

Supporting Information

**Synthesis of Indolines via Palladium-Catalyzed [4+1] Annulation of
(2-Aminophenyl) Methanols with Sulfoxonium Ylides**

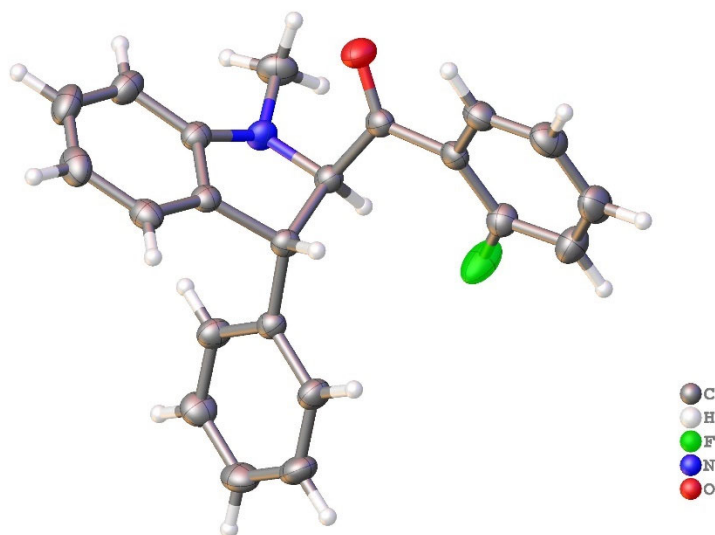
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1. The structure of **3q**



ORTEP representation (50% thermal probability ellipsoids) of the crystal structure of **complex 3q** (CCDC: 2368143)

2. Analytic data of substrates **1** and **2**

(2-(Methylamino)phenyl)(phenyl)methanol (**1a**). White solid, 89% yield, 759 mg; m.p. 127-128 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.29 (m, 5H), 7.28-7.24 (m, 1H), 6.97-6.91 (m, 1H), 6.72 (d, $J = 6.7$ Hz, 2H), 5.81 (s, 1H), 4.59 (s, 1H), 2.78 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 147.7, 141.9, 129.3, 128.6, 128.5, 127.6, 126.9, 126.7, 116.6, 111.0, 75.1, 30.6 (*Angew. Chem. Int. Ed.*, **2015**, *54*, 15540-15544). HRMS (ESI) m/z calcd for $\text{C}_{14}\text{H}_{15}\text{NO}$ ($\text{M} + \text{Na}$) $^+$ 236.1045, found 236.1049.

(2-Aminophenyl)(phenyl)methanol (**1b**). White solid, 91% yield, 1.03 g; m.p. 118-119 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.29 (m, 5H), 7.13 (t, $J = 7.6$ Hz, 1H), 7.03 (d, $J = 7.6$ Hz, 1H), 6.75 (t, $J = 7.6$ Hz, 1H), 6.67 (d, $J = 8.0$ Hz, 1H), 5.86 (s, 1H), 3.96

(s, 2H), 2.73 (s, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 144.9, 142.0, 129.0, 128.7, 128.5, 127.7, 126.6, 118.5, 117.1, 74.9 (*J. Org. Chem.*, **2023**, 88, 12367-12375). HRMS (ESI) m/z calcd for $\text{C}_{13}\text{H}_{14}\text{NO}$ ($\text{M} + \text{H}$) $^+$ 200.1069, found 200.1068.

(2-(Ethylamino)phenyl)(phenyl)methanol (**1c**). White solid, 68% yield, 618 mg; m.p. 67-68 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.40-7.34 (m, 4H), 7.32-7.28 (m, 1H), 7.22 (t, $J = 8.4$ Hz, 1H), 6.97 (d, $J = 7.2$ Hz, 1H), 6.70-6.66 (m, 2H), 5.84 (s, 1H), 4.38 (s, 1H), 3.13-3.02 (m, 2H), 2.59 (s, 1H), 1.15 (t, $J = 7.2$ Hz, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 146.8, 142.1, 129.3, 128.7, 128.5, 127.6, 127.0, 126.6, 116.6, 111.8, 75.2, 38.3, 14.7 (*J. Org. Chem.*, **2023**, 88, 12367-12375). HRMS (ESI) m/z calcd for $\text{C}_{15}\text{H}_{11}\text{NO}$ ($\text{M} + \text{Na}$) $^+$ 250.1202, found 250.1206.

(2-(Benzylamino)phenyl)(phenyl)methanol (**1d**). White solid, 60% yield, 695 mg; m.p. 72-73 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.40-7.35 (m, 5H), 7.31-7.28 (m, 3H), 7.20 (t, $J = 7.6$ Hz, 1H), 7.16 (d, $J = 7.2$ Hz, 1H), 7.03 (d, $J = 7.2$ Hz, 1H), 6.72 (t, $J = 7.6$ Hz, 1H), 6.67 (d, $J = 8.4$ Hz, 1H), 5.86 (s, 1H), 5.05 (s, 1H), 4.29 (s, 2H), 2.66 (s, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 146.3, 141.9, 139.3, 129.3, 128.8, 128.5, 128.4, 127.5, 127.3, 127.0, 126.9, 126.6, 116.7, 111.7, 75.3, 47.7 (*J. Org. Chem.*, **2023**, 88, 12367-12375). HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{20}\text{NO}$ ($\text{M} + \text{Na}$) $^+$ 290.1539, found 290.1530.

(2-(Isopropylamino)phenyl)(phenyl)methanol (**1e**). White solid, 88% yield, 899 mg; m.p. 56-57 °C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 7.36 (d, $J = 7.8$ Hz, 2H), 7.31 (t, $J = 7.5$ Hz, 2H), 7.22 (t, $J = 7.1$ Hz, 1H), 7.07 (t, $J = 7.7$ Hz, 1H), 7.01 (d, $J = 7.5$ Hz, 1H), 6.67-6.37 (m, 2H), 6.08 (q, $J = 8.0$ Hz, 1H), 5.72 (d, $J = 3.9$ Hz, 1H), 4.97 (d, $J = 7.8$ Hz, 1H), 3.59-3.44 (m, 1H), 1.09 (d, $J = 6.2$ Hz, 3H), 0.96 (d, $J = 6.2$ Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, $\text{DMSO}-d_6$) δ 145.7, 143.8, 128.3, 128.1, 127.7, 126.6, 126.3, 115.0, 111.1, 73.3, 43.0, 22.7, 22.4. HRMS (ESI) m/z calcd for $\text{C}_{16}\text{H}_{19}\text{NO}$ ($\text{M} + \text{H}$) $^+$ 242.1539, found 242.1537.

Phenyl(2-(phenylamino)phenyl)methanol (**1f**). White solid, 82% yield, 587 mg; m.p. 110-111 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.42-7.32 (m, 6H), 7.24-7.22 (m, 3H), 7.12(dd, $J = 7.6$ and 1.2 Hz, 1H), 6.97-6.89 (m, 4H), 6.43 (s, 1H), 5.94 (d, $J = 4.4$ Hz, 1H), 2.69 (d, $J = 4.4$ Hz, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 143.4, 142.1, 141.9,

132.0, 129.4, 128.9, 128.8, 128.7, 127.9, 126.8, 121.1, 120.9, 118.7, 118.1, 74.8 (*J. Org. Chem.*, **2023**, *88*, 12367-12375). HRMS (ESI) m/z calcd for $C_{19}H_{17}NO$ ($M + Na$)⁺ 262.0838, found 262.0834.

N-(2-(hydroxy(phenyl)methyl)phenyl)-4-methylbenzenesulfonamide (**1g**). White solid, 83% yield, 223mg; m.p. 147-148 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.46 (s, 1H), 7.52 (d, $J = 8.0$ Hz, 2H), 7.29 (t, $J = 6.4$ Hz, 3H), 7.26-7.21 (m, 3H), 7.19 (d, $J = 6.8$ Hz, 2H), 7.13-7.07 (m, 2H), 6.93-6.91 (m, 1H), 6.25 (s, 1H), 6.03 (s, 1H), 2.35 (s, 3H). ¹³C{¹H} NMR (101 MHz, DMSO-*d*₆) δ 143.92, 143.30, 138.36, 136.81, 134.46, 129.67, 128.45, 127.97, 127.64, 126.90, 126.78, 126.28, 125.39, 122.98, 70.67, 21.01 (*J. Org. Chem.*, **2023**, *88*, 12367-12375). HRMS (ESI) m/z calcd for $C_{20}H_{19}NO_3S$ ($M + Na$)⁺ 376.0977, found 376.0977.

N-(2-(hydroxy(phenyl)methyl)phenyl)acetamide (**1h**). White solid, 75% yield, 137 mg; m.p. 136-137 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.49 (s, 1H), 7.96 (d, $J = 8.0$ Hz, 1H), 7.35-7.27 (m, 6H), 7.12-7.06 (m, 2H), 5.88 (s, 1H), 3.58 (s, 1H), 1.90 (s, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 169.0, 141.7, 136.6, 132.8, 129.0, 128.9, 128.5, 127.7, 126.1, 124.5, 123.5, 75.2, 24.4 (*J. Org. Chem.*, **2023**, *88*, 12367-12375). HRMS (ESI) m/z calcd for $C_{15}H_{15}NO_2$ ($M + Na$)⁺ 264.0995, found 264.1009.

tert-Butyl (2-(hydroxy(phenyl)methyl)phenyl)carbamate (**1i**). White solid, 93% yield, 418 mg; m.p. 141-142 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.82 (d, $J = 8.4$ Hz, 1H), 7.60 (s, 1H), 7.36-7.27 (m, 6H), 7.05-6.99 (m, 2H), 5.92 (d, $J = 3.2$ Hz, 1H), 2.81 (d, $J = 3.2$ Hz, 1H), 1.43 (s, 9H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 153.5, 141.6, 137.2, 132.4, 128.9, 128.7, 128.5, 127.7, 126.4, 123.5, 122.6, 80.3, 75.0, 28.4 (*J. Org. Chem.*, **2023**, *88*, 12367-12375). HRMS (ESI) m/z ($M + Na$)⁺ calcd for $C_{18}H_{21}NO_3$ 322.1413, found 322.1409.

(2-(Methylamino)phenyl)(*p*-tolyl)methanol (**1l**). Yellow solid, 68% yield, 618 mg; m.p. 67-68 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.28-7.23 (m, 3H), 7.18 (d, $J = 7.9$ Hz, 2H), 6.97 (d, $J = 9.1$ Hz, 1H), 6.70 (t, $J = 8.0$ Hz, 2H), 5.81 (s, 1H), 2.79 (s, 3H), 2.37 (s, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 147.7, 139.0, 137.4, 129.3, 128.5, 127.2, 126.7, 116.6, 110.9, 75.0, 30.7, 21.3 (*Angew. Chem. Int. Ed.*, **2015**, *54*, 15540-15544). HRMS (ESI) m/z calcd for $C_{15}H_{17}NO$ [$M + H$]⁺ 228.1382, found 228.1380.

(3-Methoxyphenyl)(2-(methylamino)phenyl)methanol (**1n**). White solid, 69 % yield, 672 mg; m.p. 82 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.30-7.26 (m, 1H), 7.26-7.21 (m, 1H), 7.00-6.91 (m, 3H), 6.84 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.71-6.65 (m, 2H), 5.82 (s, 1H), 3.79 (s, 3H), 2.79 (s, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 159.9, 147.8, 143.8, 129.7, 129.5, 128.7, 126.9, 119.1, 116.7, 113.2, 112.4, 111.0, 75.2, 55.4, 30.7 (*Angew. Chem. Int. Ed.*, **2015**, *54*, 15540-15544). HRMS (ESI) *m/z* calcd for C₁₅H₁₇NO₂ (M + H)⁺ 244.1332, found 244.1335

(4-Fluorophenyl)(2-(methylamino)phenyl)methanol (**1q**). White solid, 83% yield, 768 mg; m.p. 56-57 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.34-7.30 (m, 2H), 7.27-7.23 (m, 1H), 7.05-7.01 (m, 2H), 6.92 (dd, *J* = 7.6, 1.8 Hz, 1H), 6.69 (ddd, *J* = 8.7, 5.6, 1.4 Hz, 2H), 5.80 (s, 1H), 2.77 (s, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 163.5, 161.1, 147.7, 137.7, 129.5, 128.6, 128.4, 128.3, 126.7, 116.7, 115.5, 115.2, 111.2, 74.7, 30.6 (*Angew. Chem. Int. Ed.*, **2015**, *54*, 15540-15544). HRMS (ESI) *m/z* calcd for C₁₄H₁₄FNO (M + H)⁺ 232.1132, found 232.1130.

1-(2-(Methylamino)phenyl)ethan-1-ol (**1s**). Yellow oil, 98% yield, 445 mg. ¹H NMR (400 MHz, CDCl₃) δ 7.23 (t, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 7.4 Hz, 1H), 6.69 (dd, *J* = 12.0, 8.2 Hz, 2H), 4.87 (q, *J* = 6.6 Hz, 1H), 2.86 (s, 3H), 1.58 (d, *J* = 7.6 Hz, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 147.9, 129.0, 127.8, 126.3, 116.4, 110.6, 69.9, 30.5, 21.4. HRMS (ESI) *m/z* calcd for C₉H₁₃NO (M + Na)⁺ 174.0889, found 174.0880.

1-(2-(Methylamino)phenyl)propan-1-ol (**1t**). Yellow oil, 89% yield, 441 mg. ¹H NMR (400 MHz, CDCl₃) δ 7.21 (td, *J* = 7.9, 1.6 Hz, 1H), 7.02 (dd, *J* = 7.3, 1.7 Hz, 1H), 6.71-6.63 (m, 2H), 5.04 (s, 1H), 4.55 (t, *J* = 6.9 Hz, 1H), 2.84 (s, 3H), 1.93 (p, *J* = 7.3 Hz, 2H), 0.95 (t, *J* = 7.4 Hz, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 147.9, 128.9, 127.5, 126.7, 116.2, 110.6, 76.67, 30.5, 27.7, 11.0. HRMS (ESI) *m/z* calcd for C₁₀H₁₅NO (M + H)⁺ 166.1226, found 166.1232.

2-(Dimethyl(oxo)-λ⁶-sulfaneylidene)-1-phenylethan-1-one (**2a**). White solid, 65 % yield, 3.57 g; m.p. 119 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.76 (d, *J* = 8.1 Hz, 2H), 7.45-7.30 (m, 3H), 4.99 (s, 1H), 3.47 (s, 6H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 182.4, 138.9, 130.8, 128.2, 126.6, 68.7, 42.3. HRMS (ESI) *m/z* calcd for C₁₀H₁₂O₂S (M + Na)⁺

197.0630, found 197.0628.

2-(Dimethyl(oxo)- λ^6 -sulfaneylidene)-1-(o-tolyl)ethan-1-one (**2b**). White solid, 55% yield, 3.24 g; m.p. 102 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.38 (d, $J = 8.0$ Hz, 1H), 7.22 (t, $J = 8.0$ Hz, 1H), 7.15 (d, $J = 10.3$ Hz, 2H), 4.64 (s, 1H), 3.49 (s, 3H), 2.45 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 187.0, 141.1, 135.6, 130.9, 129.0, 127.3, 125.4, 71.6, 42.3, 20.2. HRMS (ESI) m/z calcd for $\text{C}_{11}\text{H}_{14}\text{O}_2\text{S}$ ($\text{M} + \text{Na}$) $^+$ 233.0606; Found 233.0608.

2-(Dimethyl(oxo)- λ^6 -sulfaneylidene)-1-(m-tolyl)ethan-1-one (**2c**). White solid, 72% yield, 4.24 g; m.p. 114-115 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.62 (s, 1H), 7.56 (d, $J = 8.0$ Hz, 1H), 7.26-7.24 (m, 1H), 4.96 (s, 1H), 3.49 (s, 6H), 2.36 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 182.7, 138.9, 137.9, 131.6, 128.2, 127.3, 123.8, 68.4, 42.5, 21.5. HRMS (ESI) m/z calcd for $\text{C}_{11}\text{H}_{14}\text{O}_2\text{S}$ ($\text{M} + \text{Na}$) $^+$ 233.0606, found 233.0605.

2-(Dimethyl(oxo)- λ^6 -sulfaneylidene)-1-(p-tolyl)ethan-1-one (**2d**). White solid, 32% yield, 1.88 g; m.p. 141 °C. ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 7.65 (d, $J = 7.8$ Hz, 2H), 7.19 (d, $J = 7.8$ Hz, 2H), 5.56 (s, 1H), 3.54 (s, 6H), 2.32 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, $\text{DMSO-}d_6$) δ 180.40, 139.99, 136.80, 128.65, 126.45, 71.88, 40.65, 39.52, 20.99 (*Org. Lett.*, **2020**, 22, 1375-1379). HRMS (ESI) m/z calcd for $\text{C}_{11}\text{H}_{14}\text{O}_2\text{S}$ ($\text{M} + \text{Na}$) $^+$ 233.0606, found 233.0604.

2-(Dimethyl(oxo)- λ^6 -sulfaneylidene)-1-(4-methoxyphenyl)ethan-1-one (**2e**). White solid, 36% yield, 2.28 g; m.p. 132 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.76 (d, $J = 8.4$ Hz, 2H), 6.88 (d, $J = 6.7$ Hz, 2H), 4.91 (s, 1H), 3.83 (s, 3H), 3.50 (s, 6H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 181.9, 161.7, 131.5, 128.3, 113.3, 68.0, 55.3, 42.4. HRMS (ESI-TOF) m/z calcd for $\text{C}_{11}\text{H}_{14}\text{O}_3\text{S}$ ($\text{M} + \text{Na}$) $^+$ 249.0555, found 249.0555.

2-(Dimethyl(oxo)- λ^6 -sulfaneylidene)-1-(4-(trifluoromethyl)phenyl)ethan-1-one (**2f**). White solid, 77% yield, 5.70 g; m.p. 142 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.89 (d, $J = 8.1$ Hz, 2H), 7.65 (d, $J = 8.3$ Hz, 2H), 5.01 (s, 1H), 3.53 (s, 6H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 180.7, 141.7, 132.4, 132.1, 127.0, 125.3, 69.9, 42.3 (d, $J = 2.8$ Hz). HRMS (ESI) m/z calcd for $\text{C}_{11}\text{H}_{11}\text{F}_3\text{O}_2\text{S}$ ($\text{M} + \text{Na}$) $^+$ 287.0324, found 287.0328.

1-(4-Chlorophenyl)-2-(dimethyl(oxo)- λ^6 -sulfaneylidene)ethan-1-one (**2g**). White solid, 74% yield, 4.78 g; m.p. 153-154 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.70 (d, $J =$

8.6 Hz, 2H), 7.32 (d, $J = 8.6$ Hz, 2H), 4.95 (s, 1H), 3.48 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 181.0, 137.4, 136.8, 128.4, 128.0, 68.9, 42.4. HRMS (ESI) m/z calcd for $\text{C}_{10}\text{H}_{11}\text{ClO}_2\text{S}$ ($\text{M} + \text{Na}$) $^+$ 253.0060, found 253.0062.

4-(2-(Dimethyl(oxo)- λ^6 -sulfaneylidene)acetyl)benzonitrile (**2h**). Yellow solid, 13% yield, 805 mg; m.p. 197-198 °C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 7.92 (d, $J = 8.4$ Hz, 2H), 7.86 (d, $J = 8.4$ Hz, 2H), 5.76 (s, 1H), 3.57 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, $\text{DMSO}-d_6$) δ 178.0, 143.4, 132.3, 127.2, 118.8, 112.6, 74.2. HRMS (ESI) m/z calcd for $\text{C}_{11}\text{H}_{11}\text{NO}_2\text{S}$ ($\text{M} + \text{Na}$) $^+$ 244.0402, found 244.0406.

2-(Dimethyl(oxo)- λ^6 -sulfaneylidene)-1-(2-fluorophenyl)ethan-1-one (**2i**). Yellow solid, 70% yield, 4.20 g; m.p. 93-94 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.88 (td, $J = 7.8$, 1.9 Hz, 1H), 7.37–7.32 (m, 1H), 7.18–7.14 (m, 1H), 7.05–7.0 (m, 1H), 5.14 (s, 1H), 3.50 (s, 6H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 177.5, 162.1, 159.6, 132.0, 130.1, 126.8, 124.1, 116.1, 73.7, 42.3. HRMS (ESI) m/z calcd for $\text{C}_{10}\text{H}_{11}\text{FO}_2\text{S}$ ($\text{M} + \text{Na}$) $^+$ 237.0356, found 237.0332.

2-(Dimethyl(oxo)- λ^6 -sulfaneylidene)-1-(4-fluorophenyl)ethan-1-one (**2j**). White solid, 76% yield, 4.56 g; m.p. 119 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.99-7.96 (m, 2H), 7.24 (t, $J = 8.8$ Hz, 2H), 5.12 (s, 1H), 3.69 (s, 6H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 181.14, 165.71, 163.22, 135.17, 128.9, 128.8, 115.2, 115.0, 68.47, 42.45. HRMS (ESI) m/z calcd for $\text{C}_{10}\text{H}_{11}\text{FO}_2\text{S}$ ($\text{M} + \text{Na}$) $^+$ 237.0356, found 237.0357.

2-(Dimethyl(oxo)- λ^6 -sulfaneylidene)-1-(naphthalen-2-yl)ethan-1-one (**2k**). White solid, 44% yield, 3.03 g; m.p. 178-179 °C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 8.35 (s, 1H), 8.02 - 7.97 (m, 1H), 7.95 - 7.88 (m, 3H), 7.58 - 7.51 (m, 2H), 5.78 (s, 1H), 3.60 (s, 6H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, $\text{DMSO}-d_6$) δ 180.2, 136.9, 134.0, 132.5, 128.9, 127.6, 127.6, 127.1, 126.4, 126.2, 124.0, 72.8, 40.6. HRMS (ESI) m/z calcd for $\text{C}_{14}\text{H}_{14}\text{O}_2\text{S}$ ($\text{M} + \text{H}$) $^+$ 247.0787, found 247.0781.

1-(Dimethyl(oxo)- λ^6 -sulfaneylidene)propan-2-one (**2l**). Yellow oil, 54% yield, 2.03 g. ^1H NMR (400 MHz, CDCl_3) δ 4.38 (s, 1H), 3.37 (d, $J = 2.6$ Hz, 6H), 1.92 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 188.2, 69.8, 42.2, 27.8. HRMS (ESI) m/z calcd for $\text{C}_5\text{H}_{10}\text{O}_2\text{S}$ ($\text{M} + \text{H}$) $^+$ 135.0474, found 135.0470.

3. Copies of NMR spectra

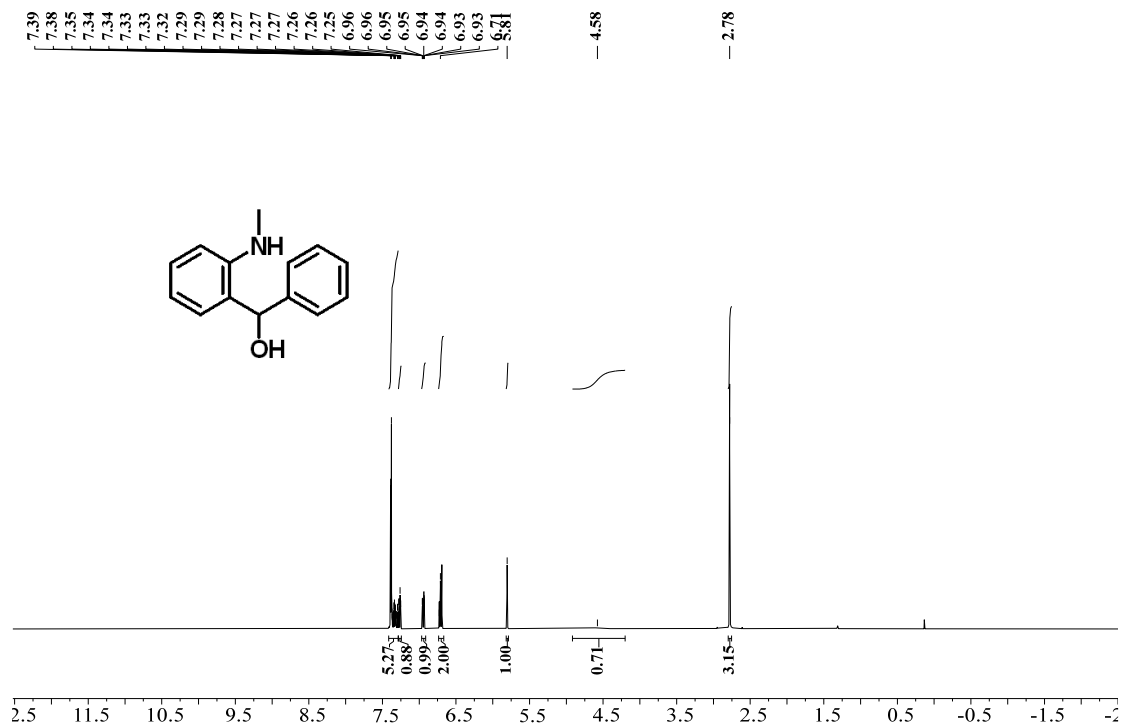


Figure S1. ^1H NMR spectrum of **1a** (CDCl_3 , 400 MHz, 23 °C)

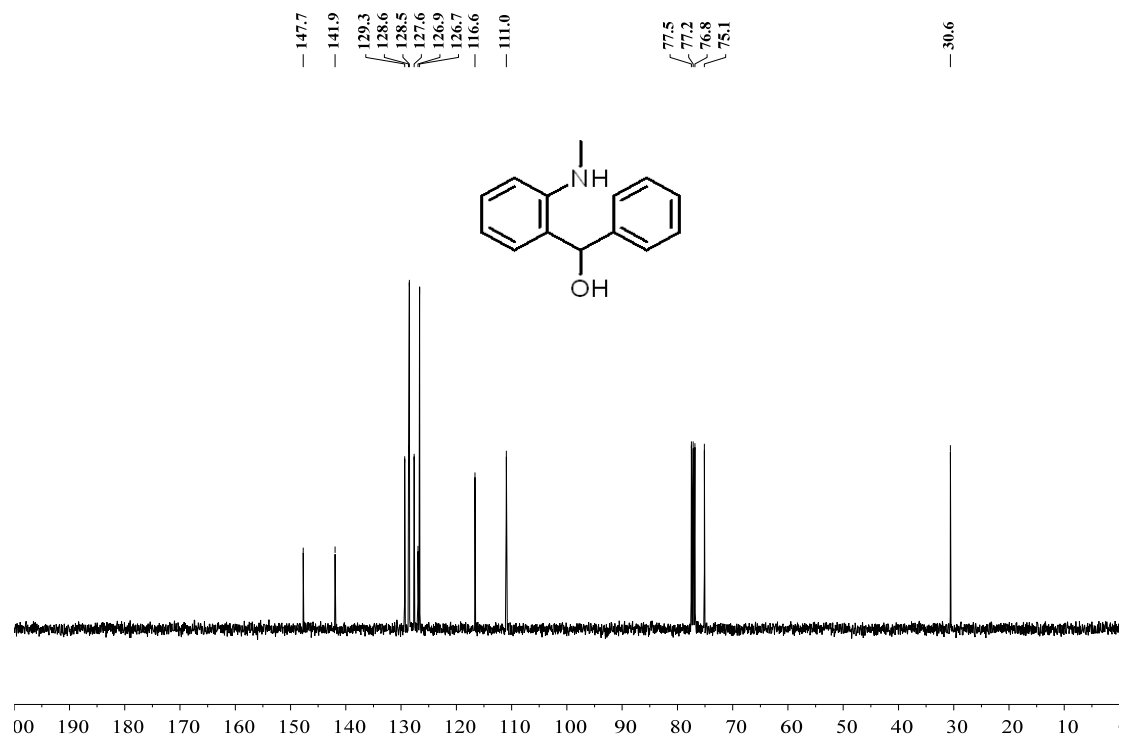


Figure S2. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1a** (CDCl_3 , 101 MHz, 23 °C)

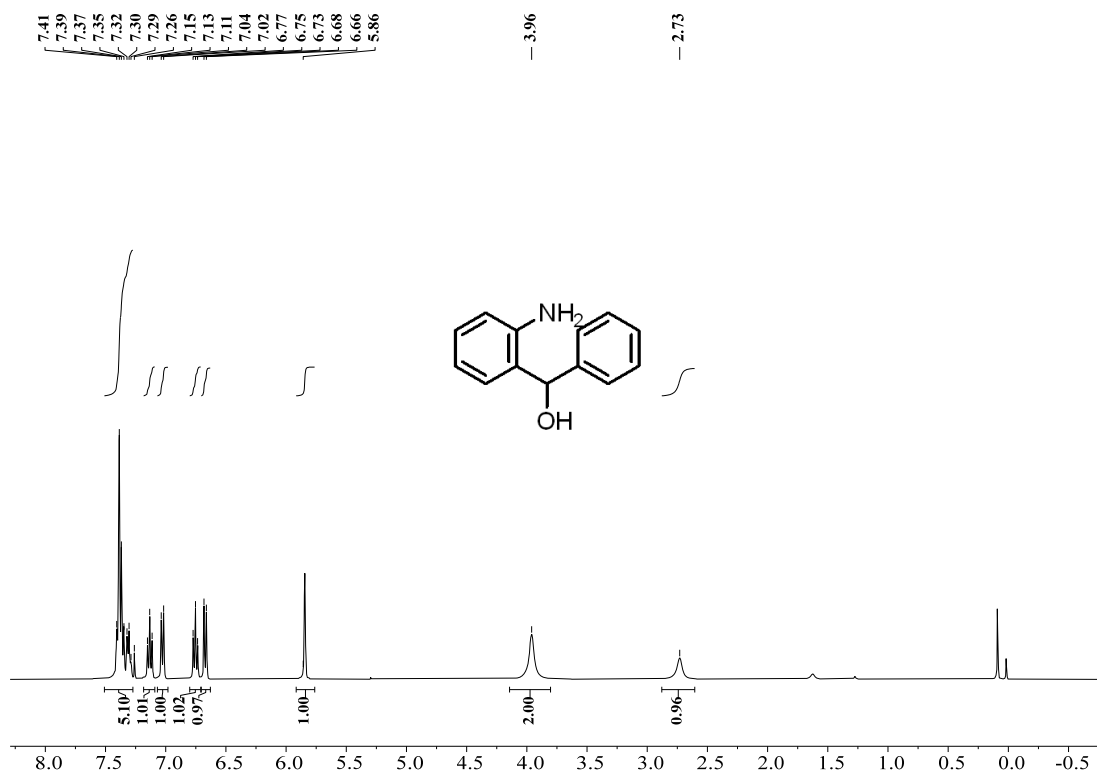


Figure S3. ^1H NMR spectrum of **1b** (CDCl_3 , 400 MHz, 23 °C)

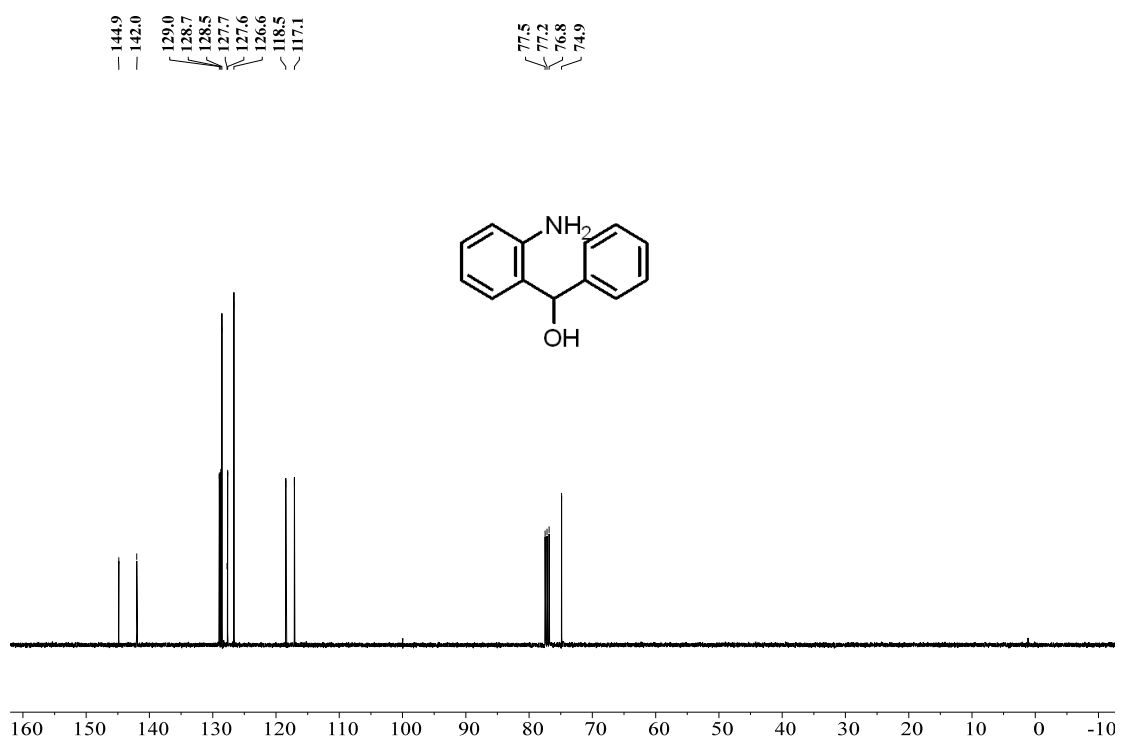


Figure S4. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1b** (CDCl_3 , 101 MHz, 23 °C)

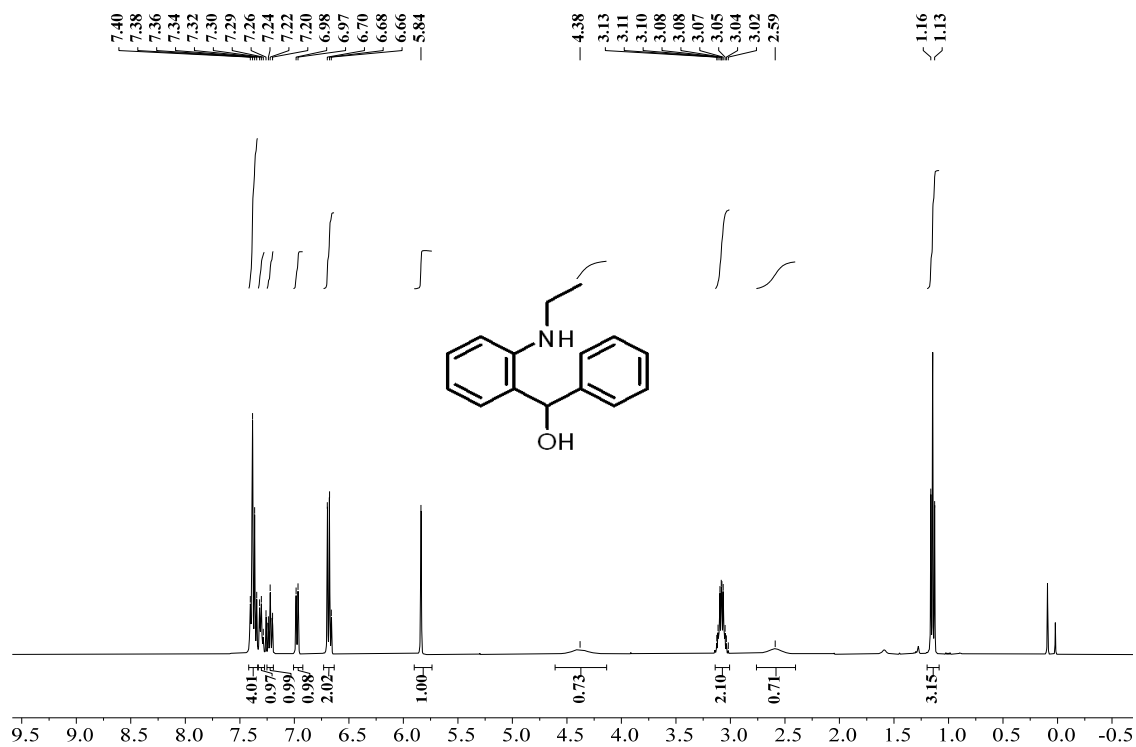


Figure S5. ^1H NMR spectrum of **1c** (CDCl_3 , 400 MHz, 23 °C)

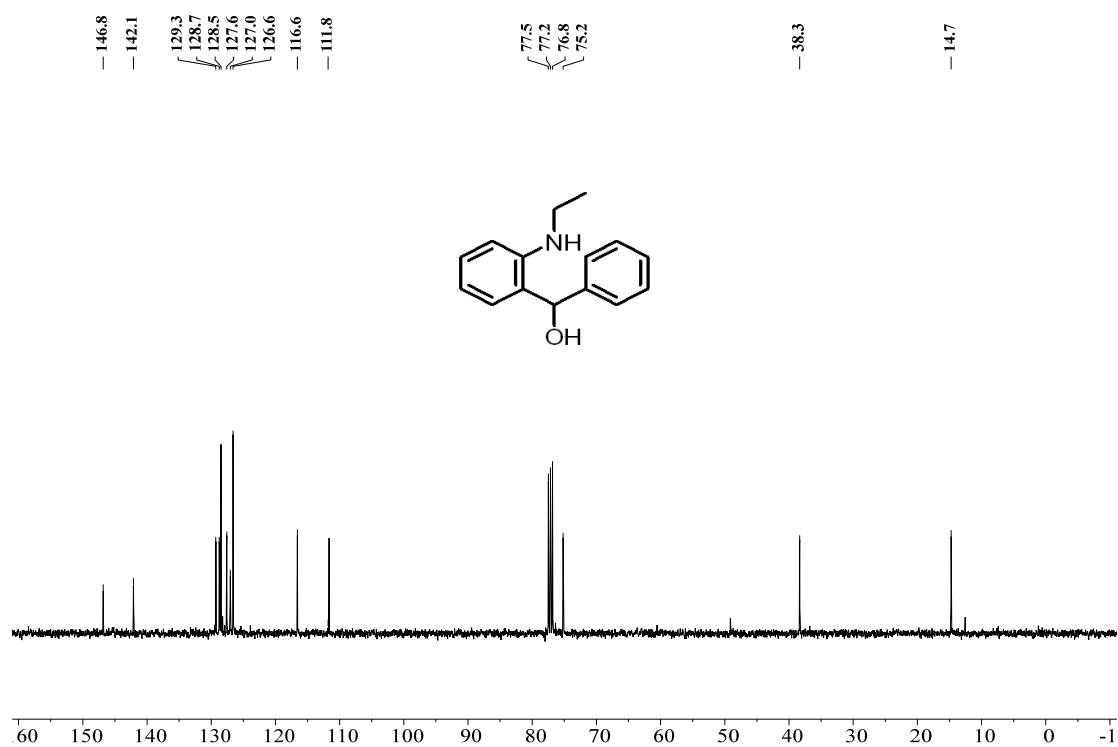


Figure S6. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1c** (CDCl_3 , 101 MHz, 23 °C)

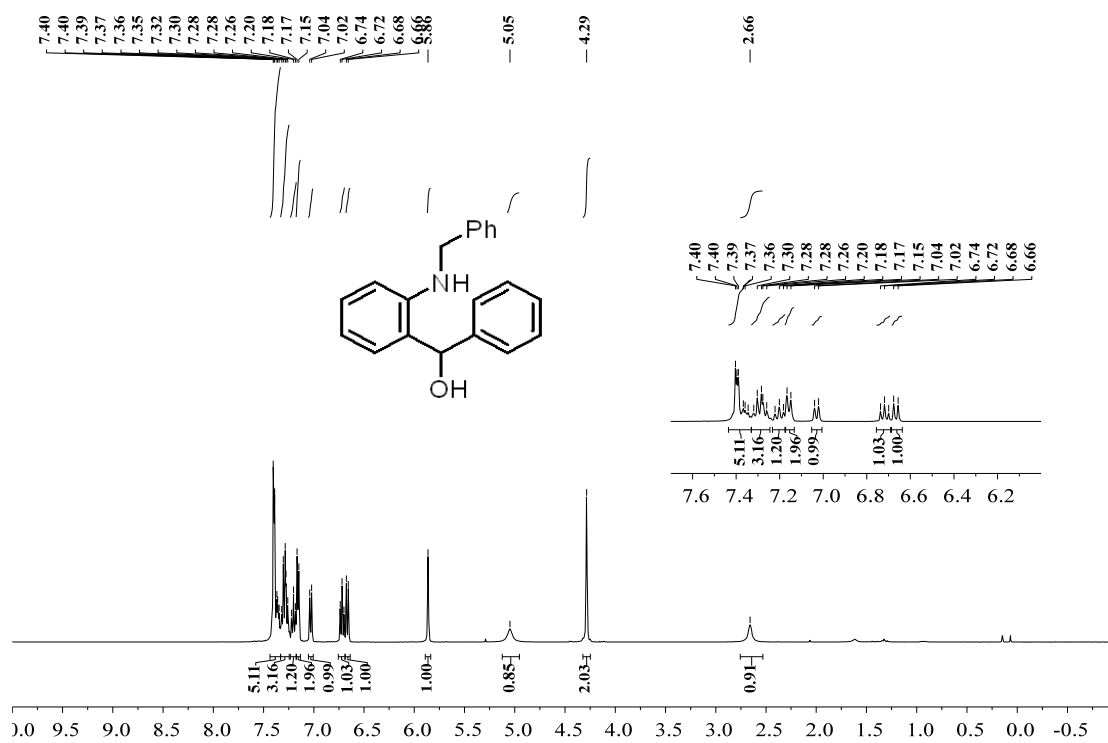


Figure S7. ^1H NMR spectrum of **1d** (CDCl₃, 400 MHz, 23 °C)

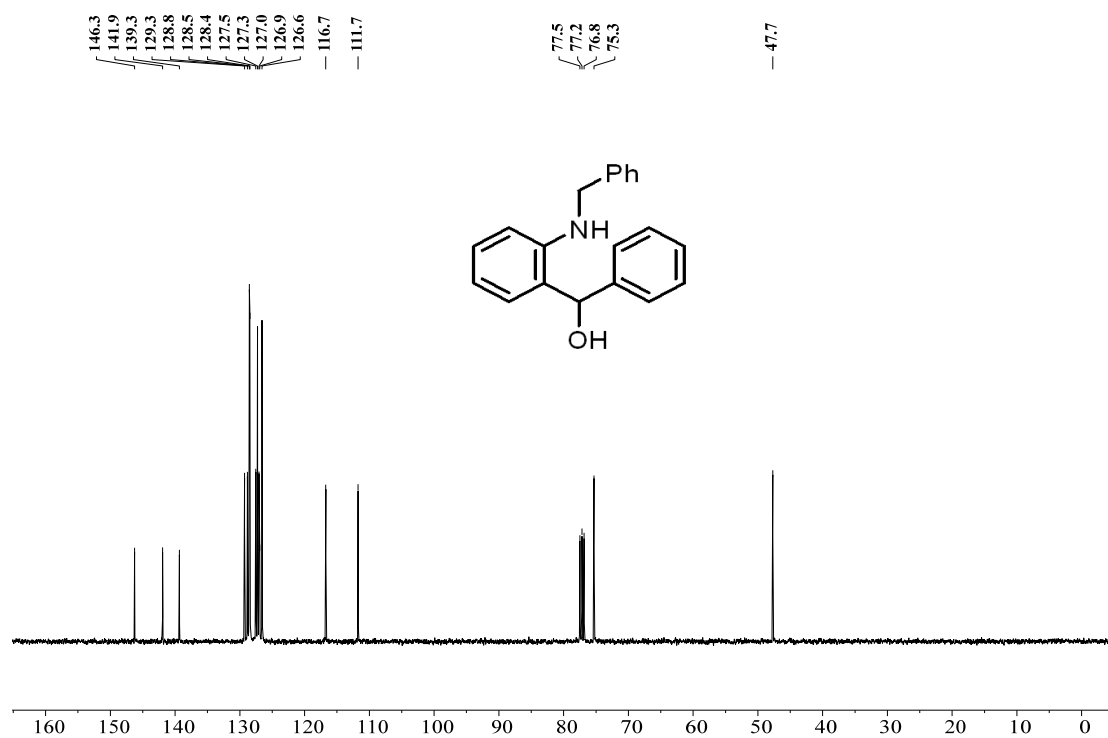


Figure S8. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1d** (CDCl₃, 101 MHz, 23 °C)

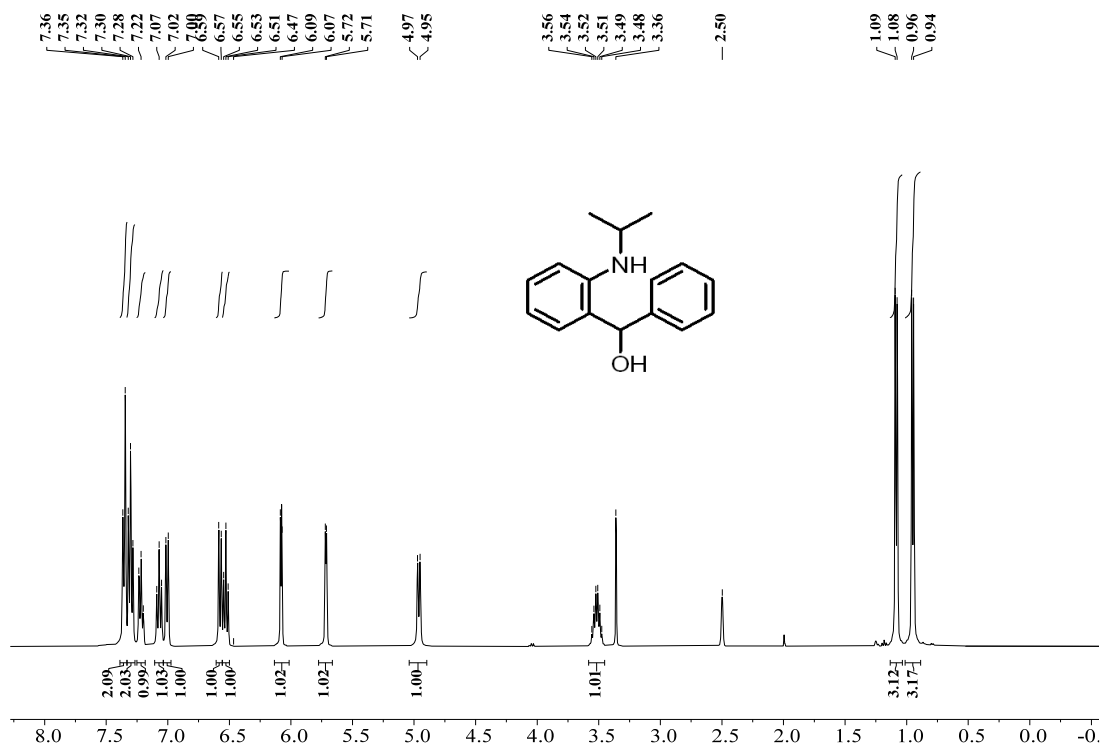


Figure S9. ¹H NMR spectrum of **1e** (DMSO-*d*₆, 400 MHz, 23 °C)

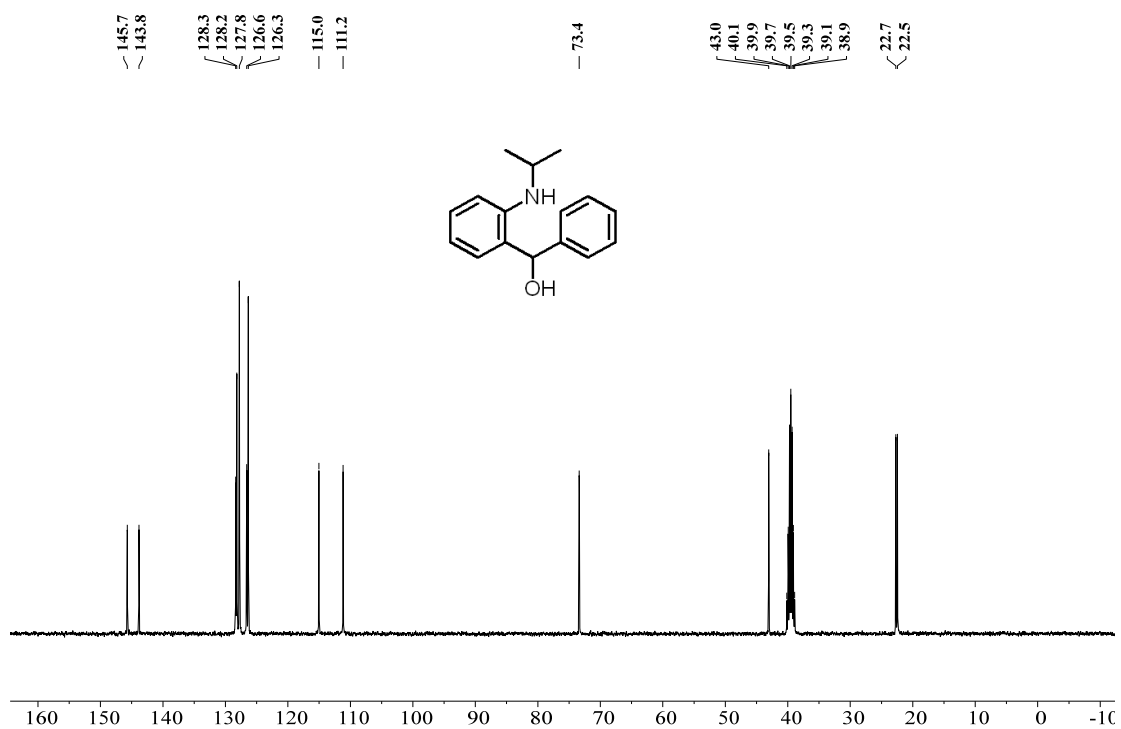


Figure S10. ¹³C{¹H} NMR spectrum of **1e** (DMSO-*d*₆, 101 MHz, 23 °C)

