

Electronic supplementary information for
***N*-Hydroxyphthalimide: a new photoredox catalyst for [4+1] radical
cyclization of *N*-methylanilines with isocyanides**

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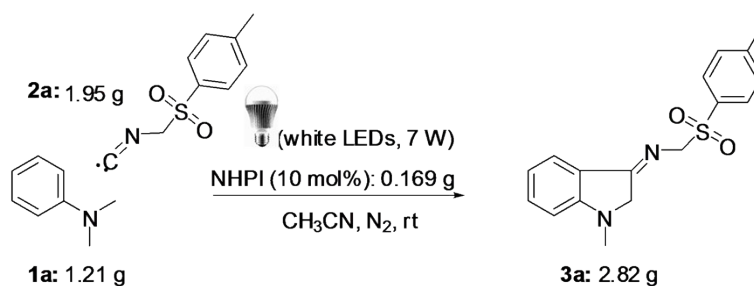
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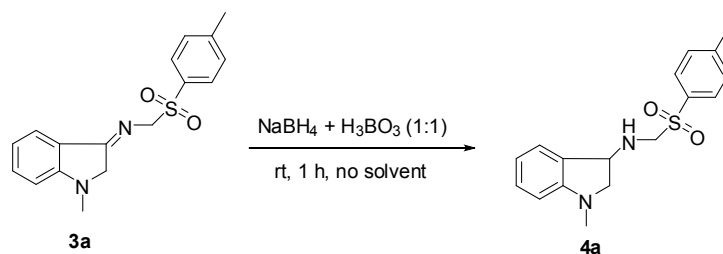


Figure. 1 Experimental setup.

- I. General Information:** All commercially available reagents were used without further purification unless otherwise specified by a reference. Solvents were purified by the usual methods and stored over molecular sieves. All reactions were performed using oven-dried glassware. Organic solutions were concentrated using a Buchi rotary evaporator. Column chromatography was carried out over silica gel (Merck 100–200 mesh) and TLC was performed using silica gel GF254 (Merck) plates. Melting points (mps) were determined by open glass tube and are uncorrected. The absorbance values of the samples were measured using potassium Reineckate as an actinometer. IR spectra in KBr were recorded on a Perkin-Elmer 993 IR spectrophotometer and ^1H (400 MHz), ^{13}C (100 MHz) NMR spectra were recorded on a Bruker AVII spectrometer in CDCl_3 using TMS as internal reference. All chemical shifts are reported in δ/ppm and coupling constants (J) in Hertz (Hz). MS (EI) spectra were recorded on double focusing mass spectrometer. White light LEDs (7 W) was purchased from commercial supplier Luxeon Star LEDs Quadica Developments Inc. 47 6th Concession Rd. Brantford, Ontario N 32 5L7 Canada.
- II. General procedure for the synthesis of compound 3:** A round bottom flask was charged with *N*-methylaniline **1** (1.0 mmol), isocyanide **2** (1.0 mmol) and NHPI (10 mol%) in CH_3CN (3 mL). The contents were irradiated from upper side of the flask using Luxeon Rebel high power white LEDs (7 W) with stirring under nitrogen atmosphere at room temperature for 10-14 h. After the completion of reaction (as indicated by TLC), it was quenched with water (5 mL) and extracted with ethyl acetate (3×5 mL). The organic phase was dried over anhydrous sodium sulfate and concentrated under reduced pressure to yield the crude product, which was purified by silica gel column chromatography using a mixture of EtOAc-hexane to give the pure product **3**.

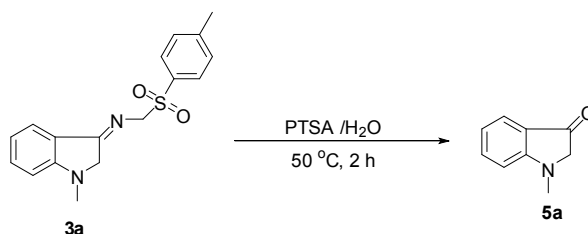


Scheme 1 Gram scale synthesis of 3-iminoindole.



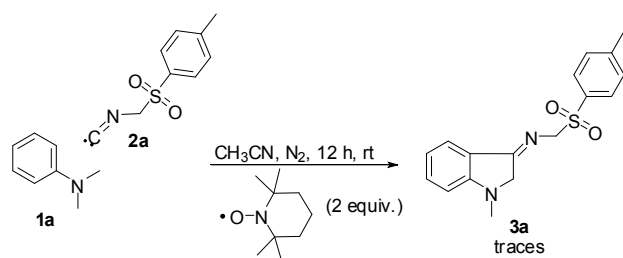
Scheme 2 Reduction of product **3a** into **4a**.

III. General procedure for the reduction of compound 3a into 4a: A mixture of imine **3a** (1.0 mmol), sodium borohydride (1.0 mmol) and boric acid (1.0 mmol) was ground with an agate mortar and pestle at room temperature for 1.0 h until TLC showed complete disappearance of the starting material. The reaction mixture was quenched with water (5 mL) and extracted with ethyl acetate (3 × 5 mL). The organic phase was dried over anhydrous sodium sulfate and concentrated under reduced pressure to yield the crude product, which was purified by silica gel column chromatography using a mixture of EtOAc-hexane to give the pure product **4a**.



Scheme 3 Hydrolysis of product **3a** into **5a**.

IV. General procedure for the hydrolysis of compound 3a into 5a: A mixture of imine **3a** (1.0 mmol) was stirred with PTSA/H₂O (1.0 equiv., 1:1) in DCE (3 mL) for 2.0 h at 50 °C until TLC showed complete disappearance of the starting material. It was extracted with ethyl acetate (3 × 5 mL). The organic phase was dried over anhydrous sodium sulfate and concentrated under reduced pressure to yield the crude product, which was purified by silica gel column chromatography using a mixture of EtOAc-hexane to give the pure product **5a**.



Scheme 4 TEMPO mediated intermolecular cyclization of *N,N*-dimethylaniline with TosMIC.

V. On/off visible light irradiation experiment:

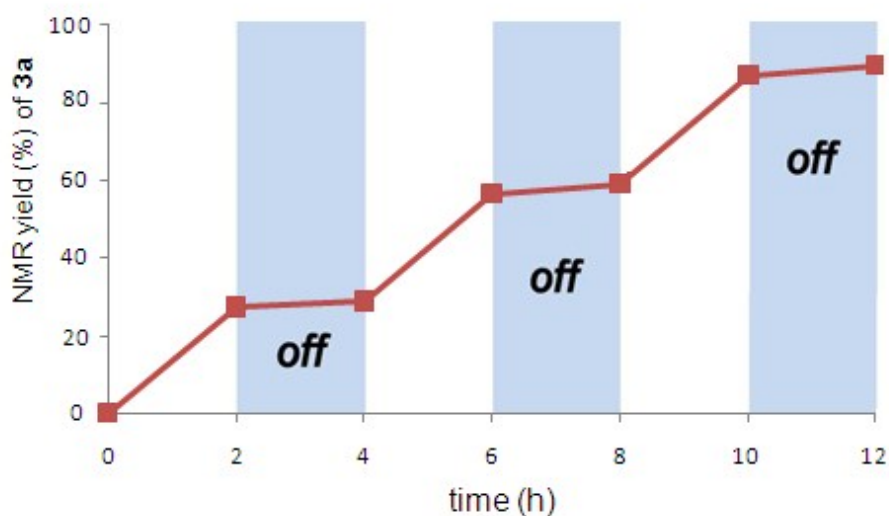


Figure 2. On/off visible light irradiation experiment.

VI. Determination of quantum yield (ϕ): The reaction mixture was irradiated with high-power LED (7 W) for 12 h (Figure 2). The photon flux was estimated to be $1.48 \times 10^{-8} \text{ E s}^{-1}$ by using potassium Reineckate as an actinometer.¹ The rate of formation of **3a** was obtained by ¹H NMR spectrum of the crude product using acetonitrile as internal standard. It was found to be $2.12 \times 10^{-9} \text{ mol s}^{-1}$, which was converted into quantum yield $\phi = 0.144$.

VII. Spectral data of synthesised compounds 3, 4a and 5a.

Compound 3a: White solid, mp: 118-120 °C; IR (KBr): 3394, 3360, 3140, 2942, 2821, 2258, 1657, 1596, 1499, 1322, 1139, 735, 681, 672 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): δ = 7.73 (d, J = 8.1 Hz, 2H), 7.40-7.33 (m, 3H), 6.90 (t, J = 7.2 Hz, 1H), 6.68 (d, J = 8.1 Hz, 2H), 4.69 (s, 2H), 3.75 (s, 2H), 2.99 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ = 164.1, 154.7, 135.2, 131.4, 128.7, 128.3, 127.2, 121.4, 118.6, 117.1, 115.0, 71.1, 47.4, 37.3, 21.1. HRMS (EI) calcd. for $\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_2\text{S}^+$: 314.1085, found 314.1087.

Compound 3b: White solid, mp: 110-113 °C; IR (KBr): 3524, 3403, 3339, 3141, 2922, 2813, 1643, 1340, 1123, 857, 672 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): δ = 7.74 (d, J = 8.1 Hz, 1H), 7.36-7.21 (m, 6H), 6.91-6.83 (m, 2H), 4.68 (s, 2H), 3.70 (s, 2H), 3.01 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ = 164.3, 154.9, 133.5, 131.3, 130.2, 128.5, 125.9, 121.6, 118.3, 117.2, 114.8, 70.4, 47.2, 37.9. HRMS (EI): calcd. for $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_2\text{S}^+$ 300.0927, found 300.0931.

Compound 3c: White solid, mp: 127-128 °C; IR (KBr): 3541, 3421, 3317, 3134, 3073, 2936, 2841, 1637, 1539, 1327, 1142, 1039, 721, 675 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): δ = 7.58 (s, 1H), 7.29 (d, J = 7.1 Hz, 4H), 7.04 (d, J = 7.2 Hz, 1H), 6.78 (d, J = 7.2 Hz, 1H), 4.67 (s, 2H), 3.61 (s, 2H), 3.01 (s, 3H), 2.34 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ = 164.7, 152.0, 135.5, 130.1, 129.7, 128.7, 128.4, 128.1, 125.3, 115.6, 115.2, 71.0, 46.9, 37.4, 21.2, 21.0. HRMS (EI) calcd. for $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_2\text{S}^+$: 328.1240, found 328.1243.

Compound 3d: White solid, mp: 135-137 °C; IR (KBr): 3473, 3342, 3152, 3042, 2978, 2831, 1634, 1547, 1321, 1137, 1119, 781 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): δ = 7.69 (s, 1H), 7.30 (d, J = 7.2 Hz, 4H), 6.79 (d, J = 8.2 Hz, 1H), 6.58 (d, J = 8.1 Hz, 1H), 4.69 (s, 2H), 3.79 (s, 3H), 3.62 (s, 2H), 3.02 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ = 164.9, 149.7, 146.2, 134.4, 129.6, 128.5, 128.1, 119.9, 117.5, 116.3, 114.7, 70.3, 54.8, 46.7, 37.3, 21.2. HRMS (EI) calcd. for $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_3\text{S}^+$: 344.1190, found 344.1192.

Compound 3e: White solid; mp: 139-140 °C; IR (KBr): 3427, 3334, 2924, 2826, 1649, 1332, 1263, 817, 642, cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): δ = 7.68 (s, 1H), 7.37 (d, J = 8.2 Hz, 1H), 7.30-7.23 (m, 4H), 6.68 (d, J = 8.2 Hz, 1H), 4.65 (s, 2H), 3.65 (s, 2H), 3.07 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ = 164.7, 154.8, 135.3, 134.6, 133.2, 130.8, 128.9, 128.1, 118.5, 116.4, 112.3, 70.6, 47.3, 37.4, 21.2. HRMS (EI) calcd. for $\text{C}_{17}\text{H}_{17}\text{BrN}_2\text{O}_2\text{S}^+$: 392.0184, found 392.0189.

Compound 3f: Yellow solid; mp: 168-170 °C; IR (KBr): 3327, 3134, 2927, 2824, 1648, 1567, 1481, 1357, 1242, 1263, 819, 643, cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): δ = 8.32 (s, 1H), 7.91 (d, J = 8.7 Hz, 1H), 7.73 (m, 4H), 7.08 (d, J = 8.2 Hz, 1H), 4.67 (s, 2H), 3.45 (s, 2H), 3.07 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ = 164.7, 160.8, 139.3, 137.4, 135.8, 130.3-2C, 128.6-2C, 127.2, 123.8, 122.9, 113.6, 71.5, 47.4, 37.3, 21.2. HRMS (EI) calcd. for $\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}_4\text{S}^+$: 359.0935, found 359.0939.

Compound 3g: Yellow oily liquid; IR (KBr): 2942, 2834, 1664, 1595, 1337, 1247, 1134, 781 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): δ = 7.72 (d, J = 8.2 Hz, 1H), 7.27 (t, J = 8.2 Hz, 1H), 7.19-6.82 (m, 5H), 6.89 (t, J = 8.2 Hz, 2H), 3.95 (s, 2H), 3.07 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ = 164.4, 154.6, 151.0, 131.3, 130.0, 128.5, 121.7, 119.8, 118.1, 117.3, 115.6, 48.1, 37.2. HRMS (EI): calcd. for $\text{C}_{15}\text{H}_{14}\text{N}_2^+$: 222.1152, found 222.1156.

Compound 3h: Yellow viscous solid; IR (KBr): 3147, 2963, 2842, 1633, 1526, 1307, 1253, 1141, 784 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): δ = 8.31 (d, J = 8.9 Hz, 1H), 7.43-7.37 (m, 2H), 7.32-7.26 (m, 2H), 7.14 (d, J = 8.9 Hz, 1H), 6.91-6.82 (m, 2H), 3.98 (s, 2H), 3.78 (s, 3H), 3.07 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ = 164.1, 158.9, 155.2, 143.5, 131.7, 122.6, 121.3, 118.5, 117.4, 115.9, 114.7, 55.6, 47.7, 36.9. HRMS (EI): calcd. for $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}^+$: 252.1258, found 252.1262.

Compound 3i: White solid, mp: 40-42 °C; IR (KBr): 2943, 2836, 1664, 1449, 1257, 831, 671 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): δ = 7.57 (d, J = 7.3 Hz, 1H), 7.50 (d, J = 8.1 Hz, 2H), 7.32 (t, J = 7.2 Hz, 2H), 6.98 (d, J = 8.2 Hz, 1H), 6.86-6.79 (m, 2H), 3.69 (s, 2H), 3.01 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ = 164.8, 155.6, 149.4,

132.2, 131.3, 130.0, 122.7, 121.1, 118.9, 117.3, 115.6, 48.7, 37.5. HRMS (EI): calcd. for $C_{15}H_{13}ClN_2^+$: 256.0762, found 256.0766.

Compound 3j: Yellow solid, mp: 70-73 °C; IR (KBr): 3203, 2946, 2837, 1672, 1567, 1481, 1357, 1242, 725, 580, cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz): δ = 8.19 (d, J = 9.2 Hz, 2H), 7.59 (d, J = 8.2 Hz, 1H), 7.34 (t, J = 7.6 Hz, 1H), 6.92 (d, J = 9.2 Hz, 2H), 6.84-6.76 (m, 2H), 3.67 (s, 2H), 3.03 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 165.3, 157.2, 155.4, 146.3, 131.8, 125.7, 123.6, 121.1, 118.9, 117.5, 115.0, 48.7, 37.5. HRMS (EI) calcd. for $C_{15}H_{13}N_3O_2^+$: 267.1003, found 267.1005.

Compound 3k: Yellow oily liquid; IR (KBr): 3221, 3141, 2952, 2834, 1669, 1254, 853, 776 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz): δ = 7.67 (d, J = 8.2 Hz, 1H), 7.35 (t, J = 8.2 Hz, 1H), 6.83-6.74 (m, 2H), 3.56 (s, 2H), 3.01 (s, 3H), 1.26 (s, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 164.1, 155.3, 130.9, 121.7, 120.4, 118.8, 117.1, 51.3, 48.5, 37.2, 30.6. HRMS (EI) calcd. for $C_{13}H_{18}N_2^+$: 202.1465, found 202.1469.

Compound 3l: Yellow oily liquid; IR (KBr): 3146, 2933, 2844, 1673, 1256, 910, 874, 776, 574 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz): δ = 7.52-7.30 (m, 2H), 7.13 (t, J = 7.2 Hz, 1H), 7.05 (t, J = 7.2 Hz, 1H), 3.58 (s, 2H), 3.02 (s, 3H), 2.96-2.90 (m, 1H), 2.06-1.99 (m, 2H), 1.74-1.67 (m, 2H), 1.61-1.59 (m, 1H), 1.55-1.50 (m, 1H), 1.47-1.36 (m, 3H), 1.32-1.26 (m, 1H), 2.95 (m, 1H), 2.05-1.98 (m, 2H), 1.72-1.65 (m, 2H), 1.60-1.57 (m, 1H), 1.54-1.51 (m, 1H), 1.46-1.35 (m, 3H), 1.30-1.24 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 164.3, 155.1, 130.7, 121.4, 121.0, 118.9, 117.2, 56.7, 47.3, 36.8, 32.3, 24.7, 24.1. HRMS (EI) calcd. for $C_{15}H_{20}N_2^+$: 228.1621, found 228.1624.

Compound 3m: Yellow oily liquid; IR (KBr): 3140, 2947, 2861, 1682, 1253, 828, 728, 700 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz): δ = 7.59 (d, J = 8.1 Hz, 1H), 6.96 (t, J = 7.2 Hz, 1H), 6.78-6.70 (m, 2H), 3.85 (t, J = 6.2 Hz, 2H), 3.65 (s, 2H), 3.02 (s, 3H), 1.67-1.62 (m, 2H), 1.60-1.31 (m, 6H), 0.83 (t, J = 6.9 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 164.7, 155.3, 131.4, 121.3, 121.1, 118.5, 117.2, 54.7, 47.6, 36.9, 31.6, 31.4, 25.6, 22.3, 13.7. HRMS (EI) calcd. for $C_{15}H_{22}N_2^+$: 230.1778, found 230.1780.

Compound 3n: Yellow oily liquid; IR (KBr): 3114, 2937, 2845, 1662, 1243, 820, 721, 707 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): $\delta = 7.56$ (d, $J = 8.2$ Hz, 1H), 7.40 (t, $J = 8.2$ Hz, 2H), 7.40-7.32 (m, 3H), 6.90 (t, $J = 8.2$ Hz, 1H), 6.86 (d, $J = 7.3$ Hz, 2H), 3.61 (s, 2H), 1.39 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 164.7$, 154.3, 151.2, 131.0, 129.4, 127.2, 121.6, 119.4, 118.0, 117.2, 115.7, 54.1, 39.8, 27.9. HRMS (EI) calcd. for $\text{C}_{18}\text{H}_{20}\text{N}_2^+$: 264.1621, found 264.1623.

Compound 3o: Yellow oily liquid; IR (KBr): 3213, 3140, 2936, 2844, 1678, 1251, 823, 710 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): $\delta = 7.56$ (d, $J = 8.2$ Hz, 1H), 7.46-7.38 (m, 2H), 7.43-7.07 (m, 3H), 6.88 (t, $J = 8.2$ Hz, 1H), 6.86 (d, $J = 7.3$ Hz, 2H), 3.64 (s, 2H), 3.28 (q, $J = 6.3$ Hz, 2H), 1.14 (t, $J = 6.4$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 168.4$, 154.7, 151.3, 131.1, 130.3, 127.0, 121.4, 119.7, 118.5, 117.3, 115.2, 45.5, 44.3, 12.9. HRMS (EI) calcd. for $\text{C}_{16}\text{H}_{16}\text{N}_2^+$: 236.1308, found 236.1312.

Compound 3p: White solid: mp 105-107 $^\circ\text{C}$; IR (KBr): 3183, 3047, 2850, 1656, 1248, 723, cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): $\delta = 7.56$ (d, $J = 8.2$ Hz, 1H), 7.63-7.27 (m, 7H), 7.18-7.10 (m, 3H), 6.99-6.94 (m, 2H), 6.72 (d, $J = 8.1$ Hz, 1H), 3.46 (s, 2H), ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 164.7$, 151.3, 148.9, 143.2, 131.3, 130.1, 129.4, 127.2, 121.8, 121.3, 120.6, 119.7, 119.1, 118.0, 116.5, 47.6. HRMS (EI) calcd. for $\text{C}_{20}\text{H}_{16}\text{N}_2^+$: 284.1308, found 284.1311.

Compound 3q: Yellow oily liquid; IR (KBr): 3227, 3140, 2957, 2834, 1667, 1250, 851, 778, cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): $\delta = 7.45$ -7.32 (m, 3H), 7.23-6.87 (m, 4H), 6.72 (t, $J = 8.1$ Hz, 1H), 3.41 (s, 2H), 3.04 (s, 3H), 2.21 (s, 3H), ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 164.5$, 151.0, 145.6, 133.2, 130.1, 128.5, 127.4, 119.3, 118.8, 118.1, 115.6, 48.2, 38.5, 17.6. HRMS (EI) calcd. for $\text{C}_{16}\text{H}_{16}\text{N}_2^+$: 236.1308, found 236.1310.

