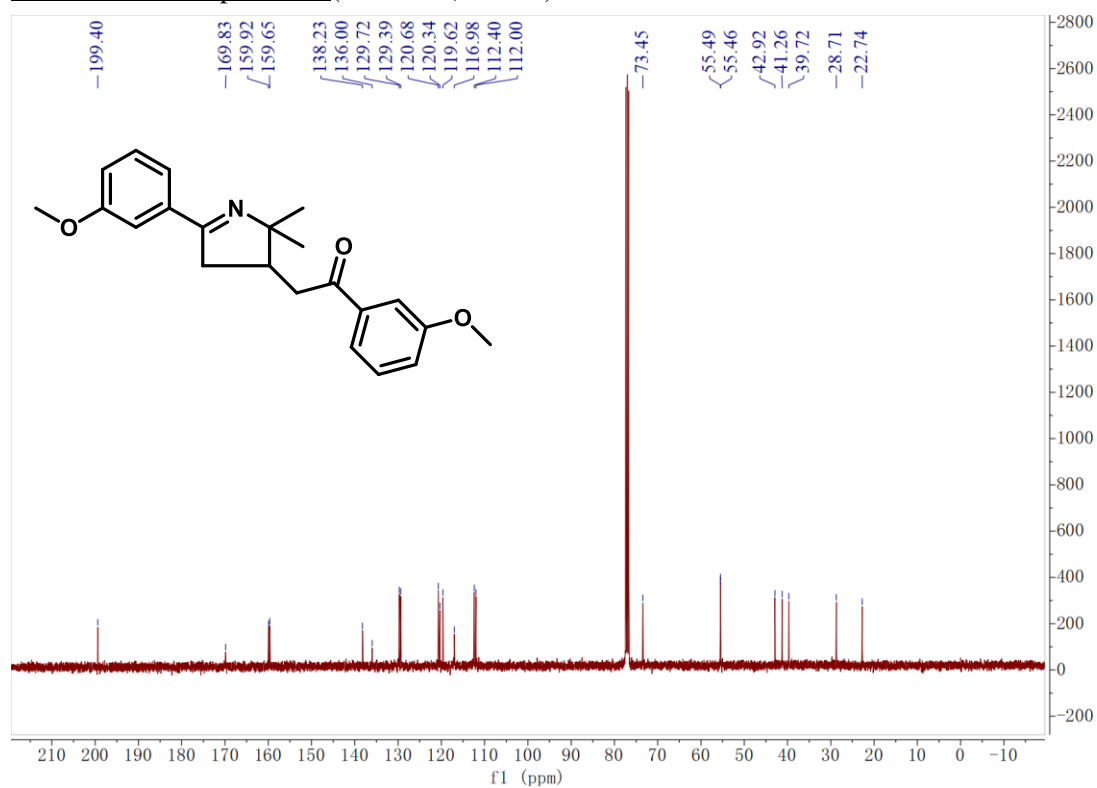
¹³C NMR of Compound 5h (101 MHz, CDCl₃)



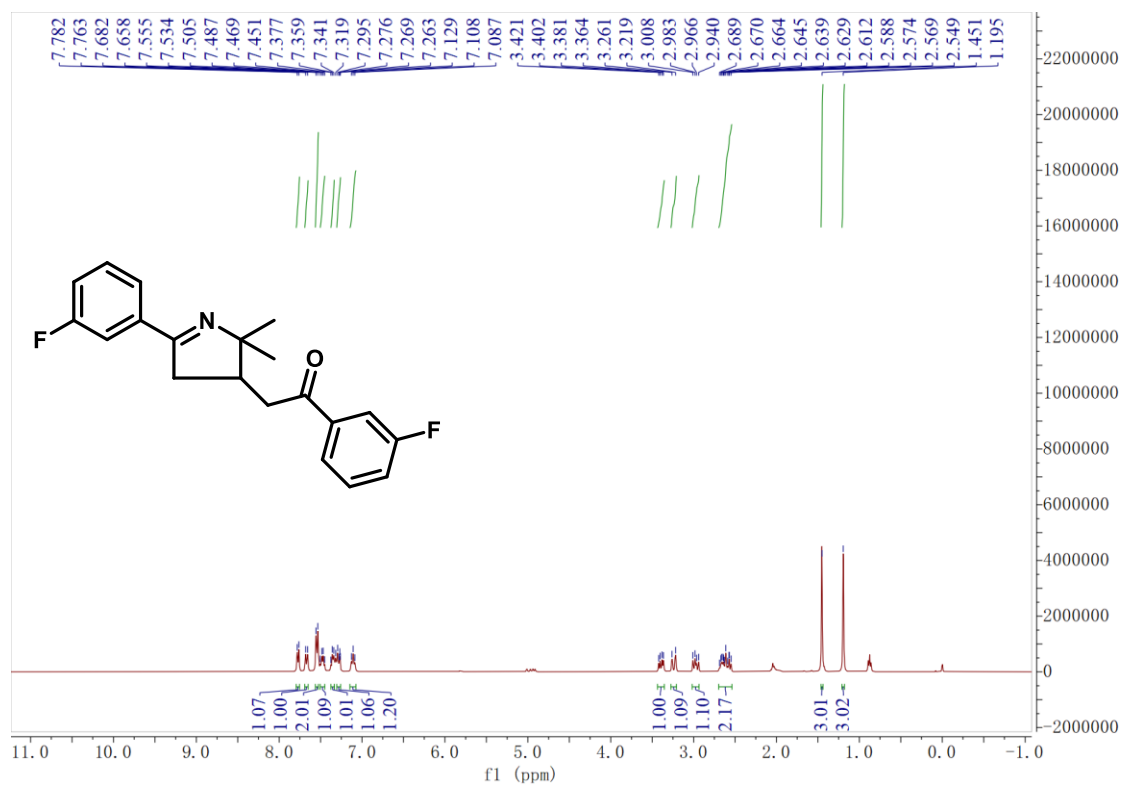
¹H NMR of Compound 5i (400 MHz, CDCl₃)