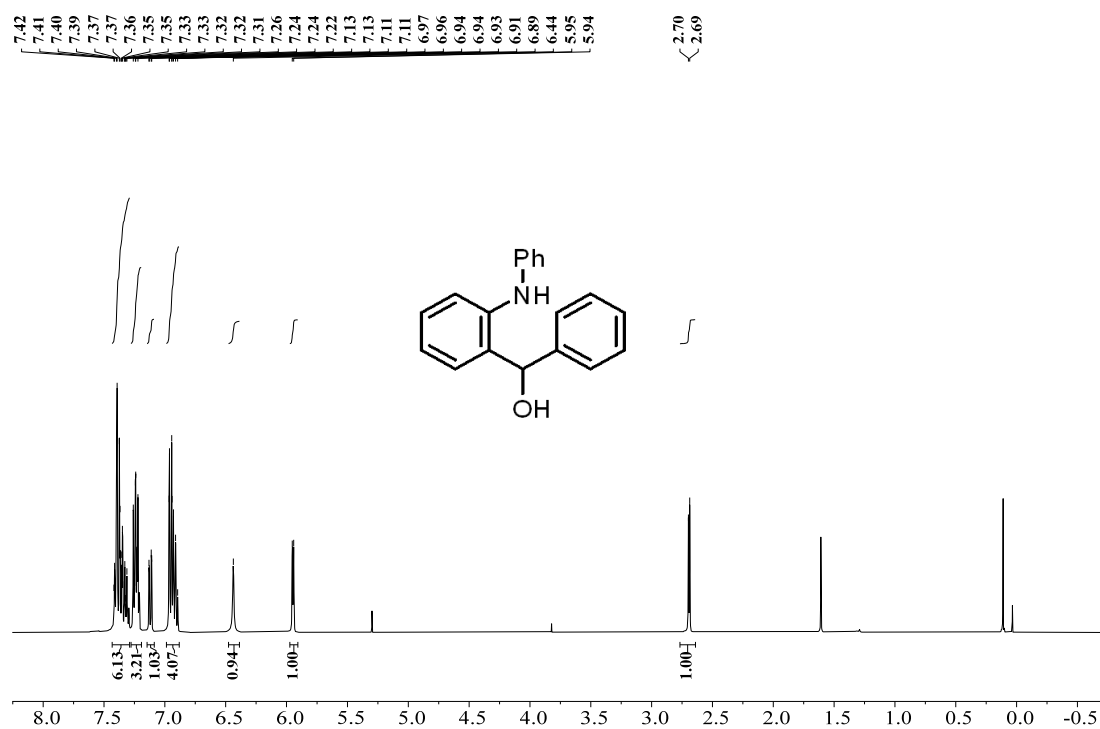


Figure S11. ^1H NMR spectrum of **1f** (CDCl_3 , 400 MHz, 23 °C)

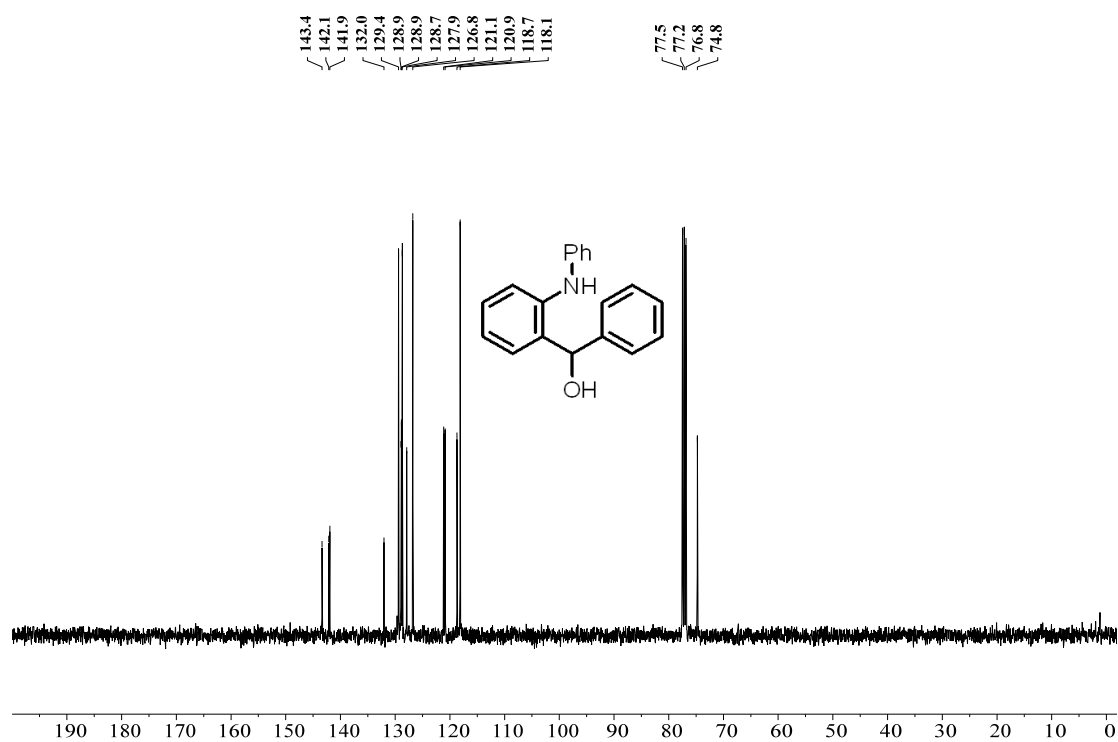


Figure S12. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1f** (CDCl_3 , 101 MHz, 23 °C)

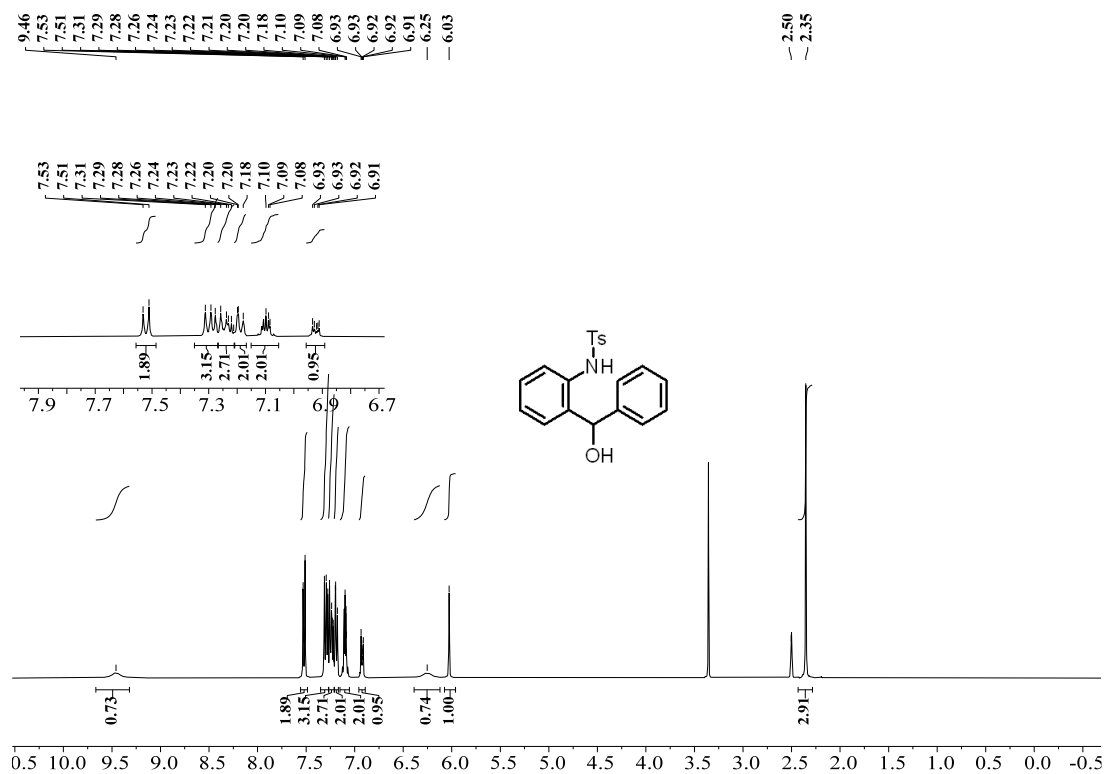


Figure S13. ^1H NMR spectrum of **1g** (DMSO- d_6 , 400 MHz, 23 °C)

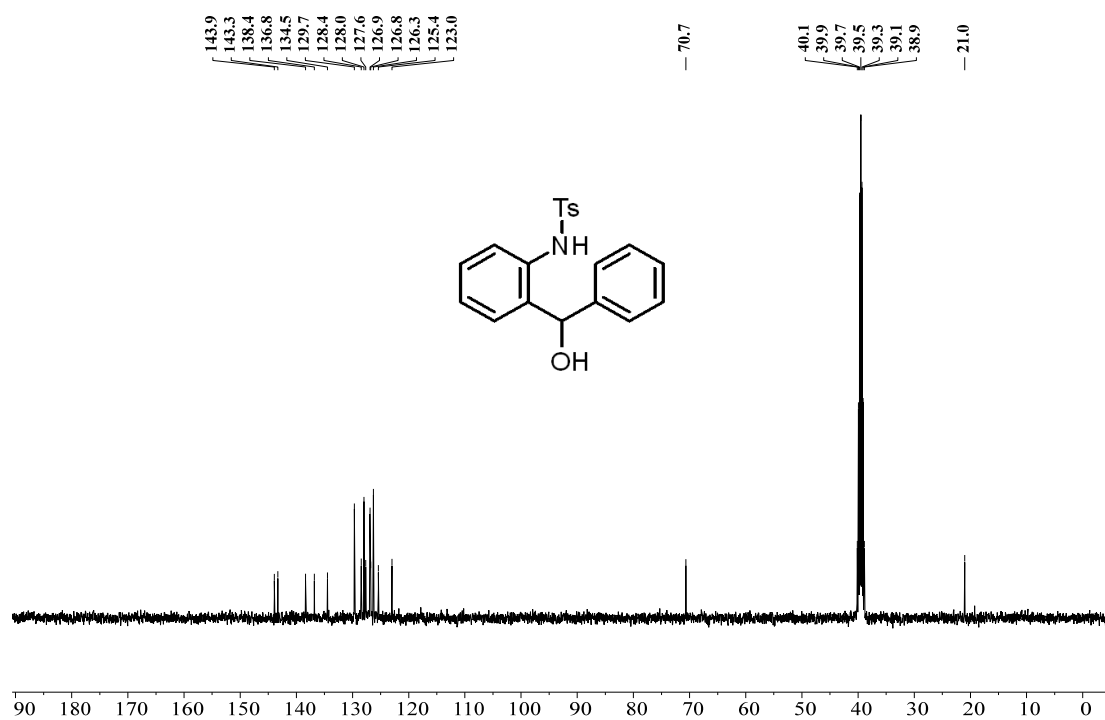


Figure S14. ^{13}C $\{^1\text{H}\}$ NMR spectrum of **1g** (DMSO- d_6 , 101 MHz, 23 °C)

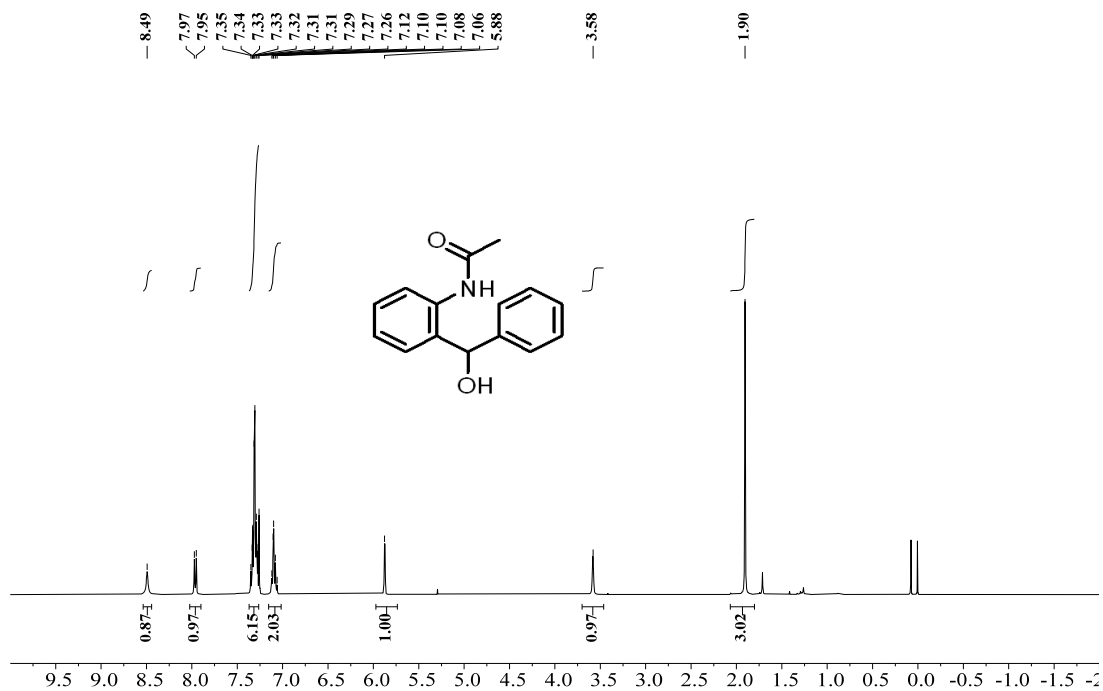


Figure S15. ^1H NMR spectrum of **1h** (CDCl_3 , 400 MHz, 23 °C)

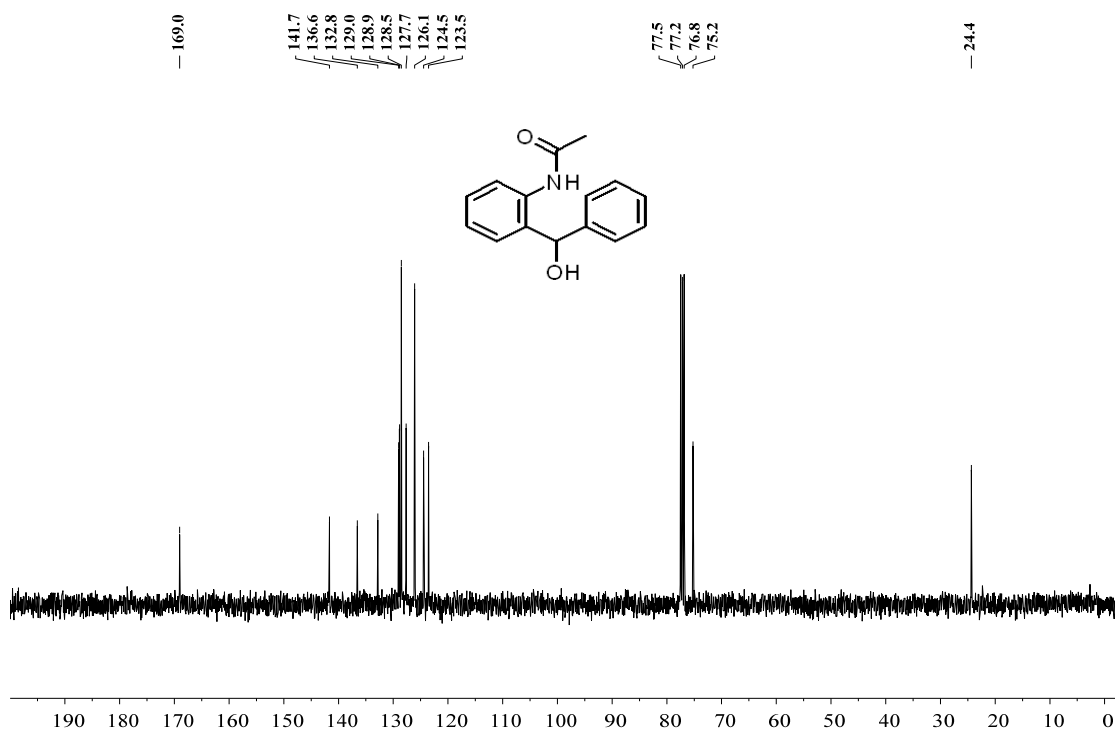


Figure S16. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1h** (CDCl_3 , 101 MHz, 23 °C)

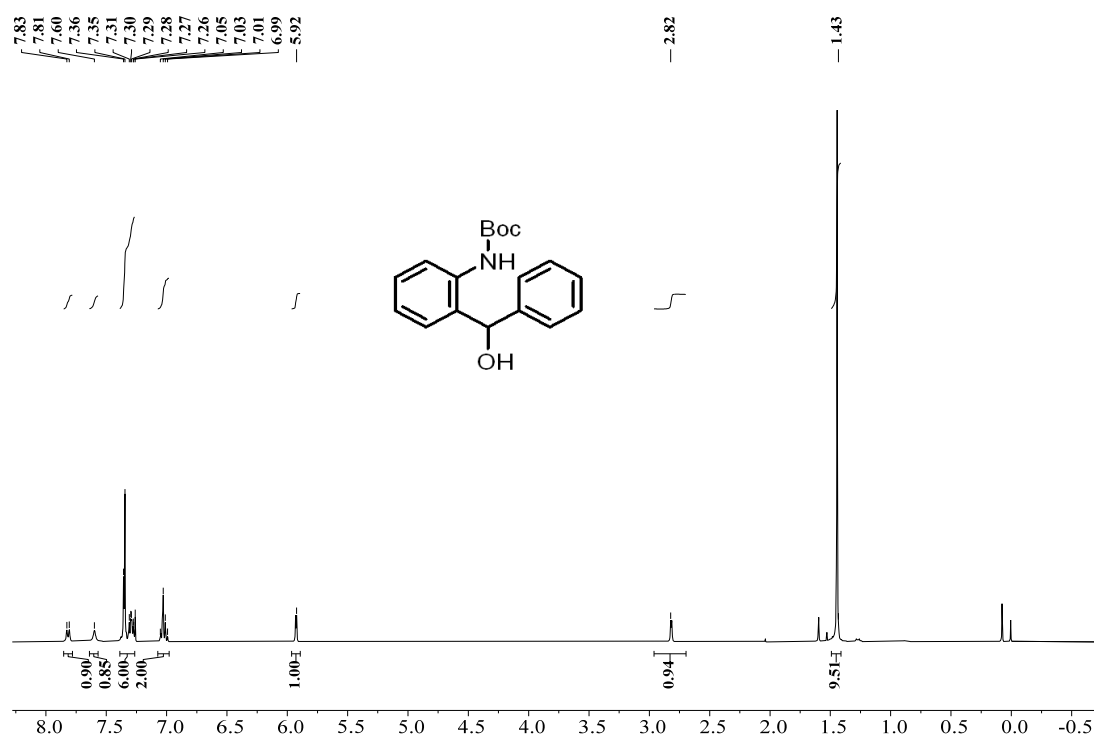


Figure S17. ^1H NMR spectrum of **1i** (CDCl_3 , 400 MHz, 23 °C)

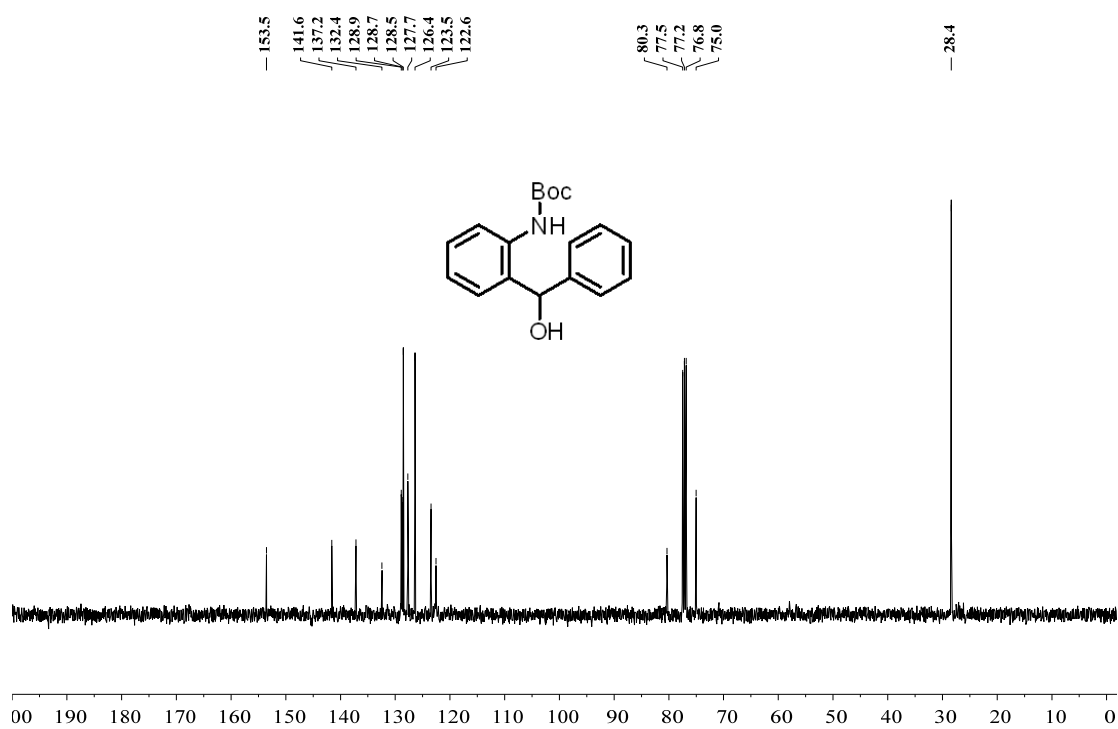


Figure S18. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1i** (CDCl_3 , 101 MHz, 23 °C)

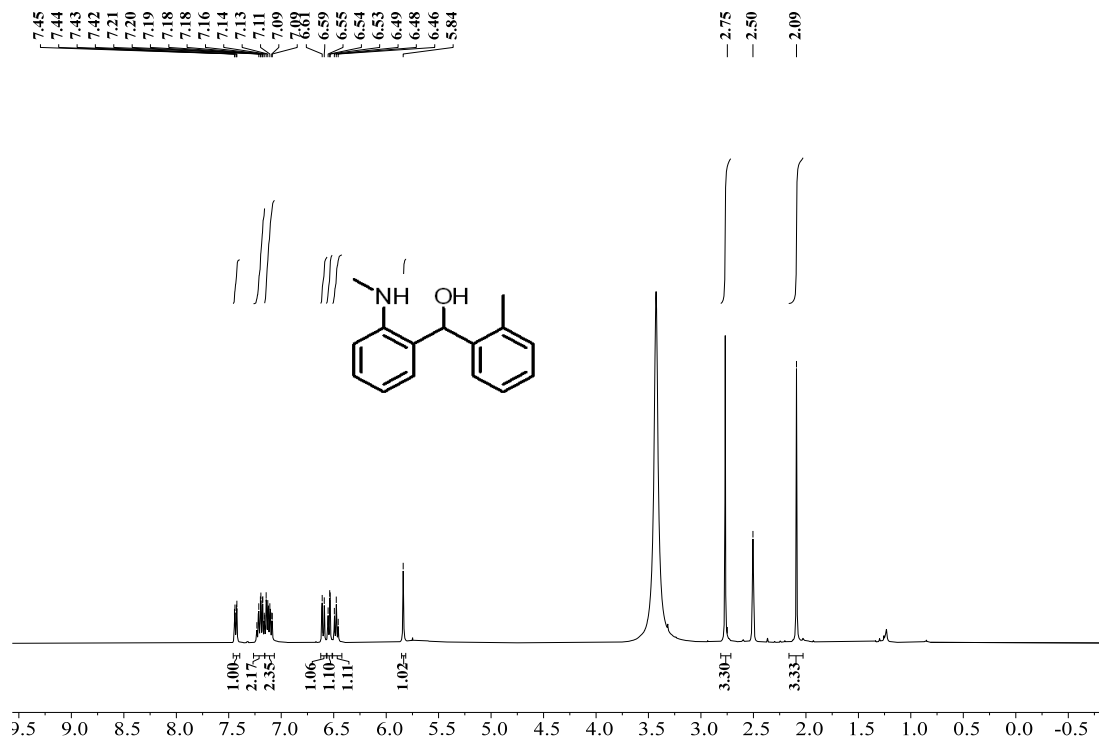


Figure S19. ¹H NMR spectrum of **1j** (DMSO-*d*₆, 400 MHz, 23 °C)

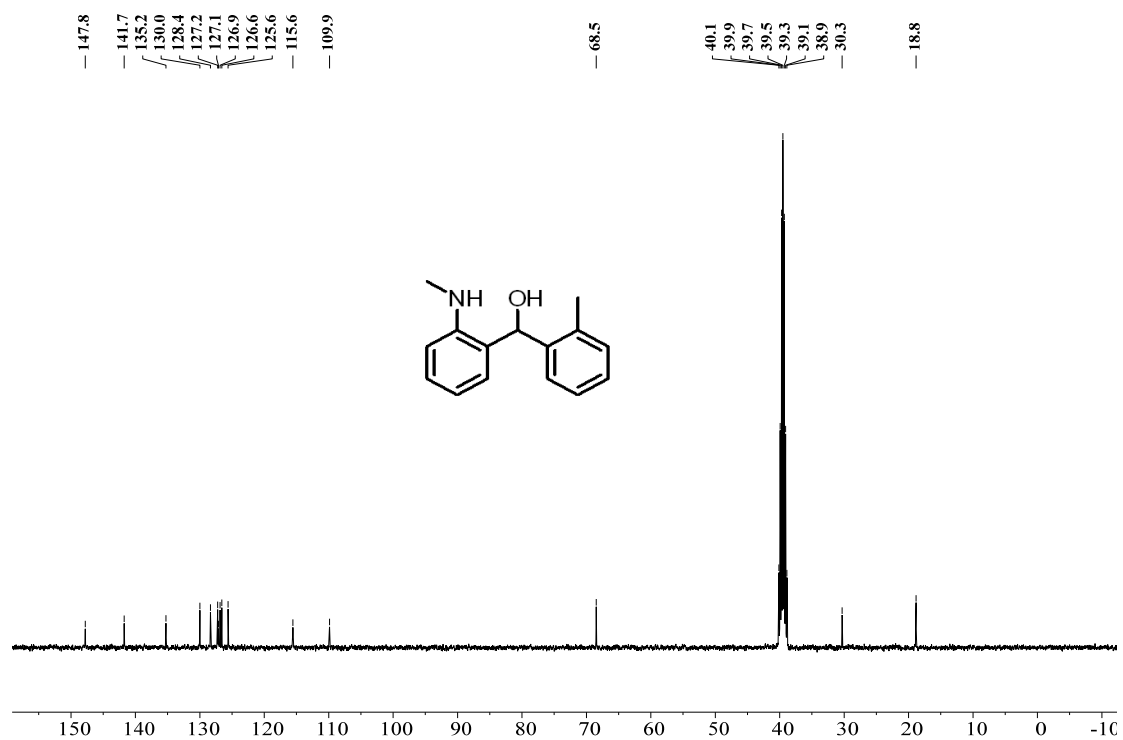


Figure S20. ¹³C{¹H} NMR spectrum of **1j** (DMSO-*d*₆, 101 MHz, 23 °C)

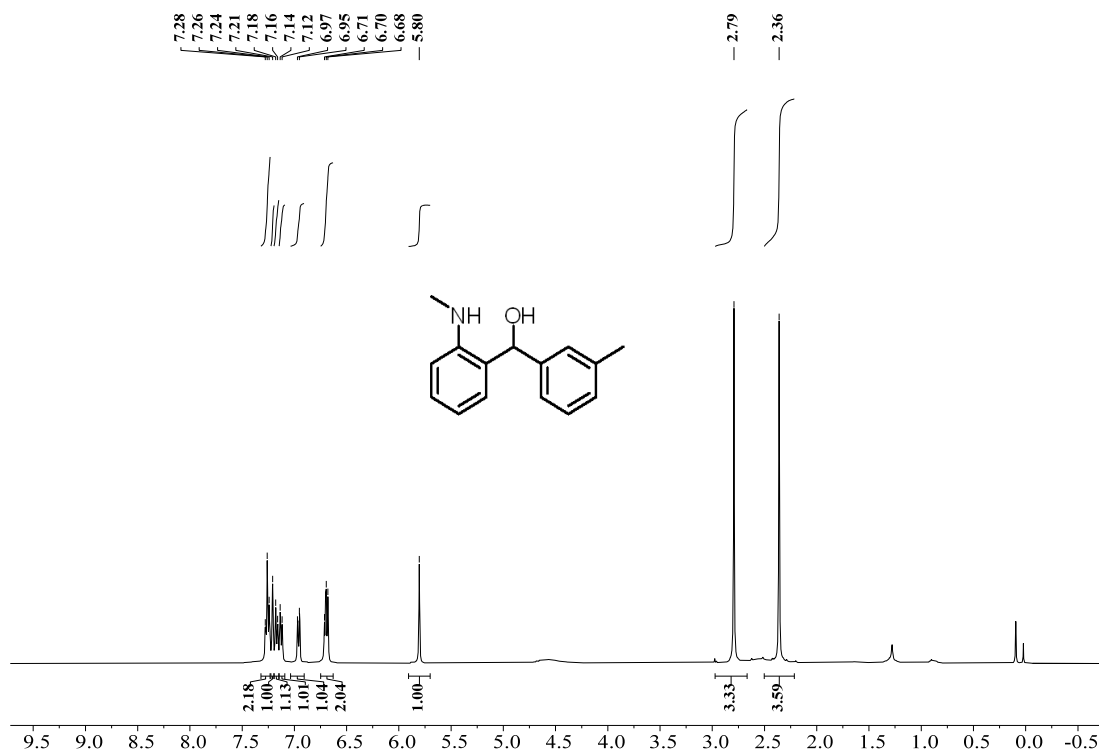


Figure S21. ^1H NMR spectrum of **1k** (CDCl_3 , 400 MHz, 23 °C)

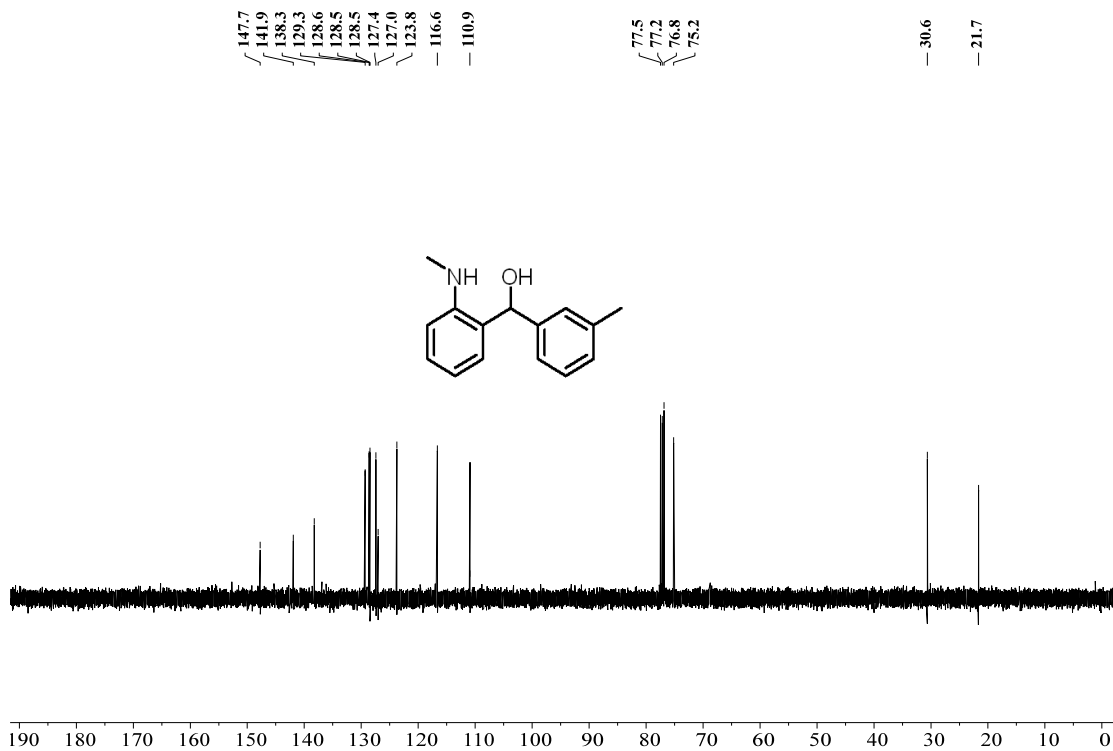


Figure S22. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1k** (CDCl_3 , 101 MHz, 23 °C)

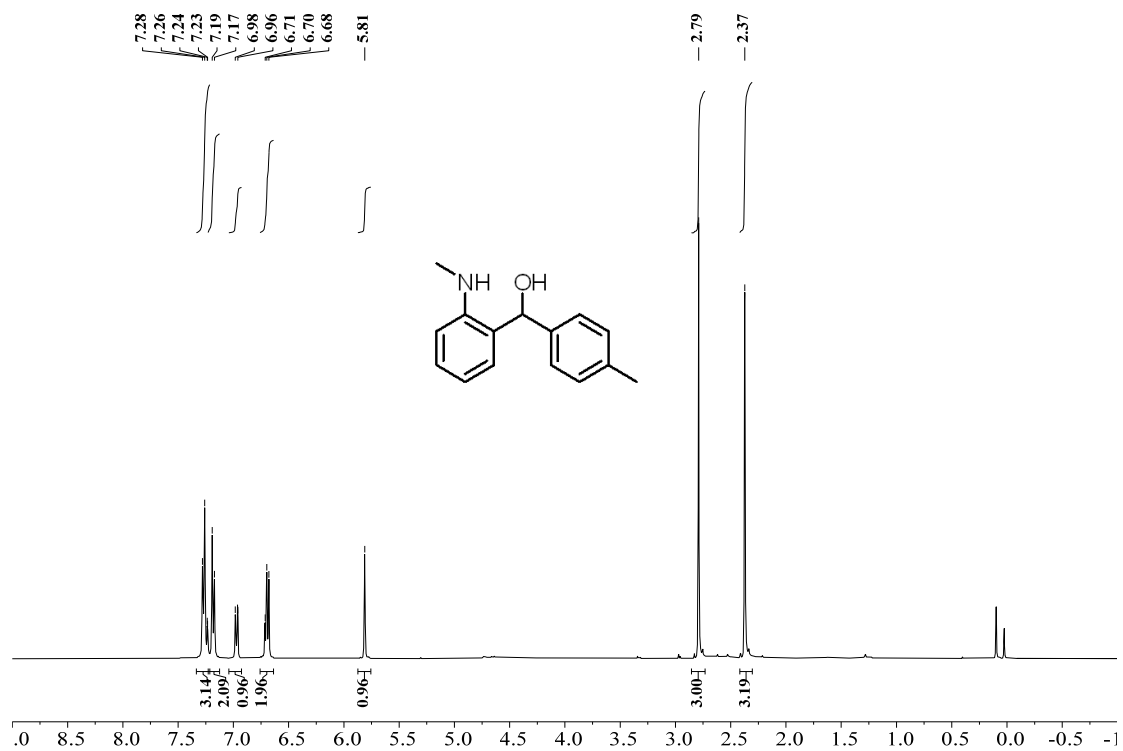


Figure S23. ^1H NMR spectrum of **11** (CDCl_3 , 400 MHz, 23 °C)

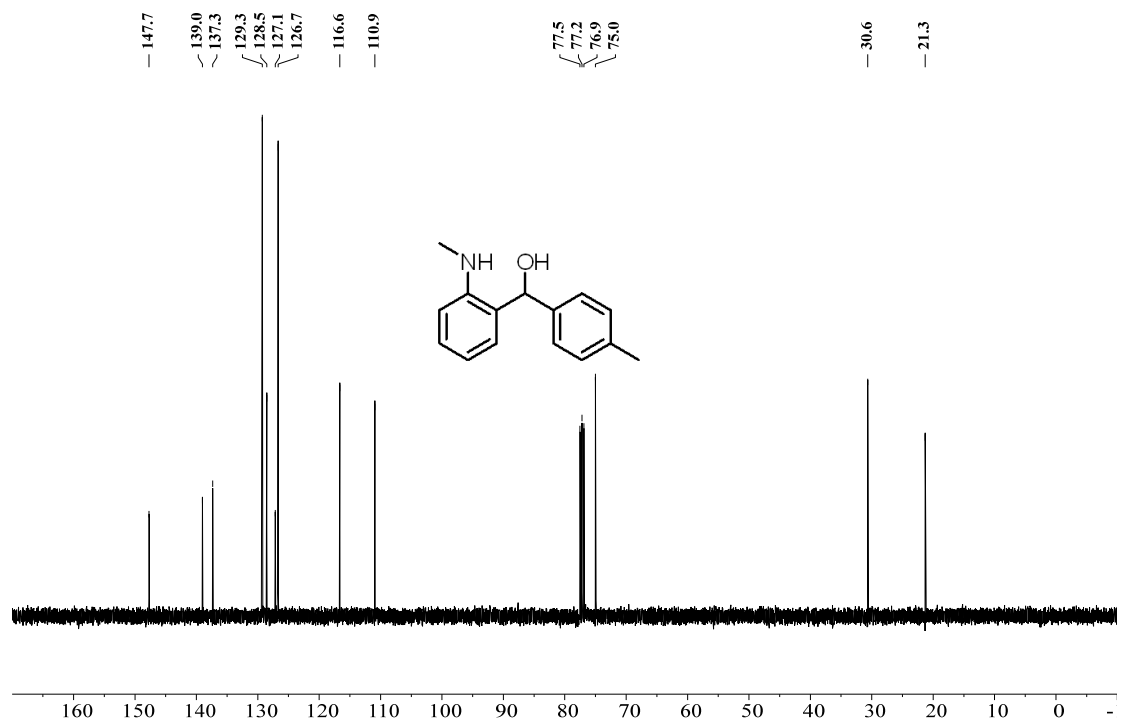


Figure S24. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **11** (CDCl_3 , 101 MHz, 23 °C)

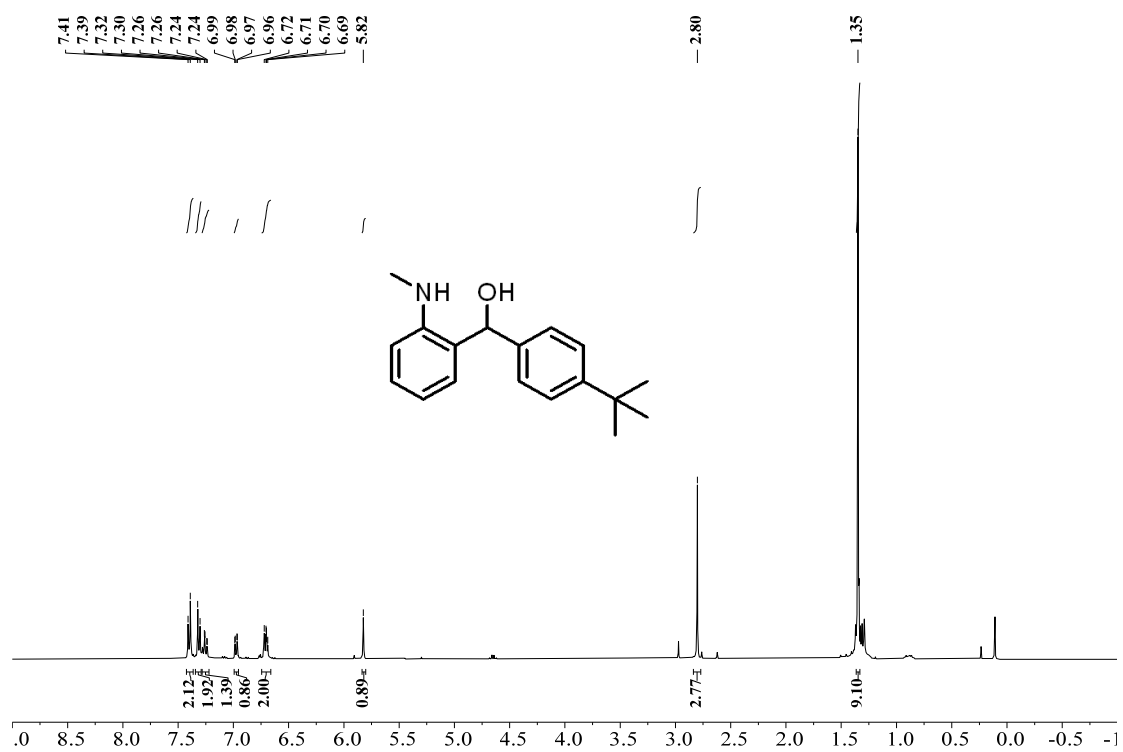


Figure S25. ¹H NMR spectrum of **1m** (CDCl₃, 400 MHz, 23 °C)

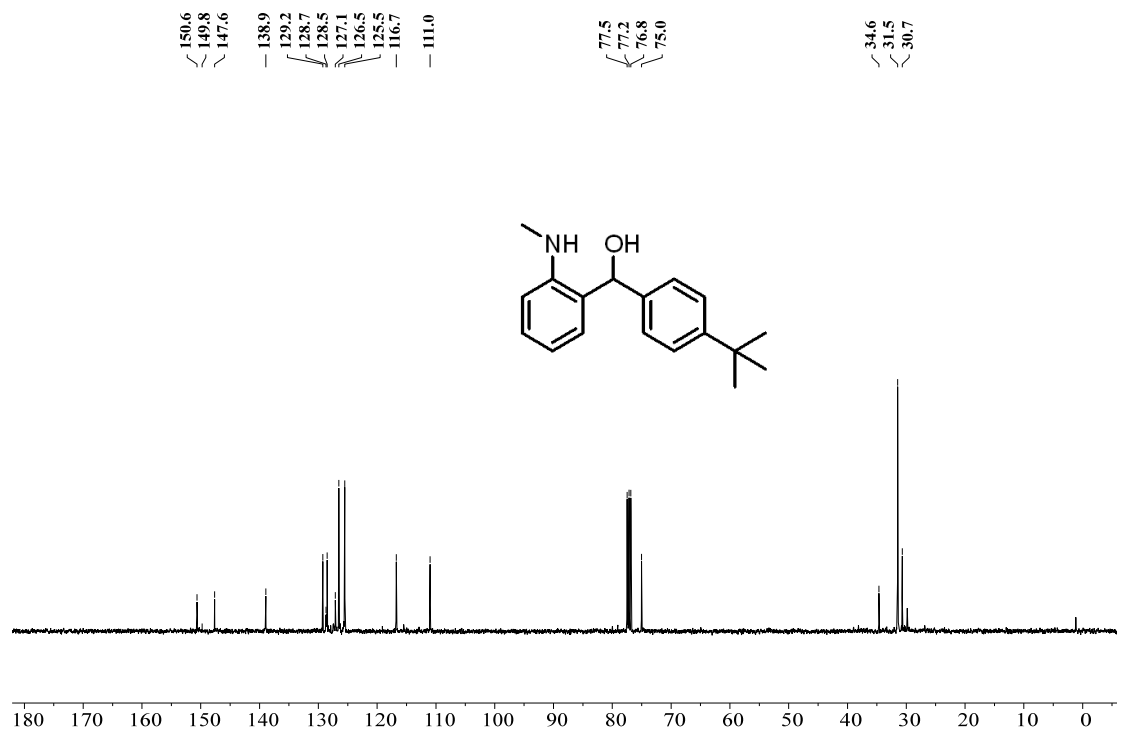


Figure S26. ¹³C{¹H} NMR spectrum of **1m** (CDCl₃, 101 MHz, 23 °C)



Figure S27. ¹H NMR spectrum of **1n** (CDCl₃, 400 MHz, 23 °C)

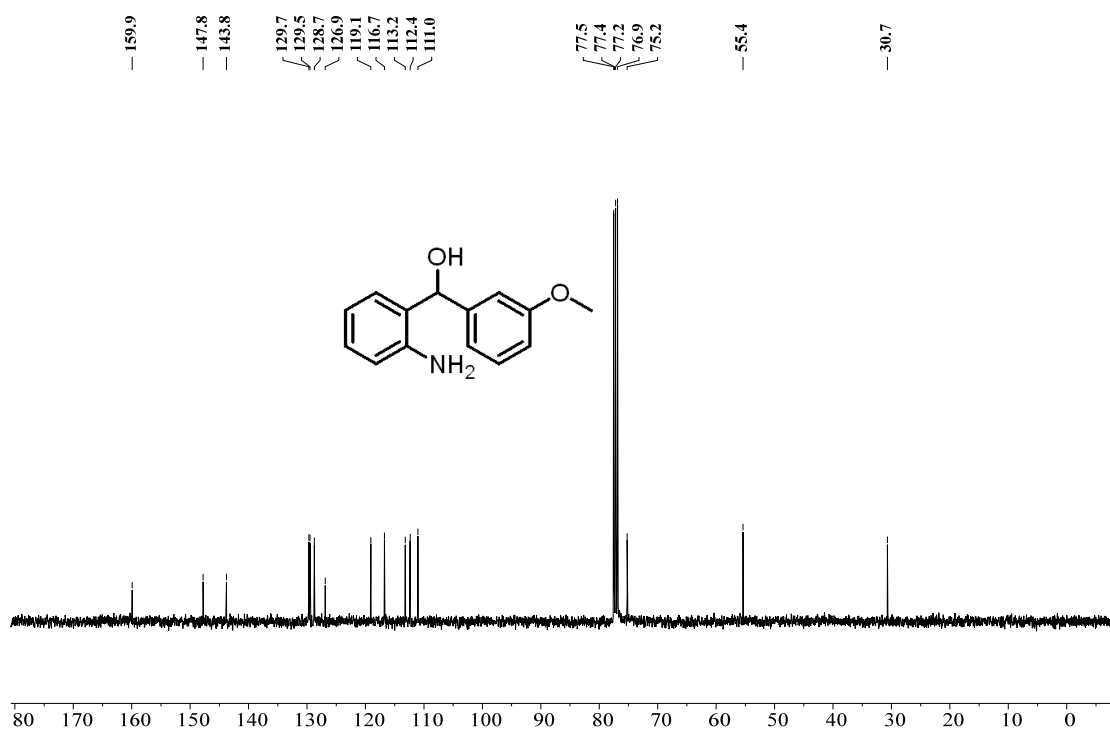


Figure S28. ¹³C{¹H} NMR spectrum of **1n** (CDCl₃, 101 MHz, 23 °C)

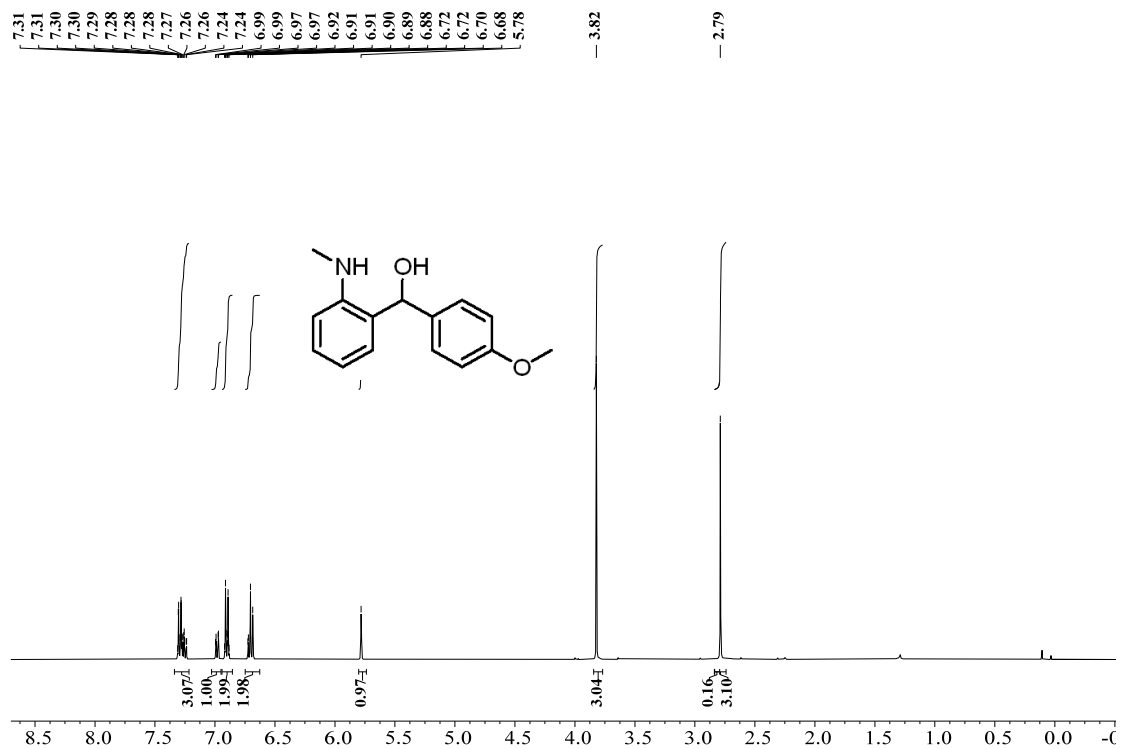


Figure S29. ^1H NMR spectrum of **1o** (CDCl_3 , 400 MHz, 23 °C)

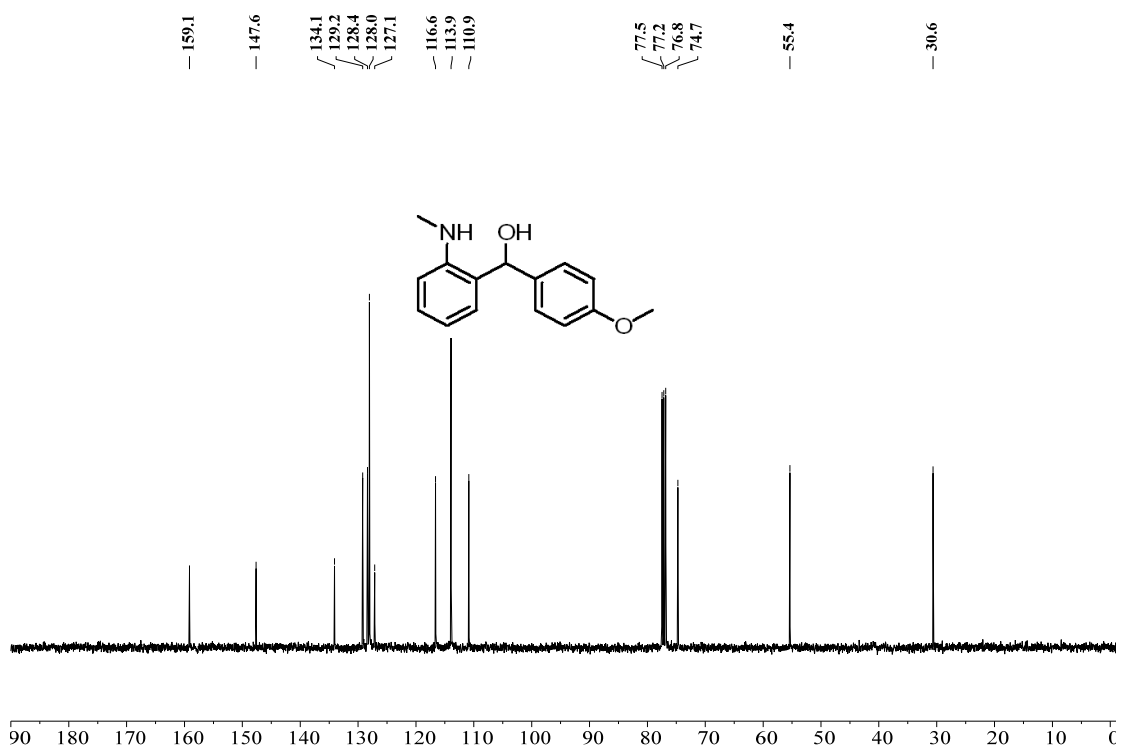


Figure S30. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1o** (CDCl_3 , 101 MHz, 23 °C)

