Supporting Information for

Microwave-assisted, ruthenium-catalyzed intramolecular amide-alkyne annulation for the rapid synthesis of fused tricyclic isoquinolinones

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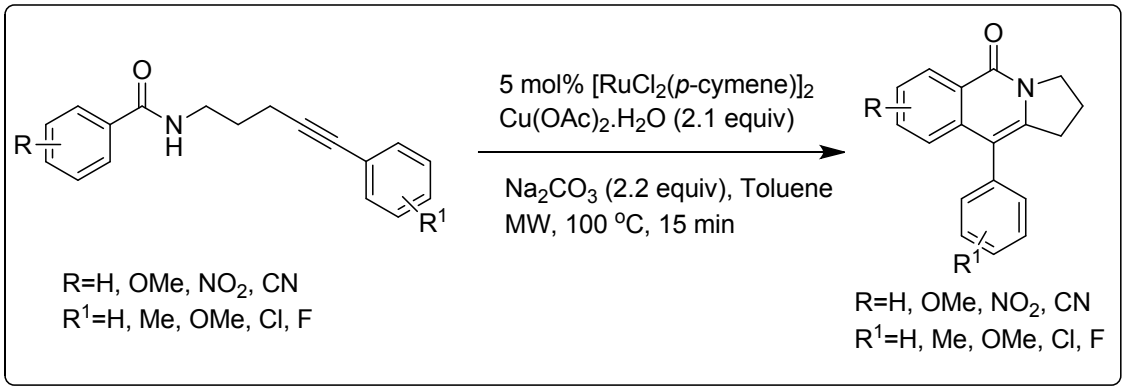
1. Experimental Procedure & Spectral data

1.1 General

IR spectra were recorded on FT-IR spectrometer (KBr) and reported in reciprocal centimeters (cm^{-1}). ^{1}HNMR spectra were recorded at 500 MHz, 300 MHz and ^{13}C NMR at 125 MHz, 75 MHz. For ^{1}H NMR, tetramethylsilane (TMS) was used as internal standard ($\delta = 0$) and the values are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), and the coupling constants in Hz. For ^{13}C NMR, $CDCl_3$ ($\delta = 77.27$) was used as internal standard and spectra were obtained with complete proton decoupling. Low-resolution MS and HRMS data were obtained using ESI ionization. Melting points were measured on micro melting point apparatus. Reaction progress was monitored by using analytical thin layer chromatography (TLC) on precoated silica gel GF_{254} plates and the spots were detected under UV light (254 nm).

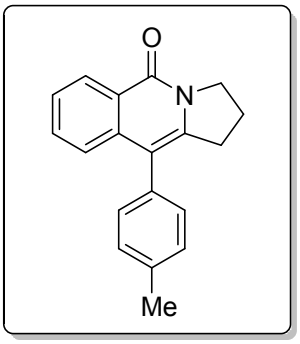
General procedure for catalytic reactions of alkynyl benzamides



An oven-dried 10 mL microwave reaction vessel containing a stir bar was charged with alkyne-amide (0.1 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (5 mol %), Na_2CO_3 (2.2 equiv) and $Cu(OAc)_2.H_2O$ (2.1 equiv) in 2 mL toluene. The vessel was sealed with a Teflon microwave septum. The vessel was placed into the CEM Discover SP system under the following conditions: 1–2 min ramp time, pre-stirring 30 sec, the stirring was set high. Power max was turn on. Maximum power and maximum pressure were set 250 W and 250 psi respectively, with a set temperature of 100 °C for 15 min (hold time). After microwave irradiation was complete, the mixture was cooled to room temperature and then diluted with ethyl acetate and filtered through celite. The filtrate was concentrated and the resulting product was purified via silica gel column chromatography, eluting with ethyl acetate/hexane mixtures.

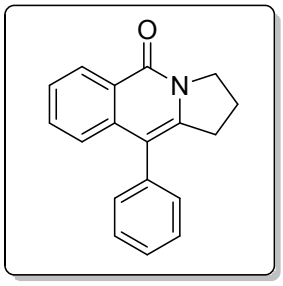
1.2 Characterization data of products (3a-p)

10-(*p*-Tolyl)-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one (3a):



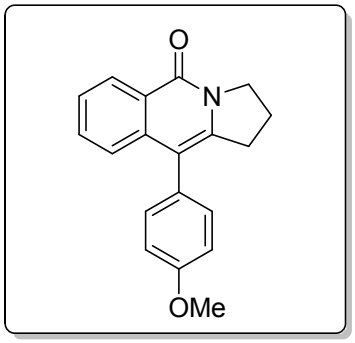
Off-white solid, m.p.165-166 ^{o}C; ^{1}HNMR (300 MHz, $CDCl_3$) δ 8.48 (d, *J* = 8.0 Hz, 1H), 7.51 (t, *J* = 8.0 Hz, 1H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.26-7.30 (m, 3H), 7.19 (d, *J* = 8.0 Hz, 2H), 4.28 (t, *J* = 7.2 Hz, 2H), 2.94 (t, *J* = 7.6 Hz, 2H), 2.42 (s, 3H), 2.25 – 2.06 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.1, 141.2, 138.2, 137.2, 133.2, 131.8, 130.4, 129.4, 127.4, 125.5, 124.9, 124.3, 113.6, 48.5, 31.08, 21.9, 21.3 ppm; IR (KBr): υ 3442, 3036, 1659, 1514, 1338, 1291, 1182, 885, 855 cm^{-1}; MS (ESI): *m/z* ([M+H]$^{+}$): 276; HRMS (ESI): *m/z* calcd for $C_{19}H_{18}NO$: 276.1380; found: 276.1388.

10-Phenyl-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one (3b):



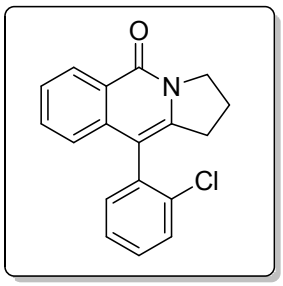
Green solid, m.p. 168-170 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.48 (d, *J* = 7.9 Hz, 1H), 7.74 – 7.07 (m, 8H), 4.28 (t, *J* = 7.2 Hz, 2H), 2.94 (t, *J* = 7.6 Hz, 2H), 2.33 – 1.97 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.1, 141.3, 138.1, 136.3, 131.8, 130.6, 128.7, 127.5, 127.4, 125.6, 124.9, 124.2, 113.7, 48.5, 31.1, 21.9 ppm; IR (KBr): υ 3441, 3032, 1660, 1515, 1340, 1292, 1183, 856.1 cm^{-1}; MS (ESI): *m/z* ([M+H]$^{+}$): 262; HRMS (ESI): *m/z* calcd for $C_{18}H_{16}NO$: 262.1232; found: 262.1221.

10-(4-Methoxyphenyl)-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one (3c):



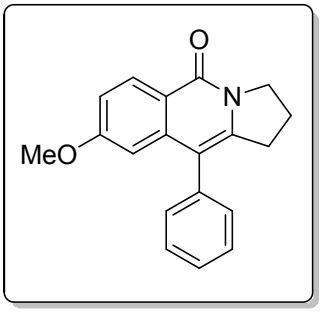
Off-white solid, m.p. 149-150 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.48 (d, *J* = 8.0 Hz, 1H), 7.57 – 7.48 (m, 1H), 7.43 (dd, *J* = 11.1, 3.9 Hz, 1H), 7.30 (d, *J* = 8.1 Hz, 1H), 7.25 – 7.19 (m, 2H), 7.04 – 6.97 (m, 2H), 4.41 (d, *J* = 8.0 Hz, 2H), 3.88 (s, 3H), 2.94 (t, *J* = 7.6 Hz, 2H), 2.35 – 2.03 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.1, 158.9, 141.4, 138.4, 131.7, 131.5, 128.4, 127.4, 125.53, 124.9, 124.3, 114.2, 114.0, 113.2, 48.5, 31.0, 21.9 ppm; IR (KBr): υ 2965, 2916, 1645, 1605, 1510, 1481, 1231, 1155, 1025, 833 cm^{-1}; MS (ESI): *m/z* ([M+H]$^{+}$): 292; HRMS (ESI): *m/z* calcd for $C_{19}H_{18}NO_2$: 292.1338; found: 292.1330.

10-(2-Chlorophenyl)-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one (3d):



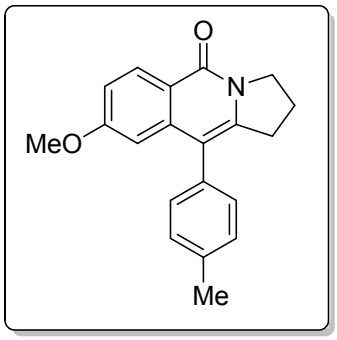
White solid, m.p.161-163 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.48 (d, *J* = 8.0 Hz, 1H), 7.71 – 7.15 (m, 6H), 7.03 (t, *J* = 10.8 Hz, 1H), 4.29 (t, *J* = 7.2 Hz, 2H), 3.02 – 2.68 (m, 2H), 2.31 – 2.00 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.2, 142.1, 137.5, 135.2, 134.9, 132.7, 132.0, 129.9, 129.5, 127.5, 127.2, 125.7, 124.8, 123.8, 111.0, 48.6, 30.7, 21.7 ppm; IR (KBr): υ 3041, 2885, 1660, 1515, 1341, 1293, 1183, 886, 855 cm^{-1}; MS (ESI): *m/z* ([M+H]$^{+}$): 296; HRMS (ESI): *m/z* calcd for $C_{18}H_{15}ClNO$: 296.0800; found: 296.0802.

8-Methoxy-10-phenyl-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one (3e):



White solid, m.p.178-179 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) d 8.40 (d, *J* = 8.9 Hz, 1H), 7.47 (t, *J* = 7.3 Hz, 2H), 7.40 (t, *J* = 7.3 Hz, 1H), 7.31 (d, *J* = 7.3 Hz, 2H), 7.01 (d, *J* = 8.7 Hz, 1H), 6.64 (s, 1H), 4.25 (t, *J* = 7.1 Hz, 2H), 3.72 (s, 3H), 2.91 (t, *J* = 7.5 Hz, 2H), 2.19 – 2.07 (m, 2H) ppm; ^{13}C NMR (125 MHz, CDCl3) δ 162.5, 160.8, 142.0, 140.2, 136.4, 130.5, 129.5, 128.7, 127.5, 119.0, 114.4, 113.3, 106.0, 55.2, 48.3, 31.1, 21.9 ppm; IR (KBr): υ 2963, 2913, 1646, 1600, 1511, 1483, 1228, 1154, 1024, 831 cm^{-1}; MS (ESI): *m/z* ([M+H]$^{+}$): 292; HRMS (ESI): *m/z* calcd for $C_{19}H_{18}NO_2$: 292.1259; found: 292.1266.

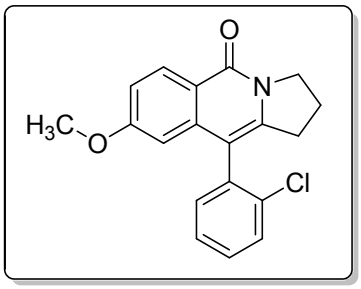
8-Methoxy-10-(*p*-tolyl)-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one (3f):



Off-white solid, m.p. 165-168 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.39 (d, *J* = 8.9 Hz, 1H), 7.27 (d, *J* = 9.0 Hz, 3H), 7.19 (d, *J* = 7.9 Hz, 2H), 7.07 – 6.95 (m, 1H), 6.66 (d, *J* = 2.4 Hz, 1H), 4.35 – 4.15 (m, 2H), 3.73 (s, 4H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.43 (s, 4H), 2.24 – 2.04 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.5, 160.8, 142.0, 140.4, 137.1, 133.3, 130.3, 129.4, 119.0, 114.4, 113.3, 106.0, 55.2, 48.3, 31.1, 21.9, 21.3 ppm; IR (KBr): υ 2964, 2912, 1645, 1599, 1510, 1482, 1227, 1153, 1024,

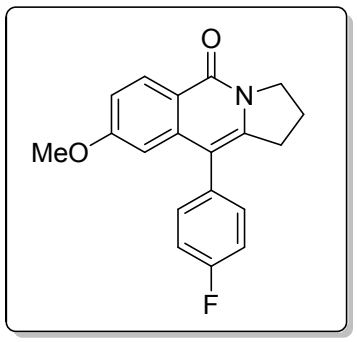
830 cm^{-1}; MS (ESI): *m/z* ($[M+H]^+$): 306; HRMS (ESI): *m/z* calcd for $C_{20}H_{20}NO_2$: 306.1494; found: 306.1482.

10-(2-Chlorophenyl)-8-methoxy-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one (3g):



White solid, m.p.160-162 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.40 (d, *J* = 8.9 Hz, 1H), 7.60 – 7.49 (m, 1H), 7.43 – 7.34 (m, 2H), 7.34 – 7.28 (m, 1H), 7.06 – 6.95 (m, 1H), 6.36 (d, *J* = 2.5 Hz, 1H), 4.26 (t, *J* = 7.2 Hz, 2H), 3.72 (s, 3H), 3.03 – 2.67 (m, 2H), 2.31 – 2.06 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.6, 160.9, 142.9, 139.6, 135.2, 134.9, 132.6, 130.0, 129.6, 129.5, 127.2, 118.9, 114.3, 110.6, 105.7, 55.2, 48.4, 30.8, 21.7 ppm; IR (KBr): υ 3011, 2911, 1645, 1595, 1511, 1484, 1228, 1154, 1025, 831 cm^{-1}; MS (ESI): *m/z* ($[M+H]^+$): 326; HRMS (ESI): *m/z* calcd for $C_{19}H_{16}NO_2Cl$: 326.0948; found: 326.0932.

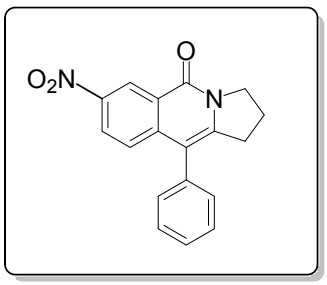
10-(4-Fluorophenyl)-8-methoxy-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1H)-one (3h):



Off-white solid, m.p. 158-160 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.40 (d, *J*= 8.9 Hz, 1H), 7.32-7.23 (m, 2H), 7.22-7.13 (m, 2H), 7.01 (d, *J* = 8.9 Hz, 1H), 6.57 (d, *J* = 2.4 Hz, 1H), 4.25 (d, *J* = 7.9 Hz, 2H), 3.74 (s, 3H), 2.89 (t, *J* = 7.6 Hz, 2H), 2.26-2.05 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.6, 160.8, 142.3, 140.2, 132.2, 132.1, 129.6, 119.0, 115.9, 115.7, 114.4, 112.2, 105.8, 55.31, 48.4,

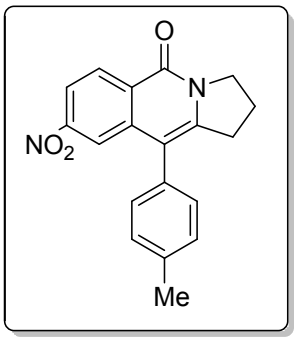
31.1, 21.8 ppm; IR (KBr): υ 2964, 2912, 1645, 1599, 1510, 1482, 1227, 1153, 1024, 830 cm^{-1}; MS (ESI): *m/z* ($[M+H]^+$): 310; HRMS (ESI): *m/z* calcd for $C_{19}H_{17}NO_2F$: 310.1243; found: 310.1232.

