

Supporting Information

Intramolecular 1,5-H transfer reaction of aryl iodides through visible-light photoredox catalysis: a concise method for the synthesis of the natural product scaffolds

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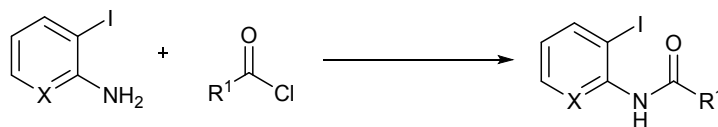
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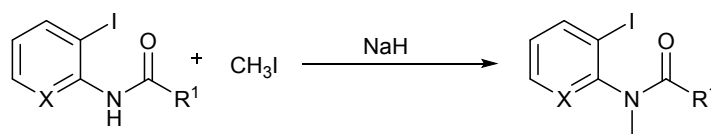
1. General information

All glassware was thoroughly oven-dried. Chemicals and solvents were either purchased from commercial suppliers or purified by standard techniques. Thin-layer chromatography plates were visualized by exposure to ultraviolet light and/or staining with phosphomolybdic acid followed by heating on a hot plate. Flash chromatography was carried out using silica gel (200–300 mesh). ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker AM-400 (400 MHz). The spectra were recorded in deuteriochloroform (CDCl_3) as solvent at room temperature, ^1H and ^{13}C NMR chemical shifts are reported in ppm relative to the residual solvent peak. The residual solvent signals were used as references and the chemical shifts were converted to the TMS scale (CDCl_3 : $\delta_{\text{H}} = 7.26$ ppm, $\delta_{\text{C}} = 77.0$ ppm). Data for ^1H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, dd = doublet, br = broad), integration, coupling constant (Hz) and assignment. Data for ^{13}C NMR are reported as chemical shift. IR spectra were recorded using Nicolet NEXUS 670 FT-IR instrument and are reported in wave numbers (cm^{-1}). HRMS were performed on a Bruker Apex II mass instrument (ESI).

2. General preparation of substrates¹

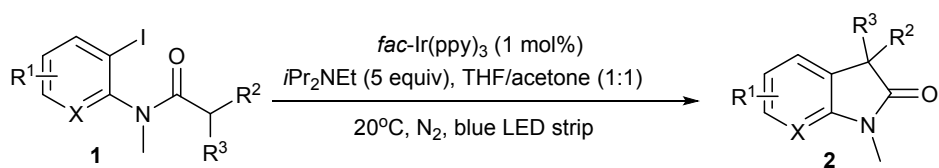


To a stirred, cooled (0–5°C) solution of 2-iodoaniline (2.190 g, 10 mmol) and Et₃N (1.113 g, 1.55 ml, 11 mmol) in 20 ml of dry THF a solution of an appropriate acyl chloride (10 mmol) in 5 ml of dry THF was added dropwise within 10 min. Then the ice bath was removed and the mixture was stirred vigorously for 30 min at room temperature. After solid Et₃N·HCl was filtered off and washed with THF (3 x 5 ml), the resulting organic fractions were combined and THF was removed under reduced pressure to yield crude amides. Recrystallization from hexane/CHCl₃ and drying in vacuum afforded analytically pure compounds.



To a stirred suspension of NaH (0.132 g; 5.5 mmol) in 5 ml of dry THF at 0°C, the respective amide (5 mmol) dissolved in 10 ml of THF was added dropwise within 10 min. The reaction mixture was stirred until the solution became clear (30 min, hydrogen gas evolved), and the solution of MeI (0.9226 g; 0.405 ml; 6.5 mmol) in 5 ml of THF was added dropwise within 10 min. The solution was warmed up to room temperature and stirred for 3 h. Then the reaction mixture was quenched with water (30 ml). The resulting solution was extracted with ethyl acetate (3 x 20 ml). Combined organic layers were washed with brine (1 x 20 ml) and dried over Na₂SO₄. Ethyl acetate was removed under reduced pressure to give crude products. Recrystallization from hexane or hexane/CHCl₃ and drying in vacuum afforded analytically pure compounds.

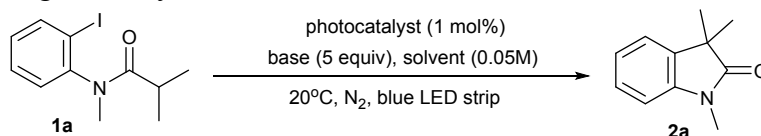
3. General procedure for intramolecular 1,5-HAT reaction



o-anilide aryl iodide **1** (0.2 mmol) and DIPEA (1.0 mmol) were added to a solution of photocatalyst *fac*-Ir(ppy)₃ (1 mol%) in dry THF/acetone (1:1) (4mL) at room temperature. The heterogenous mixture was degassed by three cycles of freeze-pump-thaw and then placed in the irradiation apparatus equipped with a 25 W blue light-emitting diode (LED) strip. The resulting mixture was stirred at 20°C until the starting material was completely consumed as monitored by TLC. Upon completion of the reaction, the reaction mixture was concentrated under reduced pressure, and the resulting crude mixture was purified by flash column chromatography on silica gel, which furnished the title compounds as described. (Attention: THF should be purified by standard techniques before using)

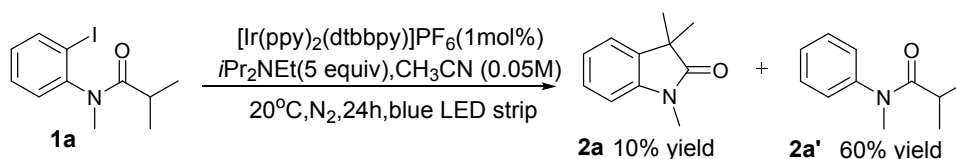
4. Initial studies and reaction optimization

Table 1 Screening of catalysts, bases, and solvents for 1,5-HAT reaction of **1a**



Entry	Photocatalyst	Base	Solvent	Light	Time	Yield(%) ^a
1	Ru(bpy) ₃ Cl ₂	iPr ₂ NEt	MeCN	blue LED	24h	0
2	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	MeCN	blue LED	24h	37
3	[Ir(ppy) ₂ (dtbbpy)]PF ₆	iPr ₂ NEt	MeCN	blue LED	24h	10
4	<i>fac</i> -Ir(ppy) ₃	Et ₃ N	MeCN/CH ₂ Cl ₂ (1:1)	blue LED	24h	46
5	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	MeCN/CH ₂ Cl ₂ (1:1)	blue LED	24h	57
6	<i>fac</i> -Ir(ppy) ₃	Bu ₃ N	MeCN/CH ₂ Cl ₂ (1:1)	blue LED	24h	49
7	<i>fac</i> -Ir(ppy) ₃	iBu ₃ N	MeCN/CH ₂ Cl ₂ (1:1)	blue LED	24h	52
8	<i>fac</i> -Ir(ppy) ₃	K ₂ HPO ₄	DMSO	blue LED	24h	0
9	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	CH ₂ Cl ₂	blue LED	24h	51
10	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	DCE	blue LED	36h	36
11	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	DMA	blue LED	36h	34
12	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	THF	blue LED	7d	78
13	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	Toluene	blue LED	7d	61
14	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	DMSO	blue LED	12h	44
15	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	Acetone	blue LED	36h	67
16	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	MeOH	blue LED	36h	46
17	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	CHCl ₃	blue LED	36h	0
18	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	DMF	blue LED	12h	51
19	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	EA	blue LED	5d	46
20	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	THF/H ₂ O(7:1)	blue LED	60h	82
21	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	THF/Acetone(1:1)	blue LED	60h	87
22	<i>fac</i> -Ir(ppy) ₃	Et ₃ N	THF/Acetone(1:1)	blue LED	60h	75
23	<i>fac</i> -Ir(ppy) ₃	Bu ₃ N	THF/Acetone(1:1)	blue LED	60h	77
24	<i>fac</i> -Ir(ppy) ₃	2,6-lutidine	THF/Acetone(1:1)	blue LED	60h	0
25	<i>fac</i> -Ir(ppy) ₃	K ₂ HPO ₄	THF/Acetone(1:1)	blue LED	60h	0
26	<i>fac</i> -Ir(ppy) ₃	-	THF/Acetone(1:1)	blue LED	60h	0
27	<i>fac</i> -Ir(ppy) ₃	iPr ₂ NEt	THF/Acetone(1:1)	-	60h	0
28	-	iPr ₂ NEt	THF/Acetone(1:1)	blue LED	60h	0

^a Isolated yield.



when [Ir(ppy)₂(dtbbpy)]PF₆ was used instead of *fac*-Ir(ppy)₃, the yield of the product was decreased to 10%.

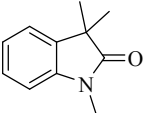
5. Devices for the photocatalytic reactions



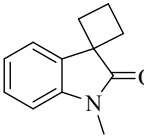
Figure 1. Devices for the photocatalytic reactions

6. Characterization of products

1,3,3-trimethylindolin-2-one (2a)

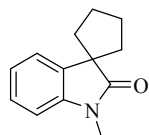
 Colorless oil; 87% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 1.37 (s, 6H), 3.22 (s, 3H), 6.85 (d, $J = 7.8$ Hz, 1H), 7.06 (m, 1H), 7.21 (dd, $J = 7.4, 0.7$ Hz, 1H), 7.24–7.28 (m, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 24.3, 26.2, 44.2, 108.0, 122.2, 122.4, 127.6, 135.8, 142.6, 181.4; IR (KBr, cm^{-1}): 3504, 2967, 2927, 1710, 1613, 1493, 1472, 1382, 1348, 1247, 1125, 757, 562. HRMS (ESI) for $\text{C}_{11}\text{H}_{13}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 176.1070, found 176.1067.

1'-methylspiro[cyclobutane-1,3'-indolin]-2'-one (2b)

 Colorless oil; 67% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 2.23–2.37 (m, 4H), 2.61–2.69 (m, 2H), 3.18 (s, 3H), 6.78 (d, $J = 7.8$ Hz, 1H), 7.07–7.11 (m, 1H), 7.23–7.28 (m, 1H), 7.51 (dd, $J = 7.3, 0.7$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 16.7, 26.1, 31.3, 48.1, 107.6, 122.2,

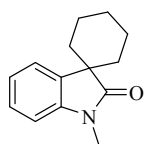
122.5, 127.8, 134.4, 143.0, 180.2; IR (KBr, cm^{-1}): 3401, 2936, 1705, 1639, 1614, 1469, 1375, 1349, 1267, 1101, 1006, 740, 543. HRMS (ESI) for $\text{C}_{12}\text{H}_{13}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd.188.1070, found 188.1067.

1'-methylspiro[cyclopentane-1,3'-indolin]-2'-one (2c)



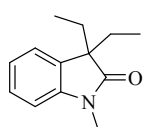
Colorless oil; 93% yield; ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 1.80–1.86 (m, 2H), 1.86–2.18 (m, 6H), 3.21 (s, 3H), 6.82 (d, $J = 7.8$ Hz, 1H), 7.02–7.06 (m, 1H), 7.20 (d, $J = 7.4$ Hz, 1H), 7.22–7.26 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 26.2, 26.6, 38.3, 53.9, 107.7, 122.2, 122.5, 127.3, 136.9, 142.9, 181.9; IR (KBr, cm^{-1}): 3405, 2954, 2867, 1709, 1656, 1612, 1468, 1375, 1347, 1264, 1124, 1077, 968, 745, 543. HRMS (ESI) for $\text{C}_{13}\text{H}_{16}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd.202.1226, found 202.1224.

1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2d)



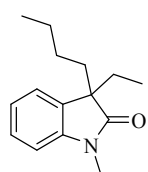
Colorless oil; 90% yield; ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 1.56–1.59 (m, 2H), 1.61–1.79 (m, 4H), 1.81–1.88 (m, 2H), 1.92–1.98 (m, 2H), 3.20 (s, 3H), 6.84 (d, $J = 7.7$ Hz, 1H), 7.02–7.06 (m, 1H), 7.25–7.27 (m, 1H), 7.25 (d, $J = 7.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 21.2, 25.2, 26.1, 33.0, 47.4, 107.8, 121.9, 123.8, 127.4, 135.4, 142.8, 180.7; IR (KBr, cm^{-1}): 3399, 3053, 2931, 2851, 1709, 1612, 1492, 1470, 1377, 1350, 1252, 1081, 1006, 744, 543. HRMS (ESI) for $\text{C}_{14}\text{H}_{18}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd.216.1383, found 216.1379.

3,3-diethyl-1-methylindolin-2-one (2e)



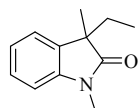
Colorless oil; 87% yield; ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 0.56 (t, $J = 7.4$ Hz, 6H), 1.74–1.83 (m, 2H), 1.88–1.97 (m, 2H), 3.20 (s, 3H), 6.84 (d, $J = 7.8$ Hz, 1H), 7.05–7.09 (m, 1H), 7.12–7.14 (m, 1H), 7.24–7.29 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 8.6, 25.9, 30.6, 54.3, 107.6, 122.3, 122.7, 127.6, 132.0, 144.4, 180.0; IR (KBr, cm^{-1}): 3407, 3054, 2966, 2929, 1709, 1612, 1493, 1468, 1377, 1338, 1254, 1124, 1079, 1021, 749, 546. HRMS (ESI) for $\text{C}_{13}\text{H}_{18}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd.204.1383, found 204.1380.

3-ethyl-1-methyl-3-propylindolin-2-one (2f)



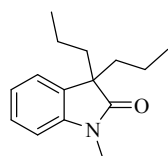
Colorless oil; 89% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 0.55 (t, $J = 7.4$ Hz, 3H), 0.72–0.84 (m, 4H), 0.89–0.97 (m, 1H), 1.14–1.21 (m, 2H), 1.70–1.82 (m, 2H), 1.84–1.94 (m, 2H), 3.21 (s, 3H), 6.83 (d, $J = 7.8$ Hz, 1H), 7.05–7.09 (m, 1H), 7.12–7.14 (m, 1H), 7.24–7.28 (m, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 8.5, 13.8, 22.9, 25.9, 26.4, 31.0, 37.5, 53.7, 107.6, 122.3, 122.6, 127.5, 132.4, 144.2, 180.2; IR (KBr, cm^{-1}): 3409, 3055, 2960, 2933, 2875, 1716, 1613, 1493, 1469, 1377, 1350, 1252, 1123, 1075, 1021, 750, 542. HRMS (ESI) for $\text{C}_{13}\text{H}_{18}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 232.1696, found 232.1693.

3-ethyl-1,3-dimethylindolin-2-one (2g)



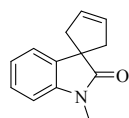
Colorless oil; 83% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 0.59 (t, $J = 7.4$ Hz, 3H), 1.35 (s, 3H), 1.73–1.82 (m, 1H), 1.89–1.97 (m, 1H), 3.21 (s, 3H), 6.84 (d, $J = 7.8$ Hz, 1H), 7.05–7.09 (m, 1H), 7.16–7.18 (m, 1H), 7.24–7.28 (m, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 8.8, 23.3, 26.0, 31.4, 48.9, 107.8, 122.4, 122.5, 127.6, 133.9, 143.5, 180.7; IR (KBr, cm^{-1}): 3409, 3055, 2966, 2928, 2877, 1713, 1613, 1493, 1471, 1378, 1349, 1258, 1124, 1078, 1017, 751, 549. HRMS (ESI) for $\text{C}_{13}\text{H}_{18}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 190.1226, found 190.1223.

1-methyl-3,3-dipropylindolin-2-one (2h)



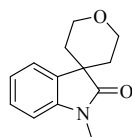
Colorless oil; 67% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 0.76 (t, $J = 6.9$ Hz, 3H), 0.79–0.85 (m, 2H), 0.94–1.03 (m, 2H), 1.67–1.74 (m, 2H), 1.82–1.90 (m, 2H), 3.20 (s, 3H), 6.82 (d, $J = 7.8$ Hz, 1H), 7.04–7.08 (m, 1H), 7.13–7.15 (m, 1H), 7.23–7.27 (m, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 14.1, 17.5, 25.9, 40.3, 53.3, 107.6, 122.3, 122.6, 127.4, 132.8, 144.0, 180.3; IR (KBr, cm^{-1}): 3409, 3054, 2958, 2933, 2873, 1711, 1613, 1493, 1467, 1378, 1347, 1248, 1126, 1076, 1019, 747, 543. HRMS (ESI) for $\text{C}_{15}\text{H}_{22}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 232.1696, found 232.1692.

1'-methylspiro[cyclopent[3]ene-1,3'-indolin]-2'-one (2i)



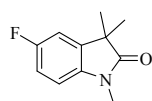
Yellow oil; 65% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 2.59 (d, J = 14.6 Hz, 2H), 3.00 (d, J = 14.7 Hz, 2H), 3.22 (s, 3H), 5.84 (s, 1H), 6.82 (dd, J = 7.9, 0.8 Hz, 1H), 7.00–7.04 (m, 1H), 7.23–7.27 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 26.3, 44.9, 52.1, 107.7, 121.6, 122.8, 127.6, 128.9, 137.4, 142.6, 181.4; IR (KBr, cm^{-1}): 3398, 2918, 2839, 1708, 1611, 1494, 1470, 1378, 1346, 1266, 1185, 1085, 1029, 756, 689, 542, 470. HRMS (ESI) for $\text{C}_{13}\text{H}_{14}\text{NO}$ [$\text{M}+\text{H}$] $^+$ calcd. 200.1070, found 200.1067.

1-methyl-2',3',5',6'-tetrahydrospiro[indoline-3,4'-pyran]-2-one (2j)



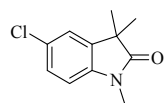
White solid; 70% yield; mp 85–86 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 1.86 (t, J = 6.0 Hz, 4H), 3.21 (s, 3H), 3.90–3.95 (m, 2H), 4.23–4.29 (m, 2H), 6.86 (d, J = 7.8 Hz, 1H), 7.06–7.10 (m, 1H), 7.28–7.32 (m, 1H), 7.38 (d, J = 7.4 Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 26.0, 32.9, 44.2, 62.9, 108.0, 122.4, 123.0, 127.9, 134.0, 142.7, 179.6; IR (KBr, cm^{-1}): 3398, 2946, 2861, 1703, 1611, 1477, 1458, 1371, 1348, 1245, 1103, 1076, 1023, 1006, 836, 760, 751, 695, 538, 490. HRMS (ESI) for $\text{C}_{13}\text{H}_{16}\text{NO}_2$ [$\text{M}+\text{H}$] $^+$ calcd. 218.1176, found 218.1172.

5-fluoro-1,3,3-trimethylindolin-2-one (2k)



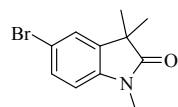
White solid; 87% yield; mp 81–83 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 1.37 (s, 6H), 3.20 (s, 3H), 6.76 (dd, J = 9.2, 4.1 Hz, 1H), 6.93–6.98 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 24.3, 26.3, 44.6 (d, J = 1.8 Hz), 108.3 (d, J = 8.1 Hz), 110.5 (d, J = 24.3 Hz), 113.7 (d, J = 23.3 Hz), 137.5 (d, J = 7.8 Hz), 138.5 (d, J = 1.8 Hz), 159.4 (d, J = 239 Hz), 180.9; IR (KBr, cm^{-1}): 3387, 3067, 2968, 2925, 2866, 1700, 1620, 1501, 1485, 1460, 1353, 1275, 1187, 1114, 1045, 886, 818, 696, 616, 559, 467. HRMS (ESI) for $\text{C}_{11}\text{H}_{13}\text{FNO}$ [$\text{M}+\text{H}$] $^+$ calcd. 194.0976, found 194.0973.

5-chloro-1,3,3-trimethylindolin-2-one (2l)



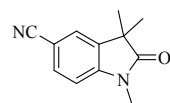
White solid; 82% yield; mp 86–87°C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 1.37 (s, 6H), 3.20 (s, 3H), 6.76 (d, $J = 8.2$ Hz, 1H), 7.18 (d, $J = 2.0$ Hz, 1H), 7.23 (dd, $J = 8.2, 2.0$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 24.3, 26.3, 44.4, 108.9, 122.9, 127.6, 127.9, 137.5, 141.2, 180.8; IR (KBr, cm^{-1}): 3395, 2976, 2930, 2866, 1706, 1610, 1490, 1467, 1426, 1345, 1266, 1242, 1125, 1087, 813, 587, 543, 468. HRMS (ESI) for $\text{C}_{11}\text{H}_{13}\text{ClNO}$ $[\text{M}+\text{H}]^+$ calcd. 210.0680, found 210.0677.