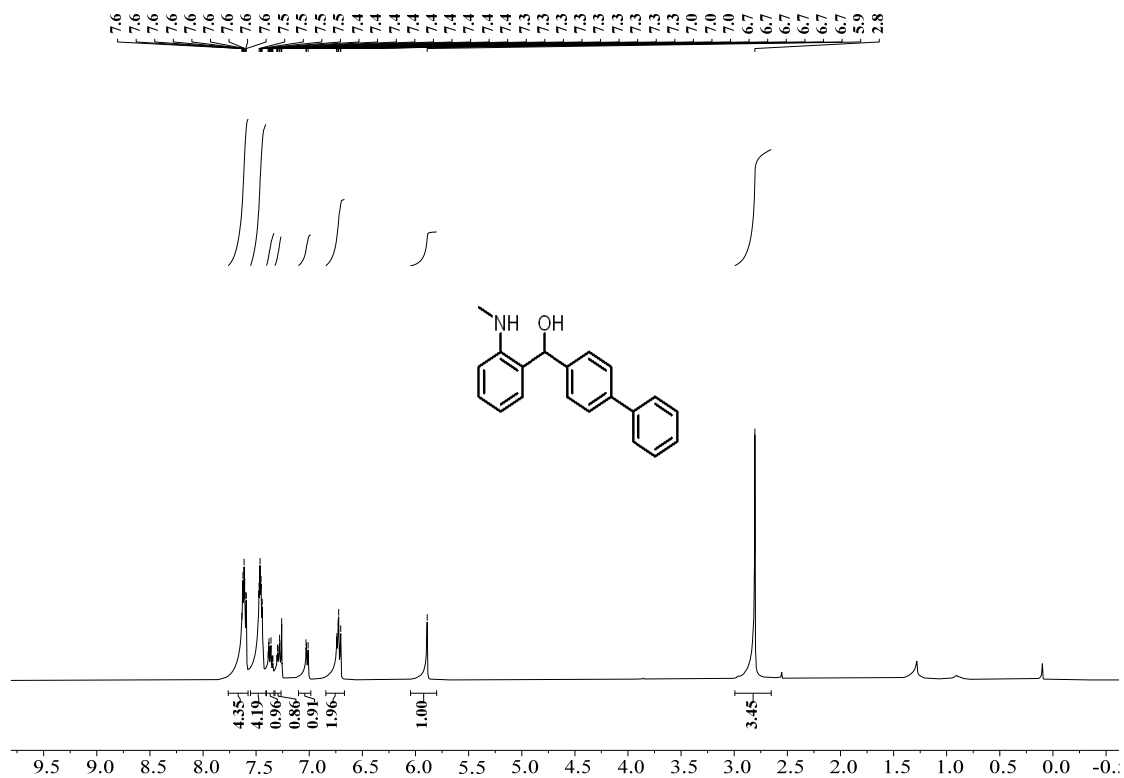


Figure S31. ^1H NMR spectrum of **1p** (CDCl_3 , 400 MHz, 23 °C)

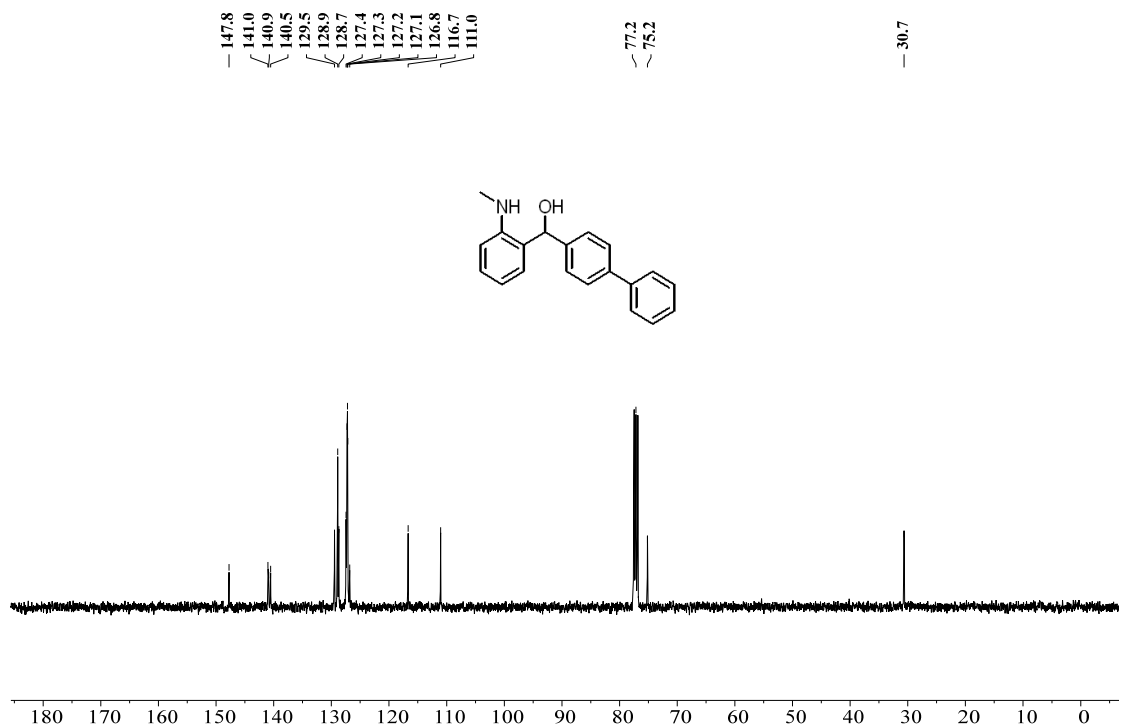


Figure S32. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1p** (CDCl_3 , 101 MHz, 23 °C)

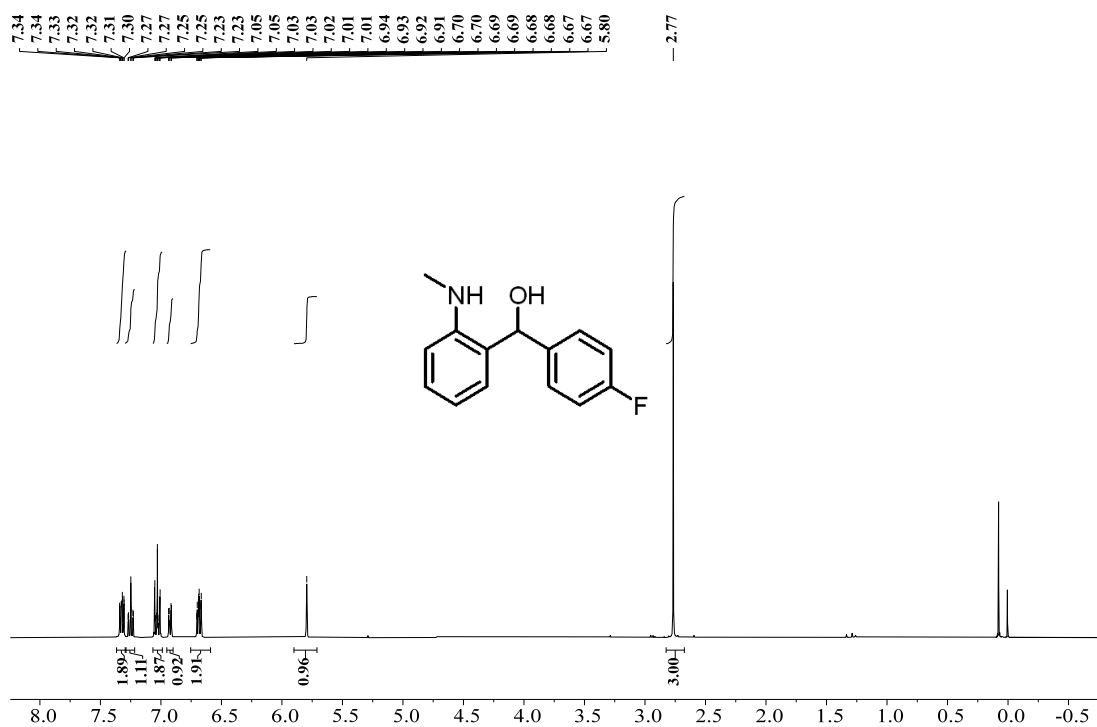


Figure S33. ¹H NMR spectrum of **1q** (CDCl₃, 400 MHz, 23 °C)

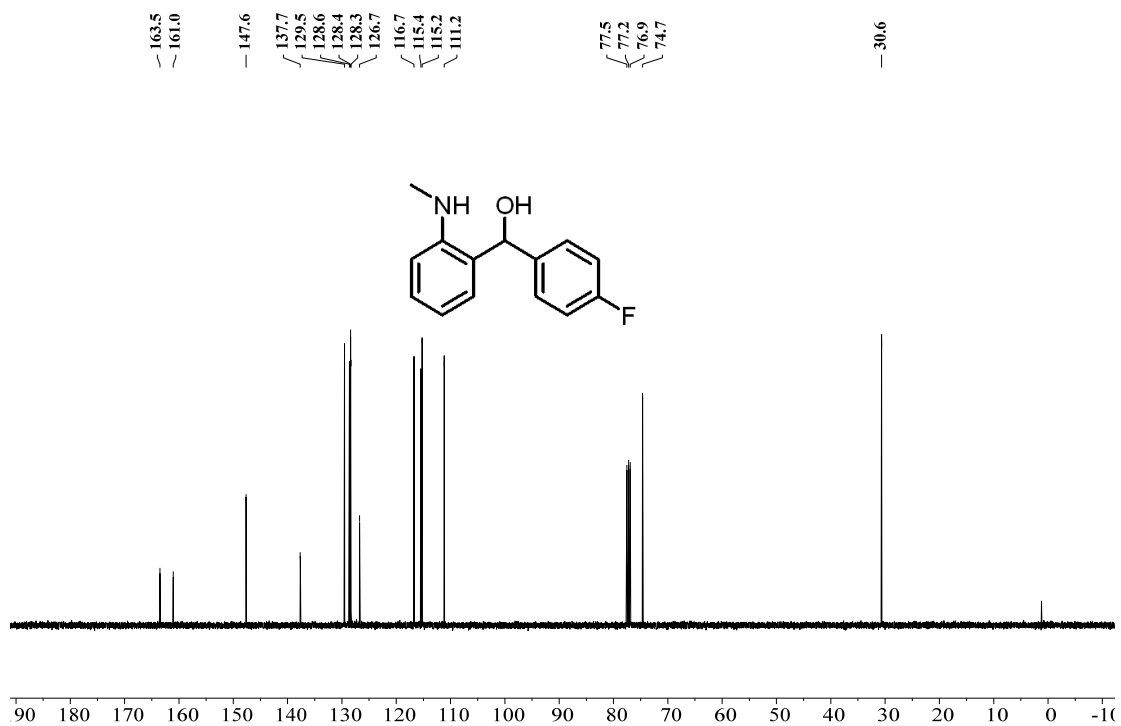


Figure S34. ¹³C{¹H} NMR spectrum of **1q** (CDCl₃, 101 MHz, 23 °C)

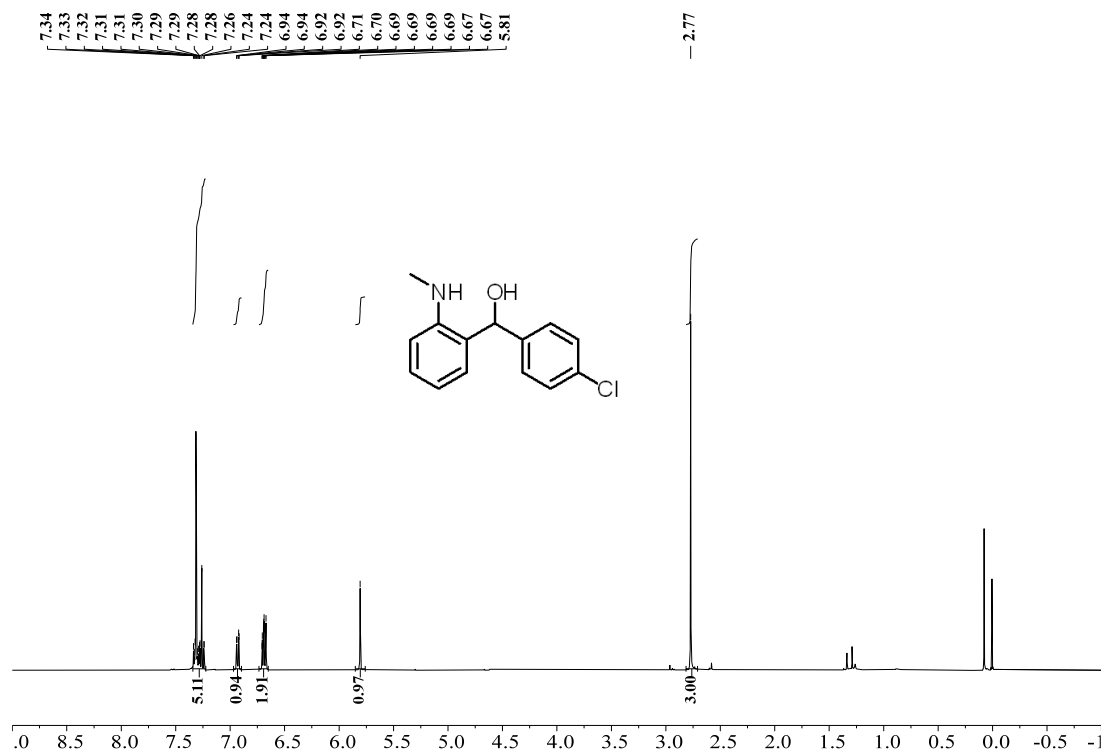


Figure S35. ^1H NMR spectrum of **1r** (CDCl_3 , 400 MHz, 23 °C)

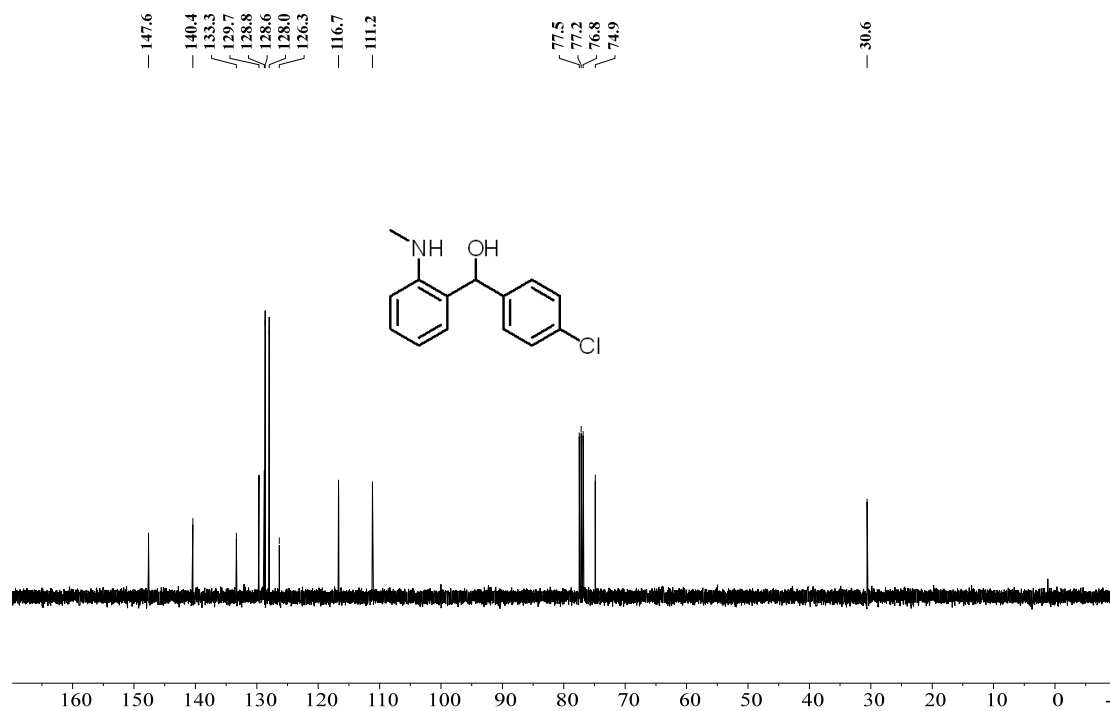


Figure S36. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1r** (CDCl_3 , 101 MHz, 23 °C)

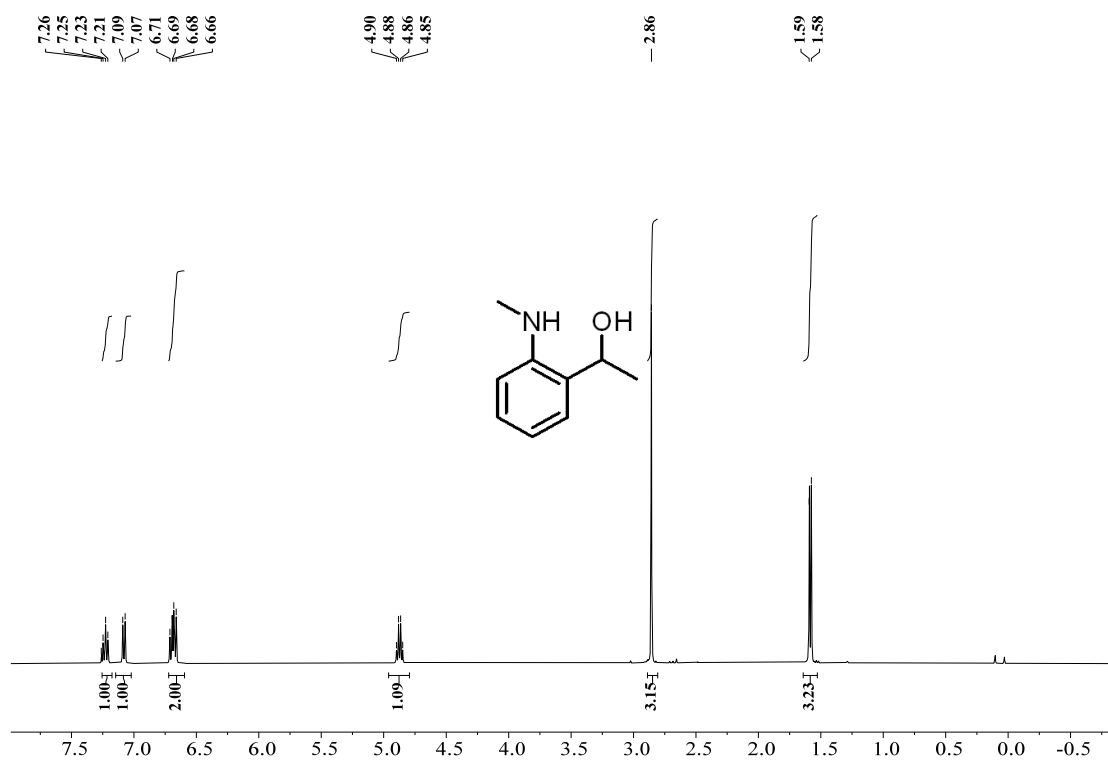


Figure S37. ^1H NMR spectrum of **1s** (CDCl_3 , 400 MHz, 23 °C)

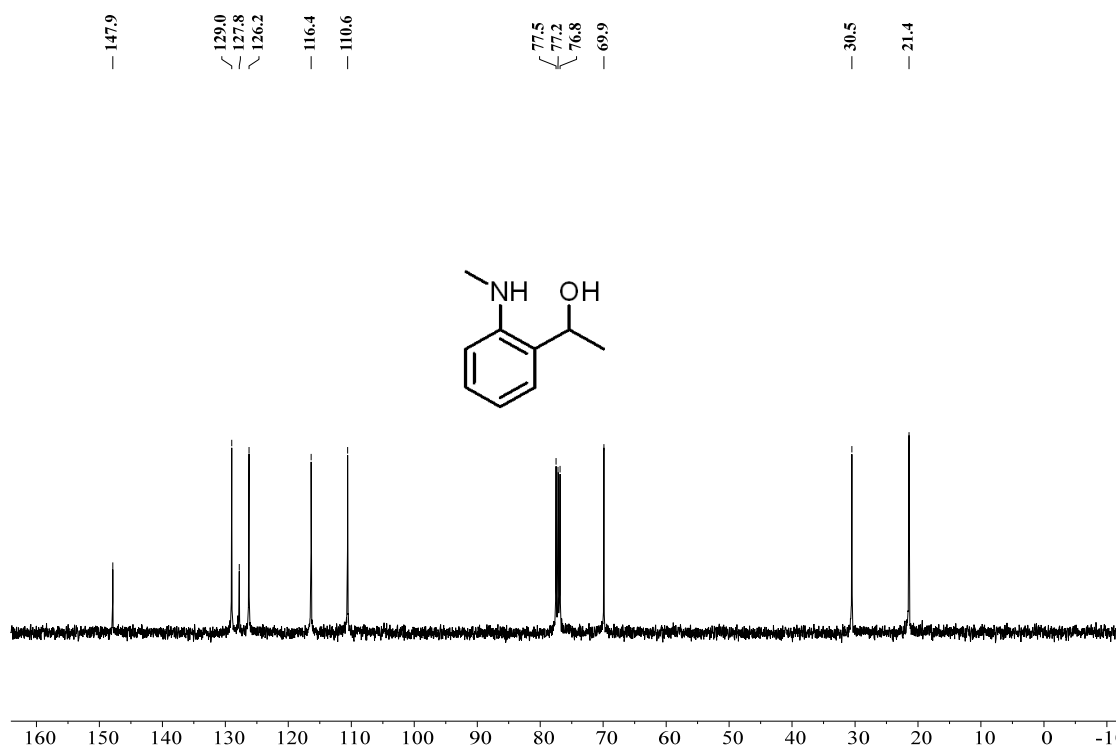


Figure S38. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1s** (CDCl_3 , 101 MHz, 23 °C)

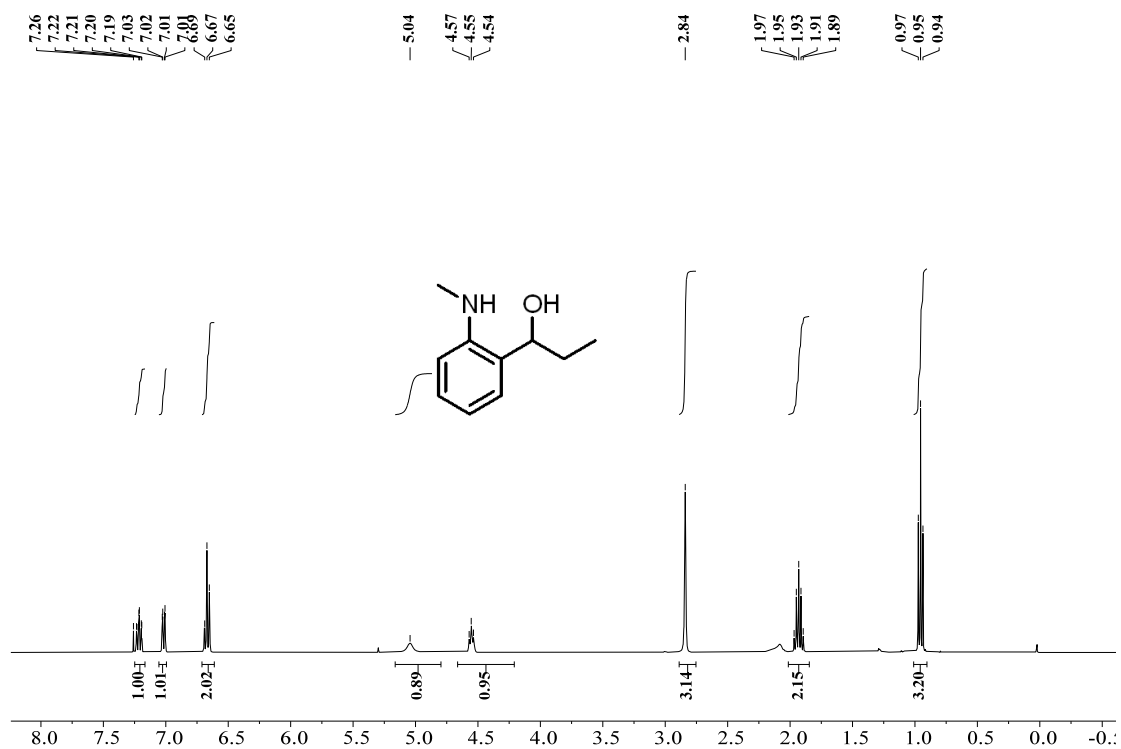


Figure S39. ^1H NMR spectrum of **1t** (CDCl_3 , 400 MHz, 23 °C)

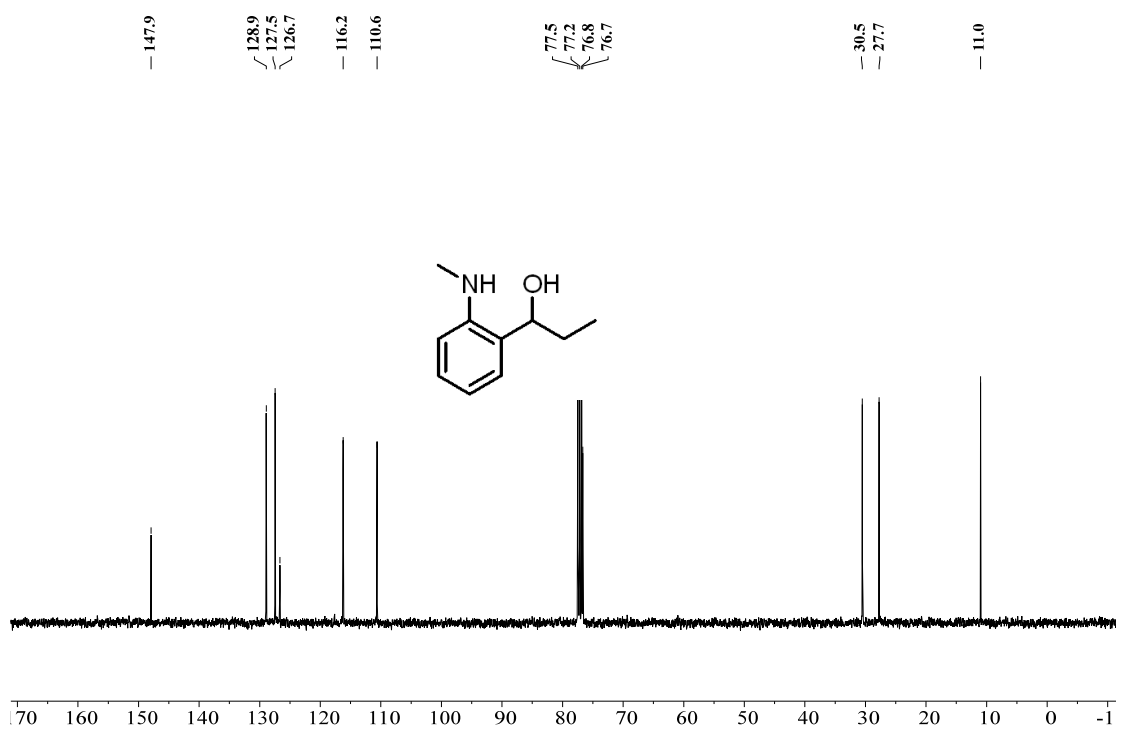


Figure S40. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1t** (CDCl_3 , 101 MHz, 23 °C)

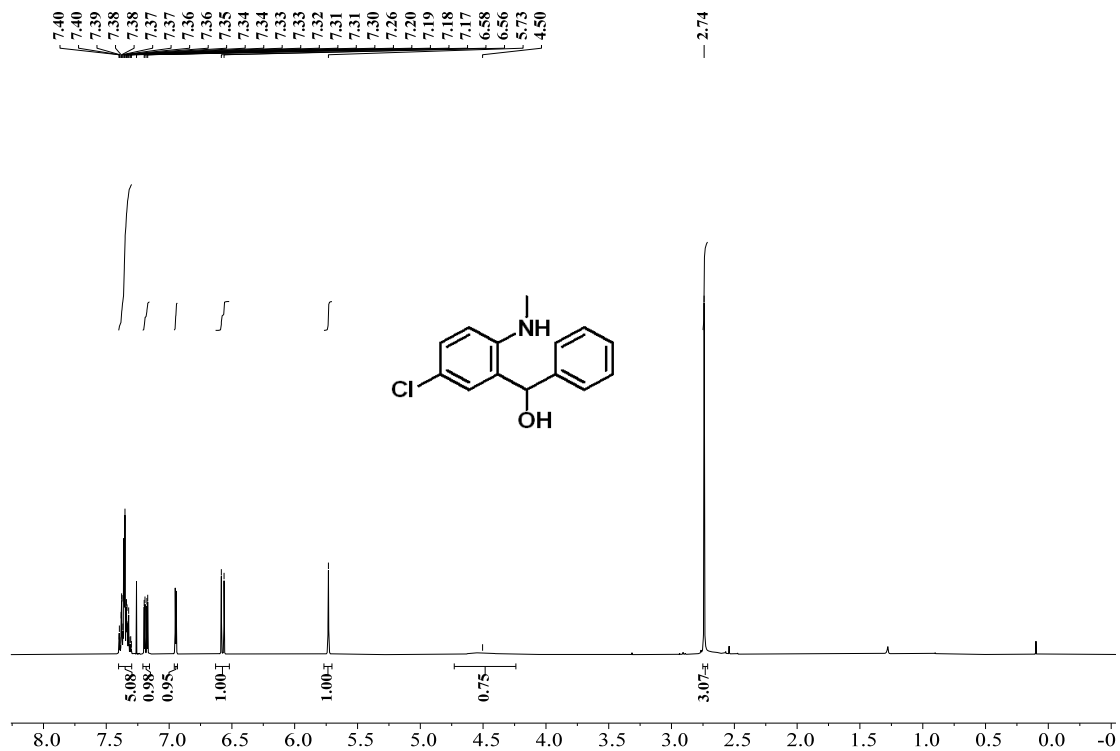


Figure S41. ^1H NMR spectrum of **1u** (CDCl_3 , 400 MHz, 23 °C)

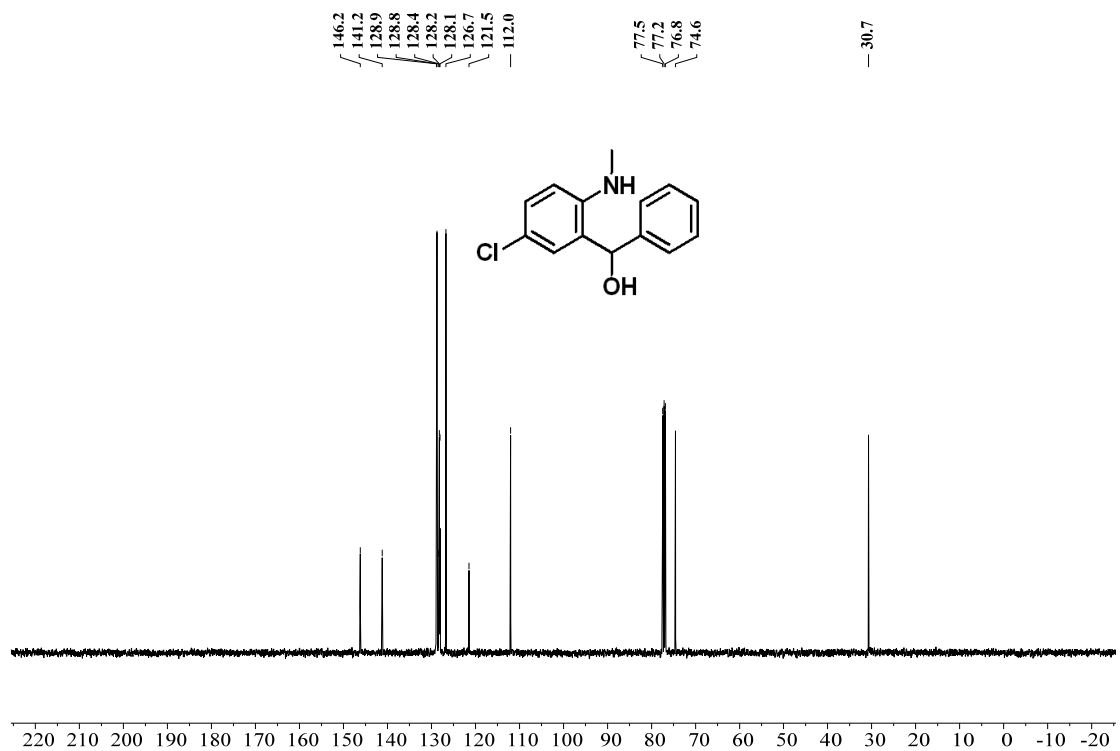


Figure S42. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1u** (CDCl_3 , 101 MHz, 23 °C)

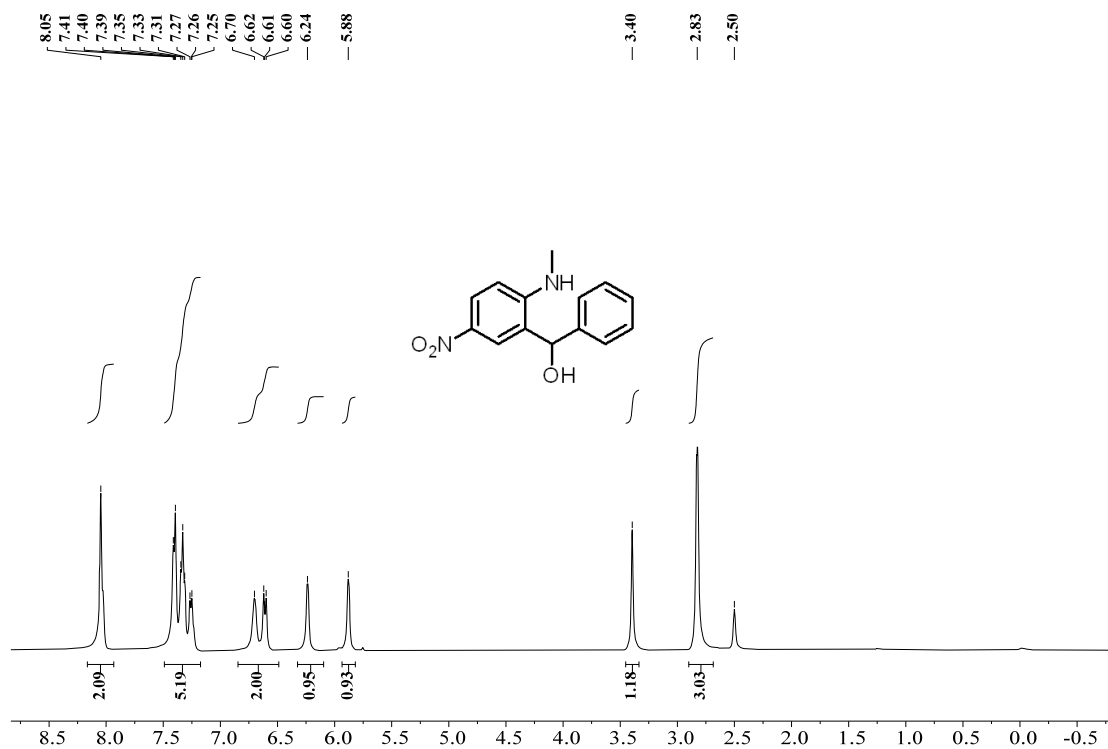


Figure S43. ^1H NMR spectrum of **1v** (DMSO- d_6 , 400 MHz, 23 °C)

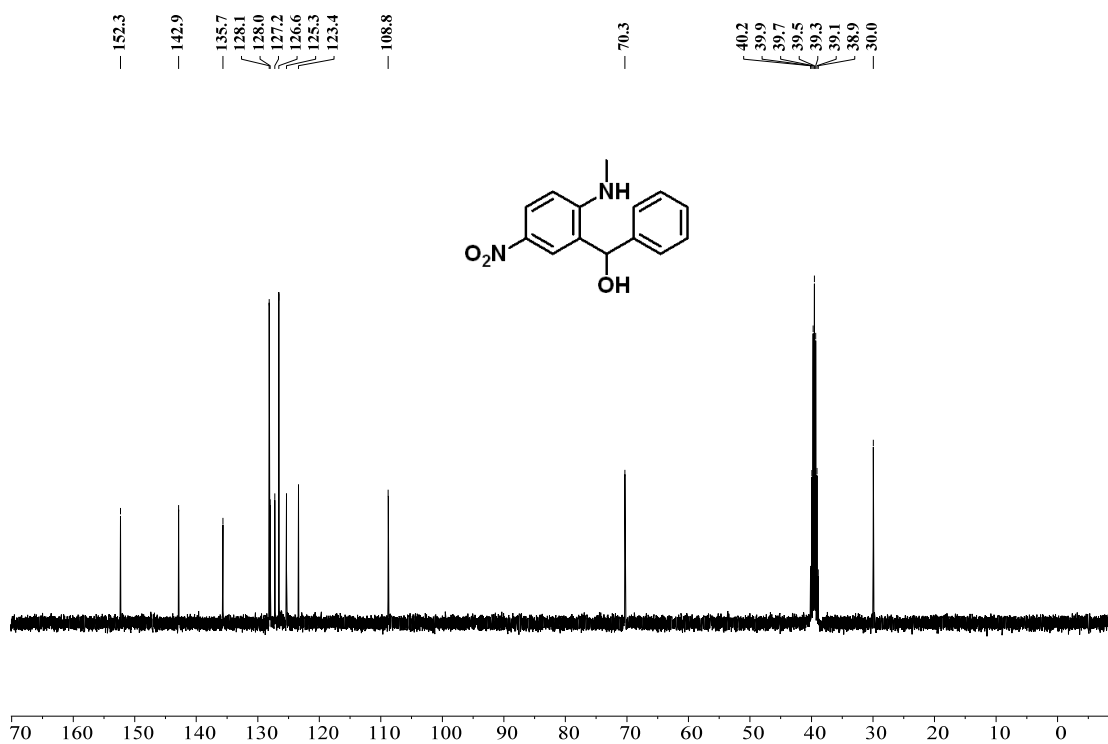


Figure S44. ^{13}C $\{^1\text{H}\}$ NMR spectrum of **1v** (DMSO- d_6 , 101 MHz, 23 °C)



Figure S45. ¹H NMR spectrum of **1w** (CDCl₃, 400 MHz, 23 °C)

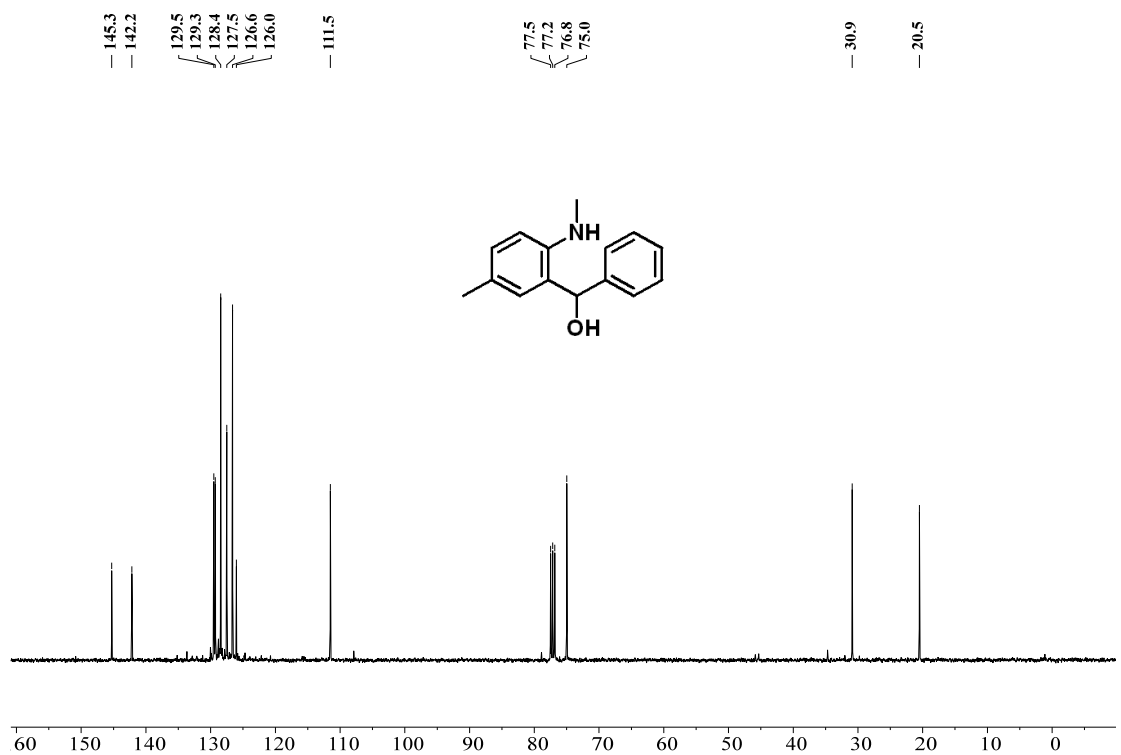


Figure S46. ¹³C {¹H} NMR spectrum of **1w** (CDCl₃, 101 MHz, 23 °C)

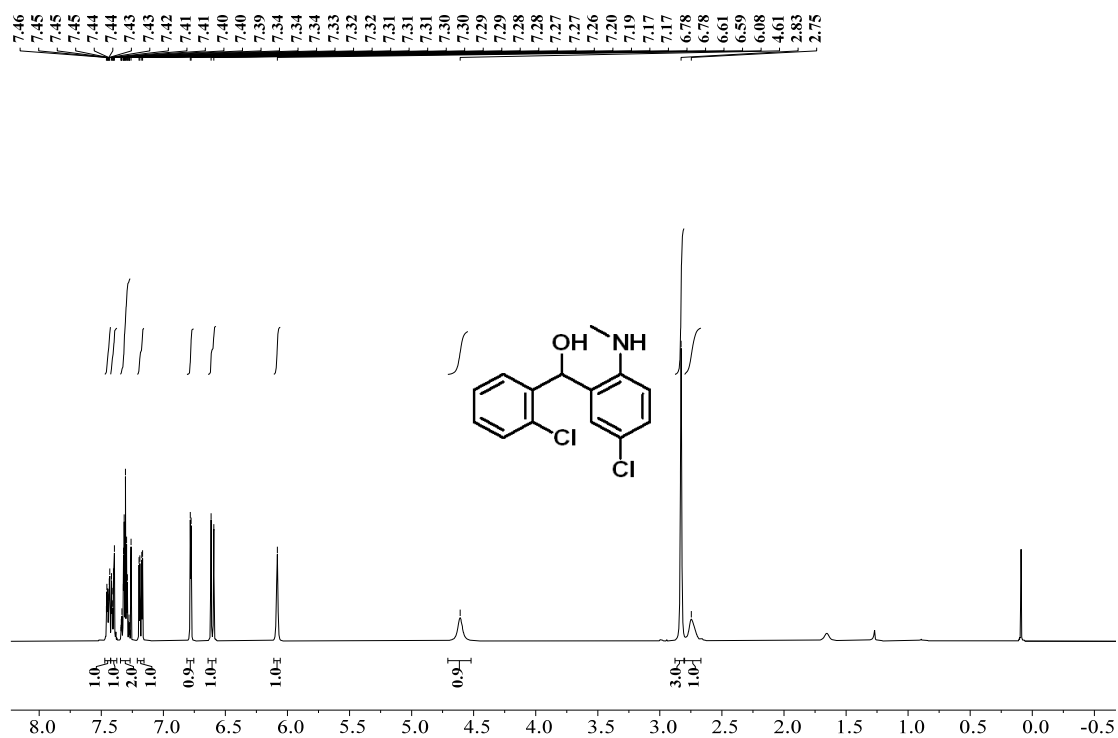


Figure S47. ¹H NMR spectrum of **1x** (CDCl₃, 400 MHz, 23 °C)

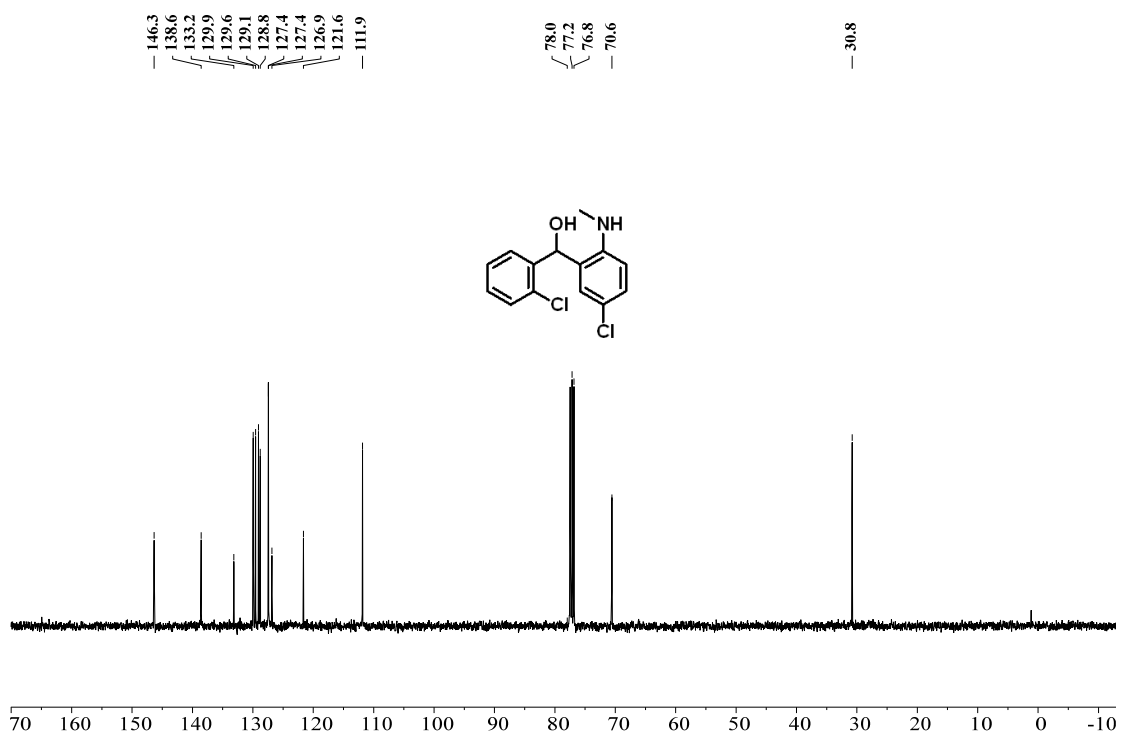


Figure S48. ¹³C{¹H} NMR spectrum of **1x** (CDCl₃, 101 MHz, 23 °C)

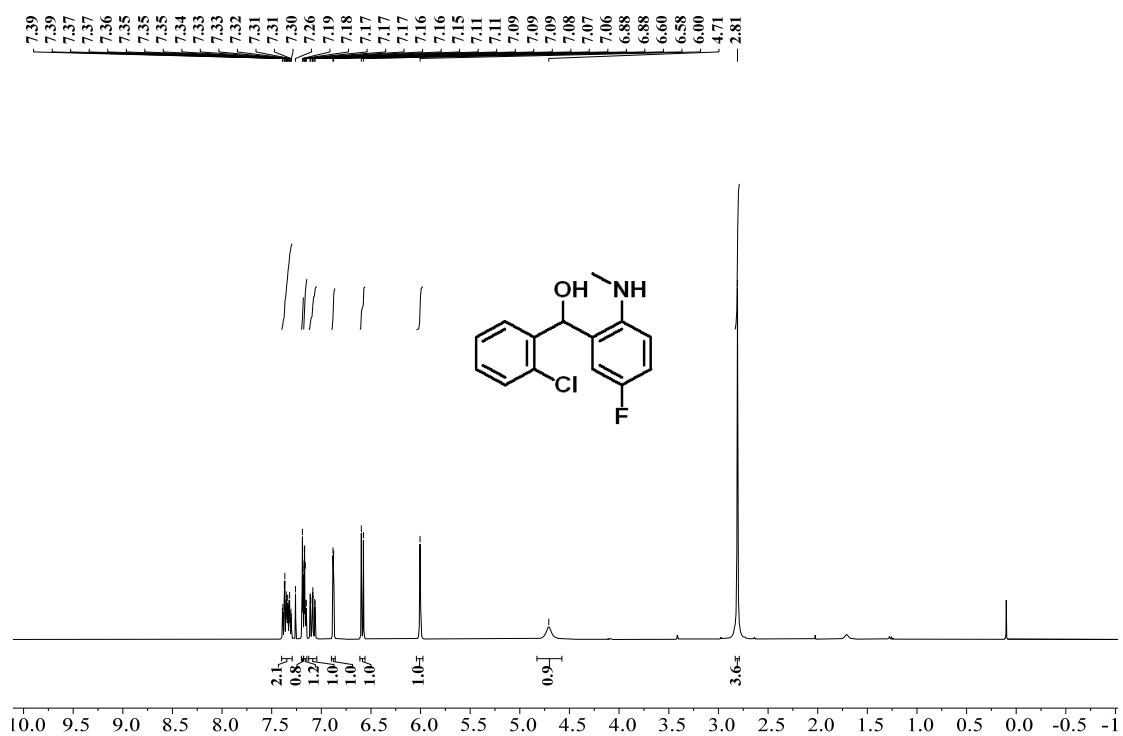


Figure S49. ^1H NMR spectrum of **1y** (CDCl₃, 400 MHz, 23 °C)

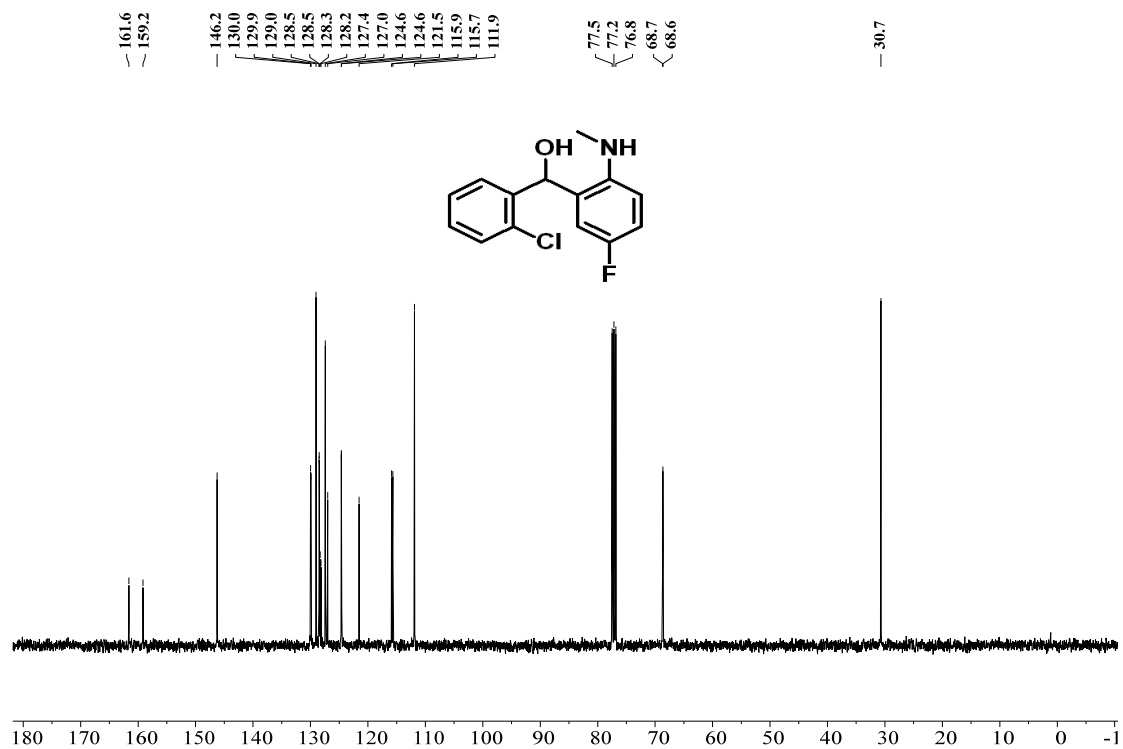


Figure S50. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1y** (CDCl₃, 101 MHz, 23 °C)