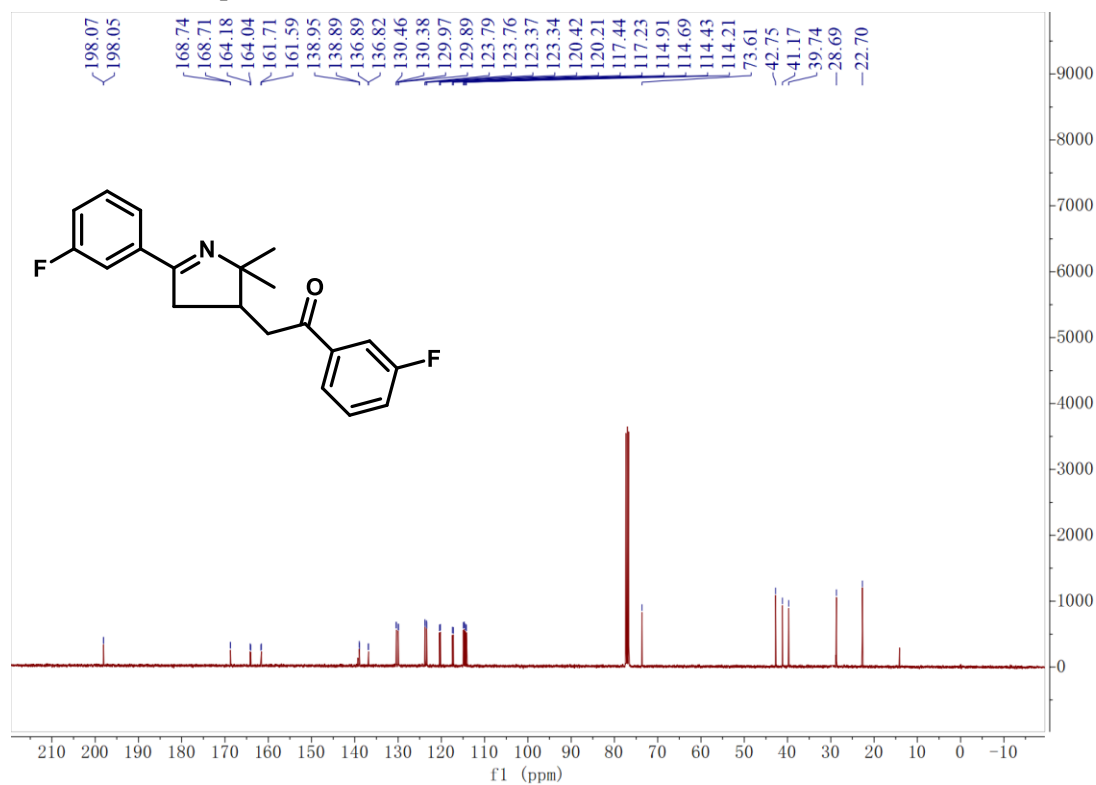
¹³C NMR of Compound 5i (101 MHz, CDCl₃)



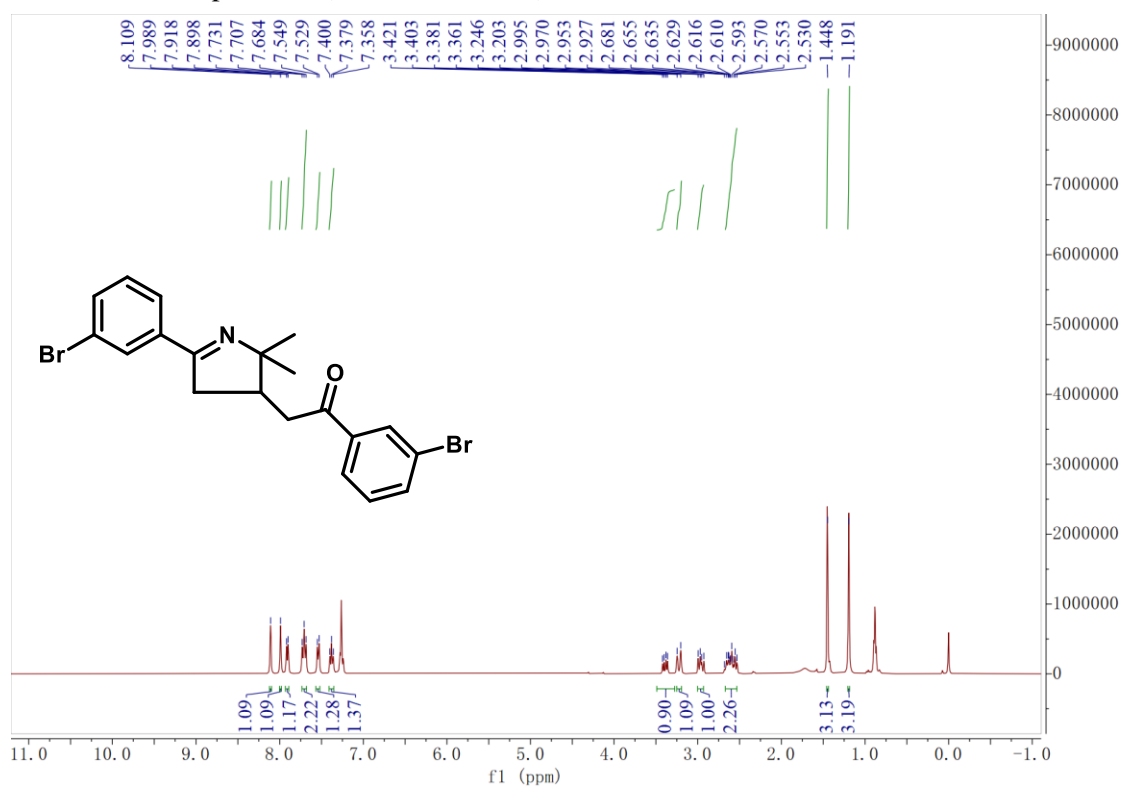
¹H NMR of Compound **5j** (400 MHz, CDCl₃)