Compound 3r: Yellow oily liquid; IR (KBr): 3173, 3053, 2856, 1650, 1241, 720, cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz): $\delta = 7.57$ -7.45 (m, 3H), 7.38-7.27 (m, 3H), 6.64 (d, $J = 8.1$ Hz, 1H), 6.21 (s, 1H), 3.43 (s, 2H), 3.01 (s, 3H), 2.32 (s, 3H), ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 164.7$, 154.6, 151.1, 141.2, 131.0, 129.2, 127.4, 119.1, 117.6,

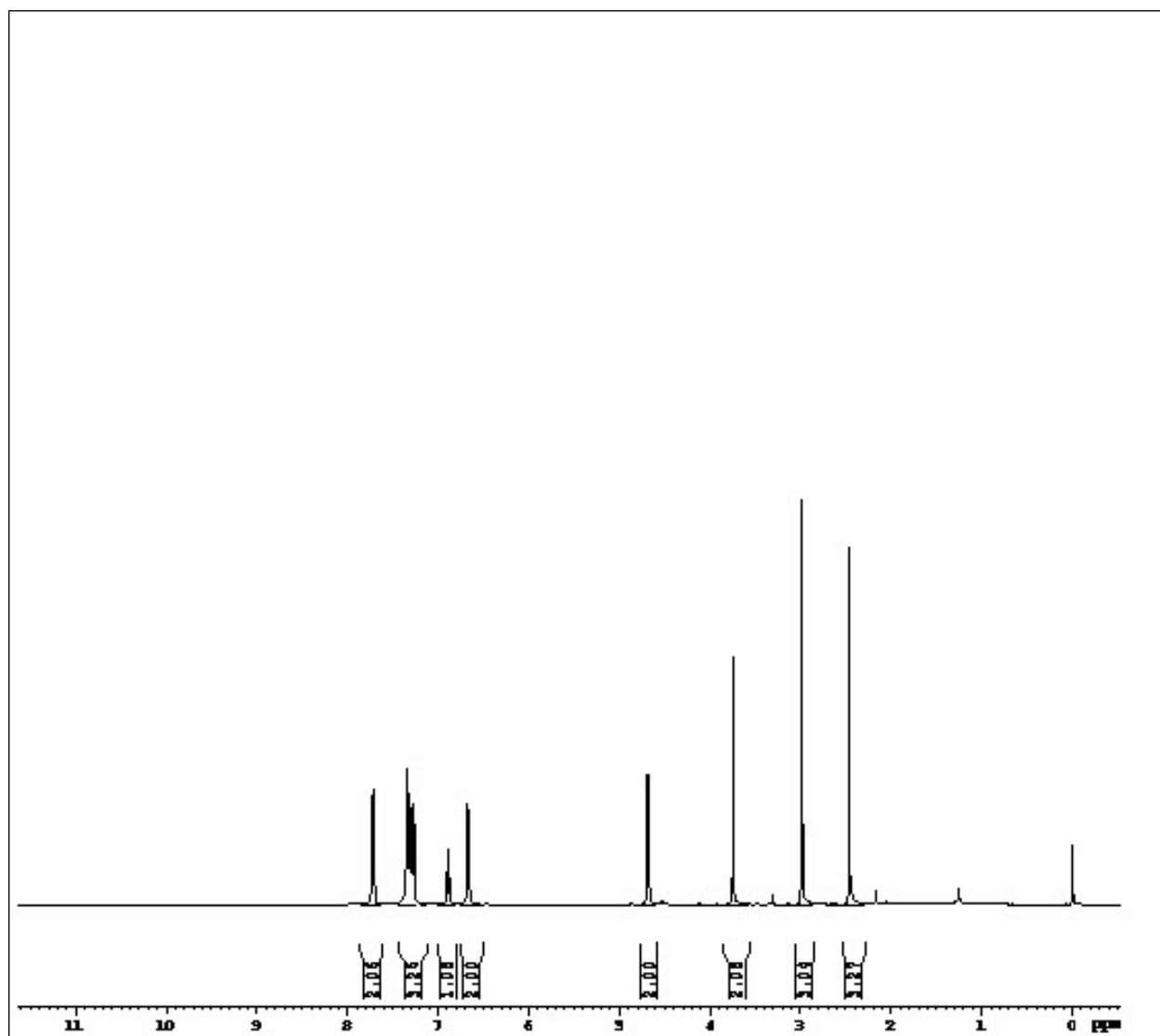
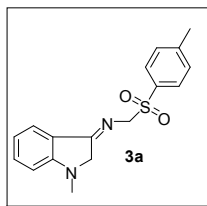
114.3, 112.5, 48.1, 37.0, 21.2. HRMS (EI) calcd. for $C_{16}H_{16}N_2^+$: 236.1308, found 236.1309.

Compound 4a: White solid, mp: 147-150 °C; IR (KBr): 3464, 3367, 3143, 2945, 2826, 2251, 1590, 1493, 1325, 1141, 737, 687, 670 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz): δ = 7.68 (d, J = 8.1 Hz, 2H), 7.41-7.35 (m, 3H), 6.92 (t, J = 7.2 Hz, 1H), 6.65 (d, J = 8.1 Hz, 2H), 5.04 (brs, 1H), 4.13 (t, J = 7.4 Hz, 1H), 3.93 (dd, J = 7.2 Hz, 1H), 3.79 (dd, J = 7.2 Hz, 1H), 3.75 (s, 2H), 2.49 (s, 3H), 2.34 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 151.9, 138.6, 135.4, 129.7, 128.1, 127.2, 126.4, 121.6, 118.1, 108.9, 70.8, 63.0, 57.3, 40.4, 21.0. HRMS (EI) calcd. for $C_{17}H_{20}N_2O_2S^+$: 316.1240, found 316.1243.

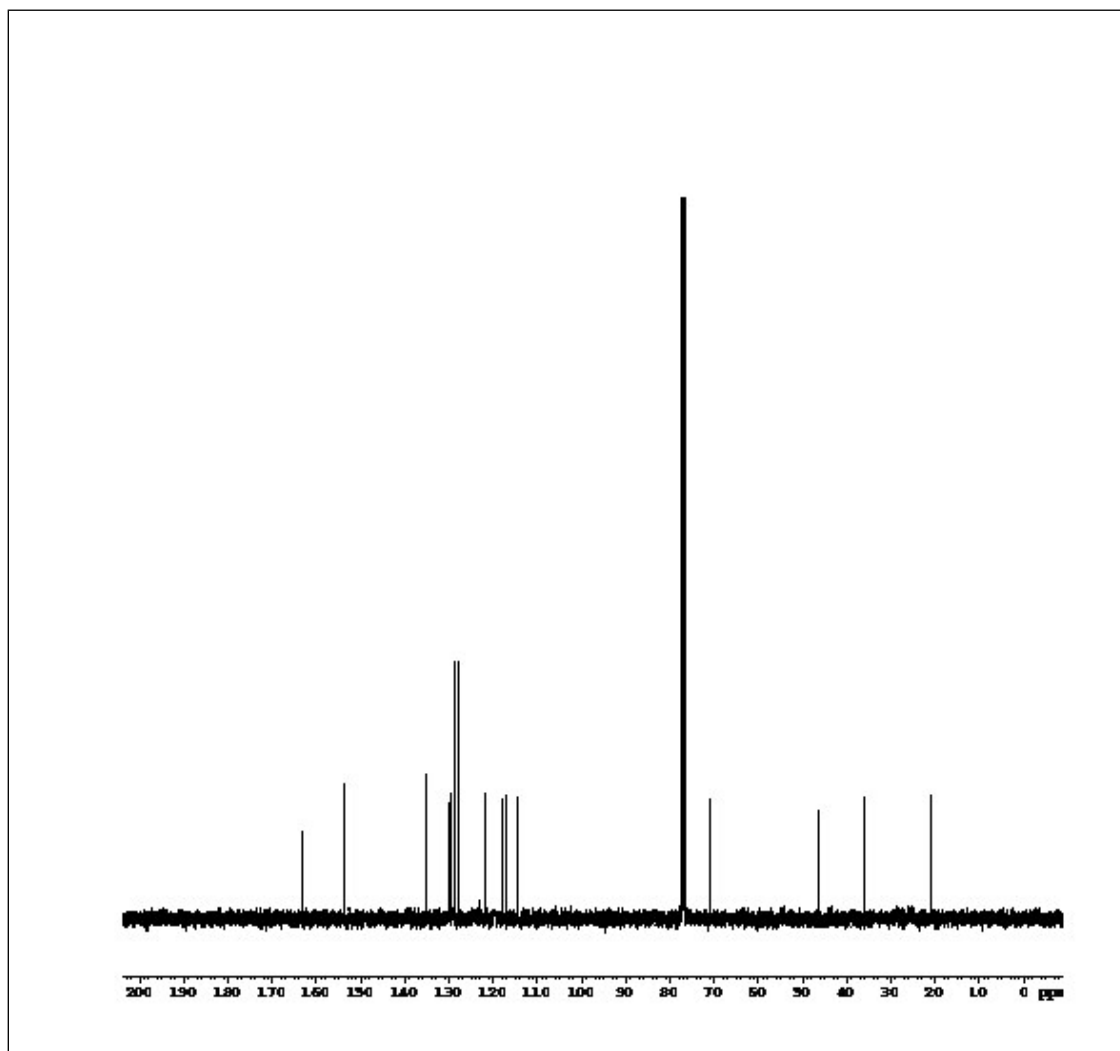
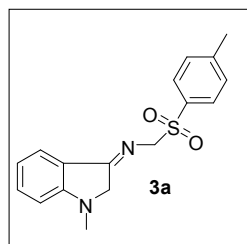
Compound 5a: Yellow oily liquid; IR (KBr): 3360, 3367, 3143, 2940, 2823, 2257, 1723, 1589, 1490, 1321, 1148, 691, 667 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz): δ = 7.63 (d, J = 8.3 Hz, 1H), 7.32-7.27 (m, 1H), 6.54-6.47 (m, 2H), 4.57 (s, 2H), 3.04 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 183.9, 154.1, 134.2, 129.4, 121.3, 118.5, 117.1, 66.9, 36.7. HRMS (EI) calcd. for $C_9H_9NO^+$: 147.0679, found 147.0681.

VIII. ^1H and ^{13}C NMR Copies of synthesised compounds.

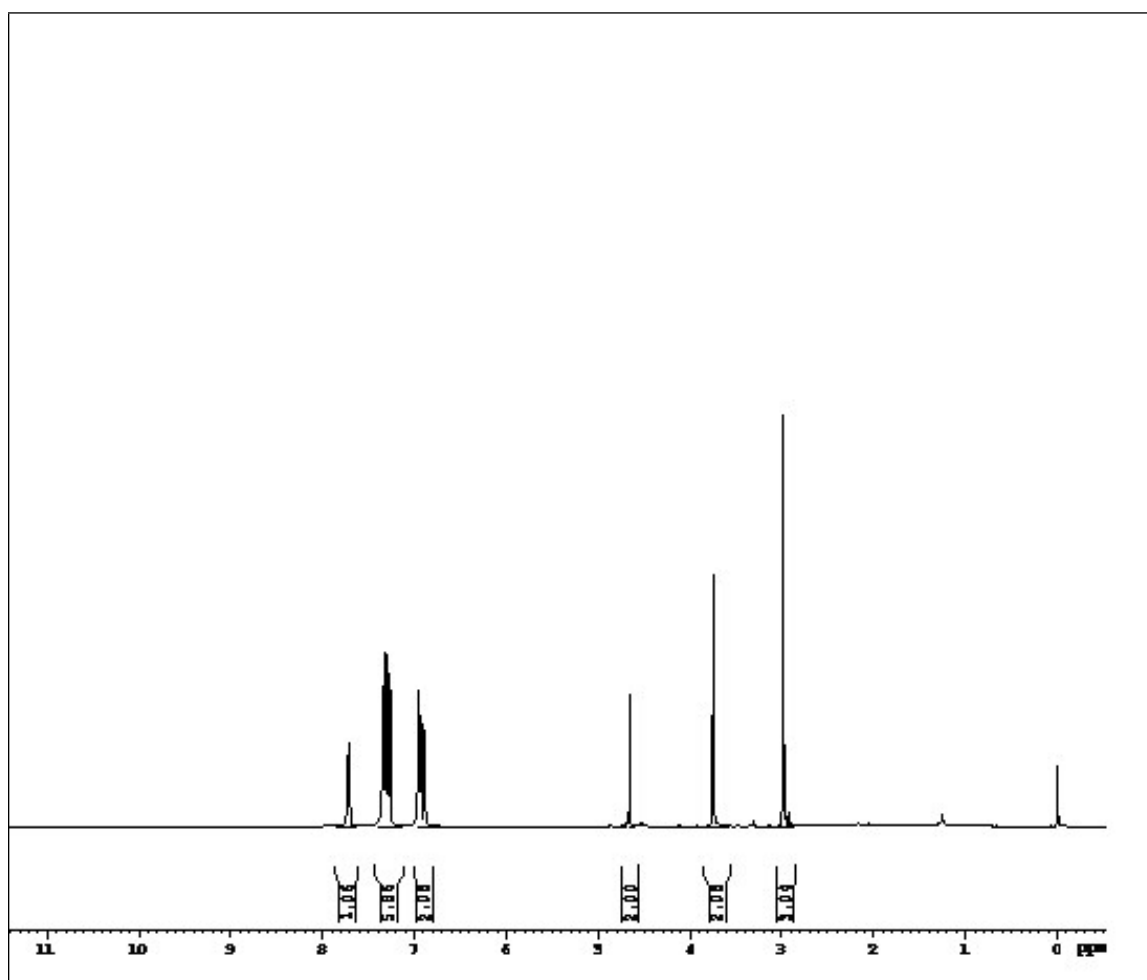
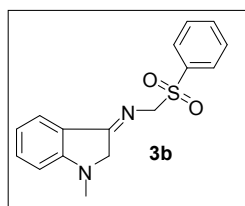
Compound 3a. ^1H NMR Spectrum (CDCl_3).



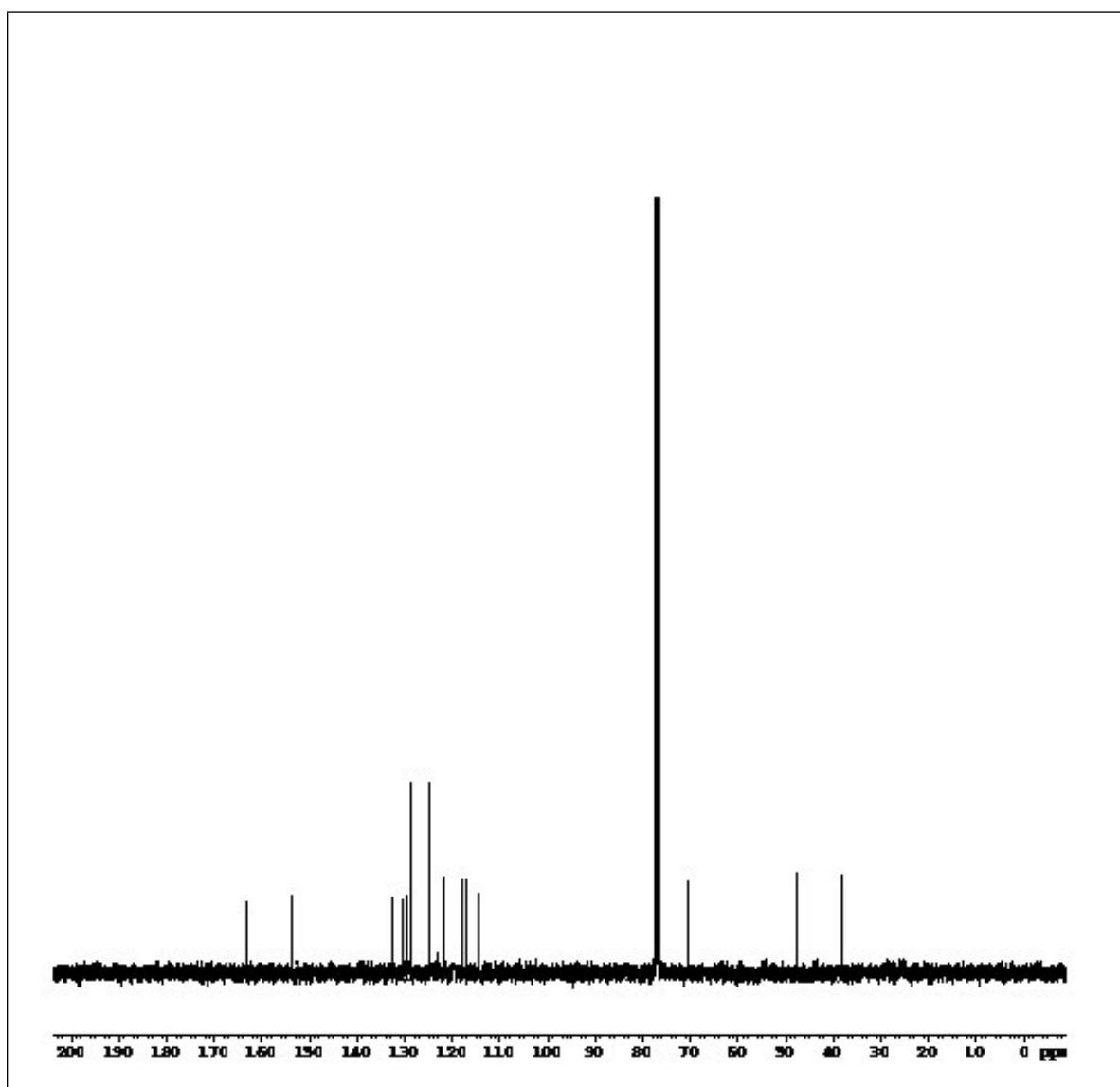
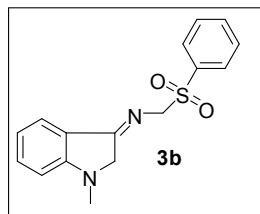
Compound 3a. ^{13}C NMR Spectrum (CDCl_3).



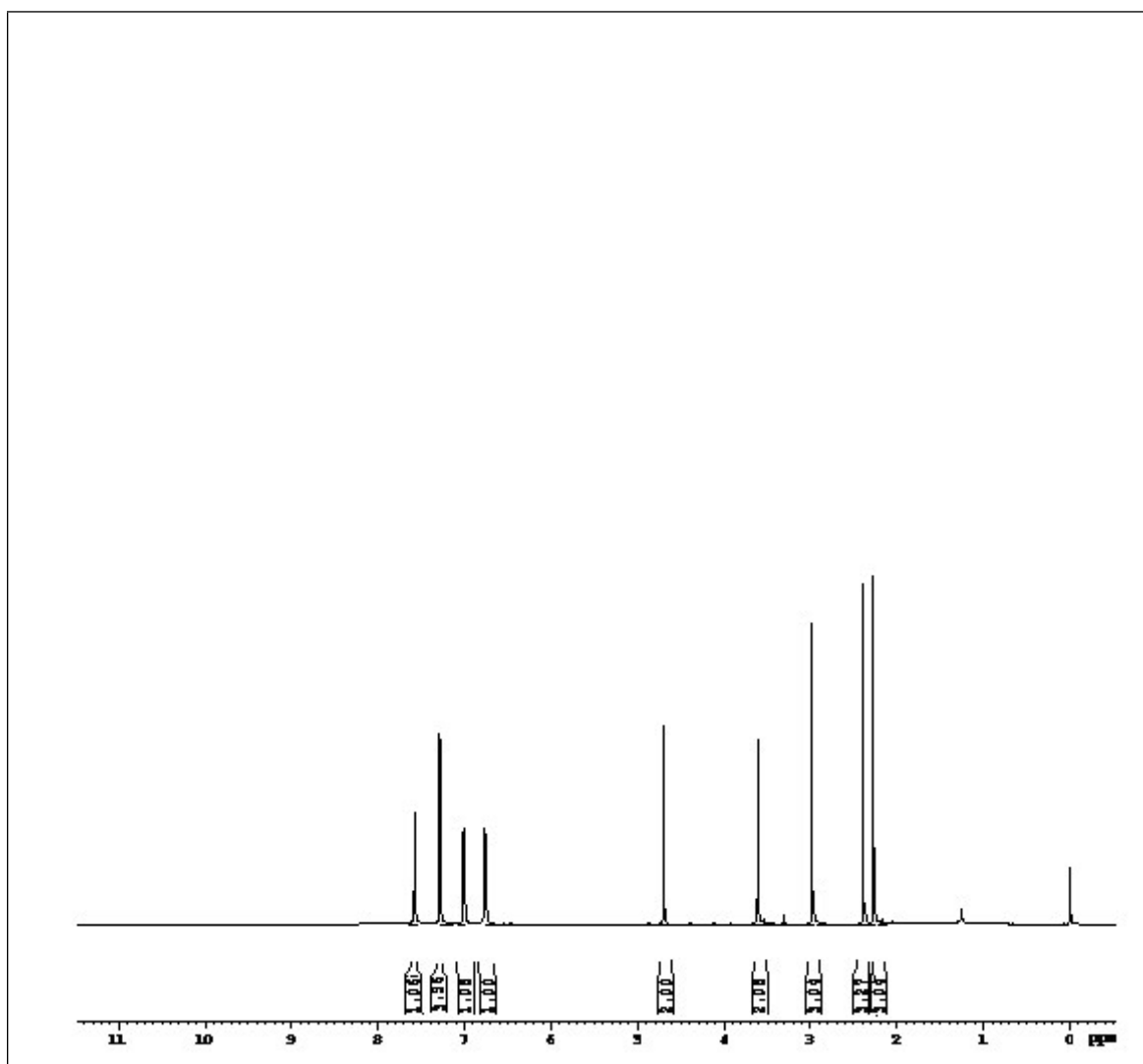
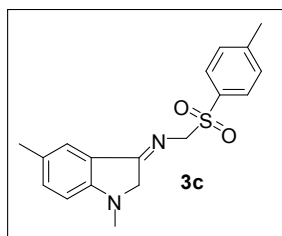
Compound 3b. ^1H NMR Spectrum (CDCl_3).