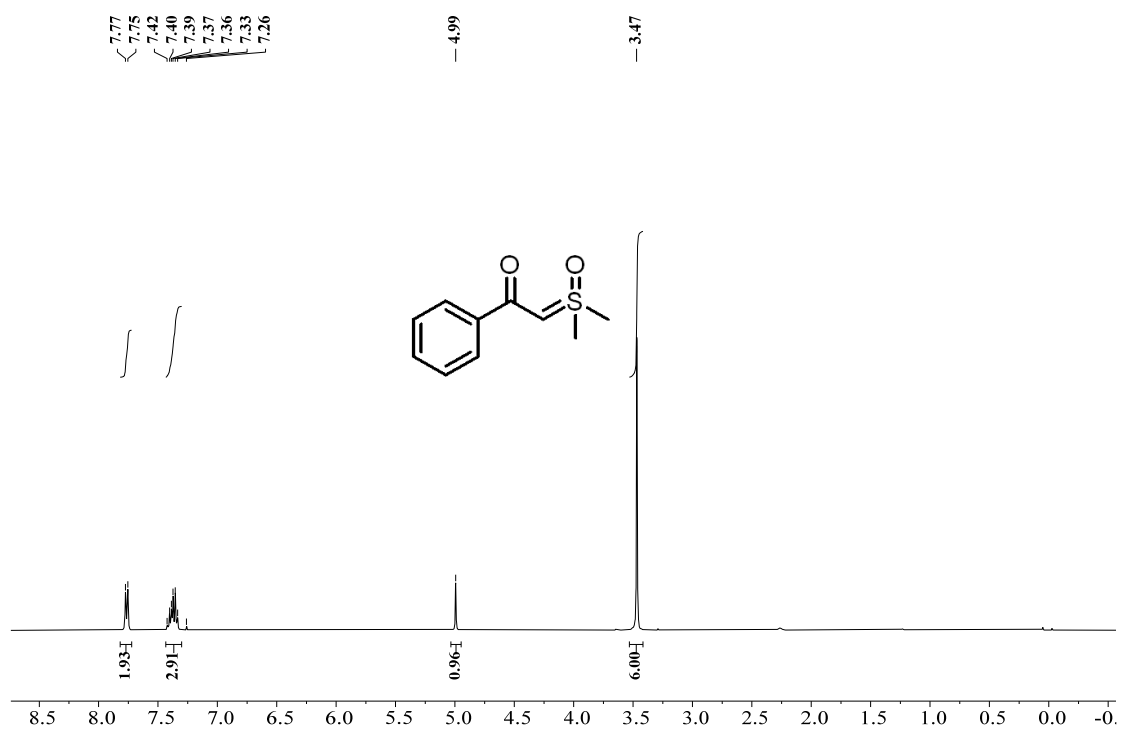


Figure S51. ^1H NMR spectrum of **2a** (CDCl_3 , 400 MHz, 23 °C)

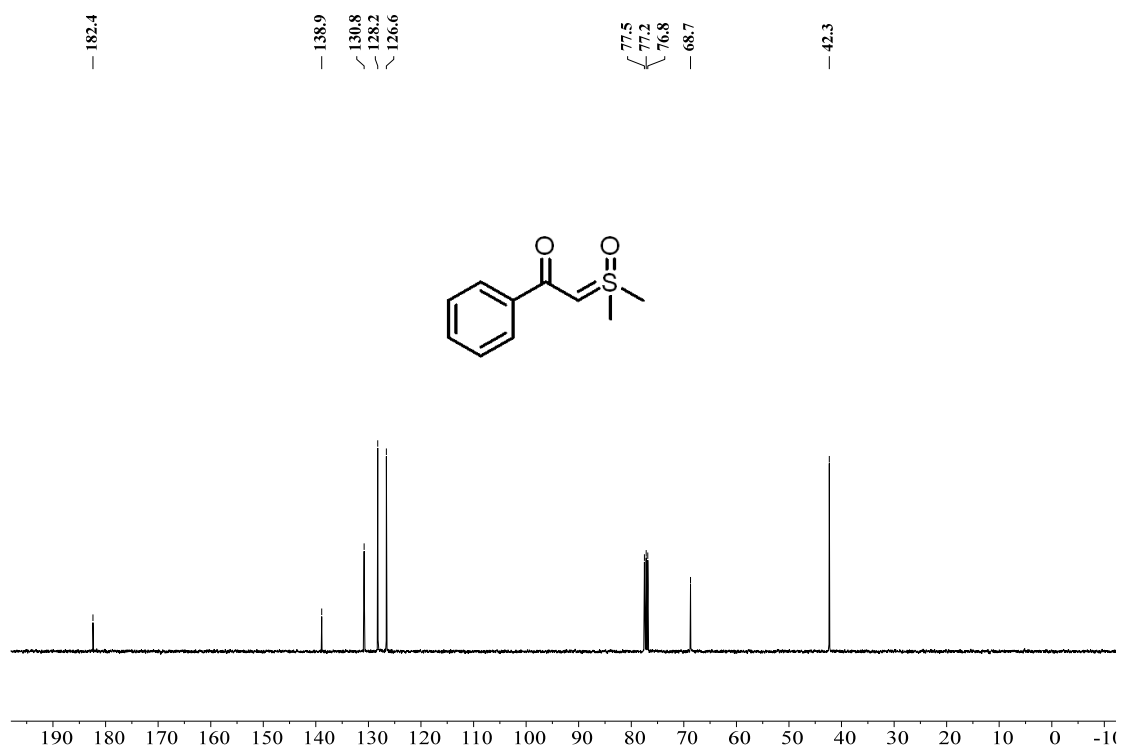


Figure S52. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2a** (CDCl_3 , 101 MHz, 23 °C)

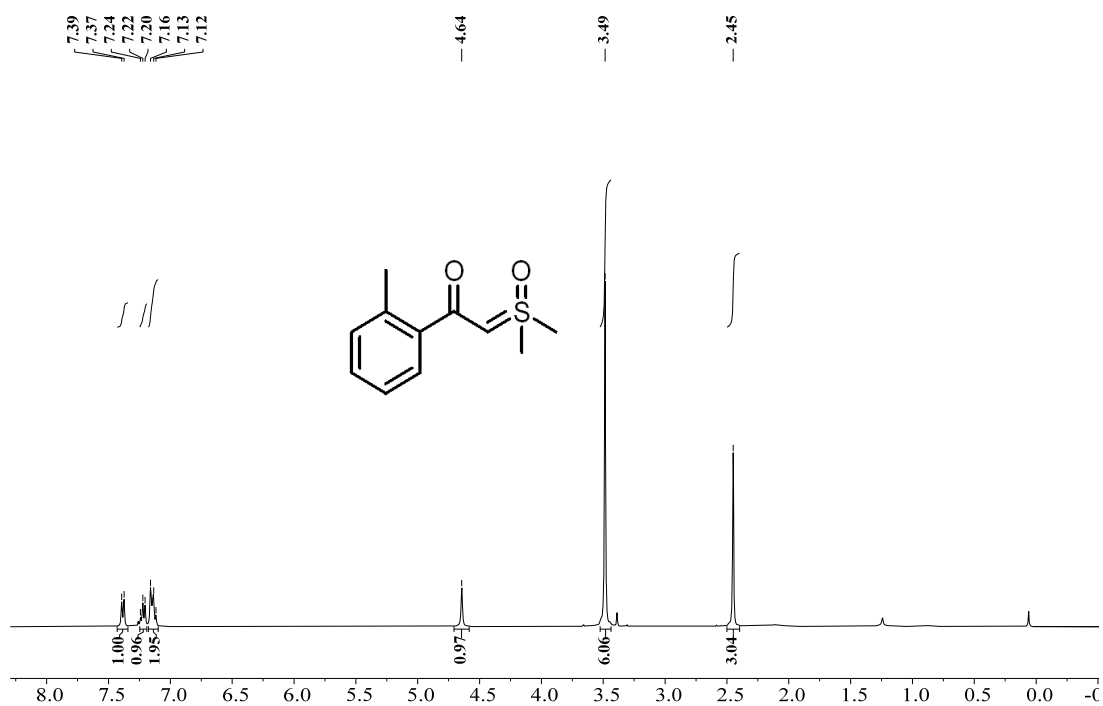


Figure S53. ^1H NMR spectrum of **2b** (CDCl_3 , 400 MHz, 23 °C)



Figure S54. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2b** (CDCl_3 , 101 MHz, 23 °C)

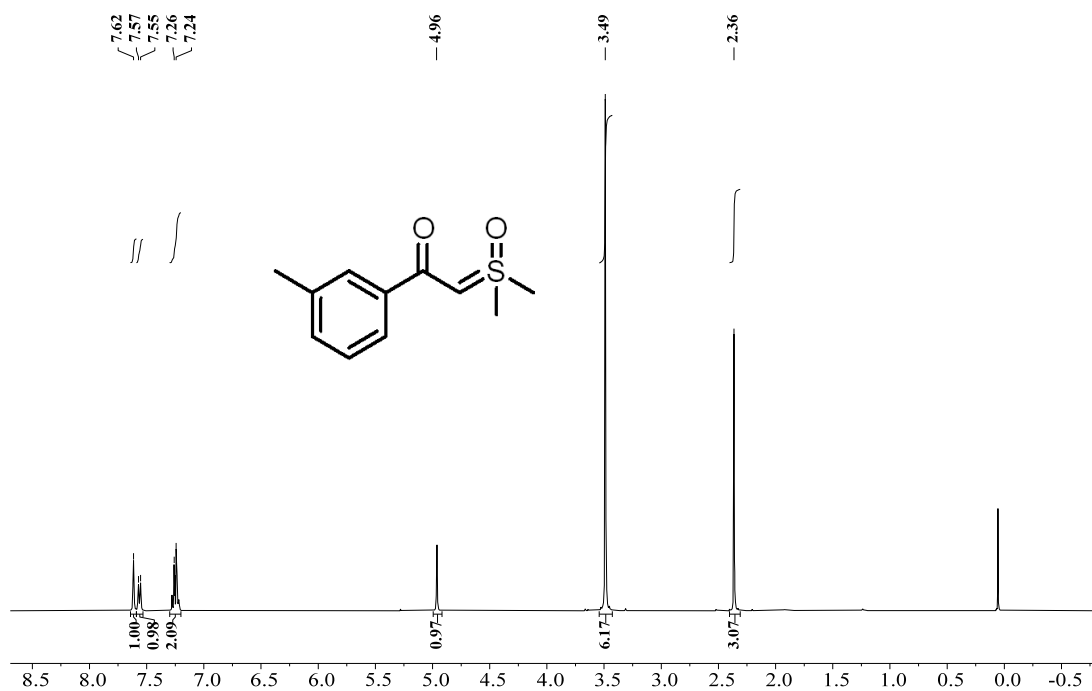


Figure S55. ^1H NMR spectrum of **2c** (CDCl_3 , 400 MHz, 23 °C)

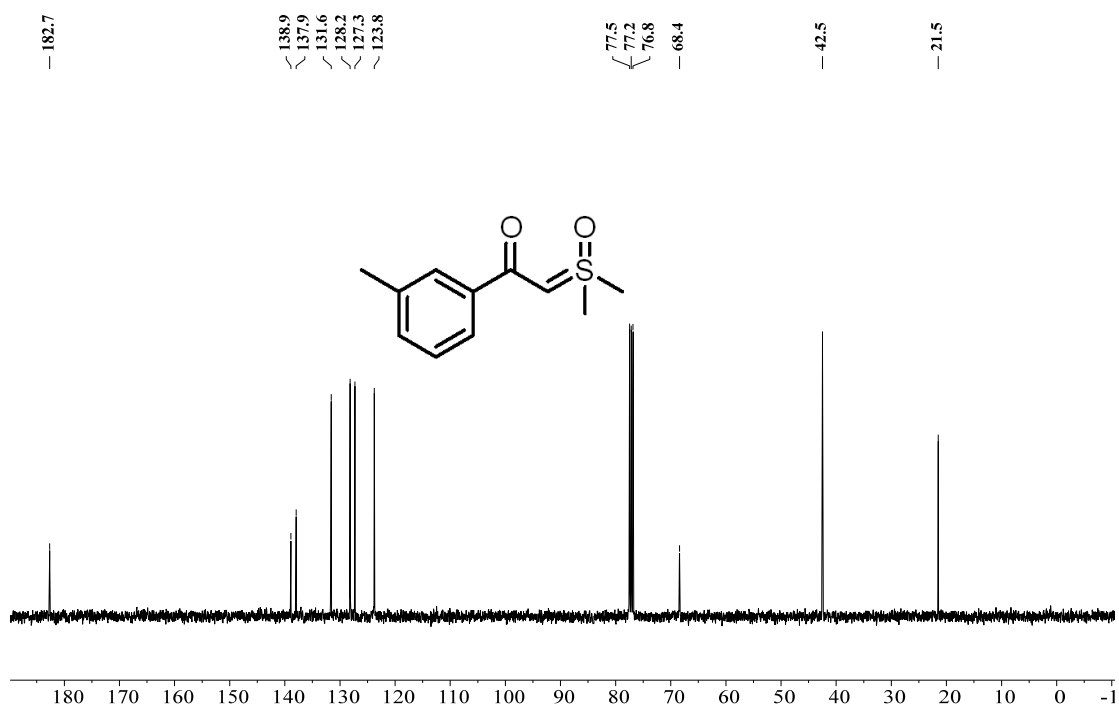


Figure S56. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2c** (CDCl_3 , 101 MHz, 23 °C)

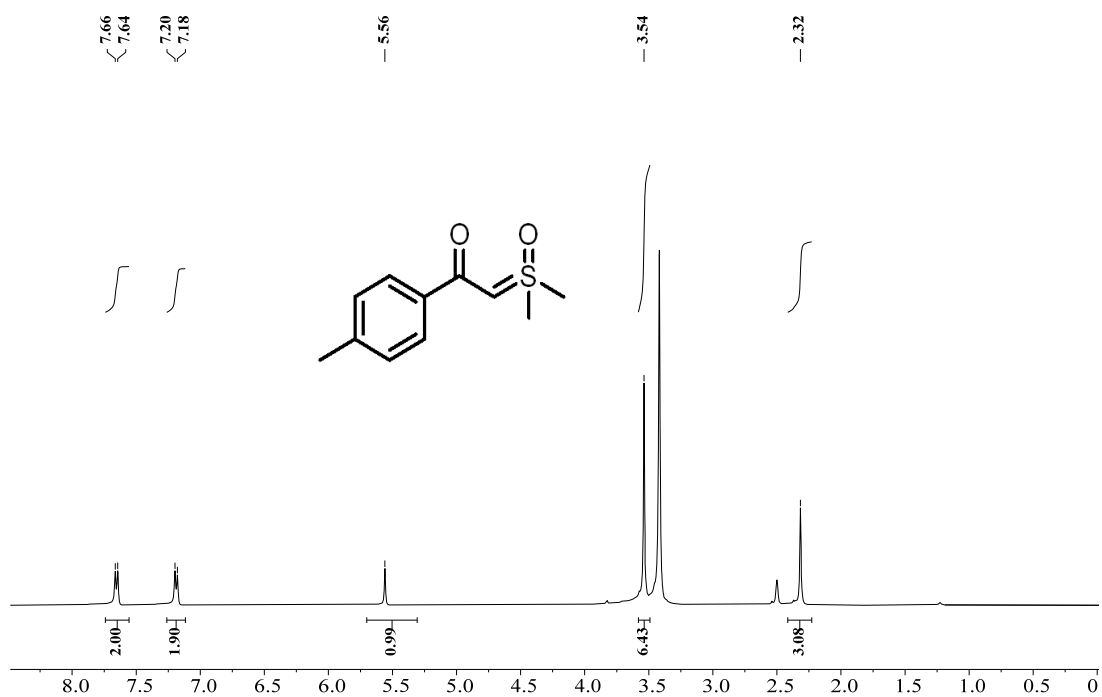


Figure S57. ^1H NMR spectrum of **2d** (DMSO- d_6 , 400 MHz, 23 °C)

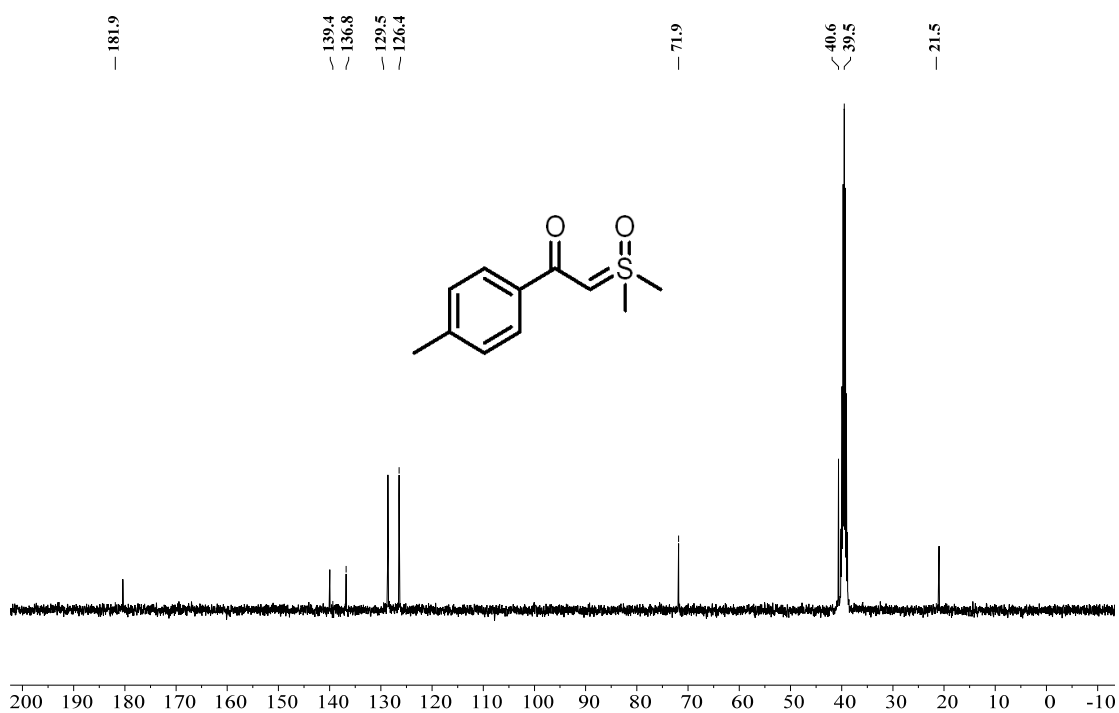


Figure S58. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2d** (DMSO- d_6 , 101 MHz, 23 °C)

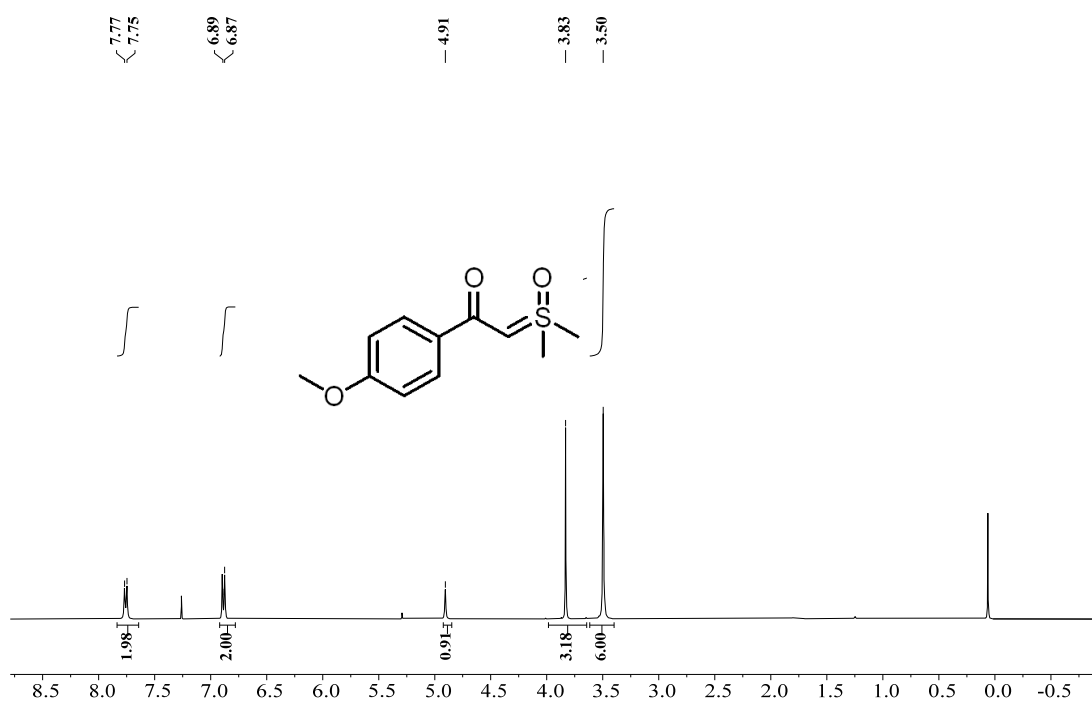


Figure S59. ^1H NMR spectrum of **2e** (CDCl_3 , 400 MHz, 23 °C)



Figure S60. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2e** (CDCl_3 , 101 MHz, 23 °C)

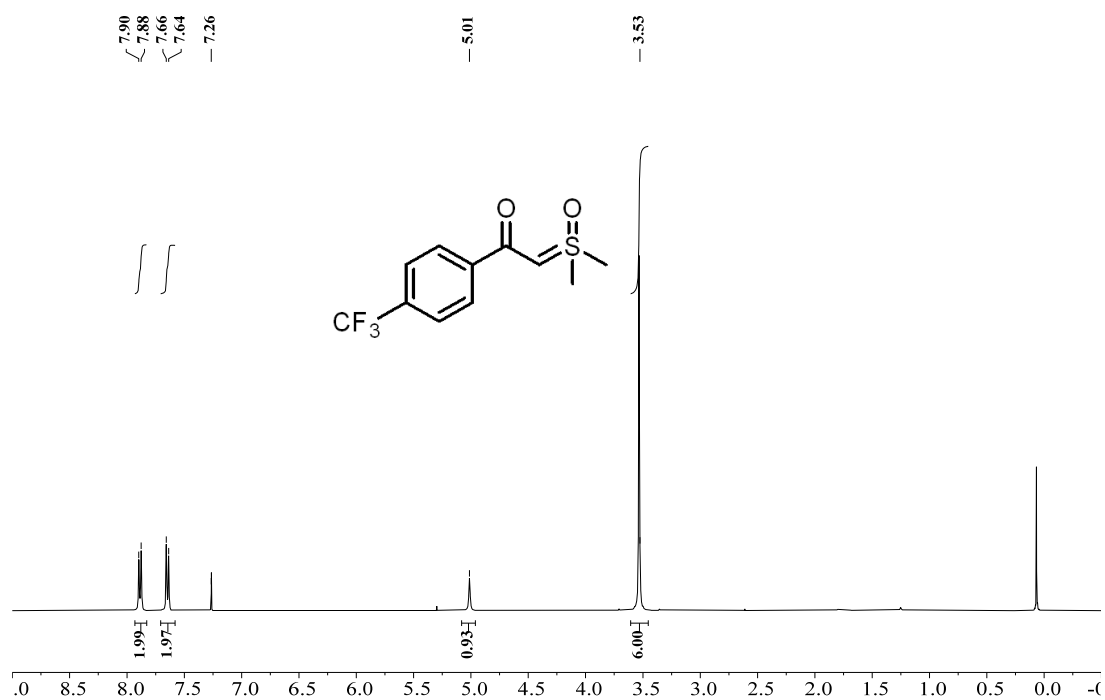


Figure S61. ¹H NMR spectrum of **2f** (CDCl₃, 400 MHz, 23 °C)

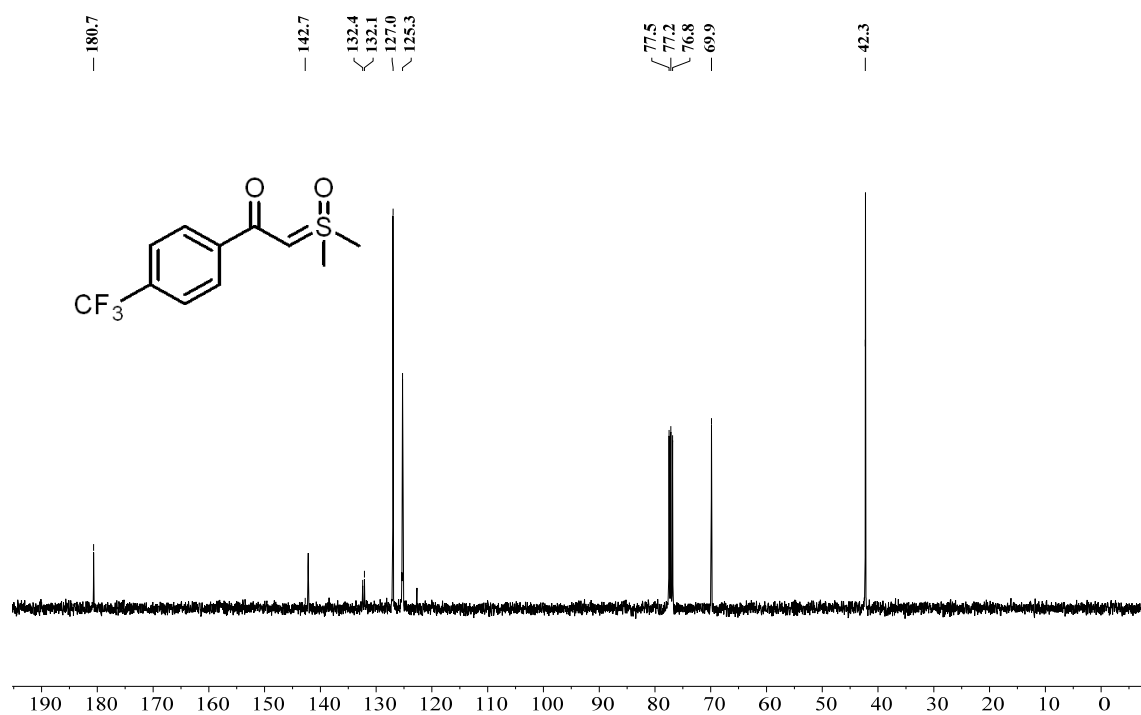


Figure S62. ¹³C{¹H} NMR spectrum of **2f** (CDCl₃, 101 MHz, 23 °C)

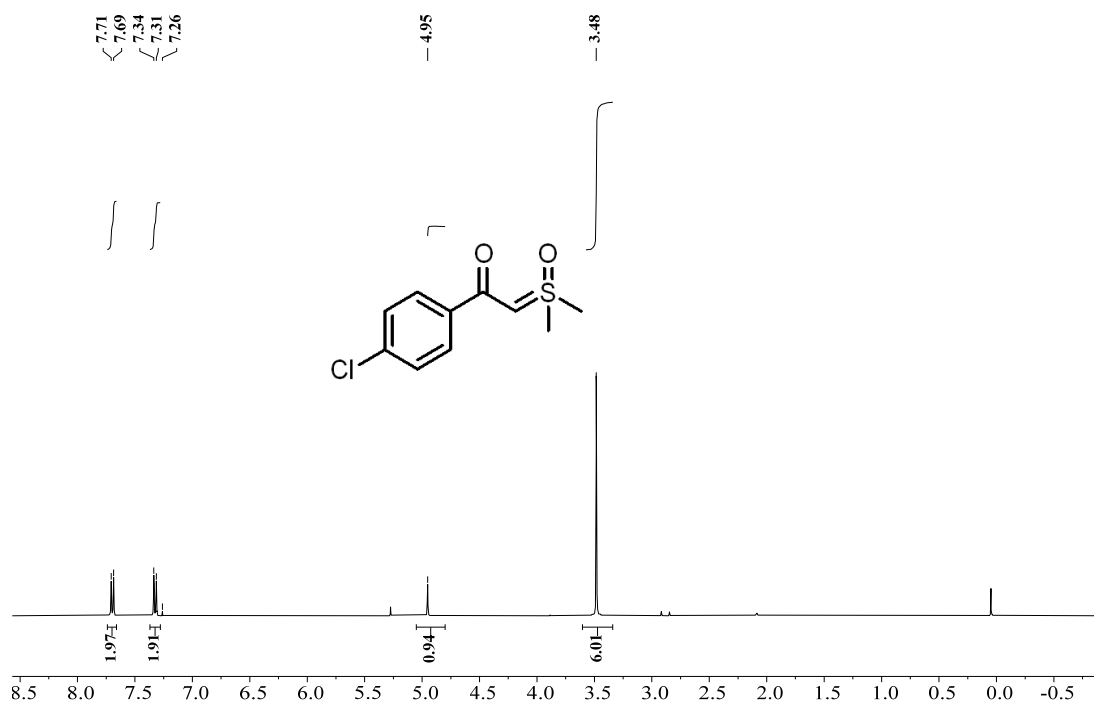


Figure S63. ^1H NMR spectrum of **2g** (CDCl_3 , 400 MHz, 23 °C)

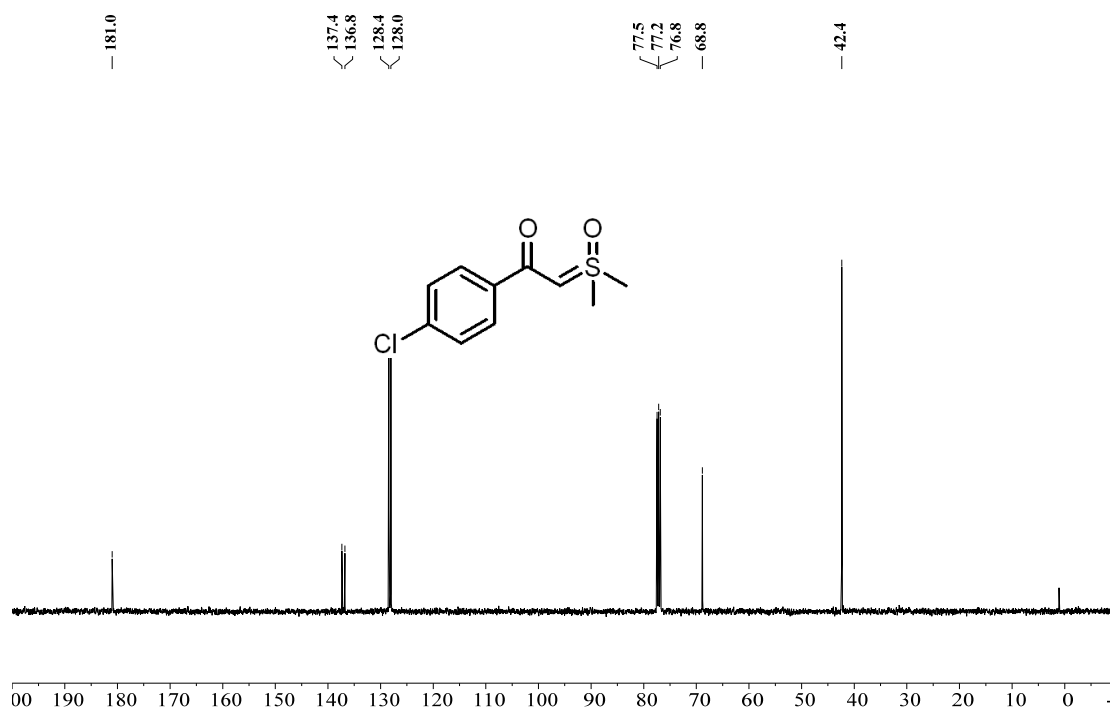


Figure S64. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2g** (CDCl_3 , 101 MHz, 23 °C)

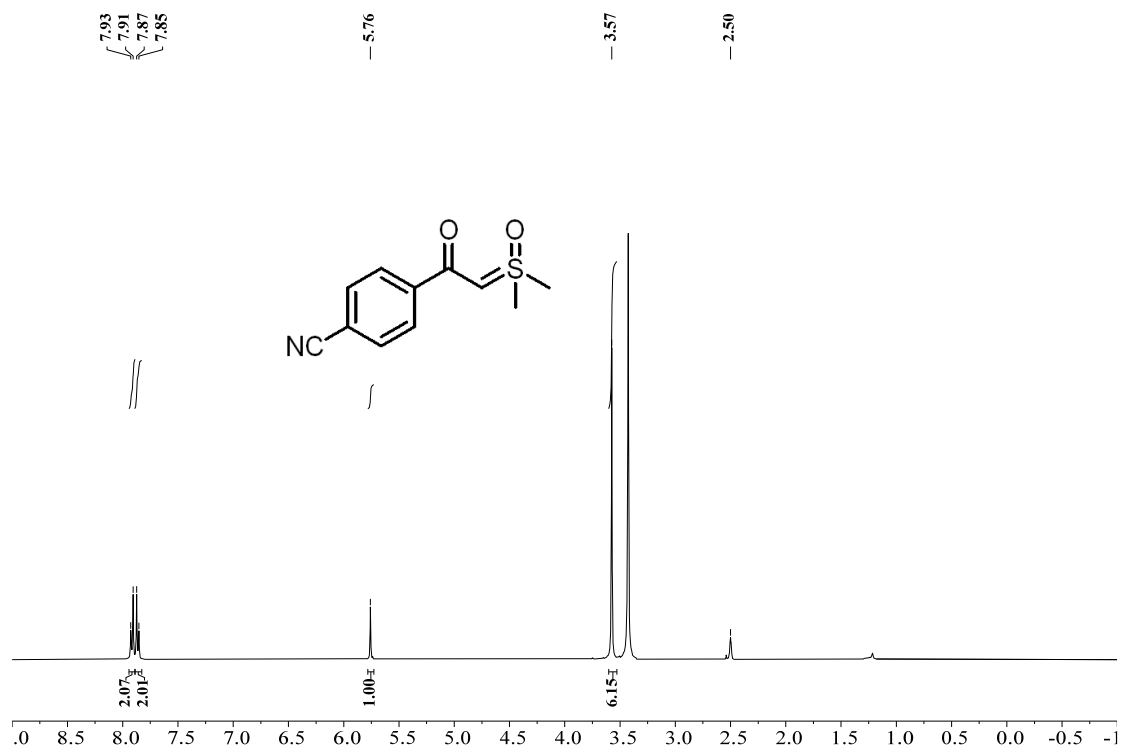


Figure S65. $^1\text{H NMR}$ spectrum of **2h** (DMSO- d_6 , 400 MHz, 23 °C)

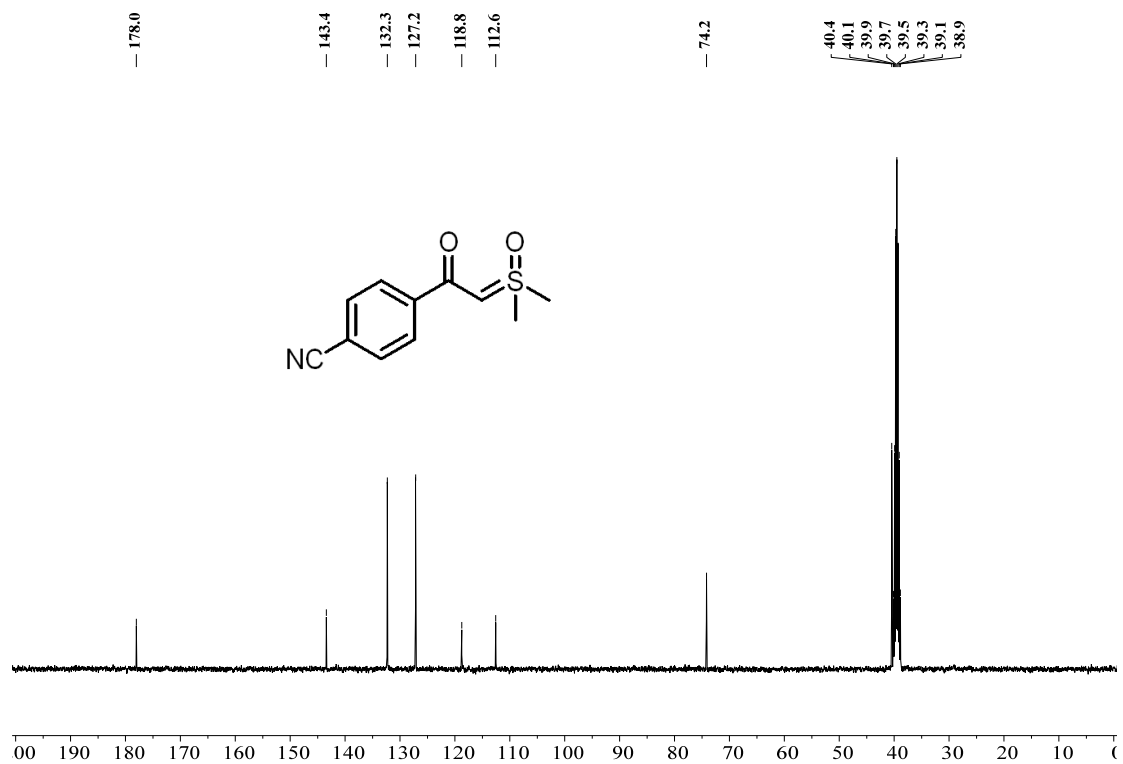


Figure S66. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2h** (DMSO- d_6 , 101 MHz, 23 °C)

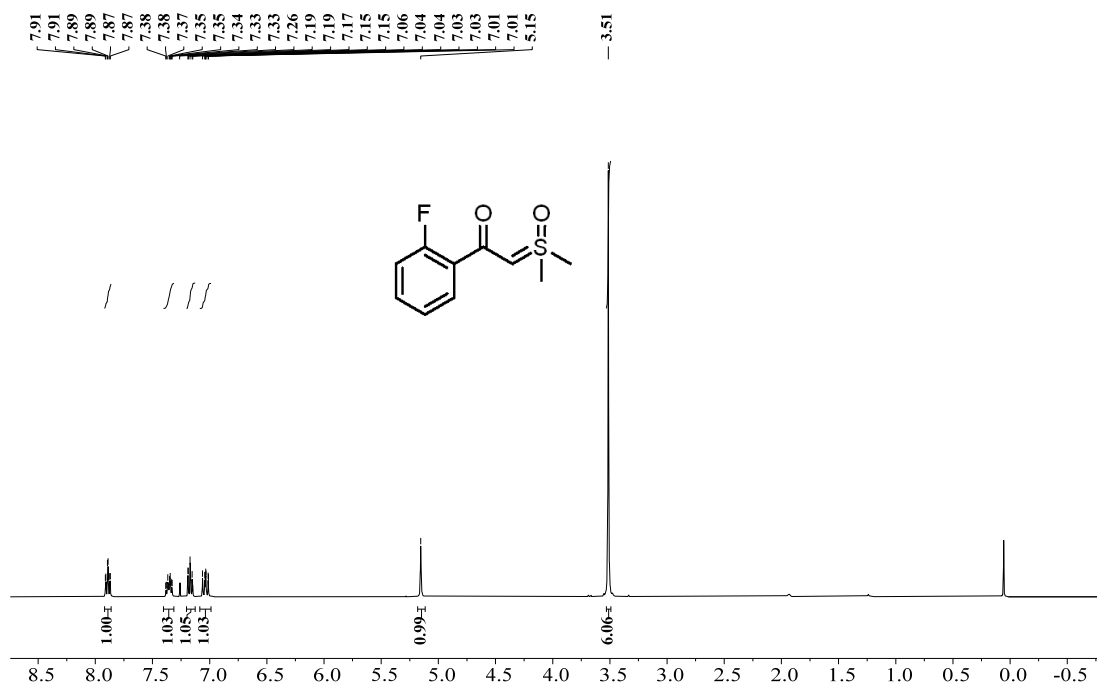


Figure S67. ¹H NMR spectrum of **2i** (CDCl₃, 400 MHz, 23 °C)

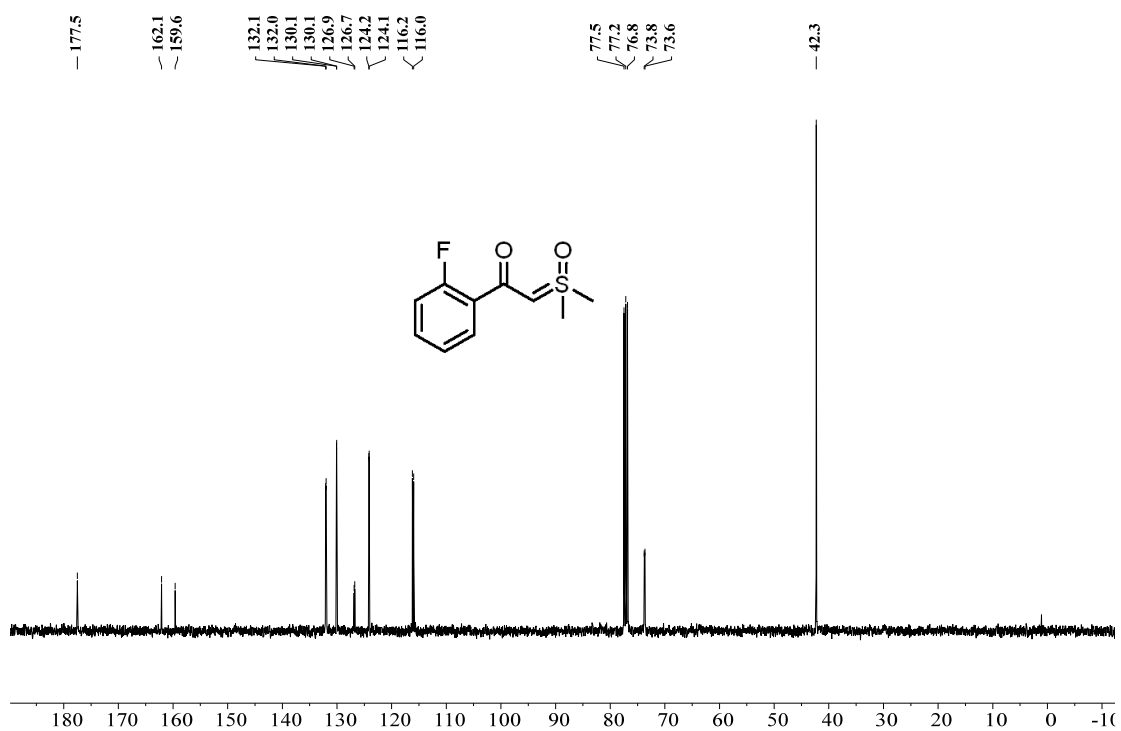


Figure S68. ¹³C{¹H} NMR spectrum of **2i** (CDCl₃, 101 MHz, 23 °C)

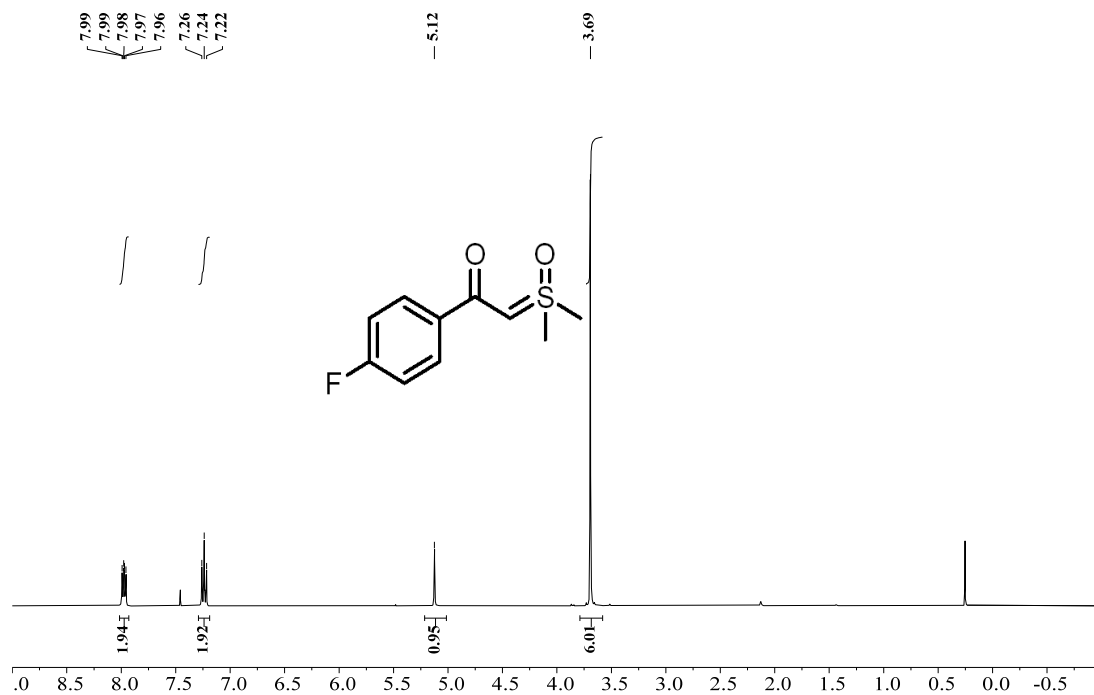


Figure S69. ^1H NMR spectrum of **2j** (CDCl_3 , 400 MHz, 23 °C)

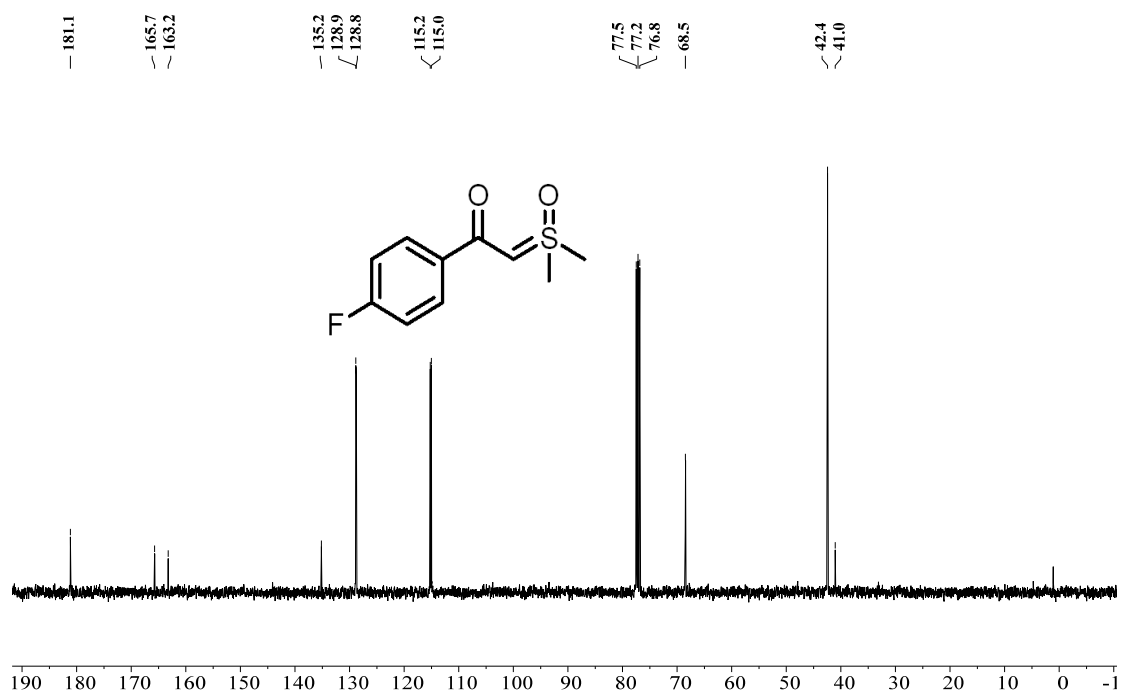


Figure S70. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2j** (CDCl_3 , 101 MHz, 23 °C)

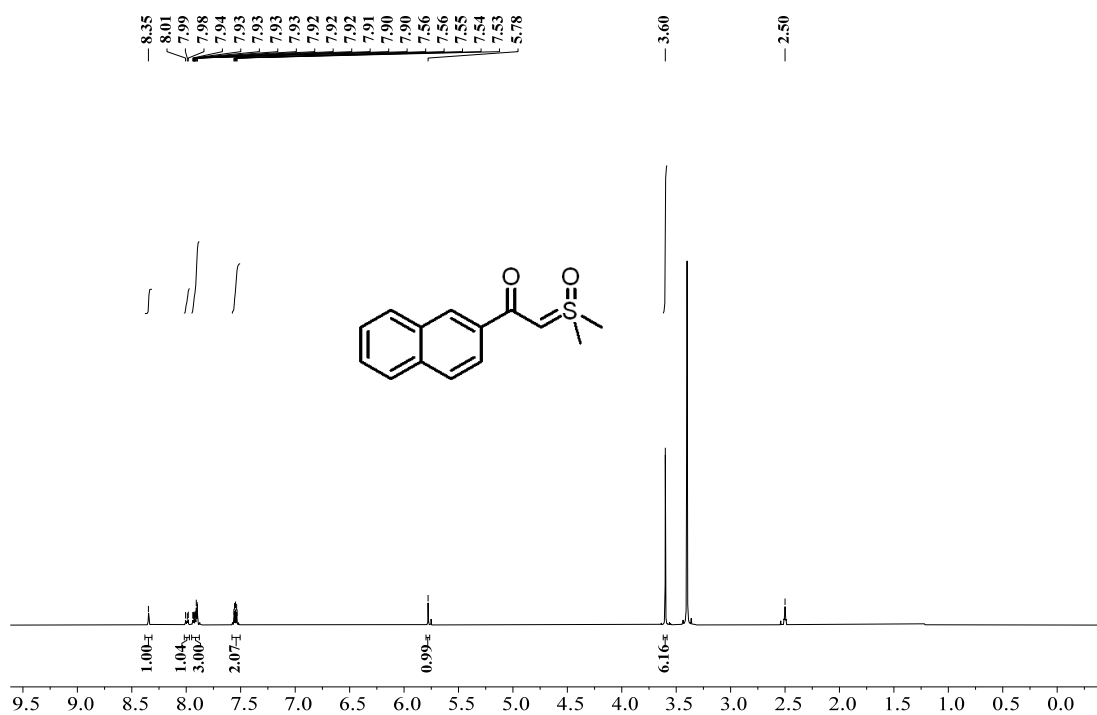


Figure S71. ^1H NMR spectrum of **2k** (DMSO- d_6 , 400 MHz, 23 °C)