5-bromo-1,3,3-trimethylindolin-2-one (2m)



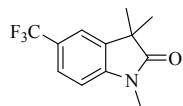
White solid; 80% yield; mp 102–104°C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 1.29 (s, 6H), 3.12 (s, 3H), 6.65 (d, $J = 8.2$ Hz, 1H), 7.23 (d, $J = 1.9$ Hz, 1H), 7.23 (dd, $J = 8.2, 1.9$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 24.3, 26.3, 44.4, 109.5, 115.2, 125.7, 130.5, 137.9, 141.7, 180.7; IR (KBr, cm^{-1}): 3398, 2966, 2925, 2863, 1716, 1606, 1488, 1468, 1415, 1361, 1342, 1264, 1242, 1126, 1077, 814, 560, 532, 463. HRMS (ESI) for $\text{C}_{11}\text{H}_{13}\text{BrNO}$ $[\text{M}+\text{H}]^+$ calcd. 254.0175, found 254.0171.

1,3,3-trimethyl-2-oxindoline-5-carbonitrile (2n)



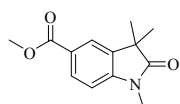
Light yellow solid; 89% yield; mp 131–132°C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 1.40 (s, 6H), 3.26 (s, 3H), 6.93 (d, $J = 8.1$ Hz, 1H), 7.46 (d, $J = 1.4$ Hz, 1H), 7.61 (dd, $J = 8.1, 1.4$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 24.2, 26.4, 44.0, 105.6, 108.5, 119.3, 125.7, 133.2, 136.7, 146.6, 180.9; IR (KBr, cm^{-1}): 3414, 2973, 2947, 2217, 1712, 1615, 1492, 1462, 1365, 1343, 1283, 1241, 1177, 1118, 1060, 1040, 826, 610, 559, 492. HRMS (ESI) for $\text{C}_{12}\text{H}_{13}\text{N}_2\text{O}$ $[\text{M}+\text{H}]^+$ calcd. 201.1022, found 201.1018.

1,3,3-trimethyl-5-(trifluoromethyl)indolin-2-one (2o)



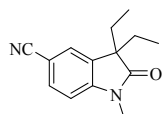
Light yellow solid; 92% yield; mp 37–39°C; **¹H NMR (400 MHz, CDCl₃)** δ (ppm) = 1.40 (s, 6H), 3.25 (s, 3H), 6.92 (d, *J* = 8.2 Hz, 1H), 7.43 (d, *J* = 1.0 Hz, 1H), 7.55 (dd, *J* = 8.2, 1.0 Hz, 1H); **¹³C NMR (100 MHz, CDCl₃)** δ (ppm) = 24.1, 26.4, 44.2, 107.7, 119.3 (q, *J* = 3.6 Hz), 124.5 (q, *J* = 270 Hz), 124.7 (q, *J* = 32.3 Hz), 125.5 (q, *J* = 3.9 Hz), 136.3, 145.7, 181.2; IR (KBr, cm⁻¹): 3424, 2973, 2930, 1721, 1624, 1503, 1467, 1384, 1327, 1261, 1158, 1116, 1076, 1053, 890, 835, 707, 653, 562, 536, 474. HRMS (ESI) for C₁₂H₁₃F₃NO [M+H]⁺ calcd. 244.0944, found 244.0940.

methyl 1,3,3-trimethyl-2-oxindoline-5-carboxylate (2p)



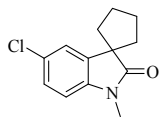
White solid; 90% yield; mp 96–97°C; **¹H NMR (400 MHz, CDCl₃)** δ (ppm) = 1.40 (s, 6H), 3.26 (s, 3H), 3.92 (s, 3H), 6.89 (d, *J* = 8.2 Hz, 1H), 7.89 (d, *J* = 1.6 Hz, 1H), 8.02 (dd, *J* = 8.2, 1.6 Hz, 1H); **¹³C NMR (100 MHz, CDCl₃)** δ (ppm) = 24.2, 26.3, 43.9, 51.9, 107.5, 123.5, 124.3, 130.4, 135.6, 146.8, 166.9, 181.5; IR (KBr, cm⁻¹): 3409, 2978, 2947, 1709, 1616, 1496, 1456, 1363, 1290, 1242, 1192, 1106, 1062, 938, 834, 773, 710, 561, 544, 472. HRMS (ESI) for C₁₃H₁₆NO₃ [M+H]⁺ calcd. 234.1125, found 234.1119.

3,3-diethyl-1-methyl-2-oxindoline-5-carbonitrile (2q)



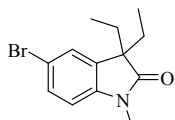
Light yellow solid; 88% yield; mp 60–62°C; **¹H NMR (400 MHz, CDCl₃)** δ (ppm) = 0.57 (t, *J* = 7.5 Hz, 6H), 1.75–1.85 (m, 2H), 1.91–2.00 (m, 2H), 3.25 (s, 3H), 6.92 (d, *J* = 8.1 Hz, 1H), 7.40 (d, *J* = 1.4 Hz, 1H), 7.62 (dd, *J* = 8.1, 1.4 Hz, 1H); **¹³C NMR (100 MHz, CDCl₃)** δ (ppm) = 8.5, 26.2, 30.5, 54.3, 105.5, 108.1, 119.4, 126.0, 133.1, 133.2, 148.2, 179.7; IR (KBr, cm⁻¹): 3416, 2963, 2928, 2878, 2218, 1719, 1612, 1495, 1460, 1359, 1262, 1122, 1069, 940, 836, 539, 513. HRMS (ESI) for C₁₄H₁₇N₂O [M+H]⁺ calcd. 229.1335, found 229.1331.

5'-chloro-1'-methylspiro[cyclopentane-1,3'-indolin]-2'-one (2r)



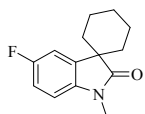
White solid; 82% yield; mp 70–71 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 1.80–1.85 (m, 2H), 1.93–1.99 (m, 2H), 2.04–2.18 (m, 4H), 3.19 (s, 3H), 6.73 (d, $J = 8.2$ Hz, 1H), 7.16 (d, $J = 2.1$ Hz, 1H), 7.21 (dd, $J = 8.2, 2.1$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 26.3, 26.6, 38.3, 54.1, 108.5, 122.8, 127.1, 127.7, 138.5, 141.4, 181.4; IR (KBr, cm^{-1}): 3405, 2947, 2872, 1705, 1610, 1490, 1469, 1429, 1365, 1346, 1274, 1246, 1039, 1067, 971, 879, 844, 806, 545, 460. HRMS (ESI) for $\text{C}_{13}\text{H}_{15}\text{ClNO}$ $[\text{M}+\text{H}]^+$ calcd. 236.0837, found 236.0832.

5-bromo-3,3-diethyl-1-methylindolin-2-one (2s)



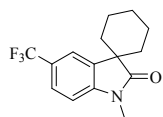
White solid; 86% yield; mp 74–75 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 0.57 (t, $J = 7.4$ Hz, 6H), 1.71–1.80 (m, 2H), 1.88–1.97 (m, 2H), 3.20 (s, 3H), 6.72 (d, $J = 8.2$ Hz, 1H), 7.25 (d, $J = 1.9$ Hz, 1H), 7.39 (dd, $J = 8.2, 1.9$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 8.6, 26.0, 30.6, 54.6, 109.3, 115.1, 125.9, 130.4, 134.2, 143.4, 179.4; IR (KBr, cm^{-1}): 3401, 3057, 3042, 2960, 2932, 2874, 1706, 1604, 1481, 1457, 1429, 1365, 1331, 1269, 1253, 1125, 1084, 905, 806, 774, 540, 489. HRMS (ESI) for $\text{C}_{13}\text{H}_{17}\text{BrNO}$ $[\text{M}+\text{H}]^+$ calcd. 282.0488, found 282.0482.

5'-fluoro-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2t)



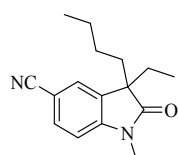
Light yellow oil; 80% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 1.51–1.57 (m, 2H), 1.61–1.75 (m, 4H), 1.82–1.88 (m, 2H), 1.92–1.98 (m, 2H), 3.19 (s, 3H), 6.75 (dd, $J = 8.5, 4.3$ Hz, 1H), 6.95–7.00 (m, 1H), 7.20 (dd, $J = 8.5, 2.5$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 21.1, 25.0, 26.2, 32.8, 47.9 (d, $J = 1.3$ Hz), 108.1 (d, $J = 8.1$ Hz), 112.1 (d, $J = 24.8$ Hz), 113.4 (d, $J = 23.2$ Hz), 136.9 (d, $J = 7.6$ Hz), 138.7 (d, $J = 1.4$ Hz), 158.8 (d, $J = 238$ Hz), 180.3; IR (KBr, cm^{-1}): 3403, 3060, 2933, 2856, 1709, 1620, 1495, 1470, 1446, 1352, 1276, 1137, 1097, 1006, 932, 869, 809, 760, 702, 643, 560, 483. HRMS (ESI) for $\text{C}_{14}\text{H}_{17}\text{FNO}$ $[\text{M}+\text{H}]^+$ calcd. 234.1289, found 234.1282.

1'-methyl-5'-(trifluoromethyl)spiro[cyclohexane-1,3'-indolin]-2'-one (2u)



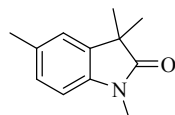
White solid; 92% yield; mp 37–39°C; **¹H NMR (400 MHz, CDCl₃)** δ (ppm) = 1.56–1.62 (m, 2H), 1.67–1.75 (m, 4H), 1.82–1.88 (m, 2H), 1.97–2.01 (m, 2H), 3.23(s, 3H), 6.91 (d, *J* = 8.2 Hz, 1H), 7.56 (dd, *J* = 8.2, 0.6 Hz, 1H), 7.64(s, 1H); **¹³C NMR (100 MHz, CDCl₃)** δ (ppm) = 21.0, 25.0, 26.3, 32.9, 47.3, 107.5, 120.5 (q, *J* = 3.6 Hz), 124.1 (q, *J* = 32.1 Hz), 124.5 (q, *J* = 270 Hz), 125.2 (q, *J* = 4.1 Hz), 135.8, 145.8, 180.5; IR (KBr, cm⁻¹): 3424, 3083, 2972, 2930, 1721, 1624, 1503, 1467, 1384, 1327, 1297, 1261, 1116, 1076, 1054, 890, 835, 707, 653, 562, 536, 474. HRMS (ESI) for C₁₂H₁₃F₃NO [M+H]⁺calcd. 244.0944, found 244.0940.

3-butyl-3-ethyl-1-methyl-2-oxoindoline-5-carbonitrile (2v)



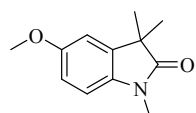
Light yellow oil; 86% yield; **¹H NMR (400 MHz, CDCl₃)** δ (ppm) = 0.55 (t, *J* = 7.4 Hz, 3H), 0.72–0.84 (m, 4H), 0.89–0.97 (m, 1H), 1.14–1.21 (m, 2H), 1.70–1.82 (m, 2H), 1.84–1.94 (m, 2H), 3.21 (s, 3H), 6.83 (d, *J* = 7.8 Hz, 1H), 7.05–7.09 (m, 1H), 7.12–7.14 (m, 1H), 7.24–7.28 (m, 1H); **¹³C NMR (100 MHz, CDCl₃)** δ (ppm) = 8.5, 13.8, 22.9, 25.9, 26.4, 31.0, 37.5, 53.7, 107.6, 122.3, 122.6, 127.5, 132.4, 144.2, 180.2; IR (KBr, cm⁻¹): 3513, 3432, 2962, 2934, 2875, 2223, 1721, 1614, 1592, 1493, 1460, 1371, 1348, 1254, 1173, 1123, 1068, 822, 737, 569, 521, 508. HRMS (ESI) for C₁₆H₂₀NO [M+Na]⁺calcd. 279.1468, found 279.1462.

1,3,3,5-tetramethylindolin-2-one (2w)



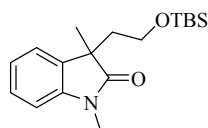
White solid; 70% yield; mp 54–56°C; **¹H NMR (400 MHz, CDCl₃)** δ (ppm) = 1.35 (s, 6H), 2.34 (s, 3H), 3.19 (s, 3H), 6.73 (d, *J* = 7.8 Hz, 1H), 7.02–7.06 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ (ppm) = 21.1, 24.4, 26.2, 44.2, 107.7, 123.1, 127.8, 132.0, 135.9, 140.3, 181.3; IR (KBr, cm⁻¹): 3400, 2961, 2925, 2863, 1710, 1620, 1603, 1505, 1458, 1380, 1349, 1348, 1244, 1133, 1117, 1066, 1045, 882, 800, 617, 555, 465. HRMS (ESI) for C₁₂H₁₆NO [M+H]⁺calcd. 190.1226, found 190.1223.

5-methoxy-1,3,3-trimethylindolin-2-one (2x)



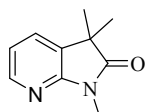
Colorless oil; 77% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 1.36 (s, 6H), 3.19 (s, 3H), 3.81 (s, 3H), 6.73–6.80 (m, 2H), 6.83 (d, J = 2.3 Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 24.4, 26.2, 44.6, 55.8, 108.2, 110.0, 111.5, 136.1, 137.2, 156.0, 181.0; IR (KBr, cm^{-1}): 3487, 2966, 2929, 2867, 1708, 1662, 1602, 1500, 1472, 1436, 1383, 1356, 1289, 1220, 1120, 1048, 1028, 882, 805, 697, 558, 463. HRMS (ESI) for $\text{C}_{12}\text{H}_{16}\text{NO}_2$ $[\text{M}+\text{H}]^+$ calcd. 206.1176, found 206.1171.

3-((tert-butyldimethylsilyloxy)ethyl)-1,3-dimethylindolin-2-one (2y)



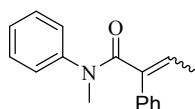
Colorless oil; 64% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm)= -0.13 (s, 6H), 0.77 (s, 9H), 1.36(s, 3H), 1.94–2.00(m, 1H), 2.20–2.27 (m, 1H), 3.19 (s, 3H), 3.36 (t, J = 6.7 Hz, 2H), 6.82 (d, J = 7.8 Hz, 1H), 7.04 (t, J = 7.5 Hz, 1H), 7.17 (d, J = 7.4 Hz, 1H), 7.25 (t, J = 7.7 Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm)= -5.7, -5.6, 18.1, 24.8, 25.8, 26.2, 40.3, 46.5, 59.5, 107.8, 122.2, 122.7, 127.6, 133.7, 143.3, 180.4; IR (KBr, cm^{-1}): 3426, 2954, 2928, 2856, 1719, 1614, 1494, 1471, 1377, 1348, 1253, 1110, 835, 752. HRMS (ESI) for $\text{C}_{18}\text{H}_{30}\text{NO}_2\text{Si}$ $[\text{M}+\text{H}]^+$ calcd. 320.2040, found 320.2033.

1,3,3-trimethyl-1,3-dihydro-2H-pyrrolo[2,3-b]pyridin-2-one (2z)



Yellow oil; 65% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 1.40 (s, 6H), 3.31 (s, 3H), 6.96 (dd, J = 7.2, 5.3 Hz, 1H), 7.43 (dd, J = 7.2, 1.5 Hz, 1H), 8.18 (dd, J = 5.3, 1.5 Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 23.8, 25.3, 118.0, 129.5, 130.0, 146.5, 156.2, 180.8; IR (KBr, cm^{-1}): 3425, 2971, 2932, 2870, 1720, 1608, 1595, 1471, 1360, 1258, 1143, 1046, 939, 803, 782, 559, 497. HRMS (ESI) for $\text{C}_{10}\text{H}_{13}\text{N}_2\text{O}$ $[\text{M}+\text{H}]^+$ calcd. 177.1022, found 177.1025.

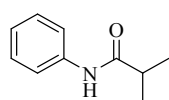
(E)-N-methyl-N,2-diphenylbut-2-enamide (2aa)



Colorless oil; 20% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 1.63 (d, J = 7.1 Hz, 3H), 3.29 (s, 3H), 6.25 (q, J = 7.1 Hz, 1H),

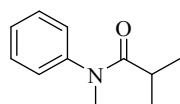
6.80–6.84 (m, 4H), 7.10–7.12 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 14.4, 37.9, 126.4, 126.7, 127.3, 127.7, 128.7, 128.7, 131.6, 135.7, 139.3, 144.1, 171.8; IR (KBr, cm^{-1}): 3390, 3057, 2963, 2932, 1720, 1650, 1595, 1495, 1455, 1382, 1367, 1267, 1134, 1075, 766, 737, 700, 553. HRMS (ESI) for $\text{C}_{17}\text{H}_{18}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 252.1383, found 252.1386

N-phenylisobutyramide (2ab)



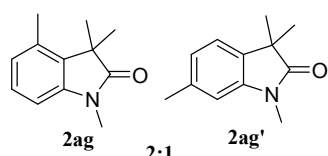
White solid; 95% yield; mp 98–99°C; ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 1.21 (d, J = 1.4 Hz, 3H), 1.23 (d, J = 1.4 Hz, 3H), 2.52 (m, 1H), 7.08 (t, J = 7.3 Hz, 1H), 7.28 (t, J = 7.2 Hz, 2H), 7.54 (d, J = 8.0 Hz, 2H), 7.58–7.80 (br, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 19.6, 36.5, 119.9, 124.0, 128.8, 138.1, 175.6; IR (KBr, cm^{-1}): 3302, 3263, 2969, 2931, 1662, 1600, 1549, 1490, 1442, 1386, 1310, 1252, 1204, 1099, 942, 883, 761, 734, 694, 589, 510. HRMS (ESI) for $\text{C}_{10}\text{H}_{14}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 164.1070, found 164.1067.

N-methyl-N-phenylisobutyramide (2a')



White solid; 60% yield; mp 98–99°C; ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 1.03 (d, J = 6.7 Hz, 6H), 2.51 (m, 1H), 3.25 (s, 3H), 7.19 (dd, J = 8.3, 1.4 Hz, 2H), 7.35 (t, J = 7.4 Hz, 1H), 7.43 (t, J = 7.8 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 19.6, 30.9, 37.4, 127.2, 127.6, 129.7, 144.3, 177.4; IR (KBr, cm^{-1}): 3442, 3054, 2962, 2933, 2870, 1652, 1595, 1496, 1471, 1420, 1387, 1268, 1117, 1039, 783, 708, 572, 411. HRMS (ESI) for $\text{C}_{11}\text{H}_{16}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 178.1226, found 178.1222.

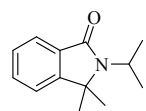
1,3,3,4-tetramethylindolin-2-one (2ag); 1,3,3,6-tetramethylindolin-2-one (2ag')



White solid; 71% yield; ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 1.35 (s, 3H), 1.45 (s, 6H), 2.38 (s, 1.5H), 2.40 (s, 3H), 3.20 (d, 4.7H), 6.67 (s, 0.5H), 6.69 (d, J = 7.8 Hz,

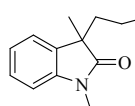
1H), 6.83 (d, $J = 7.8$ Hz, 1H), 6.87 (dd, $J = 7.5, 0.6$ Hz, 0.5H), 7.08 (d, $J = 7.5$ Hz, 0.5H), 7.16 (t, $J = 7.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 18.1, 21.7, 22.4, 24.5, 26.1, 26.3, 43.9, 45.0, 105.8, 109.0, 122.0, 122.9, 125.0, 127.4, 132.6, 133.0, 134.0, 137.7, 142.7, 142.9, 181.4, 181.7; IR (KBr, cm^{-1}): 3396, 2979, 2965, 2928, 1712, 1608, 1598, 1468, 1442, 1381, 1331, 1300, 1243, 1073, 1047, 954, 826, 786, 760, 600, 560, 474. HRMS (ESI) for $\text{C}_{12}\text{H}_{16}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 190.1226, found 190.1222.

2-isopropyl-3,3-dimethylisoindolin-1-one (1af)



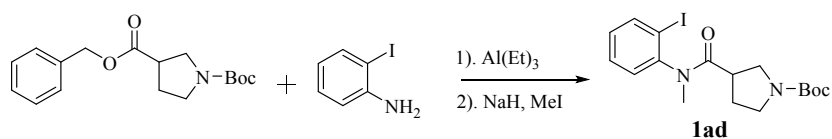
White solid; 53% yield; ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 1.28 (s, 6H), 1.56 (s, $J = 6.9$ Hz, 6H), 3.65 (m, 1H), 7.34 (d, $J = 7.6$ Hz, 1H), 7.40 (t, $J = 7.4$ Hz, 1H), 7.50 (t, $J = 7.5$ Hz, 1H), 7.77 (d, $J = 7.5$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 20.5, 25.4, 44.5, 63.2, 120.5, 123.1, 127.8, 131.2, 132.0, 151.1, 167.2; IR (KBr, cm^{-1}): 3424, 2970, 2932, 2874, 1647, 1578, 1471, 1428, 1389, 1280, 1128, 1051, 1019, 769, 727. HRMS (ESI) for $\text{C}_{13}\text{H}_{17}\text{NONa}$ $[\text{M}+\text{Na}]^+$ calcd. 226.1202, found 226.1202.