7-Nitro-10-phenyl-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one (3i):



Yellow solid, m.p.180-182 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 8.62 (d, *J* = 8.7 Hz, 1H), 8.15 (d, *J* = 11.5 Hz, 2H), 7.53 (t, *J* = 7.4 Hz, 2H), 7.47 (t, *J* = 7.4 Hz, 1H), 7.31 (d, *J* = 7.0 Hz, 2H), 4.31 (t, *J* = 7.2 Hz, 2H), 3.00 (t, *J* = 7.6 Hz, 2H), 2.27 – 2.14 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.7, 150.1, 144.1, 138.7, 134.7, 130.3, 130.2, 129.2, 128.3, 128.3, 119.9, 119.1, 113.6, 48.9, 31.3, 21.7 ppm; IR (KBr): υ 3423, 2920, 1663, 1629, 1470, 1340, 1294, 837 cm^{-1}; MS (ESI): *m/z* ($[M+H]^+$): 307; HRMS (ESI): *m/z* calcd for $C_{18}H_{15}N_2O_3$: 307.1083; found: 307.1073.

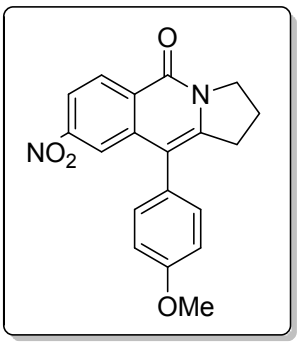
8-Nitro-10-(*p*-tolyl)-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one (3j):



Yellow solid, m.p.199-200 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 8.67 – 8.56 (m, 1H), 8.15 (d, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 10.7 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 4.28 (t, *J* = 7.2 Hz, 2H), 2.99 (t, *J* = 7.7 Hz, 2H), 2.46 (s, 3H), 2.27 – 2.13 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.8, 150.1, 144.1,

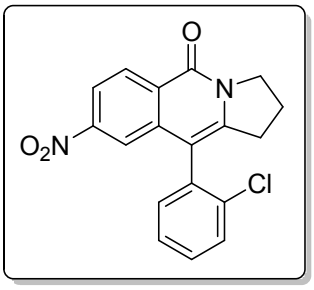
138.94, 138.1, 131.6, 130.1, 129.9, 129.4, 128.4, 120.0, 119.1, 113.7, 48.9, 31.3, 21.7, 21.3 ppm; IR (KBr): υ 3423, 2920, 1663, 1629, 1470, 1340, 1294, 837 cm^{-1}; MS (ESI): *m/z* ($[M+H]^+$): 321; HRMS (ESI): *m/z* calcd for $C_{19}H_{17}N_2O_3$: 321.1233; found: 321.1234.

10-(4-Methoxyphenyl)-8-nitro-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one (3k):



Orange Solid, m.p.132-134 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.65 – 8.58 (m, 1H), 8.15 (d, *J* = 7.4 Hz, 2H), 7.25 – 7.18 (m, 2H), 7.05 (d, *J* = 8.6 Hz, 2H), 4.30 (t, *J* = 7.2 Hz, 2H), 2.99 (t, *J* = 7.7 Hz, 2H), 2.26 – 2.16 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.4, 150.1, 144.3, 139.1, 131.4, 129.5, 126.8, 120.0, 119.1, 114.6, 55.4, 48.9, 31.3, 21.7 ppm; IR (KBr): υ 3102, 2975, 2838, 1657, 16178, 1517, 1467, 1342, 1247, 1025, 843 cm^{-1}; MS (ESI): *m/z* ($[M+H]^+$): 337; HRMS (ESI): *m/z* calcd for $C_{19}H_{17}N_2O_4$: 337.1188; found: 337.1179.

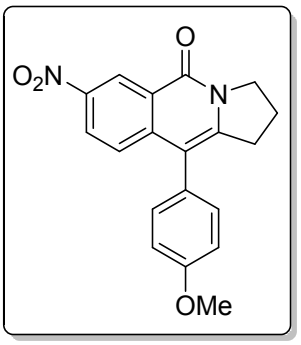
10-(2-Chlorophenyl)-8-nitro-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one(3l):



Yellow solid, m.p.205-206 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 8.8 Hz, 1H), 8.17 (d, *J* = 8.8 Hz, 1H), 7.86 (s, 1H), 7.60 (t, *J* = 7.8 Hz, 1H), 7.50 – 7.39 (m, 2H), 7.34 – 7.29 (m, 1H), 4.30(t, *J* = 7.2 Hz, 2H), 2.99 – 2.82 (m, 2H), 2.30 – 2.17 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.9, 150.3, 145.1, 138.2, 135.0, 133.3, 132.4, 130.4, 130.3, 129.6, 128.3, 127.6, 119.5, 119.3, 110.9, 49.02,

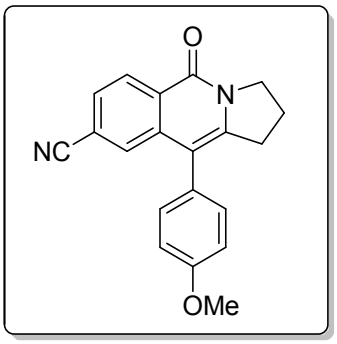
31.0, 21.5 ppm; IR (KBr): υ 3105, 2975, 2839, 1658, 16179, 1518, 1468, 1343, 1248, 1026, 844 cm^{-1}; MS (ESI): *m/z* ($[M+H]^+$): 341; HRMS (ESI): *m/z* calcd for $C_{18}H_{14}ClN_2O_3$: 341.0693; found: 341.0690.

10-(4-Methoxyphenyl)-7-nitro-2,3-dihydropyrrolo[1,2-*b*]isoquinolin-5(1*H*)-one (3m):



Orange solid, m.p. 161-162 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 9.31 (d, *J* = 2.4 Hz, 1H), 8.27 (dd, *J* = 9.0, 2.4 Hz, 1H), 7.40 (d, *J* = 9.0 Hz, 1H), 7.24 – 7.16 (m, 2H), 7.08 – 7.00 (m, 2H), 4.29 (t, *J* = 7.2 Hz, 2H), 3.90 (s, 2H), 2.99 (t, *J* = 7.7 Hz, 2H), 2.28 – 2.14 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.1, 159.4, 146.5, 145.1, 142.8, 131.5, 127.0, 125.7, 124.7, 124.1, 114.5, 113.0, 55.4, 49.0, 31.6, 21.6 ppm; IR (KBr): υ 3099, 2974, 2839, 1655, 16179, 1516, 1454, 1344, 1249, 1025, 844 cm^{-1}; MS (ESI): *m/z* ($[M+H]^+$): 337; HRMS (ESI): *m/z* calcd for $C_{19}H_{17}N_2O_4$: 337.1188; found: 337.1184.

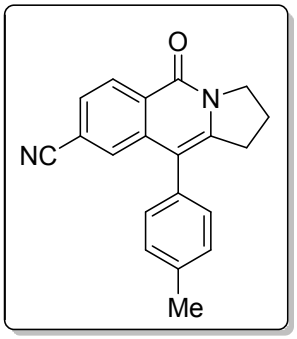
10-(4-Methoxyphenyl)-5-oxo-1,2,3,5-tetrahydropyrrolo[1,2-*b*]isoquinoline-8-carbonitrile (3n):



Off-white solid, m.p. 164-165 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.54 (d, *J* = 8.2 Hz, 1H), 7.70 – 7.51 (m, 2H), 7.30 (dd, *J* = 9.1, 2.3 Hz, 2H),7.05 (dd, *J* = 9.1, 2.3 Hz, 2H), 4.29 (t, *J* = 7.2 Hz, 2H), 3.90 (s, 3H), 2.97 (t, *J* = 7.7 Hz, 2H), 2.34 – 2.06 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.0, 159.4,

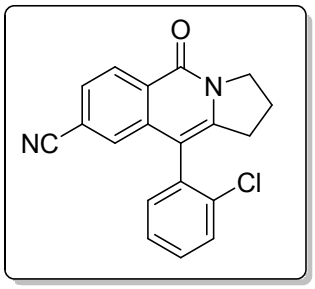
143.8, 138.5, 131.4, 129.4, 128.6, 127.1, 126.8, 118.5, 115.3, 114.6, 112.5, 55.4, 48.9, 31.2, 21.7 ppm; IR (KBr): υ 3448, 3038, 2886, 2224, 1649, 1515, 1340, 1293, 1185, 887, 855 cm^{-1}; MS (ESI): *m/z* ($[M+H]^+$): 317; HRMS (ESI): *m/z* calcd for $C_{20}H_{17}N_2O_2$: 317.1284; found: 317.1296.

5-Oxo-10-(*p*-tolyl)-1,2,3,5-tetrahydropyrrolo[1,2-b]isoquinoline-8-carbonitrile (3o):



Off-white solid, m.p. 169-171 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.54 (d, *J* = 8.2 Hz, 1H), 7.65 – 7.56 (m, 2H), 7.32 (d, *J* = 7.8 Hz, 2H), 7.16 (d, *J* = 7.9 Hz, 2H), 4.28 (t, *J* = 7.2 Hz, 2H), 2.97 (t, *J* = 7.7 Hz, 2H), 2.46 (s, 3H), 2.26 – 2.12 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.0, 143.7, 138.3, 138.0, 131.7, 130.2, 129.8, 129.4, 128.5, 127.2, 127.1, 118.5, 115.3, 112.8, 48.9, 31.2, 21.7, 21.3 ppm; IR (KBr): υ 3447, 3037, 2886, 2225, 1658, 1513, 1339, 1292, 1183, 886, 855 cm^{-1}; MS (ESI): *m/z* ($[M+H]^+$): 301; HRMS (ESI): *m/z* calcd for $C_{20}H_{17}N_2O$: 301.1341; found: 301.1327.

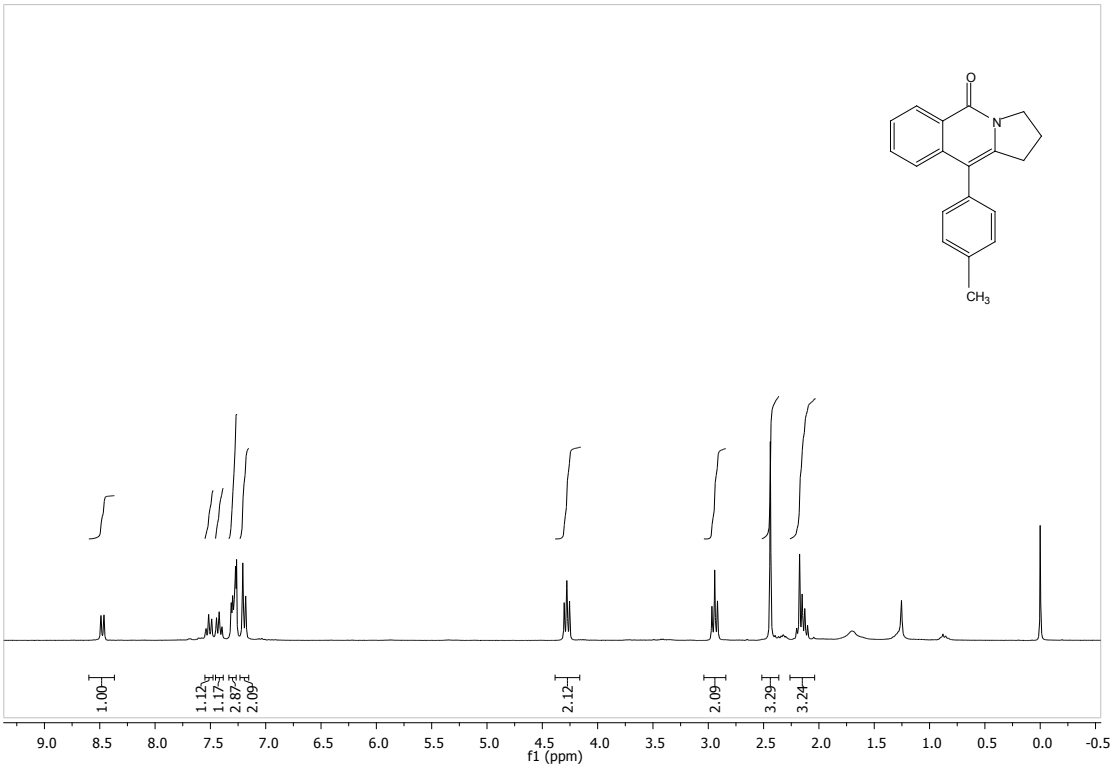
10-(2-Chlorophenyl)-5-oxo-1,2,3,5-tetrahydropyrrolo[1,2-*b*]isoquinoline-8-carbonitrile (3p):



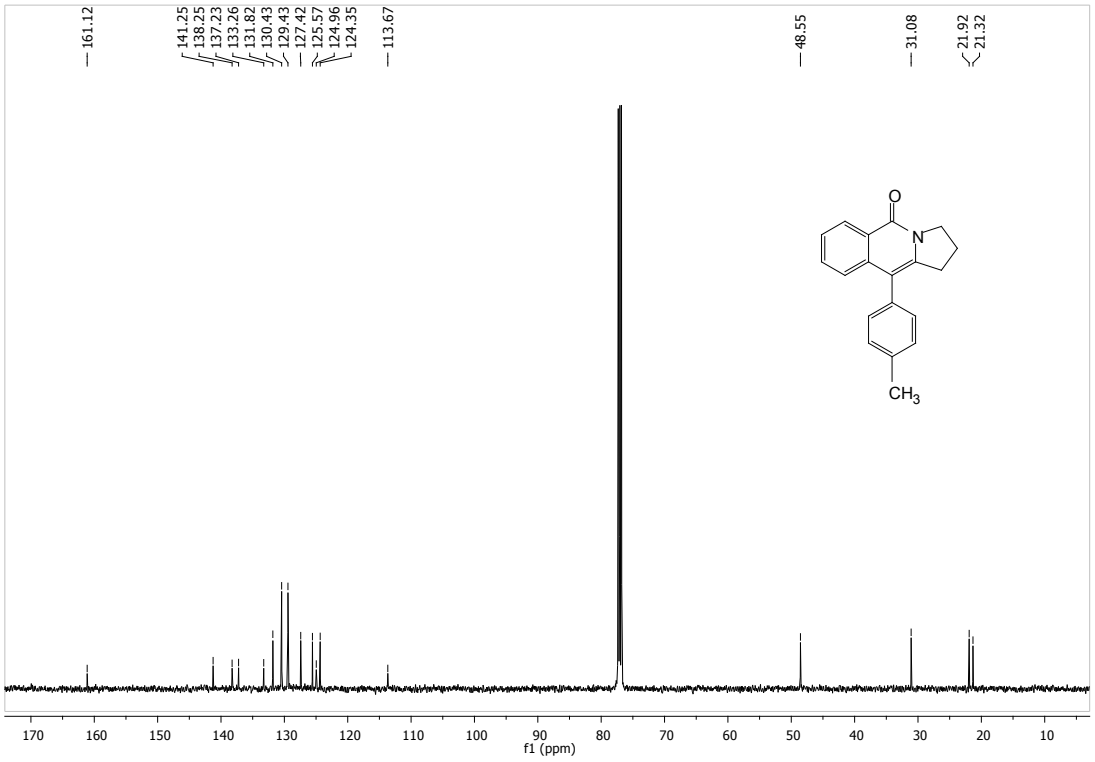
Off-white solid, m.p. 155-157 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.55 (d, *J* = 8.3 Hz, 1H), 7.66 – 7.56 (m, 2H), 7.50 – 7.38 (m, 2H), 7.33 (t, *J* = 3.8 Hz, 1H), 7.31 – 7.24 (m, 1H), 4.39 – 4.23 (t, *J* = 7.2 Hz, 2H), 3.01 – 2.80 (m, 2H), 2.32 – 2.14 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.1, 144.7, 137.6, 135.1, 133.4, 132.4, 130.3, 130.2, 128.9, 128.7, 127.6, 127.3, 127.1, 118.5, 115.6, 110.1, 48.96,

30.9, 21.5 ppm; IR (KBr): υ 3442, 3040, 2885, 2226, 1659, 1514, 1340, 1292, 1182, 885, 854 cm^{-1}; MS (ESI): *m/z* ($[M+H]^+$): 321; HRMS (ESI): *m/z* calcd for $C_{19}H_{14}ClN_2O$: 321.0792; found: 321.0788.