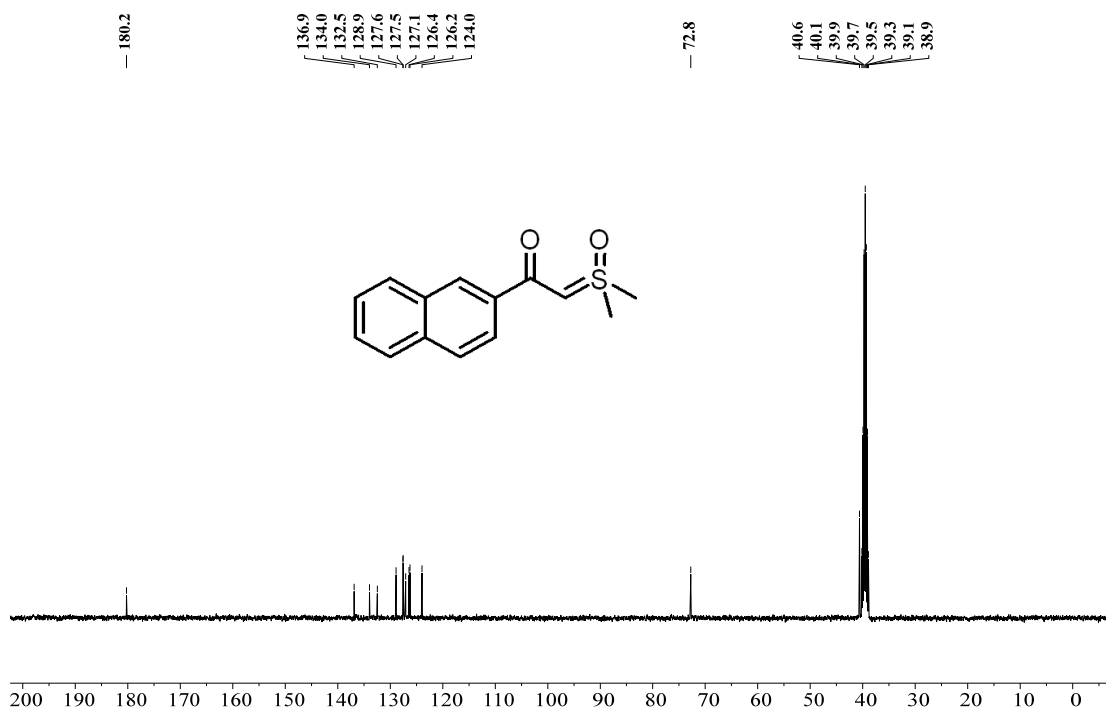


Figure S72. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2k** (DMSO- d_6 , 101 MHz, 23 °C)

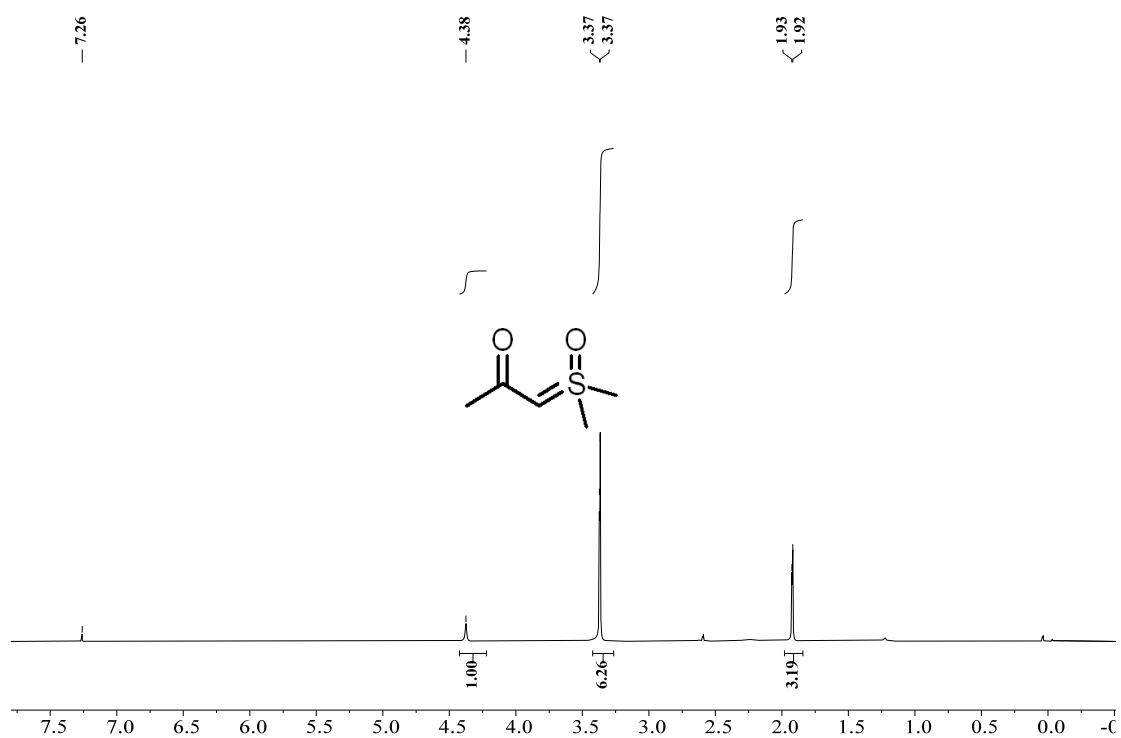


Figure S73. ^1H NMR spectrum of **2I** (CDCl_3 , 400 MHz, 23 °C)

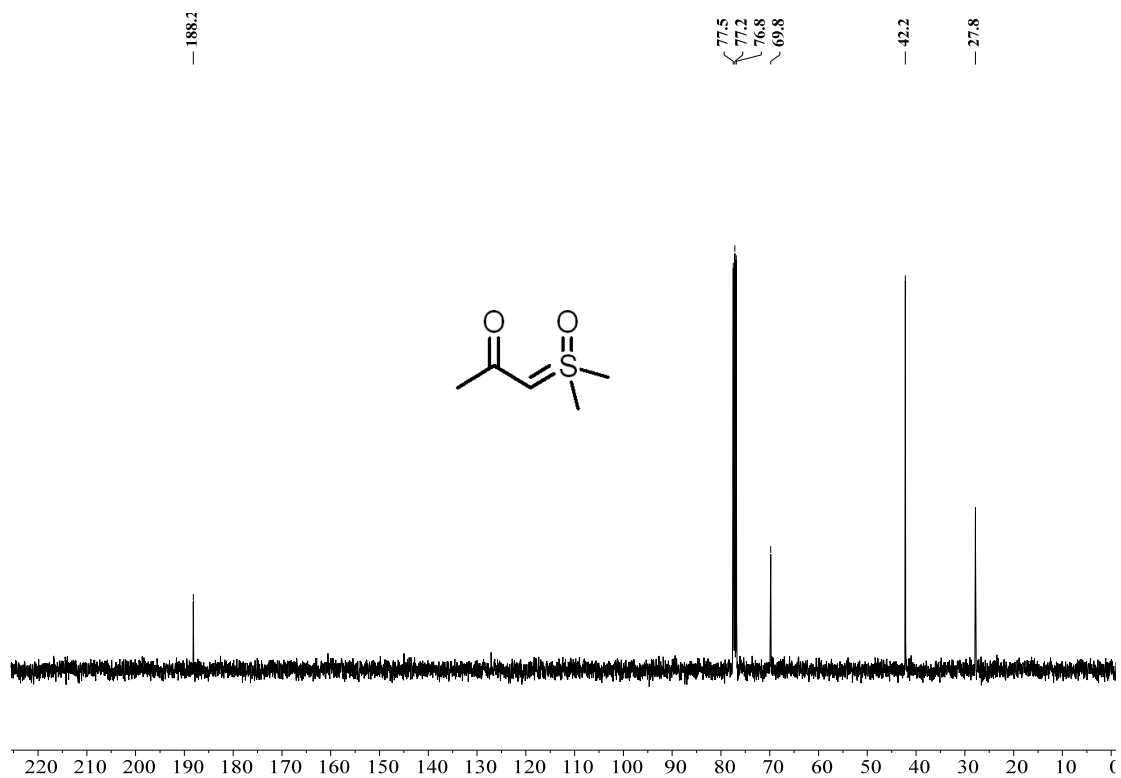


Figure S74. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2I** (CDCl_3 , 101 MHz, 23 °C)

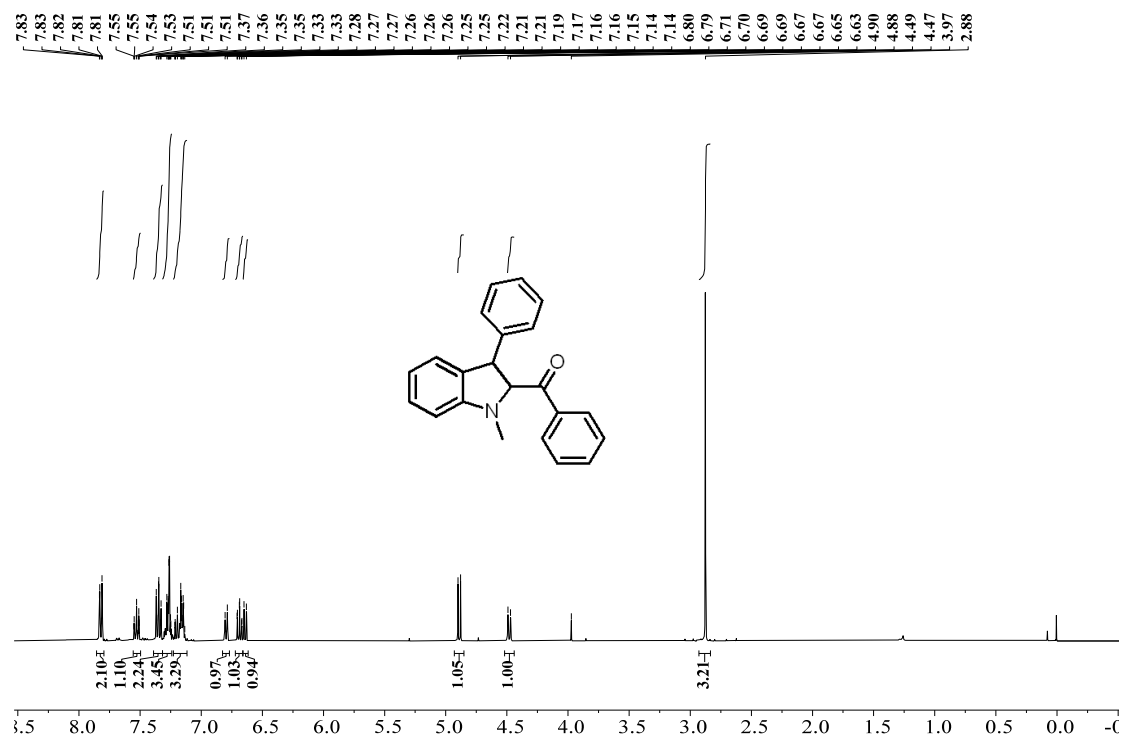


Figure S75. ^1H NMR spectrum of **3a** (CDCl_3 , 400 MHz, 23 °C)

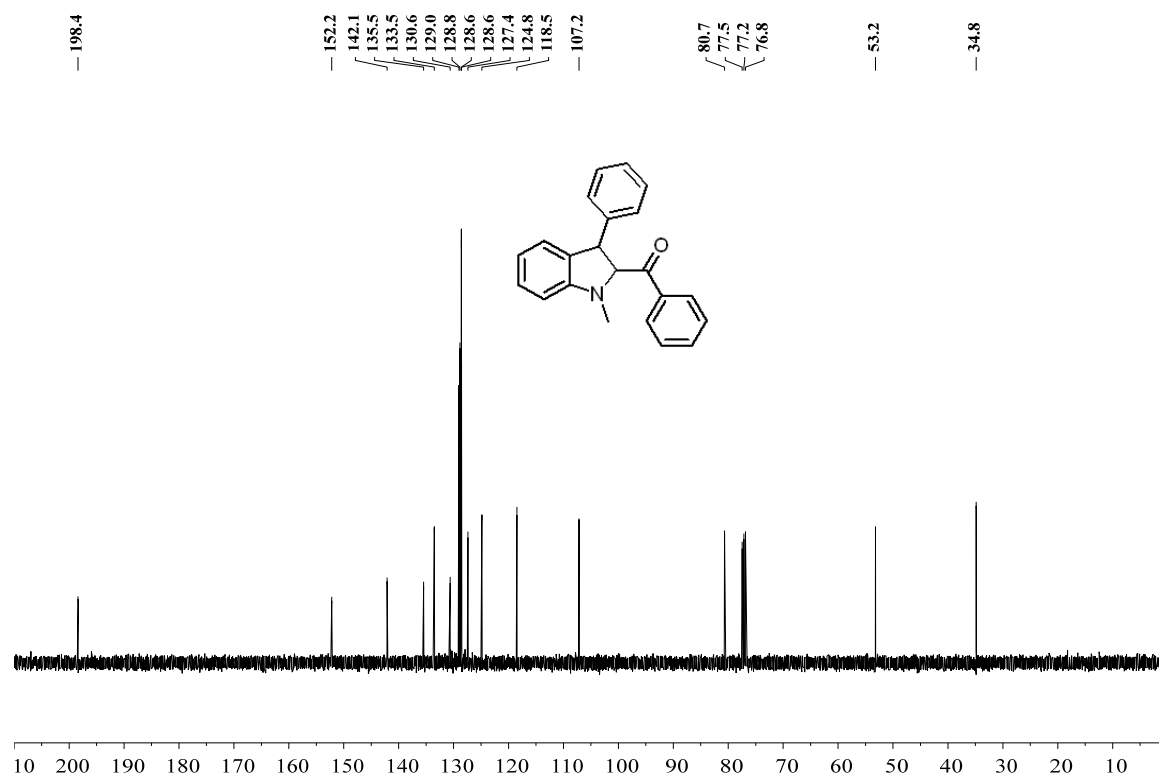


Figure S76. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3a** (CDCl_3 , 101 MHz, 23 °C)

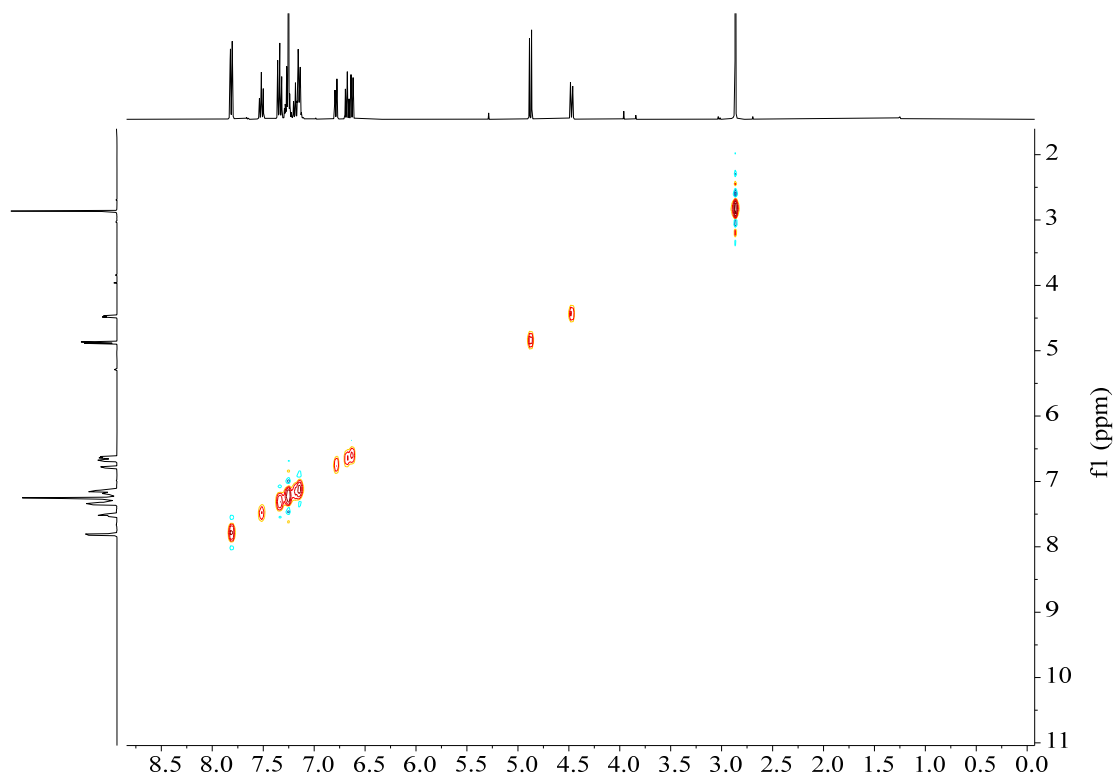


Figure S77. NOE spectrum of **3a** (CDCl_3 , $23\text{ }^\circ\text{C}$)

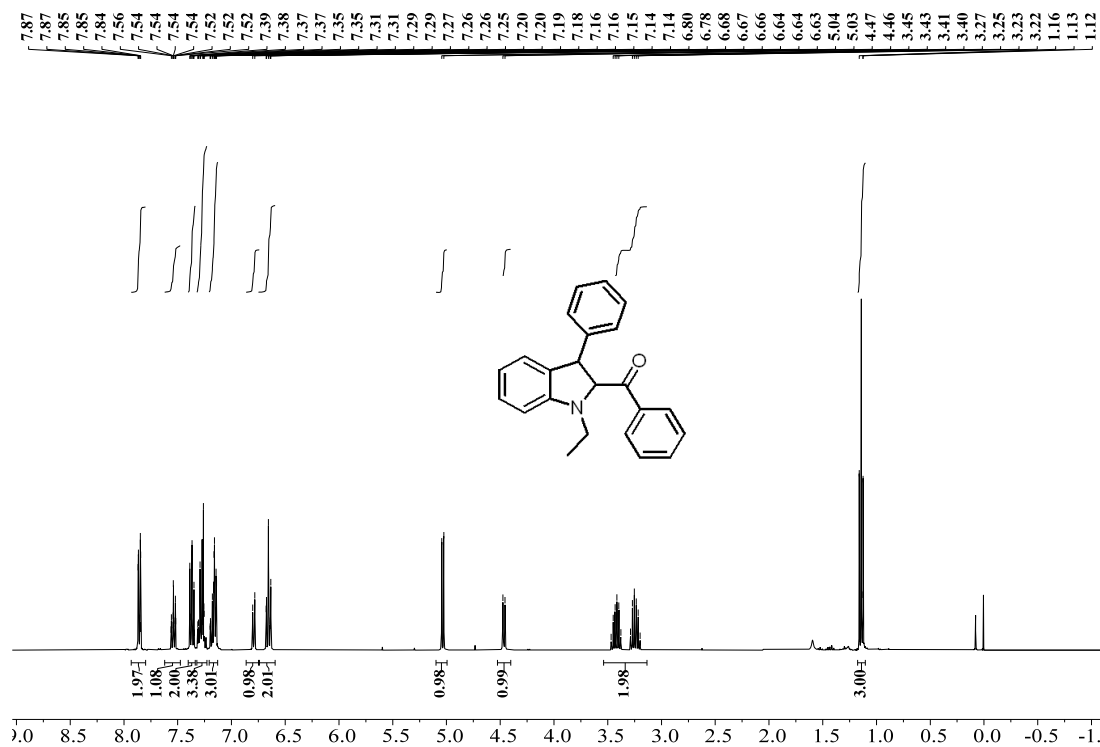


Figure S78. ¹H NMR spectrum of **3c** (CDCl₃, 400 MHz, 23 °C)

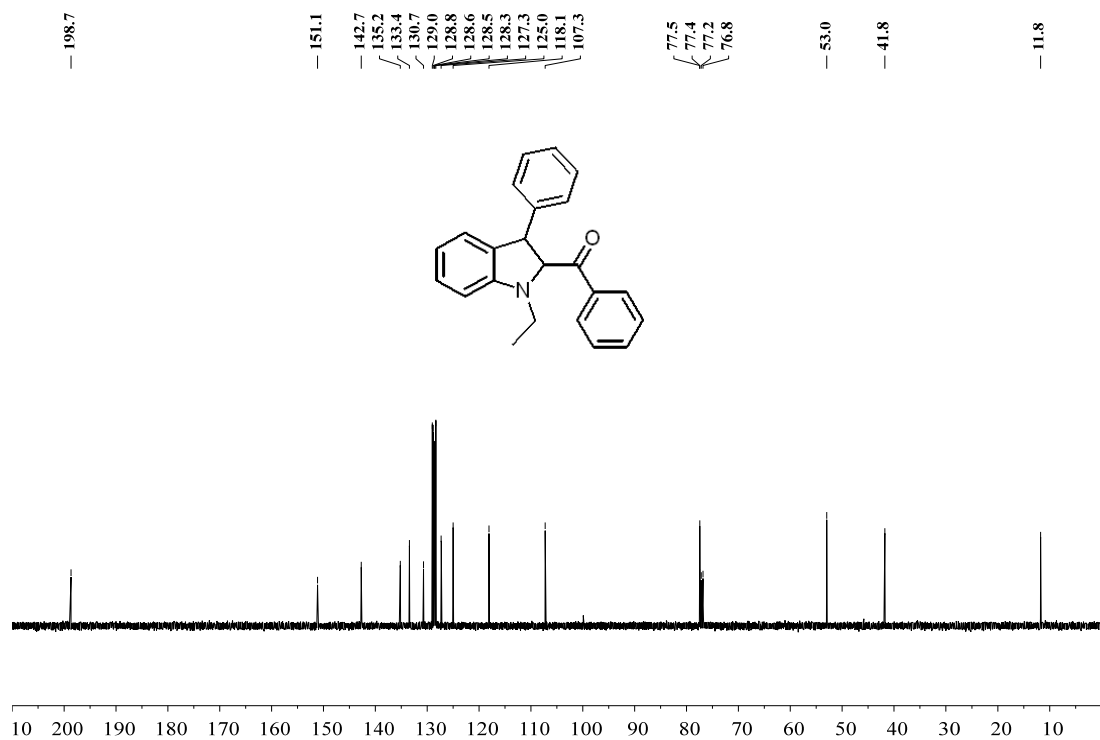


Figure S79. ¹³C {¹H} NMR spectrum of **3c** (CDCl₃, 101 MHz, 23 °C)

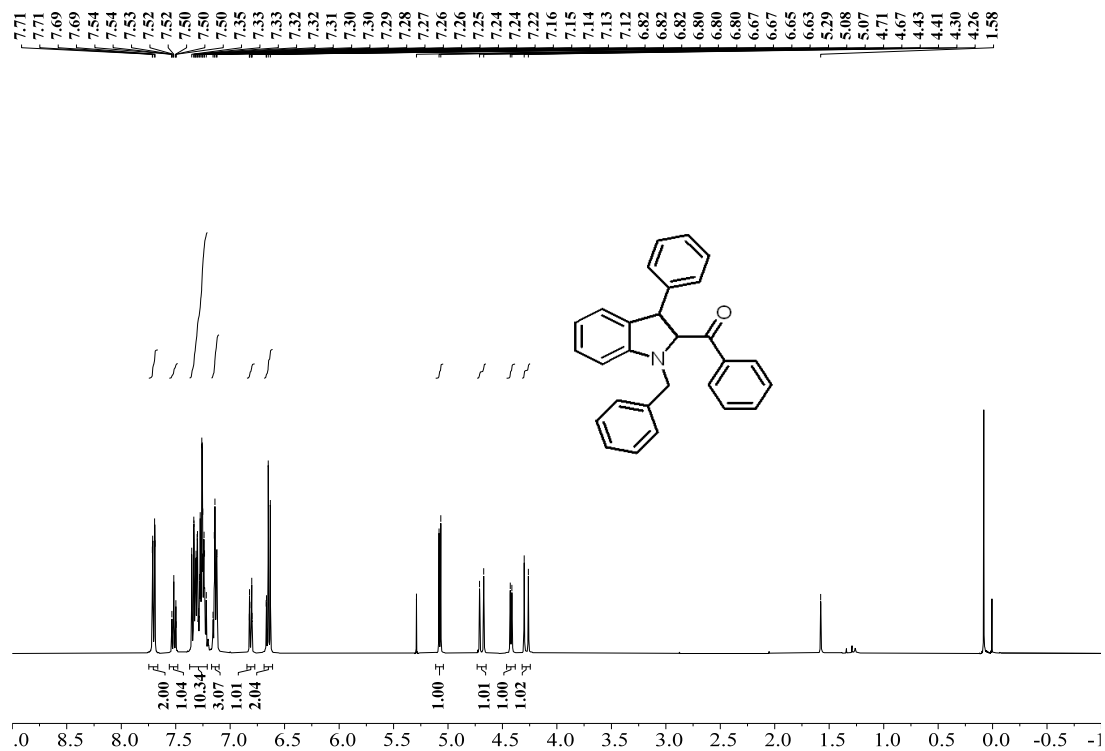


Figure S80. ¹H NMR spectrum of **3d** (CDCl₃, 400 MHz, 23 °C)

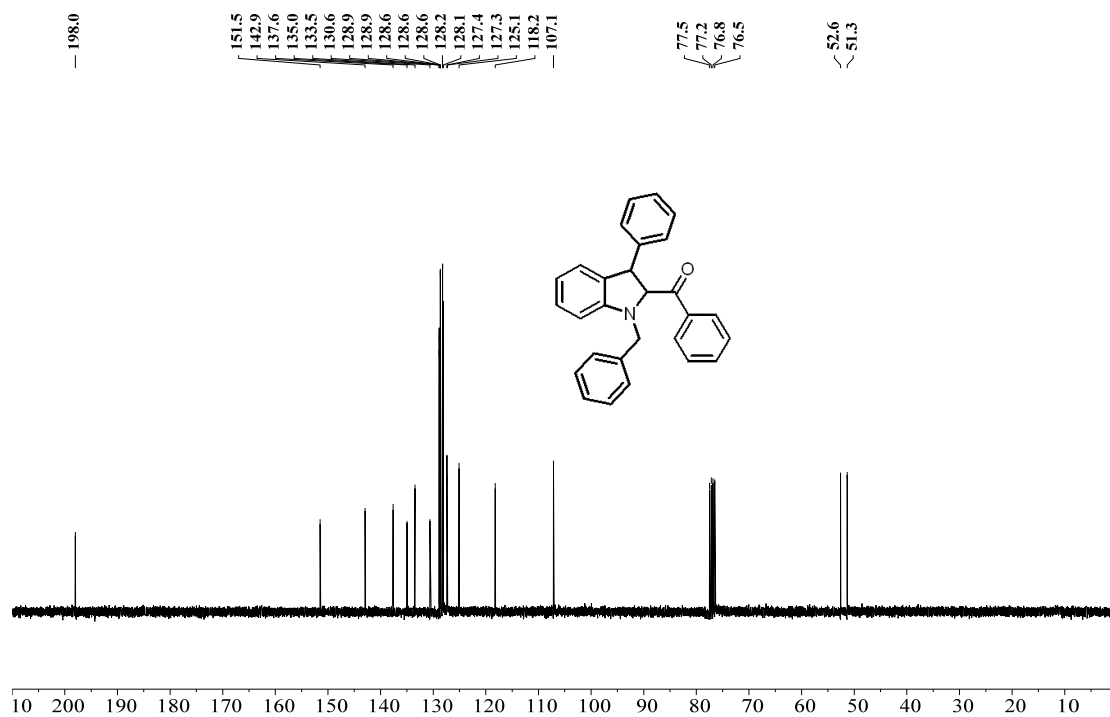


Figure S81. ¹³C{¹H} NMR spectrum of **3d** (CDCl₃, 101 MHz, 23 °C)

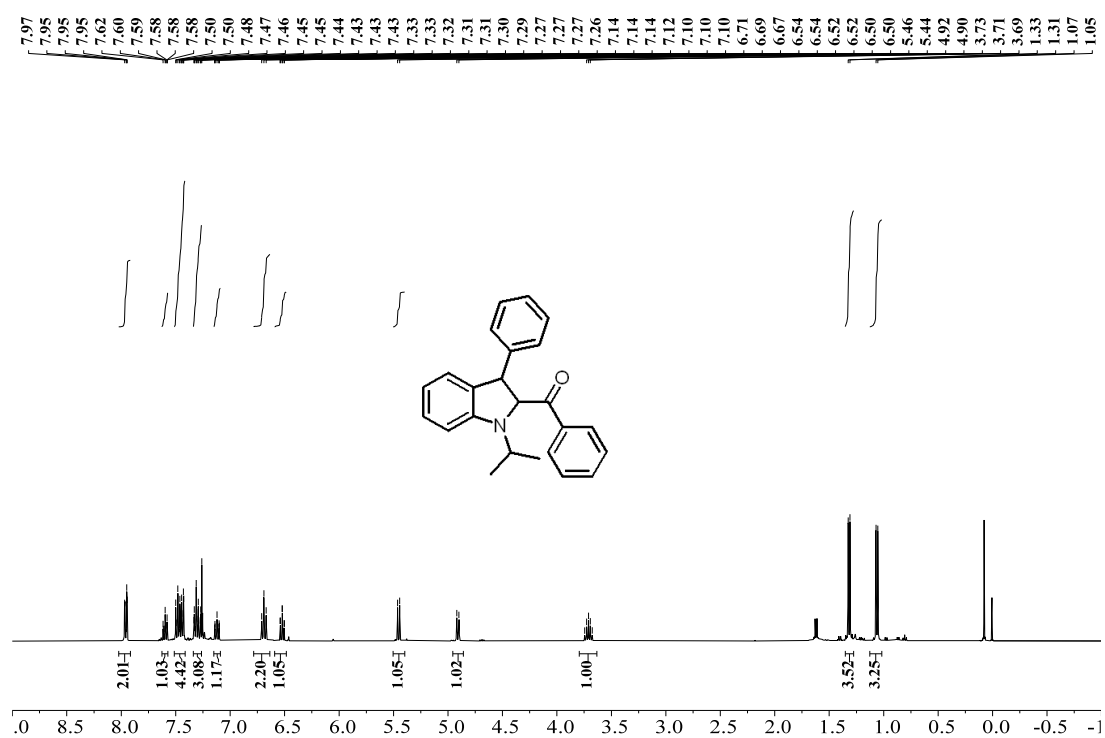


Figure S82. ^1H NMR spectrum of **3e** (CDCl_3 , 400 MHz, 23 °C)

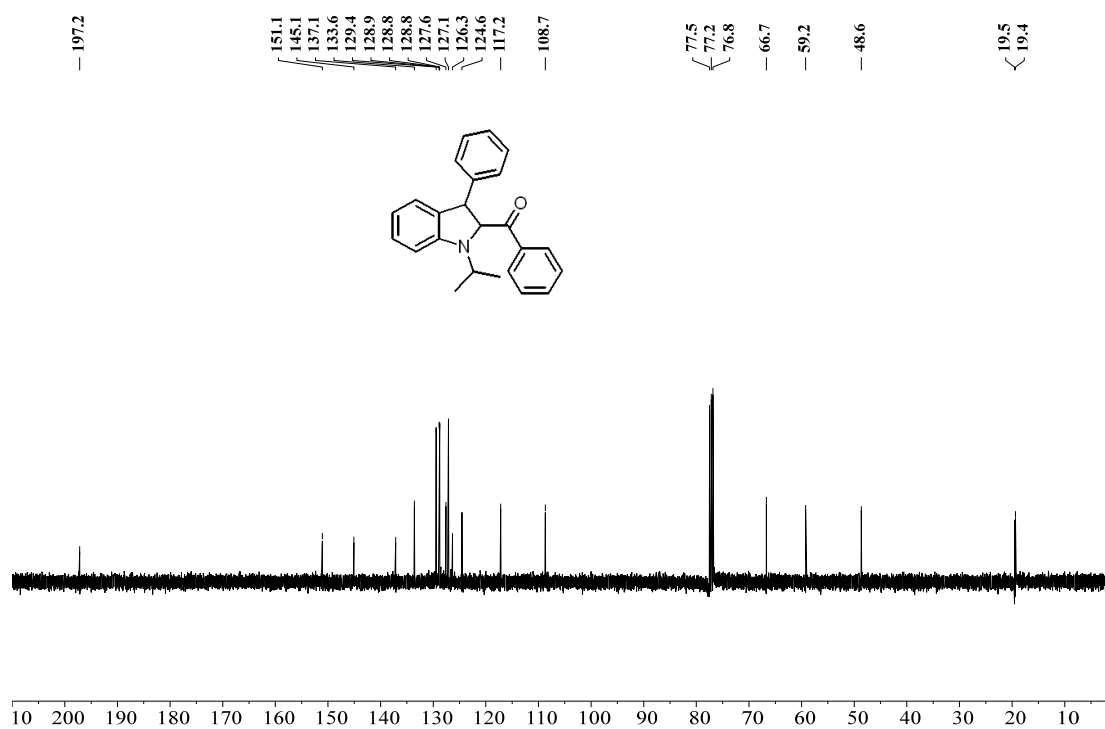


Figure S83. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3e** (CDCl_3 , 101 MHz, 23 °C)

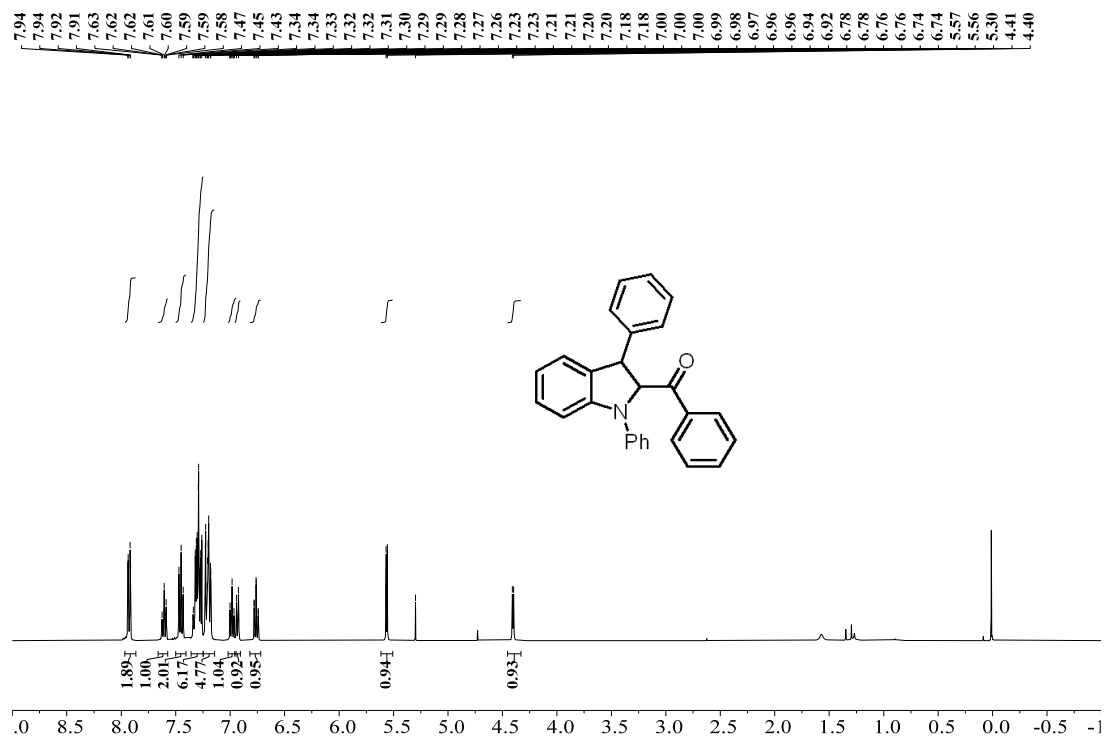


Figure S84. ^1H NMR spectrum of **3f** (CDCl_3 , 400 MHz, 23 °C)

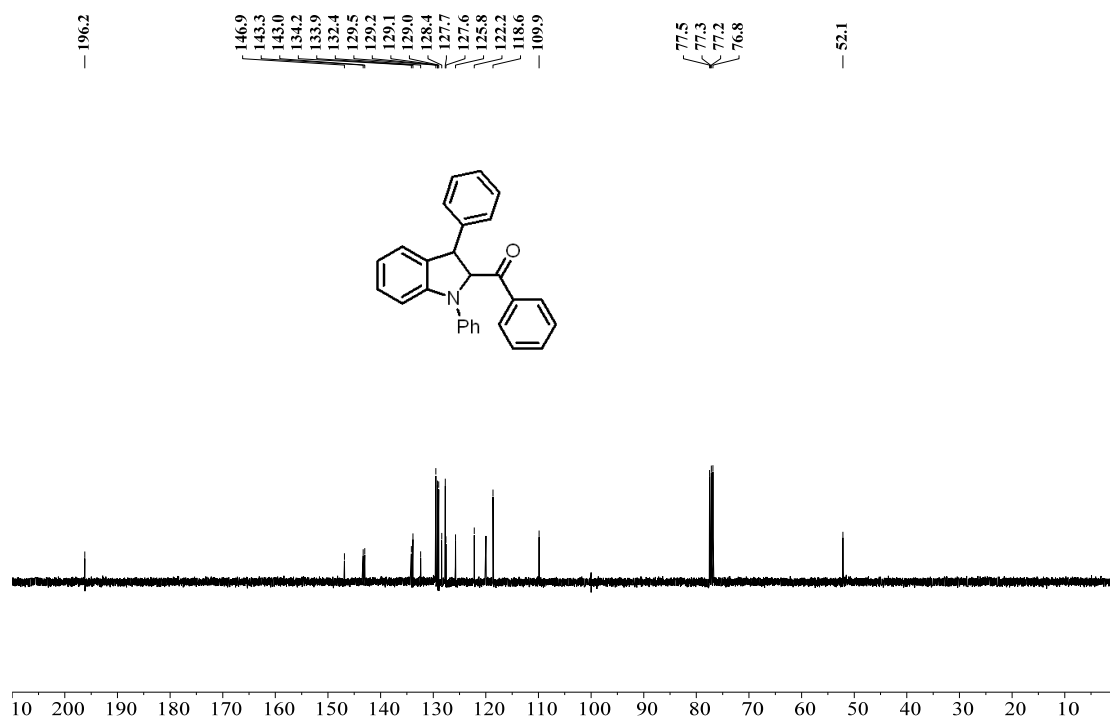


Figure S85. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3f** (CDCl_3 , 101 MHz, 23 °C)

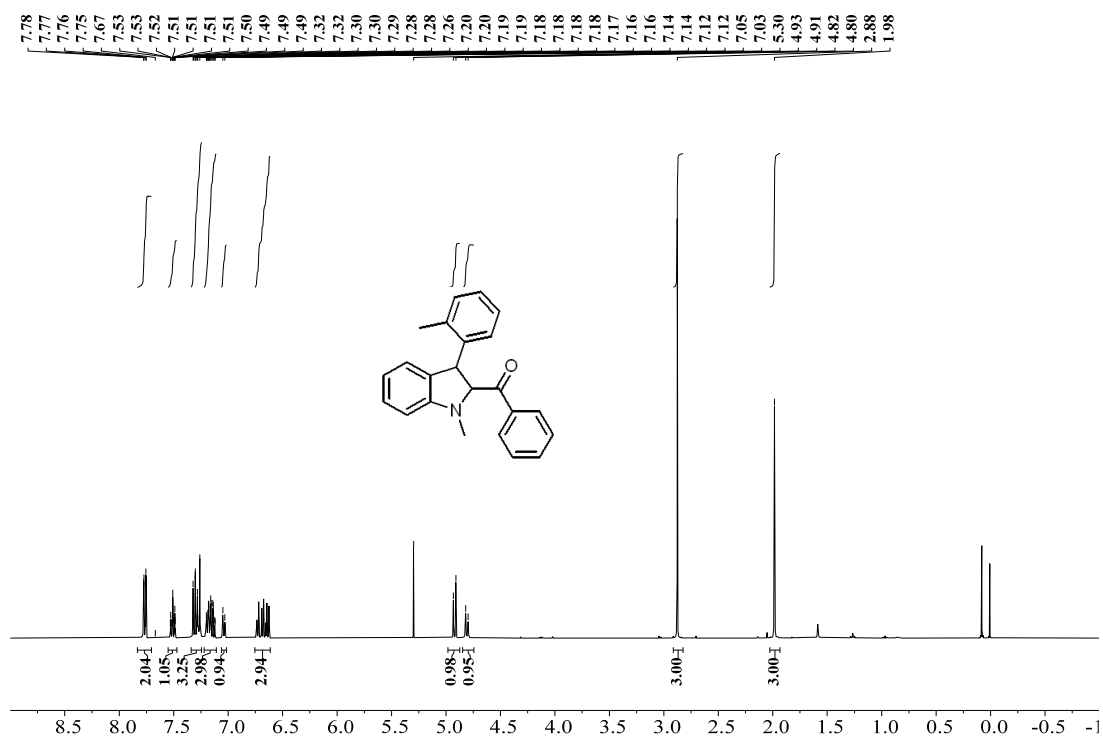


Figure S86. ¹H NMR spectrum of **3j** (CDCl₃, 400 MHz, 23 °C)

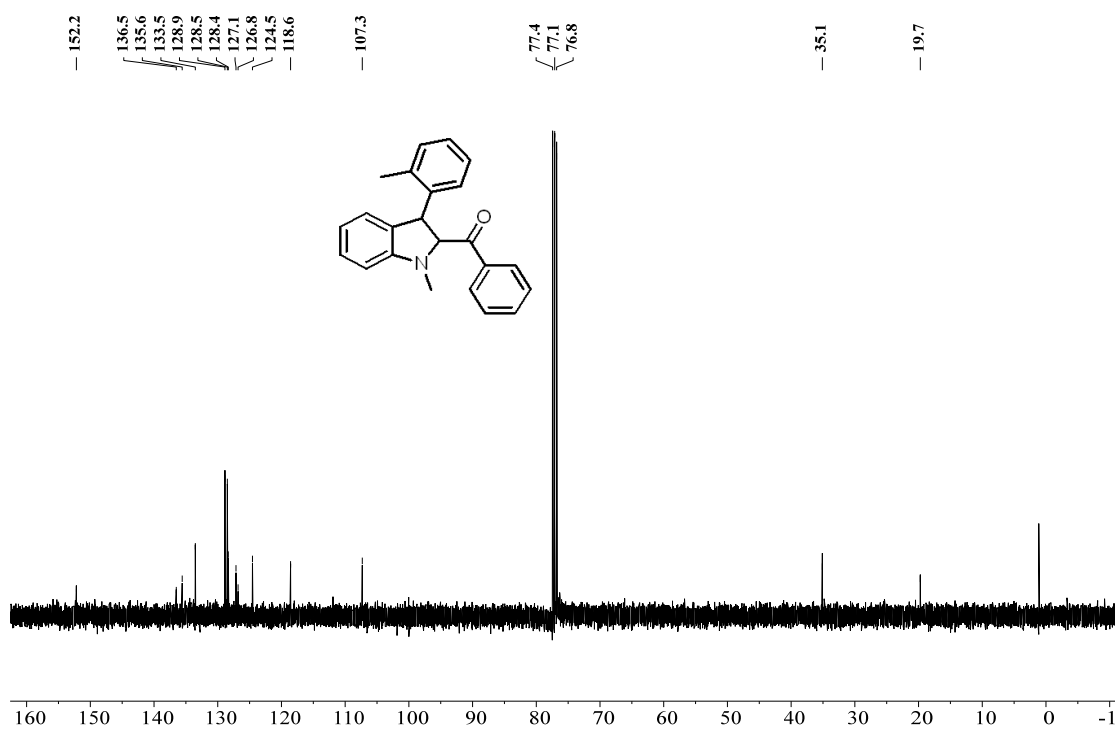


Figure S87. ¹³C{¹H} NMR spectrum of **3j** (CDCl₃, 101 MHz, 23 °C)

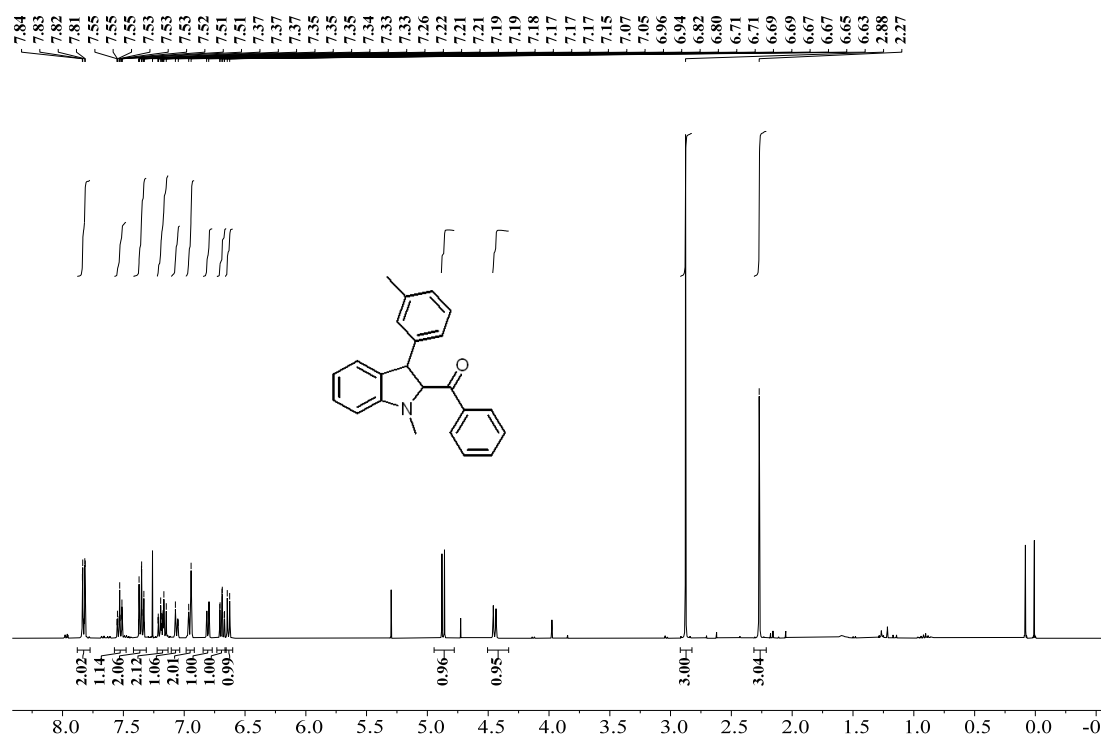


Figure S88. ^1H NMR spectrum of **3k** (CDCl_3 , 400 MHz, 23 °C)

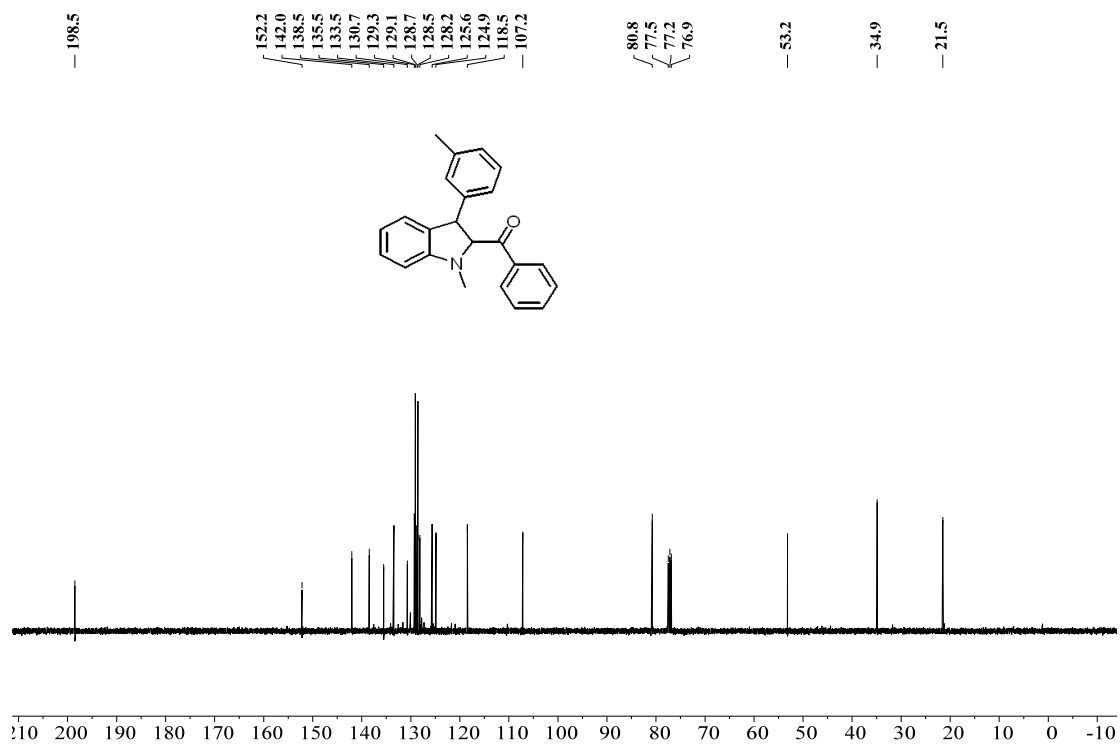


Figure S89. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3k** (CDCl_3 , 101 MHz, 23 °C)

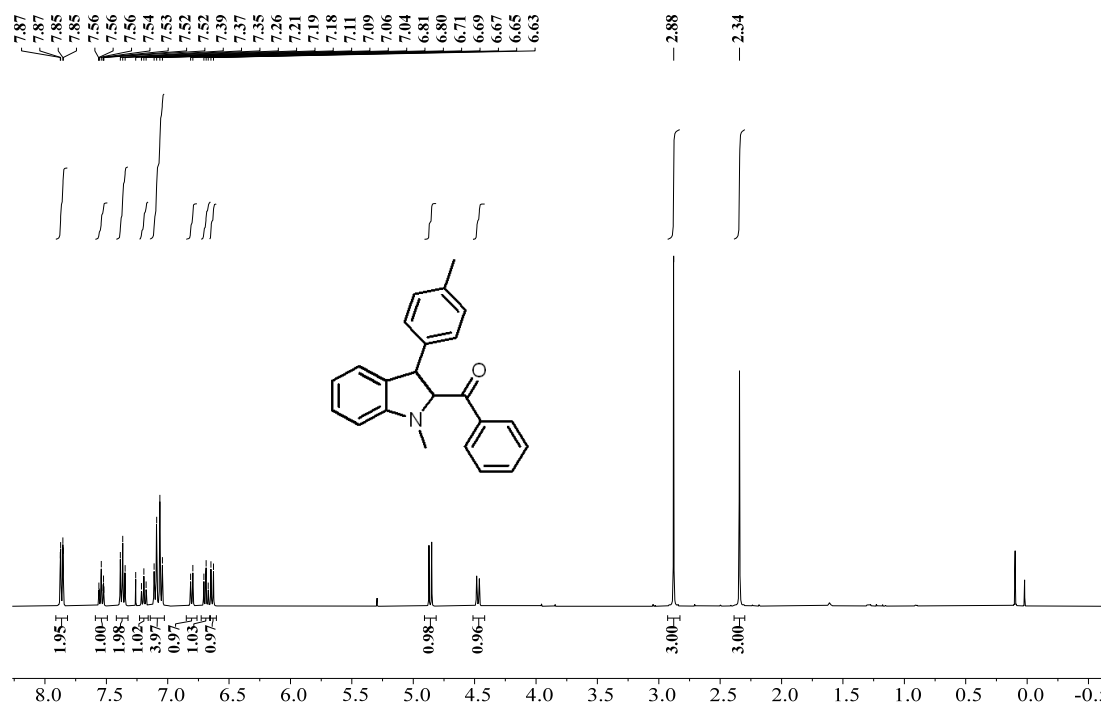


Figure S90. ^1H NMR spectrum of **3I** (CDCl_3 , 400 MHz, 23 °C)