3-(2-hydroxyethyl)-1,3-dimethylindolin-2-one (2ae)



Colorless oil; 88% yield; ^1H NMR (400 MHz, CDCl_3): δ (ppm)= 1.41 (s, 3H), 1.96–2.02 (m, 1H), 2.13–2.19 (m, 1H), 2.62 (br, 1H), 3.22 (s, 3H), 3.41–3.47 (m, 1H), 3.61–3.67 (m, 1H), 6.87 (d, $J = 7.8$ Hz, 1H), 7.09 (t, $J = 7.4$ Hz, 1H), 7.18 (d, $J = 7.0$ Hz, 1H), 7.29 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 23.5, 26.3, 40.0, 46.9, 59.2, 108.3, 122.4, 122.7, 127.9, 134.0, 142.8, 181.5; IR (KBr, cm^{-1}): 3417, 3055, 2968, 2925, 1703, 1613, 1493, 1471, 1453, 1379, 1352, 1265, 1126, 1105, 1043, 737, 701. HRMS (ESI) for $\text{C}_{12}\text{H}_{15}\text{NO}_2\text{Na}$ $[\text{M}+\text{Na}]^+$ calcd. 228.0995, found 228.0995.

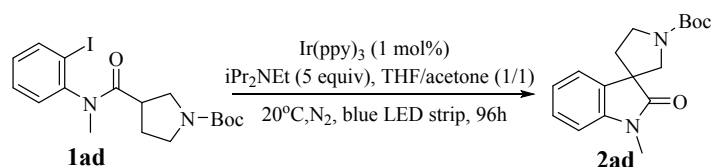
7. Synthesis of the (\pm)-coerulescine scaffold



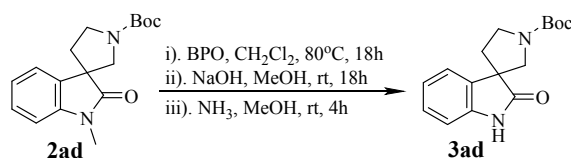
A solution of triethylaluminum in toluene (1.0 M, 2.5 ml, 2.5 mmol, 2.5 equiv.) was added to a solution of 2-iodoaniline (0.219 g, 1.0 mmol, 1 equiv.) in toluene (3 ml) at 0°C under inert atmosphere. The mixture was then warmed to room temperature. After stirring for 3h, a solution of benzyl 1-tert-{butyloxycarbonyl}-pyrrolidine-3-carboxylate (0.305 g, 1.0 mmol, 1 equiv.) in toluene (3 ml) was added, and the mixture was heated at 80°C for 12 h. On completion of the reaction, a saturated aqueous solution of ammonium chloride and a few drops of hydrochloric acid (2M) were added. The mixture was washed with a solution of sodium hydroxide (2M), and extracted with diethyl ether, and the combined organic phase was dried over sodium sulfate. The product was purified by column chromatography (70:30, hexanes-ethyl acetate) giving tert-butyl-3-((2-iodophenyl)carbamoyl)pyrrolidine-1-carboxylate as a white powder.^{2,4}

To a stirred suspension of NaH (0.027 g; 1.1 mmol) in 1 ml of dry THF at 0°C , the tert-butyl-3-((2-iodophenyl)carbamoyl)pyrrolidine-1-carboxylate (1 mmol) dissolved in 2 ml of THF was added dropwise within 10 min. The reaction mixture was stirred until the solution became clear (30 min, hydrogen gas evolved), and the solution of MeI (0.22 g; 1.5 mmol) in 1 ml of THF was added dropwise within 10 min. The solution was warmed up to room temperature and stirred for 3 h. Then the reaction mixture was quenched with water (5 ml). The resulting solution was extracted with ethyl acetate (3 x 5 ml). Combined organic layers were washed with brine (1 x 4 ml) and dried over Na_2SO_4 . Ethyl acetate was removed under reduced pressure to give crude products. The product was purified by column chromatography (7:3, hexanes-ethyl acetate) giving tert-butyl-3-((2-iodophenyl)(methyl)carbamoyl)pyrrolidine-1-carboxylate **1ad** as a pale oil (0.38 g, 88% over 2 steps); $^1\text{H NMR}$ (400

MHz, CDCl₃): δ (ppm)= 1.42 (2 x s, 9H), 1.95–2.02 (m, 1H), 2.09 (br, 1H), 2.62–2.70 (m, 1H), 3.05–3.14 (m, 1H), 3.19 (s, 3H), 3.31–3.36 (m, 1H), 3.51–3.62 (m, 2H), 7.11–7.55 (m, 1H), 7.26–7.30 (m, 1H), 7.46 (t, $J = 7.6$ Hz, 1H), 7.96 (d, $J = 8.0$ Hz, 1H); **¹³C NMR (100 MHz, CDCl₃)** δ (ppm)= 28.4, 36.2, 36.2, 45.4, 45.5, 49.1, 79.1, 79.1, 99.7, 99.8, 128.8, 128.8, 129.0, 129.8, 130.0, 130.1, 130.1, 140.2, 140.3, 145.5, 154.1, 154.1, 172.2, 172.3; IR (KBr, cm⁻¹): 3315, 2974, 2931, 2884, 2372, 1688, 1663, 1578, 1470, 1409, 1386, 1250, 1169, 1125, 1019, 875, 771, 728, 552. HRMS (ESI) for C₁₇H₂₃N₂O₃INa [M+Na]⁺calcd. 453.0646, found 453.0657.



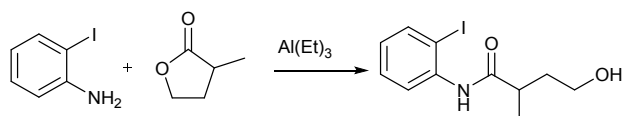
The title compound **2ad** was prepared according to the general method described above and purified by flash column chromatography (2:1, hexanes-ethyl acetate) in 65% yield. **¹H NMR (400 MHz, CDCl₃)**: δ (ppm)= 1.48 (2 x s, 9H), 2.03 (br, 1H), 2.38–2.45 (m, 1H), 3.24 (s, 3H), 3.52–3.86 (m, 4H), 6.88 (d, $J = 7.7$ Hz, 1H), 7.08 (t, $J = 7.4$ Hz, 1H), 7.4 (br, 1H), 7.32 (t, $J = 7.6$ Hz, 1H); **¹³C NMR (100 MHz, CDCl₃)** δ (ppm)= 26.4, 28.4, 35.4, 36.2, 45.2, 45.4, 51.9, 52.8, 53.8, 54.3, 79.7, 108.2, 122.3, 122.9, 128.3, 142.7, 154.3, 177.2; IR (KBr, cm⁻¹): 3414, 3056, 2975, 2934, 2885, 1717, 1696, 1614, 1494, 1472, 1402, 1375, 1350, 1254, 1171, 1124, 1022, 988, 882, 752, 544, 463. HRMS (ESI) for C₁₇H₂₂N₂O₃Na [M+Na]⁺calcd. 325.1523, found 325.1531.



A solution of **2ad** (60.4 mg, 0.2 mmol) and benzoyl peroxide (96.8 mg, 0.4 mmol) in CH₂Cl₂ (1 mL) in a sealed tube was heated slowly to 80 °C. After stirring for 18 h, the reaction mixture was cooled to rt and the solvent was evaporated. The residue was dissolved in MeOH (1 mL), NaOH (29.2 mg, 0.8 mmol) was added and the reaction

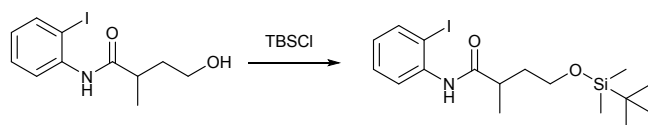
mixture was stirred for 18 h at rt. Then the slurry was poured onto saturated aqueous NH_4Cl (1 mL) and extracted with CH_2Cl_2 (3 x 2 mL). The combined organic layers were dried (MgSO_4) and concentrated. The residue was dissolved in a saturated methanolic NH_3 solution (0.5 mL) and stirred for 4 h at rt. The solvent was evaporated and the residue chromatographed to give **3ad** as a pale oil (25 mg, 43%);³ $^1\text{H NMR}$ (400 MHz, CDCl_3): $\delta(\text{ppm}) = 1.49$ (2 x s, 9H), 2.04–2.14 (m, 1H), 2.39–2.46 (m, 1H), 3.55–3.91 (m, 4H), 6.97 (t, $J = 6.3$ Hz, 1H), 7.06 (t, $J = 7.3$ Hz, 1H), 7.16–7.26 (m, 2H), 9.19 (2 x s, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta(\text{ppm}) = 28.4, 28.5, 35.5, 36.3, 45.2, 45.4, 52.4, 53.3, 53.8, 54.3, 79.8, 110.1, 122.7, 122.9, 128.4, 132.6, 133.0, 140.1, 140.2, 154.4, 180.1, 180.4$; IR (KBr, cm^{-1}): 3248, 2977, 2932, 2887, 1719, 1700, 1621, 1472, 1404, 1367, 1345, 1228, 1172, 1130, 879, 751, 737, 702, 674, 636. HRMS (ESI) for $\text{C}_{16}\text{H}_{20}\text{N}_2\text{O}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ calcd. 311.1366, found 311.1357.

8. Synthesis of the (\pm) - physovenine scaffold

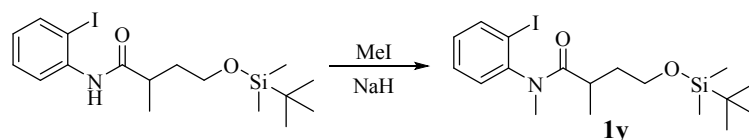


A solution of triethylaluminum in toluene (1.0 M, 10 ml, 10 mmol, 2.5 equiv.) was added to a solution of 2-iodoaniline (0.876 g, 4.0 mmol, 1 equiv.) in toluene (15 ml) at 0°C under inert atmosphere. The mixture was then warmed to room temperature. After stirring for 3h, a solution of 3-methyldihydrofuran-2(3H)-one (0.40 g, 4.0 mmol, 1 equiv.) in toluene (15 ml) was added, and the mixture was heated at 80°C for 12 h. On completion of the reaction, a saturated aqueous solution of ammonium chloride and a few drops of hydrochloric acid (2M) were added. The mixture was washed with a solution of sodium hydroxide (2M), and extracted with diethyl ether, and the combined organic phase was dried over sodium sulfate and concentrated. The product was purified by column chromatography (1:1,

dichloromethane-ethyl acetate) giving the product as a white powder (1.13 g, 89%).^{2,4}

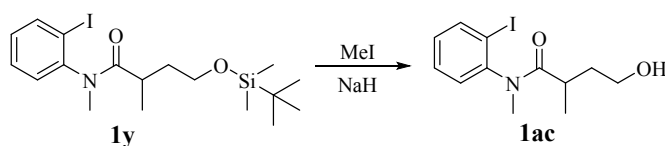


To a reaction flask containing the 4-hydroxy-N-(2-iodophenyl)-2-methylbutanamide (0.64 g, 2 mmol) and imidazole (0.2 g, 3 mmol) was added CH₂Cl₂ (3 mL). The solution was cooled to 0°C in an ice bath before adding TBSCl (0.34 g, 2.2 mmol). The reaction was allowed to warm to room temperature overnight (12 h) and a white precipitate emerged. The reaction was diluted with CH₂Cl₂ (3 mL) and quenched with saturated NH₄Cl solution. The aqueous layer was extracted with CH₂Cl₂, and the combined organic layers were washed with saturated NaHCO₃ solution, water, and brine; dried over sodium sulfate; and concentrated. The resulting crude mixture was purified by flash column chromatography (4:1, hexanes-ethyl acetate) giving the product as a pale oil (0.85 g, 95%).⁵ **¹H NMR (400 MHz, CDCl₃):** δ(ppm)= 0.04 (2 x s, 6H), 0.88 (s, 9H), 1.28 (d, *J* = 6.9 Hz, 3H), 1.64–1.72 (m, 1H), 1.95–2.03 (m, 1H), 2.65–2.73 (m, 1H), 3.70 (t, *J* = 6.0 Hz, 2H), 6.81 (m, 1H), 7.31 (m, 1H), 7.59 (br, 1H), 7.75 (dd, *J* = 8.0, 1.4 Hz, 1H), 8.22 (d, *J* = 8.1 Hz, 1H); **¹³C NMR (100 MHz, CDCl₃):** δ(ppm)= -5.4, -5.3, 17.7, 18.2, 25.9, 37.0, 38.7, 60.4, 90.0, 122.0, 125.7, 129.1, 138.2, 138.6, 174.6; IR (KBr, cm⁻¹): 3388, 3278, 2955, 2930, 2856, 1671, 1584, 1516, 1462, 1431, 1387, 1288, 1255, 1176, 1102, 1014, 903, 835, 776, 749, 665, 649. HRMS (ESI) for C₁₇H₂₈NO₂INa [M+Na]⁺calcd. 456.0826, found 456.0820.

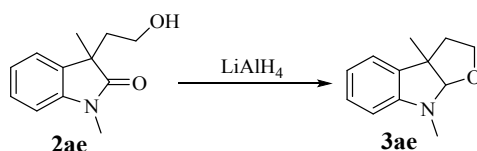


The title compound **1y** was prepared according to the general method described above and purified by flash column chromatography (6:1, hexanes-ethyl acetate) in 98% yield. Colorless oil; **¹H NMR (400 MHz, CDCl₃):** δ(ppm)= -0.06– -0.03 (m, 6H), 0.78 (2 x s, 9H), 1.06 (4 x s, 3H), 1.44–1.58 (m, 1H), 1.79–1.05 (m, 1H), 2.25–2.40 (m, 1H), 3.17 (2 x s, 3H), 3.52–3.58 (m, 2H), 7.04–7.09 (m, 1H), 7.26–7.32 (m, 1H),

7.38–7.44 (m, 1H), 7.91–7.94 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) $\delta(\text{ppm}) = -5.5, -5.4, -5.3, 17.1, 17.2, 18.1, 18.2, 25.9, 33.4, 33.5, 36.1, 36.2, 36.3, 37.0, 60.4, 60.6, 99.5, 99.8, 128.8, 129.4, 129.6, 129.6, 129.7, 140.1, 140.3, 146.1, 176.5, 176.7$; IR (KBr, cm^{-1}): 3413, 2930, 2857, 2372, 1647, 1578, 1468, 1429, 1388, 1280, 1252, 1112, 1051, 1020, 878, 836, 772, 726, 666, 575. HRMS (ESI) for $\text{C}_{18}\text{H}_{30}\text{NO}_2\text{INa}$ $[\text{M}+\text{Na}]^+$ calcd. 470.0983, found 470.0999.



To a solution of the silyl ether (447 mg, 1 mmol) in THF (5 mL) at 0°C was added TBAF (1.0 M in THF, 1.2 mL, 1.2 mmol). The reaction mixture was stirred at room temperature for 4 h and quenched by saturated NH_4Cl solution. The aqueous layer was extracted with EtOAc, and the combined organic layers were washed with water and brine, dried, and concentrated. The residue was purified by flash chromatography (EtOAc). The alcohol as a colorless oil which solidified upon standing (318 mg, 96%).



To a solution of the oxindole-3-ethanol (41 mg, 0.2 mmol) in dry THF (4 mL) at 0°C was added LAH (31 mg, 0.8 mmol). The reaction mixture was stirred at this temperature for 40 min and quenched by saturated NaHCO_3 (5 mL) solution. The aqueous layer was extracted with EtOAc, and the combined organic layers were washed with brine, dried, and concentrated. The residue was purified by flash chromatography (EtOAc) gave **3ae** (15.1 mg, 80%) as a slightly white solid:⁶ ^1H NMR (400 MHz, CDCl_3): $\delta(\text{ppm}) = 1.46$ (s, 3H), 2.00–2.15 (m, 2H), 2.92 (s, 3H), 3.42–3.48 (m, 1H), 3.92–3.96 (m, 1H), 5.06 (s, 1H), 6.36 (d, $J = 7.8$ Hz, 1H), 6.67 (m, 1H), 7.04 (d, $J = 6.9$ Hz, 1H), 7.07–7.12 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) $\delta(\text{ppm}) = 24.7, 30.9, 41.7, 52.3, 67.3, 104.8, 104.9, 117.3, 122.4, 128.1, 134.4,$

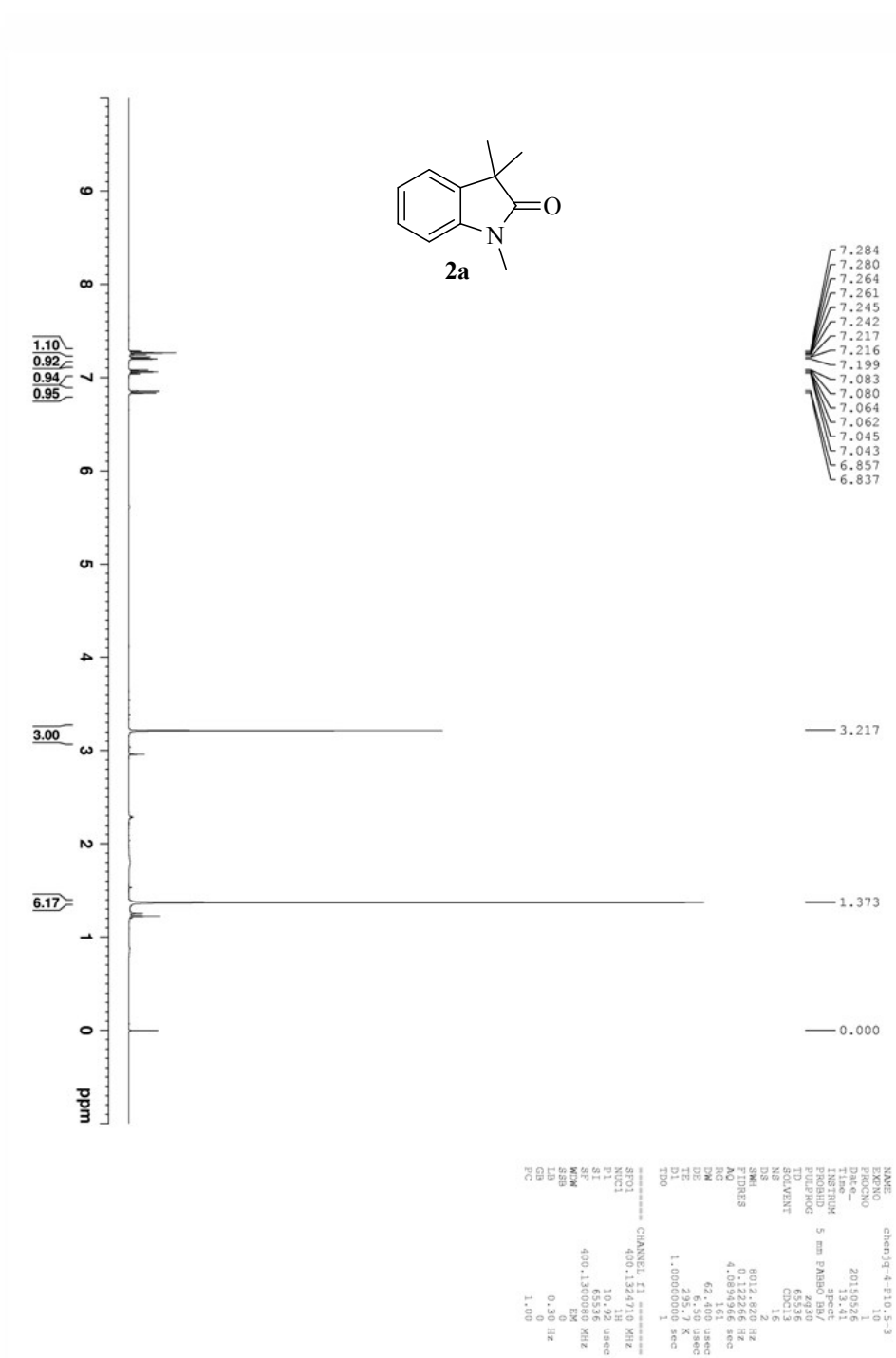
150.4; IR (KBr, cm^{-1}): 2959, 2926, 2866, 1608, 1494, 1448, 1388, 1301, 1216, 124, 1033, 1011, 918, 740. HRMS (ESI) for $\text{C}_{12}\text{H}_{15}\text{NONa}$ $[\text{M}+\text{Na}]^+$ calcd. 212.1046, found 212.1041.