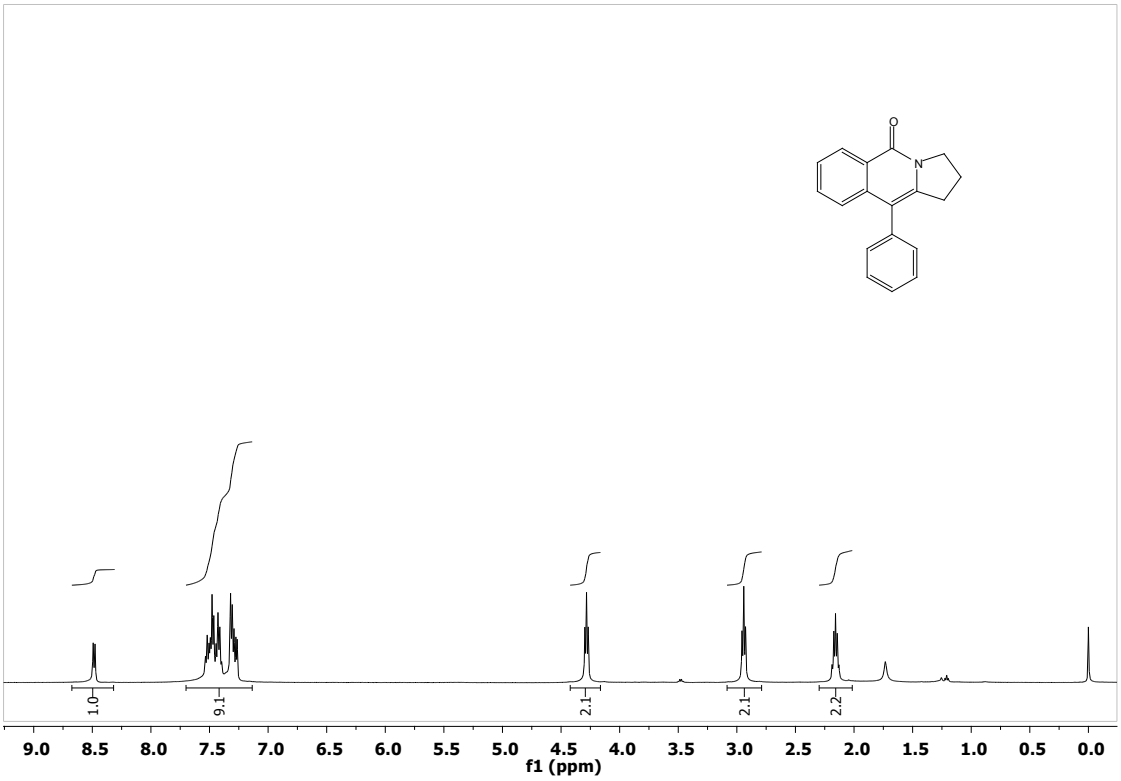
(2) NMR Spectra of Products:



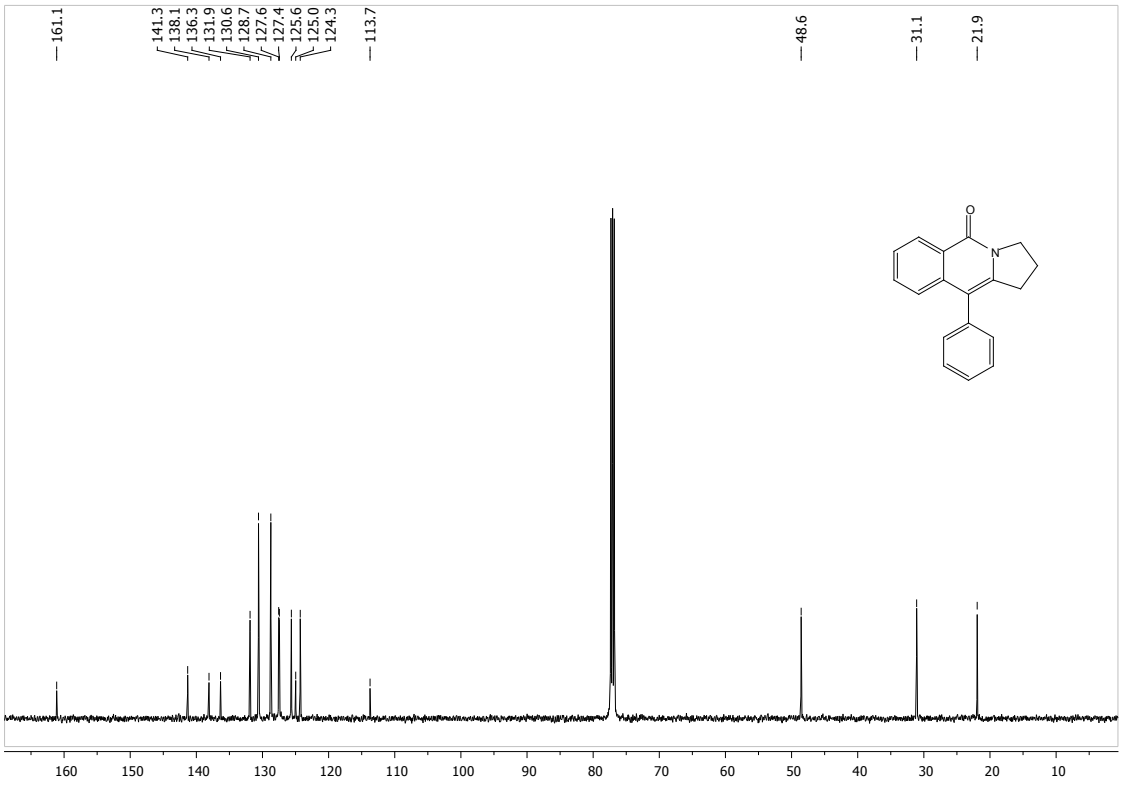
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3a



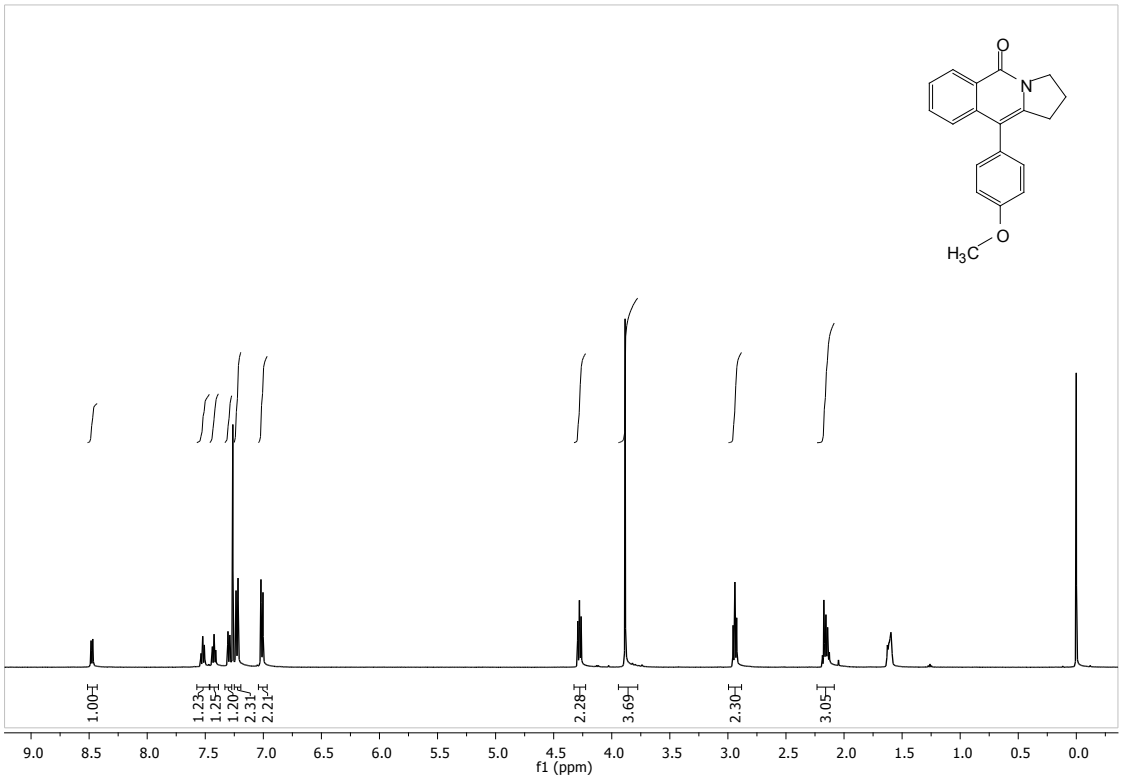
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3a



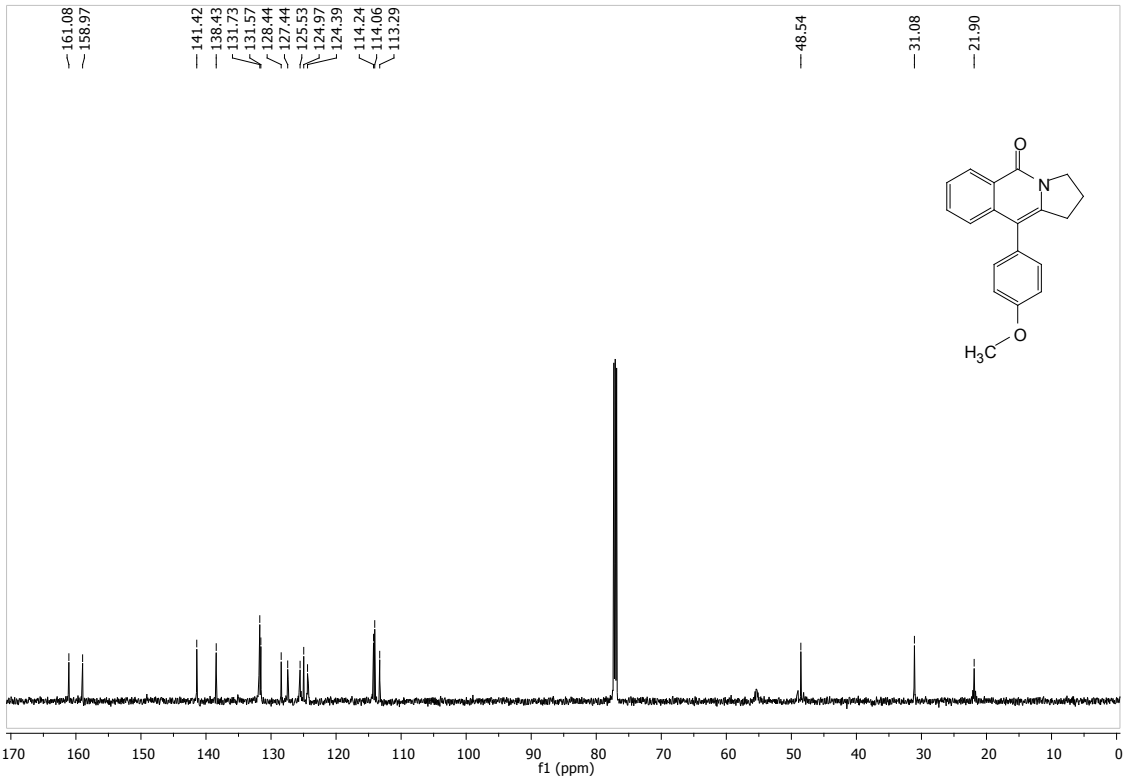
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3b



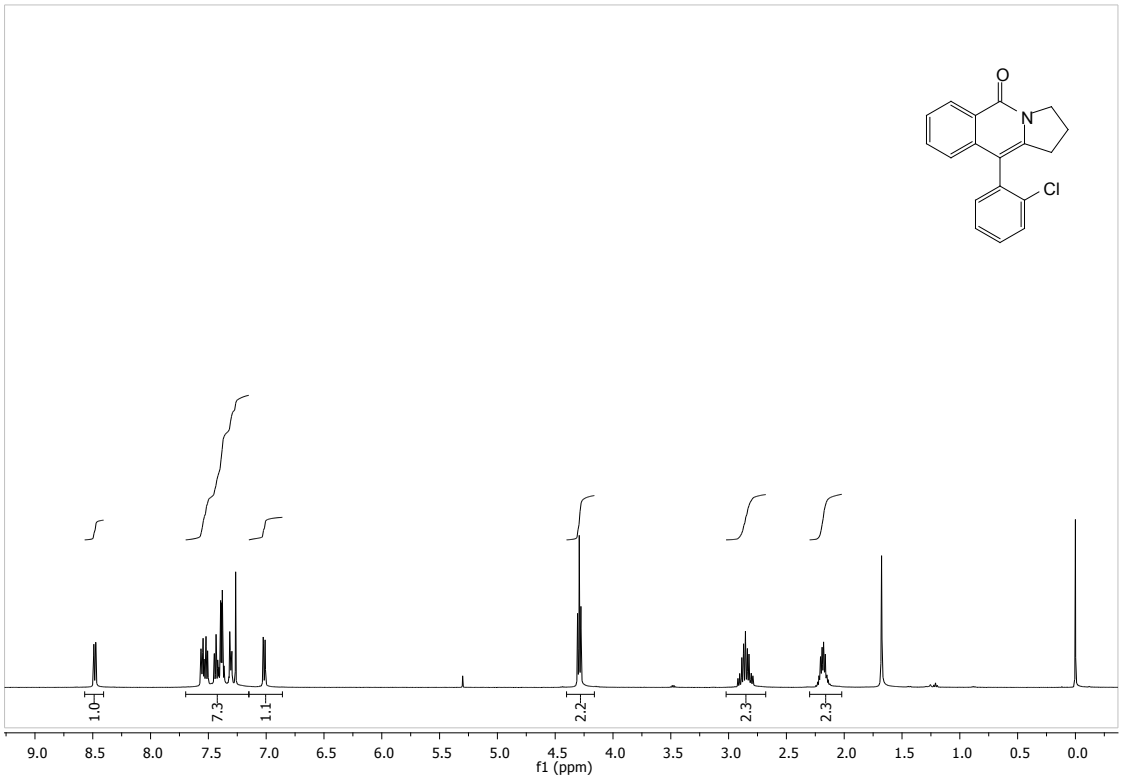
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3b