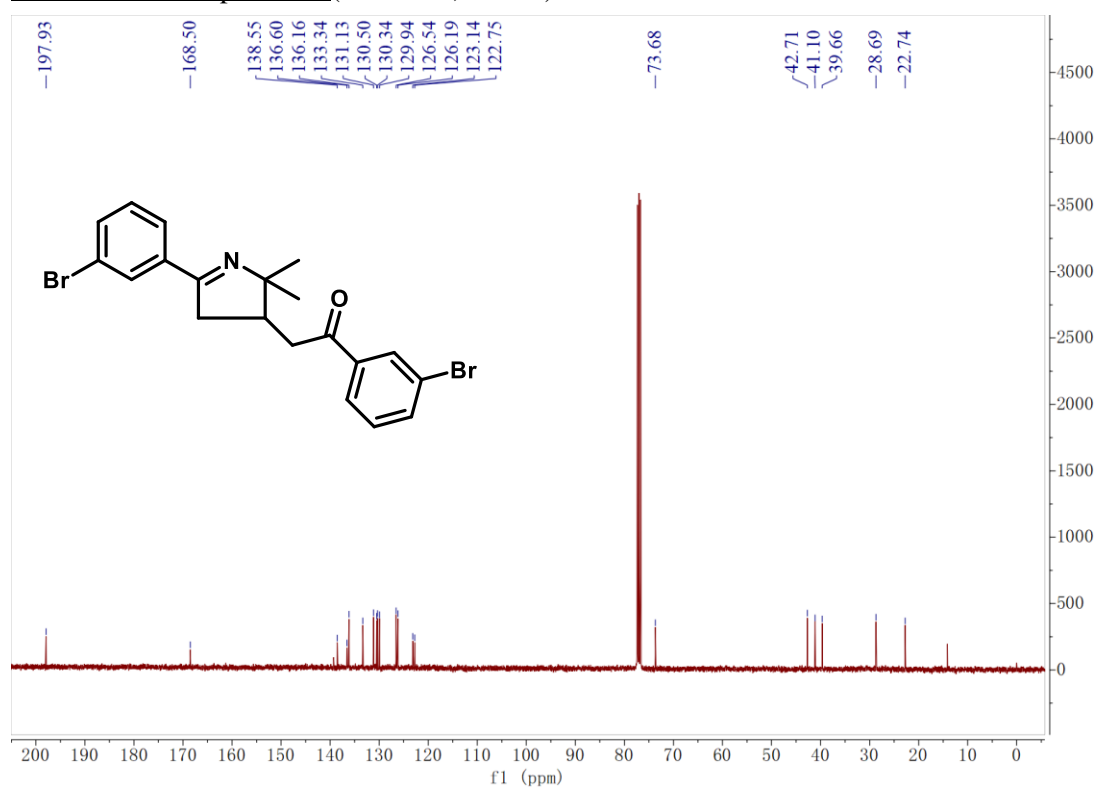
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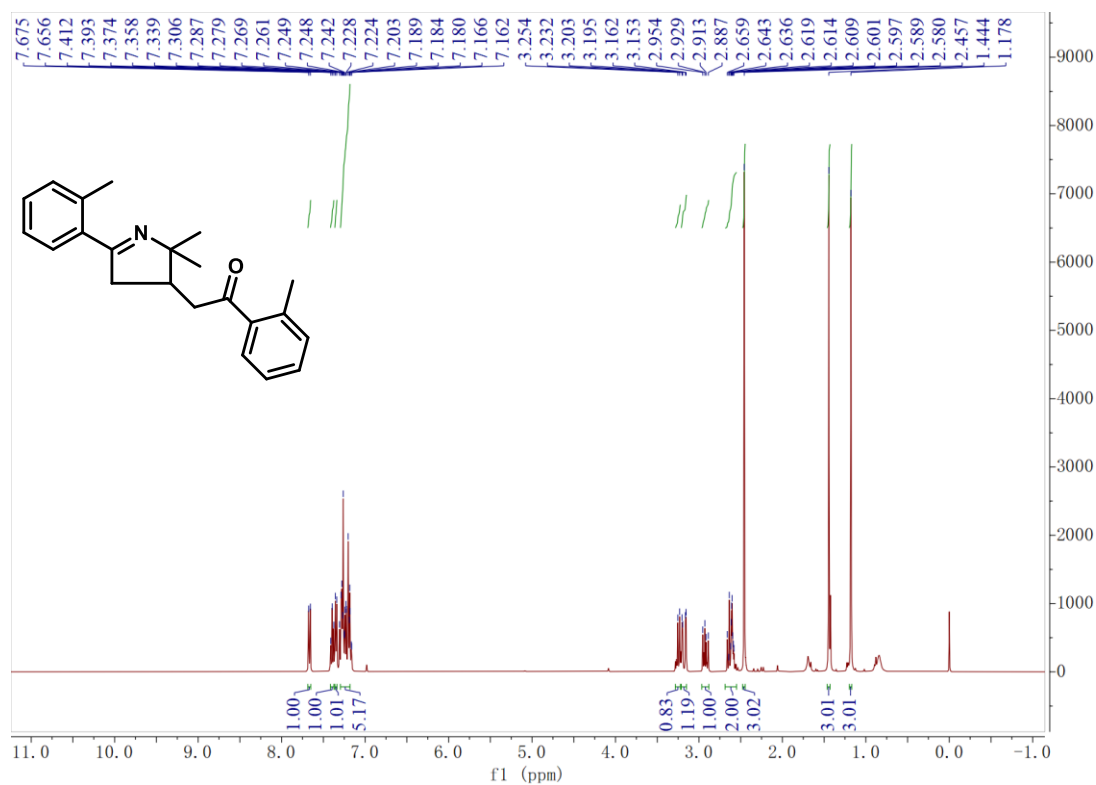
¹H NMR of Compound **5I** (400 MHz, CDCl₃)