References:

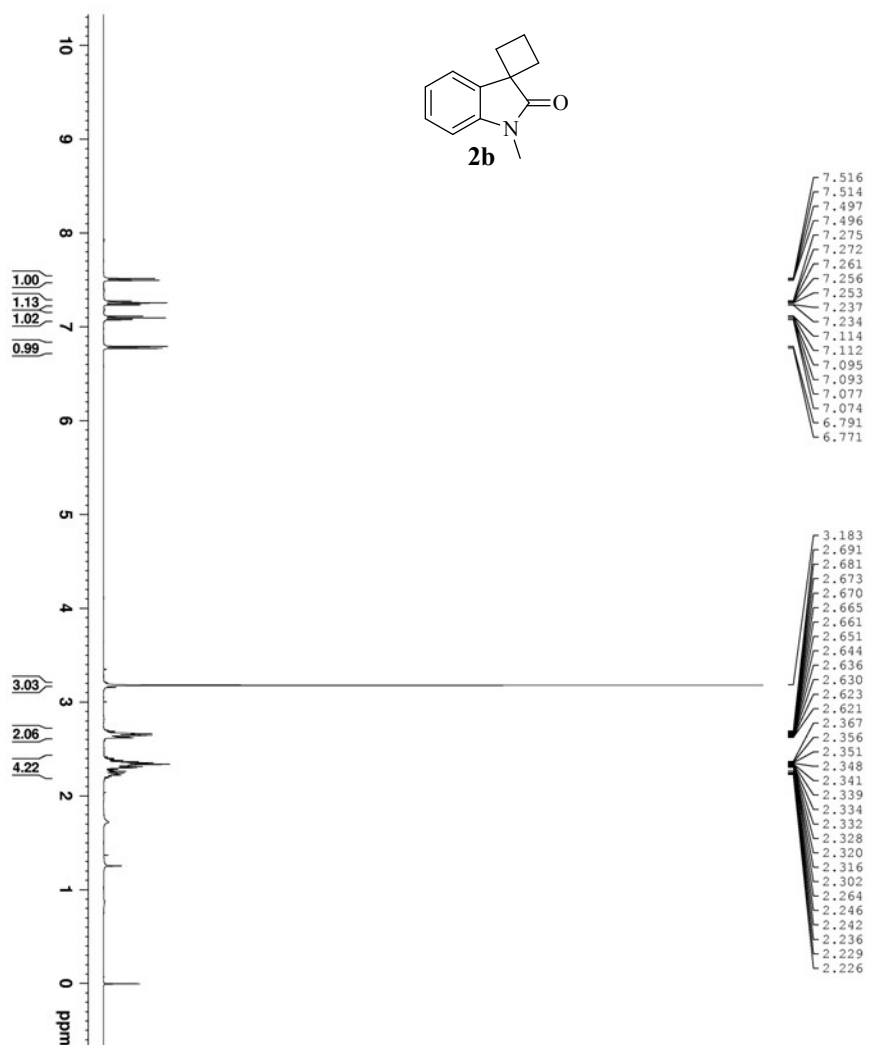
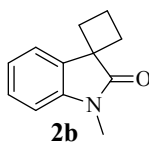
- 1 U. Ladziata, A. Y. Koposov, K. Y. Lo, J. Willging, V. N. Nemykin and V. V. Zhdankin, *Angew. Chem. Int. Ed.*, 2005, **44**, 7127.
- 2 J. A. Murphy, R. Tripoli, T. A. Khan, and Umesh W. Mali, *Org. Lett.*, 2005, **7**, 3287.
- 3 W. G. B. van Henegouwen, R. M. Fieseler, F. P. J. T. Rutjes, and H. Hiemstra, *J. Org. Chem.*, 2000, **65**, 8317.
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- 5 K. Kong, J. A. Enquist, J. M. E. McCallum, G. M. Smith, T. Matsumaru, E. Menhaji-Klotz, and J. L. Wood, *J. Am. Chem. Soc.*, 2013, **135**, 10890.
- 6 B. Zhou, W. Hou, Y. Yang, H. Feng, and Y. Li, *Org. Lett.*, 2014, **16**, 1322.

9. NMR spectra of compounds

1,3,3-trimethylindolin-2-one (2a)

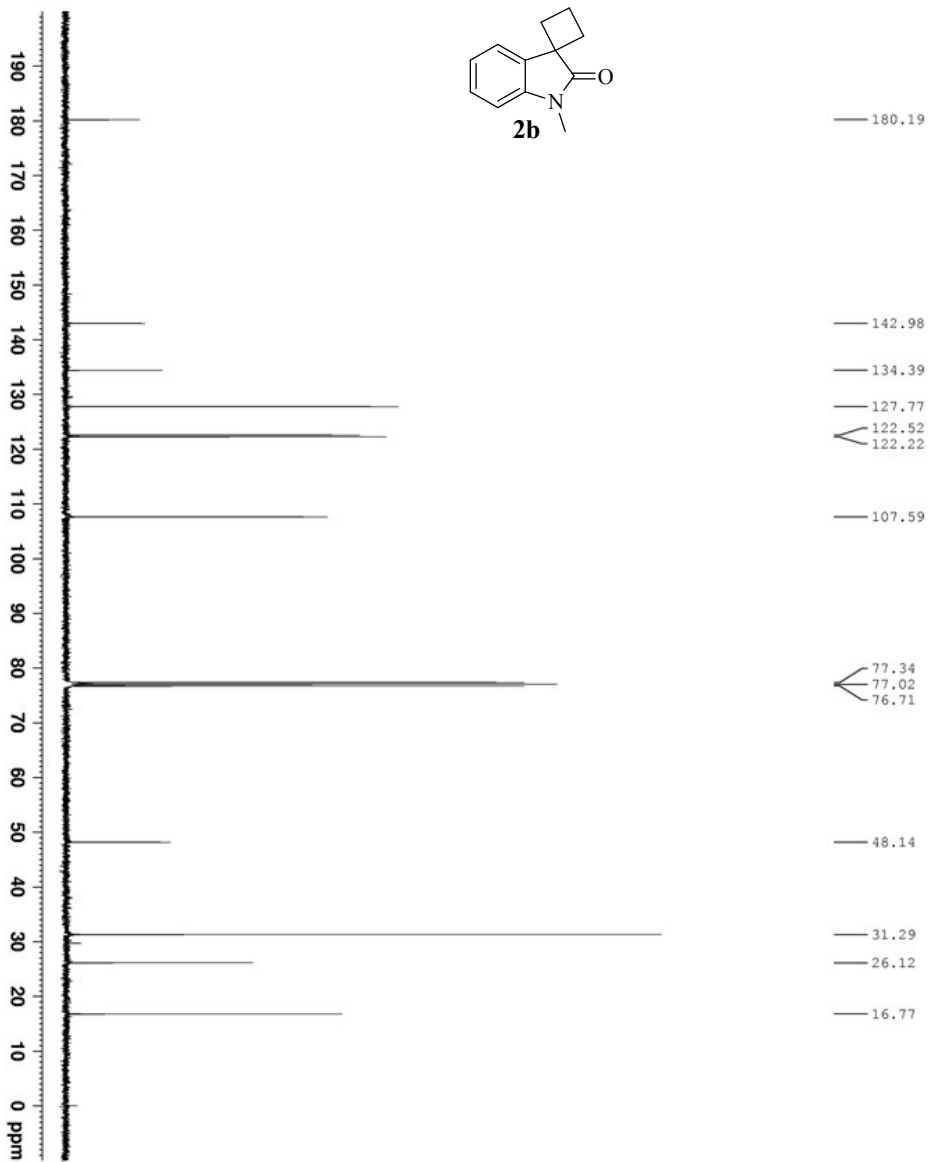
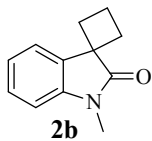


1'-methylspiro[cyclobutane-1,3'-indolin]-2'-one (**2b**)



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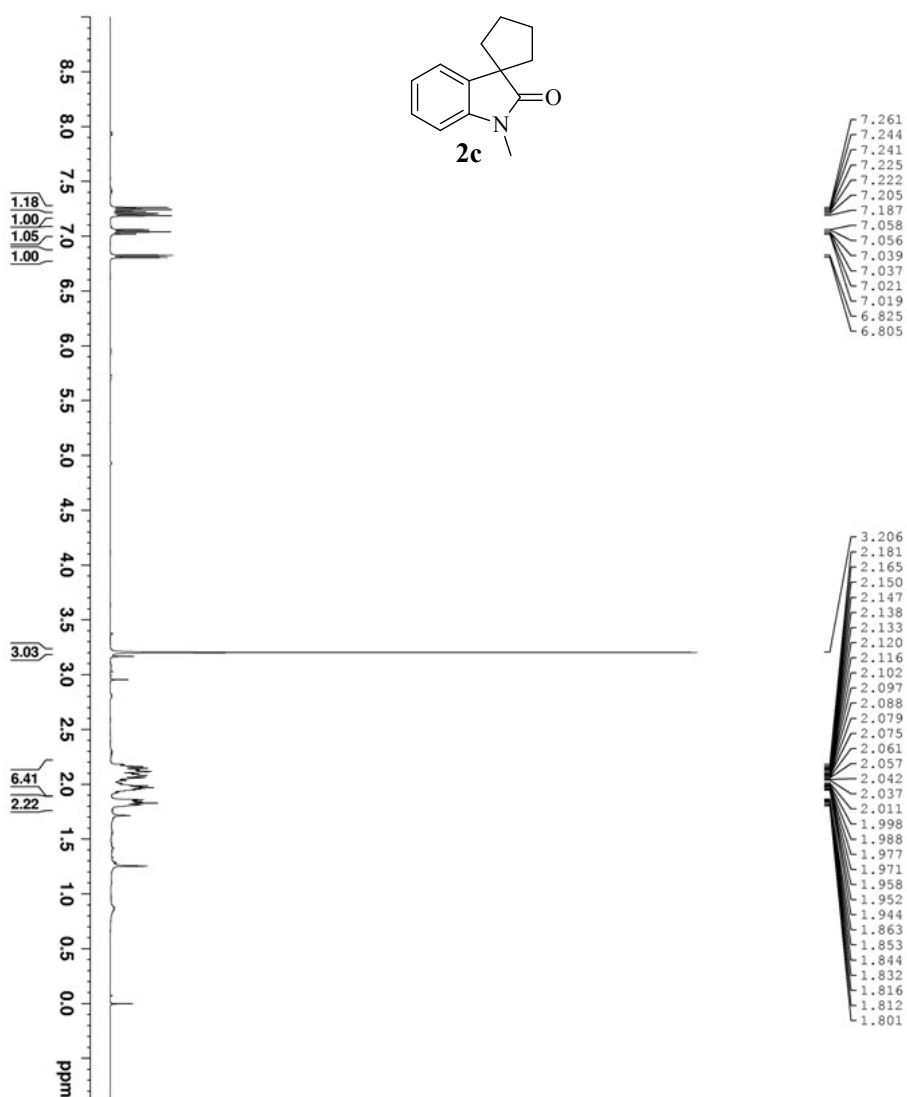
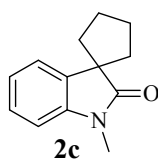
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1'-methylspiro[cyclopentane-1,3'-indolin]-2'-one (**2c**)



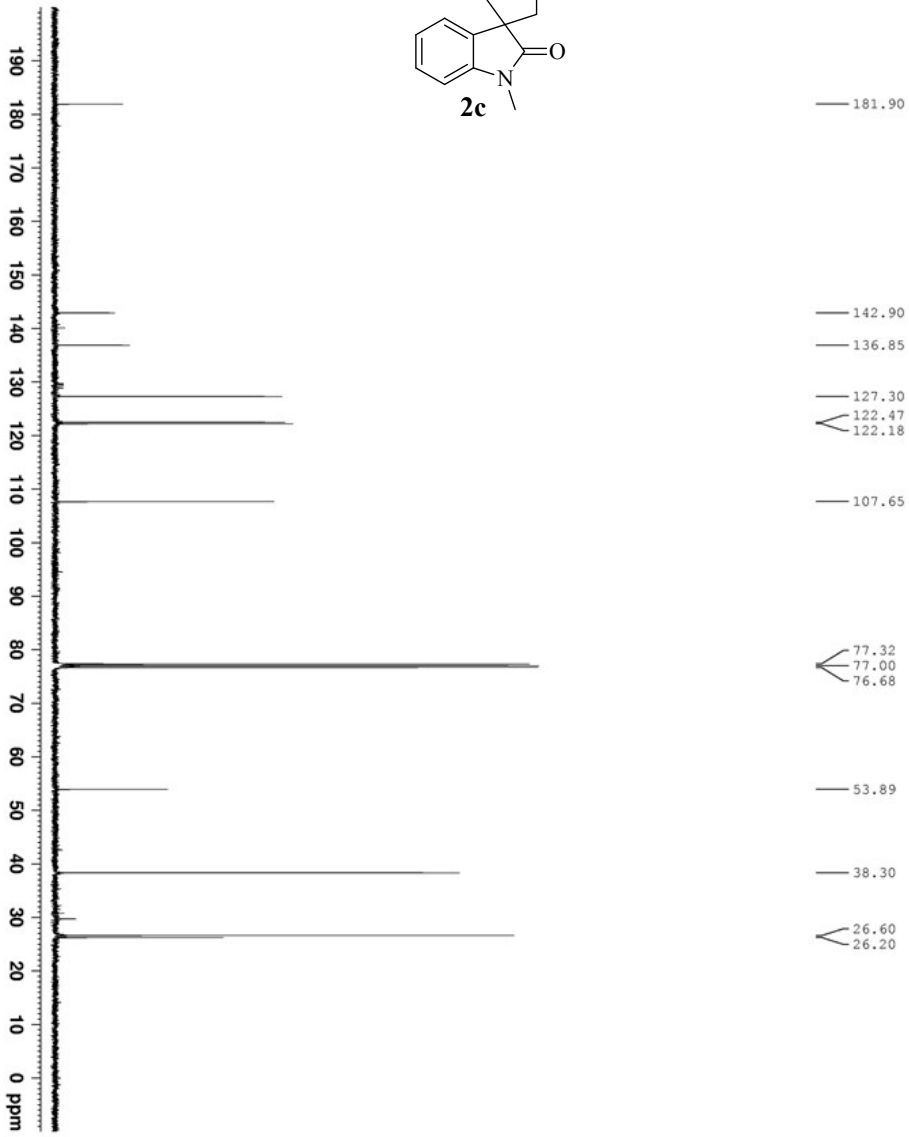
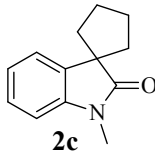
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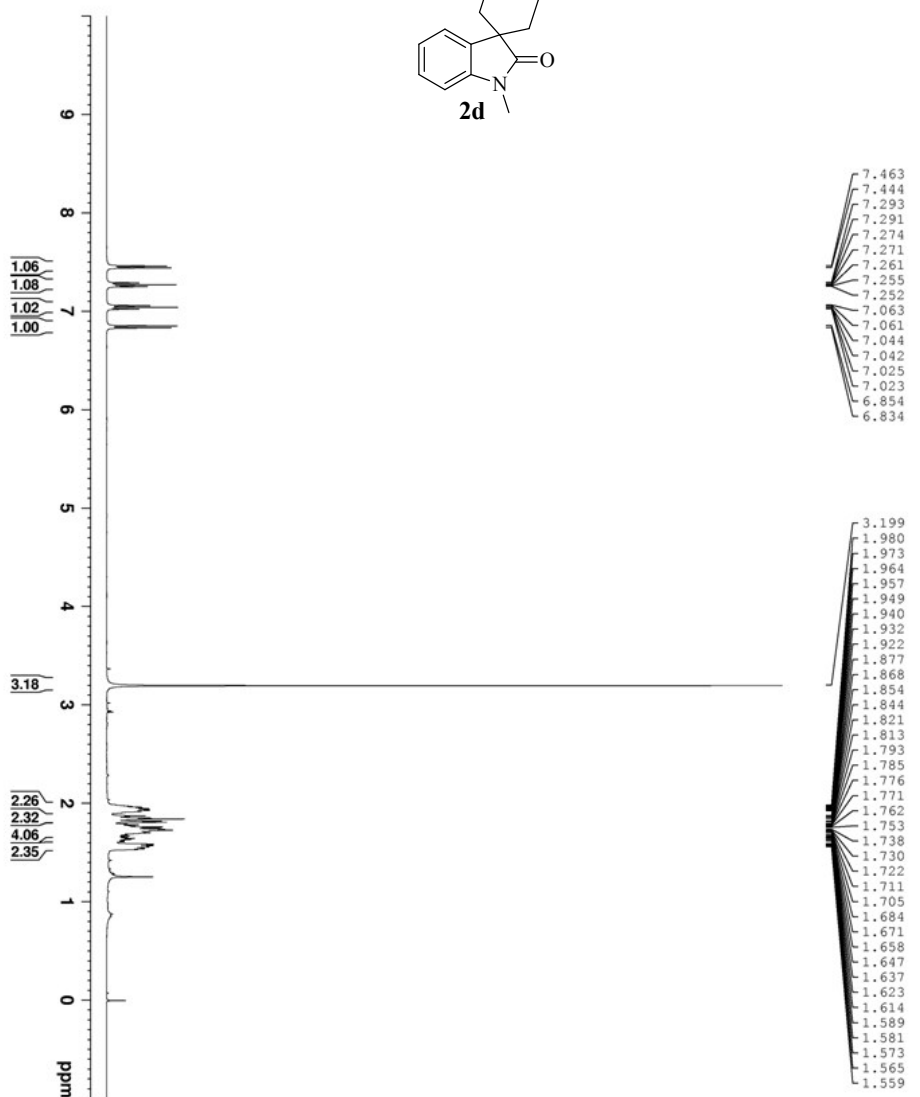
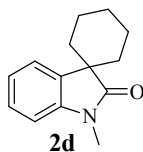
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```

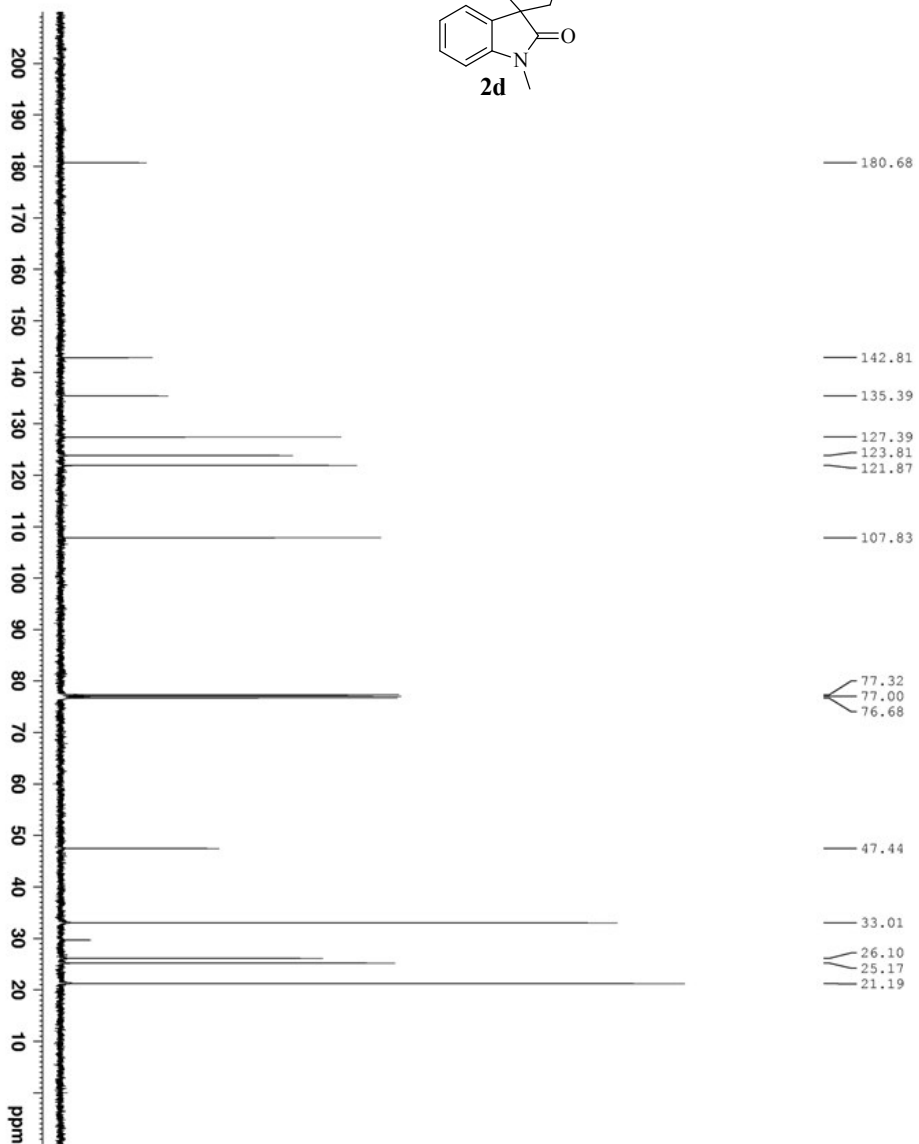
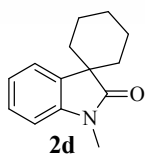
NAME      chem3c-4-p31-1
EXPNO    1
PROCNO   1
Date_    20150727
Time     14.02
INSTRUM  spect
PROBHD   5 mm PABBO BB7
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
DS        2
SWH       24038.461 Hz
FIDRES    0.455928 Hz
AQ         1.459328 sec
RG         812
DW         20.800 usec
DE         5.500 usec
TE         29.2 K
D1         2.00000000 sec
D11        0.03000000 sec
D12        1
===== CHANNEL f1 =====
SFO1     100.628298 MHz
NUC1      13C
P1        14.20
PL1       0.00 usec
SI        32768
SF        100.612722 MHz
K10W      EX
L1        0
L2        0
L3        0
GB        0
PC        1.40
  
```

1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (**2d**)