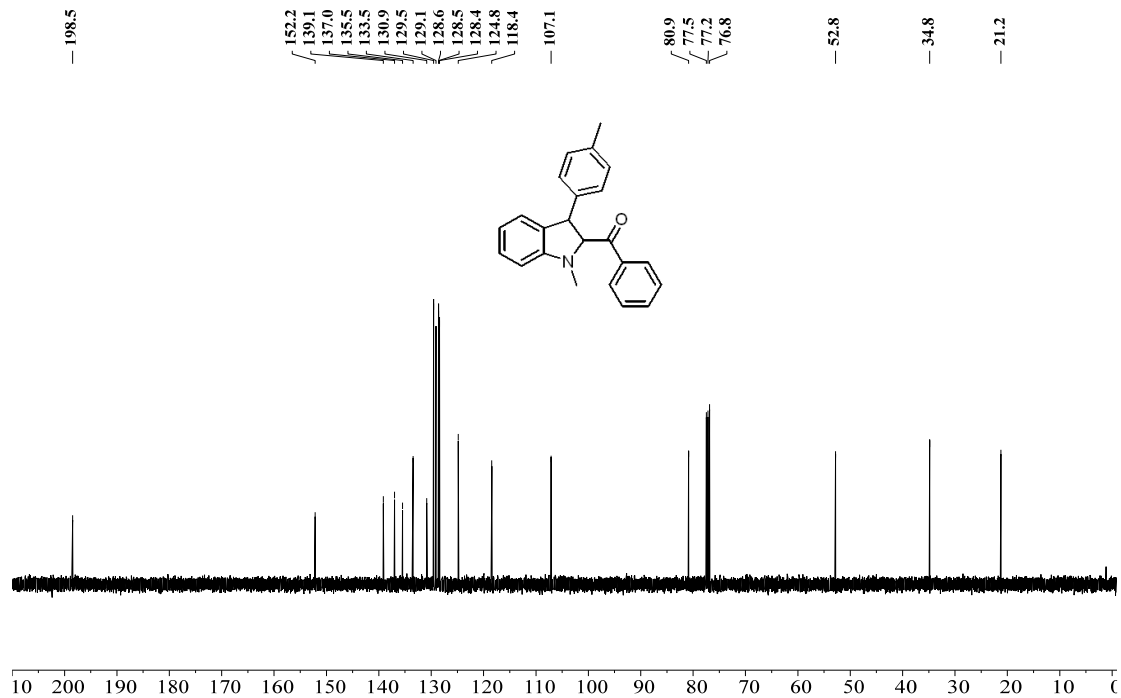


Figure S91. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3I** (CDCl_3 , 101 MHz, 23 °C)

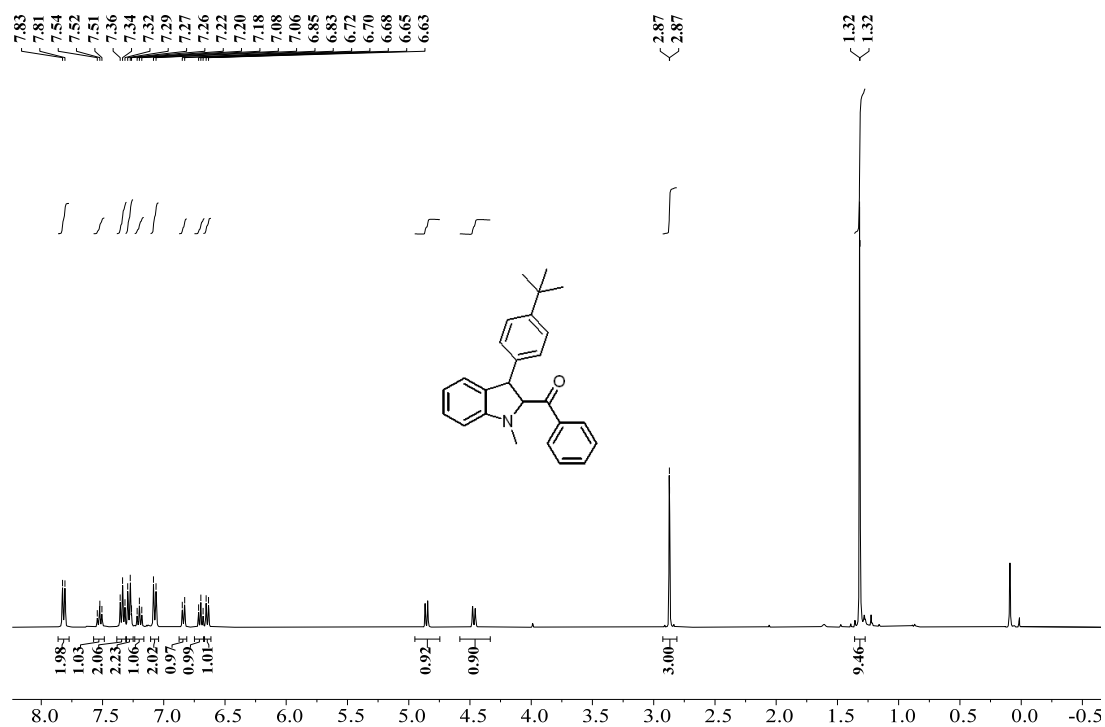


Figure S92. ^1H NMR spectrum of **3m** (CDCl_3 , 400 MHz, 23 °C)

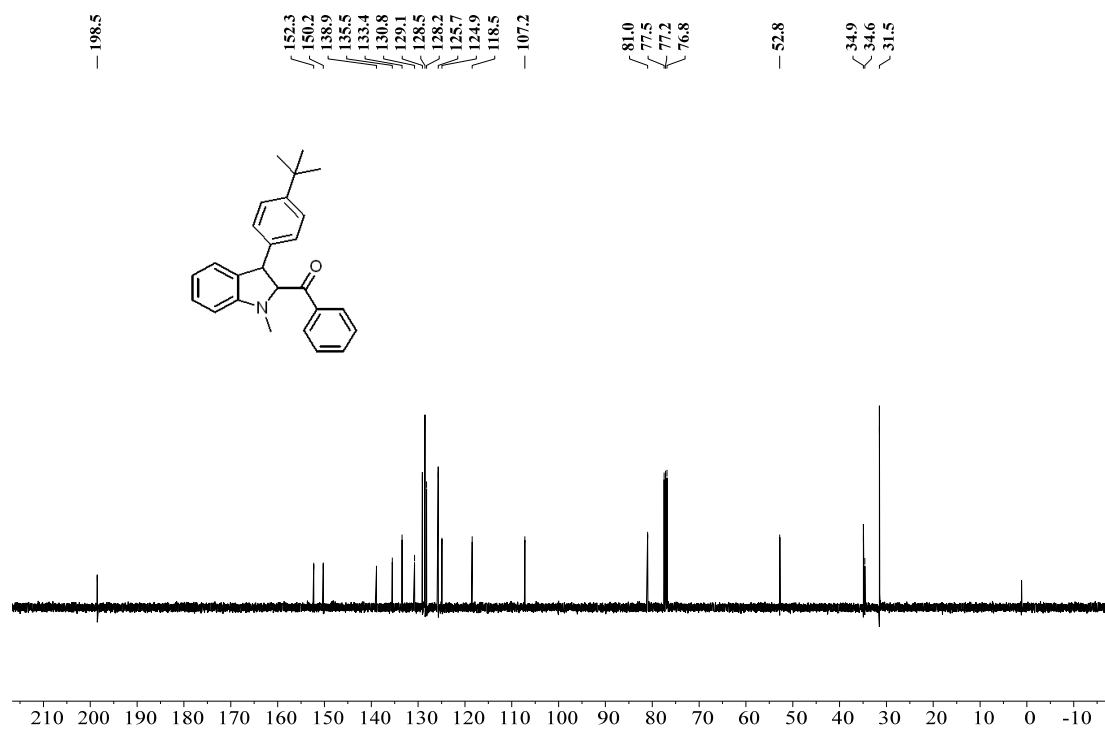


Figure S93. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3m** (CDCl_3 , 101 MHz, 23 °C)

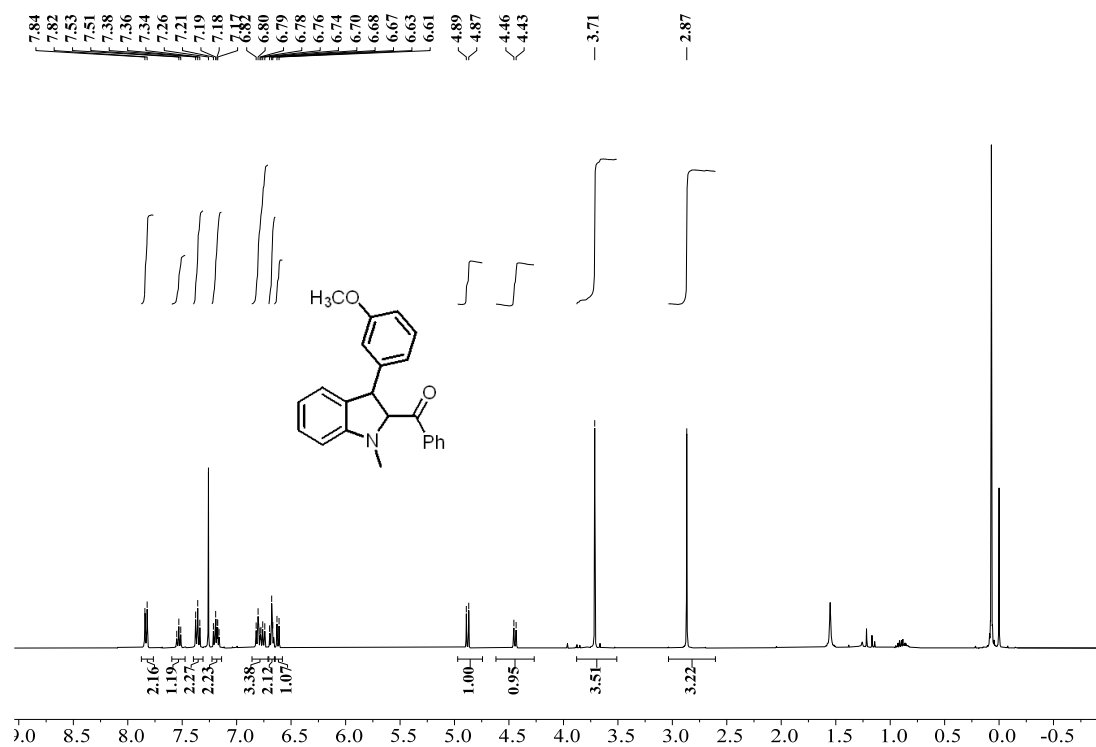


Figure S94. ^1H NMR spectrum of **3n** (CDCl_3 , 400 MHz, 23 $^\circ\text{C}$)

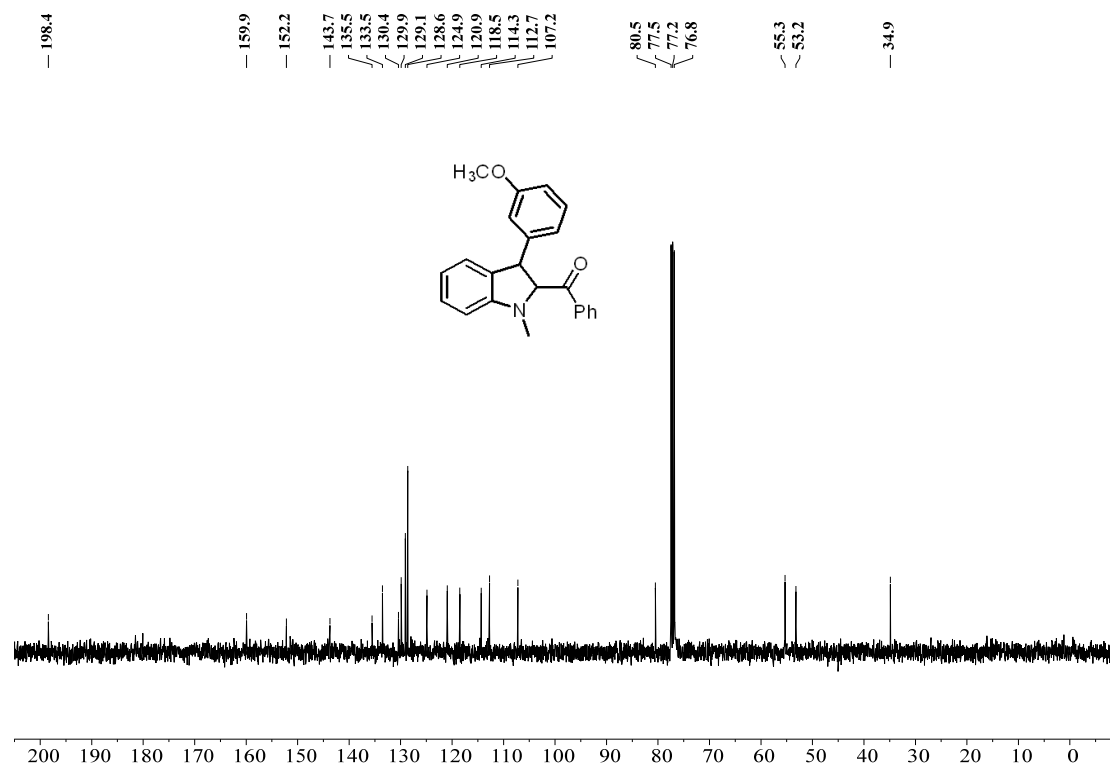


Figure S95. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3n** (CDCl_3 , 101 MHz, 23 $^\circ\text{C}$)

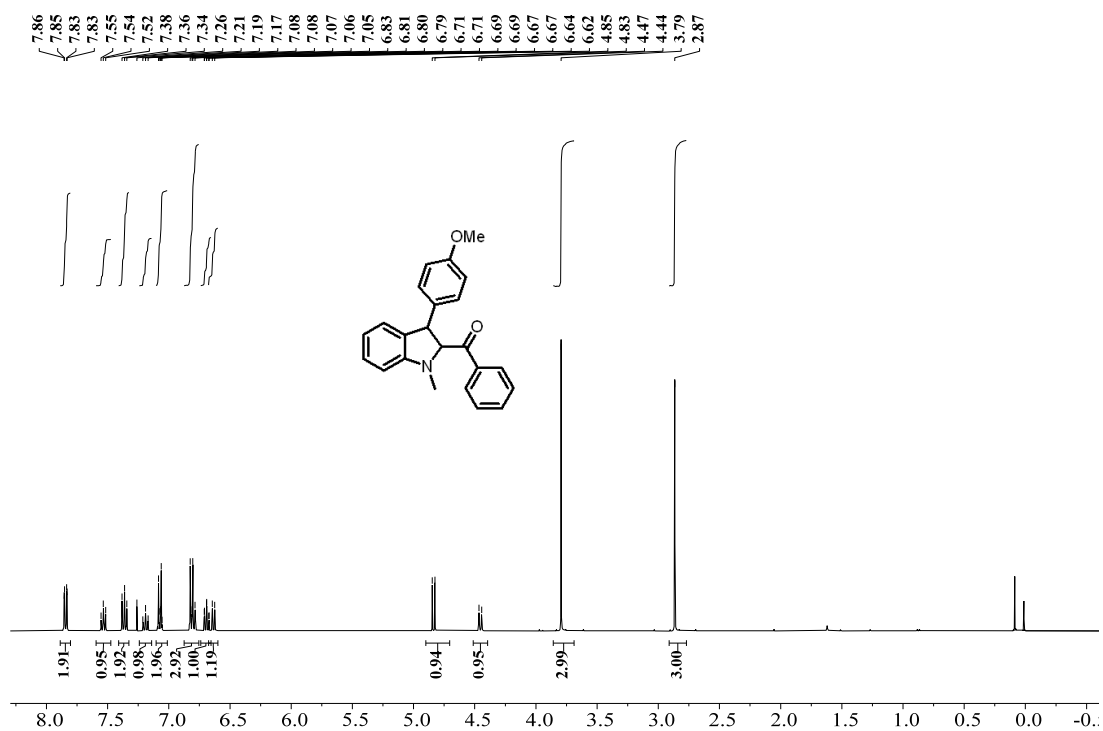


Figure S96. ^1H NMR spectrum of **3o** (CDCl_3 , 400 MHz, 23 °C)

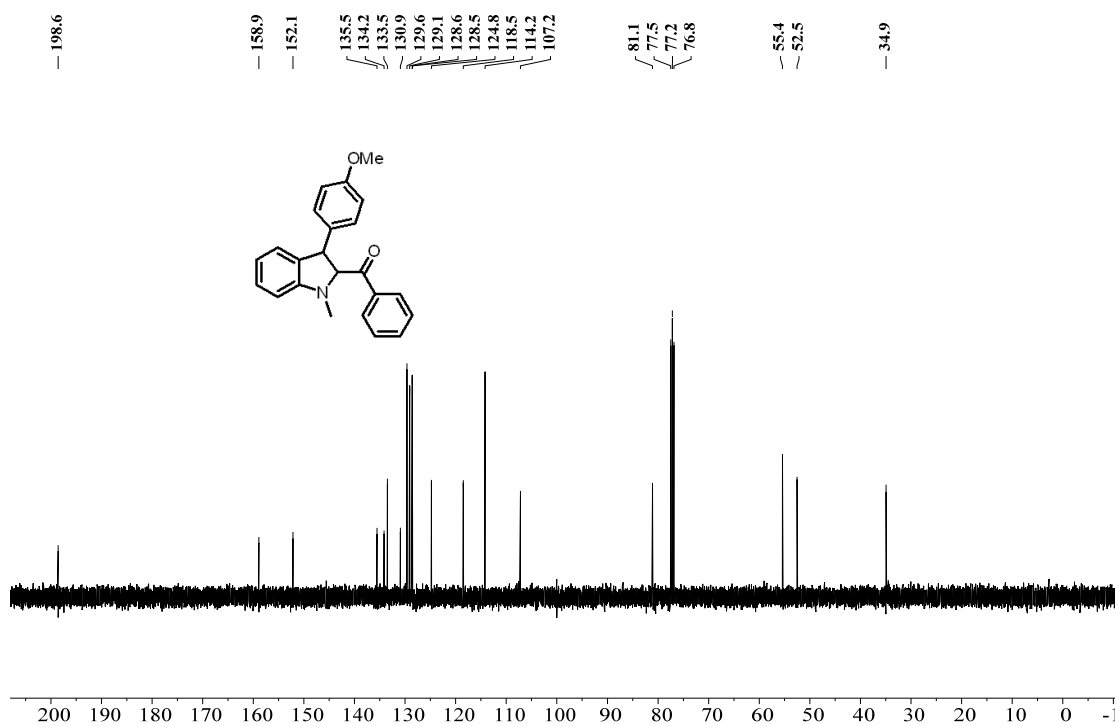


Figure S97. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3o** (CDCl_3 , 101 MHz, 23 °C)

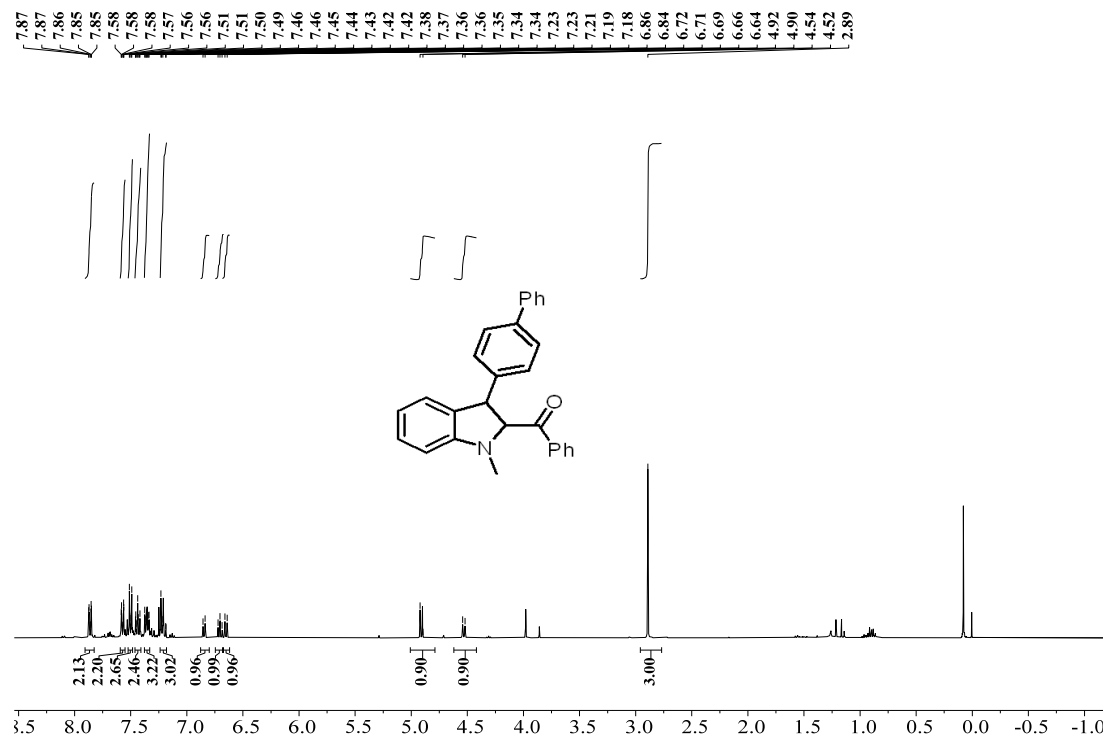


Figure S98. ^1H NMR spectrum of **3p** (CDCl_3 , 400 MHz, 23 °C)

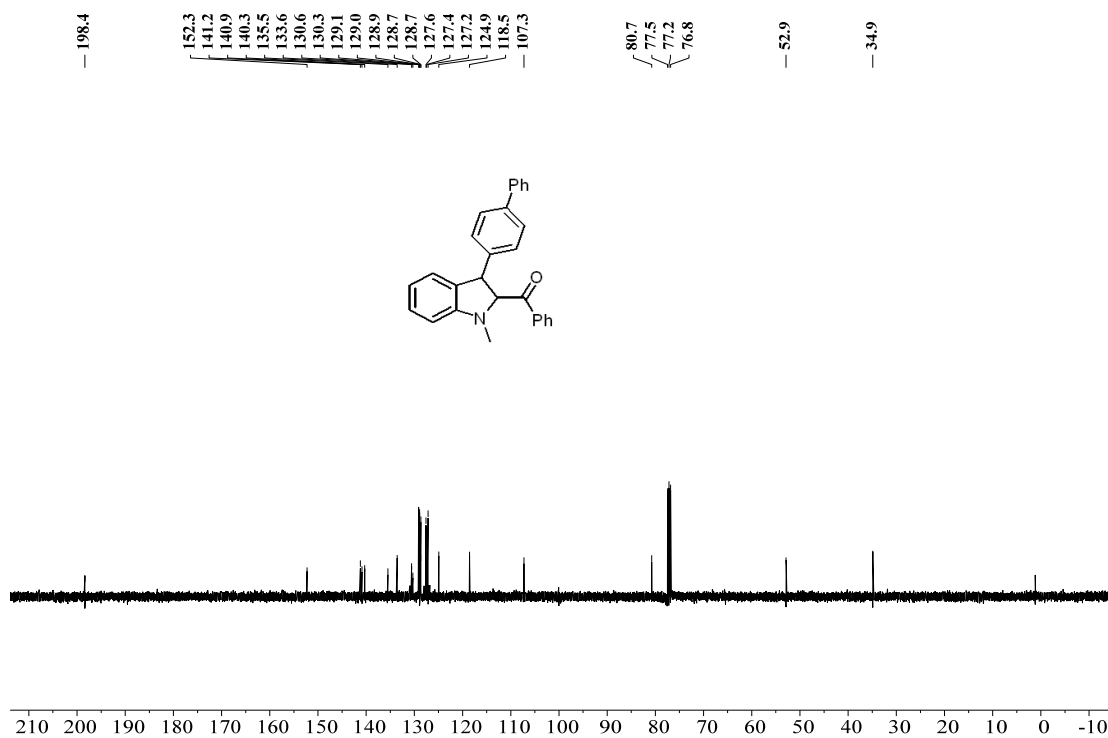


Figure S99. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3p** (CDCl_3 , 101 MHz, 23 °C)

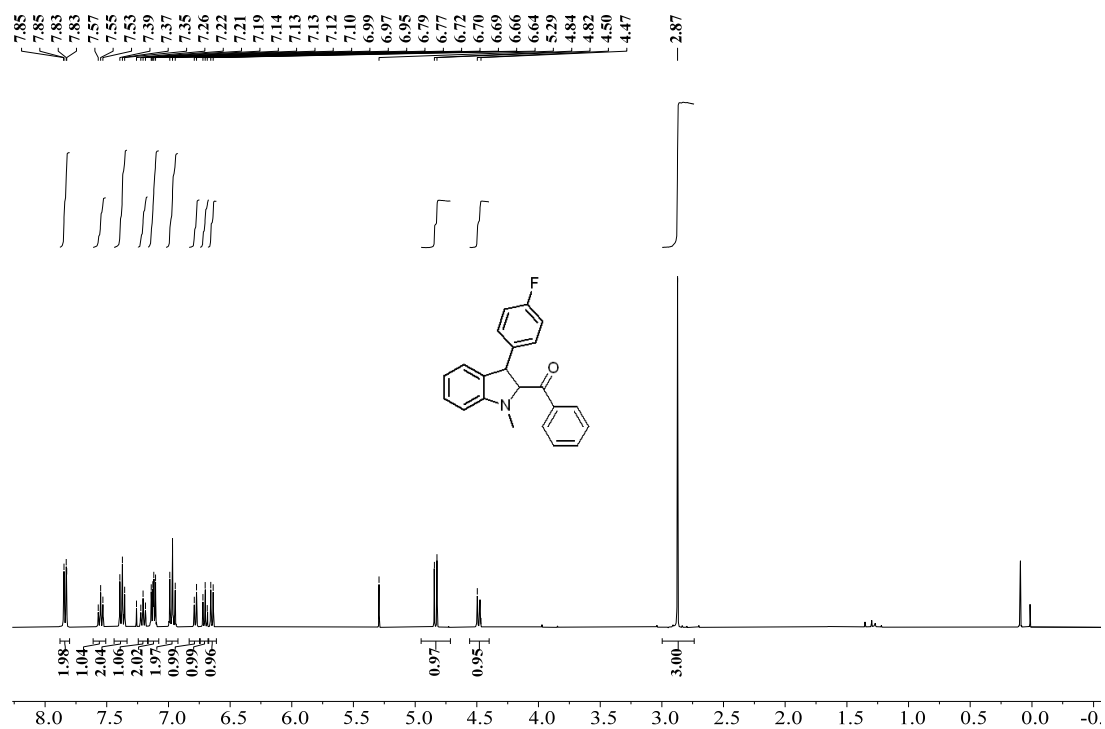


Figure S100. ^1H NMR spectrum of **3q** (CDCl_3 , 400 MHz, 23 $^\circ\text{C}$)

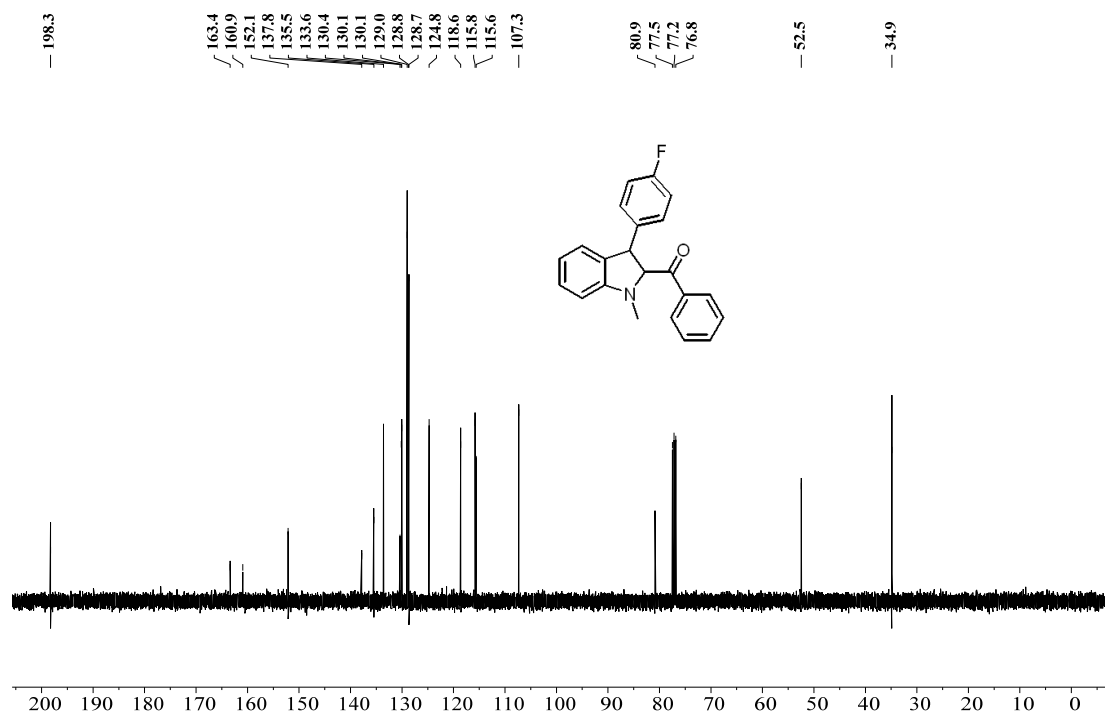


Figure S101 $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3q** (CDCl_3 , 101 MHz, 23 $^\circ\text{C}$)

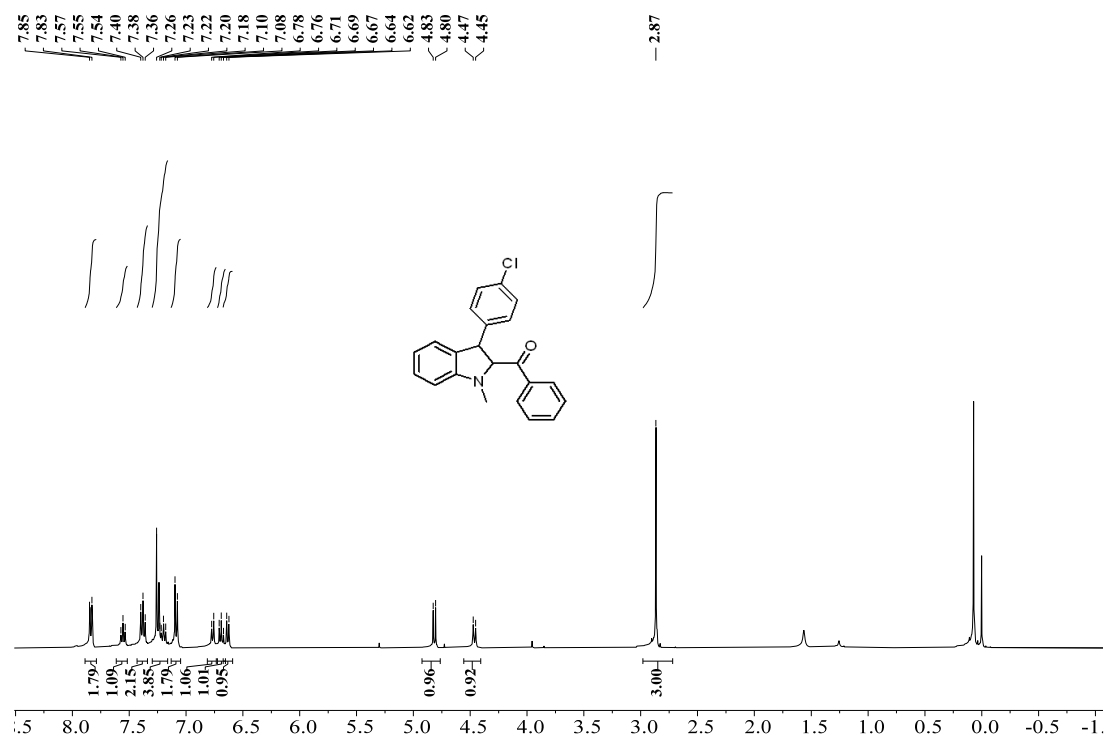


Figure S102. ^1H NMR spectrum of **3r** (CDCl_3 , 400 MHz, 23 °C)

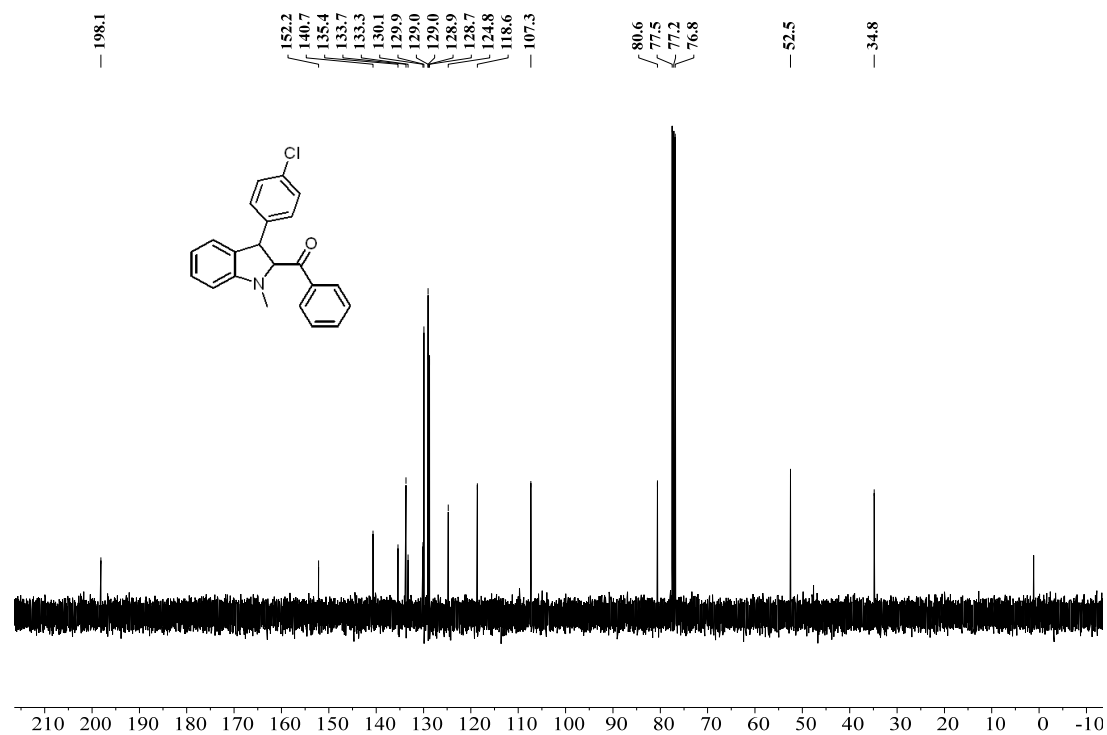


Figure S103. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3r** (CDCl_3 , 101 MHz, 23 °C)

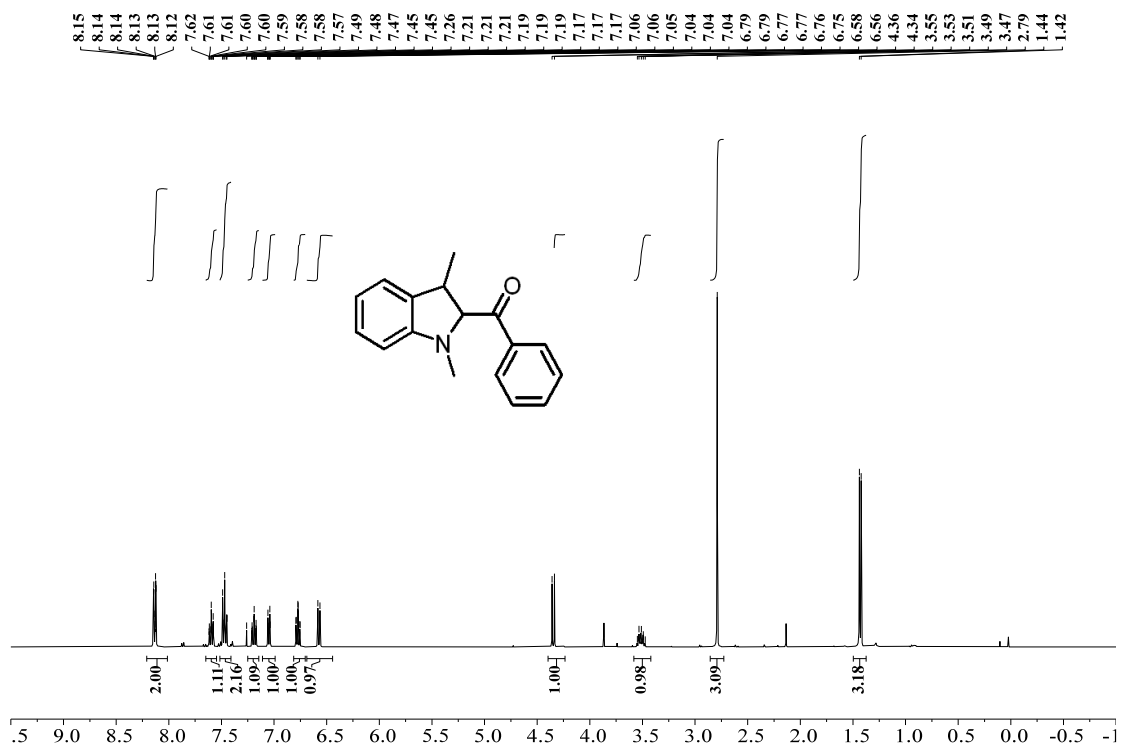


Figure S104. ¹H NMR spectrum of **3s** (CDCl₃, 400 MHz, 23 °C)

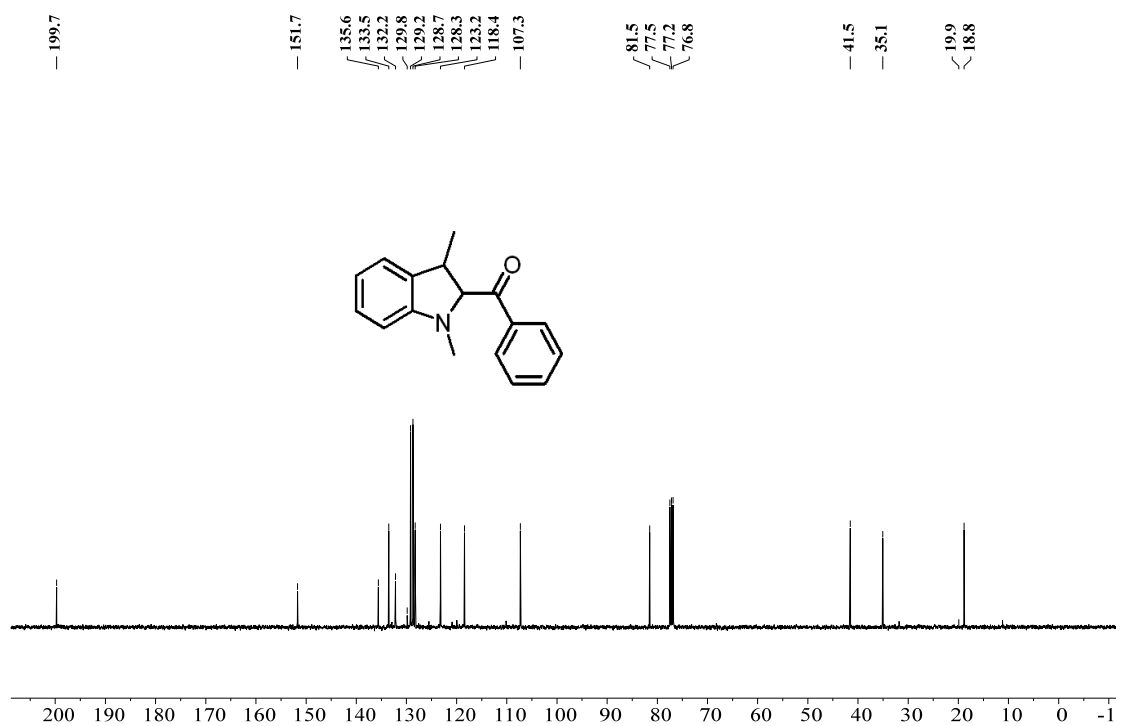


Figure S105. ¹³C{¹H} NMR spectrum of **3s** (CDCl₃, 101 MHz, 23 °C)

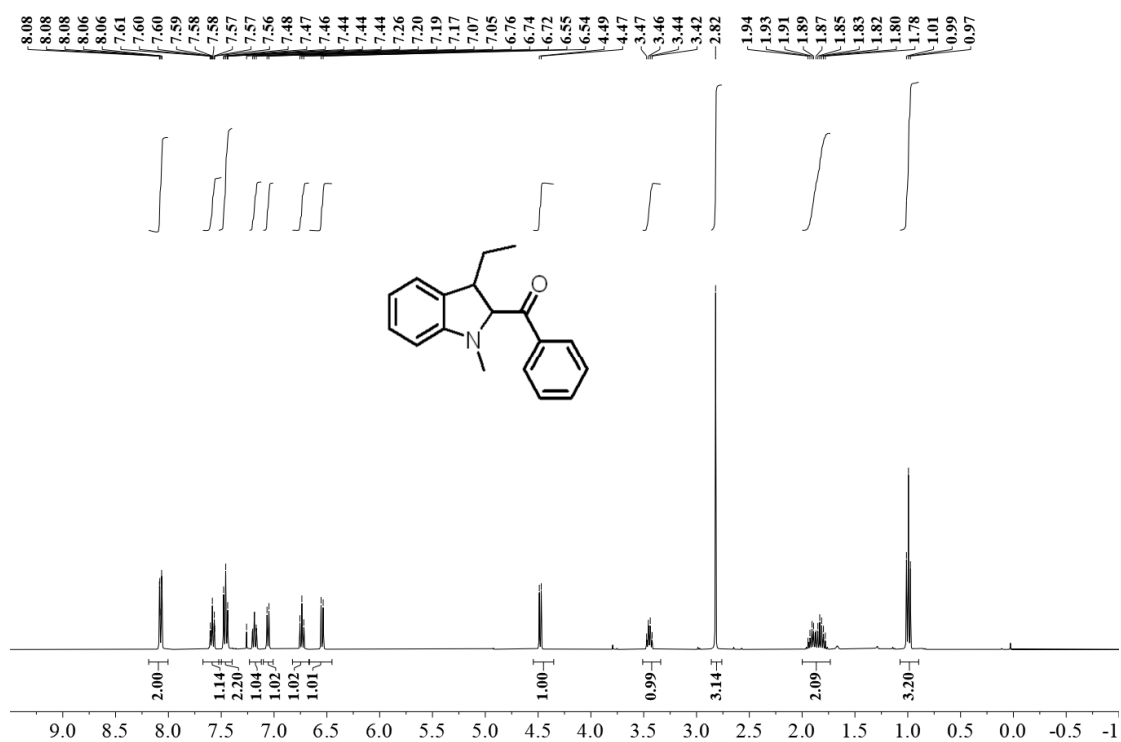


Figure S106. ^1H NMR spectrum of **3t** (CDCl_3 , 400 MHz, 23 °C)

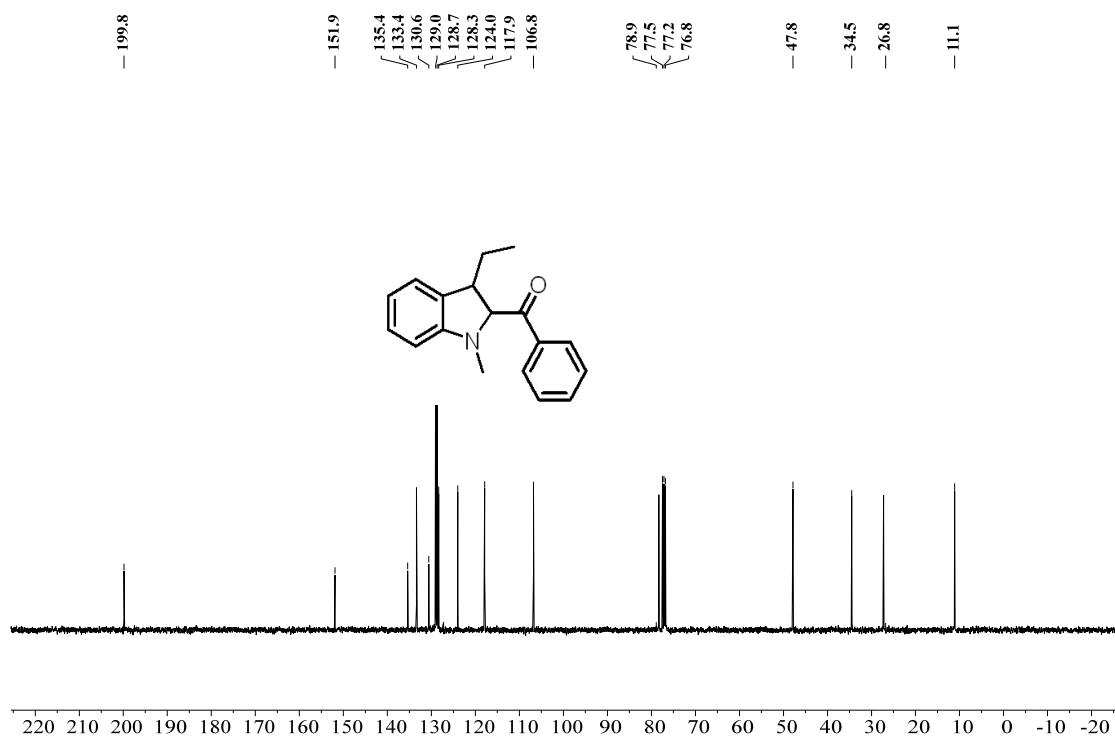


Figure S107. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3t** (CDCl_3 , 101 MHz, 23 °C)

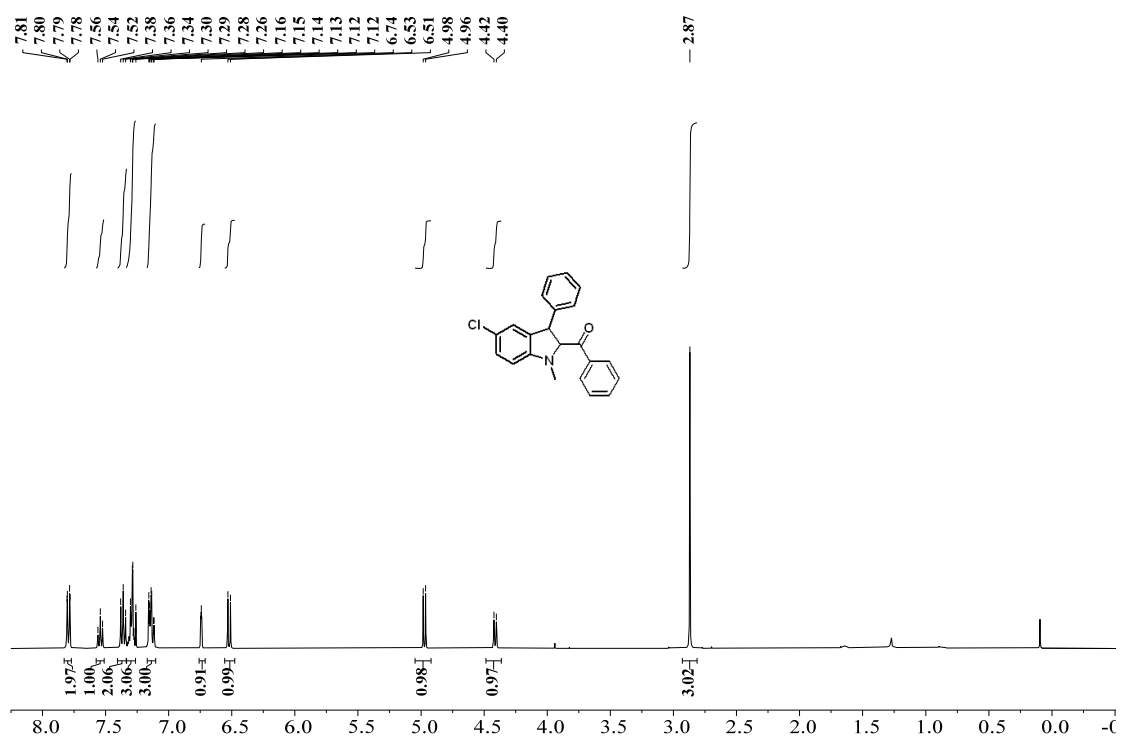


Figure S108. ^1H NMR spectrum of **3u** (CDCl_3 , 400 MHz, 23 °C)