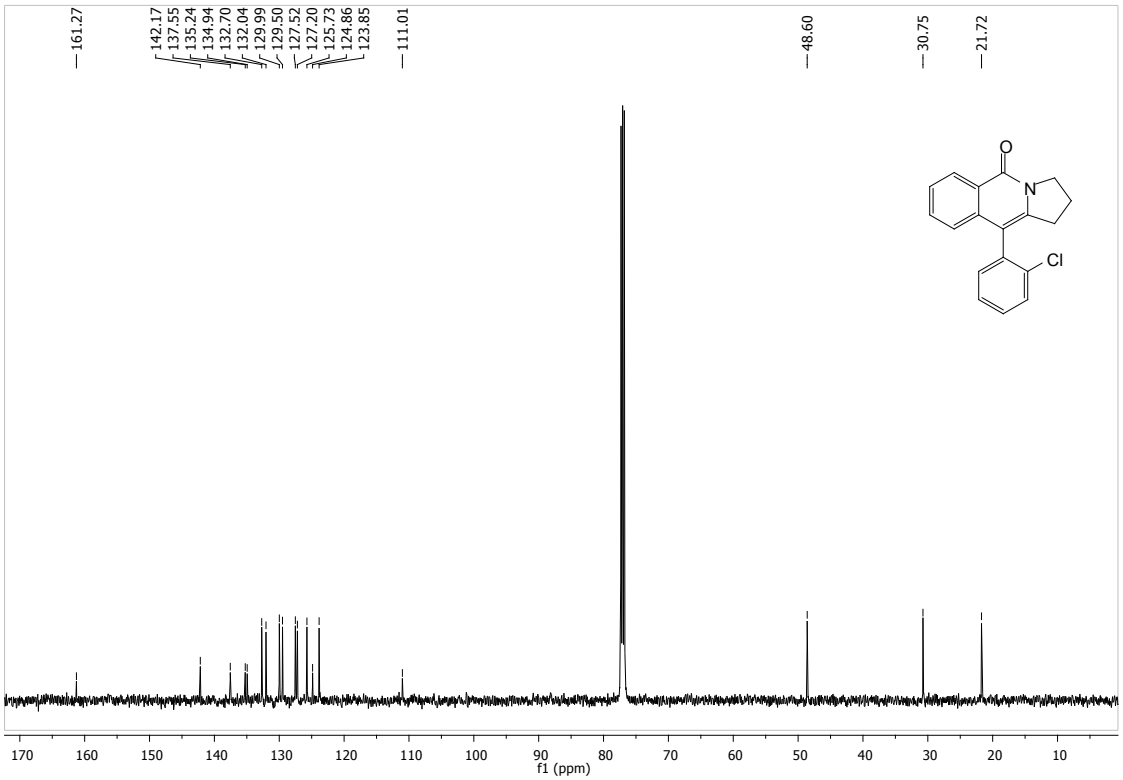
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3c



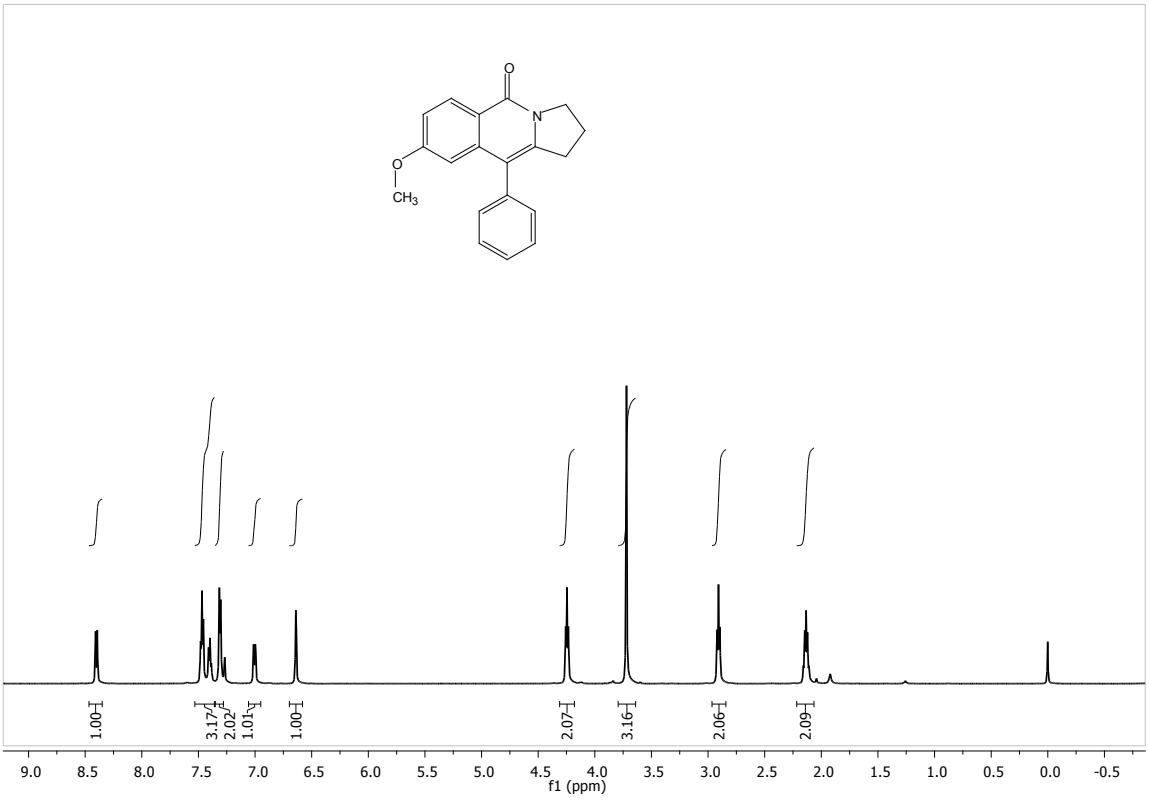
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3c



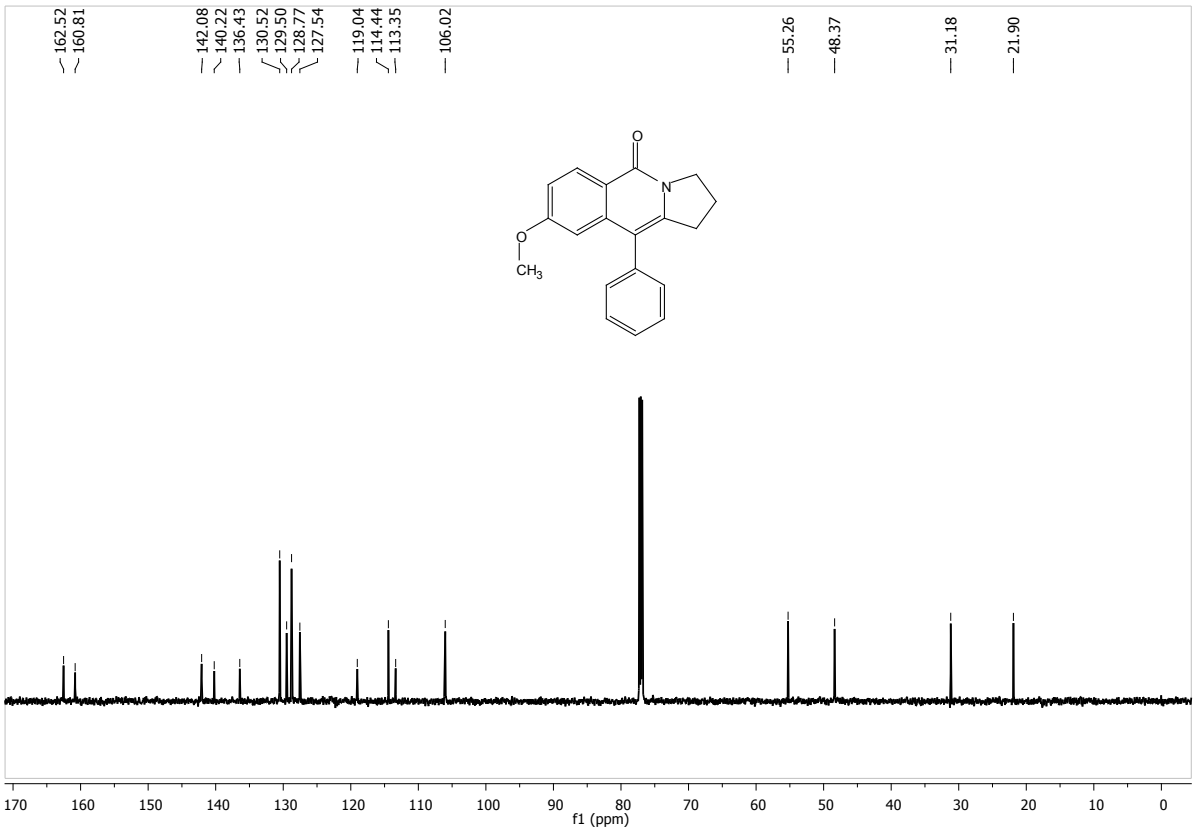
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3d



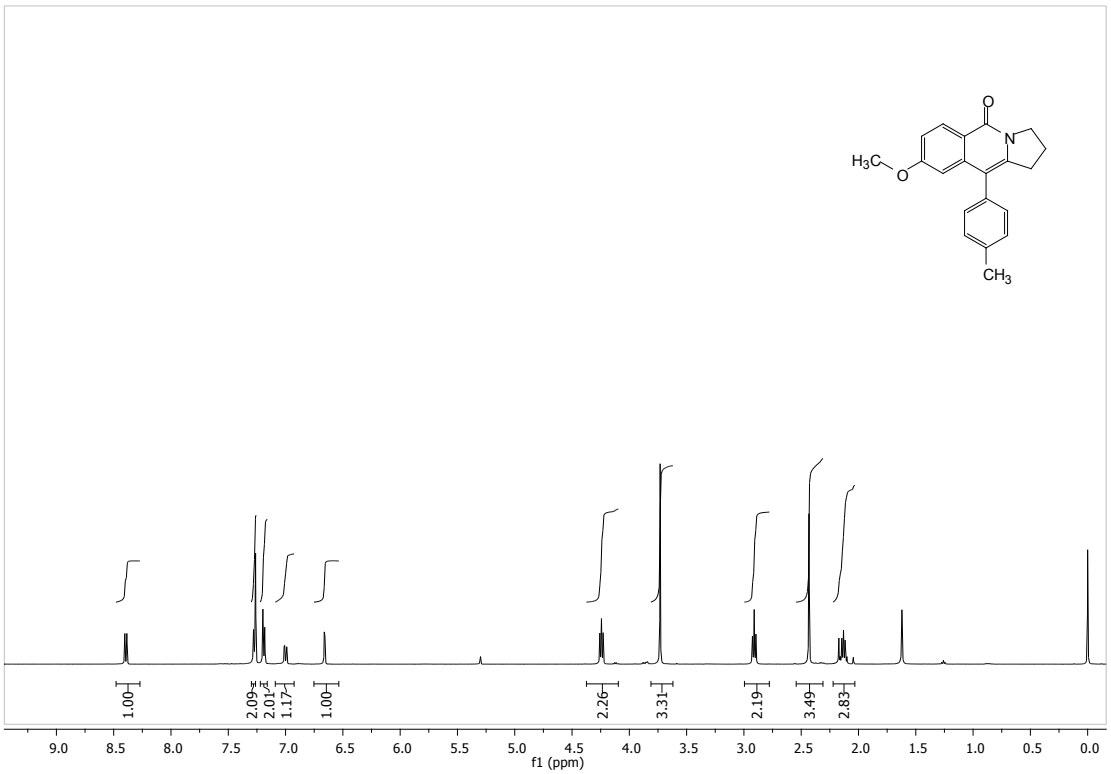
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3d



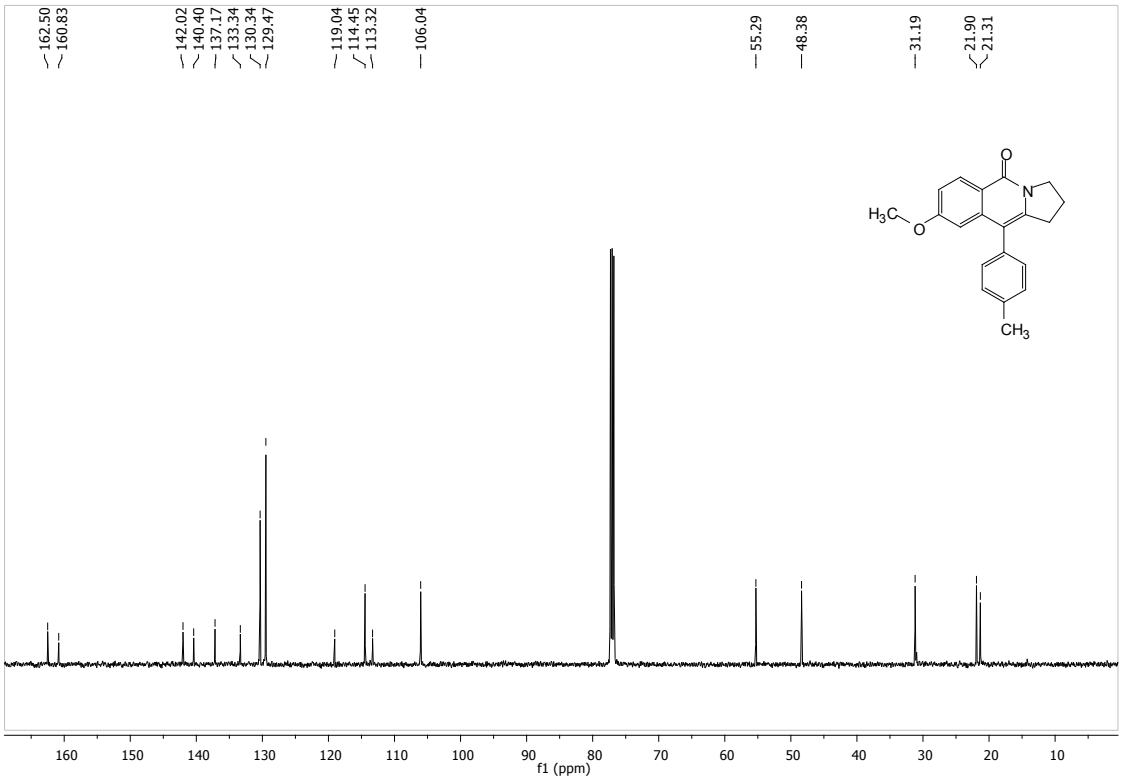
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3e