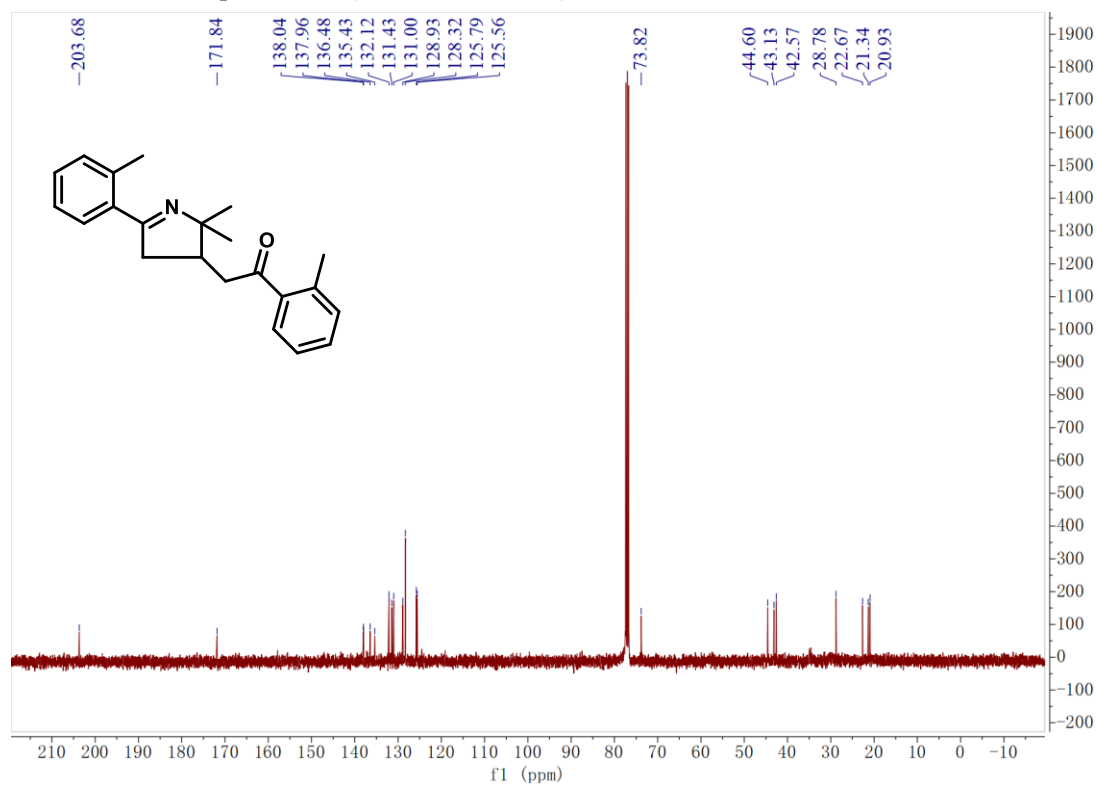
¹³C NMR of Compound **5I** (101 MHz, CDCl₃)



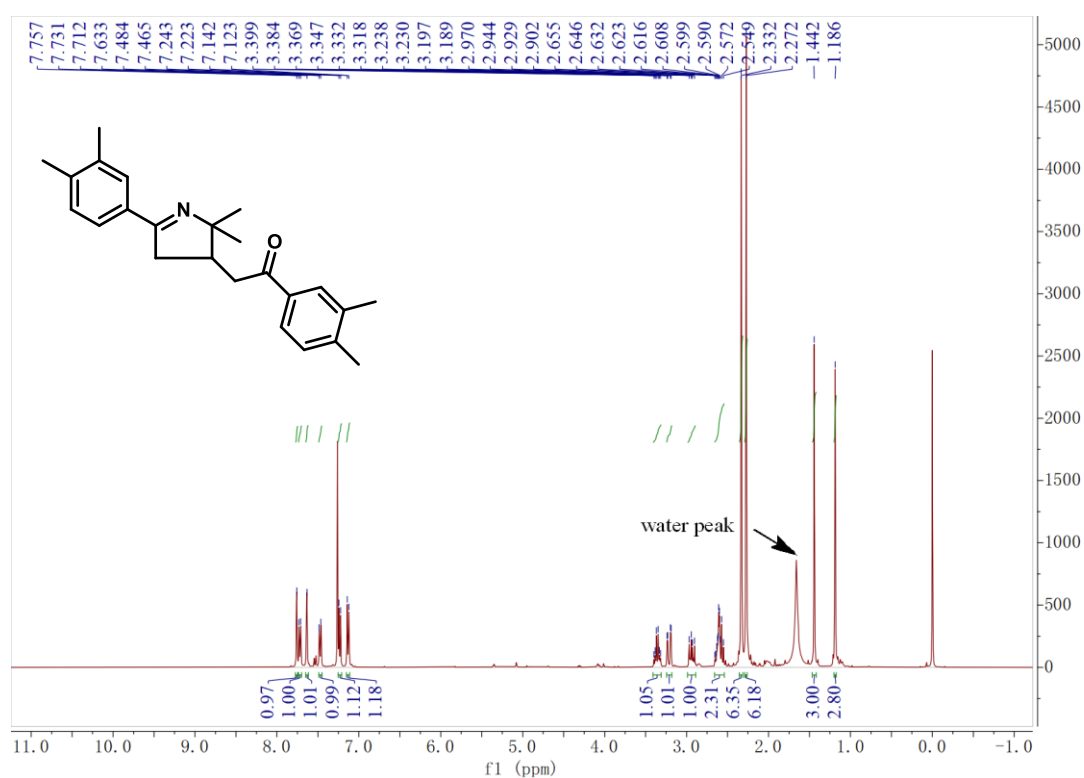
¹H NMR of Compound **5m** (400 MHz, CDCl₃)