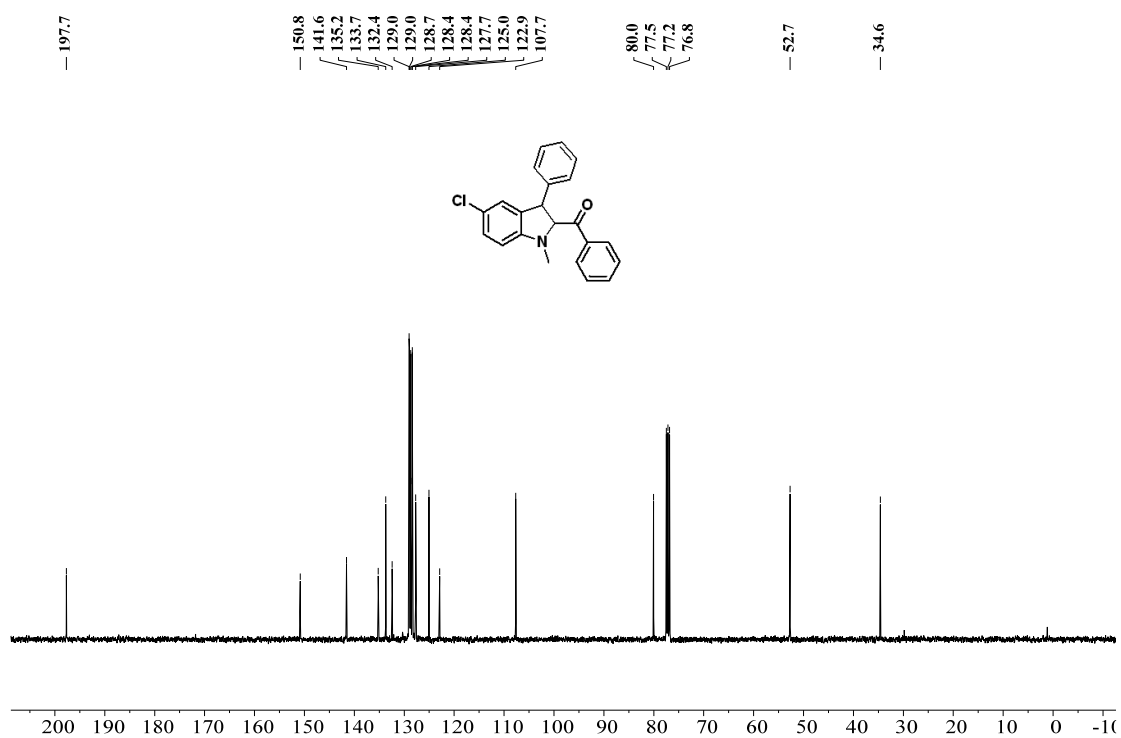


Figure S109. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3u** (CDCl_3 , 101 MHz, 23 °C)

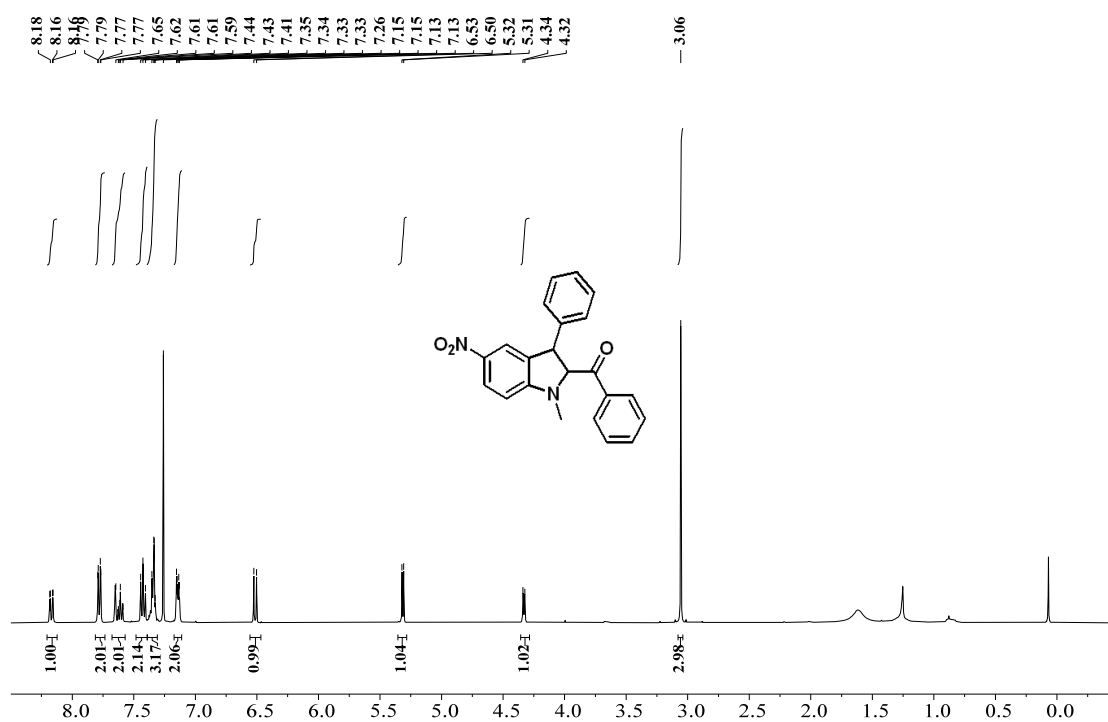


Figure S110. ^1H NMR spectrum of **3v** (CDCl_3 , 400 MHz, 23 °C)

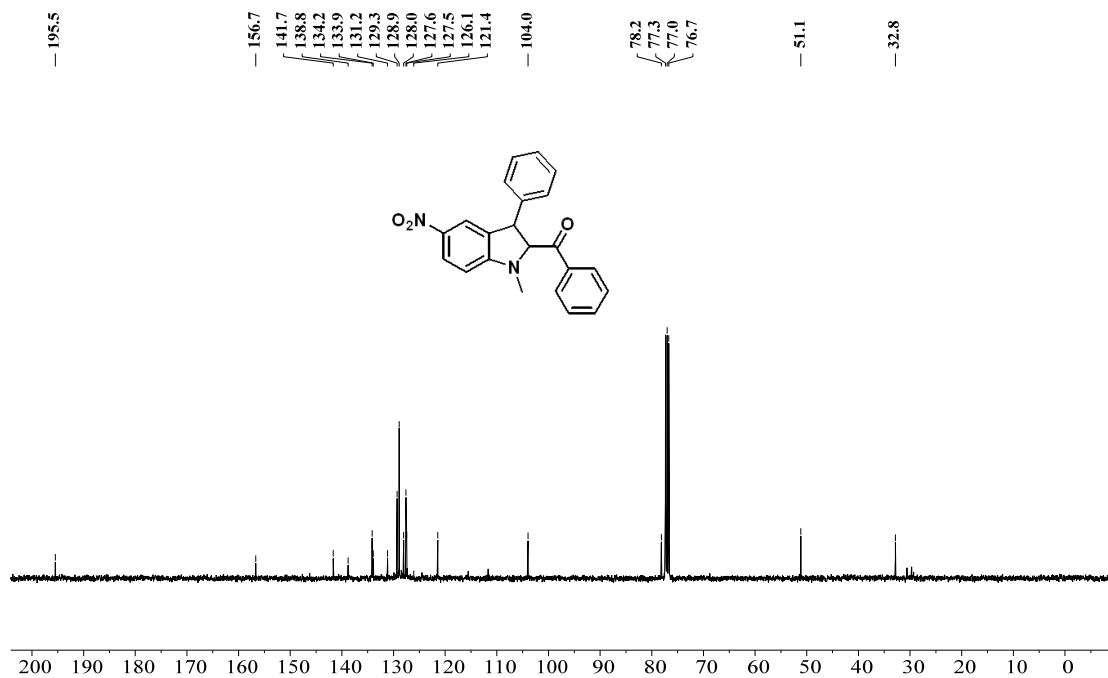


Figure S111. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3v** (CDCl_3 , 101 MHz, 23 °C)

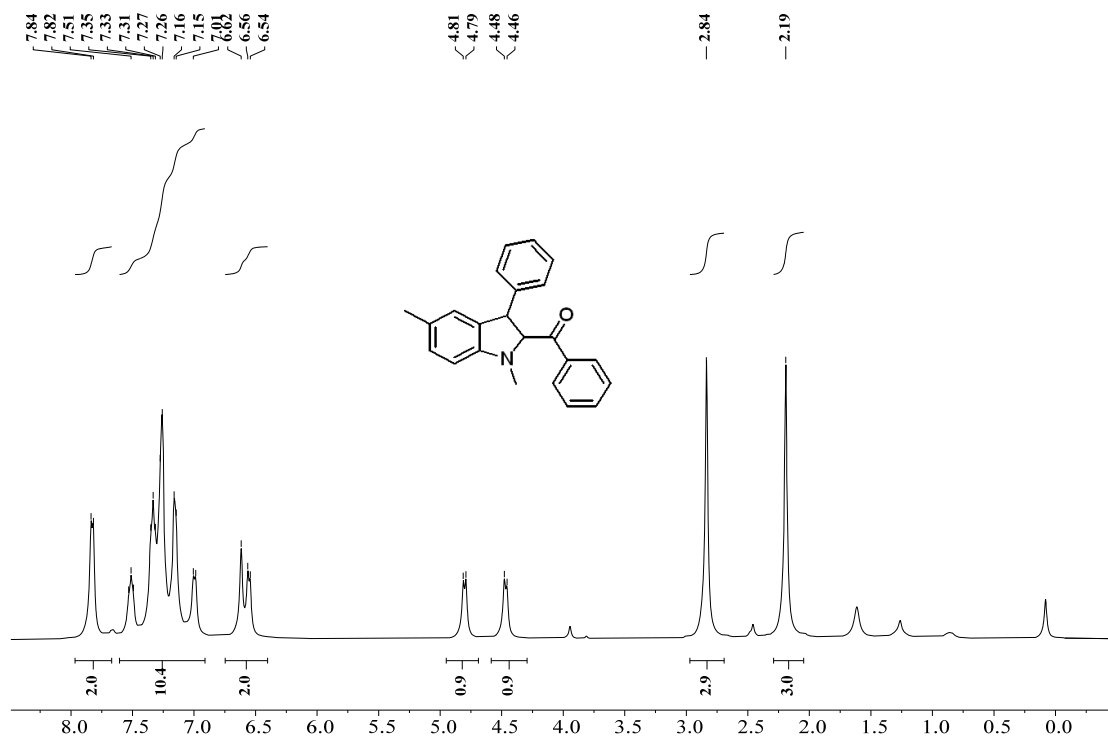


Figure S112. $^1\text{H NMR}$ spectrum of **3w** (CDCl_3 , 400 MHz, 23 °C)

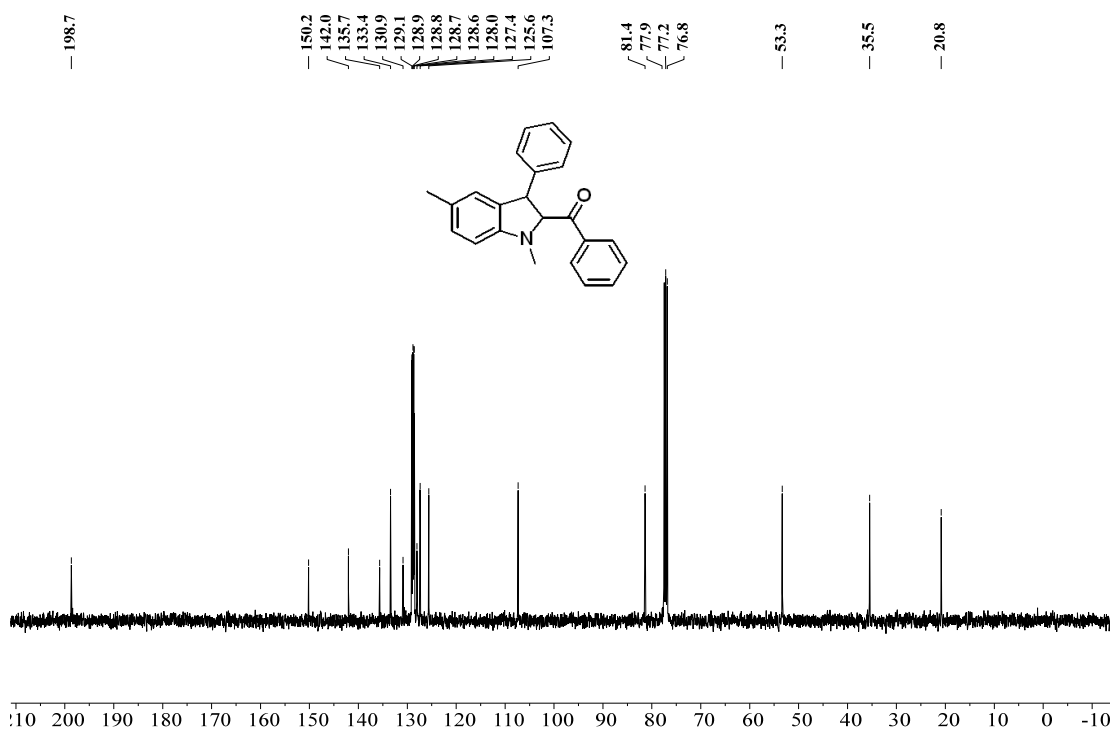


Figure S113. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3w** (CDCl_3 , 101 MHz, 23 °C)

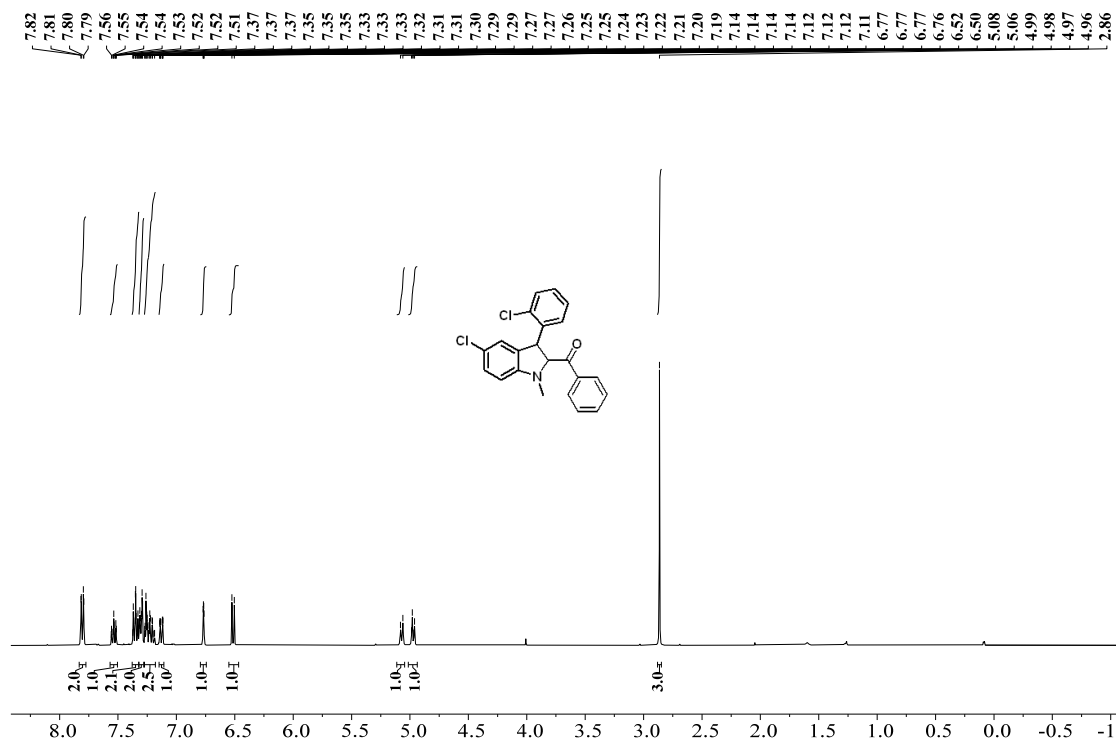


Figure S114. ^1H NMR spectrum of **3x** (CDCl_3 , 400 MHz, 23 °C)

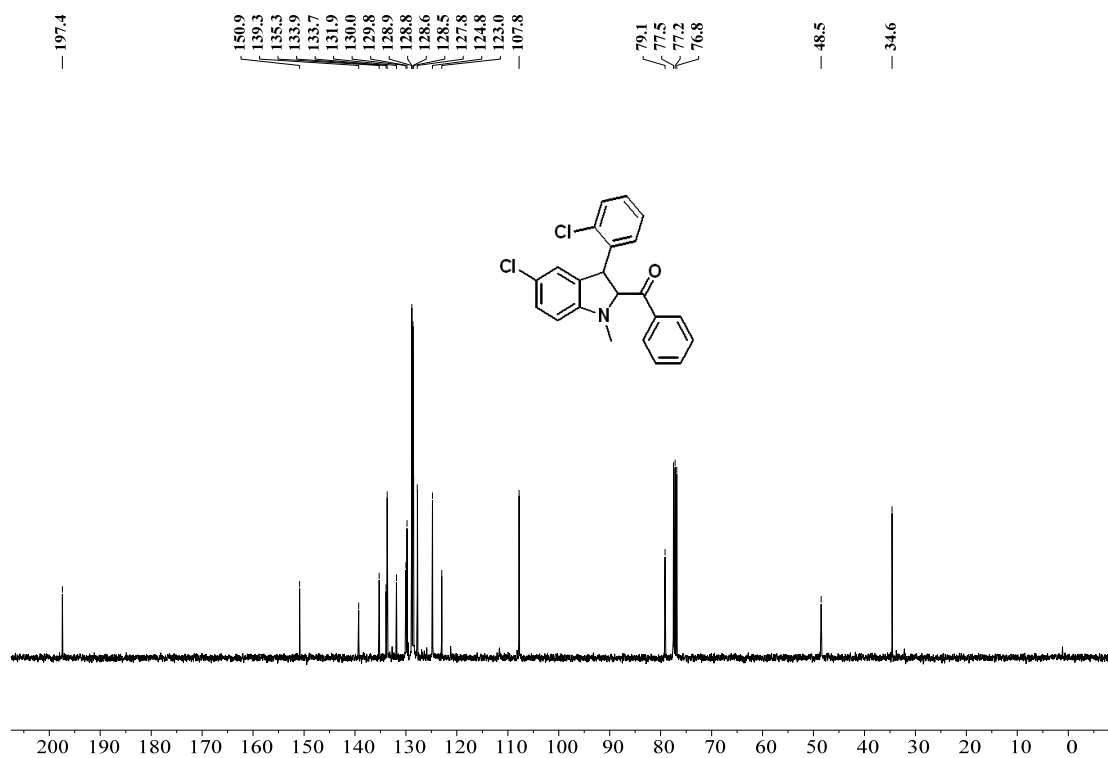


Figure S115. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3x** (CDCl_3 , 101 MHz, 23 °C)

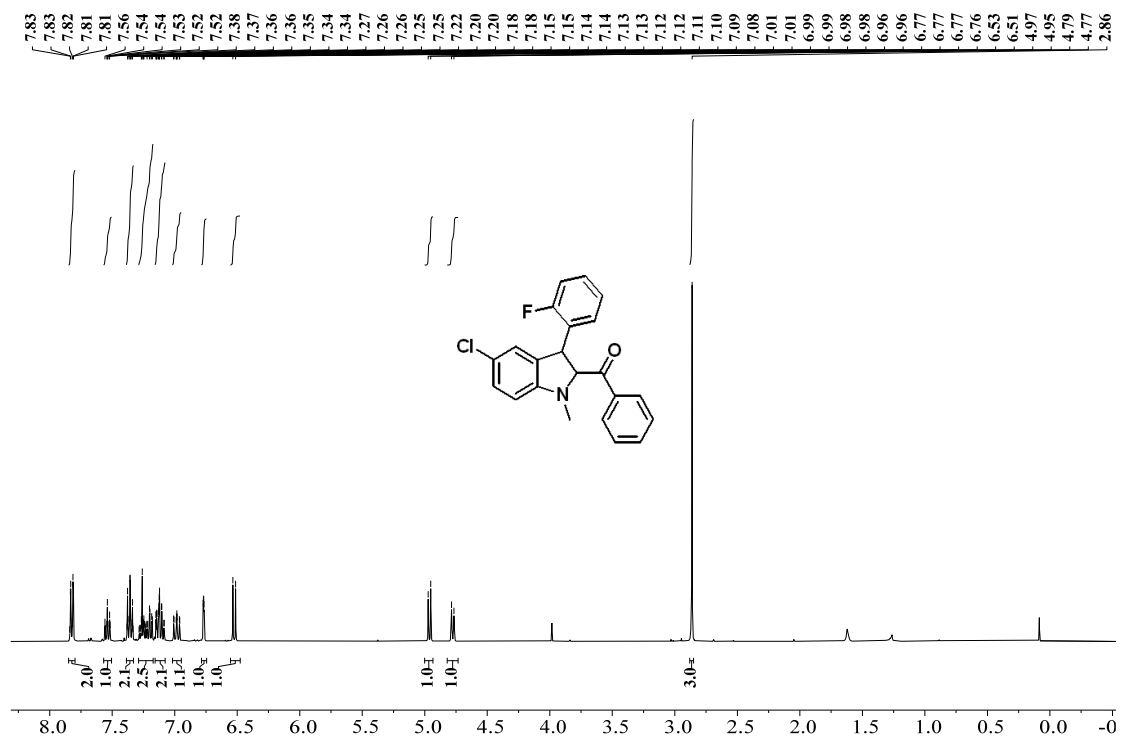


Figure S116. ¹H NMR spectrum of **3y** (CDCl₃, 400 MHz, 23 °C)

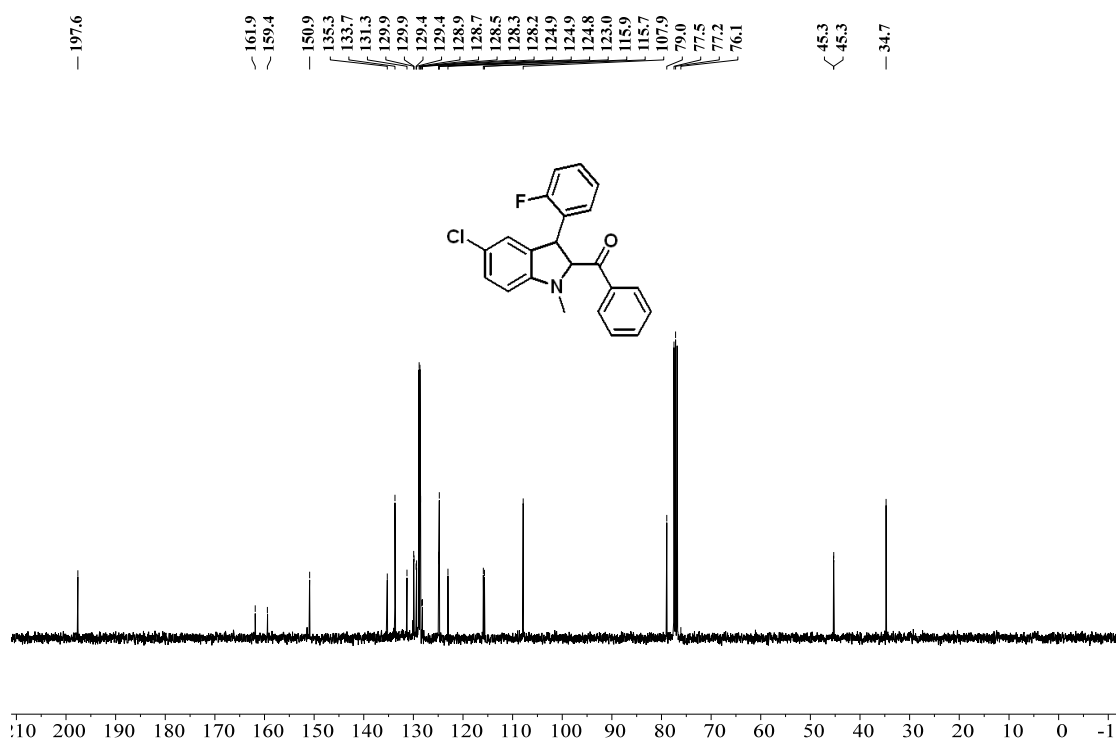


Figure S117. ¹³C{¹H} NMR spectrum of **3y** (CDCl₃, 101 MHz, 23 °C)

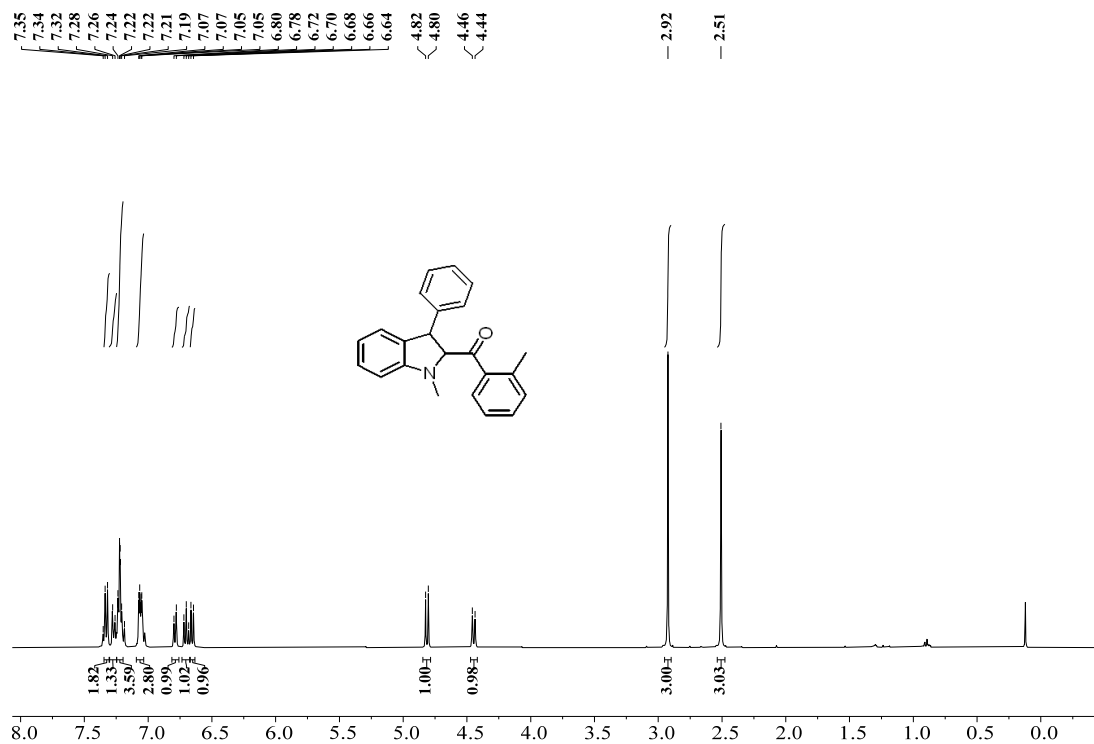


Figure S118. ^1H NMR spectrum of **3z** (CDCl_3 , 400 MHz, 23 °C)

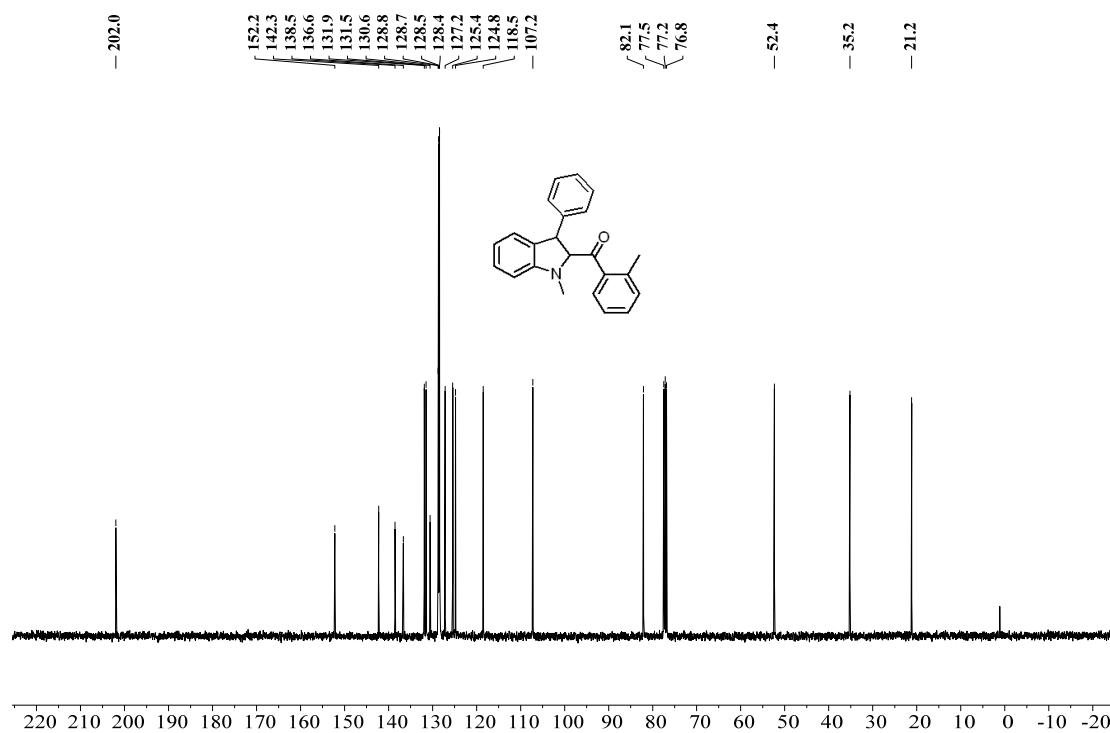


Figure S119. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3z** (CDCl_3 , 101 MHz, 23 °C)

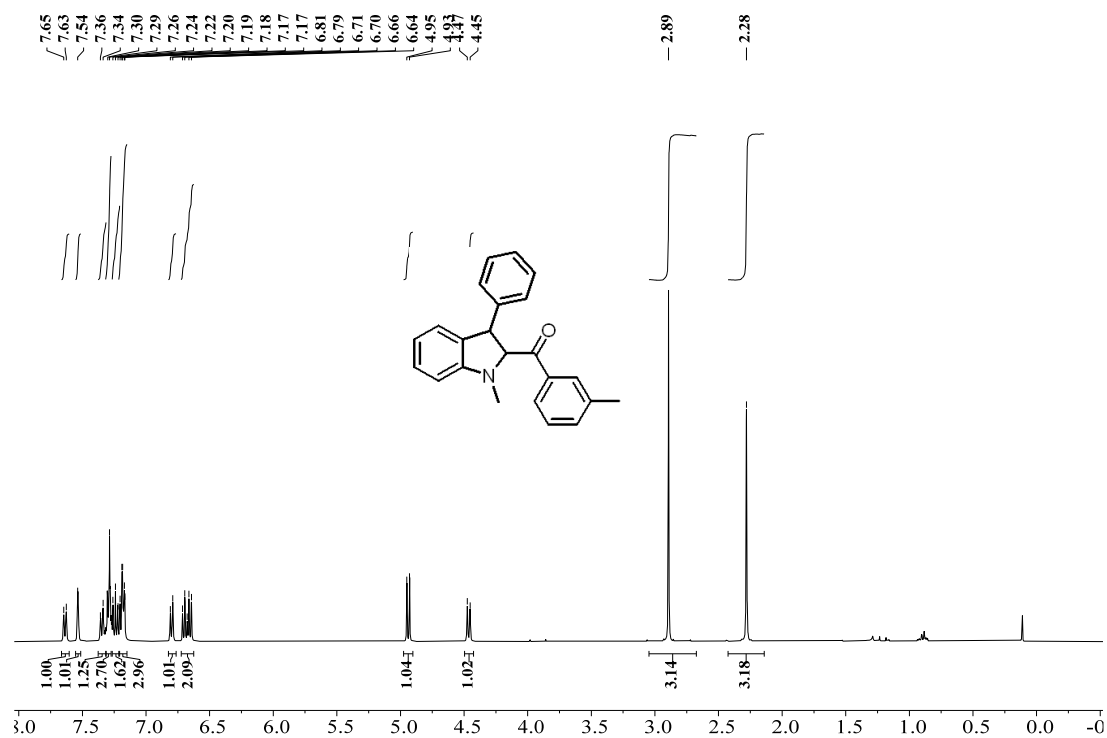


Figure S120. ^1H NMR spectrum of **3aa** (CDCl_3 , 400 MHz, 23 °C)

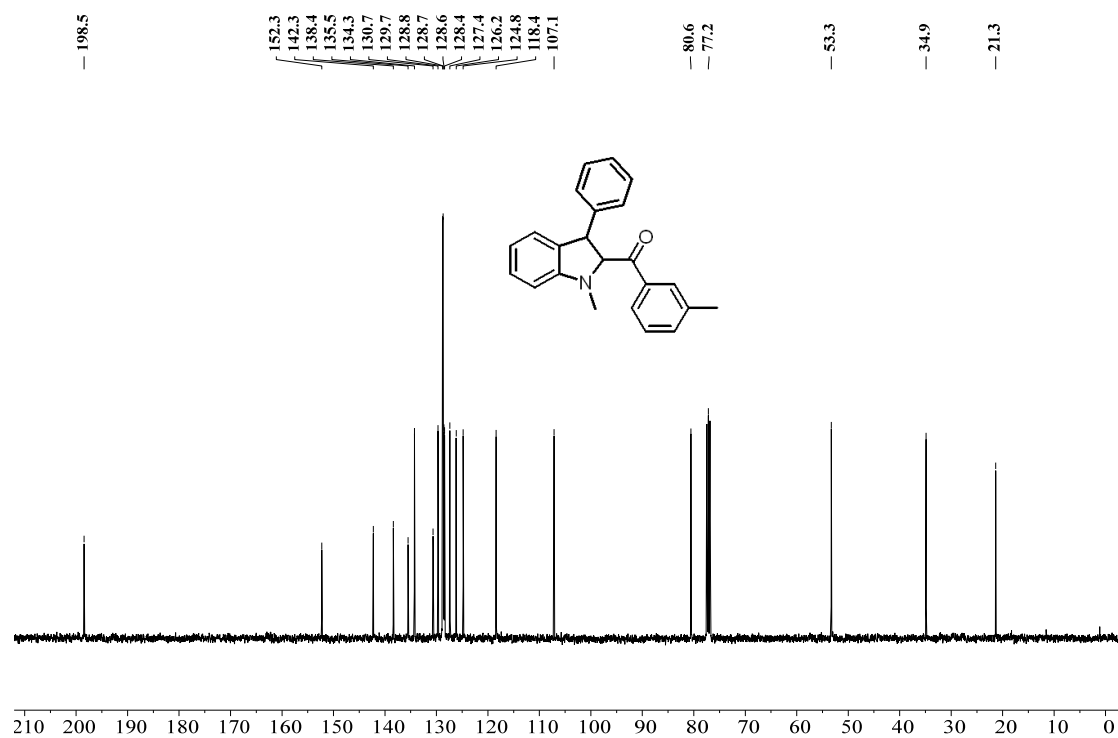


Figure S121. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3aa** (CDCl_3 , 101 MHz, 23 °C)

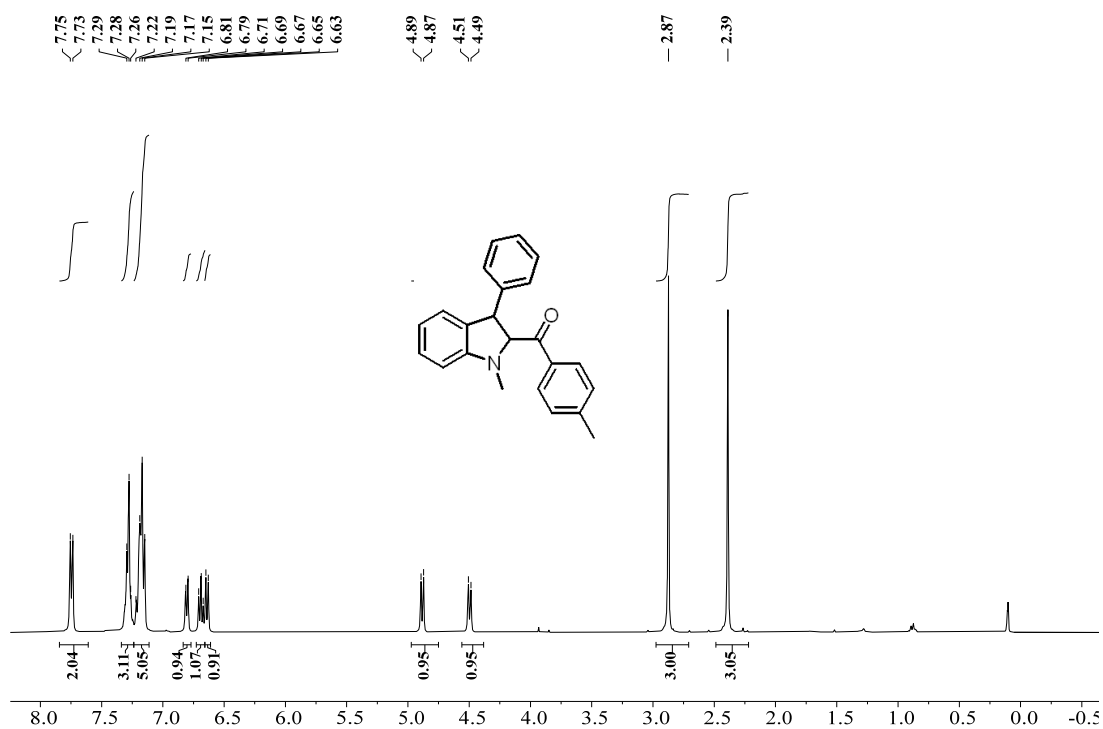


Figure S122. ^1H NMR spectrum of **3ab** (CDCl_3 , 400 MHz, 23 °C)

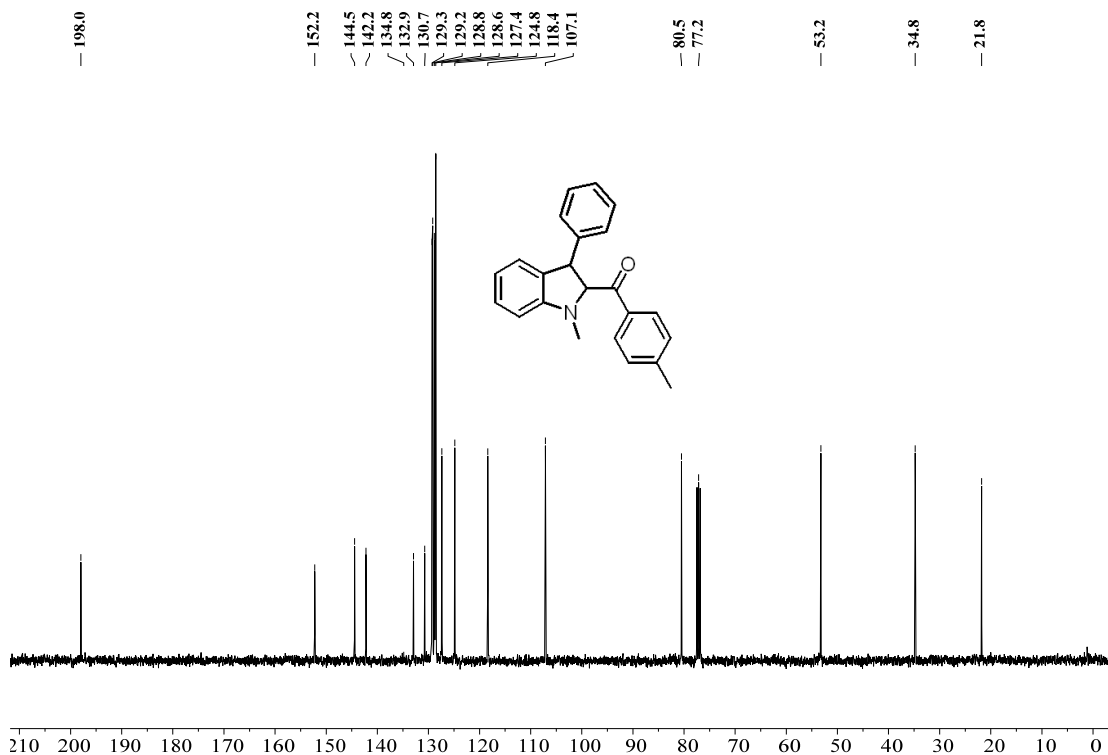


Figure S123. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3ab** (CDCl_3 , 101 MHz, 23 °C)

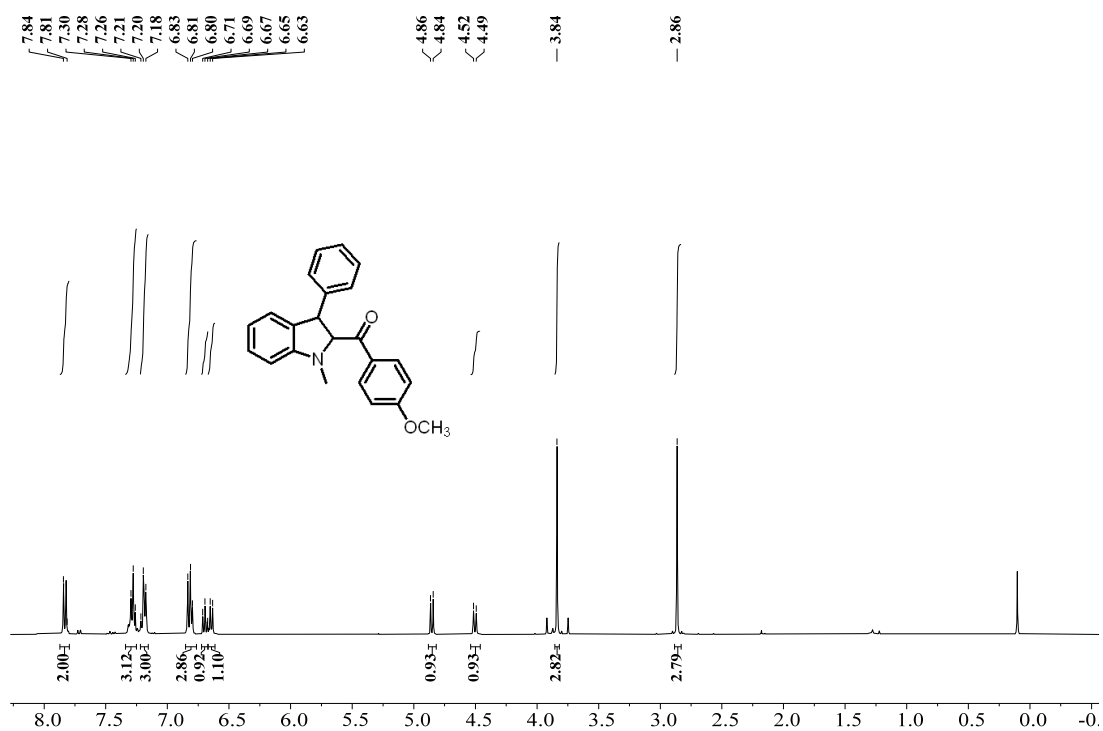


Figure S124. ^1H NMR spectrum of **3ac** (CDCl_3 , 400 MHz, 23 °C)

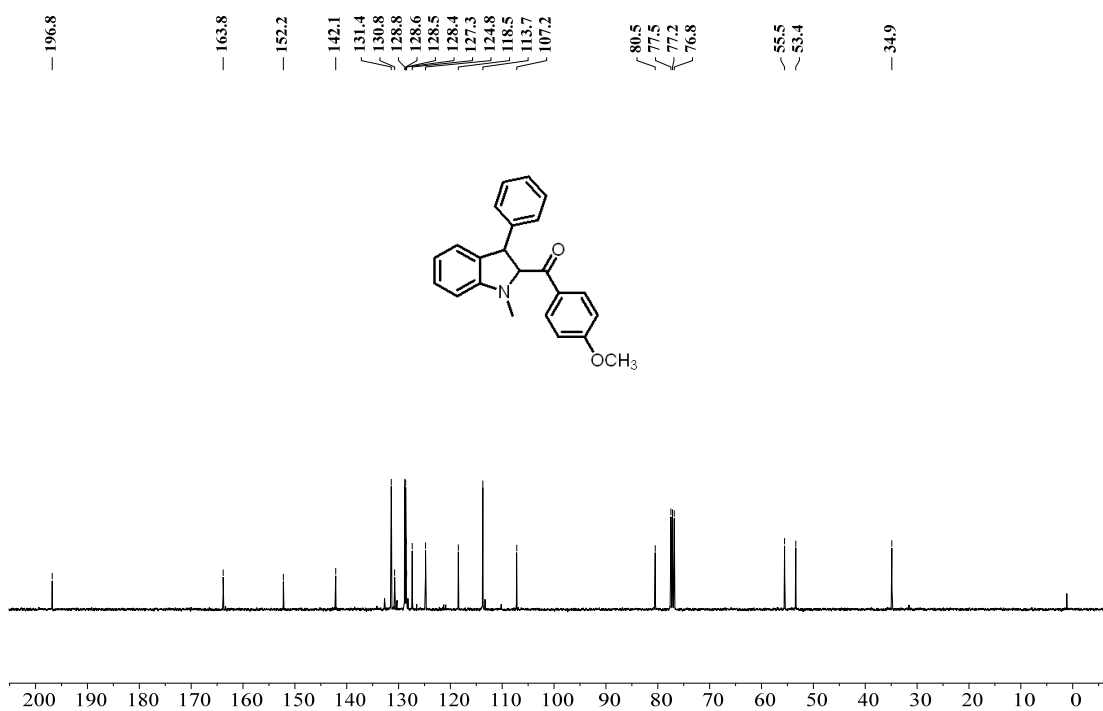


Figure S125. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3ac** (CDCl_3 , 101 MHz, 23 °C)

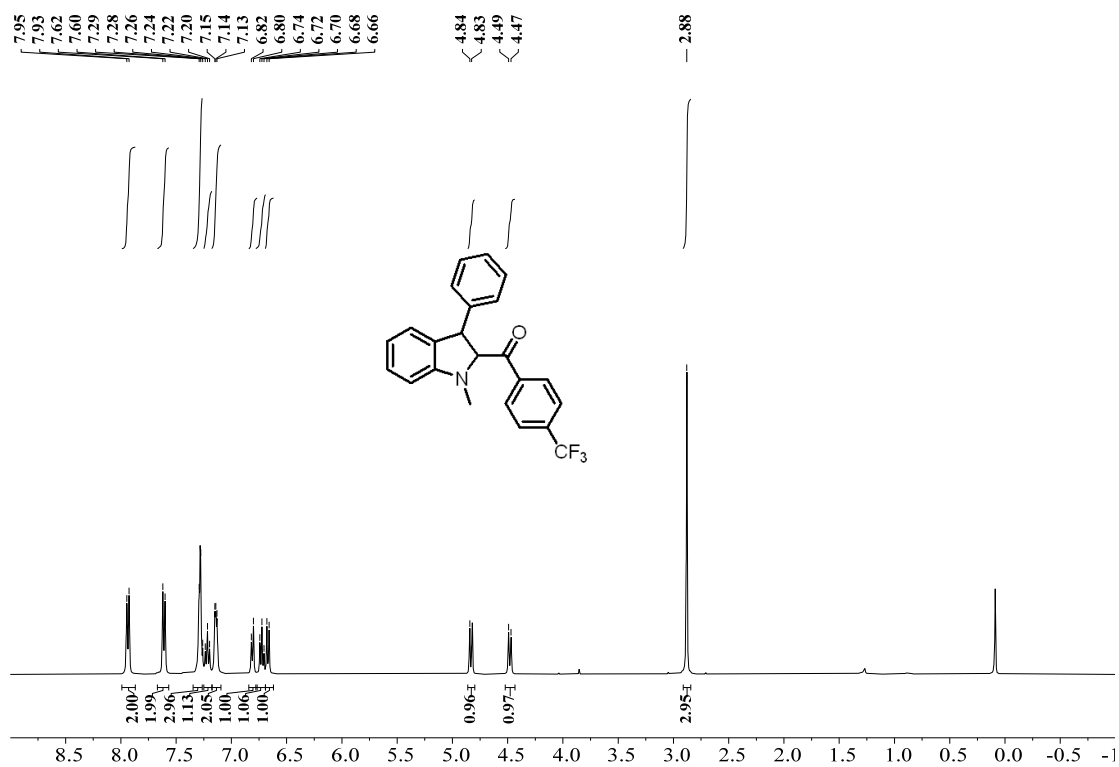


Figure S126. ^1H NMR spectrum of **3ad** (CDCl_3 , 400 MHz, 23 °C)

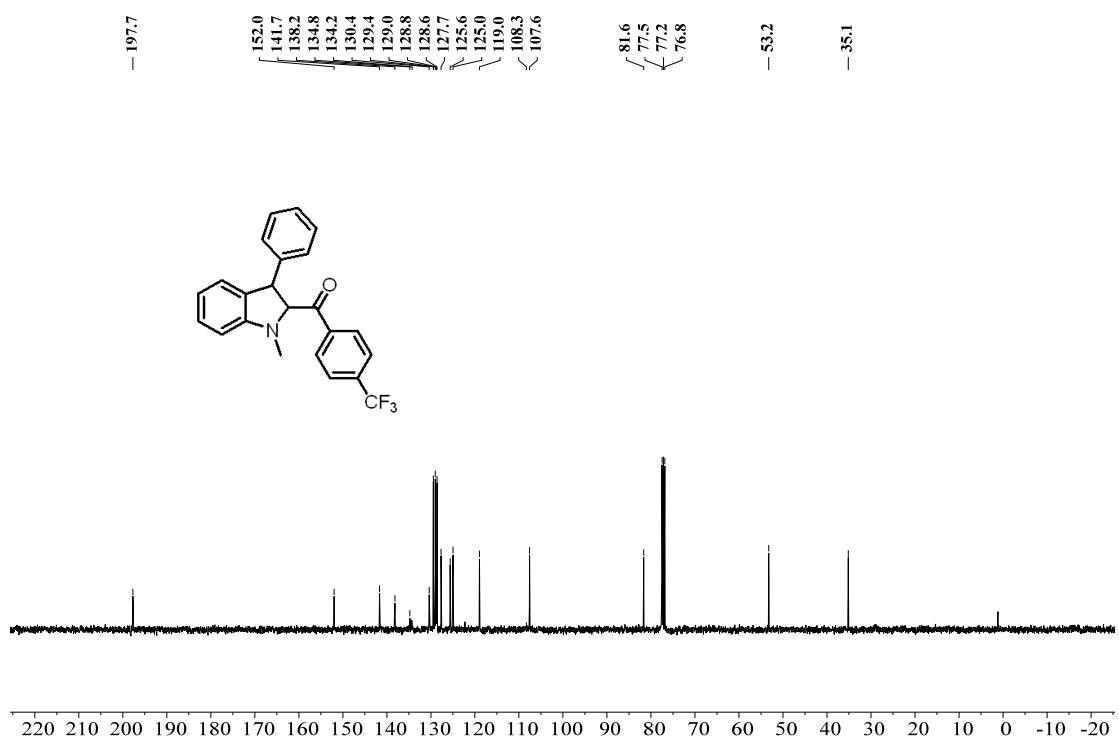


Figure S127. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3ad** (CDCl_3 , 101 MHz, 23 °C)