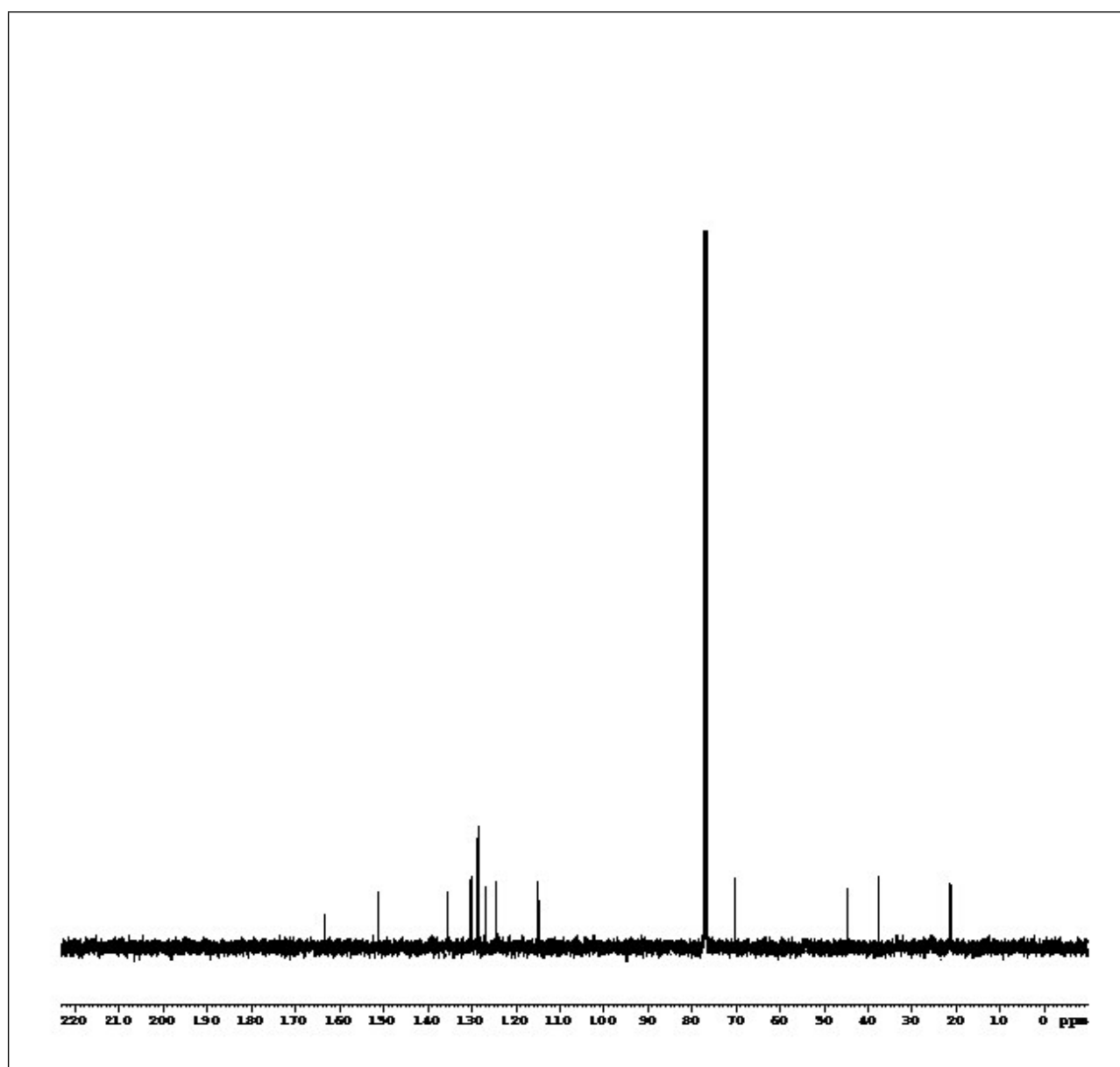
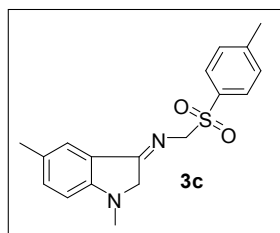
Compound 3b. ^{13}C NMR Spectrum (CDCl_3).



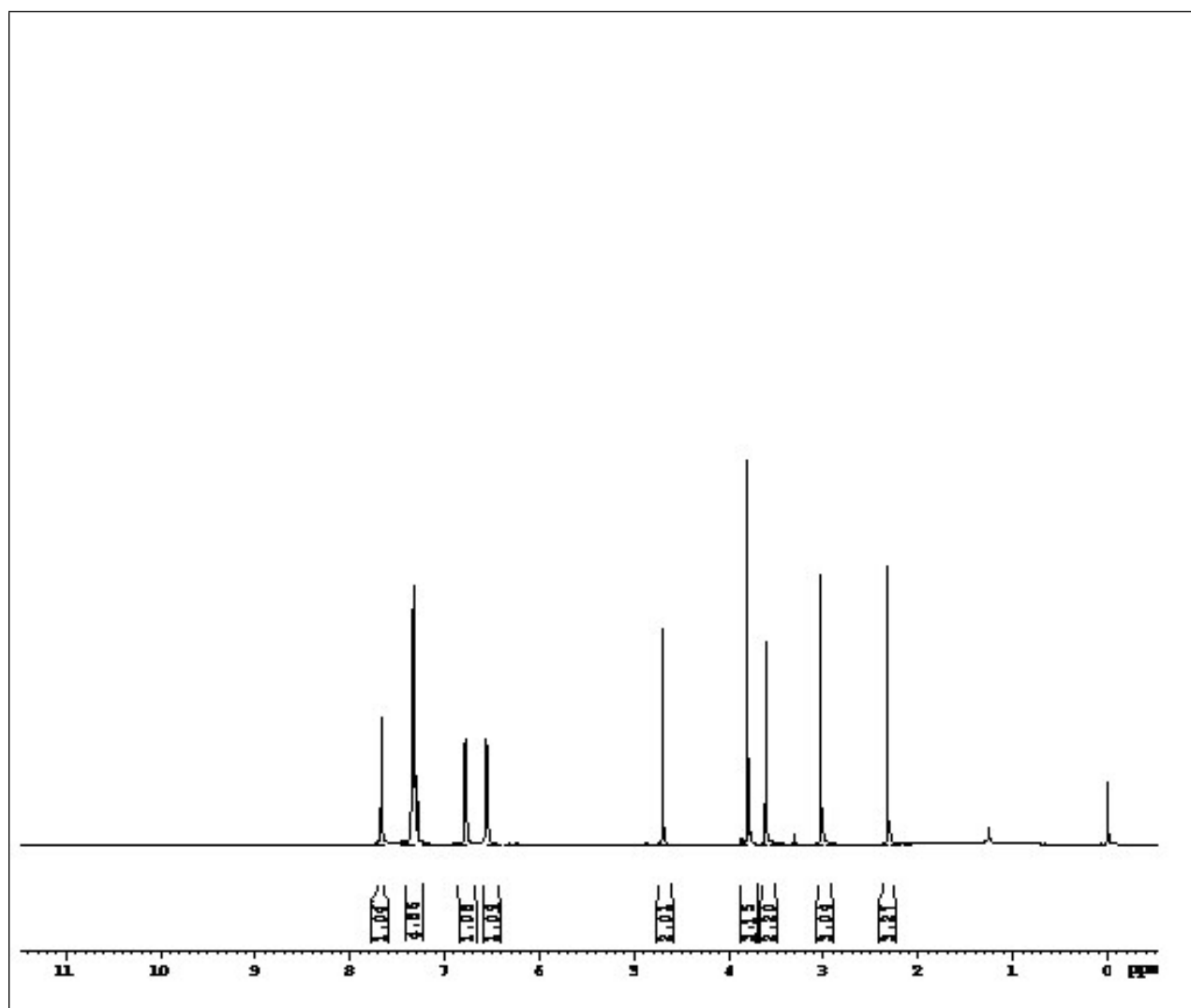
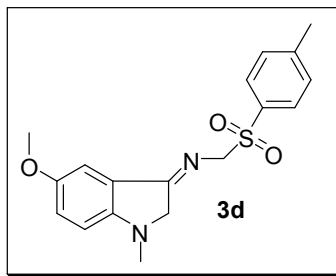
Compound 3c. ^1H NMR Spectrum (CDCl_3).



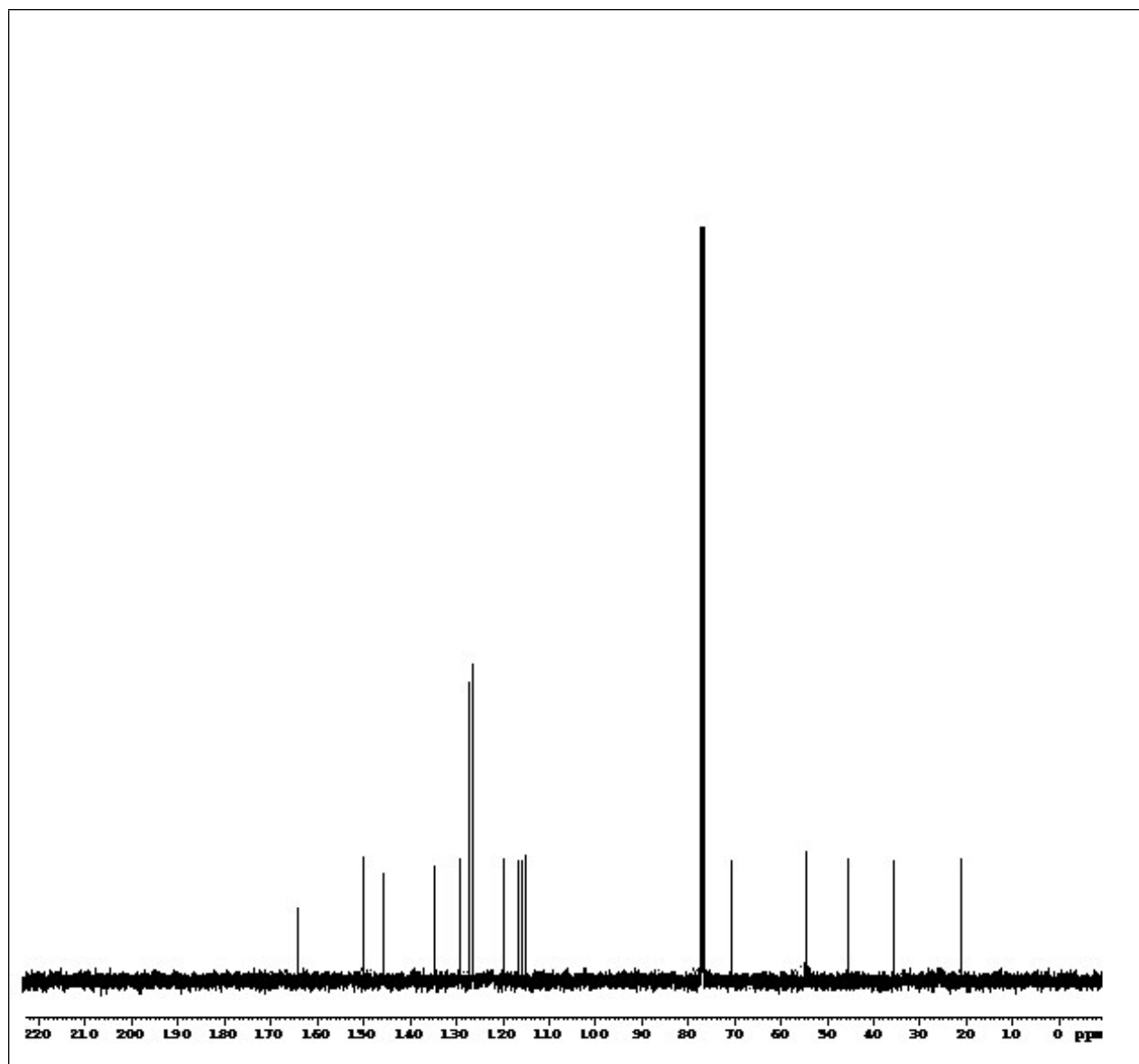
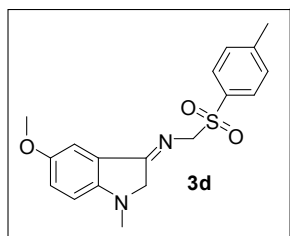
Compound 3c. ^{13}C NMR Spectrum (CDCl_3).



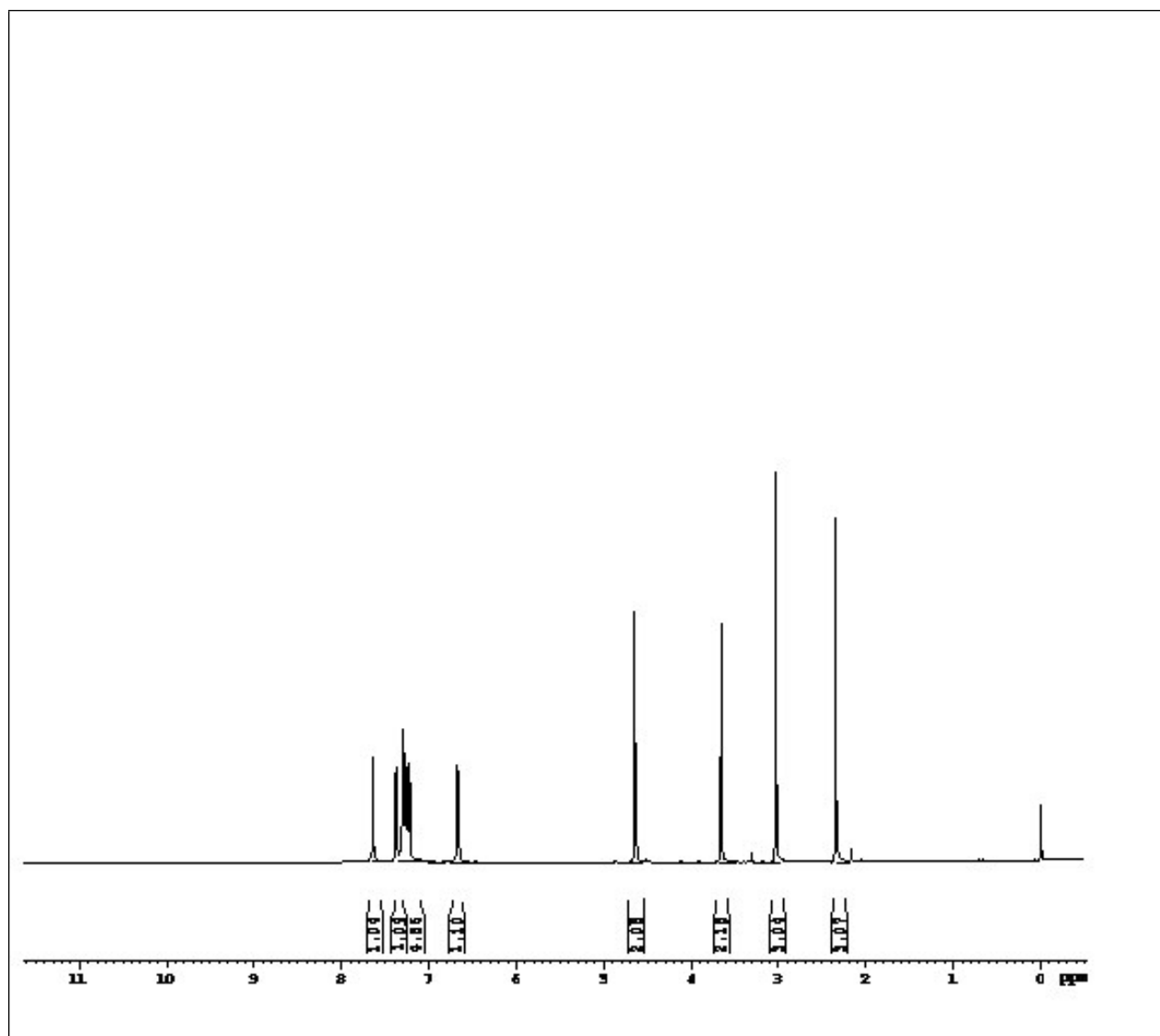
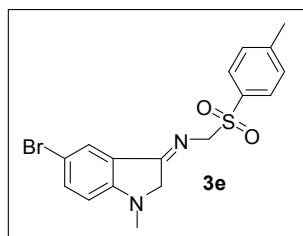
Compound 3d. ^1H NMR Spectrum (CDCl_3).



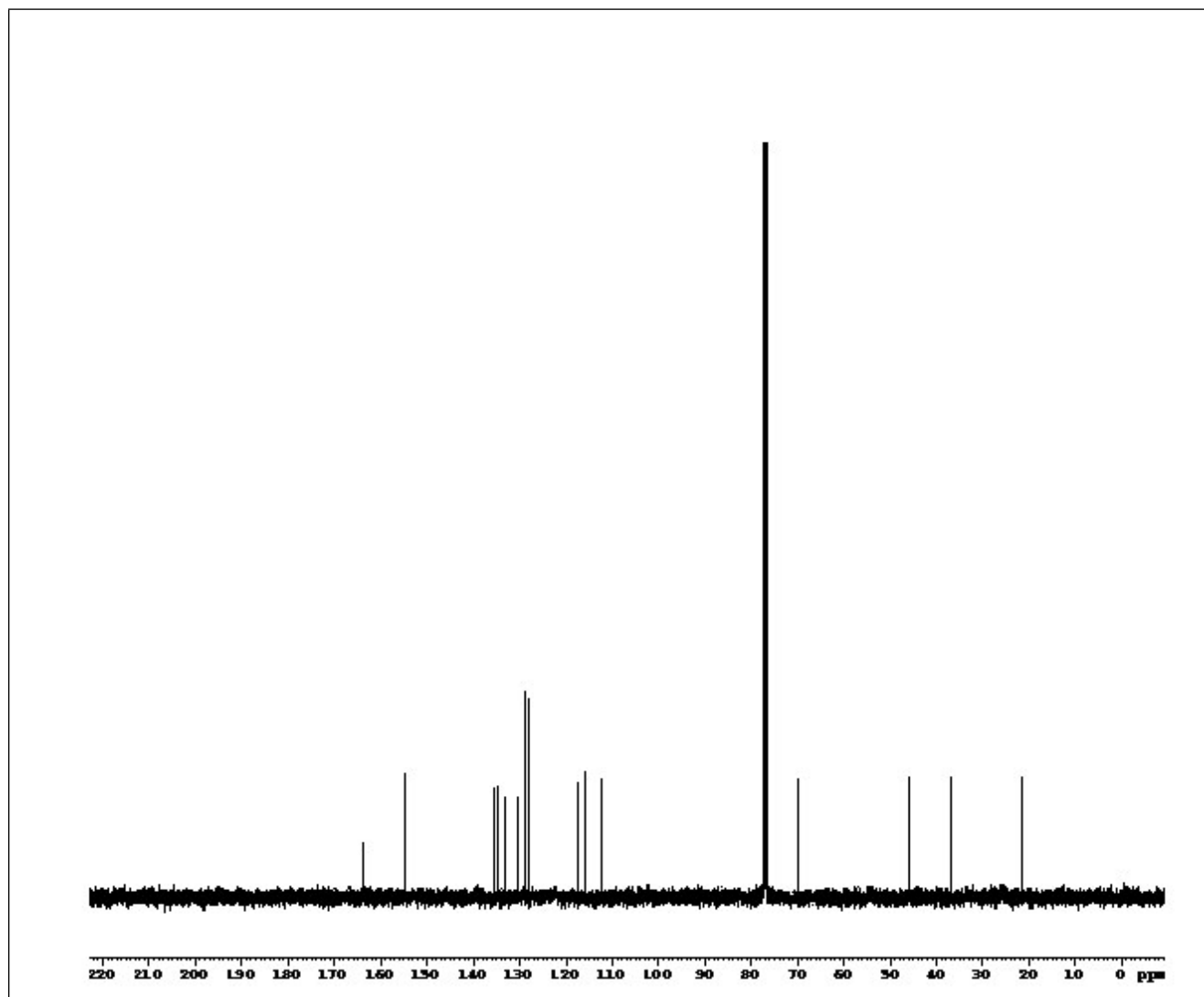
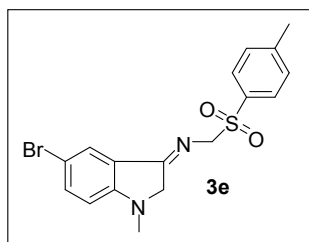
Compound 3d. ^{13}C NMR Spectrum (CDCl_3).