```

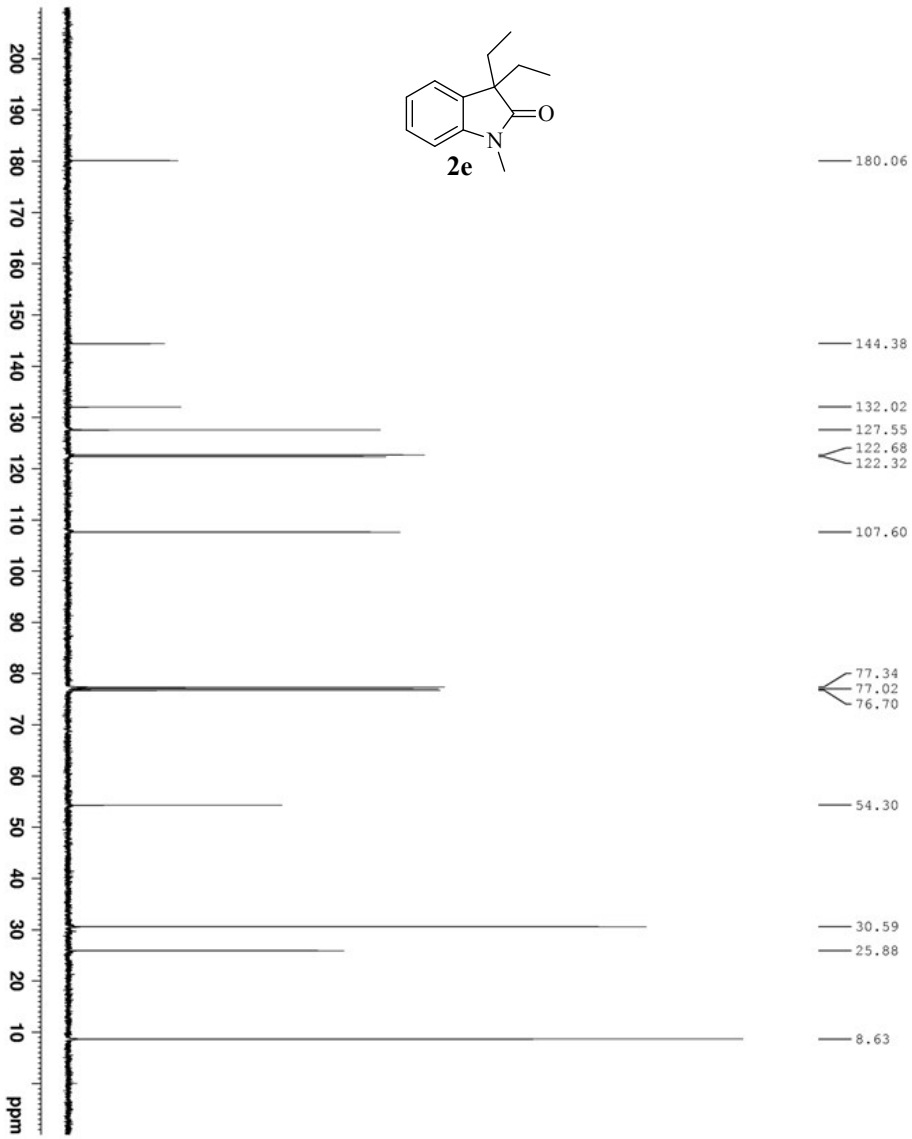
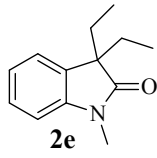
NAME: chem19-4-p31-2
EXPNO: 10
PROCNO: 10
Date_=: 20150127
Time: 14.09
PROBHD: 5 mm PABBO
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 12
DS: 2
SWH: 8012.820 Hz
AQ: 0.0212386 sec
RG: 4.0829266 sec
SM: 114
AQ: 62.400 usec
TE: 296.2 K
DE: 1.00000000 sec
TD0: 1
===== CHANNEL f1 =====
NUC1: 400.1324710 MHz
P1: 10.10 usec
PT: 1.00 usec
SI: 65536
SF: 400.1300093 MHz
WDW: EM
SSB: 0
LB: 0.50 Hz
GB: 0
PC: 1.00
    
```



```

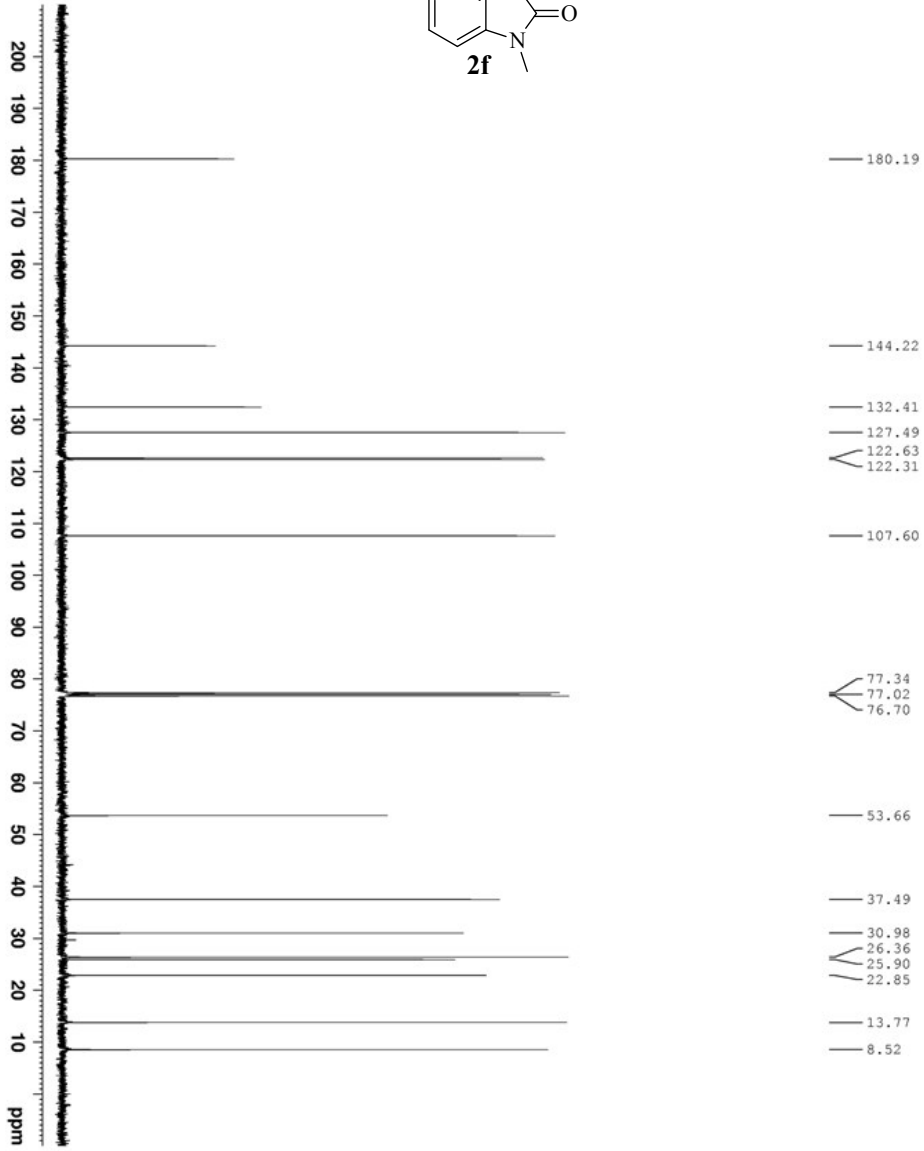
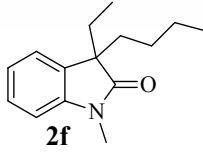
NAME          Chem14-4-p31-2
EXPNO         1
PROCNO        1
Date_         20150727
Time         14:29
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS           340
DS           2
SWH           24038.461 Hz
F2           100.627714 MHz
AQ           1.361368 sec
RG           1050
DM           20.800 usec
DE           297.9 K
TE           297.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1

===== CHANNEL f1 =====
SFO1         100.627714 MHz
NUC1         13C
P1           1.40 usec
SI           32768
SF           100.627714 MHz
WDW          EM
SS           0
LB           1.00 Hz
GB           0
PC           1.40
  
```

```

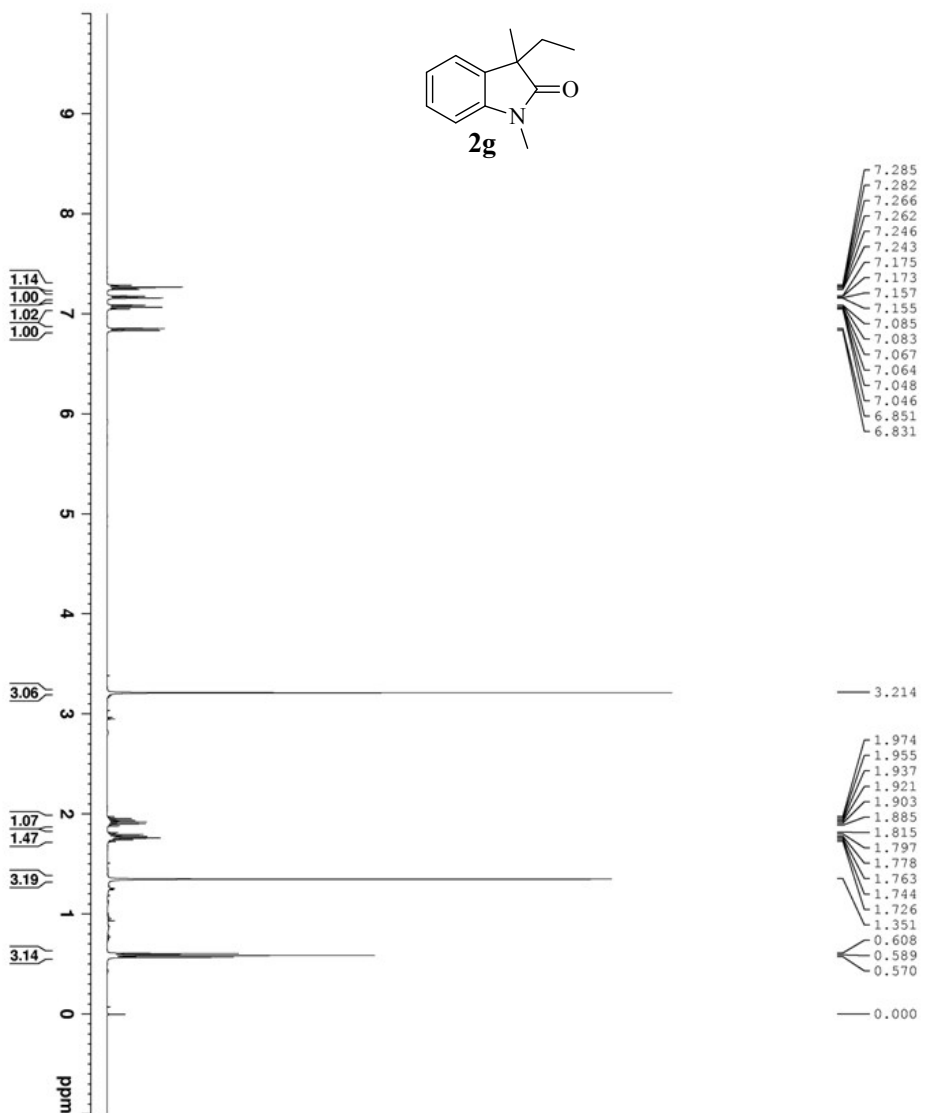
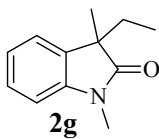
NAME: chem34-4-p34-1
EXPNO: 1
PROCNO: 1
Date_: 20150825
Time: 15.38
F2:
PROBHD: 5 mm BBOBO/BB/
PULPROG: zgpg30
TD: 65536
SFO1: 100.6228298 MHz
SF: 100.6228298 MHz
WDW: EM
SSB: 0
GB: 1.40
PC: 1.40
===== CHANNEL f1 =====
SFO1: 100.6228298 MHz
NUC1: 13C
P1: 12.00 usec
SI: 32768
SF: 100.6127690 MHz
WDW: EM
SSB: 0
GB: 1.40
=====
  
```

```

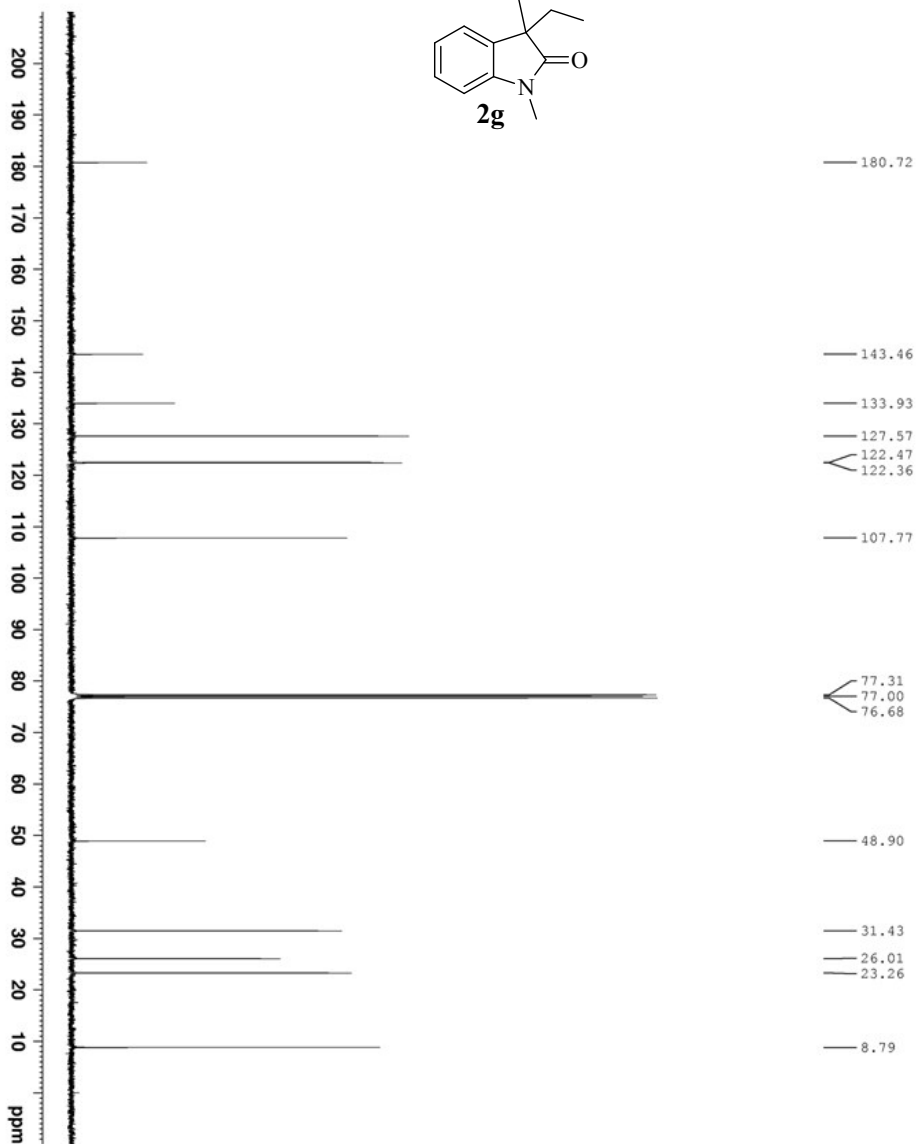
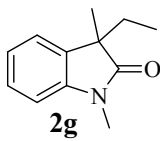
NAME: chenJ9-4-p34-2
EXPNO: 27
PROCNO: 1
Date_ : 20150825
Time: 16.09
INSTRUM: spect
PROBHD: 5 mm PABBO BB/
PULPROG: zgpg30
TD: 65536
SOLVENT: CD400
NS: 400
DS: 2
SWH: 24038.461 Hz
AQ: 0.0475181 sec
RG: 1.5631968 sec
DM: 20.800 usec
DE: 29.30 usec
TE: 300.2 K
D1: 2.00000000 sec
D11: 0.03000000 sec
D10: 1
===== CHANNEL f1 =====
SF01: 100.628298 MHz
NUC1: 13C
P1: 1.00 usec
SI: 32768
SF: 100.6127690 MHz
WDW: EM
SSB: EX
GB: 0
PC: 1.40
  
```

3-ethyl-1,3-dimethylindolin-2-one (2g)



```

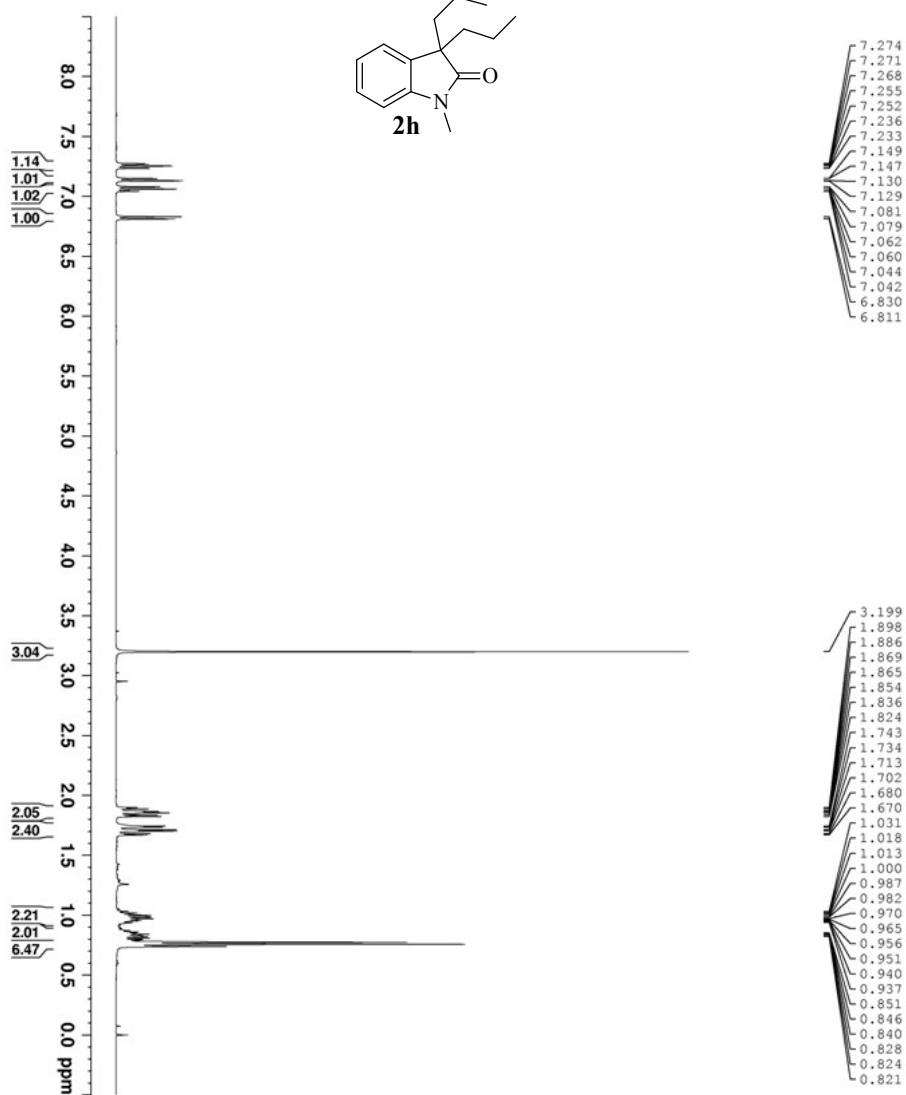
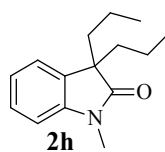
NAME: chem13-4-p127 5-4
EXPNO: 12
PROCNO: 1
Date_ : 20150905
Time: 14.48
INSTRUM: spect
PROBHD: 5 mm F400 BBI/
PULPROG: zgpg30
AQ: 6.2930
SFO1: 400.1360772 MHz
SFO2: 101.6251250 MHz
SOLVENT: CDCl3
NS: 16
DS: 2
AQ: 8012.882 Hz
FIDRES: 0.122266 Hz
AQ: 4.0894966 sec
RG: 144
DM: 62.430 usec
TE: 296.7 K
D1: 1.00000000 sec
D11: 1
===== CHANNEL f1 =====
SFO1: 400.1364710 MHz
NUC1: 13
P1: 10.00 usec
SI: 65536
SF: 400.1360072 MHz
MWM: 8K
SFO2: 101.6251250 MHz
P2: 0.30 Hz
SI: 0
SFO3: 0
SI: 1.00
PC: 1.00
    