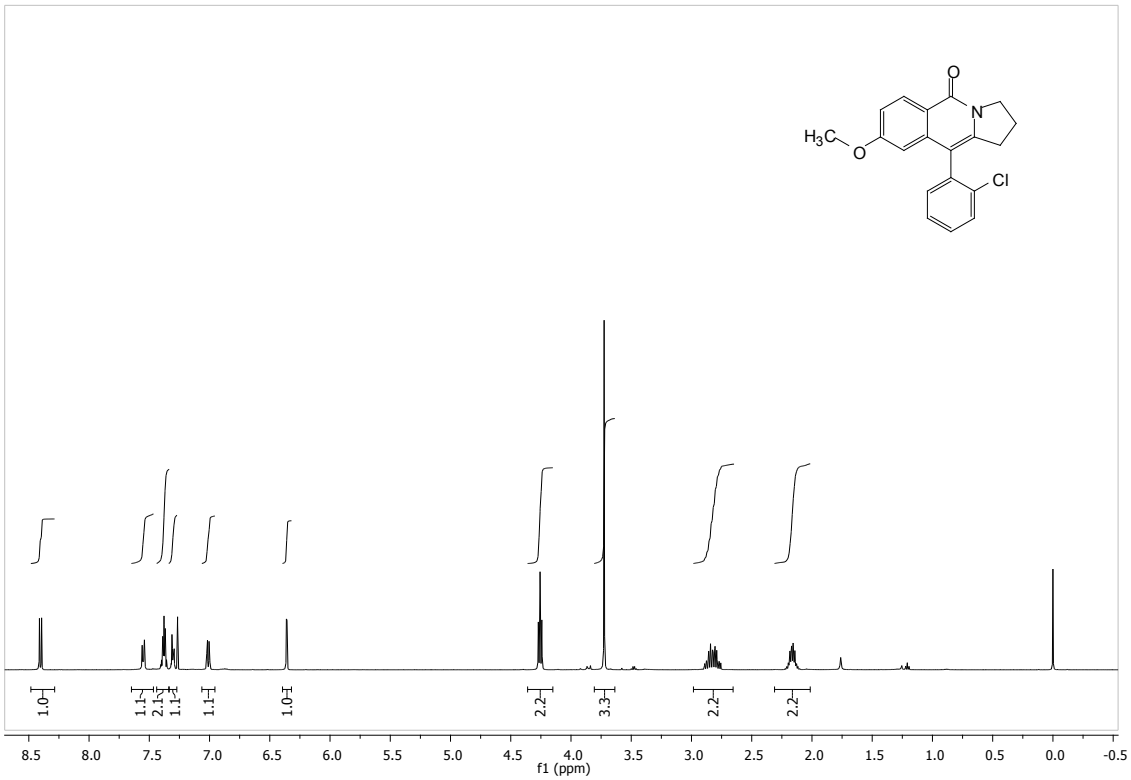
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3e



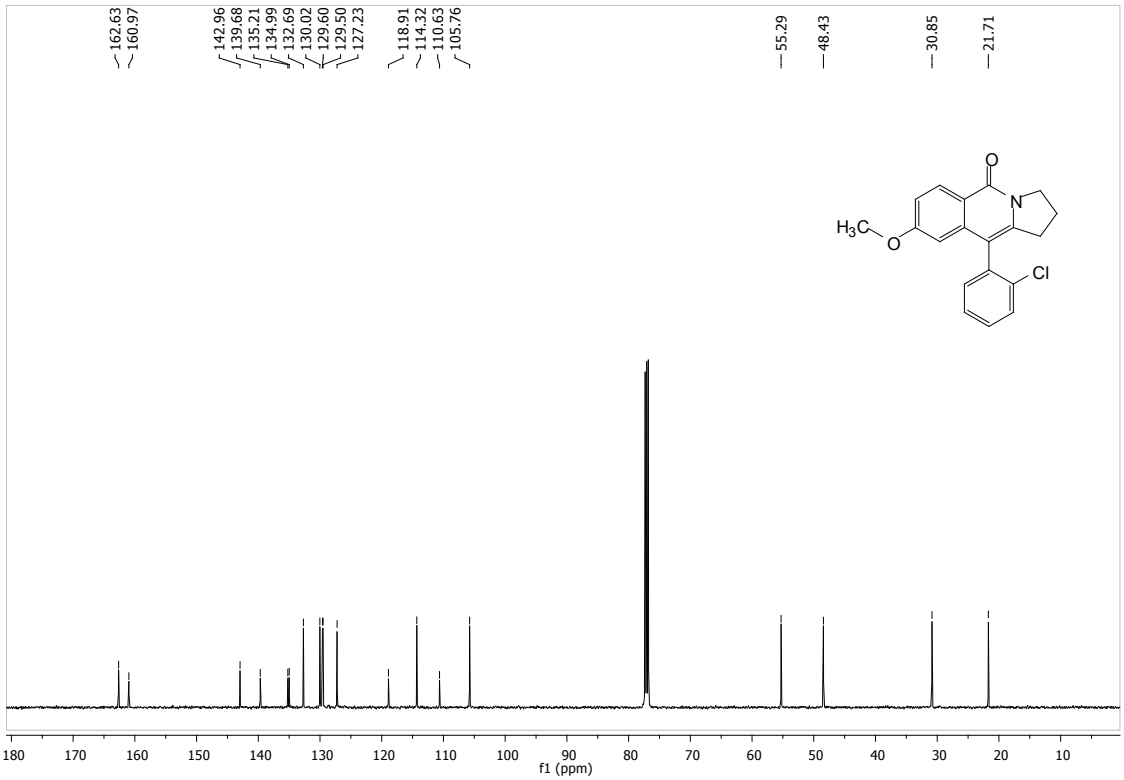
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3f



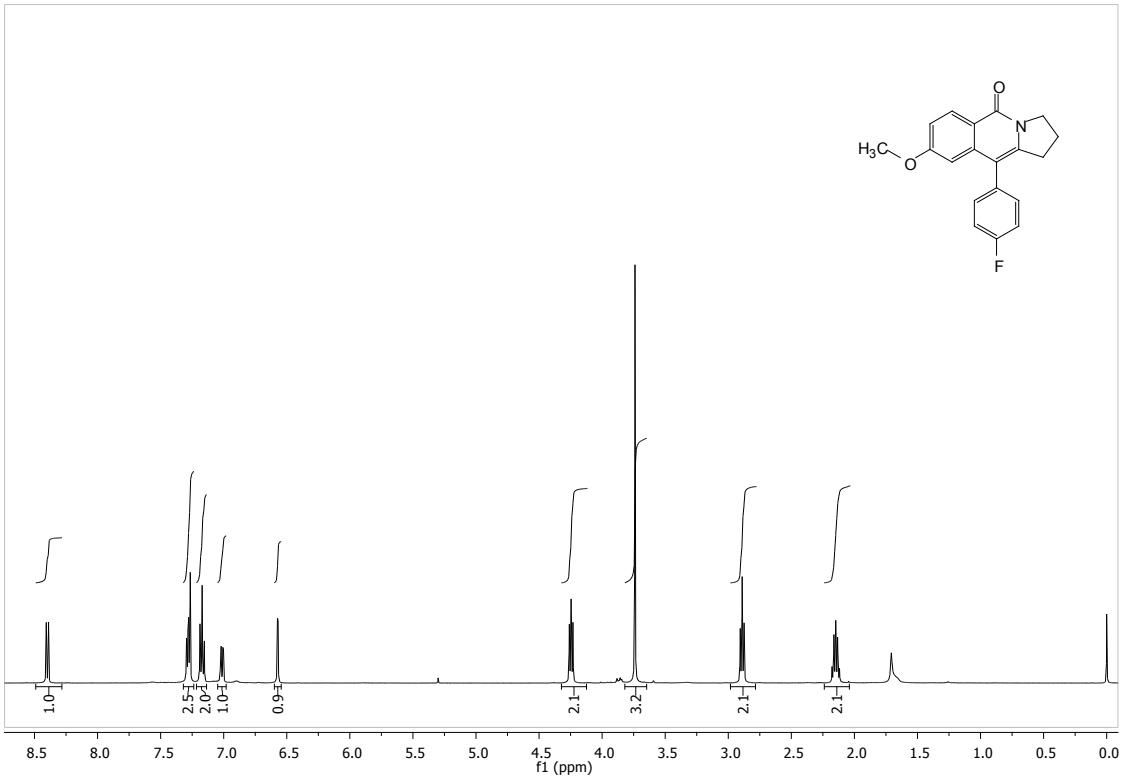
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3f



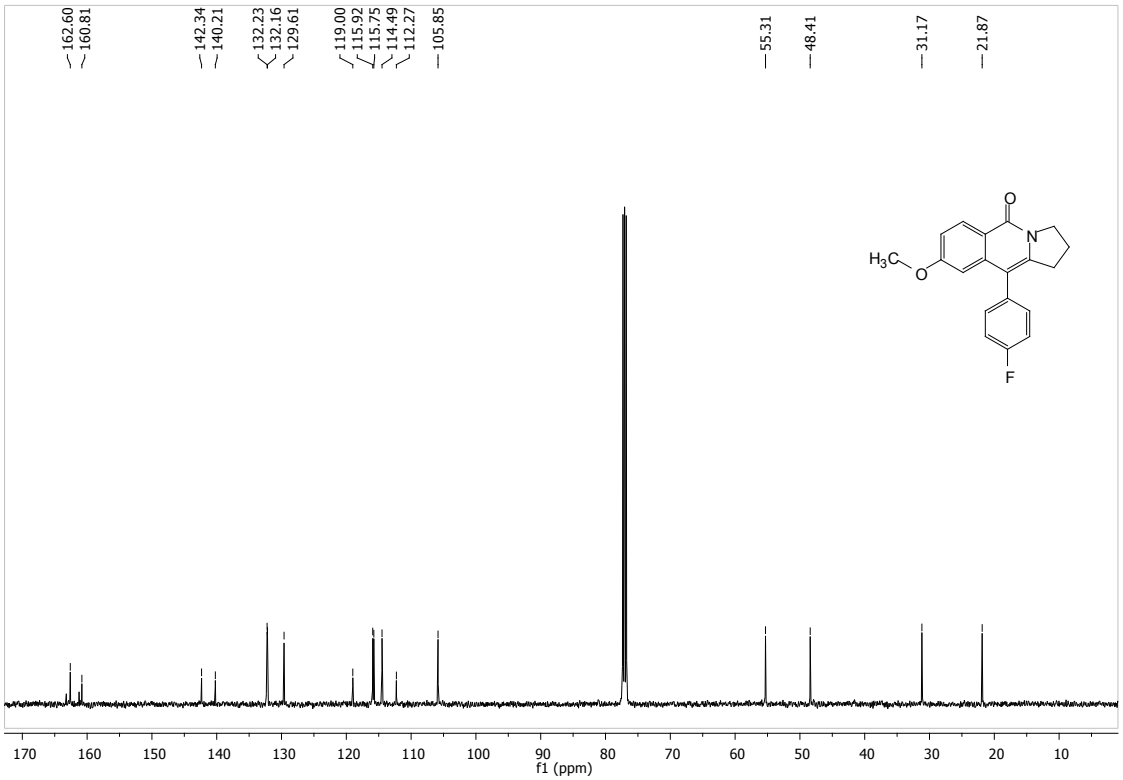
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3g



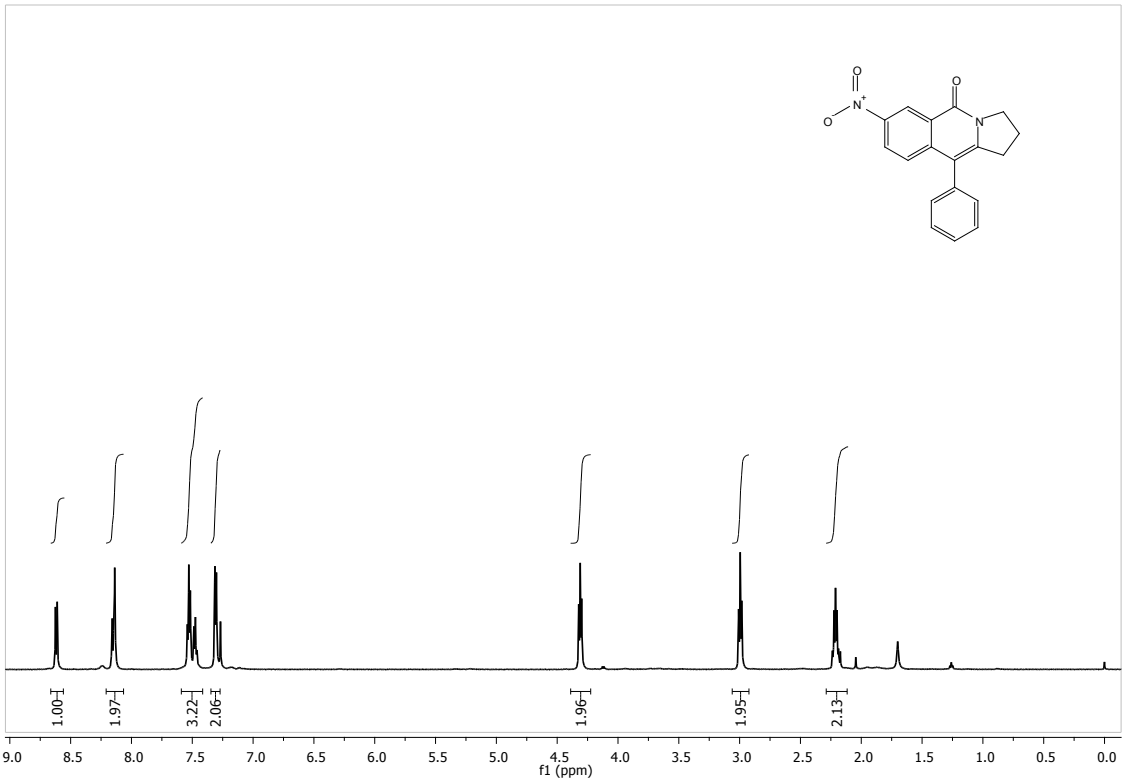
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3g