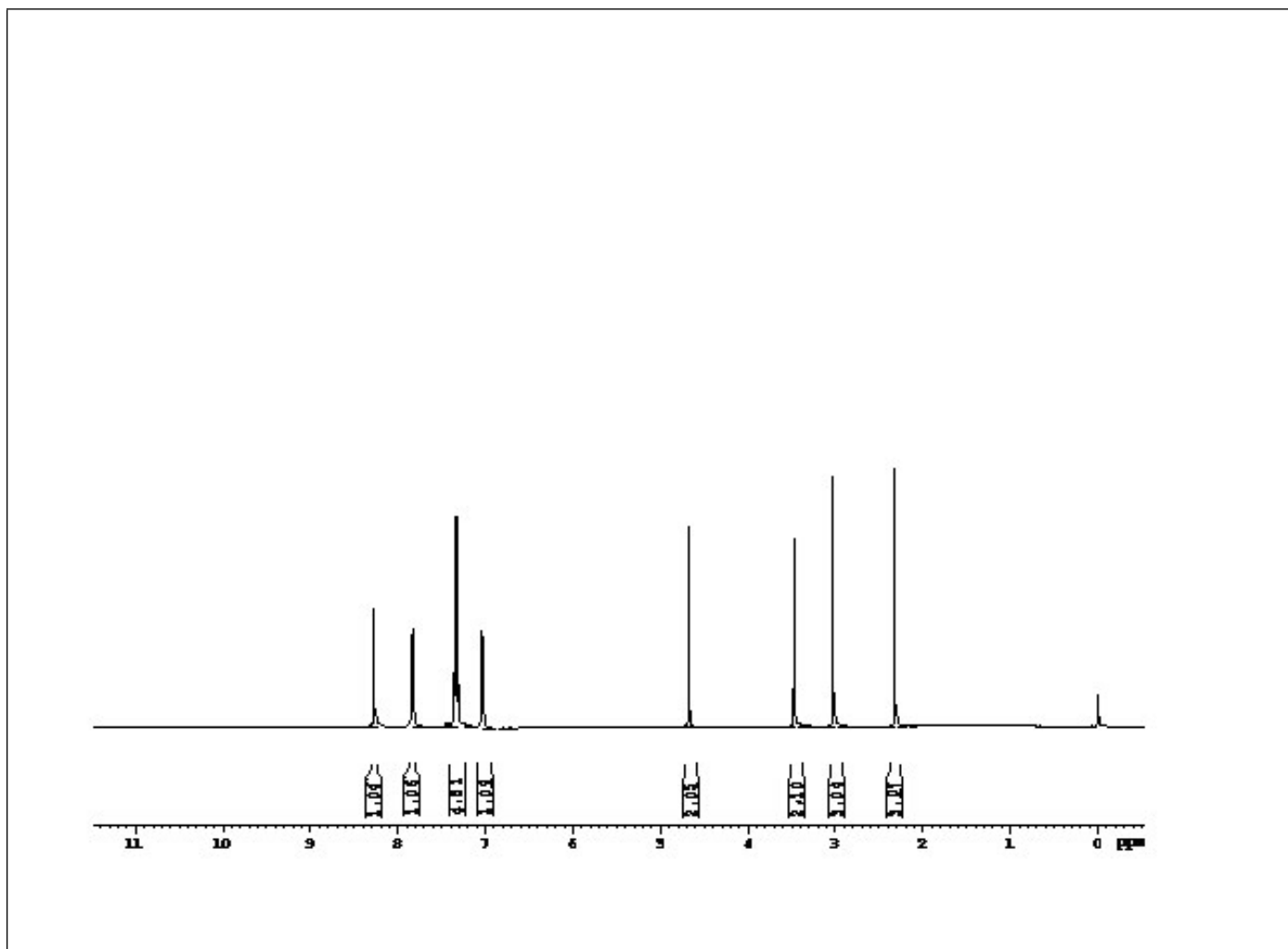
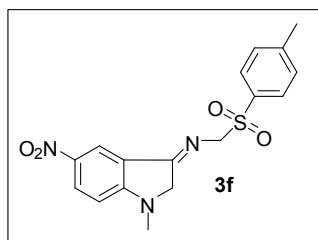
Compound 3e. ^1H NMR Spectrum (CDCl_3).



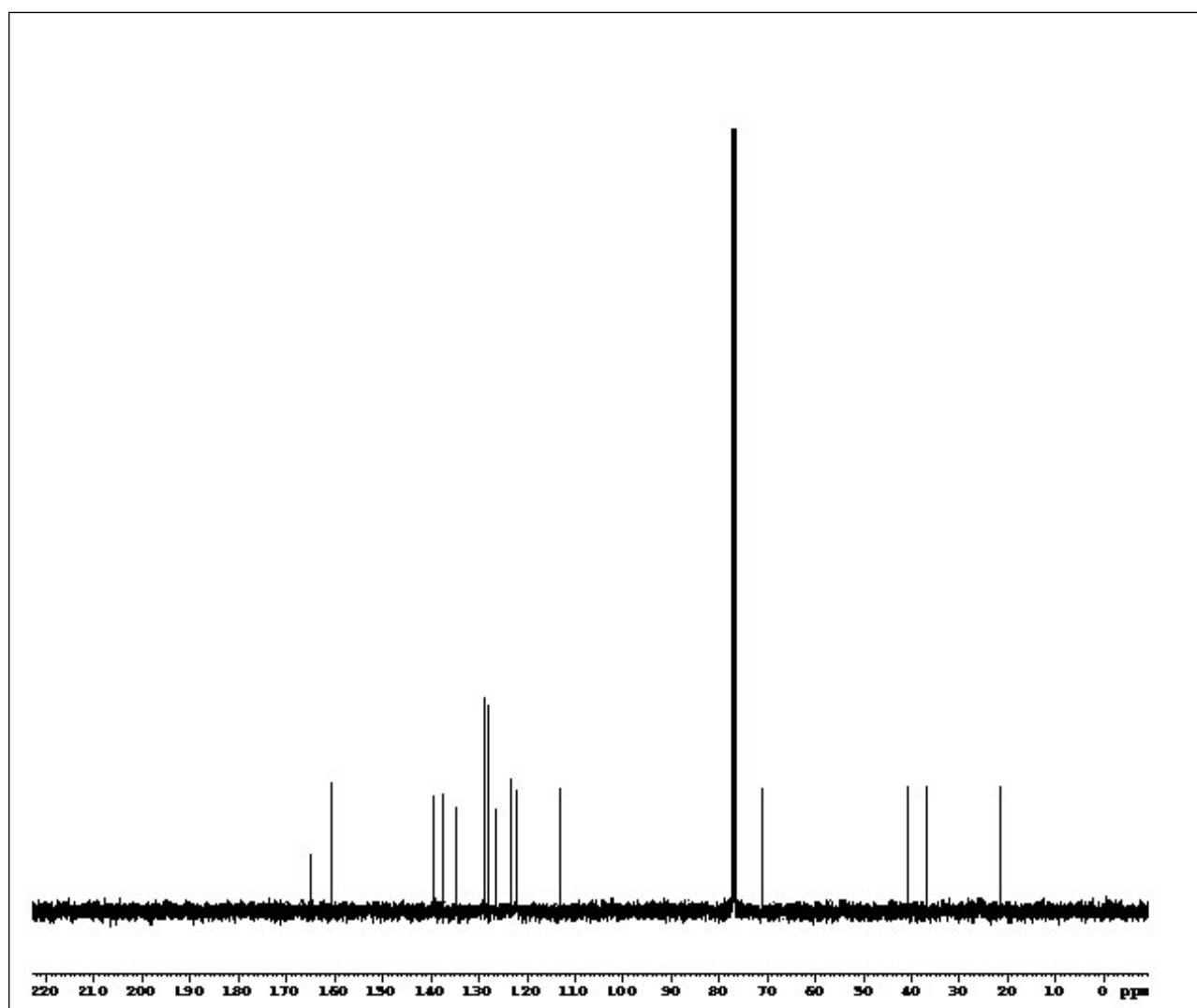
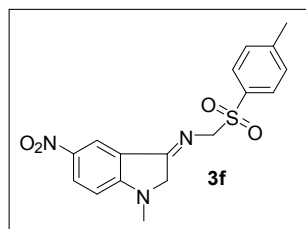
Compound 3e. ^{13}C NMR Spectrum (CDCl_3).



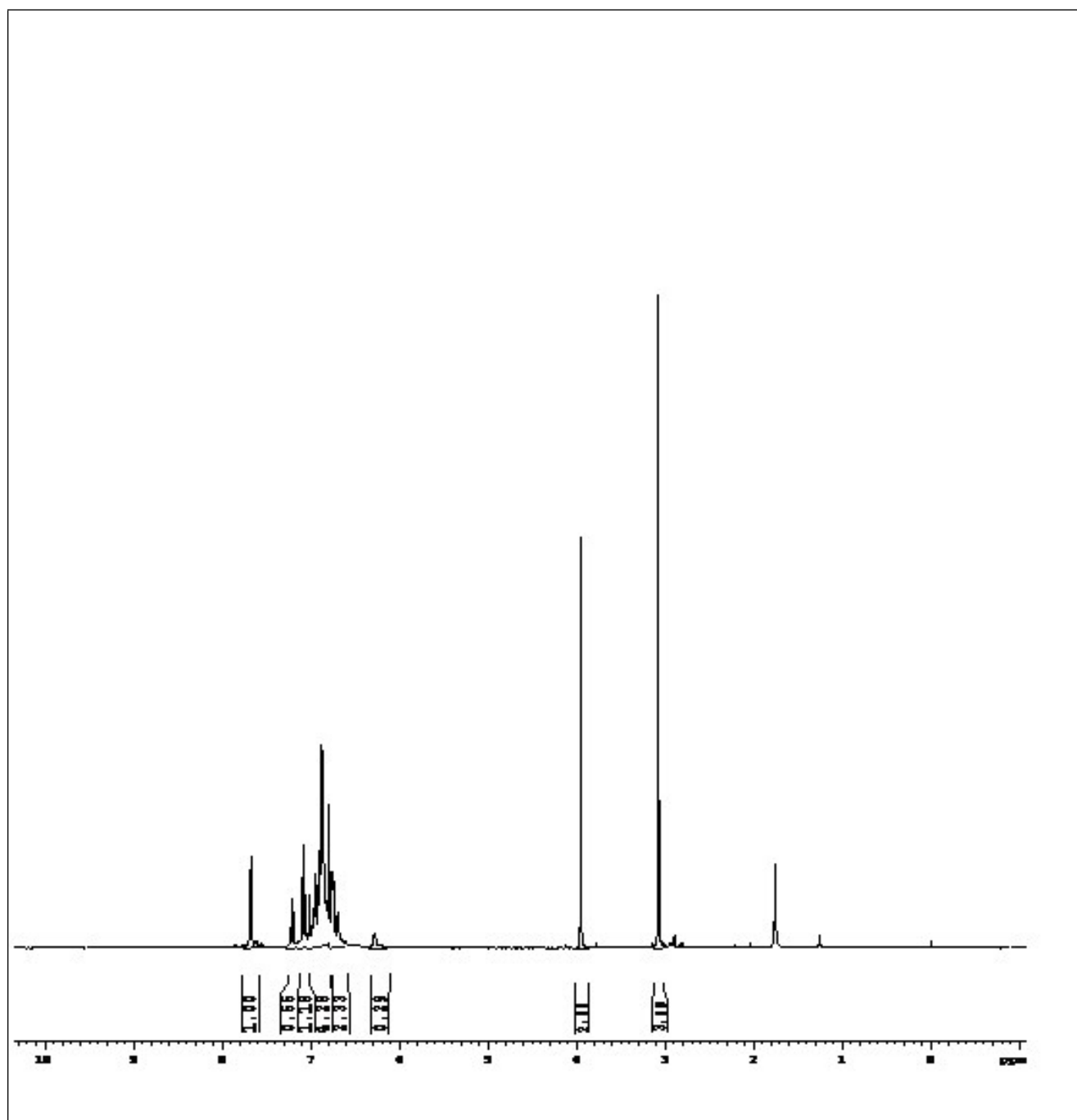
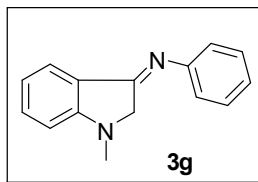
Compound 3f. ^1H NMR Spectrum (CDCl_3).



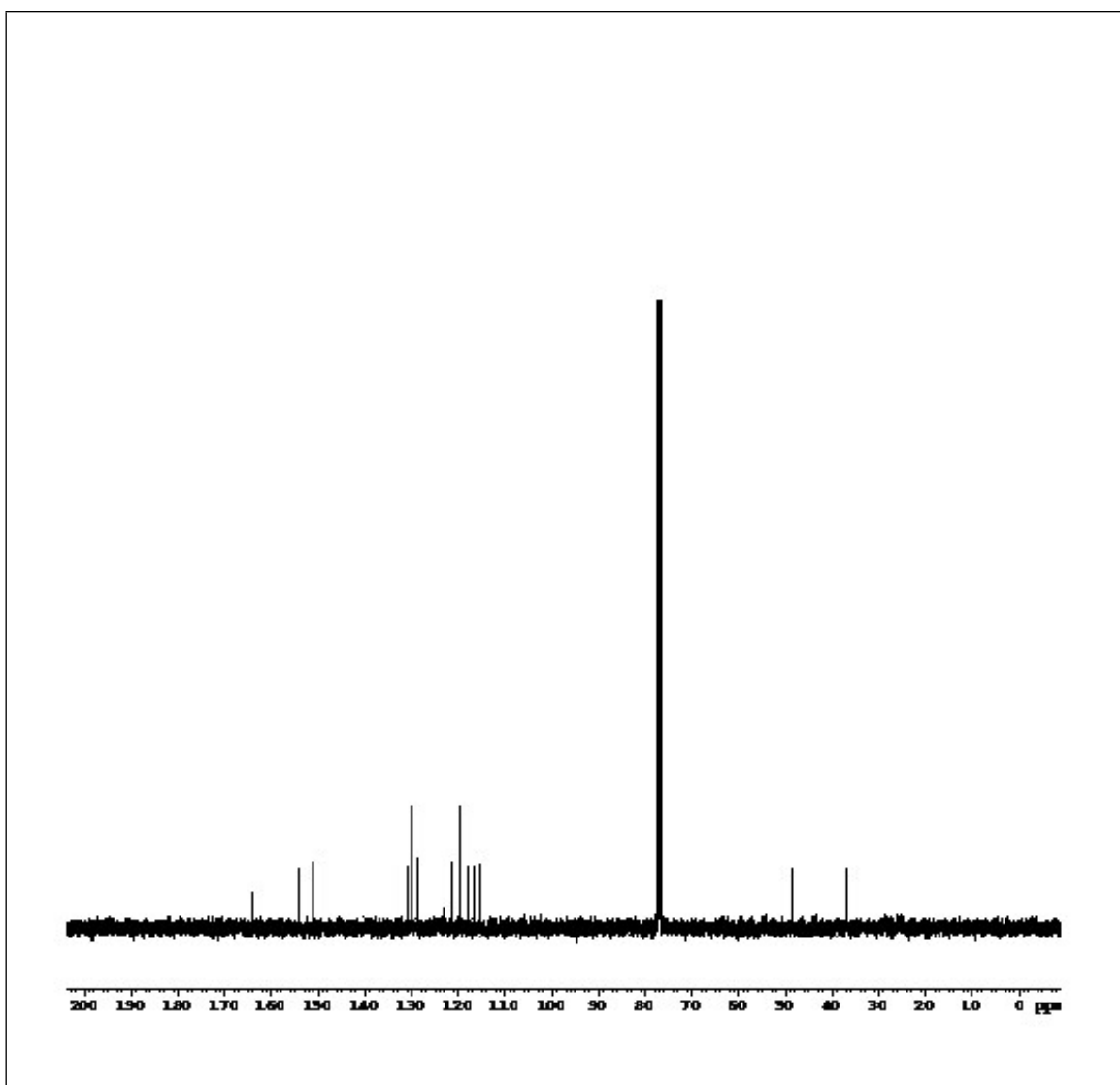
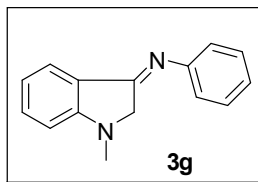
Compound 3f. ^{13}C NMR Spectrum (CDCl_3).



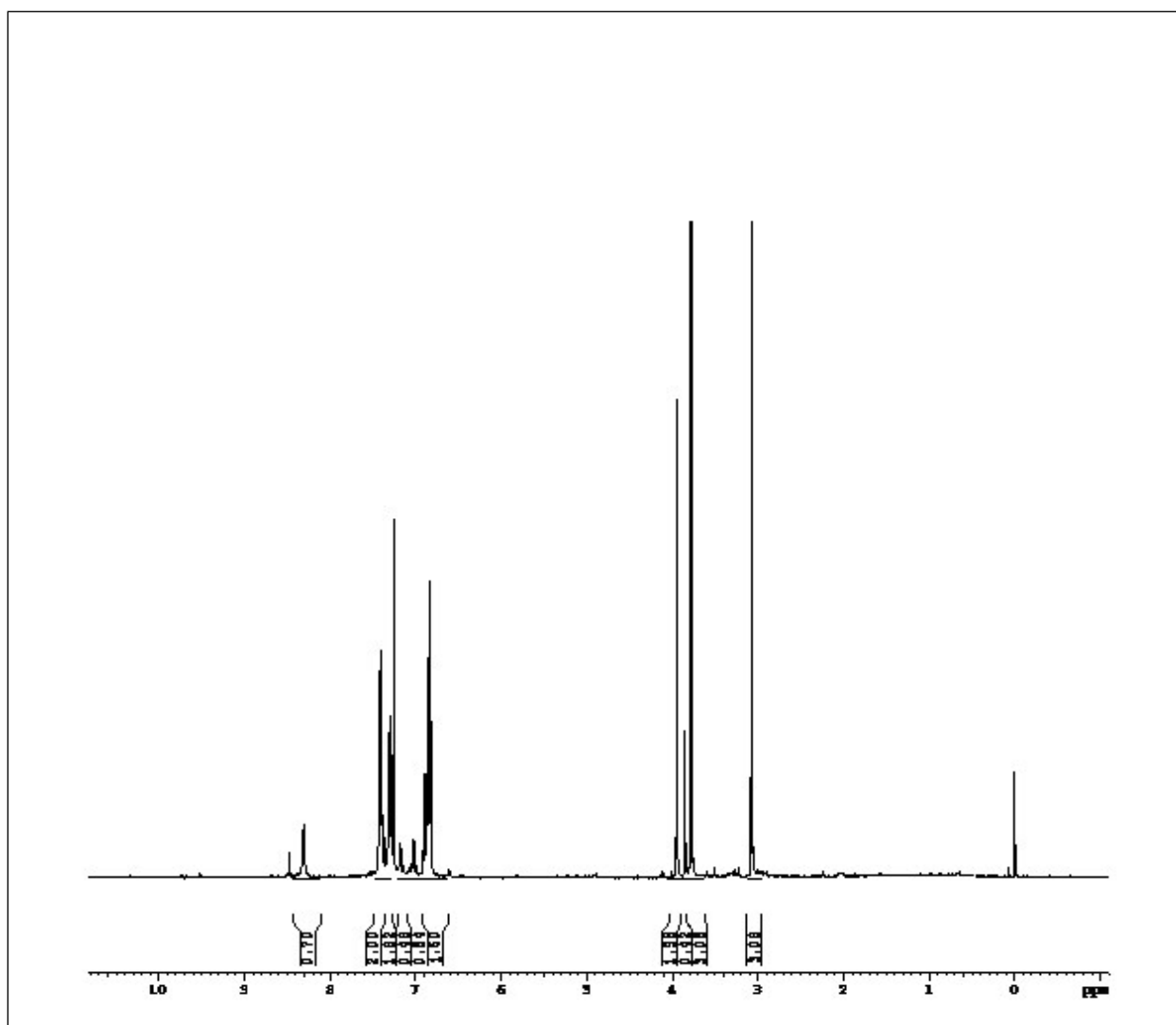
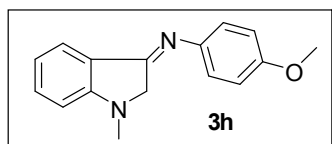
Compound **3g**. ^1H NMR Spectrum (CDCl_3).