```



```

NAME: Chem14-4-p37 3-4
EXPNO: 1
PROCNO: 1
Date_: 20150905
Time: 18.29
INSTRUM: zgpg30
PROBHD: 5 mm PABBO BB/
PULPROG: zgpg30
TD: 65536
SOLVENT: DMSO
NS: 500
DS: 2
SWH: 24038.461 Hz
F2: 100.628298 MHz
AQ: 1.361368 sec
RG: 1020
DM: 20.800 usec
DE: 297.9 K
TE: 297.2 K
D1: 2.00000000 sec
D11: 0.03000000 sec
TD0: 1
===== CHANNEL f1 =====
SFO1: 100.628298 MHz
NUC1: 13C
P1: 14.00 usec
SI: 32768
SF: 100.6127721 MHz
WDM: EX
EX: EX
LB: 1.00 Hz
GB: 0
PC: 1.40
  
```

1-methyl-3,3-dipropylindolin-2-one (**2h**)



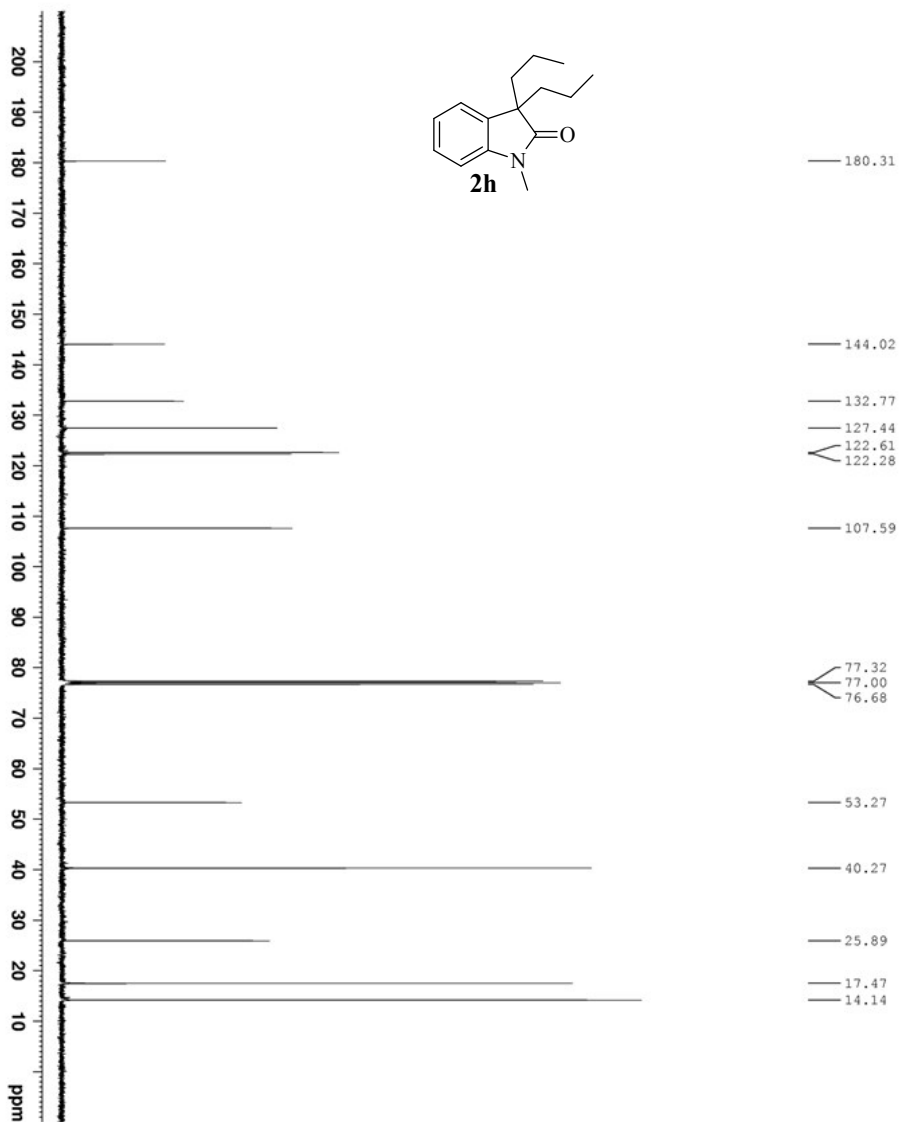
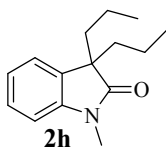
7.274
7.271
7.268
7.255
7.252
7.236
7.233
7.149
7.147
7.130
7.129
7.081
7.079
7.062
7.060
7.044
7.042
6.830
6.811

3.199
1.898
1.886
1.869
1.865
1.854
1.836
1.824
1.743
1.734
1.713
1.702
1.680
1.670
1.031
1.018
1.013
1.000
0.987
0.982
0.970
0.965
0.956
0.951
0.940
0.937
0.851
0.846
0.840
0.828
0.824
0.821

```

NAME: chen[4-4-p38--3
EXPNO: 12
PROCNO: 1
Date_ : 20150907
Time: 13.38
INSTRUM: spect
PROBHD: 5 mm EASY-1
PULPROG: zg30
TD: 65536
SOLVENT: CDCl3
DS: 2
SWH: 8012.820 Hz
AQ: 0.182466 Hz
RG: 4.139246 Hz
AQ: 128 sec
RG: 62.400 usec
DE: 301.9 Ksec
TE: 301.9 K
D1: 1.00000000 sec
ID0: 1

===== CHANNEL f1 =====
SFO1: 400.1324710 MHz
NUC1: 13
P1: 19.00 usec
SI: 65536
SF: 400.1300065 MHz
WGM: EX
LB: 0
GB: 0
PC: 1.00
    
```

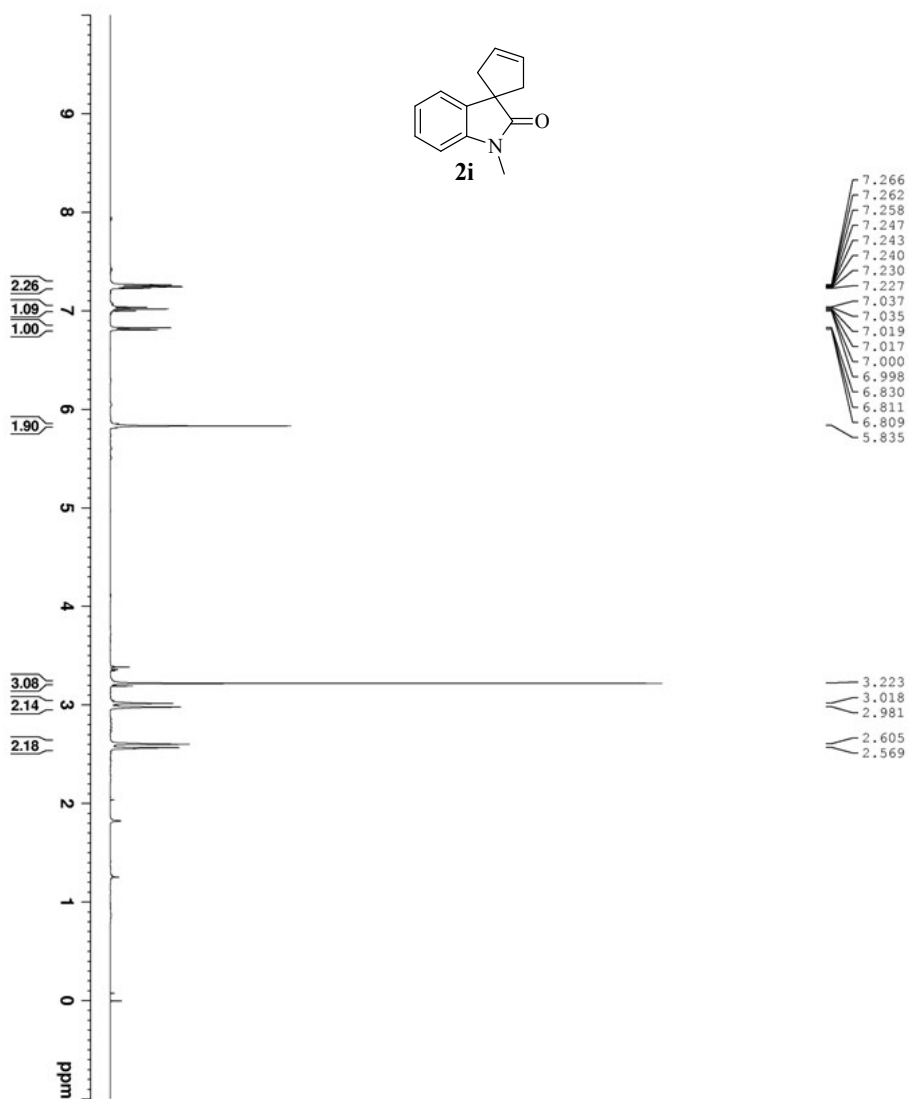
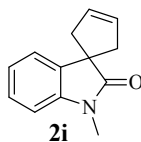


```

NAME          chen3d-4-f38-3
EXPNO         13
PROCNO        1
Date_         20150901
Time         14-08
INSTRUM       5 mm BBOBO
PROBHD        5mmBBO
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
DS            500
AQ            2.0000000
FIDRES        0.366798 Hz
AQ           1.369295
RG            200
DM            20.000 usec
DE            6.50 usec
TE            300.2 K
D1            2.0000000 sec
D11           0.03000000 sec
TDD           1

===== CHANNEL f1 =====
NUC1          13C
P1            13C usec
SFO1          100.628298 MHz
SFO2          125.761350 MHz
SF           100.612714 MHz
WDW           EM
SSB           0
GB            0
PC            1.40
  
```

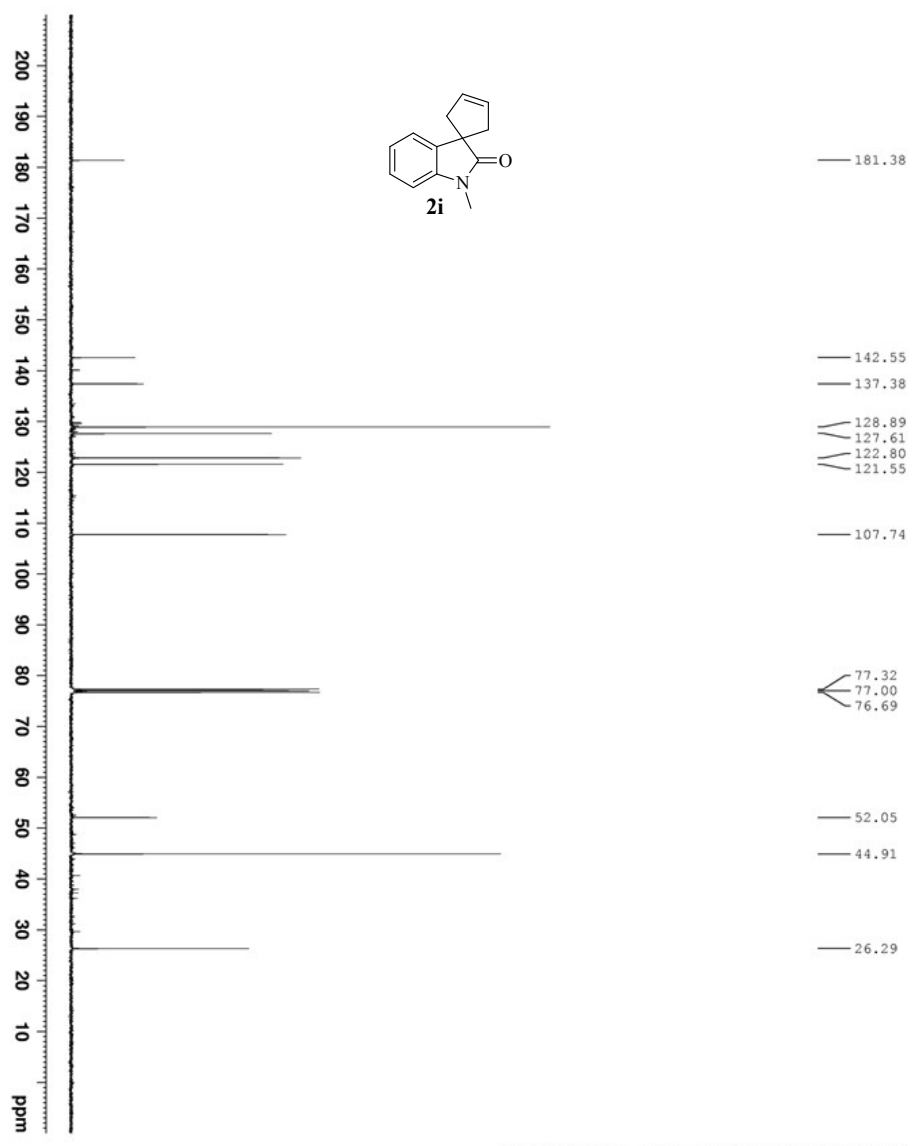
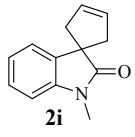
1'-methylspiro[cyclopent[3]ene-1,3'-indolin]-2'-one (**2i**)



```

NAME: chem3g-4-237.15-3
EXPNO: 1
PROCNO: 1
Date_=: 20150905
Time: 17.54
INSTRUM: spect
PROBHD: 5 mm PABBO
PULPROG: zg30
TD: 65536
F2: 400.1300833
SOLVENT: CHL12
NS: 12
DS: 2
SWH: 8012.820 Hz
AQ: 0.122966 sec
RG: 4.0839966
RG2: 1.14
SM: 62.400 usec
TE: 296.2 K
DE: 2.963 X
D1: 1.00000000 sec
TD0: 1

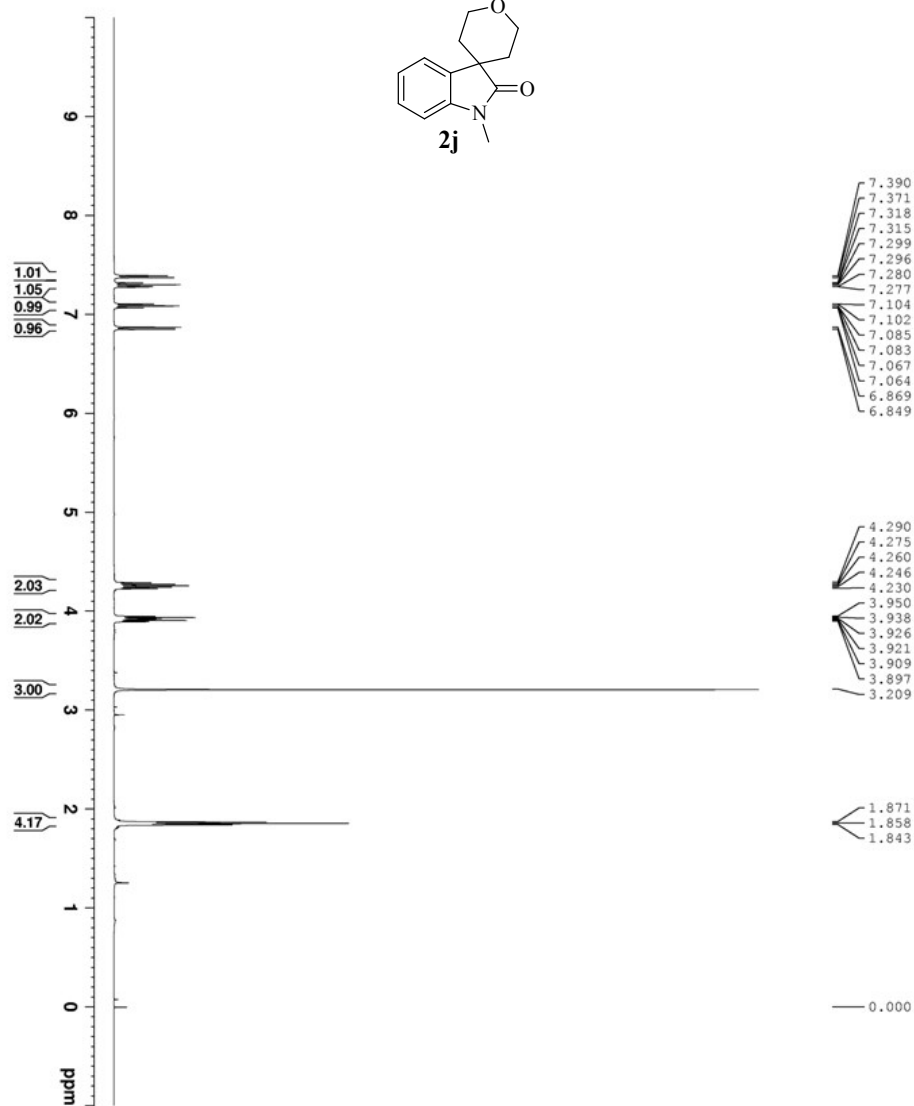
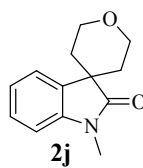
===== CHANNEL f1 =====
SFO1: 400.1324710 MHz
NUC1: 13
P1: 10.10 usec
PT: 1.00 usec
SI: 65536
SF: 400.1300833 MHz
WDM: EX
WDW: EM
SSB: 0
LB: 0.50 Hz
GB: 0
PC: 1.00
    
```



```

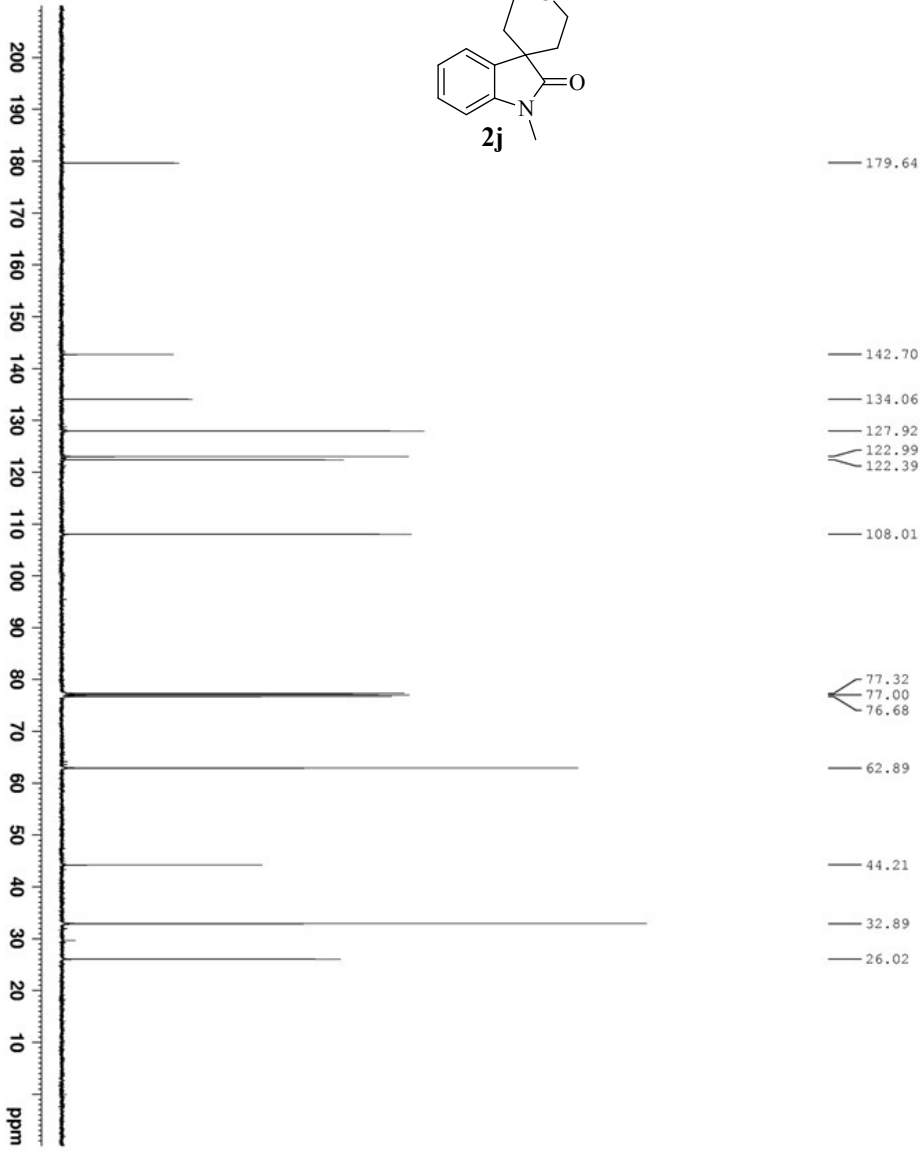
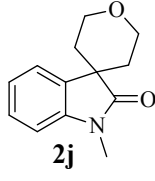
NAME: chenJg-4-p37,5-3
EXPNO: 12
PROCNO: 1
Date_ : 20150905
Time: 18.24
INSTRUM: spect
PROBHD: 5 mm PABBO
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 512
DS: 2
SWH: 24028.461 HZ
FIDRES: 0.266798 HZ
AQ: 0.366798 SEC
RG: 1.5051812
DM: 20.800 USEC
DE: 6.50 USEC
TE: 297.2
D1: 2.00000000 SEC
D11: 0.03000000 SEC
TD0: 1
===== CHANNEL f1 =====
SFO1: 100.6282298 MHZ
NUC1: 13C
P1: 1.40
PL1: 0.00 USEC
SI: 32768
SF: 100.6127744 MHZ
NUC2: EX
P2: 1.00 USEC
PL2: 0
GB: 1.40
PC: 0
  
```


1-methyl-2',3',5',6'-tetrahydrospiro[indoline-3,4'-pyran]-2-one (**2j**)