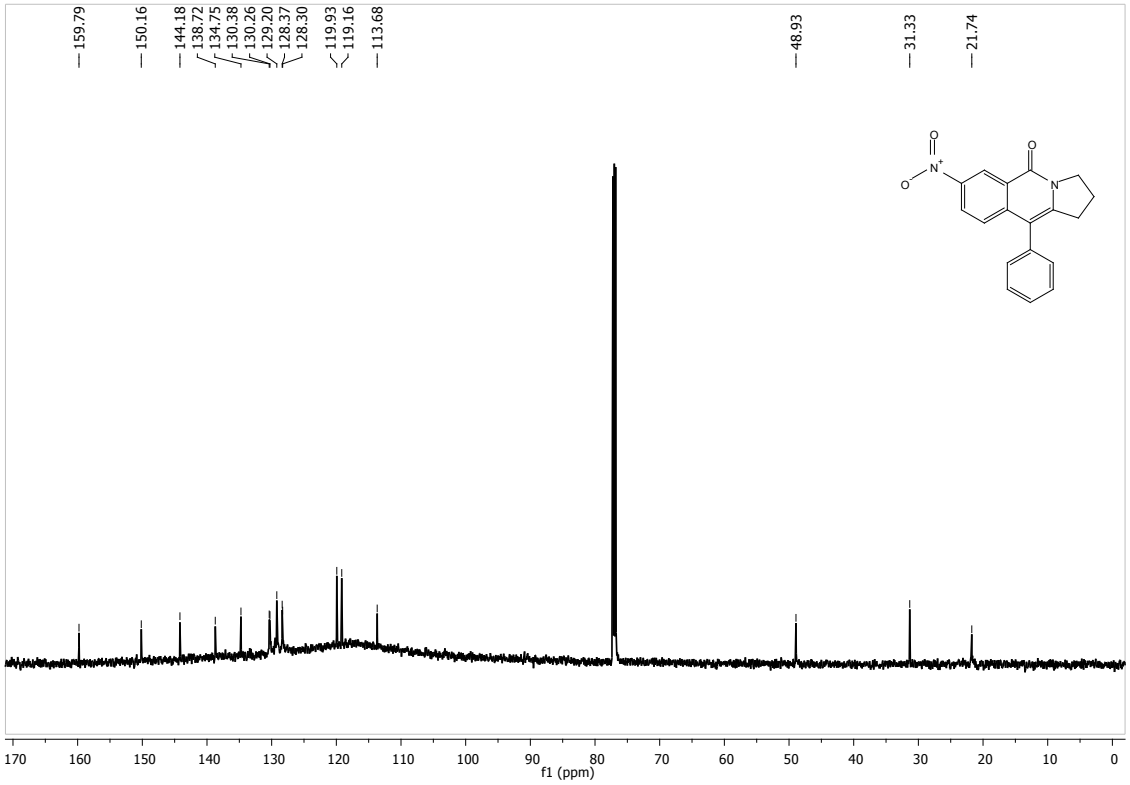
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3h



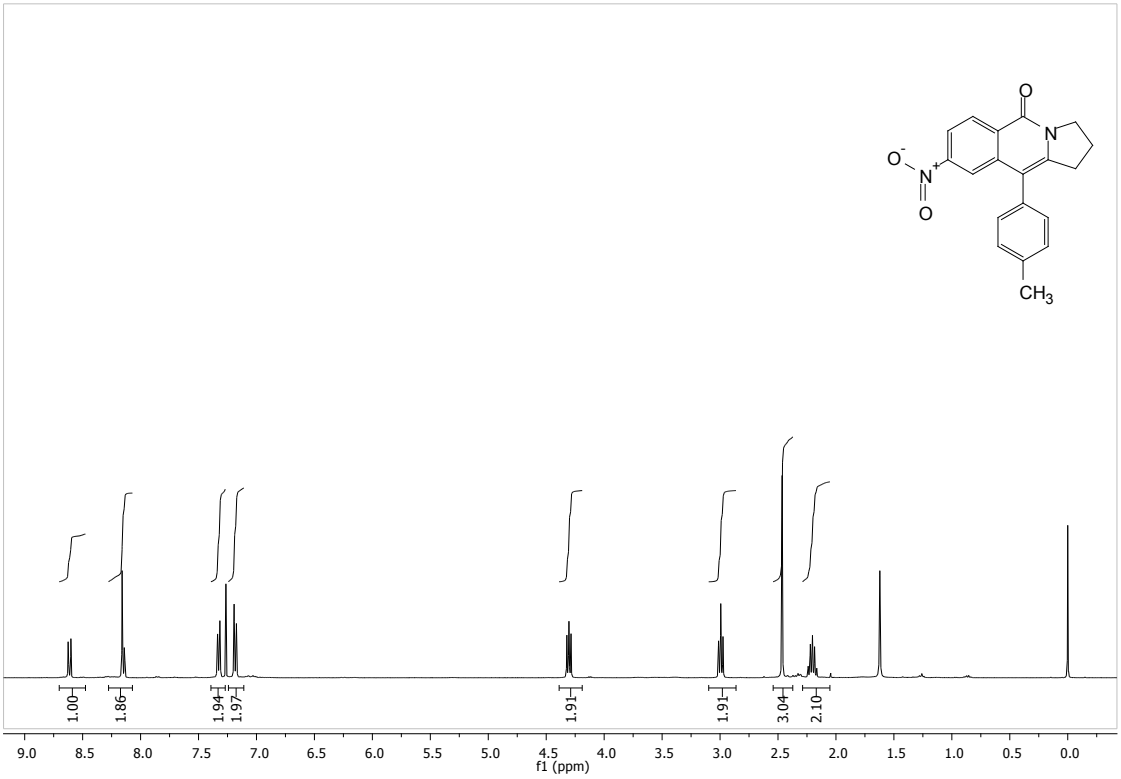
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3h



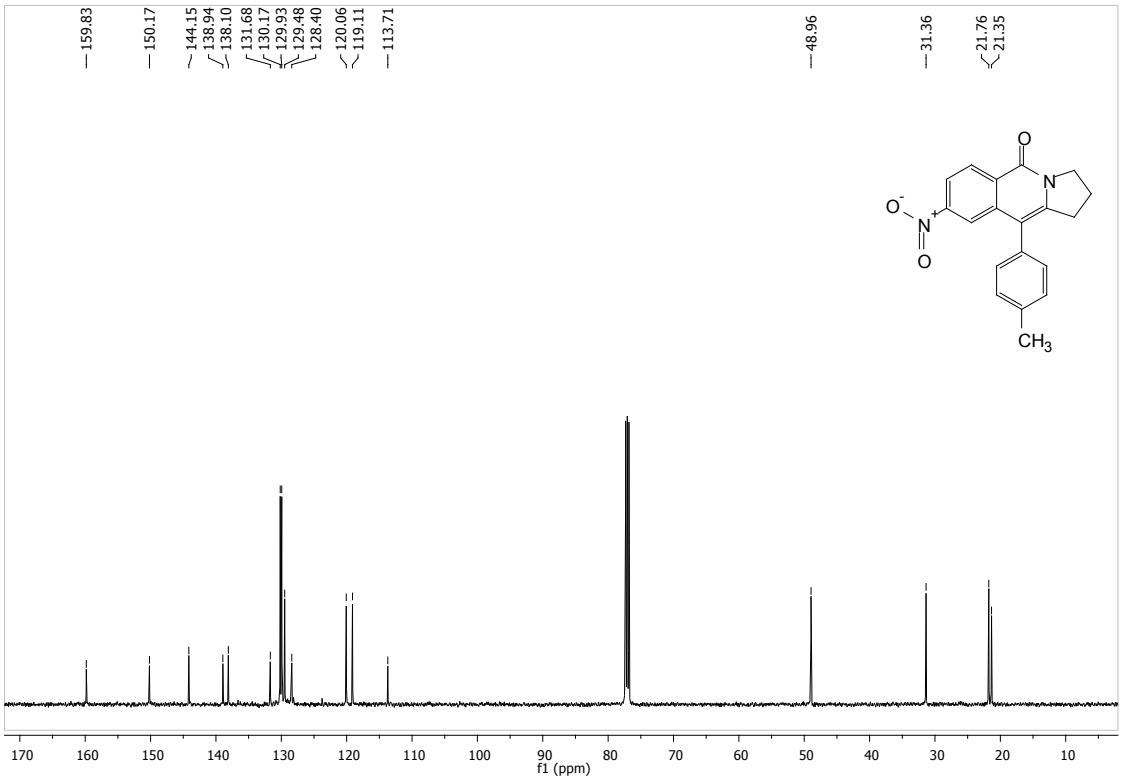
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3i



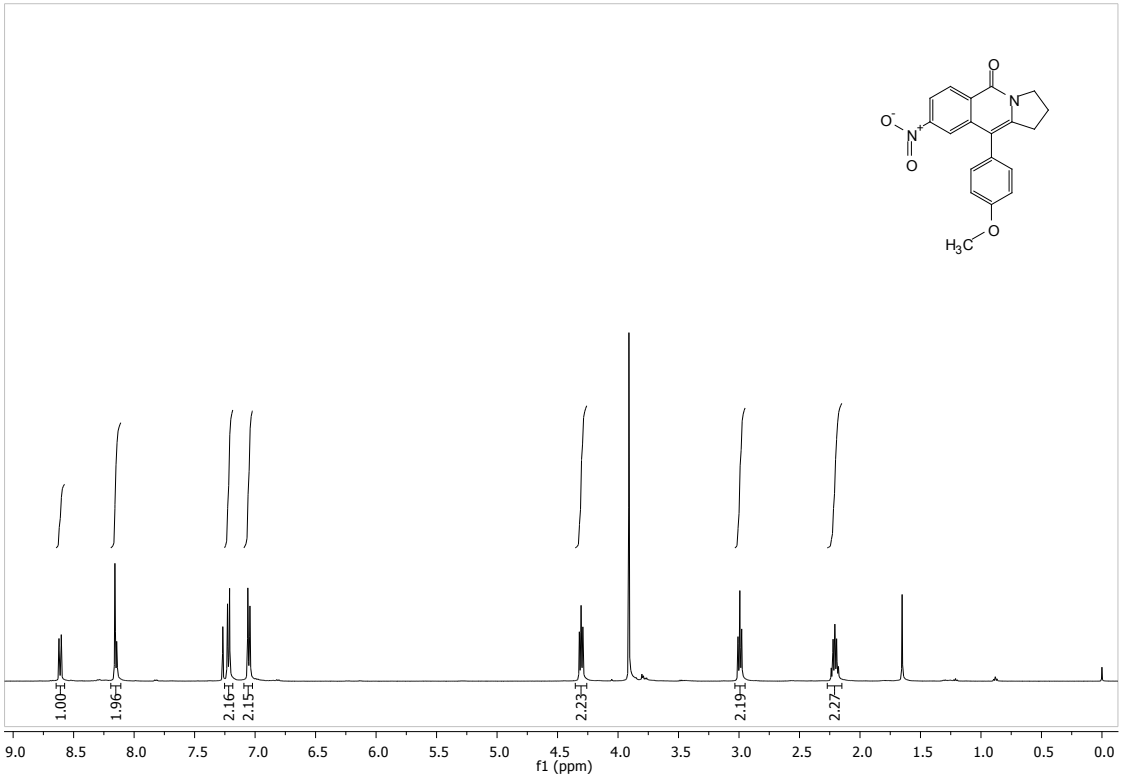
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3i



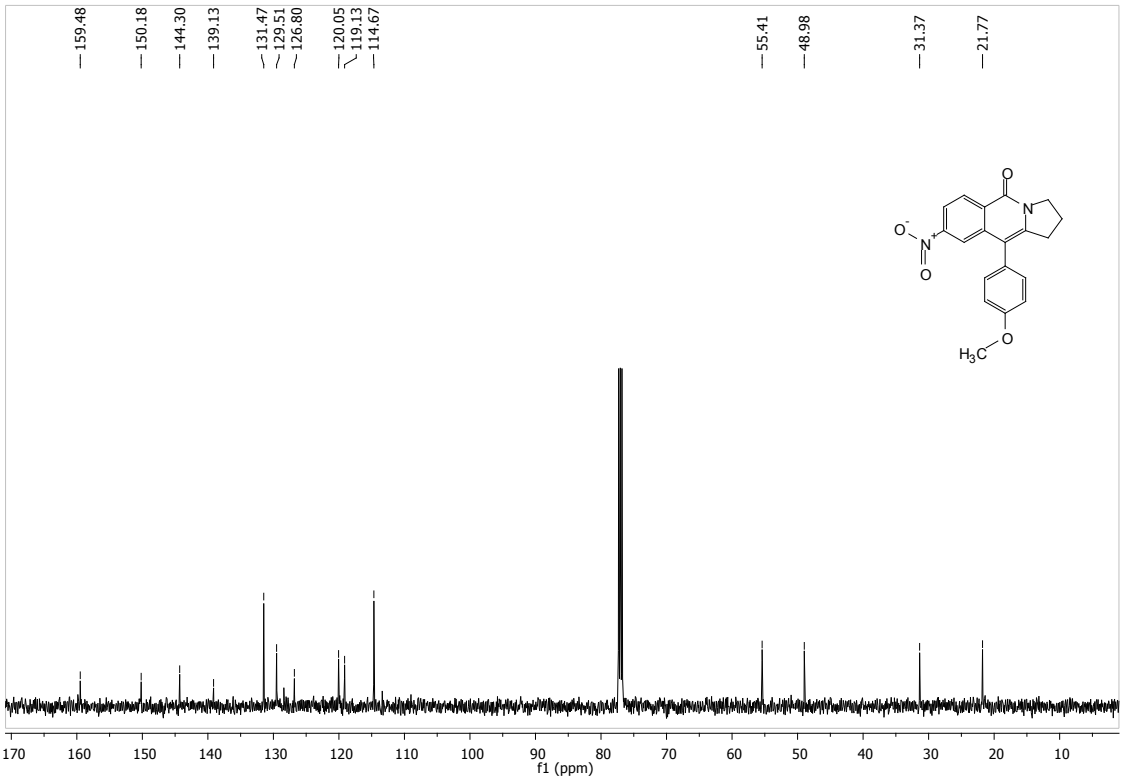
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3j