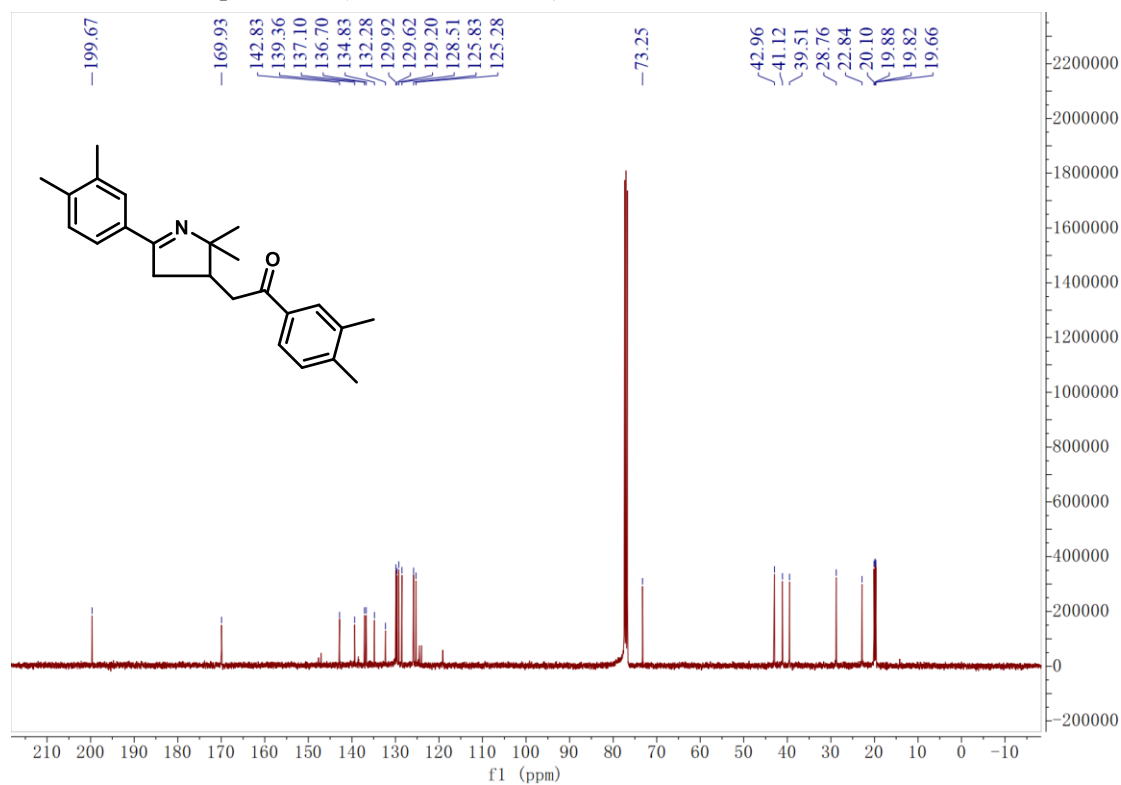
¹³C NMR of Compound **5m** (101 MHz, CDCl₃)



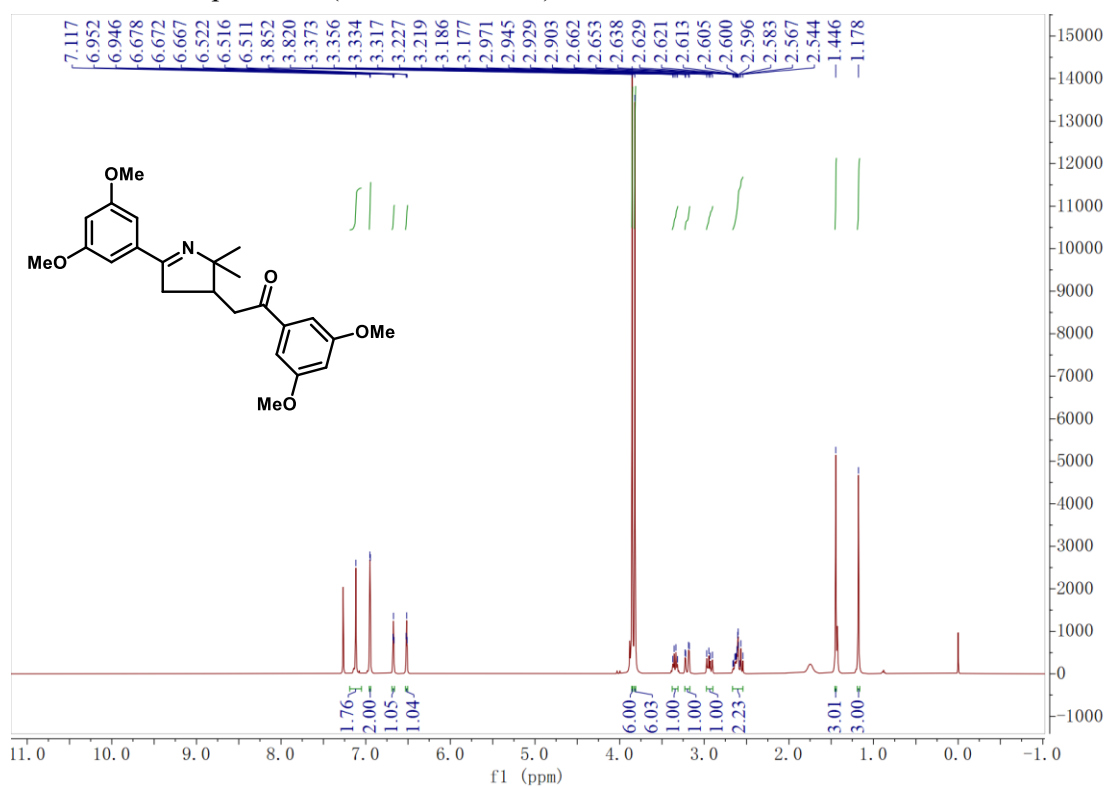
¹H NMR of Compound **5n** (400 MHz, CDCl₃)