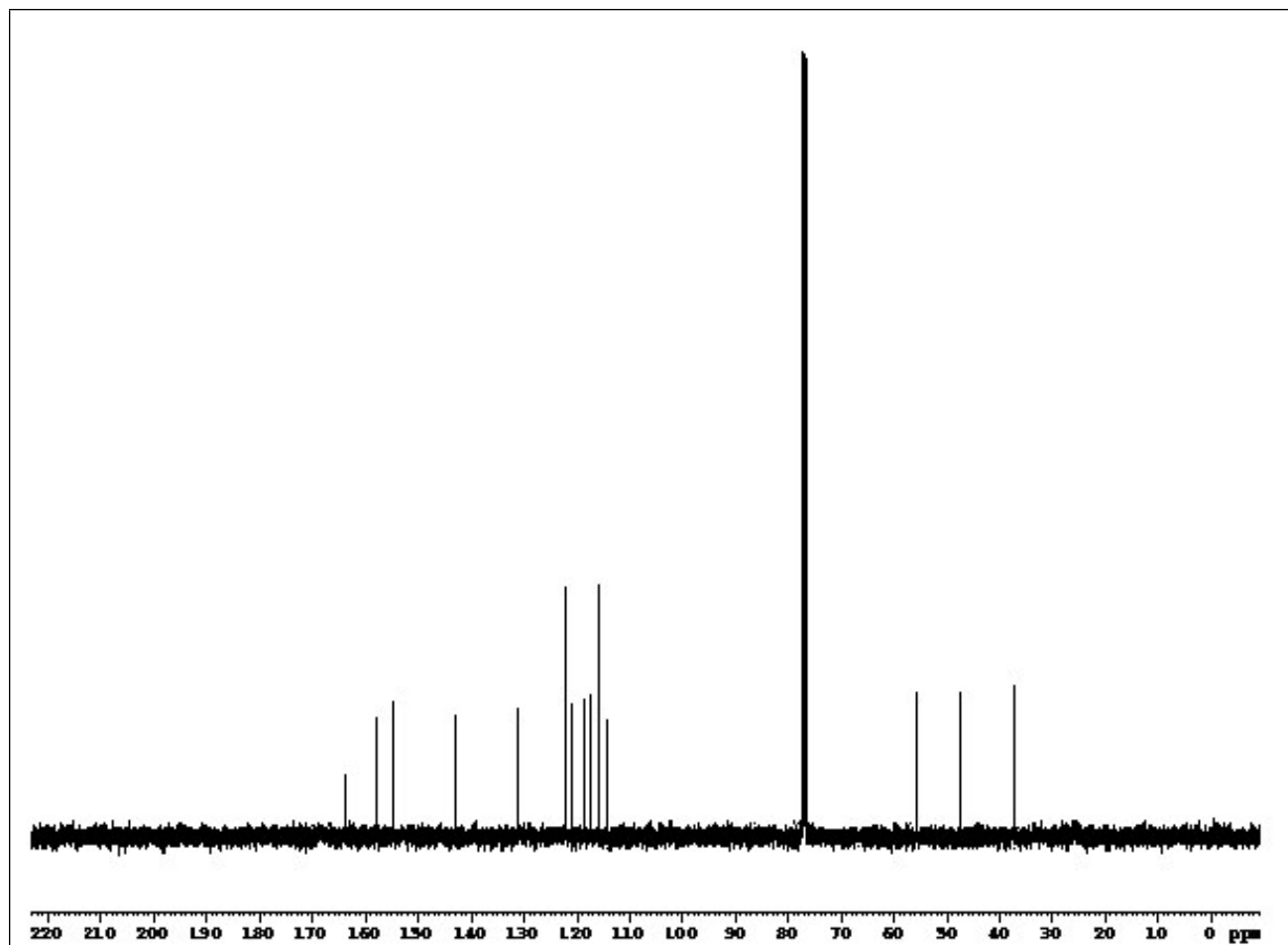
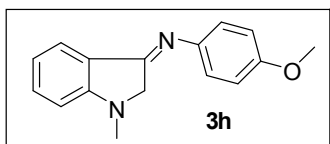
Compound **3g**. ^{13}C NMR Spectrum (CDCl_3).



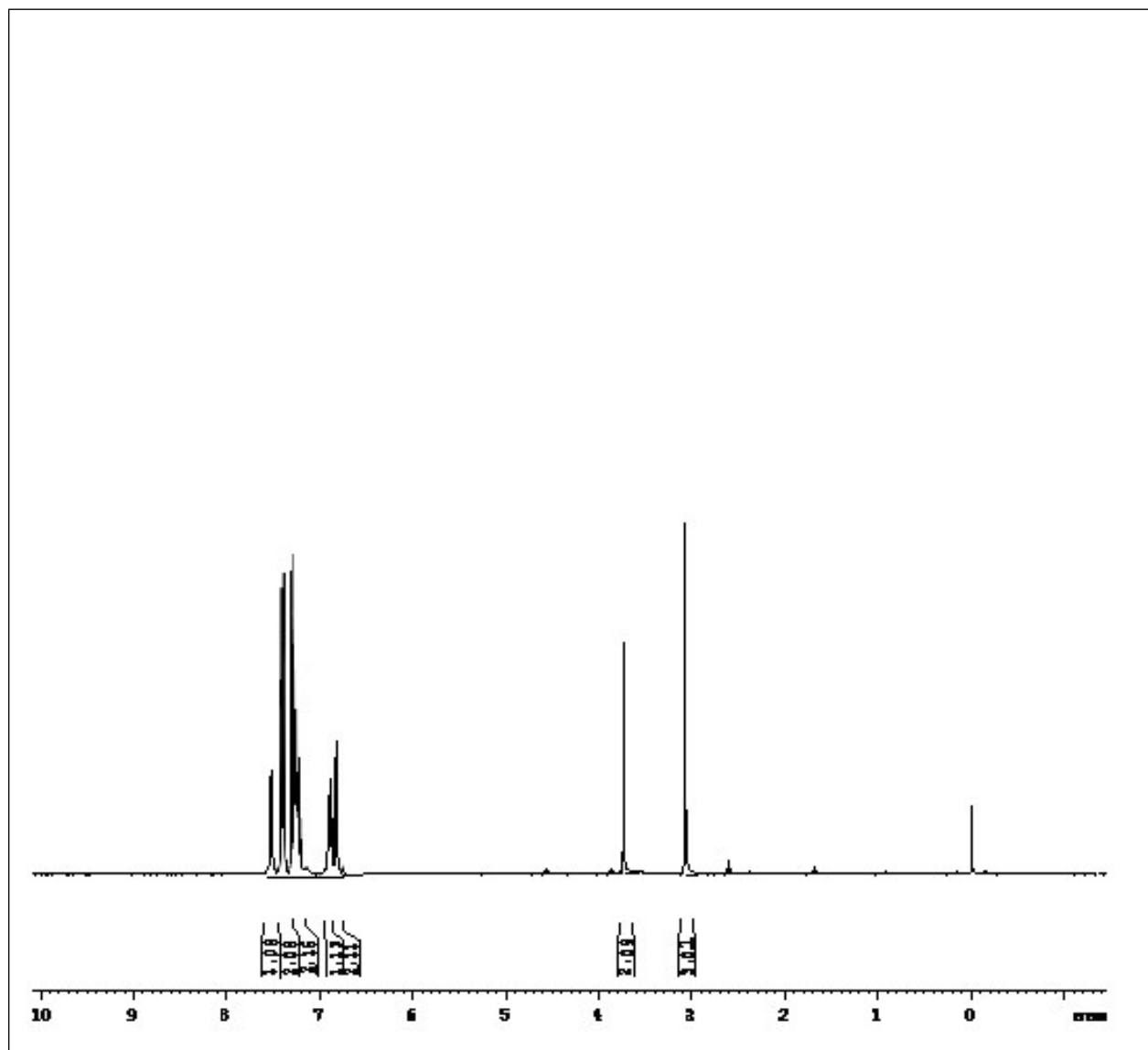
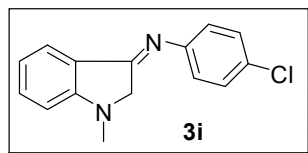
Compound 3h. ^1H NMR Spectrum (CDCl_3).



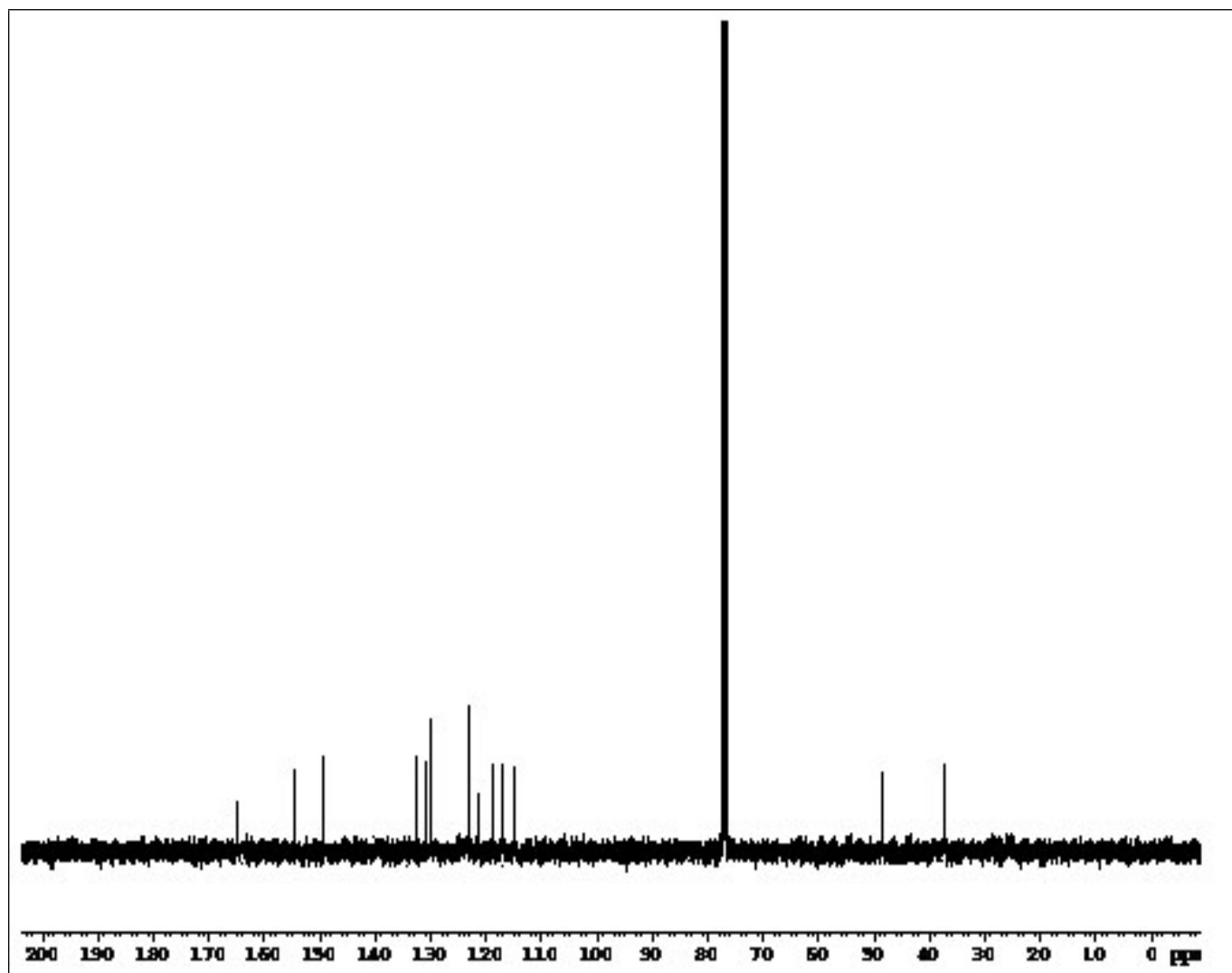
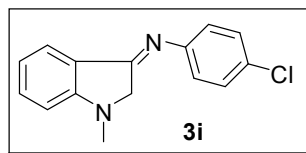
Compound 3h. ^{13}C NMR Spectrum (CDCl_3).



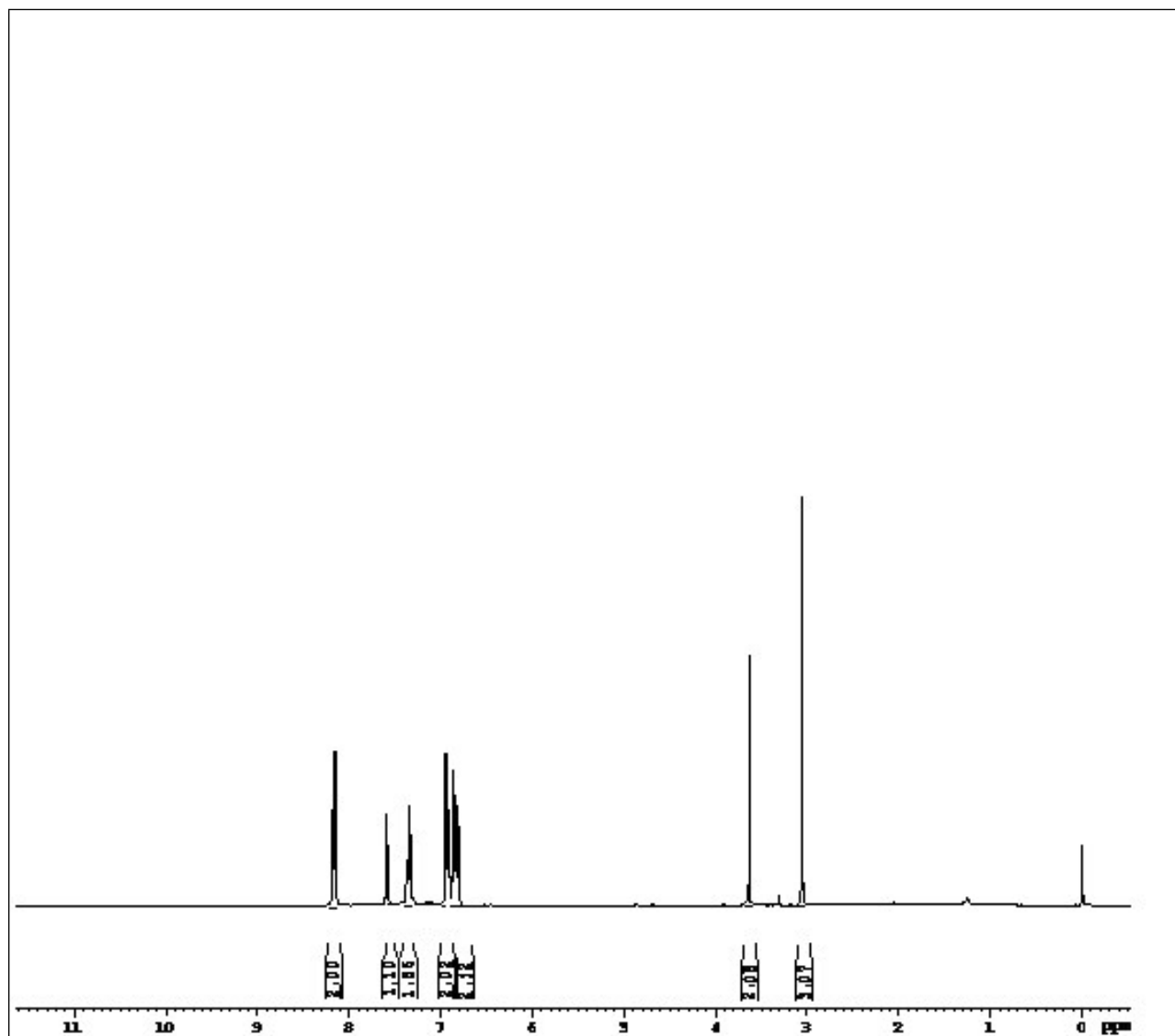
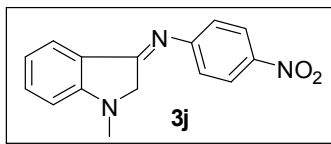
Compound 3i. ^1H NMR Spectrum (CDCl_3).



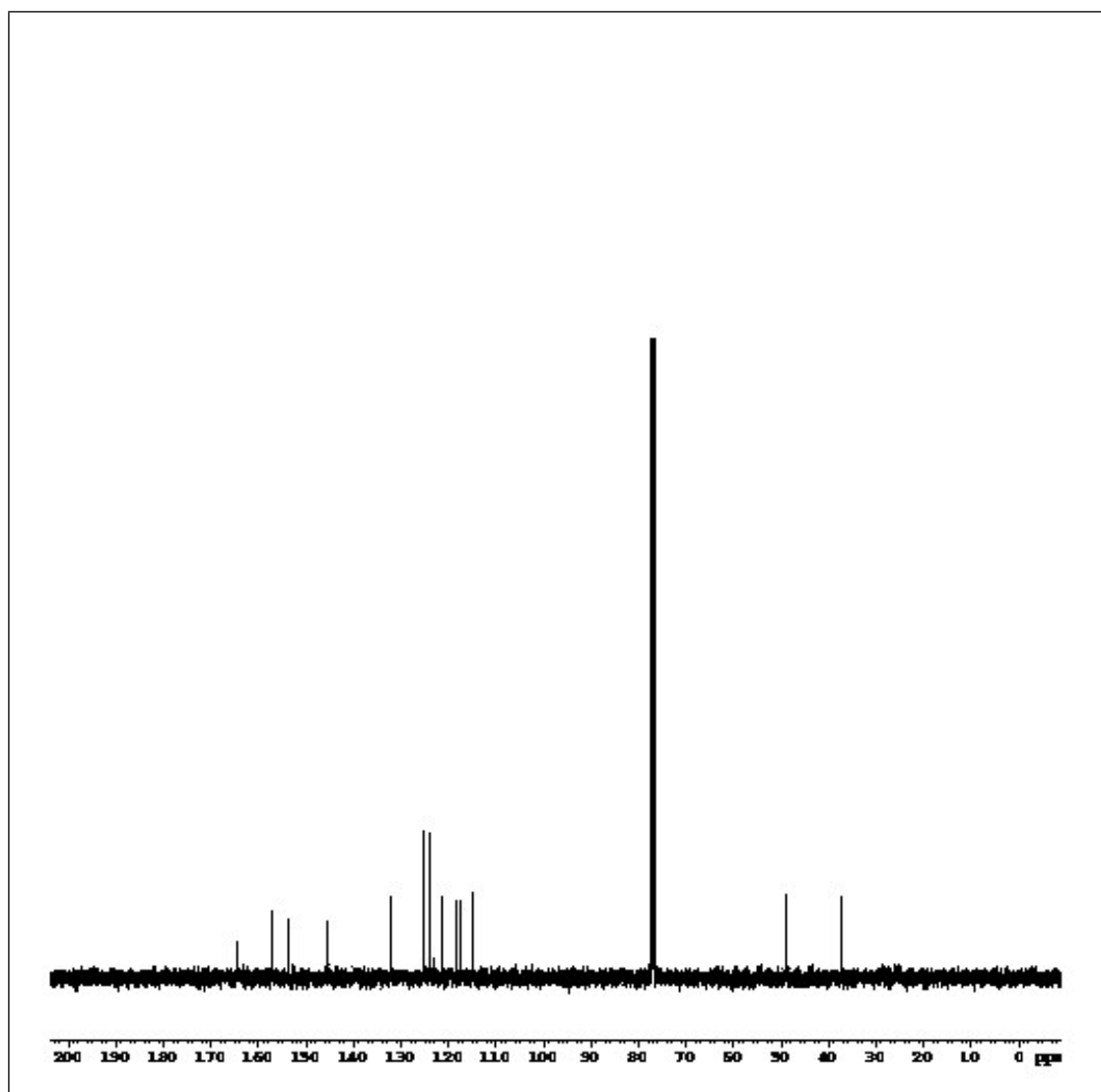
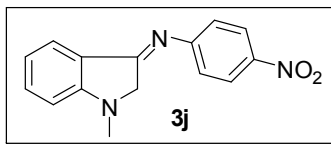
Compound 3i. ^{13}C NMR Spectrum (CDCl_3).