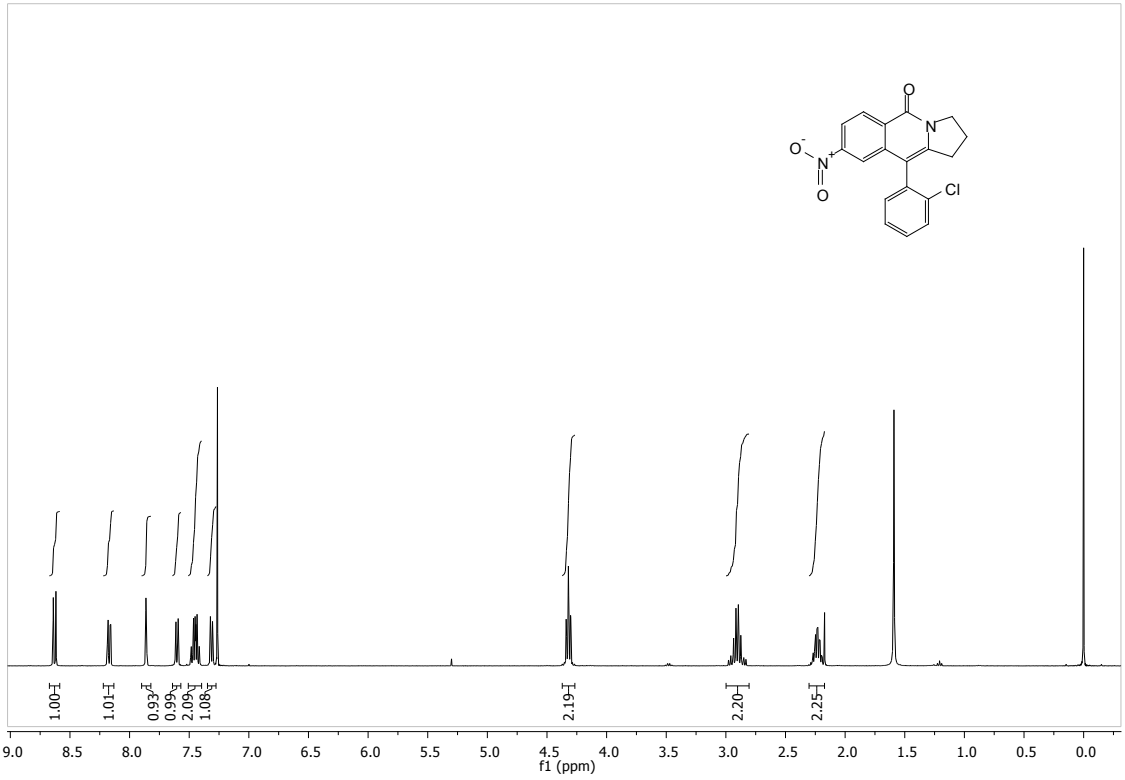
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3j



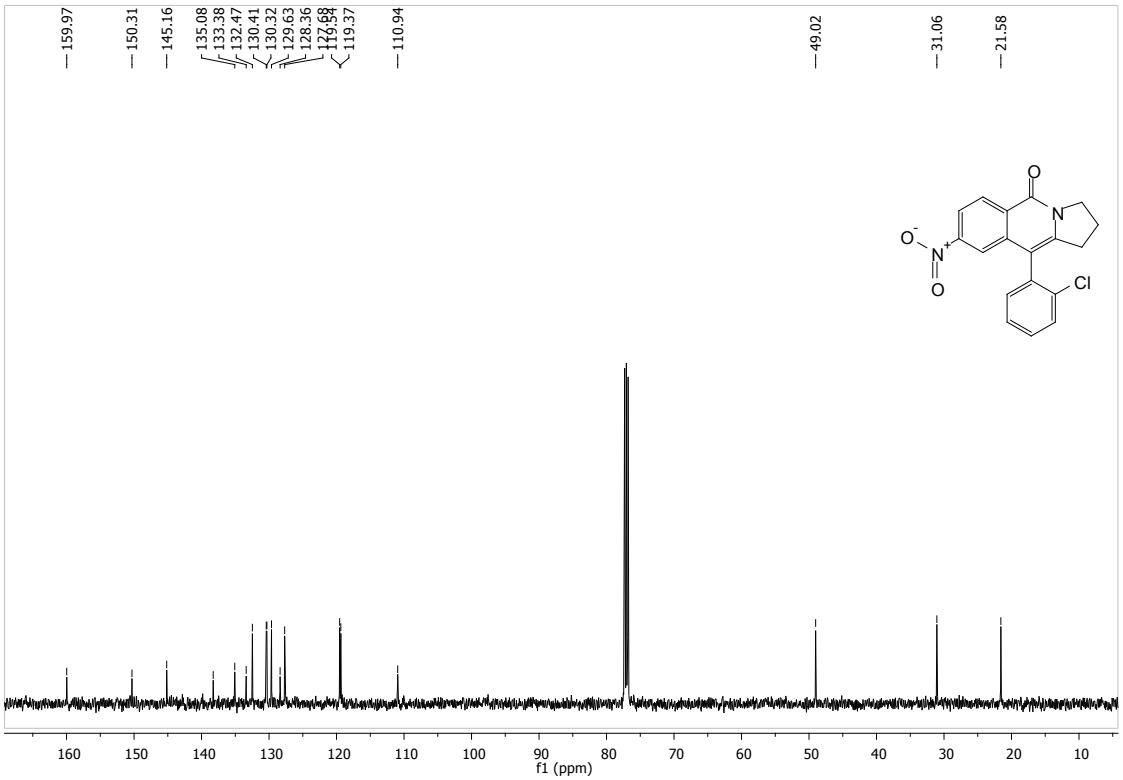
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3k



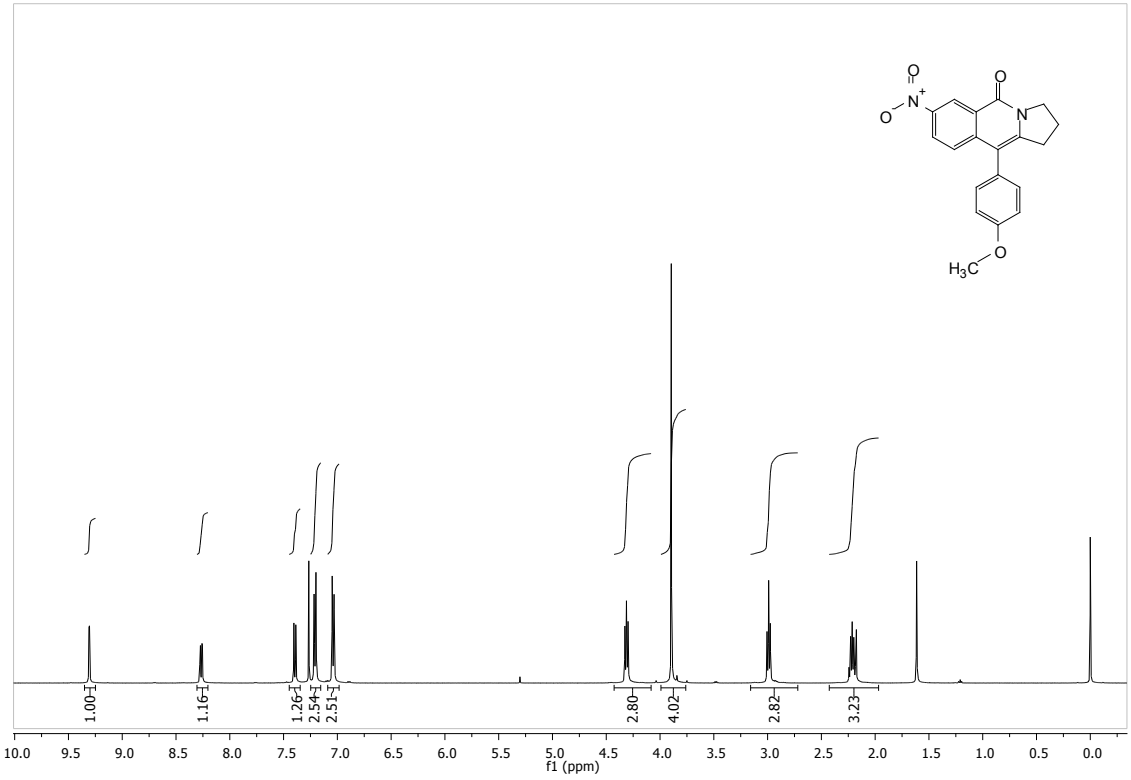
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3k



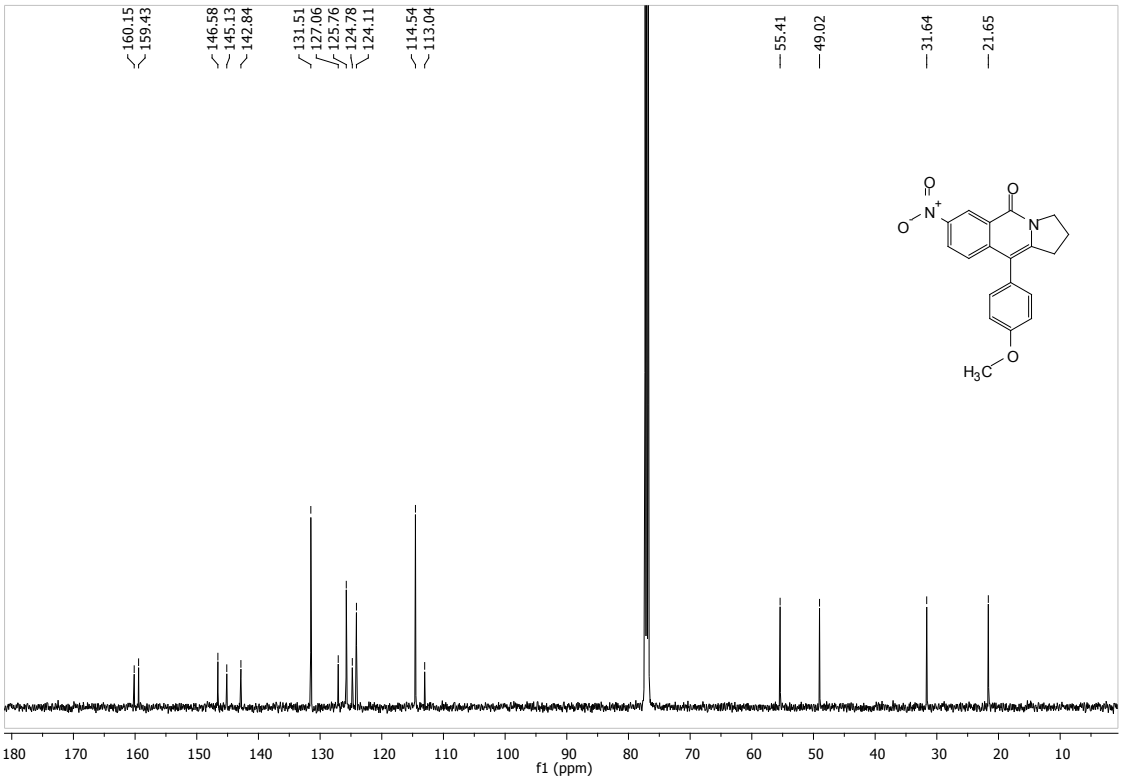
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3l



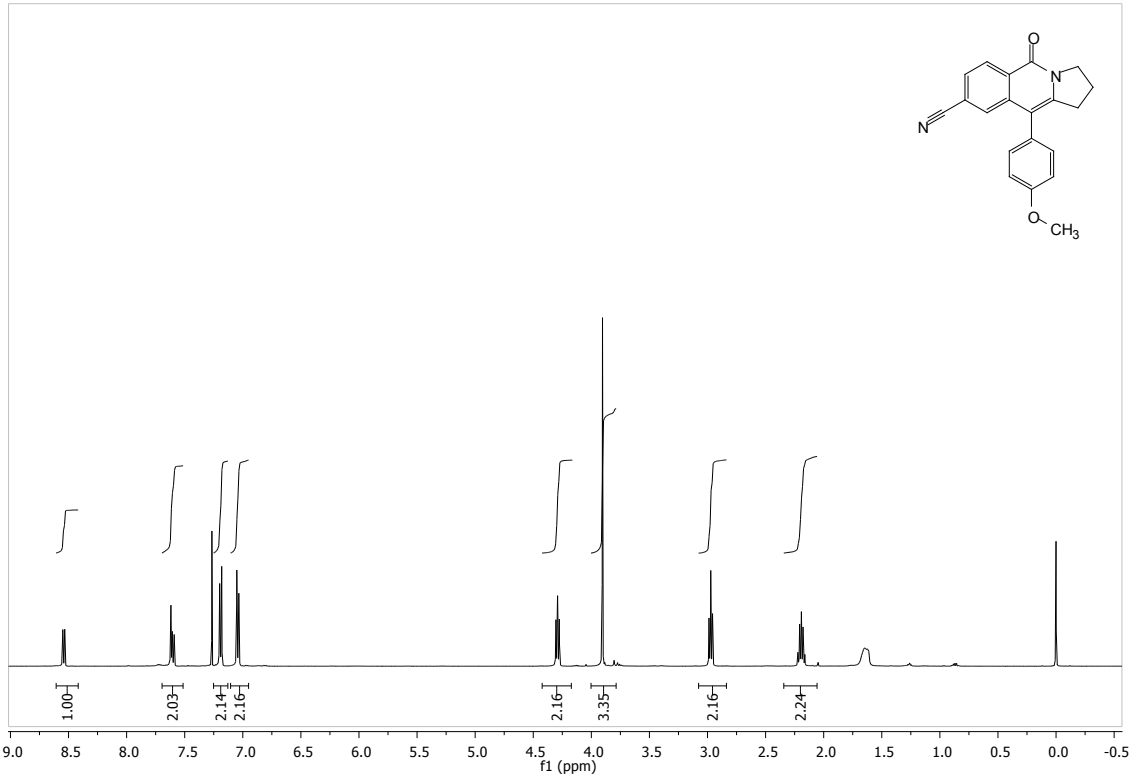
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3l



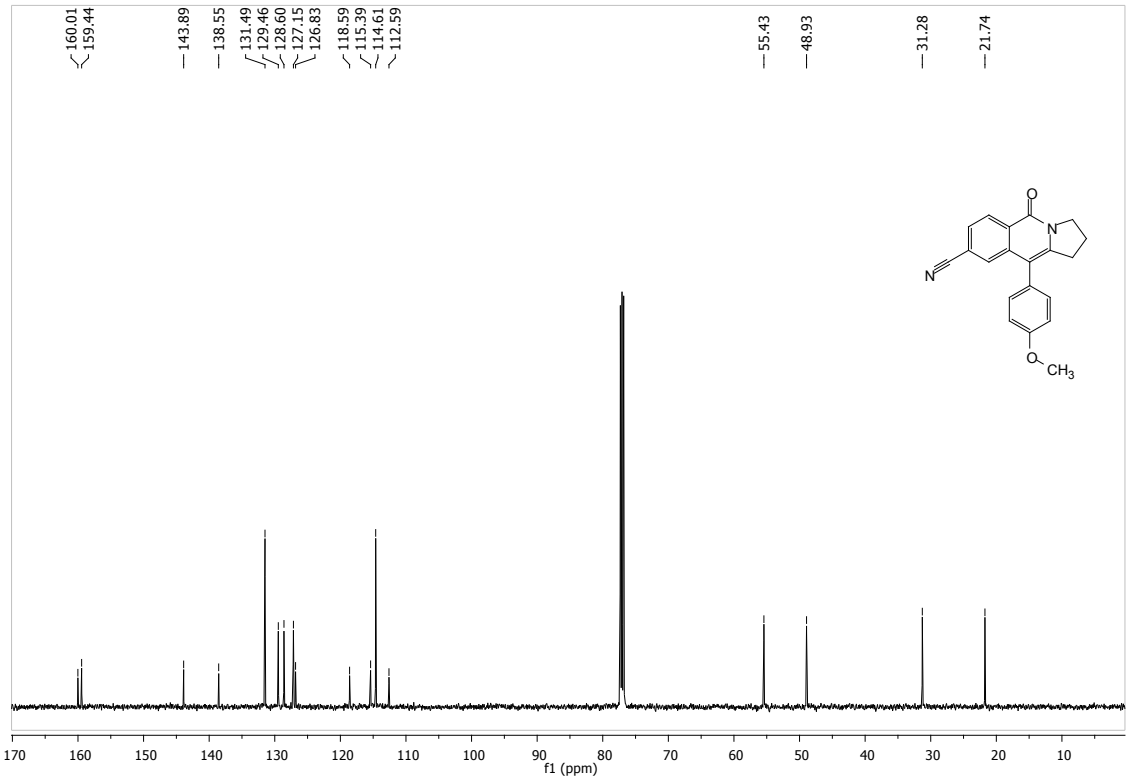
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3m