```

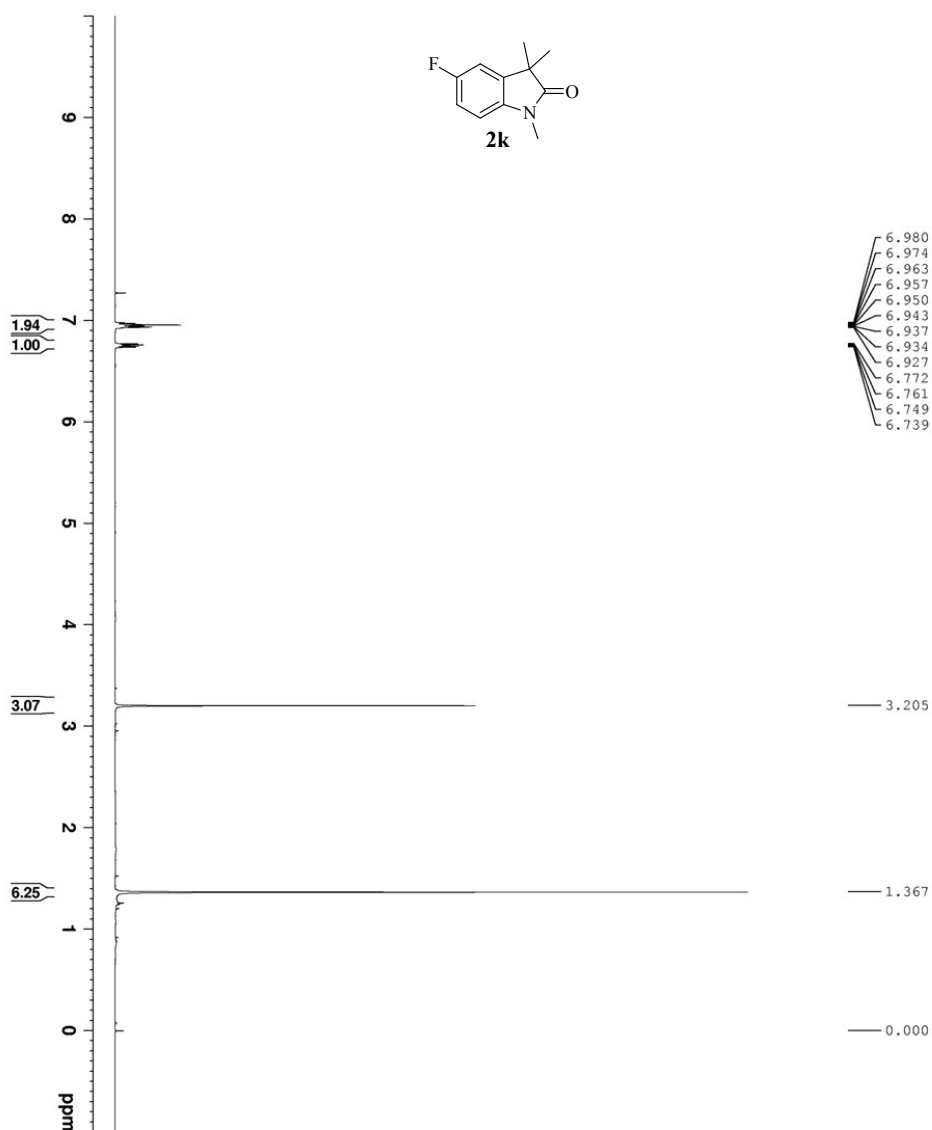
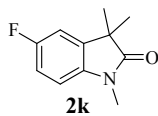
NAME          chenjg-4-p37.5-2
EXPNO         1
PROCNO        1
Date_         20150905
Time          17.19
PULPROG       5 mm PABPO 9B/
PROBHD        2930
TD            65536
SOLVENT       CDCl3
NS           16
DS           2
SH            812.820 Hz
AQ           0.12490 sec
RG           4.089966 sec
RG           101
AQ           62.400 usec
TE           296.2 K
DE           1.00000000 sec
D1           1
===== CHANNEL f1 =====
SFO1          400.1324710 MHz
NUC1          13
SI           10.42 usec
SF           400.1300044 MHz
WDW          EM
SS           0
LB           0.30 Hz
GB           0
PC           1.00
    
```



```

NAME: chen19-4-p37-5-2
EXPNO: 12
PROCNO: 1
Date_ : 20150905
Time: 14.43
INSTRUM: spect
PROBHD: 5 mm PABBO BB/
PULPROG: zgpg30
SOLVENT: CDCl3
NS: 500
DS: 2
F2: 24038.461 Hz
F3: 0.366791 Hz
AQ: 1.3631988 sec
RG: 1030
DM: 20.800 uSinc
DE: 4.500 uSinc
TE: 297.8 K
D1: 2.00000000 sec
D11: 0.03000000 sec
D10: 1
===== CHANNEL f1 =====
SF01: 100.6282298 MHz
P1: 14.00 uSinc
SI: 32768
SF: 100.6127751 MHz
KRM: EX
L8: 1.00 Hz
GB: 0
FC: 1.40
  
```

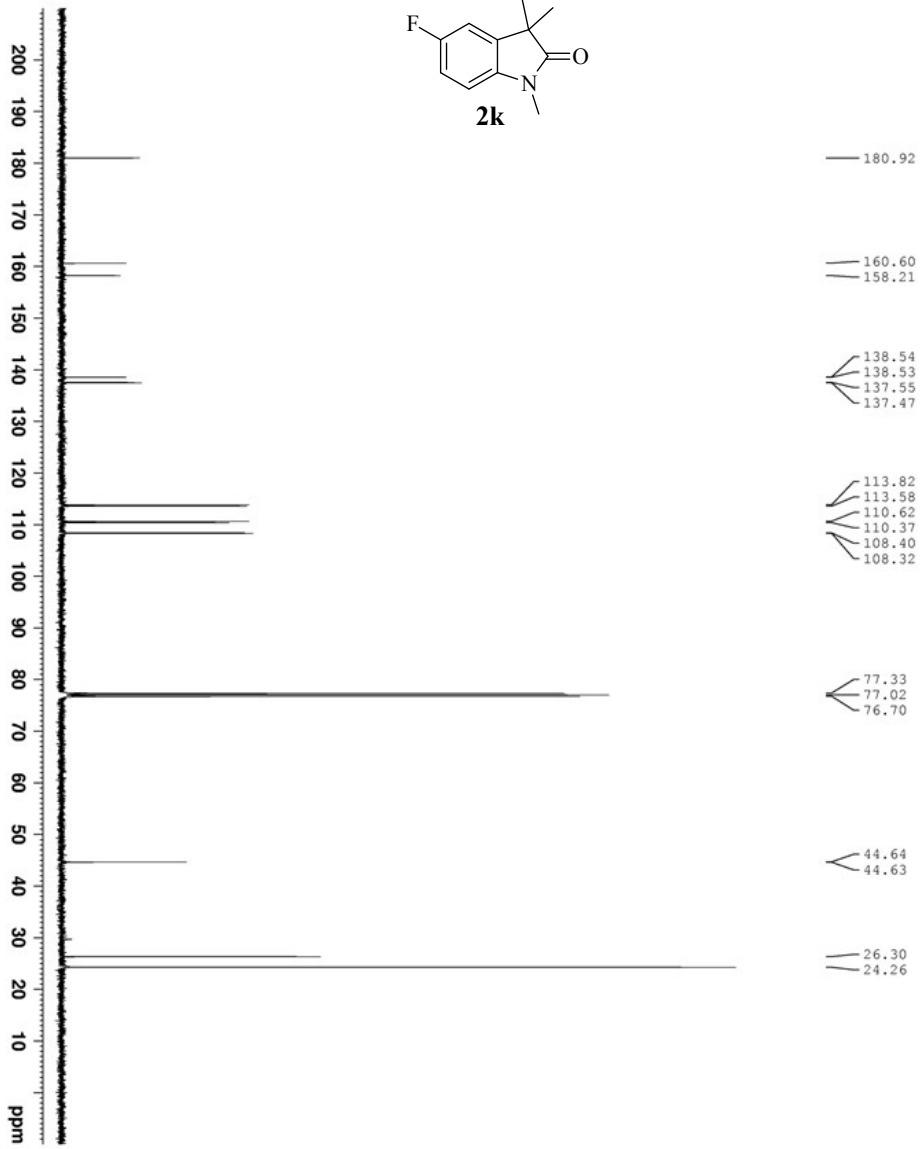
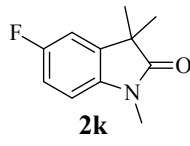
5-fluoro-1,3,3-trimethylindolin-2-one (**2k**)



```

NAME          chem|9-4-p34-4
EXPNO        11
PROCNO       1
F2 - Time    20150921
F2 - Time    17.40
INSTRUM      5 mm BBOBO HS/
PROBHD       spect
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           2
SWH           8012.822 Hz
FIDRES       0.122266 Hz
AQ           4.0894966 sec
RG           62.101 usec
DE           5.50 usec
TE           299.3 K
D1           1.00000000 sec
D10          1

===== CHANNEL f1 =====
SFO1         400.1324710 MHz
NUC1         13C
P1           10.70 usec
SI           65536
SF           400.1300041 MHz
Waltz16     EX
SFO2         0.30 Hz
L8           0
GB           1.00
PC
    
```

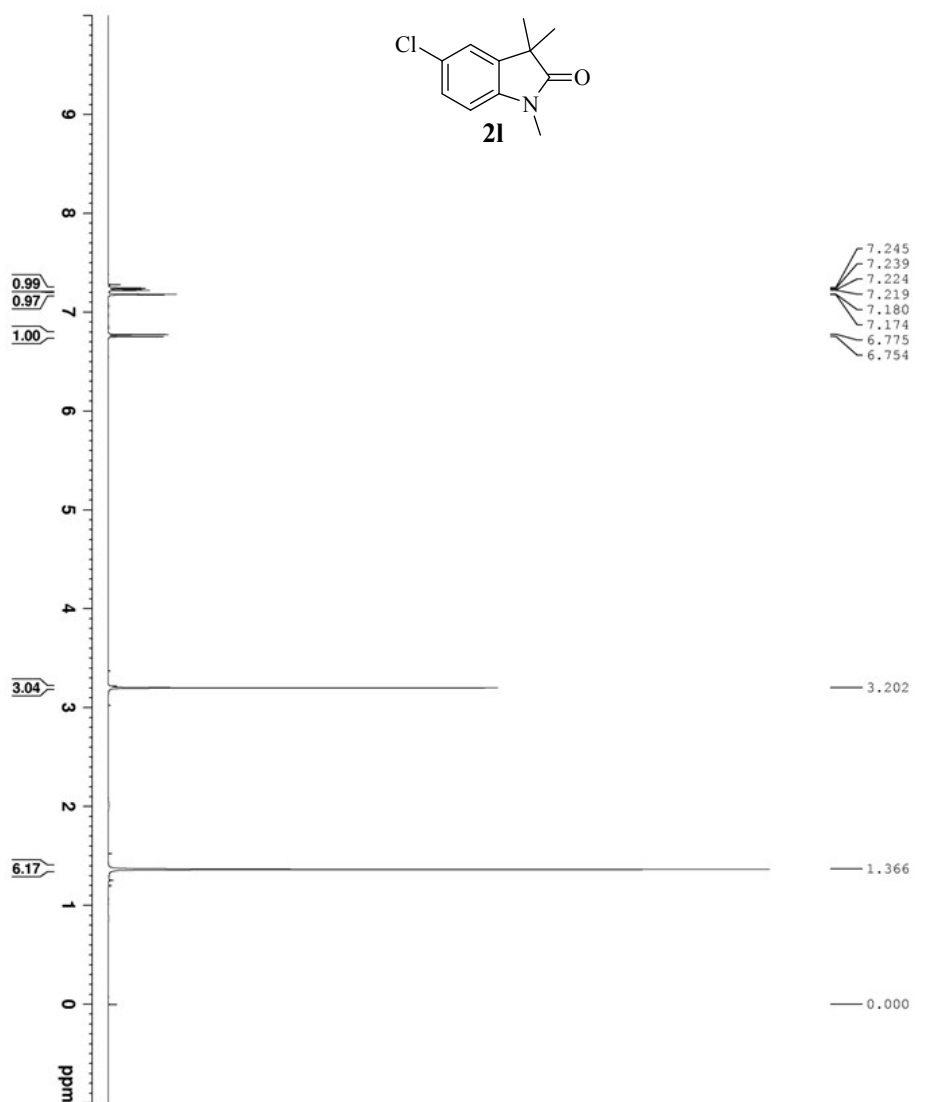
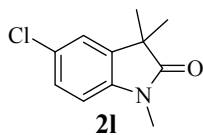


```

NAME: ChenJq-4-p34-4
EXPNO: 13
PROCNO: 1
Date_: 20150825
Time: 18.04
INSTRUM: spect
PROBHD: 5 mm PABBO 90/
PULPROG: zgpg30
TD: 65536
SFO1: 100.627690
AQ: 0.0300000
RG: 2050
DM: 20.800
DE: 300.5
TE: 300.2
D1: 2.00000000
D11: 0.03000000
D12: 0.03000000
D13: 0.03000000
D14: 0.03000000
D15: 0.03000000
DS: 2
SOLVENT: CDCl3
NS: 400
DS: 2
SOLVENT: CDCl3
AQ: 0.03000000
RG: 2050
DM: 20.800
DE: 300.5
TE: 300.2
D1: 2.00000000
D11: 0.03000000
D12: 0.03000000
D13: 0.03000000
D14: 0.03000000
D15: 0.03000000
===== CHANNEL f1 =====
SFO1: 100.627690 MHz
NUC1: 13C
P1: 12.00 usec
SI: 32768
SF: 100.627690 MHz
KCMW: EX
EX: 0
LBS: 0
GB: 0
PC: 1.40

```

5-chloro-1,3,3-trimethylindolin-2-one (**21**)



7.245
7.239
7.224
7.219
7.180
7.174
6.775
6.754

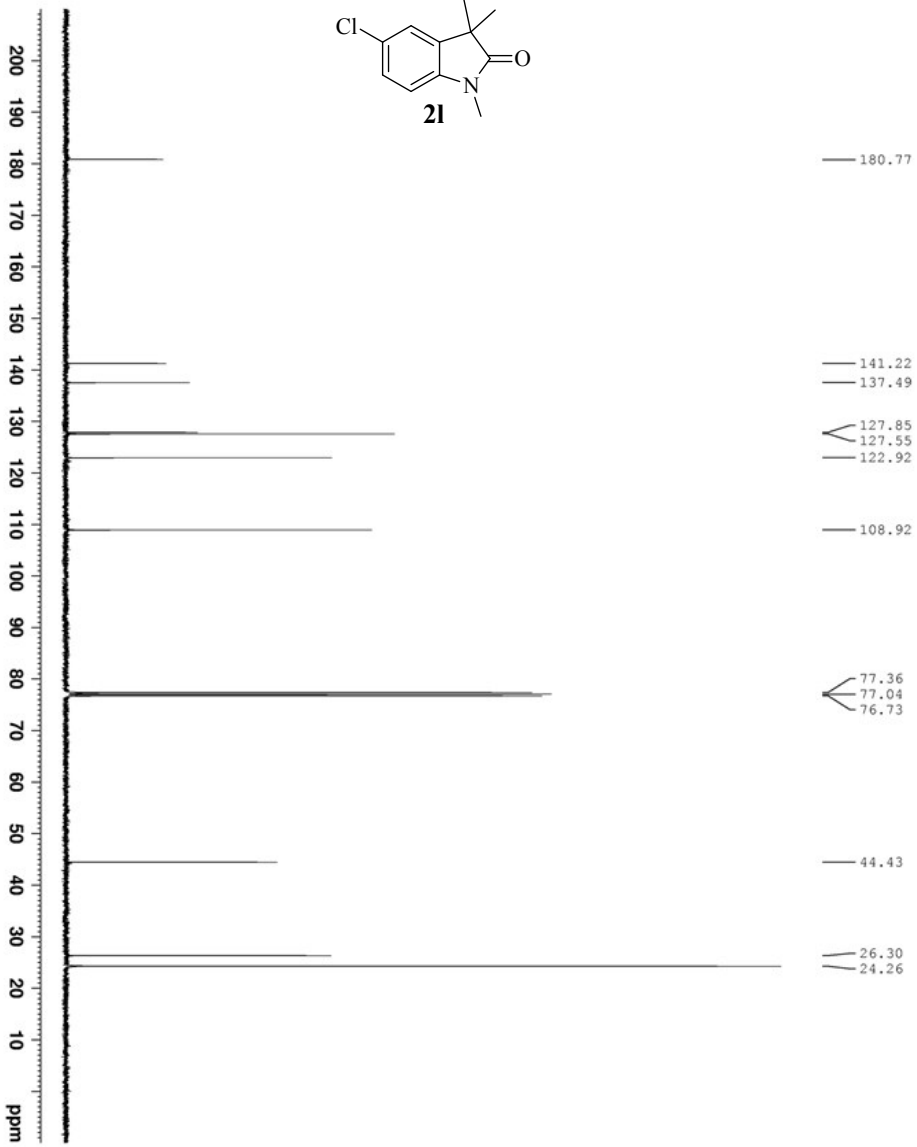
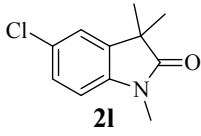
3.202

1.366

0.000