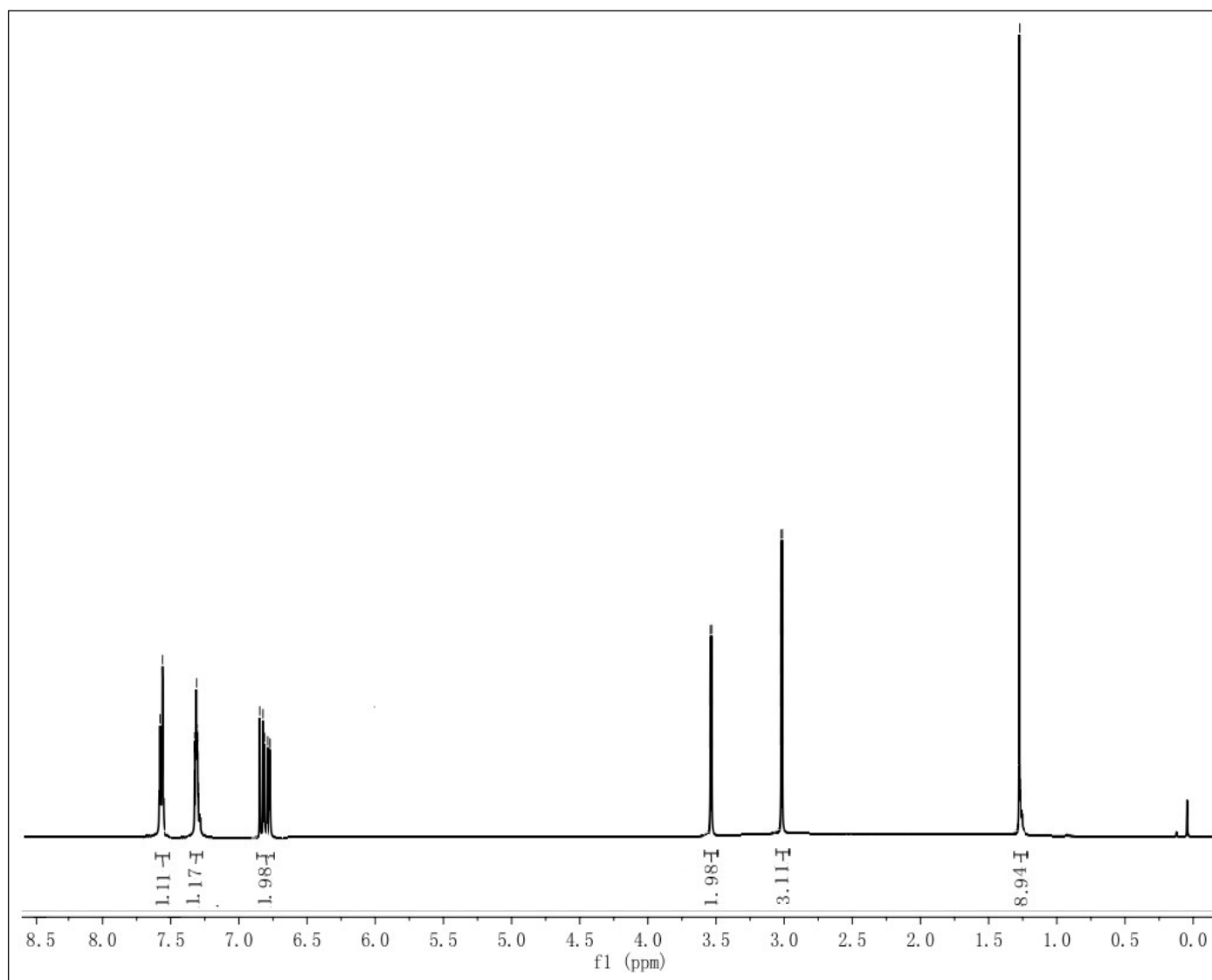
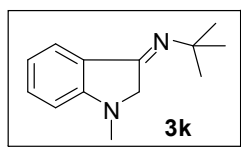
Compound 3j. ^1H NMR Spectrum (CDCl_3).



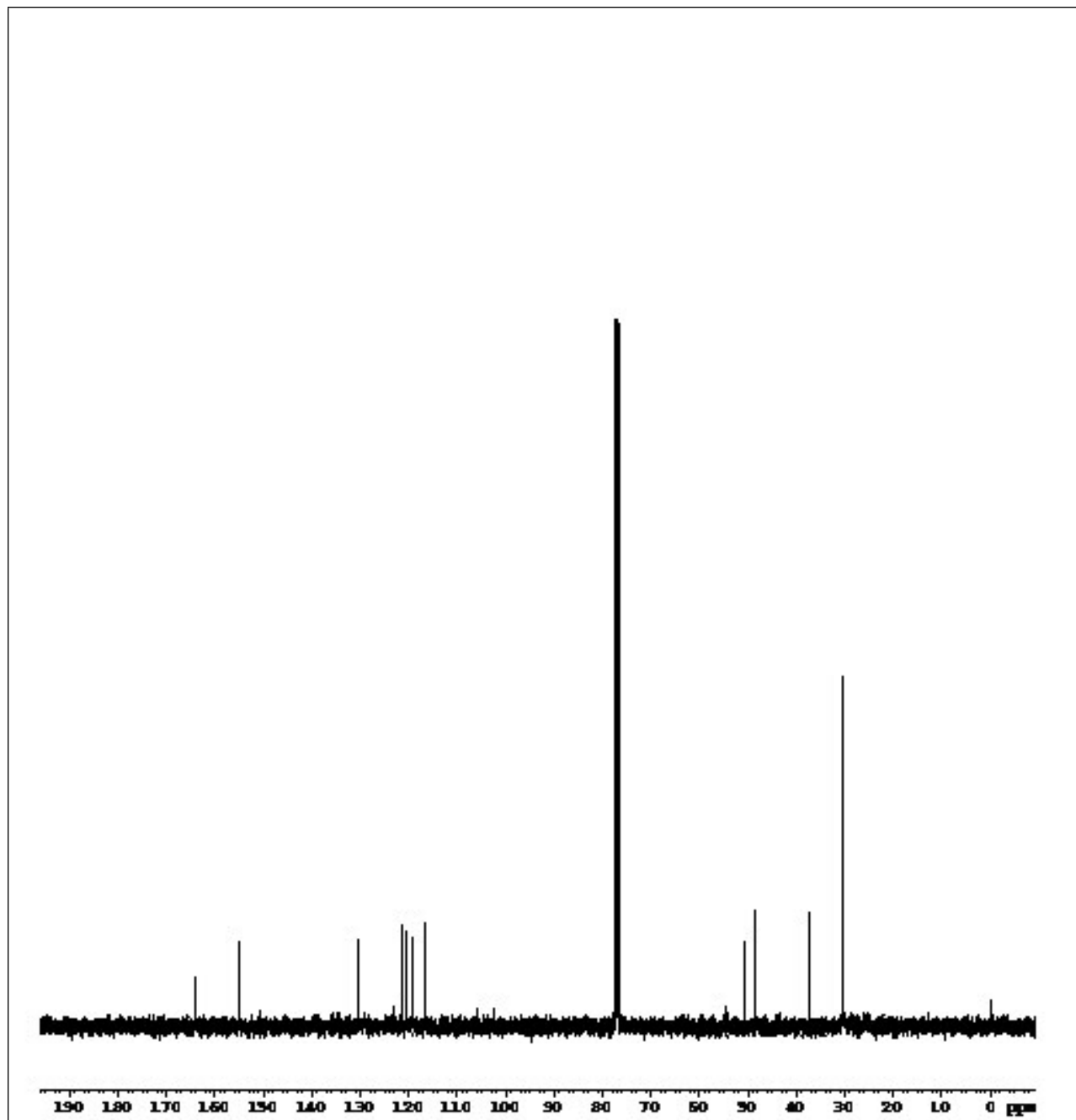
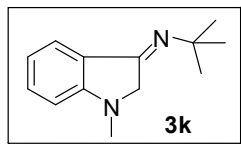
Compound **3j**. ^{13}C NMR Spectrum (CDCl_3).



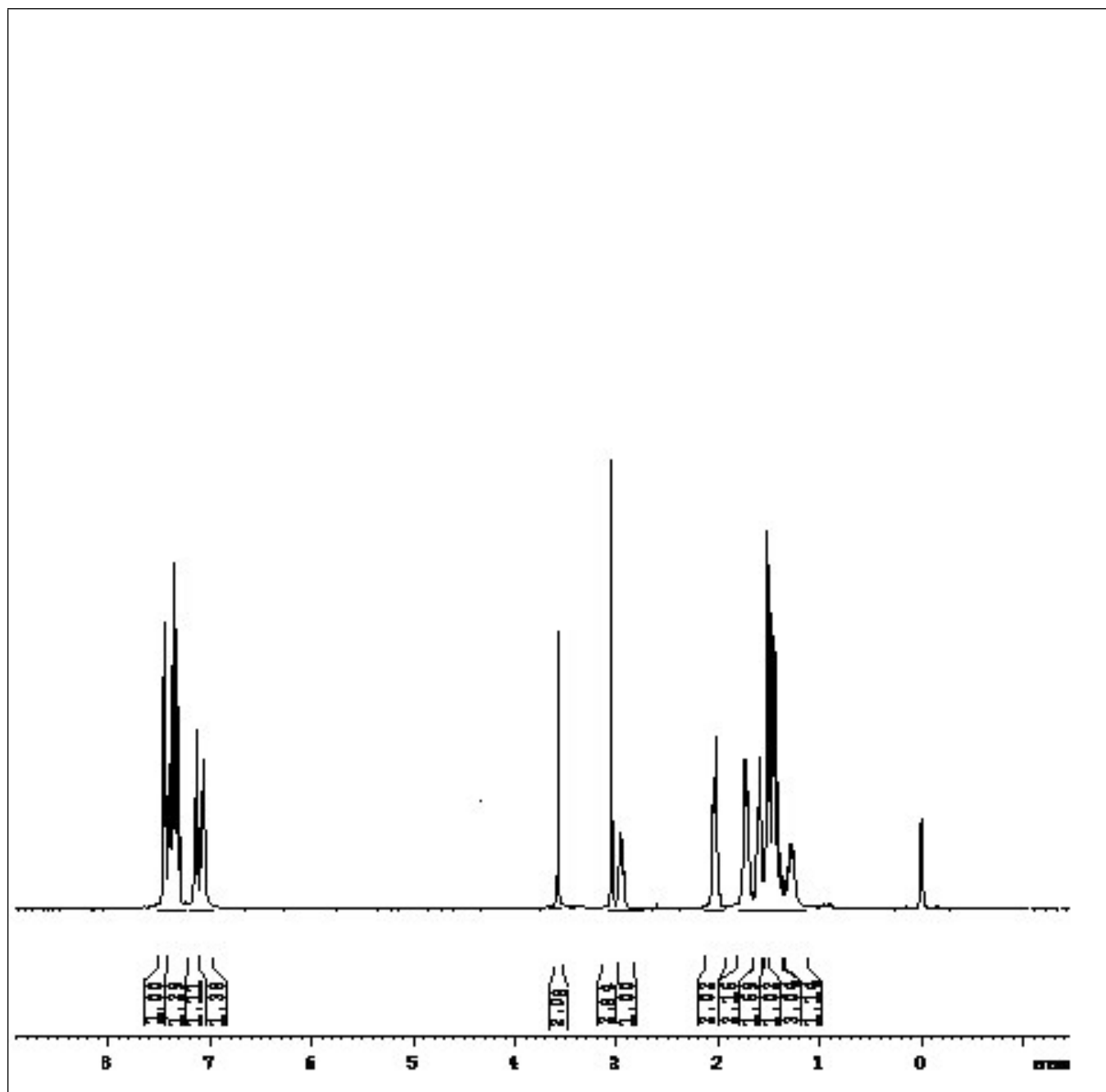
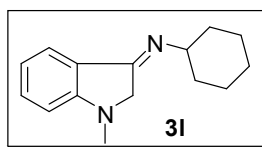
Compound 3k. ^1H NMR Spectrum (CDCl_3).



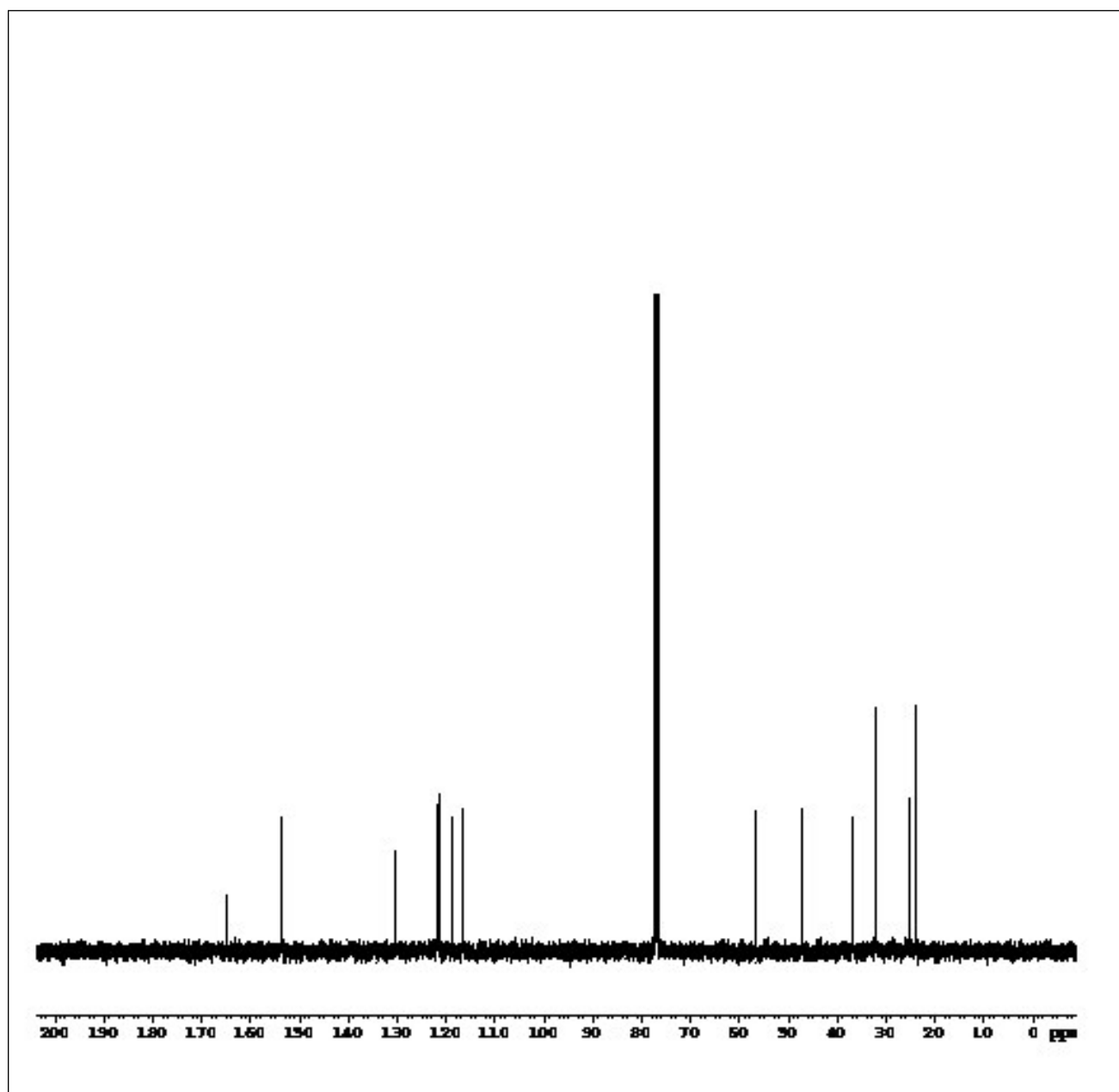
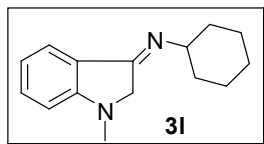
Compound 3k. ^{13}C NMR Spectrum (CDCl_3).



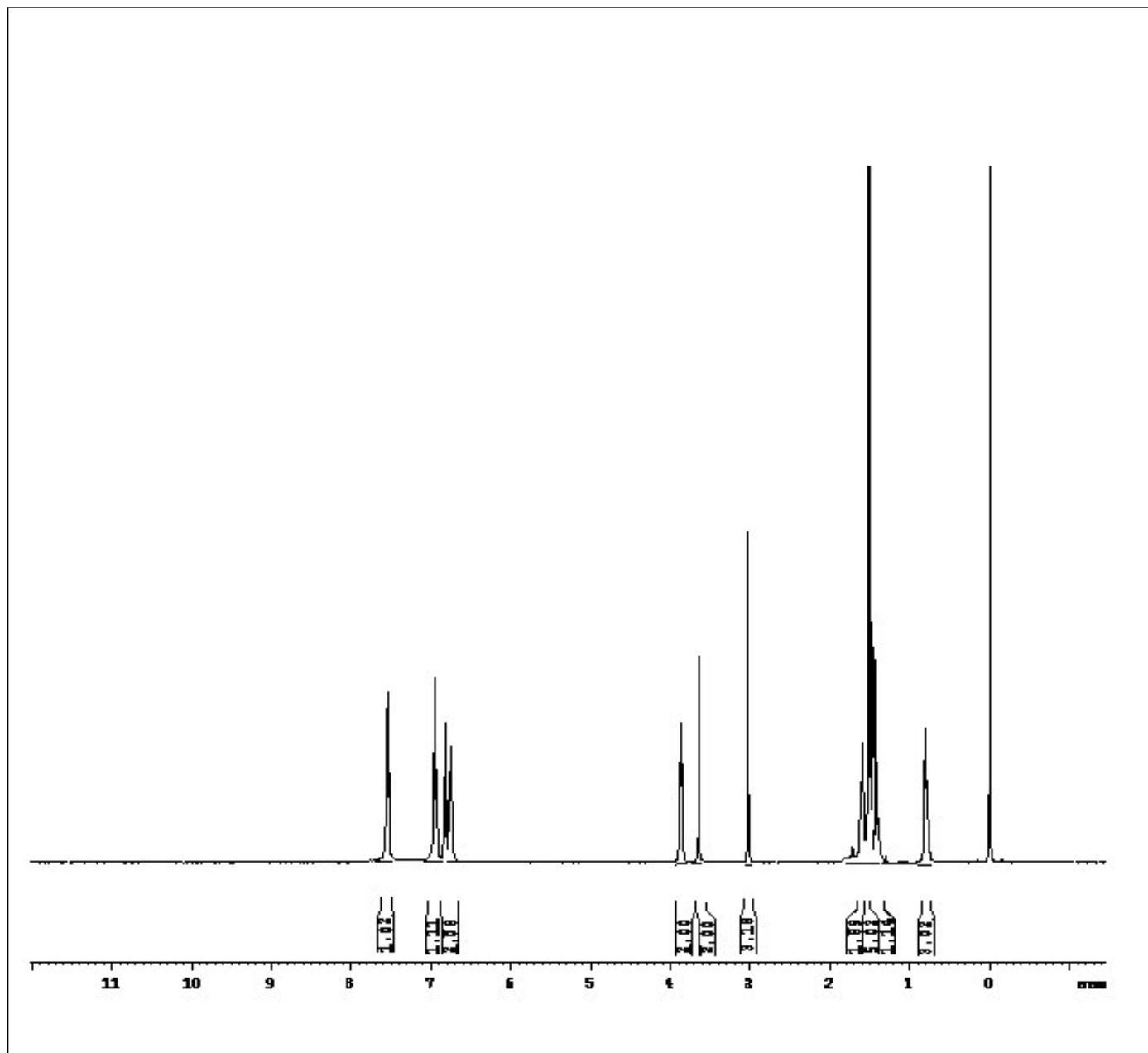
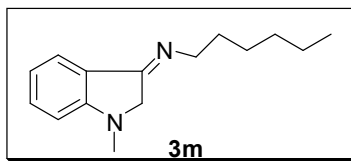
Compound 31. ^1H NMR Spectrum (CDCl_3).