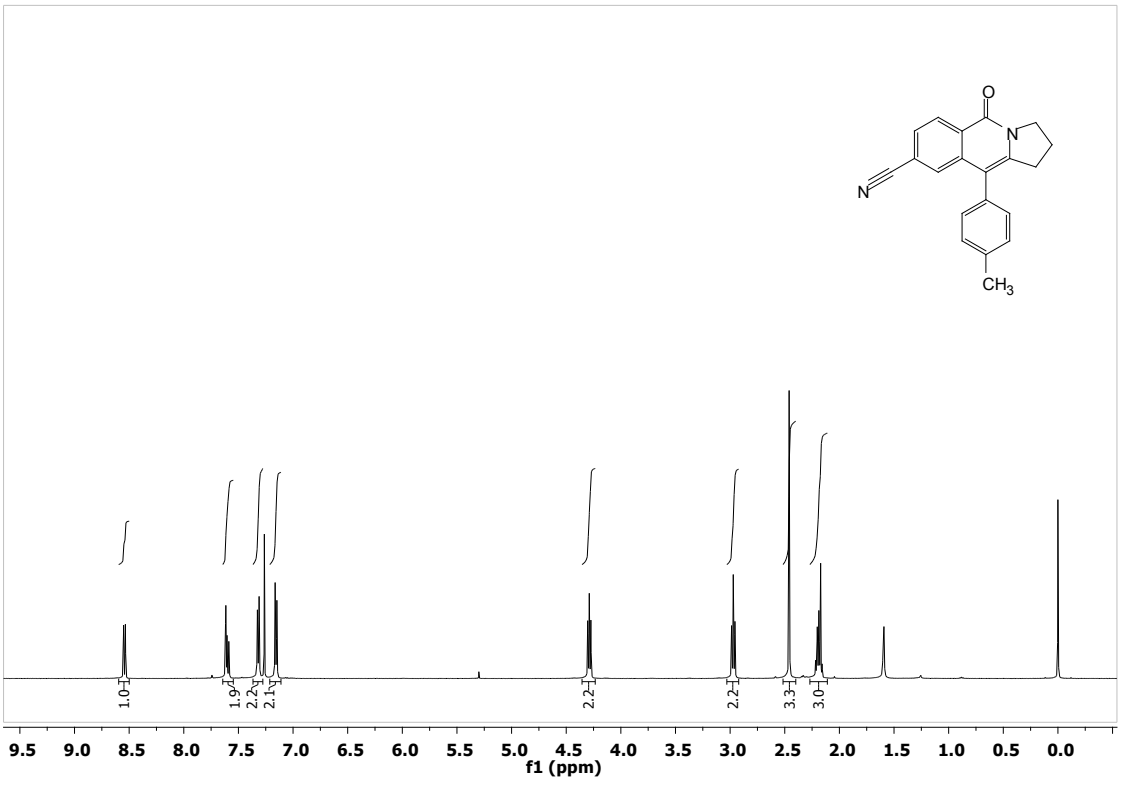
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3m



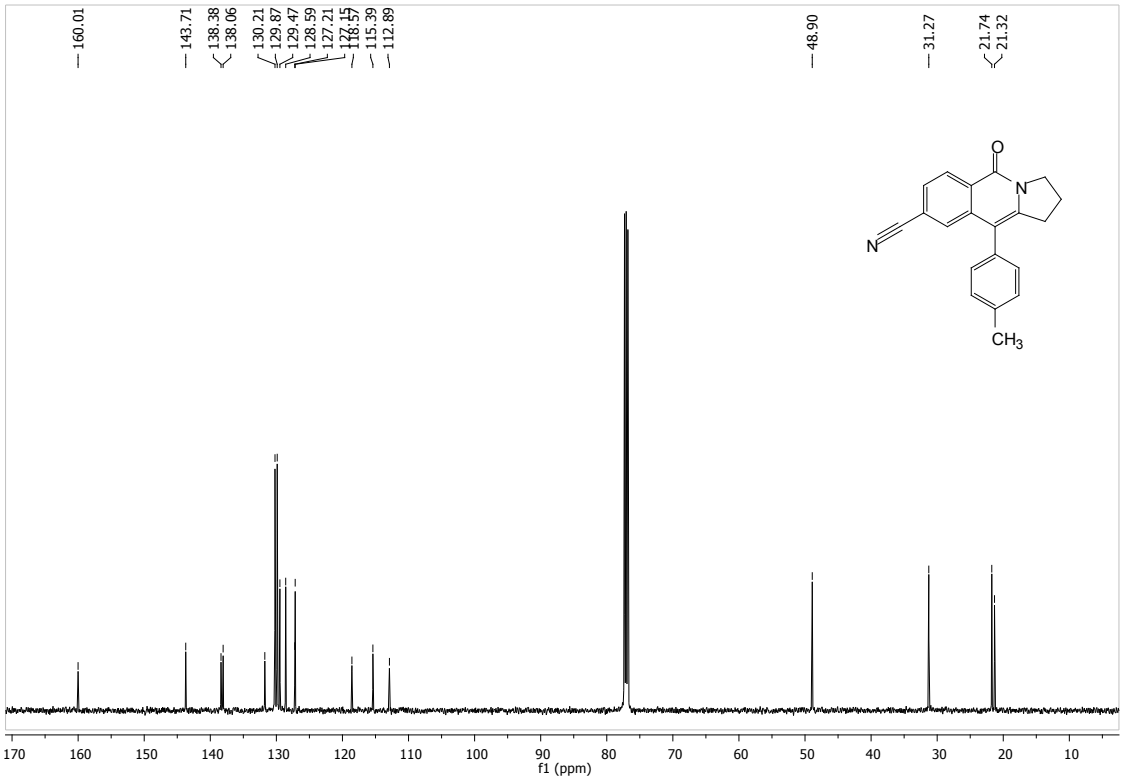
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3n



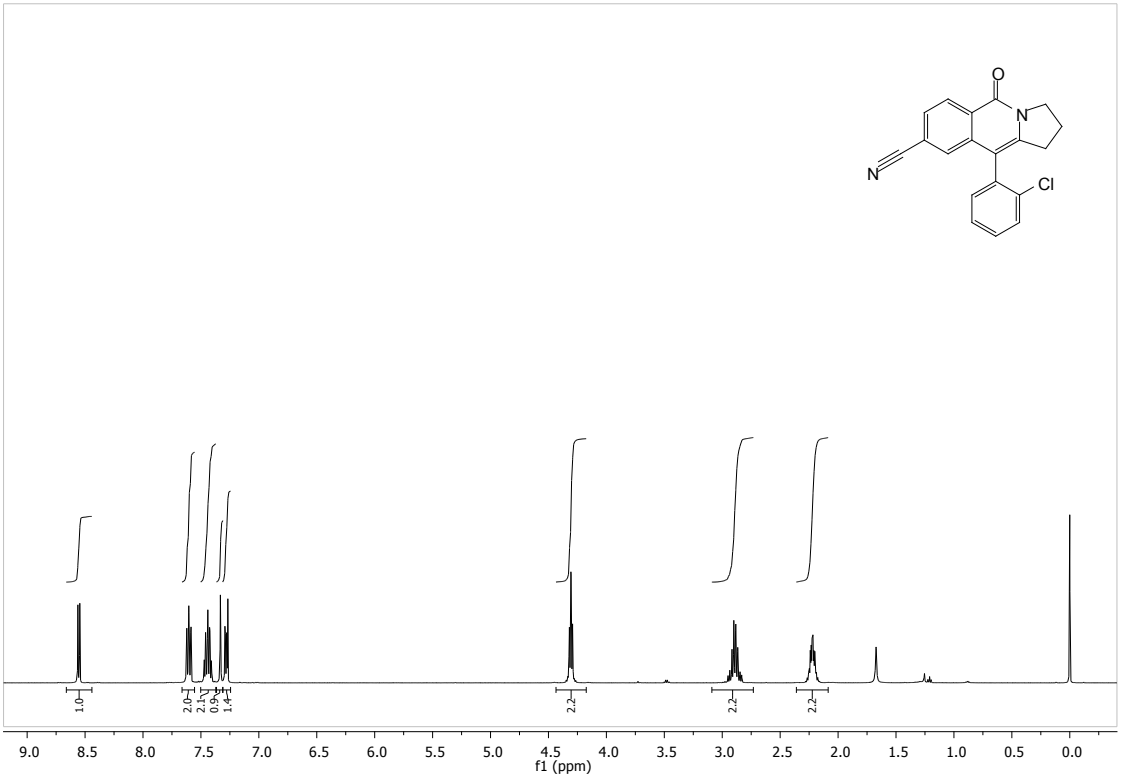
^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3n



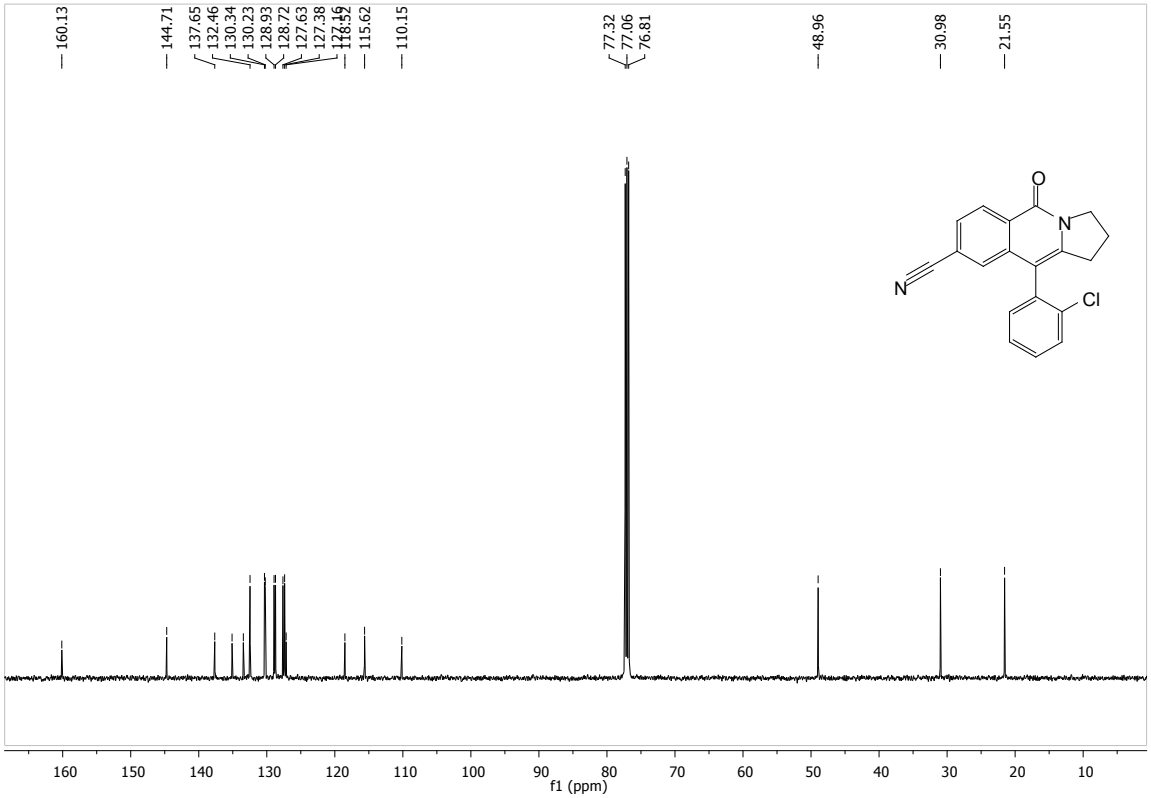
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3o



^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3o



^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound 3p



^{13}CNMR (125 MHz, $CDCl_3$) spectrum of compound 3p

3. X-ray Crystallography for 3e

X-ray data for the compounds were collected at room temperature using a Bruker Smart Apex CCD diffractometer with graphite monochromated MoKα radiation (λ=0.71073Å) with ω-scan method [1]. Preliminary lattice parameters and orientation matrices were obtained from four sets of frames.

Integration and scaling of intensity data were accomplished using SAINT program [1]. The structure was solved by direct methods using SHELXS [2] and refinement was carried out by full-matrix least-squares technique using SHELXL [2]. Anisotropic displacement parameters were included for all non-hydrogen atoms. The O-bound H atom of the water molecule (lying on the inversion centre) was located in a difference density map and was refined isotropically. However the isotropic displacement parameter of O bound H atom of (I) was fixed at 1.2 times the U_{eq} value of the parent atom. Distance restraint was also applied O-H bond distance. All other H atoms were positioned geometrically and treated as riding on their parent C atoms [C-H = 0.93-0.97 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H or $1.2U_{eq}(c)$ for other H atoms]. The methyl groups were allowed to rotate but not to tip.

Crystal Data for 3e: $2(C_{19}H_{17}NO_2)$, H_2O (M =600.69): monoclinic, space group C2/c (no. 15), a = 14.3475(11) Å, b = 11.0883(11) Å, c = 20.423(2) Å, β = 107.064(2)°, V = 3106.0(5) Å^3, Z = 4, T = 294(2) K, μ(MoKα) = 0.085 mm^{-1}, *Dcalc* = 1.285 g/mm^3, 16119 reflections measured ($4.172 \leq 2\Theta \leq 52.498$), 3145 unique ($R_{int}$ = 0.0183) which were used in all calculations. The final R_1 was 0.0524 ($I > 2\sigma(I)$) and wR_2 was 0.1545 (all data). CCDC 1061398 contains supplementary Crystallographic data for the structure. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0) 1223 336 033; email: deposit@ccdc.cam.ac.uk].

1. Bruker (2001). SAINT (Version 6.28a) & SMART (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.
2. Sheldrick G. M. (2015**)** Acta Crystallogr C71: 3-8.

Figure Caption

Fig.1. A view of **3e**, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by circles of arbitrary radii. The water molecule is sitting on the inversion centre.