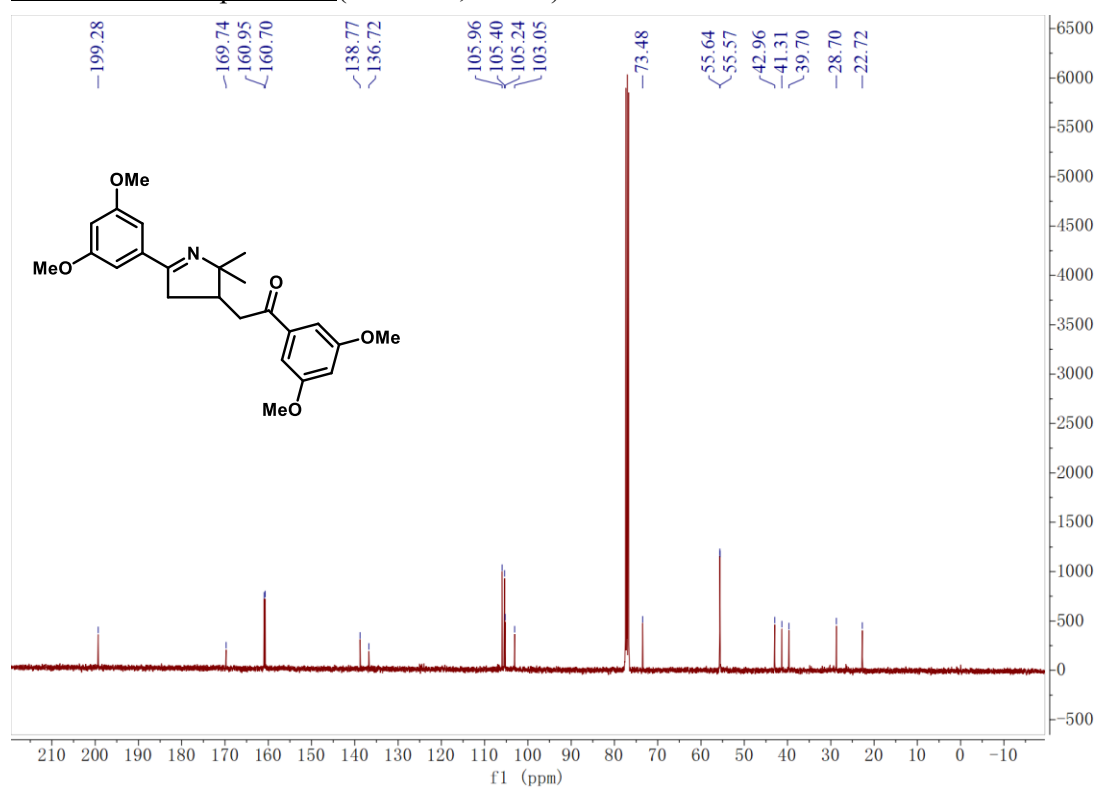
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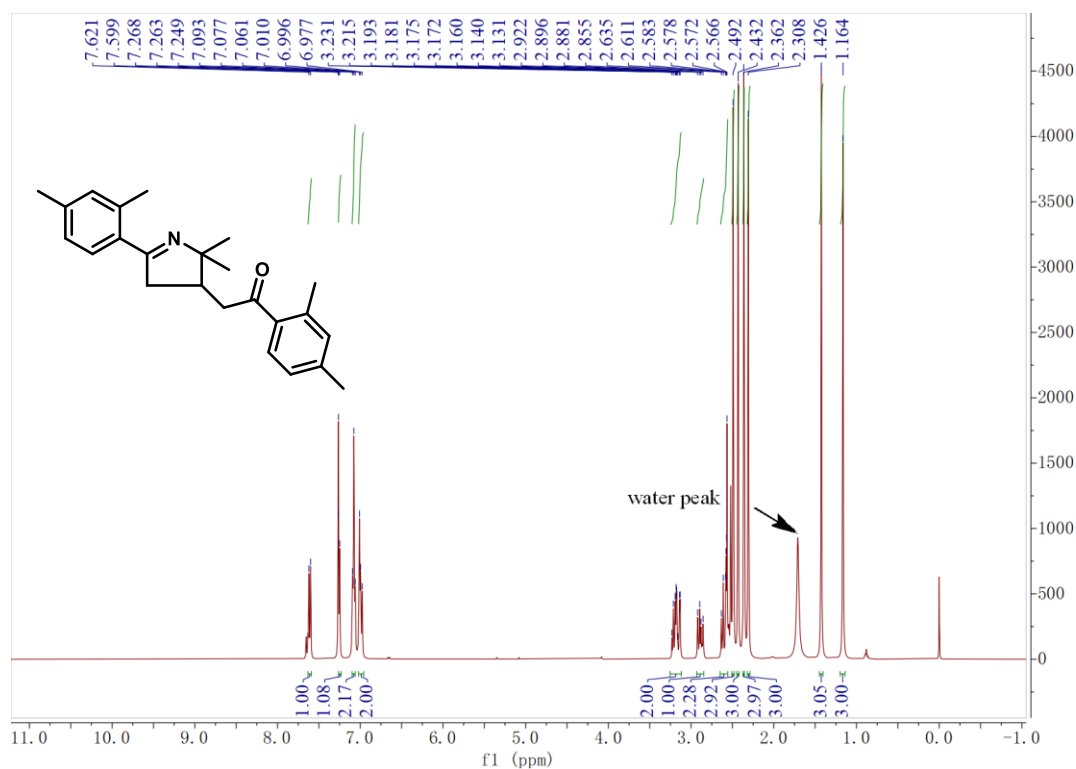
¹H NMR of Compound **5o** (400 MHz, CDCl₃)