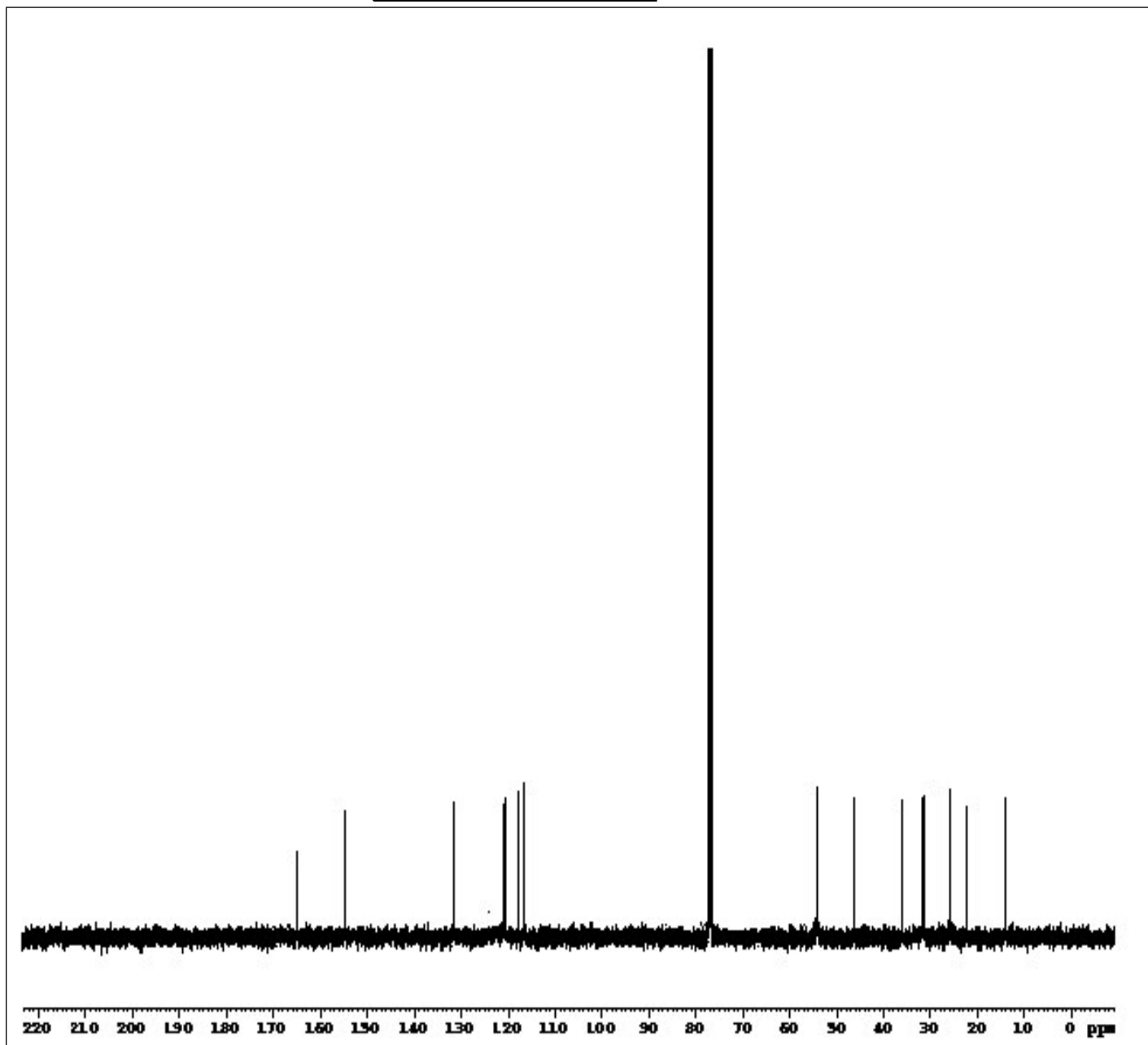
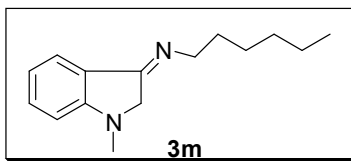
Compound 31. ^{13}C NMR Spectrum (CDCl_3).



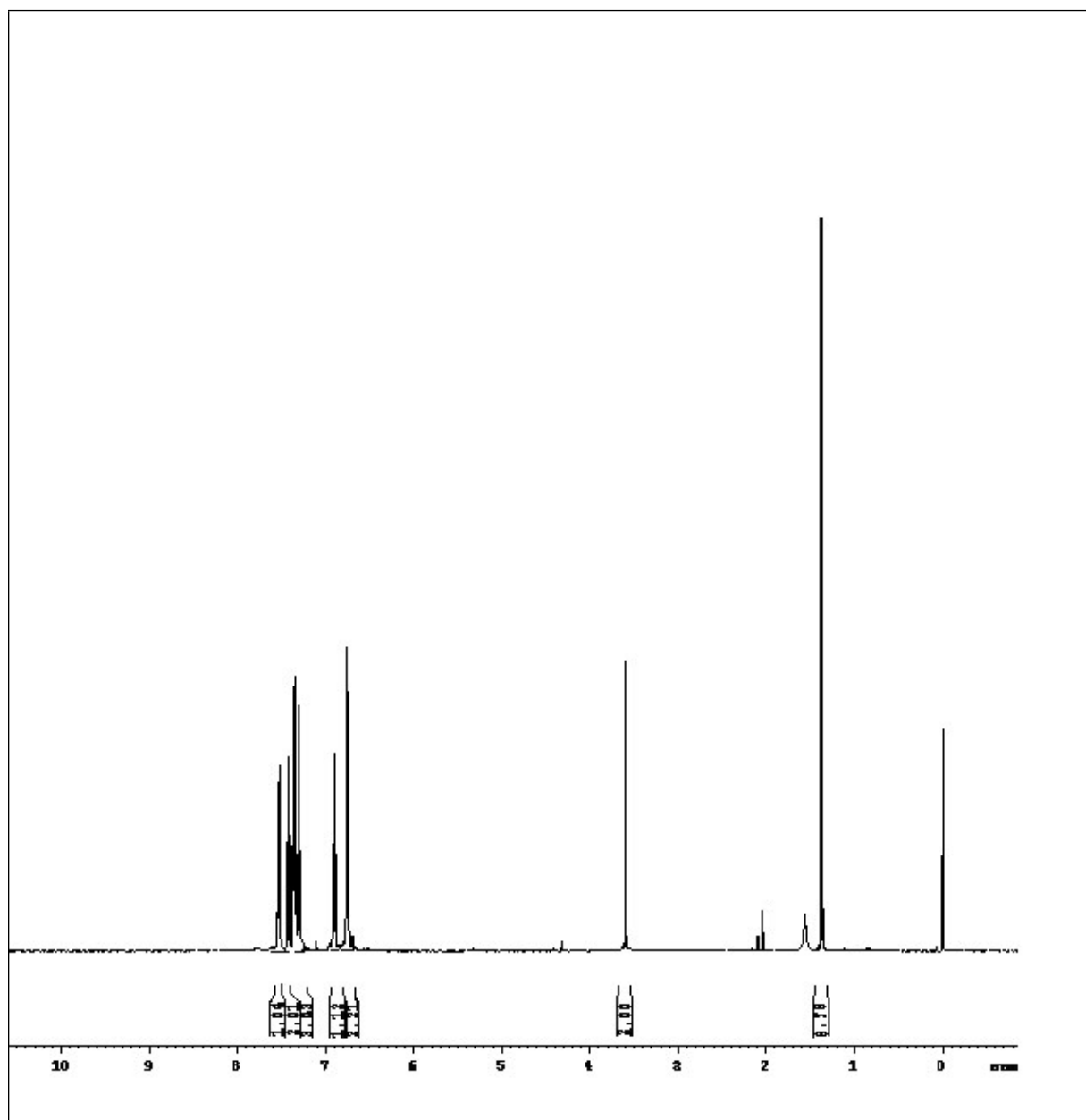
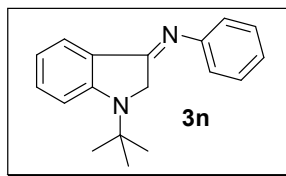
Compound 3m. ^1H NMR Spectrum (CDCl_3).



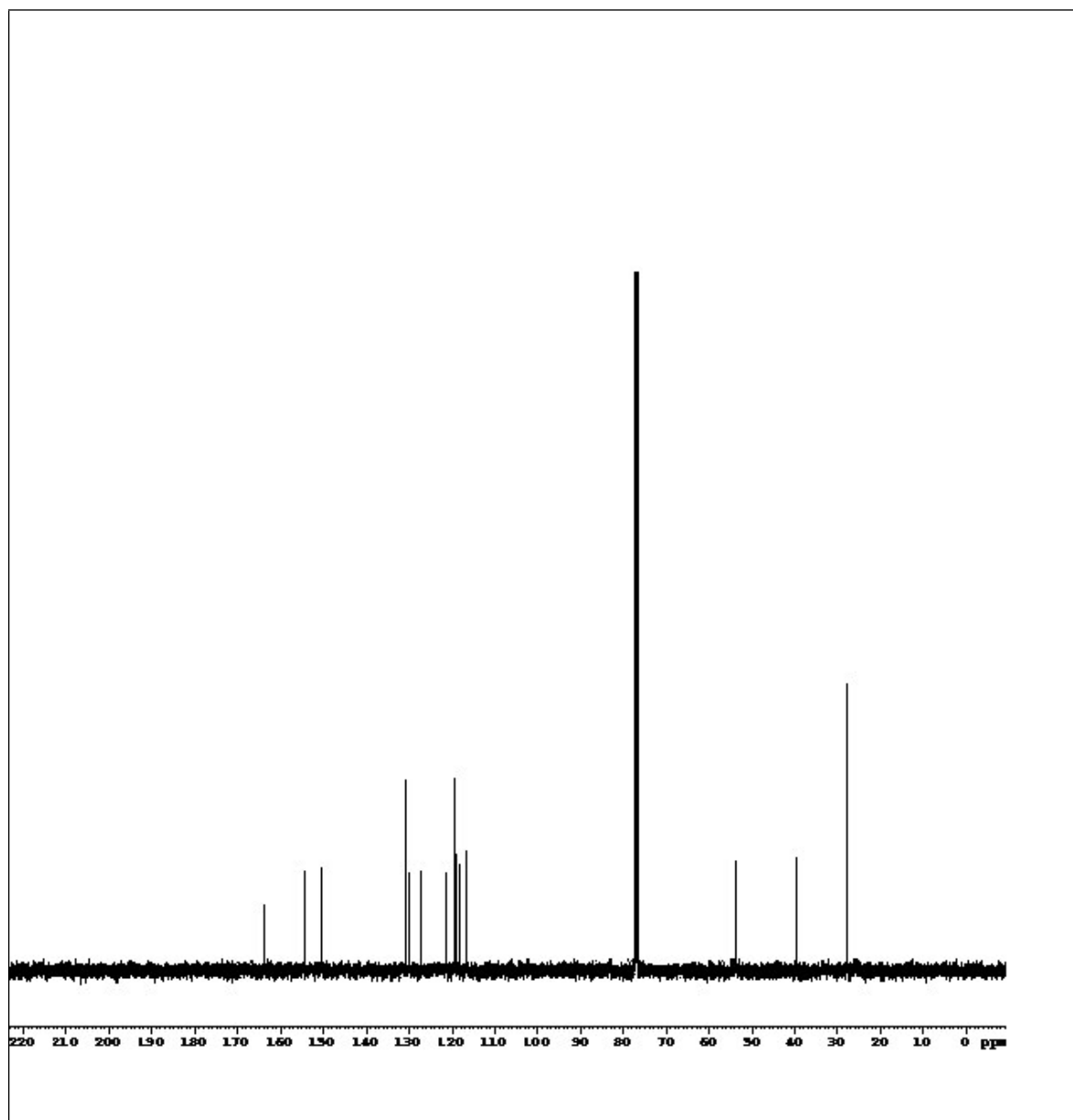
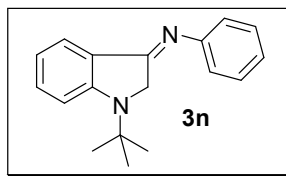
Compound 3m. ^{13}C NMR Spectrum (CDCl_3).



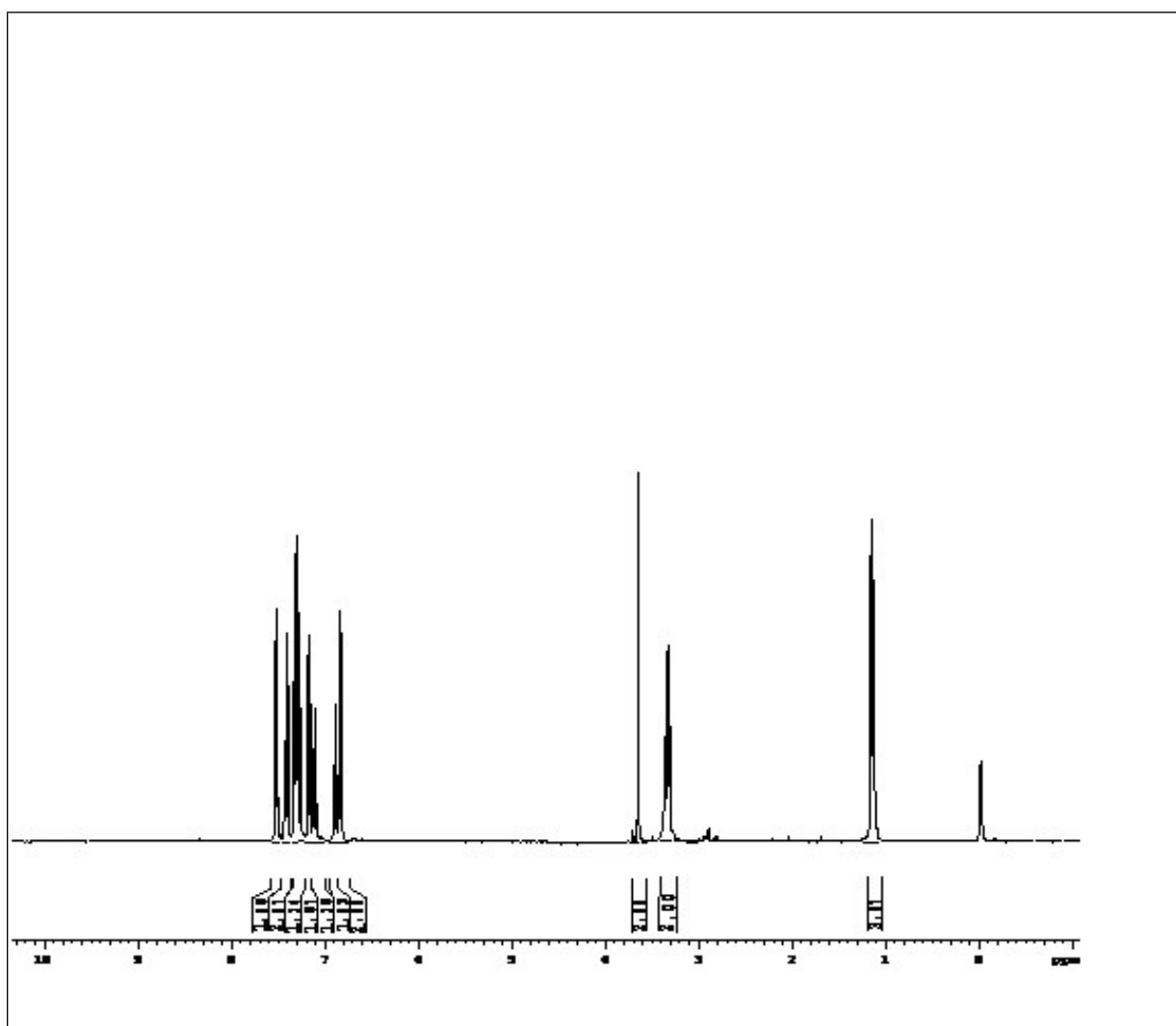
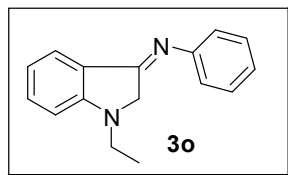
Compound 3n. ^1H NMR Spectrum (CDCl_3).



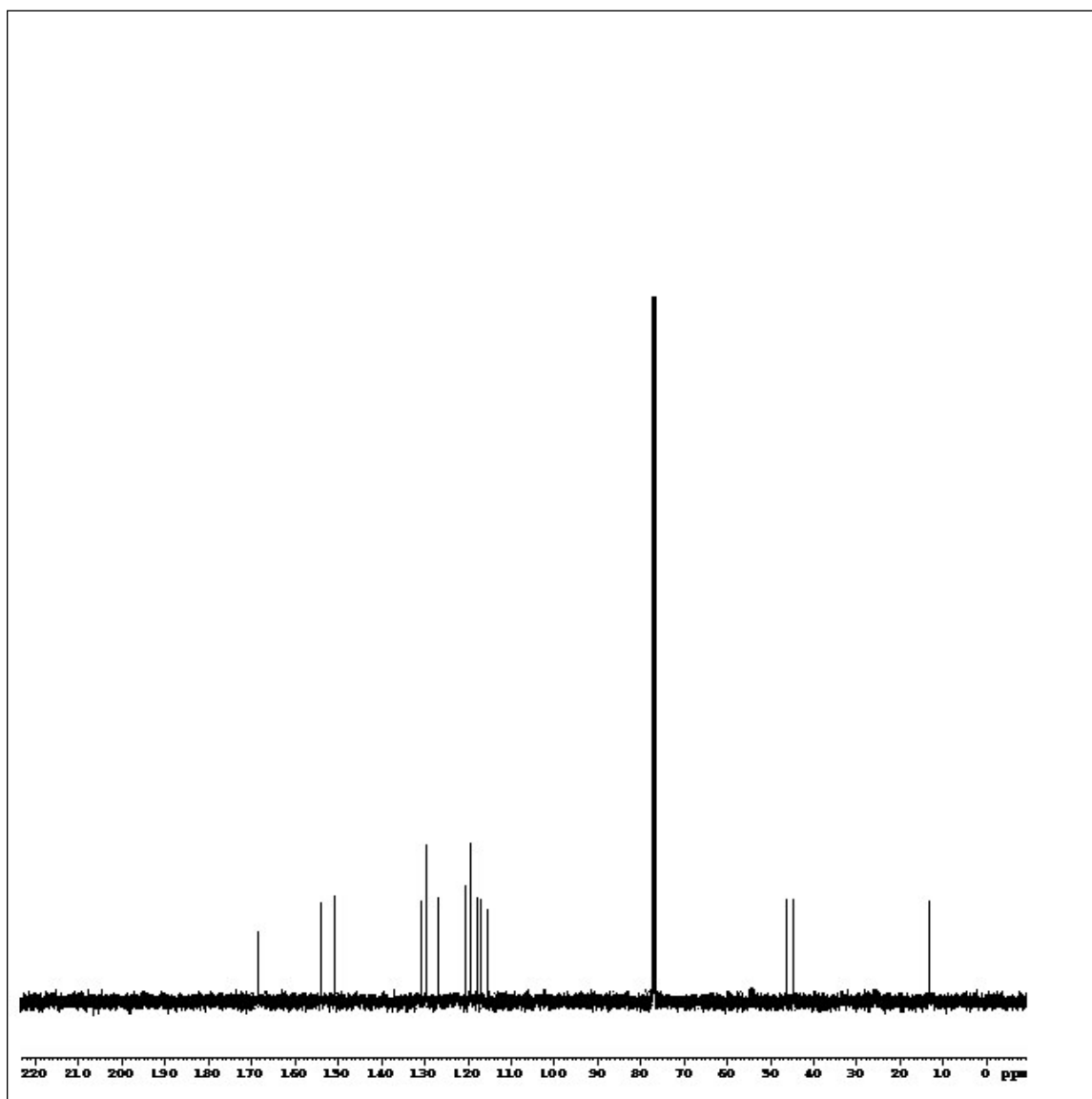
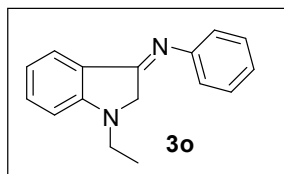
Compound 3n. ^{13}C NMR Spectrum (CDCl_3).



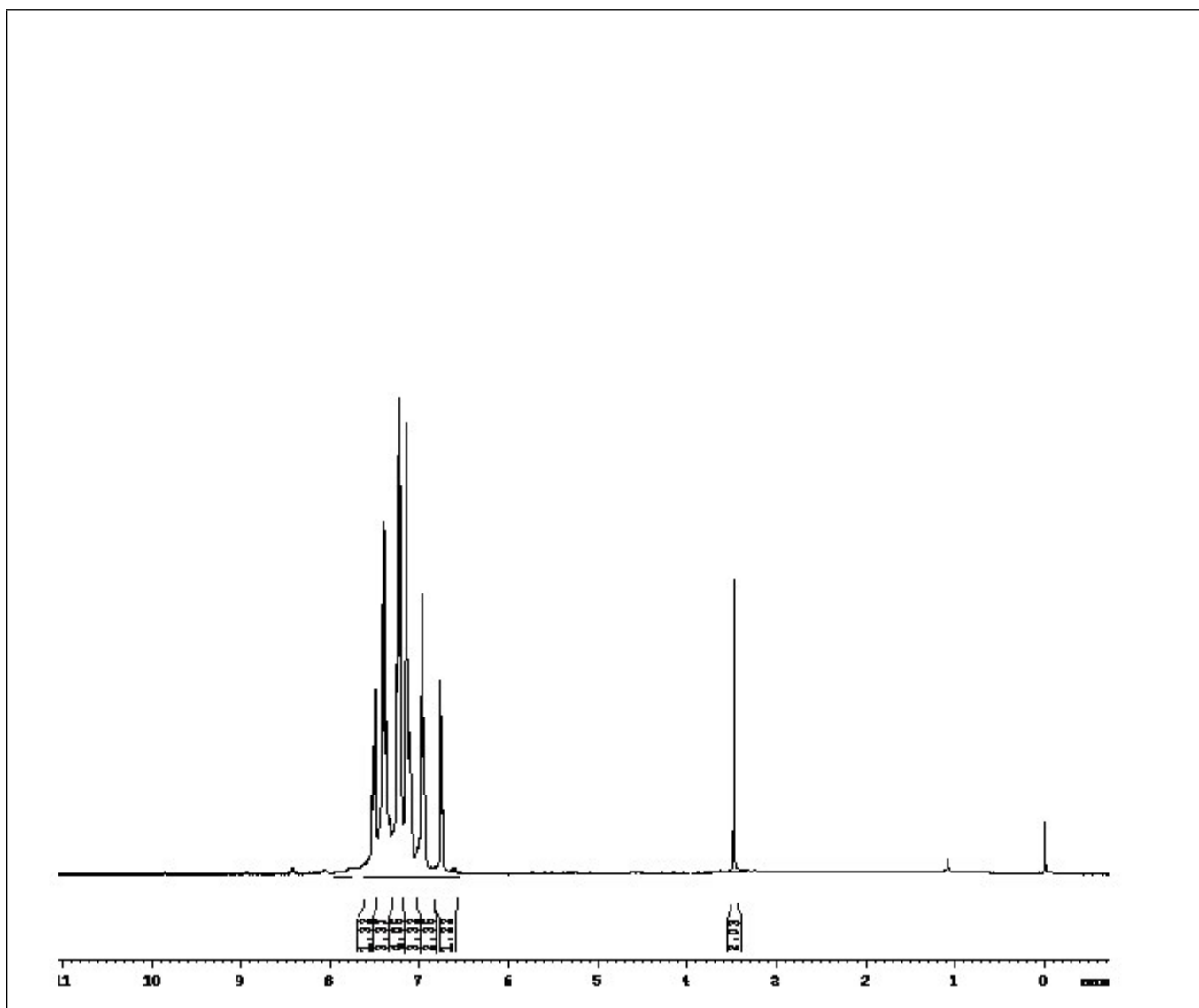
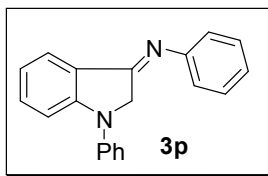
Compound 3o. ^1H NMR Spectrum (CDCl_3).