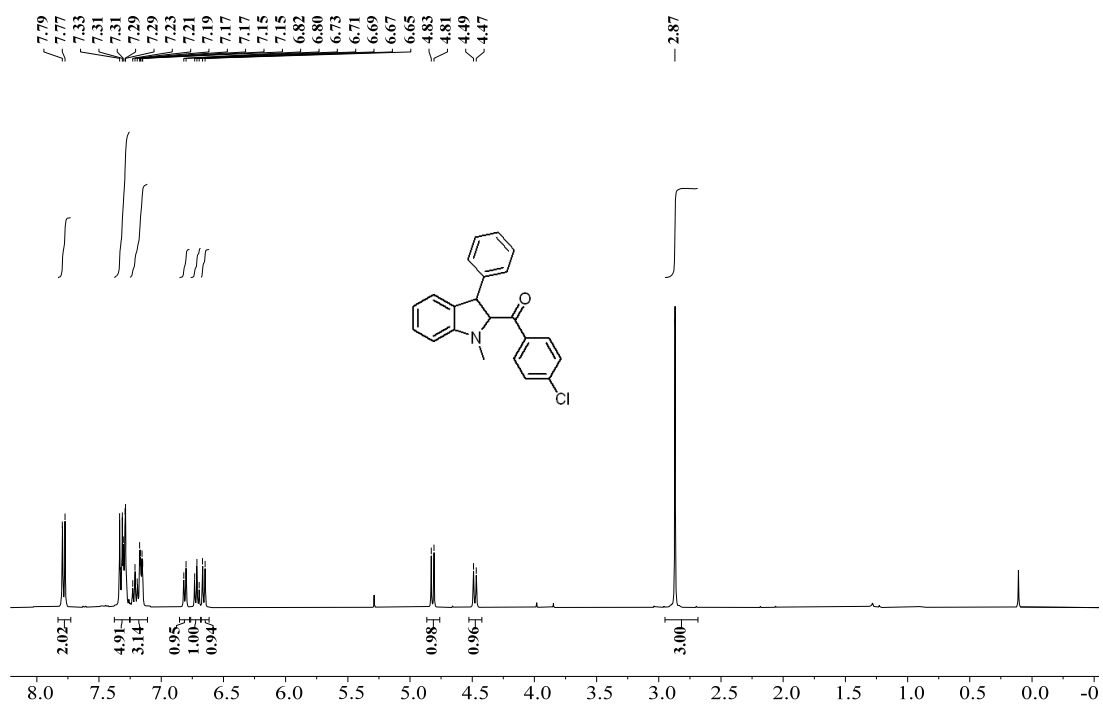


Figure S128. ^1H NMR spectrum of **3ae** (CDCl_3 , 400 MHz, 23 °C)

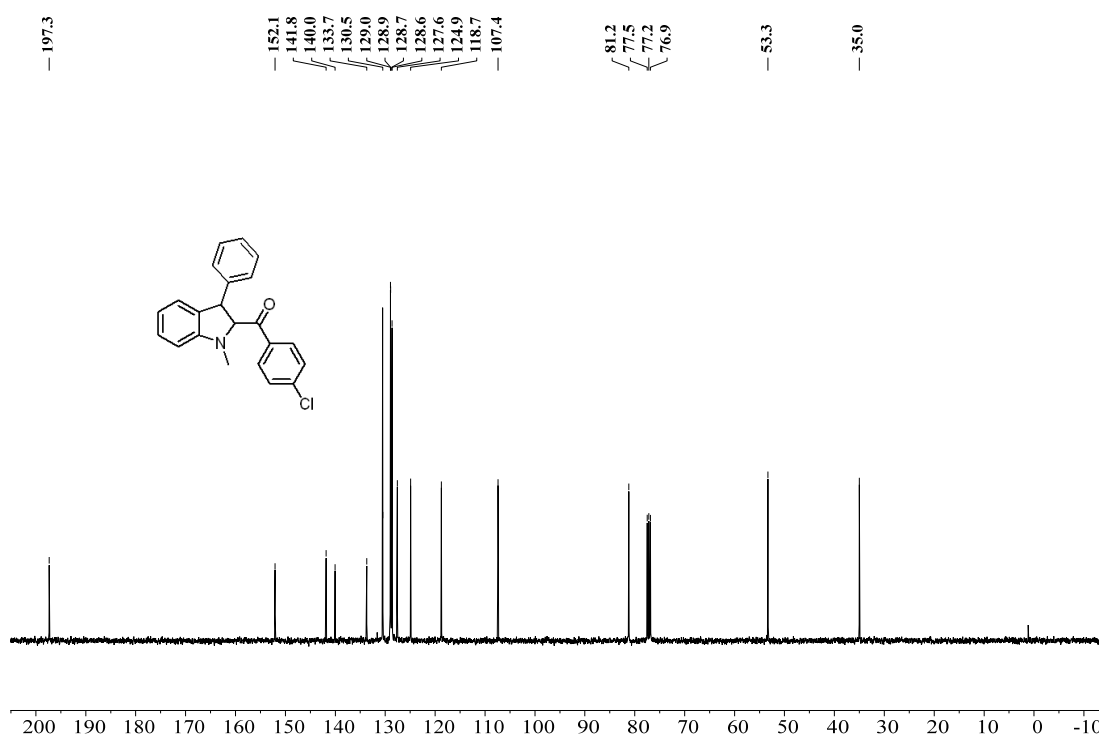


Figure S129. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3ae** (CDCl_3 , 101 MHz, 23 °C)

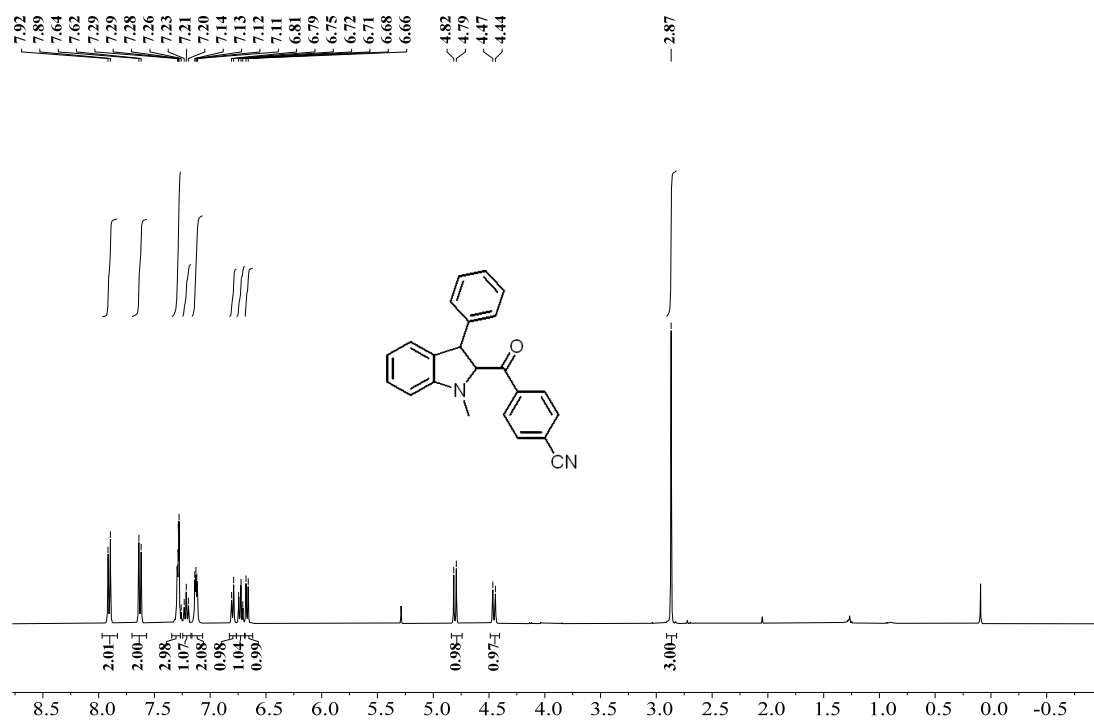


Figure S130. ¹H NMR spectrum of **3af** (CDCl₃, 400 MHz, 23 °C)

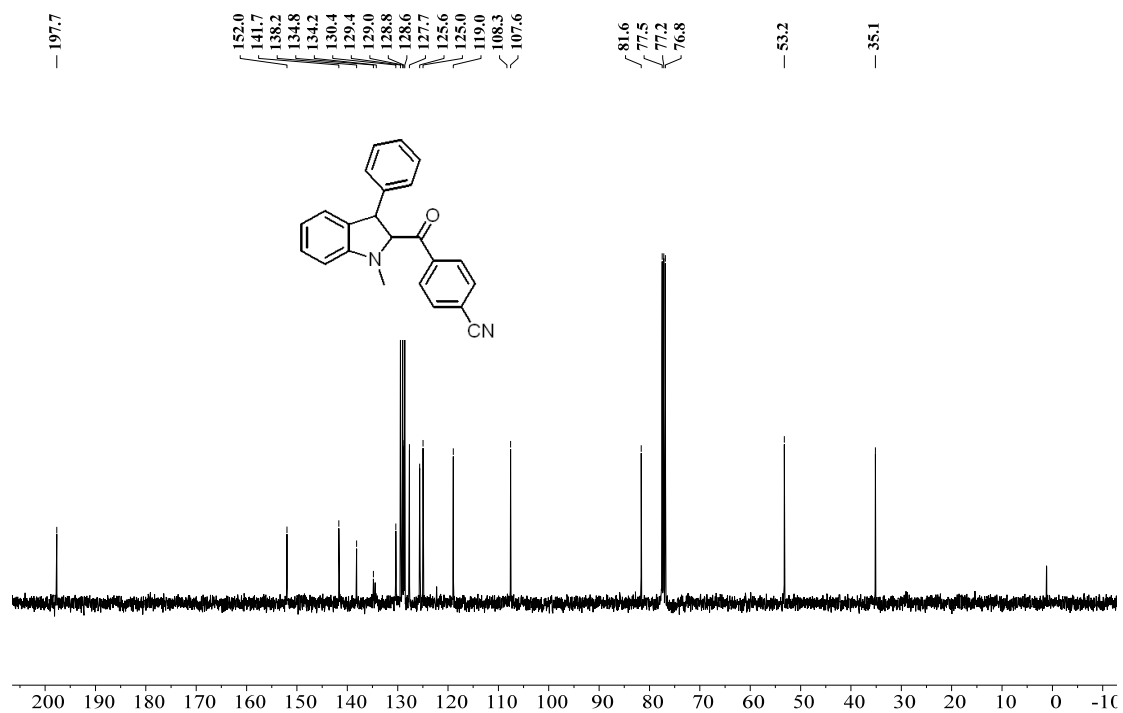


Figure S131. ¹³C{¹H} NMR spectrum of **3af** (CDCl₃, 101 MHz, 23 °C)

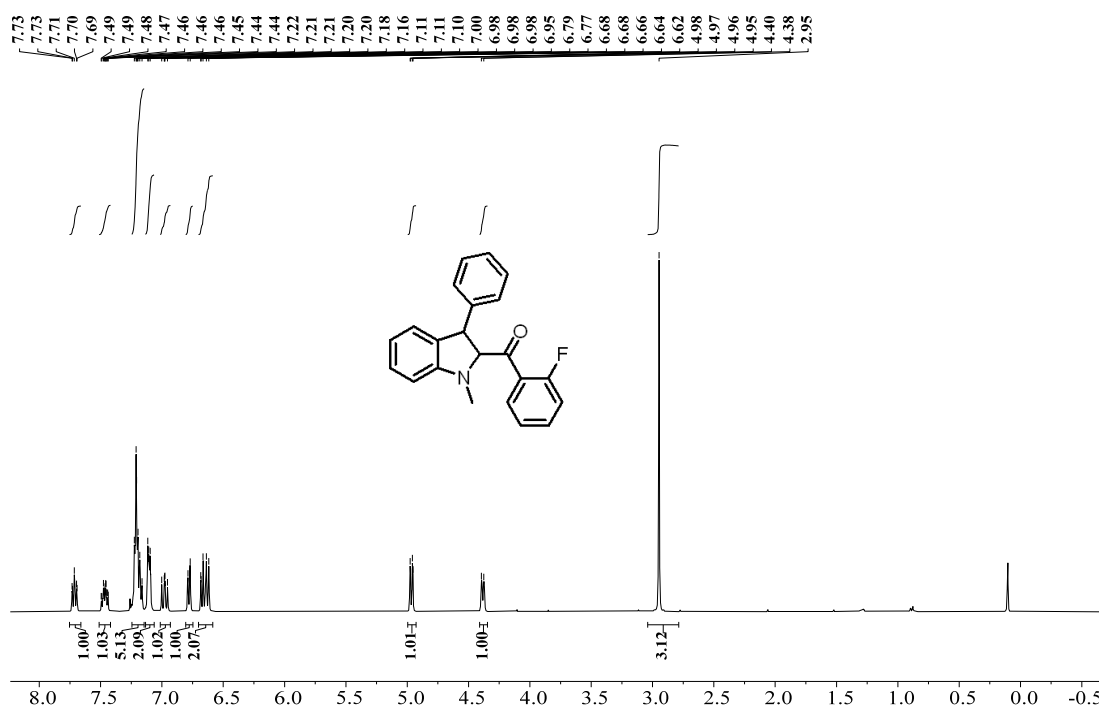


Figure S132. ¹H NMR spectrum of **3ag** (CDCl₃, 400 MHz, 23 °C)

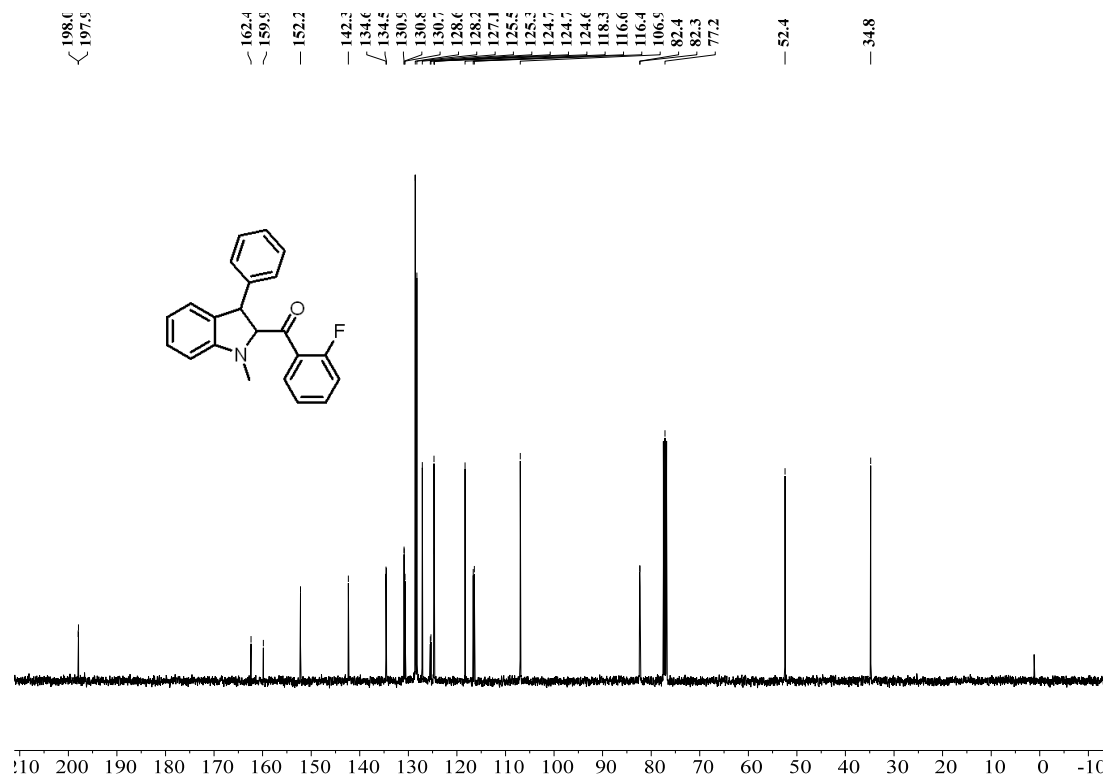


Figure S133. ¹³C{¹H} NMR spectrum of **3ag** (CDCl₃, 101 MHz, 23 °C)

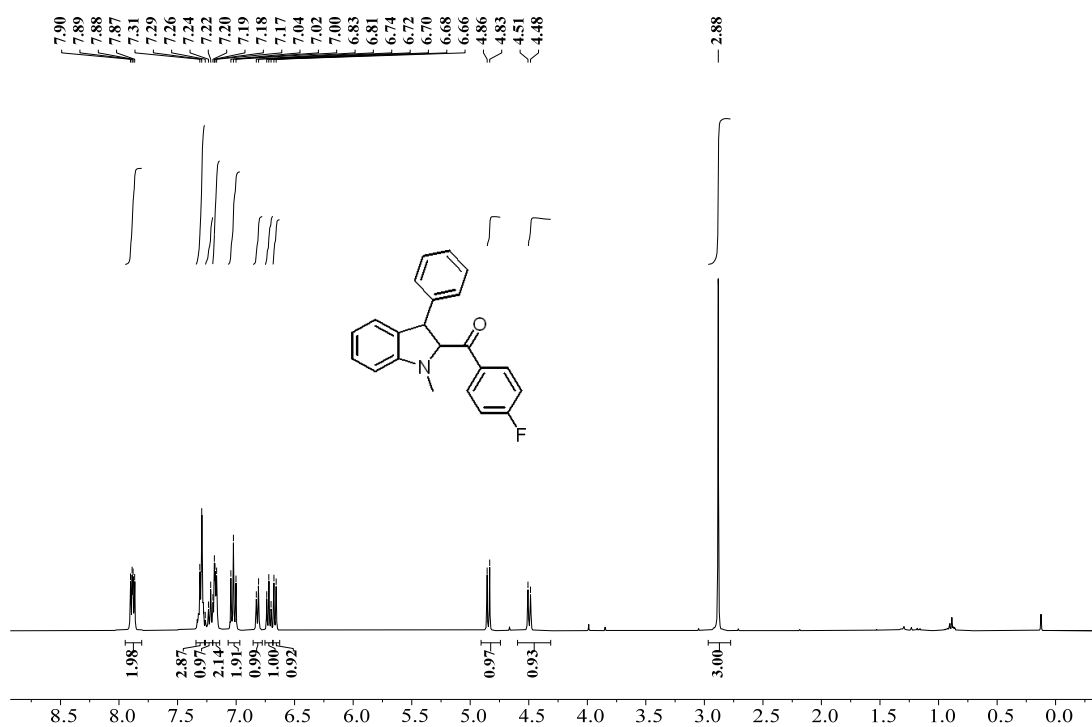


Figure S134. ^1H NMR spectrum of **3ah** (CDCl_3 , 400 MHz, 23 °C)

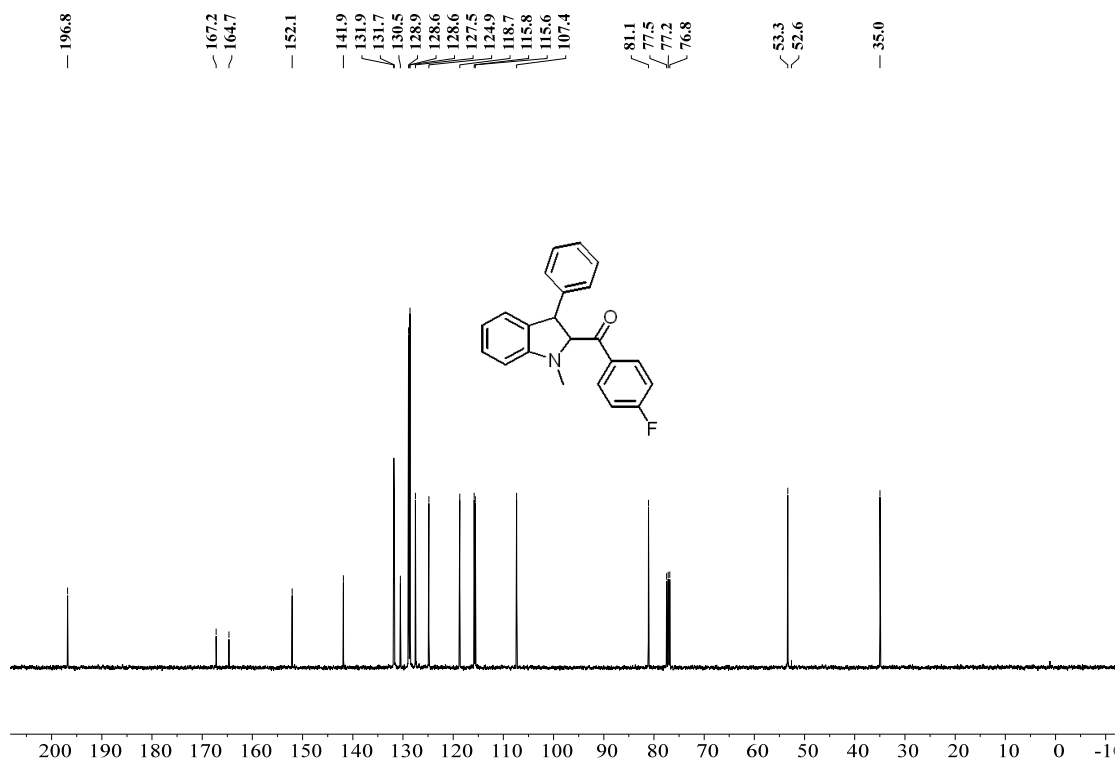


Figure S135. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3ah** (CDCl_3 , 101 MHz, 23 °C)

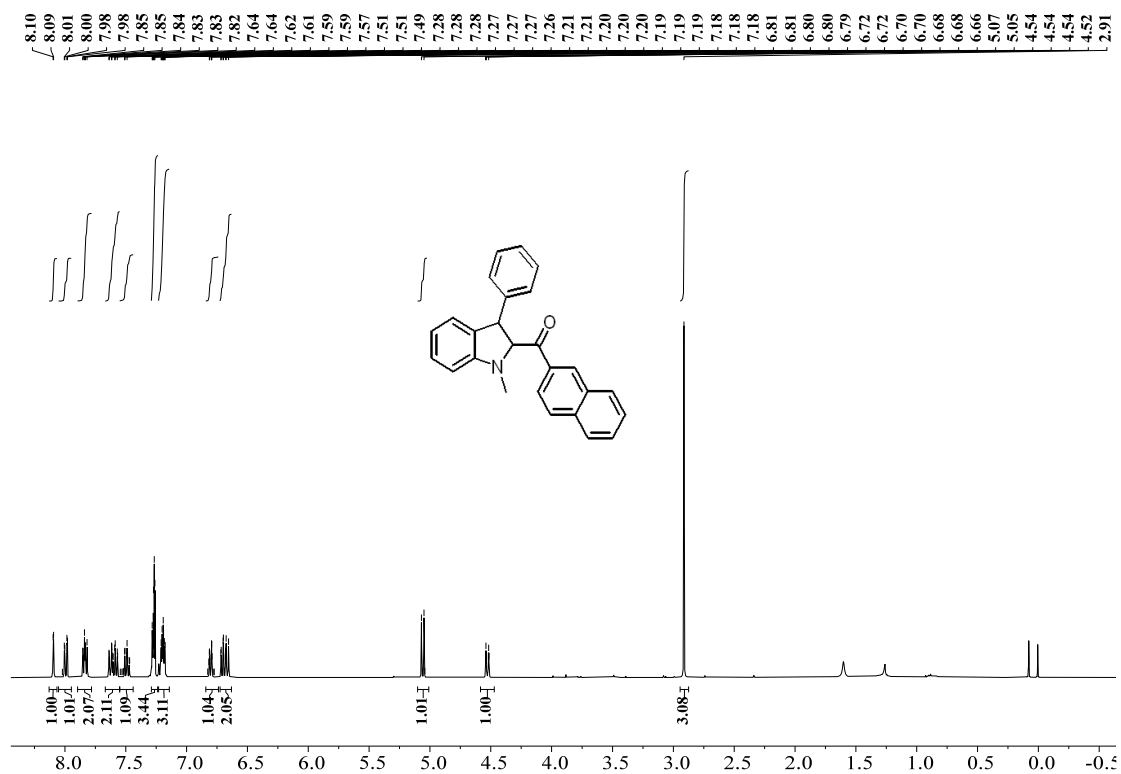


Figure S136. ^1H NMR spectrum of **3ai** (CDCl_3 , 400 MHz, 23 °C)

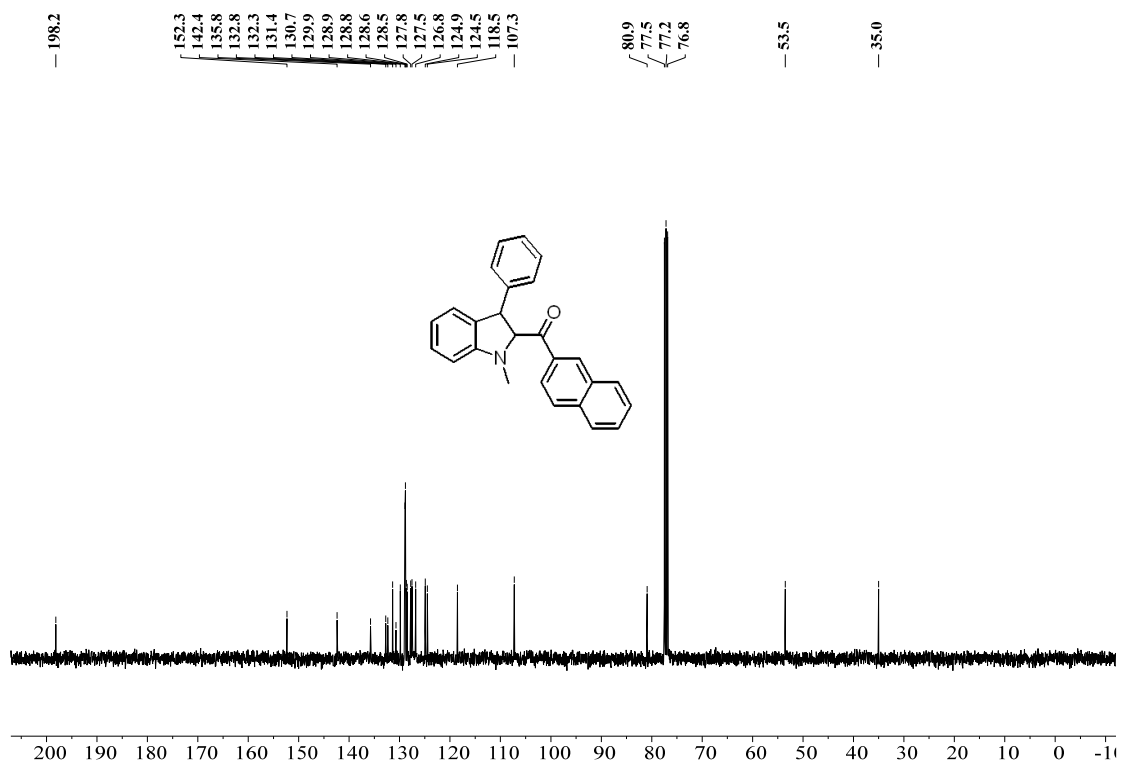
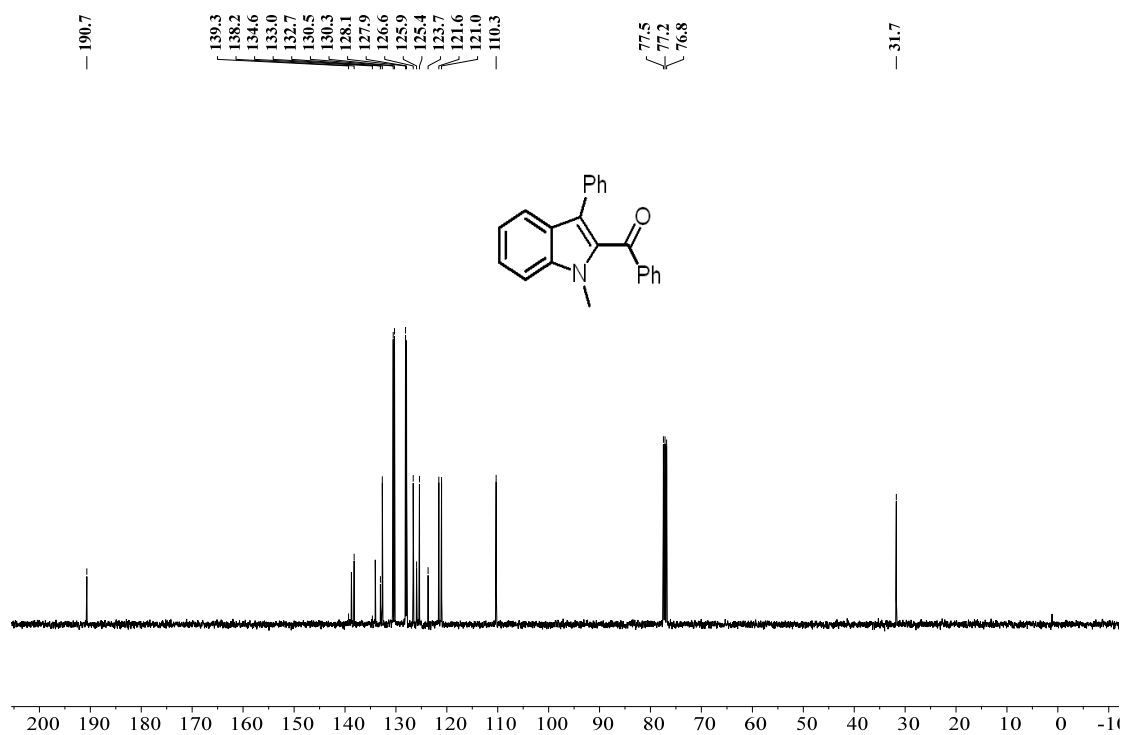
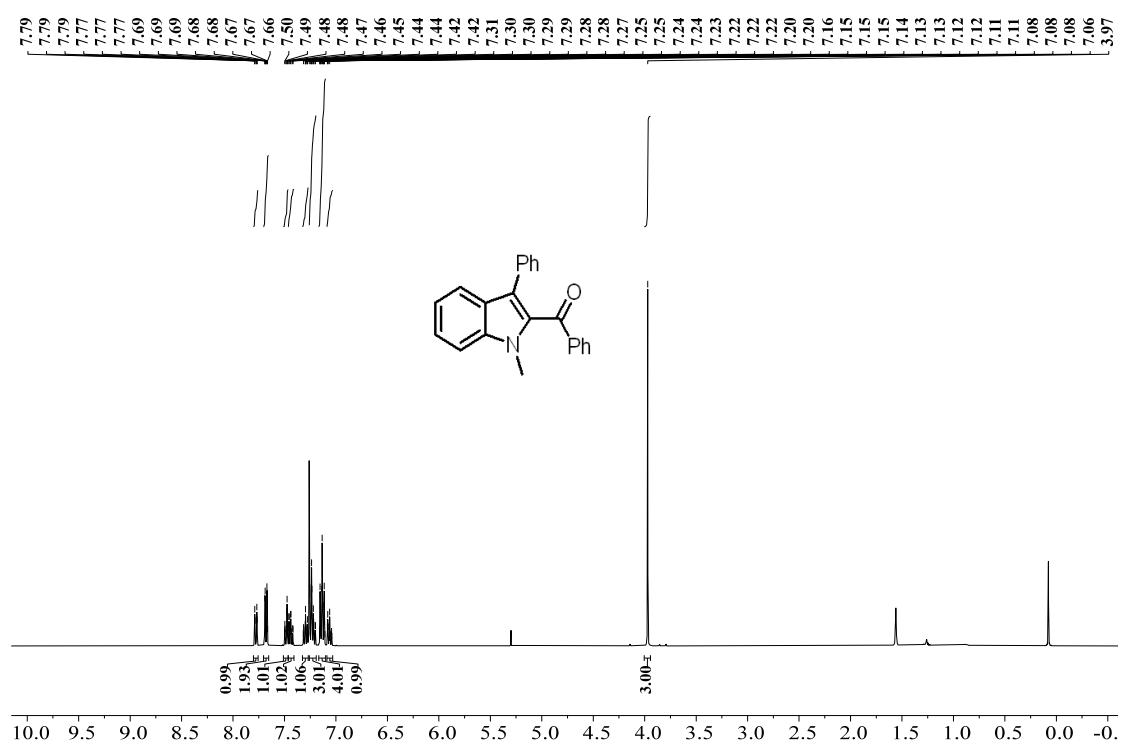


Figure S137. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3ai** (CDCl_3 , 101 MHz, 23 °C)



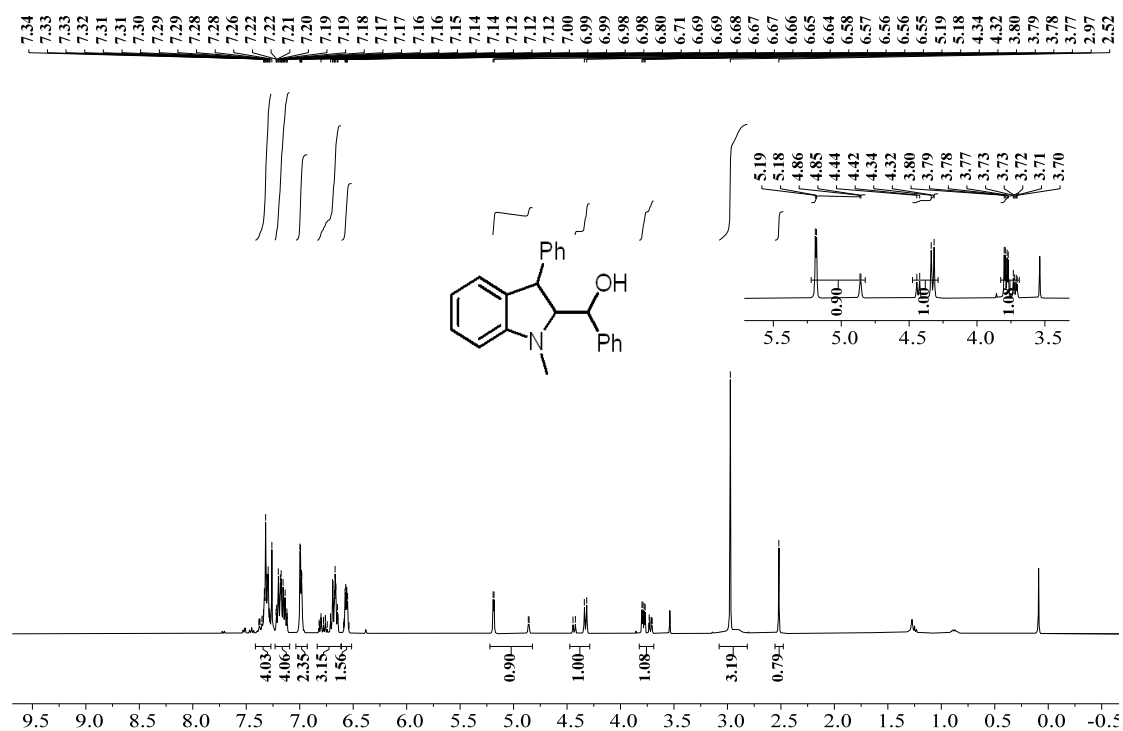


Figure S140. ^1H NMR spectrum of **5** (CDCl_3 , 400 MHz, 23 °C)

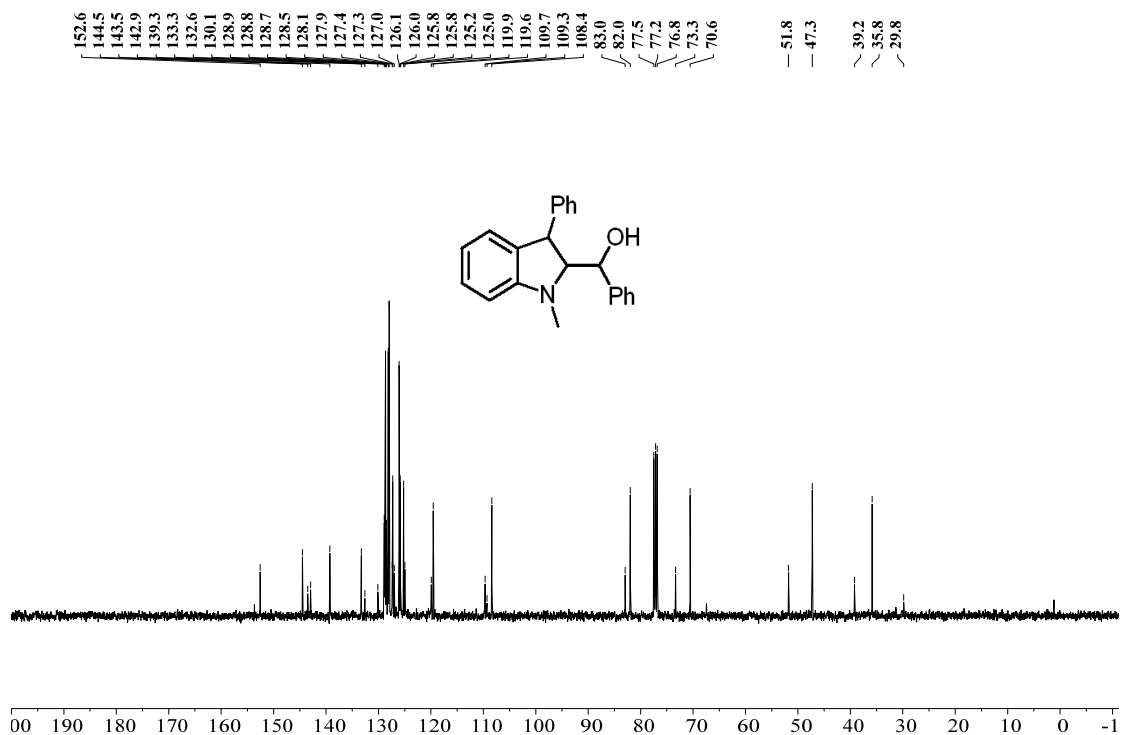


Figure S141. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **5** (CDCl_3 , 101 MHz, 23 °C)

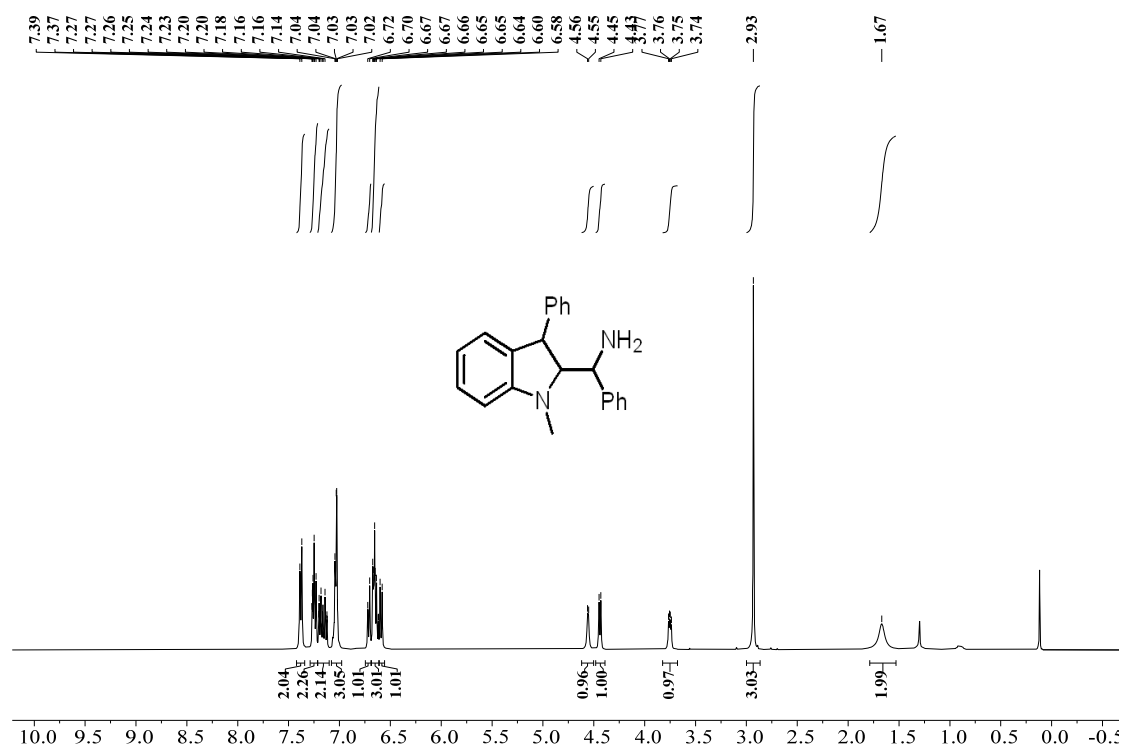


Figure S142. ^1H NMR spectrum of **6** (CDCl_3 , 400 MHz, 23 °C)

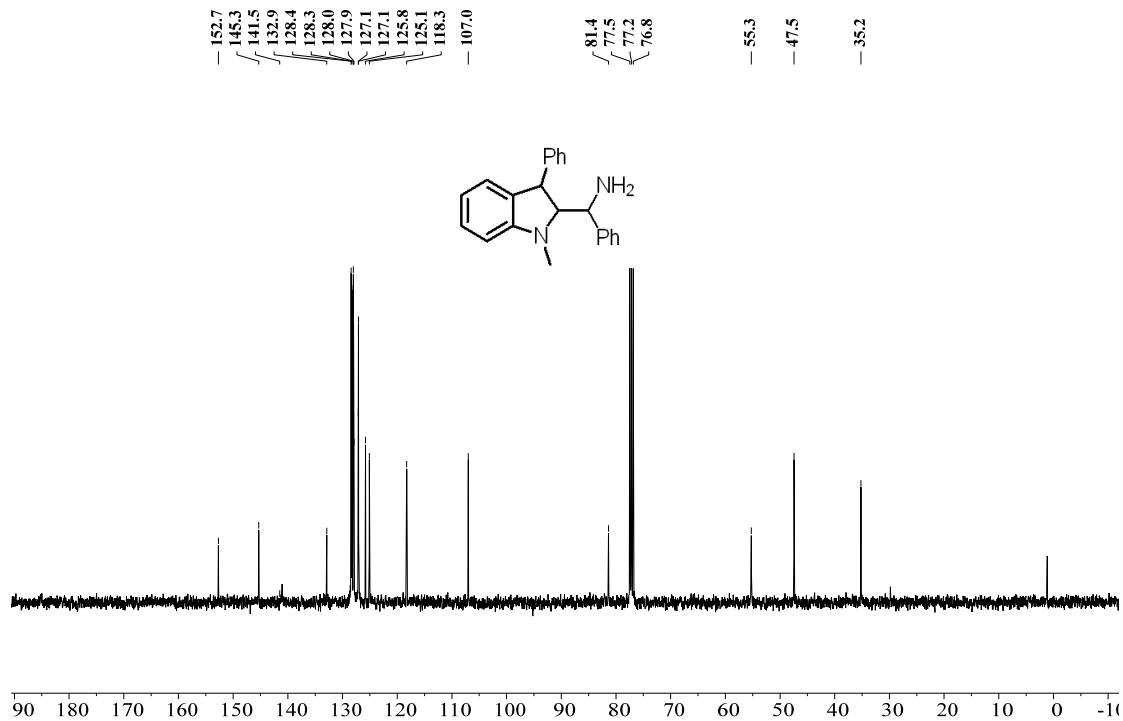


Figure S143. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **6** (CDCl_3 , 101 MHz, 23 °C)

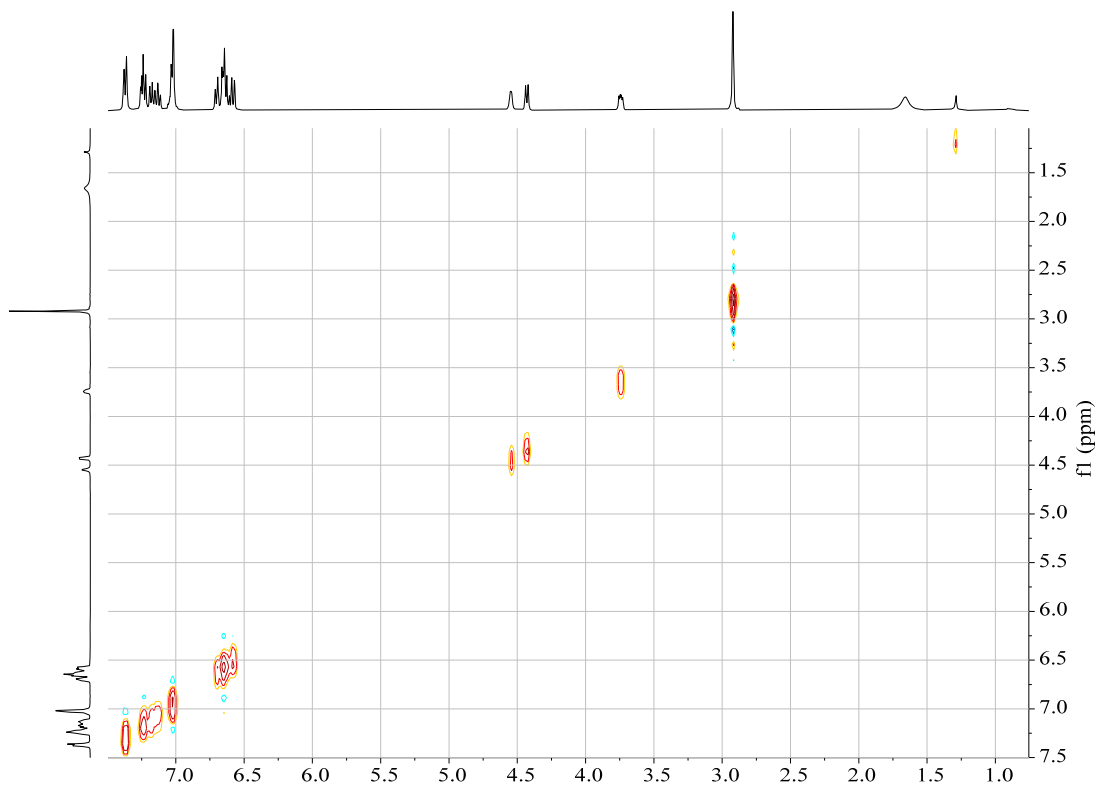


Figure S144. NOE spectrum of **6** (CDCl₃, 23 °C)

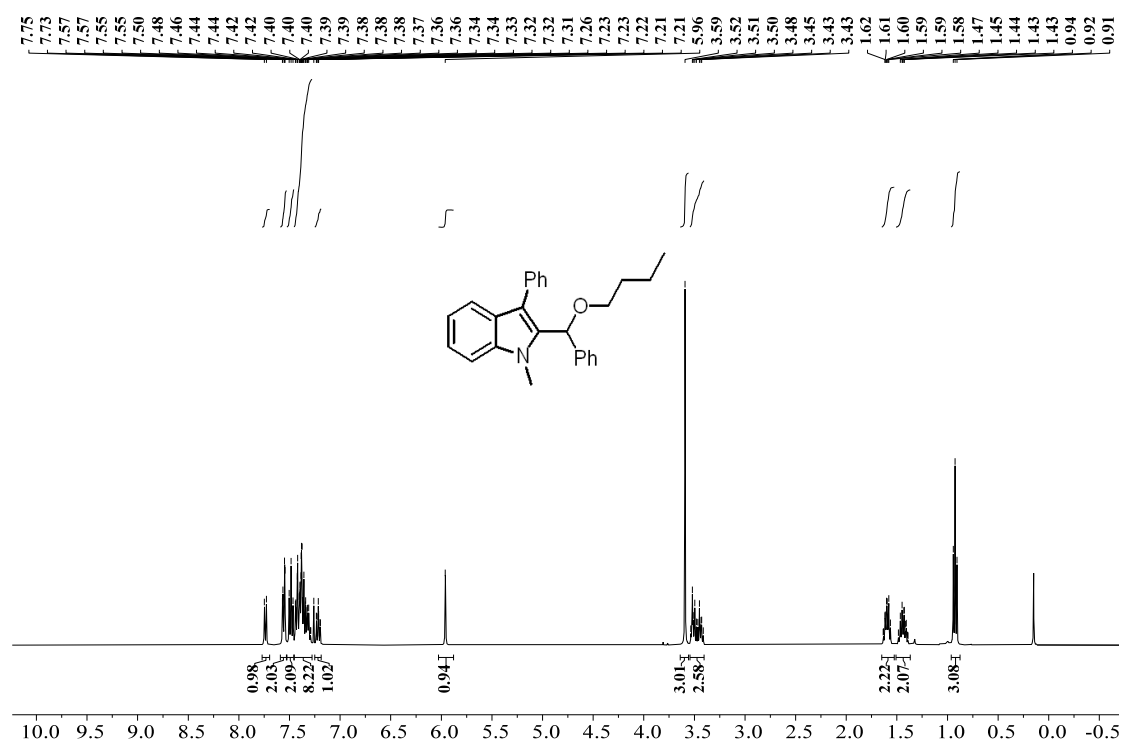


Figure S145. ^1H NMR spectrum of 7 (CDCl_3 , 400 MHz, 23 °C)

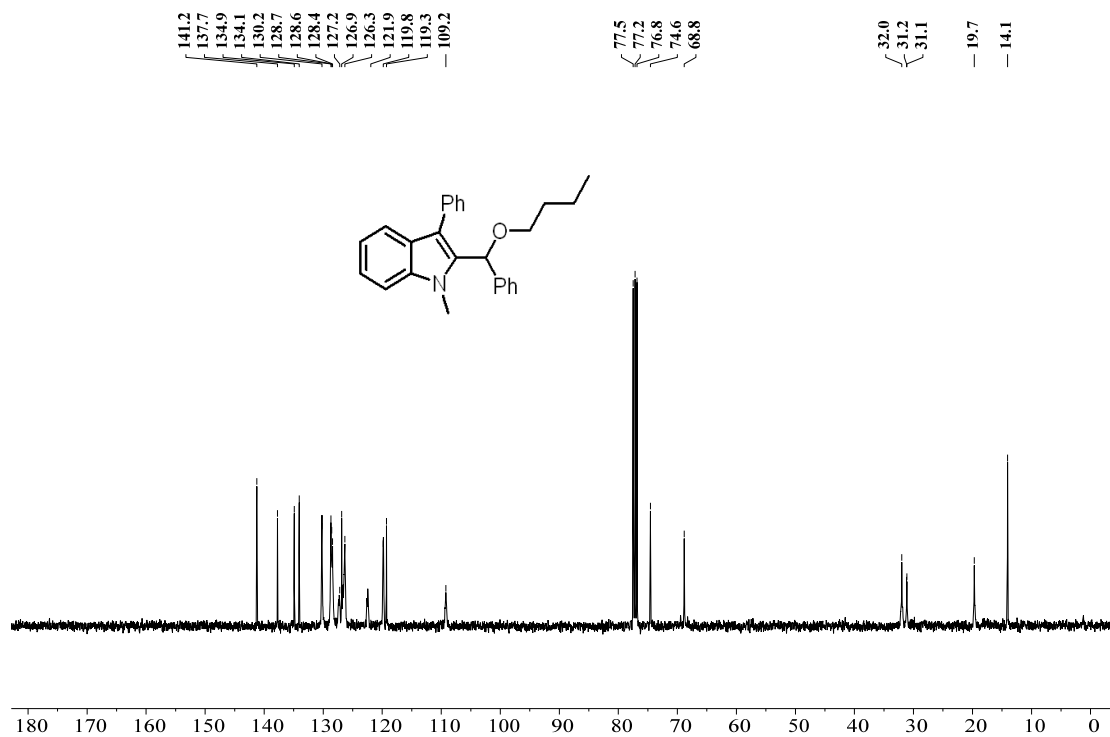


Figure S146. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of 7 (CDCl_3 , 101 MHz, 23 °C)