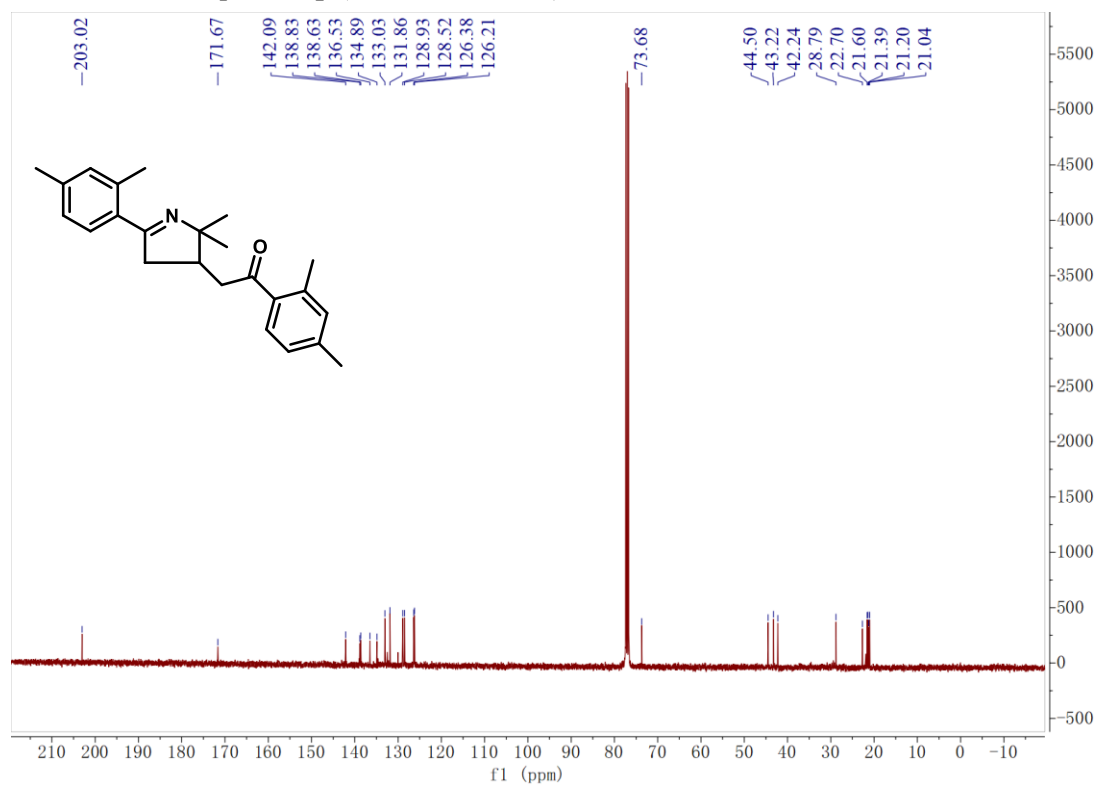
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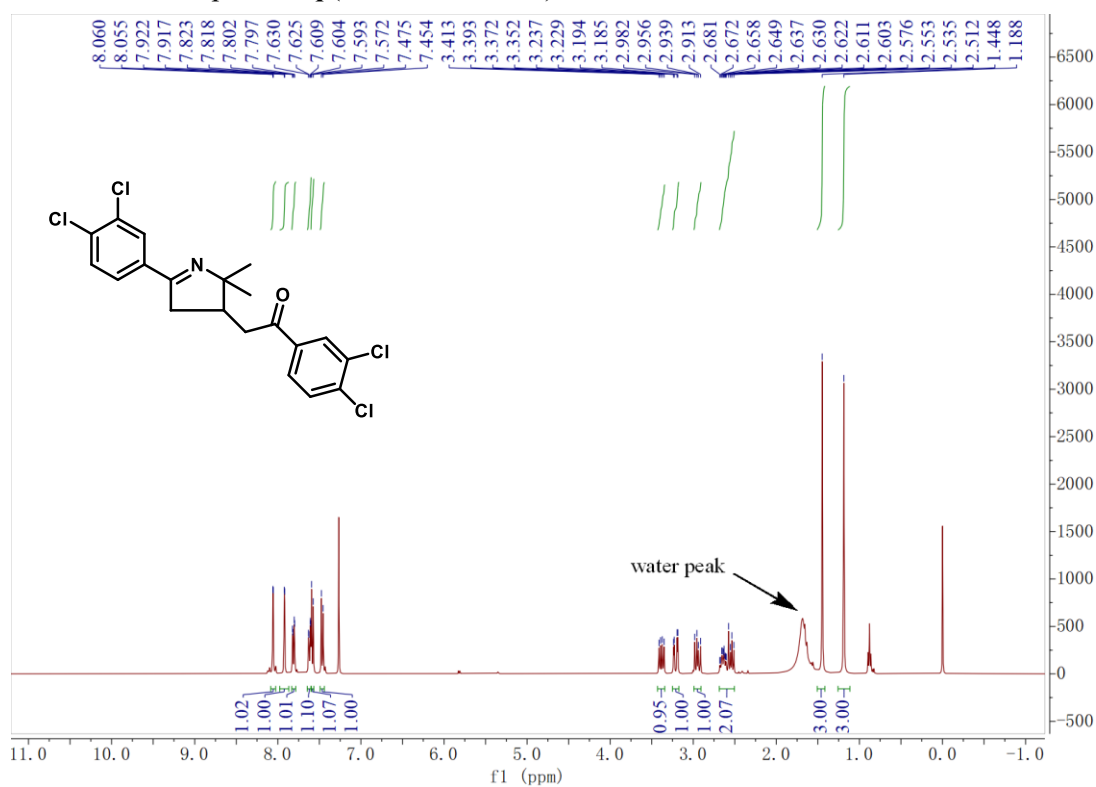
¹H NMR of Compound **5p** (400 MHz, CDCl₃)