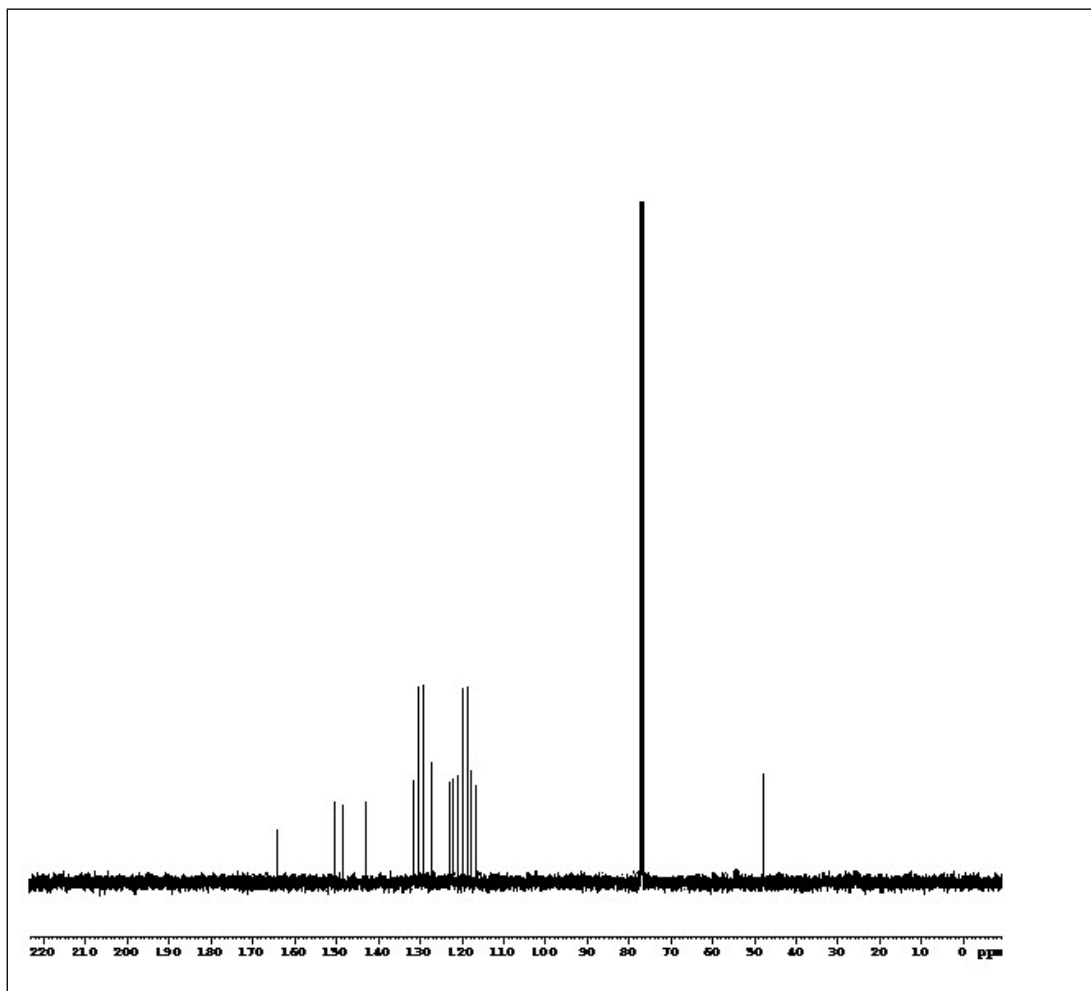
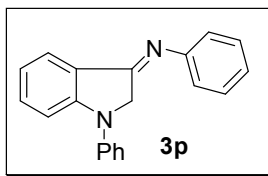
Compound 3o. ^{13}C NMR Spectrum (CDCl_3).



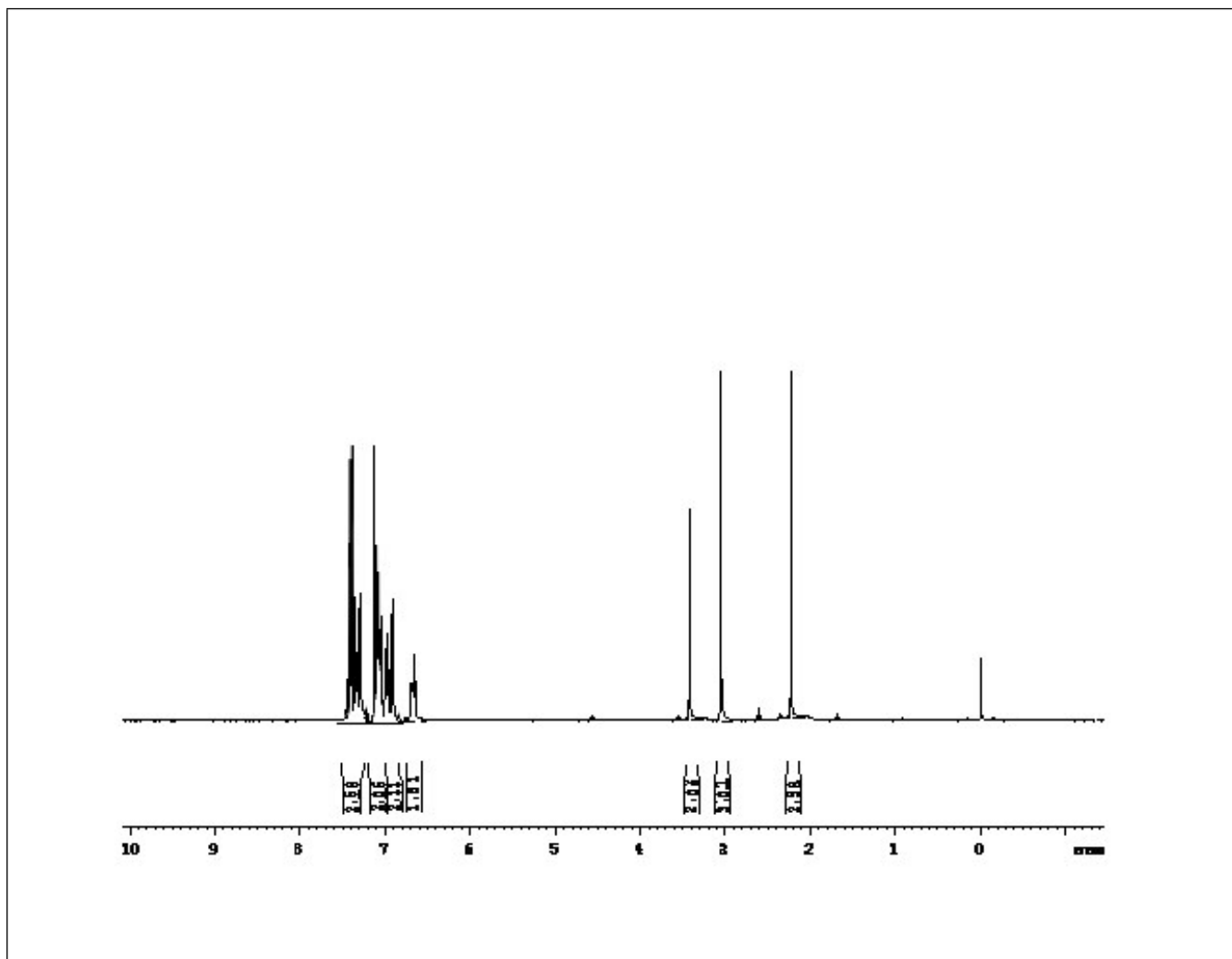
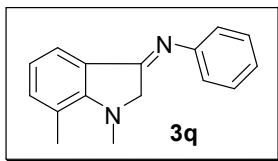
Compound 3p. ¹H NMR Spectrum (CDCl₃).



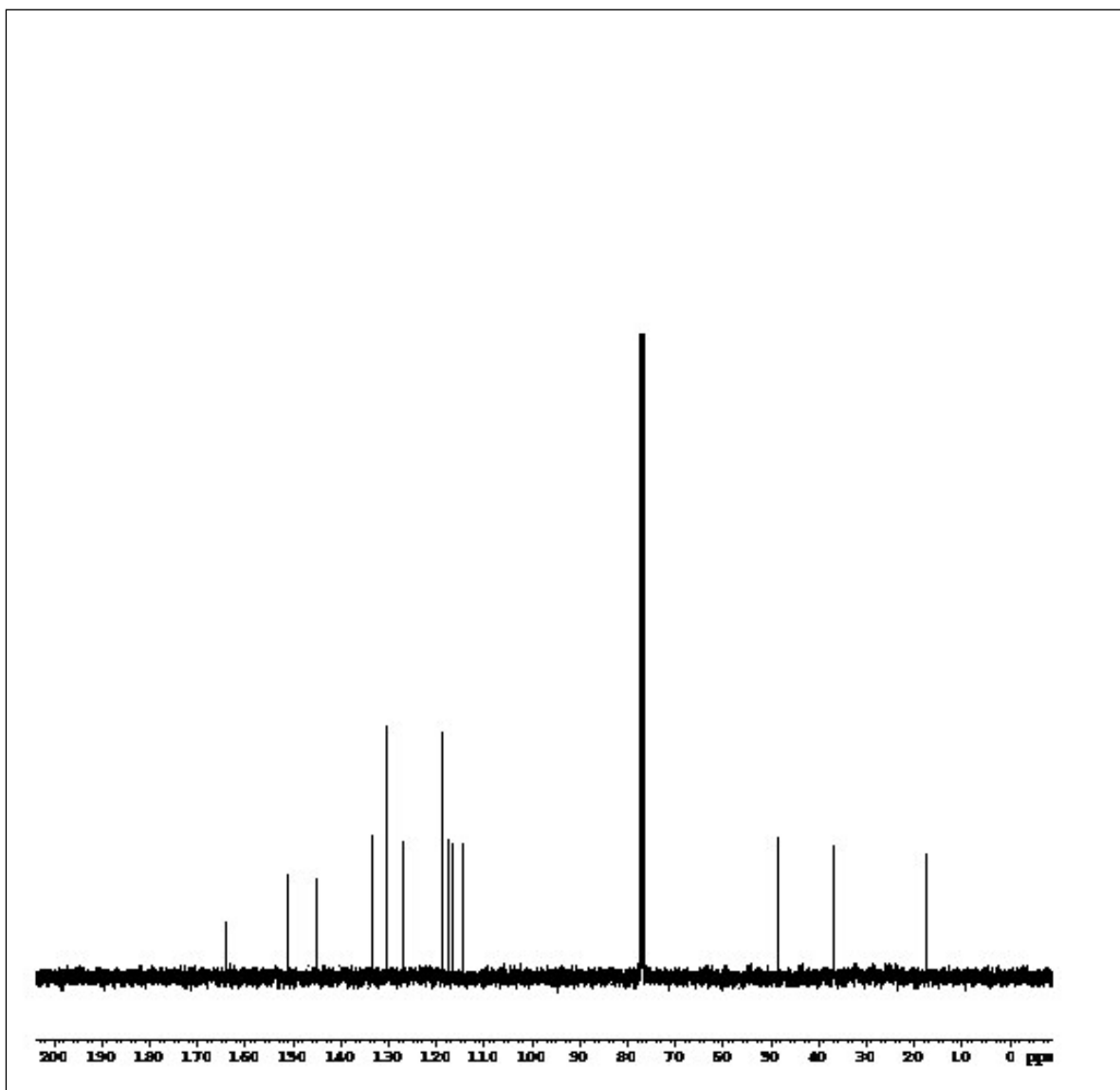
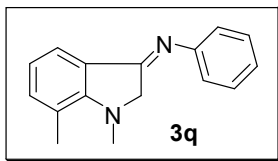
Compound 3p. ^{13}C NMR Spectrum (CDCl_3).



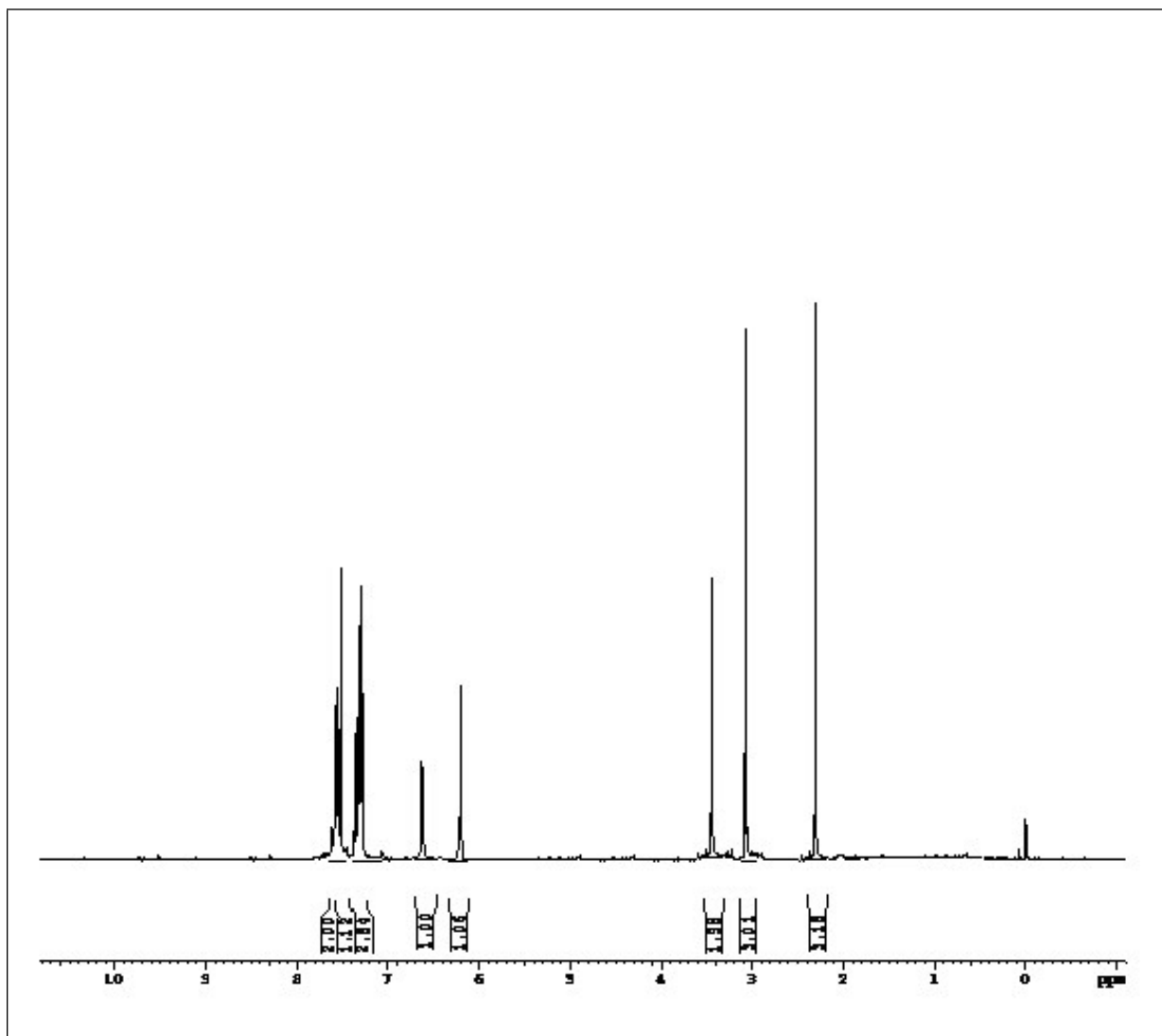
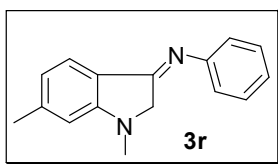
Compound **3q**. ^1H NMR Spectrum (CDCl_3).



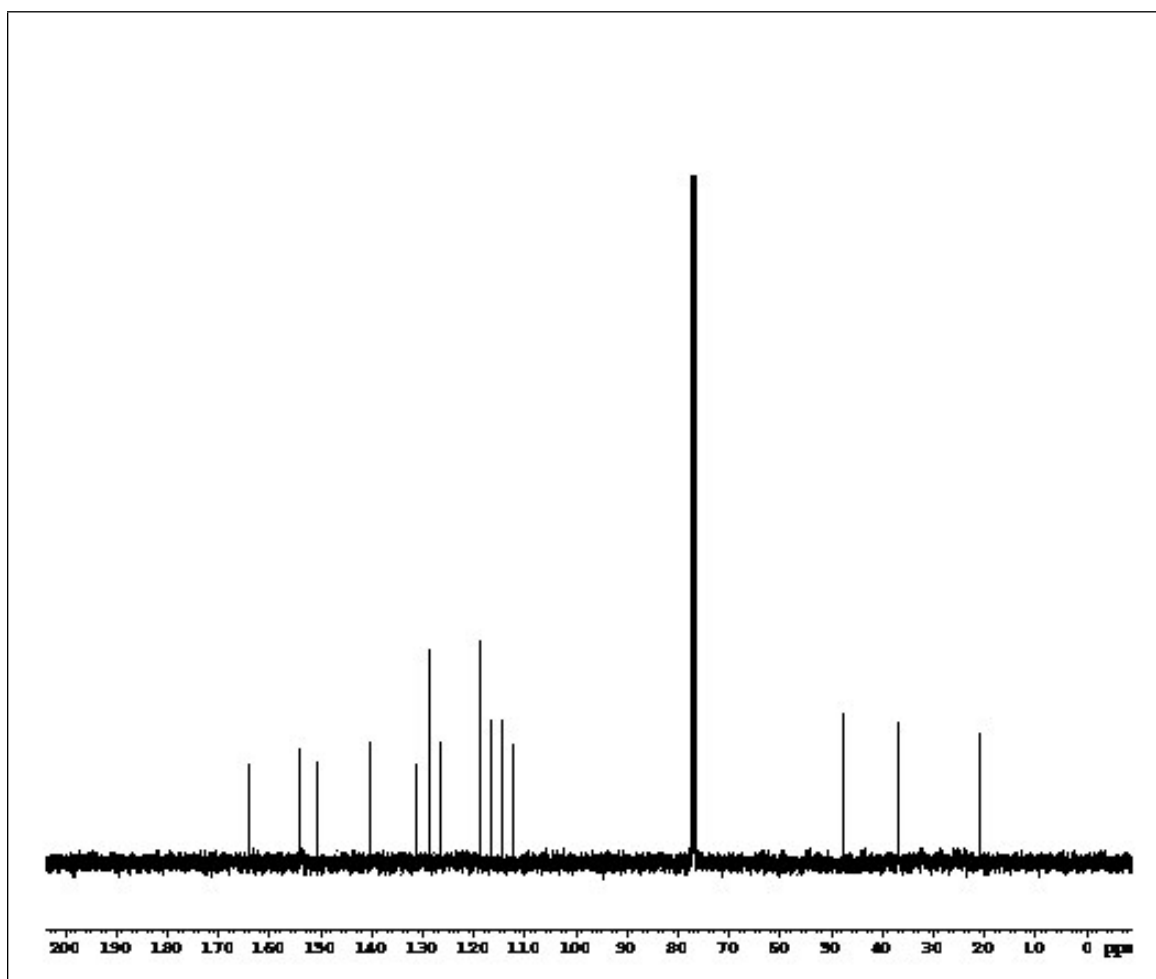
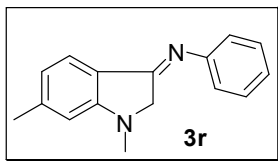
Compound 3q. ^{13}C NMR Spectrum (CDCl_3).



Compound 3r. ^1H NMR Spectrum (CDCl_3).



Compound 3r. ^{13}C NMR Spectrum (CDCl_3).



IX. Reference: 1 M. Majek, F. Filace and A. J. Wangelin, *Beilstein J. Org. Chem.*, 2014, **10**, 981.