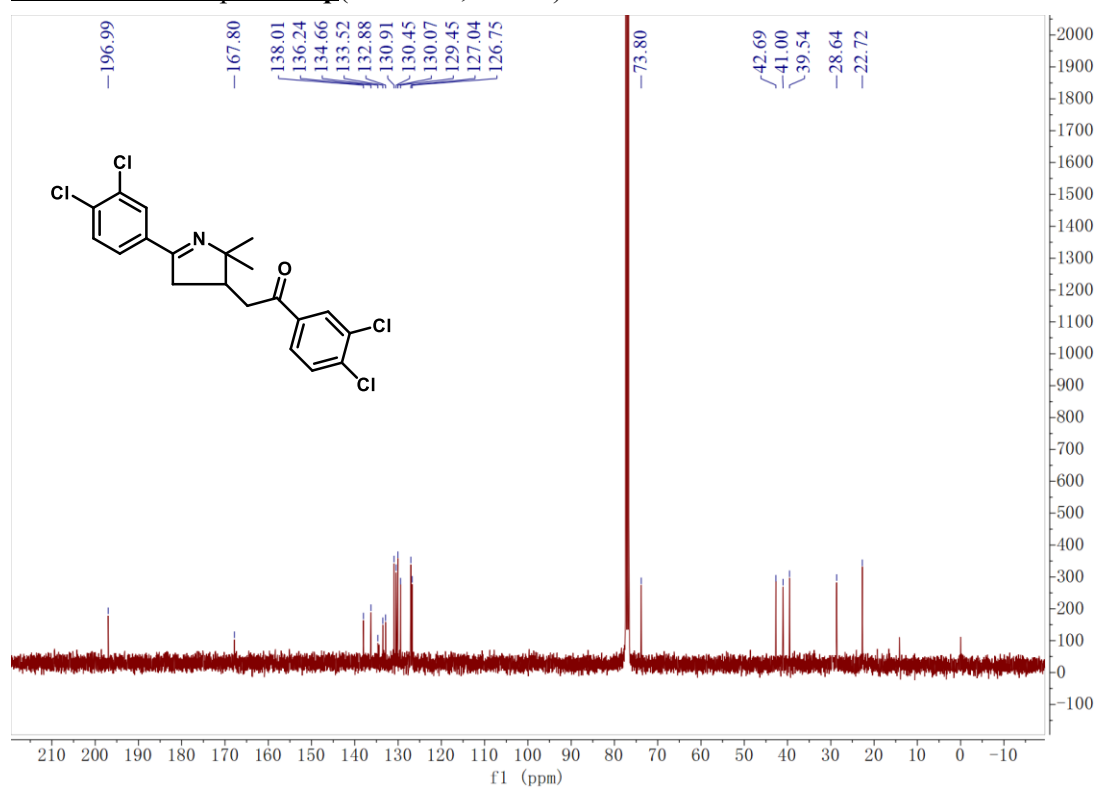
¹³C NMR of Compound **5p** (101 MHz, CDCl₃)



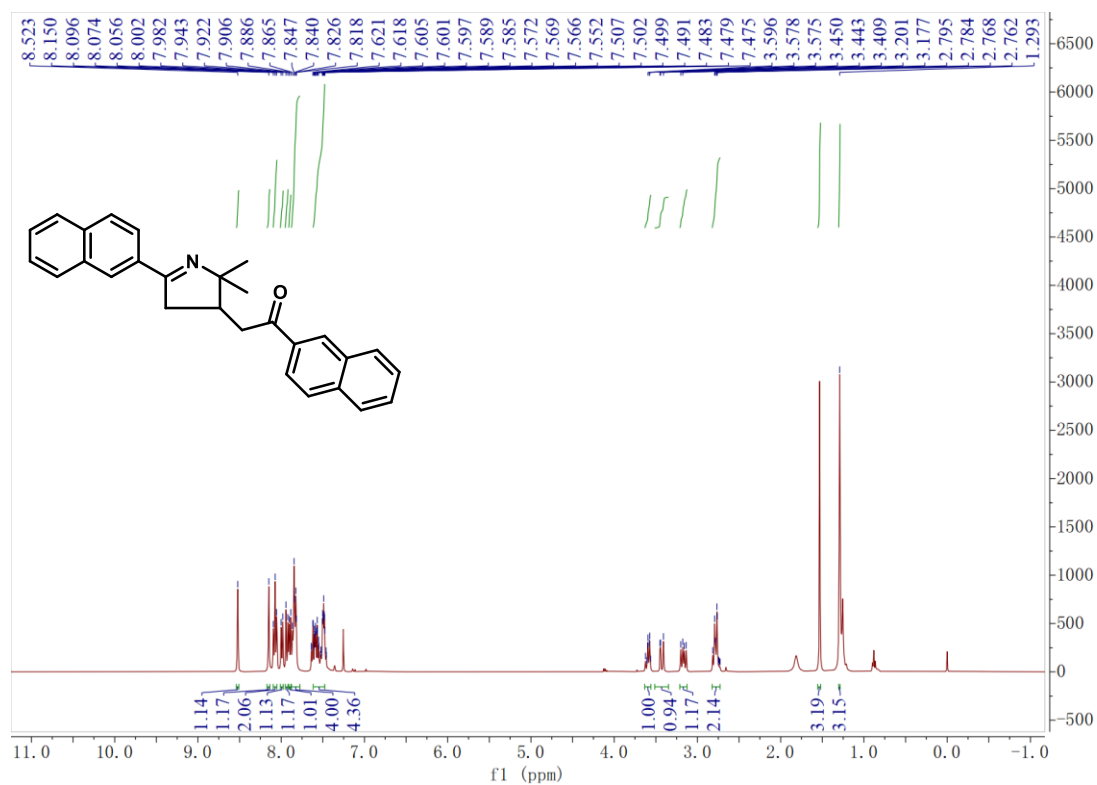
¹H NMR of Compound **5q** (400 MHz, CDCl₃)



¹³C NMR of Compound **5q** (101 MHz, CDCl₃)



¹H NMR of Compound **5r** (400 MHz, CDCl₃)



¹³C NMR of Compound **5r** (101 MHz, CDCl₃)