```

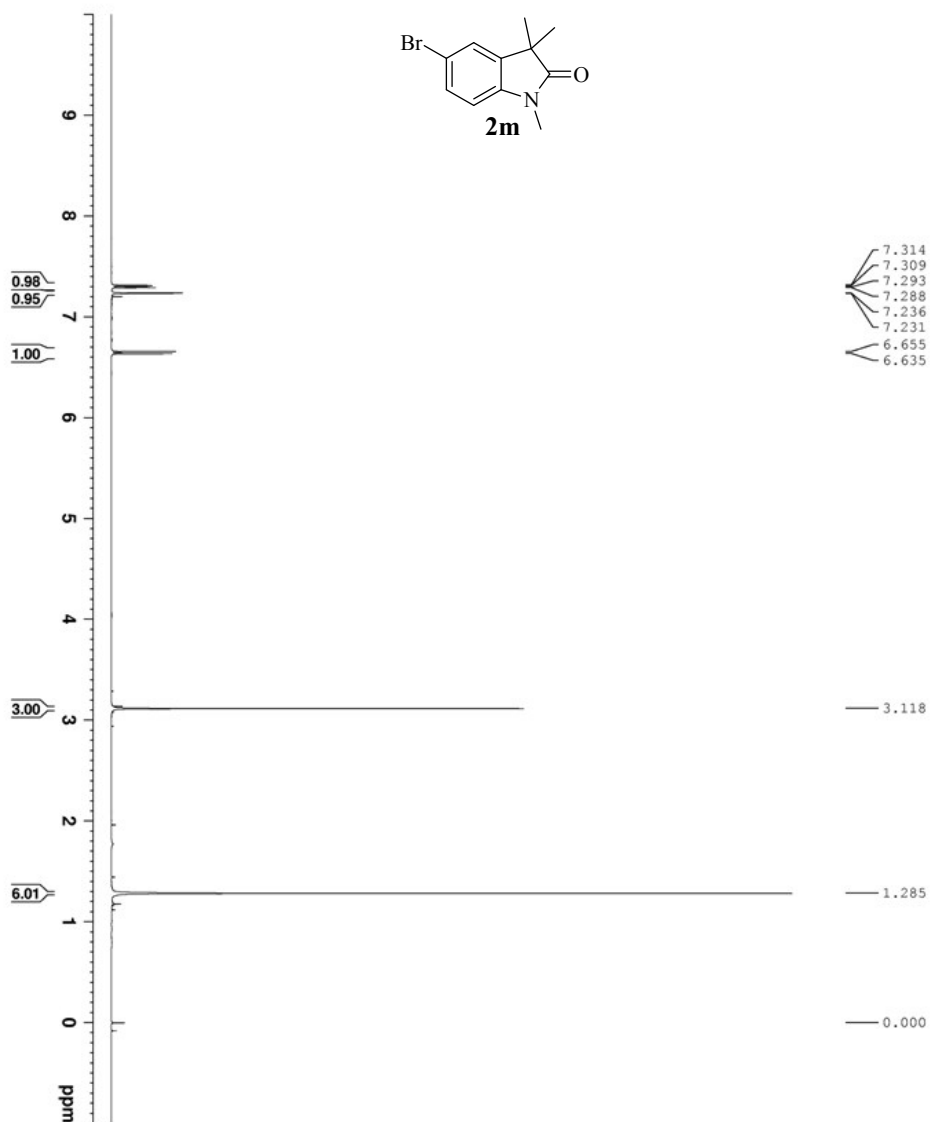
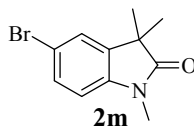
NAME: chen19-4-p3-3
PROCNO: 1
Date_: 20150828
Time: 11.28
INSTRUM: spect
PROBHD: 5 mm PABBO 5B/
PULPROG: zg30
TD: 65536
AQ: 4.0899962 sec
RG: 144
SD: 62.400 usec
DE: 294.1 K
TE: 1.00000000 sec
D1: 1
===== CHANNEL f1 =====
SFO1: 400.132719 MHz
NUC1: 13
P1: 19.00 usec
PT: 1.00 usec
SI: 65536
SF: 400.130036 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00
    
```



```

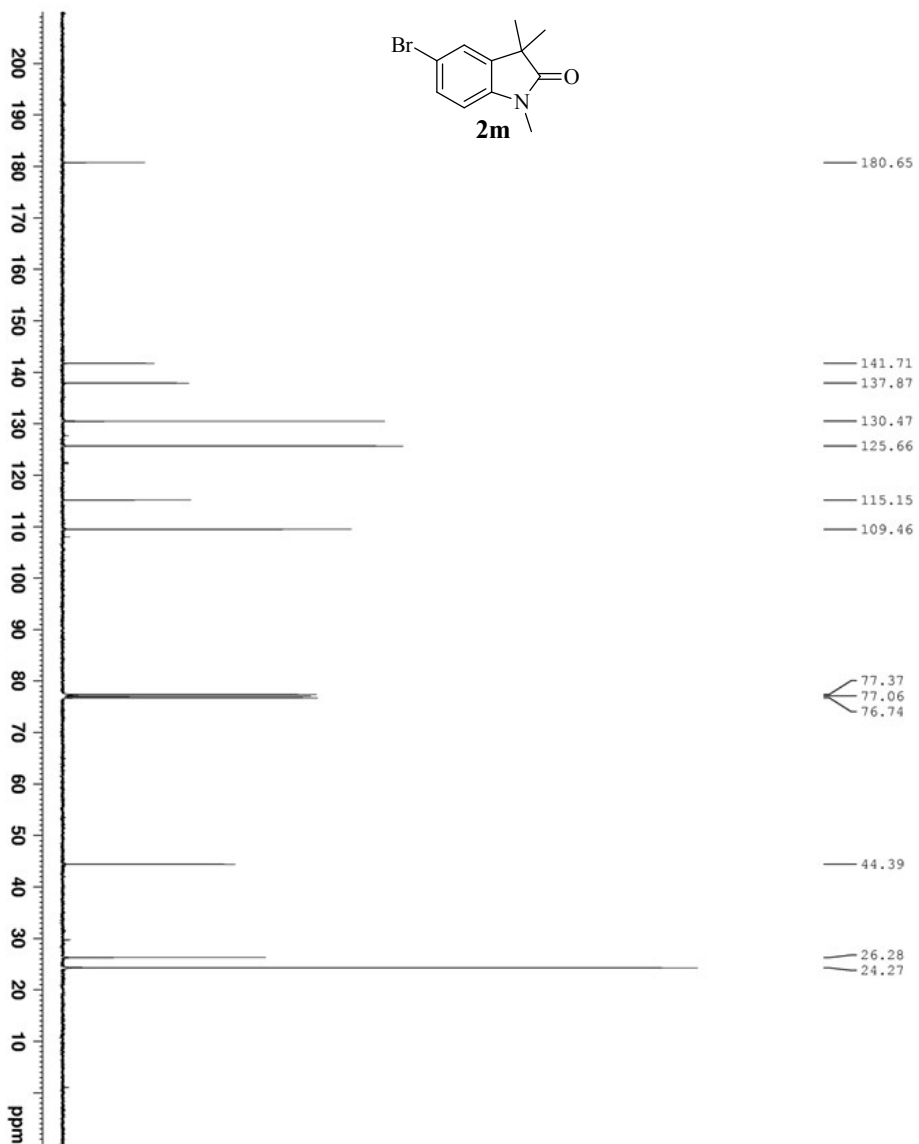
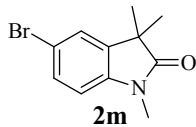
NAME          dhen19-4-13-3
EXPNO         11
PROCNO        1
Date_         20150928
Time          11.21
INSTRUM       spect
PROBHD        5 mm PABBO AB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            363
DS            2
SWH           24032.463 Hz
AQ            1.3631968 sec
RG            20.2050
DM            20.800 usec
DE            297.4 K
TE            2.00000000 sec
D1            0.03000000 sec
D11           1
D10           1
===== CHANNEL f1 =====
SFO1          100.628295 MHz
NUC1          13C
P1            12.00 usec
SI            32768
SF            100.627690 MHz
WGM           EX
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

5-bromo-1,3,3-trimethylindolin-2-one (2m)



```

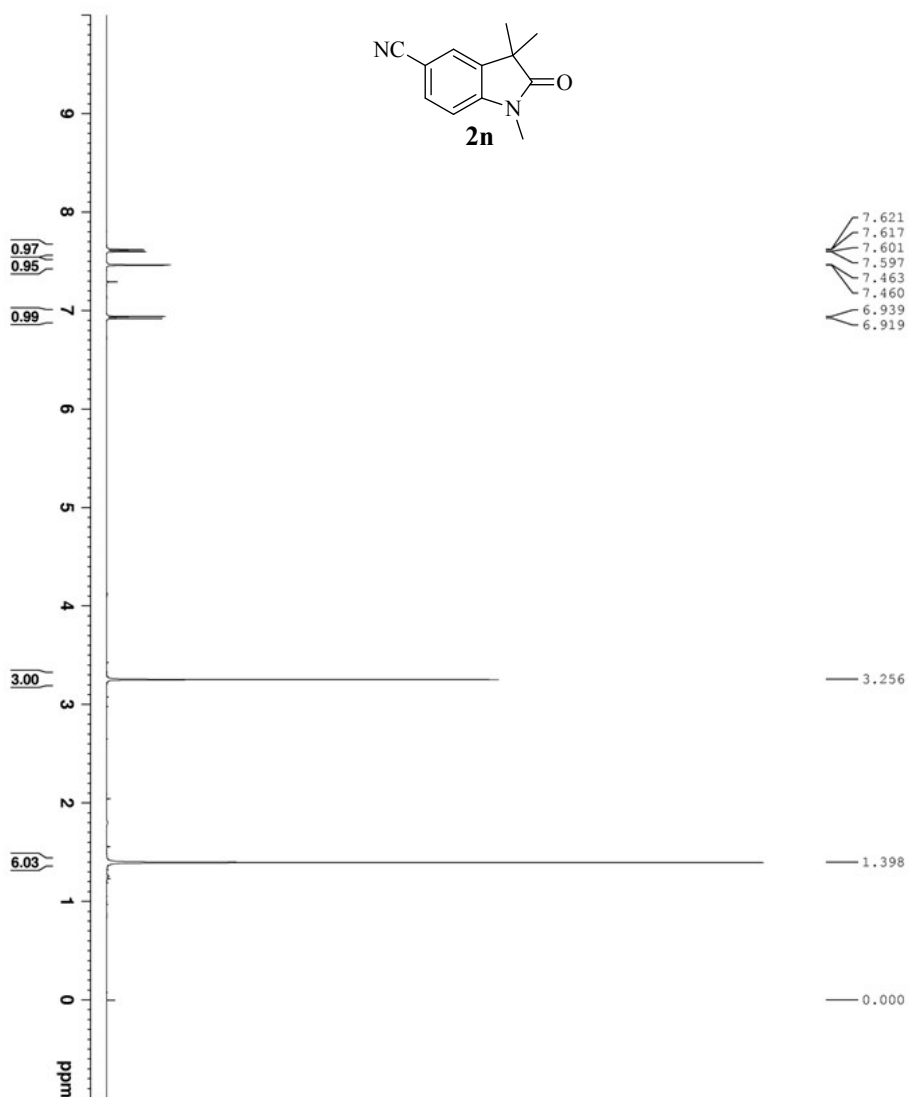
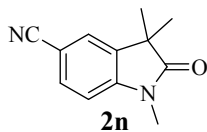
NAME: chemj3-4-p37-1
EXPNO: 20
PROCNO: 1
Date_ : 20160901
Time: 17.17
INSTRUM: spect
PROBHD: 5 mm PABBO BB/
PULPROG: zgpg30
F2 - F4: 64.44
SOLVENT: CDCl3
NS: 16
DS: 2
SWH: 8012.624 Hz
FIDRES: 0.122246 Hz
AQ: 4.0893966 sec
RG: 114
AQ: 62.400 usec
DE: 4.400 usec
TE: 296.5 K
D1: 1.00000000 sec
TD0: 1
===== CHANNEL f1 =====
SFO1: 400.1324710 MHz
NUC1: 13
P1: 10.00 usec
PT: 65.536
SR: 400.1300334 MHz
WDM: EM
LB: 0.30 Hz
GB: 0
FC: 1.00
    
```



```

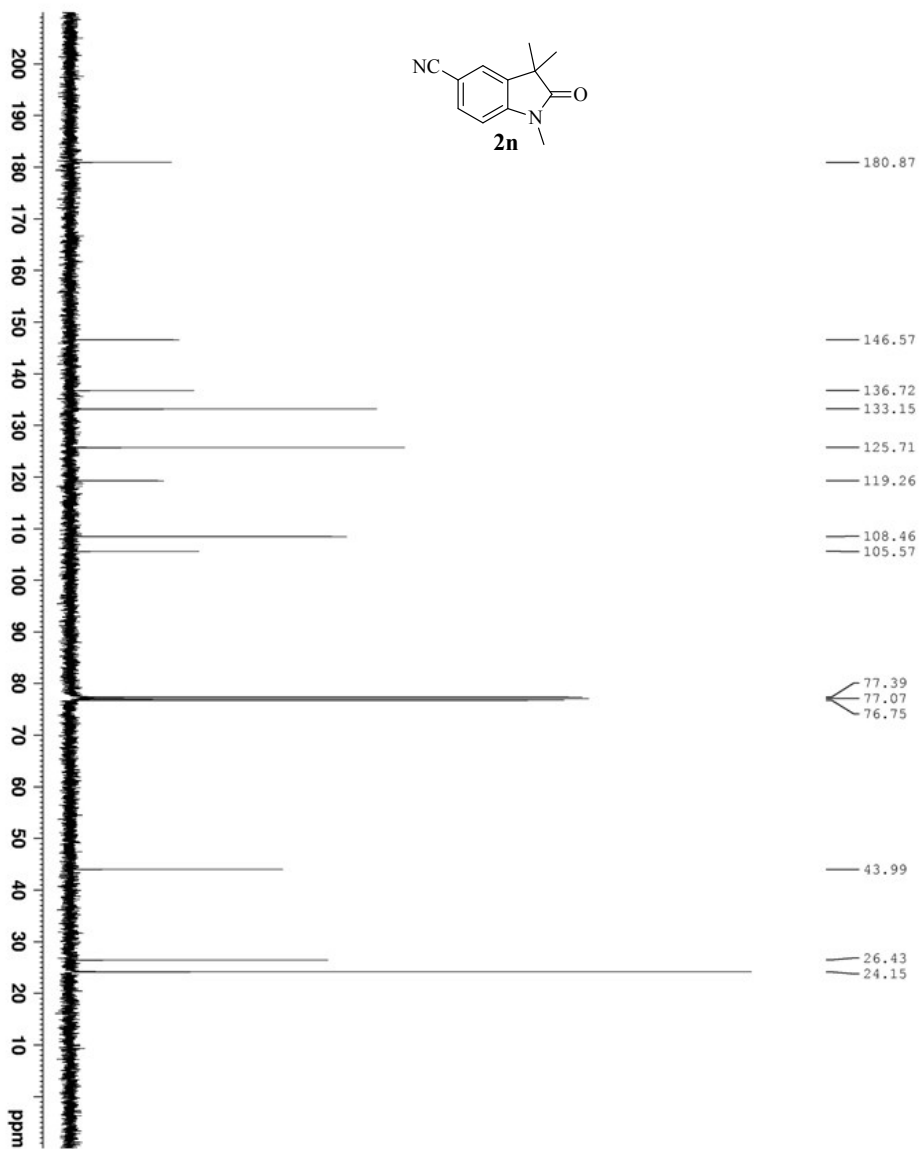
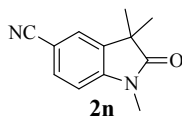
NAME          Chem14-4-p37-1
EXPNO         1
PROCNO        1
Date_         20150901
Time         14.27
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            500
DS            2
SWH           24038.461 Hz
AQ            1.361398 sec
RG            912
DM            20.800 usec
DE            297.4 K
TE            297.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
===== CHANNEL f1 =====
SFO1          100.628298 MHz
NUC1          13C
P1            14.00 usec
SI            32768
SF            100.627690 MHz
WDW           EM
SS            0
LB            1.00 Hz
GB            0
PC            1.40
  
```


1,3,3-trimethyl-2-oxoindoline-5-carbonitrile (**2n**)



```

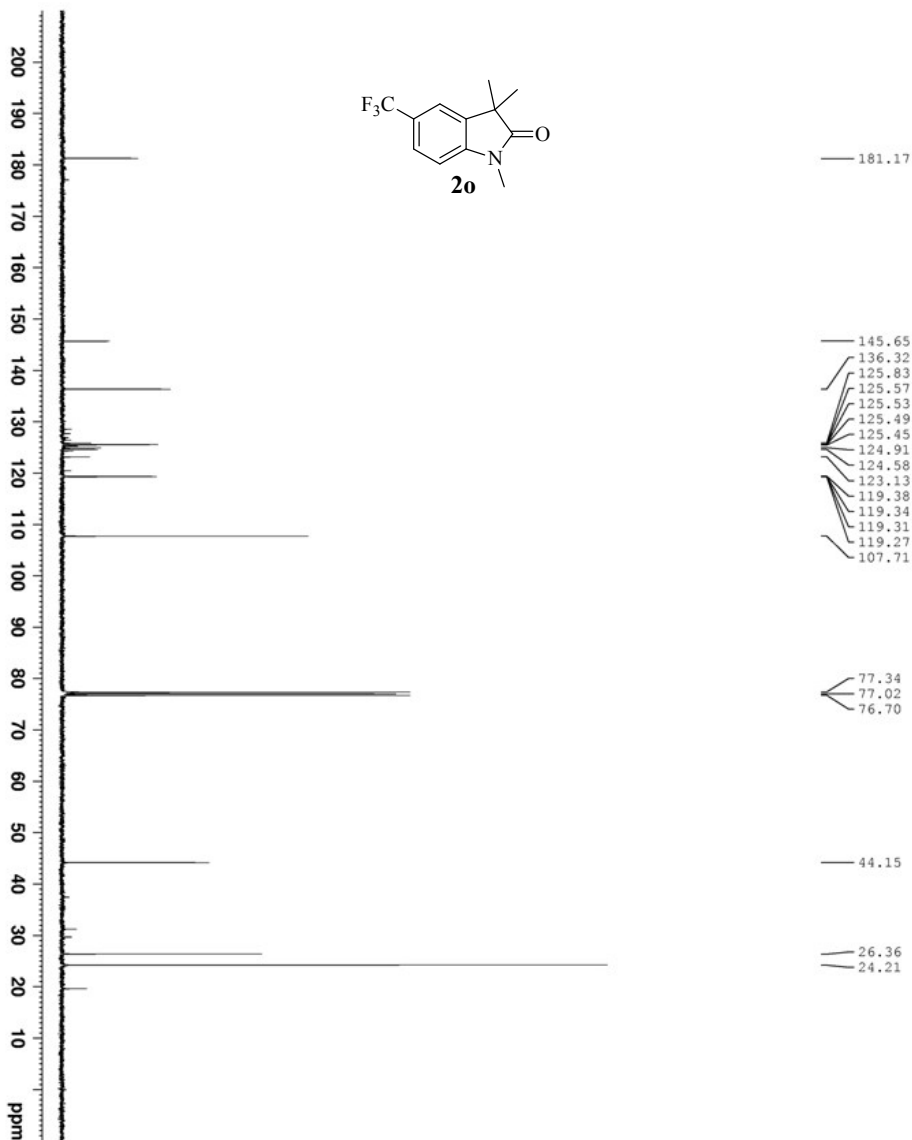
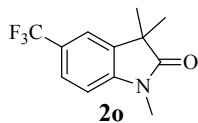
NAME: chem19-4-p35-2
EXPNO: 10
PROCNO: 10
Date_=: 20150827
Time: 21.44
INSTRUM: spect
PROBHD: 5 mm PABBO
PULPROG: zgpg30
TD: 65536
F2: 400.1299977
SOLVENT: CDCl3
NS: 1024
DS: 2
SWH: 8012.820 Hz
AQ: 0.001299977 sec
RG: 4.0833966
RG2: 1.44
AQ2: 62.400 usec
TE: 296.2 K
D1: 1.00000000 sec
===== CHANNEL f1 =====
NUC1: 13C
P1: 10.00 usec
PT: 1.00 usec
SI: 65536
SF: 400.1299977 MHz
WDM: EX
WDW: EM
SSB: 0
LB: 0.50 Hz
GB: 0
PC: 1.00
    
```



```

NAME: ChenJq-4-p33-2
EXPNO: 11
PROCNO: 1
Date_ : 20150827
Time: 21.46
INSTRUM: zgpg30
PROBHD: 5 mm PABBO BB/
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 51
DS: 2
SWH: 24038.461 Hz
AQ: 0.048444 sec
RG: 1.561398 sec
DM: 20.4800 usec
DE: 2.00000000 sec
TE: 297.1 K
D1: 2.00000000 sec
D11: 0.03000000 sec
D12: 0.03000000 sec
D13: 0.03000000 sec
D14: 0.03000000 sec
D15: 0.03000000 sec
===== CHANNEL f1 =====
SF01: 100.6228298 MHz
NUC1: 13C
PC1: 12.00 usec
SI: 32768
SF: 100.6127690 MHz
K1: 1.00 usec
K2: 1.00 usec
K3: 1.00 usec
K4: 1.00 usec
K5: 1.00 usec
K6: 1.00 usec
K7: 1.00 usec
K8: 1.00 usec
K9: 1.00 usec
K10: 1.00 usec
K11: 1.00 usec
K12: 1.00 usec
K13: 1.00 usec
K14: 1.00 usec
K15: 1.00 usec
K16: 1.00 usec
K17: 1.00 usec
K18: 1.00 usec
K19: 1.00 usec
K20: 1.00 usec
K21: 1.00 usec
K22: 1.00 usec
K23: 1.00 usec
K24: 1.00 usec
K25: 1.00 usec
K26: 1.00 usec
K27: 1.00 usec
K28: 1.00 usec
K29: 1.00 usec
K30: 1.00 usec
K31: 1.00 usec
K32: 1.00 usec
K33: 1.00 usec
K34: 1.00 usec
K35: 1.00 usec
K36: 1.00 usec
K37: 1.00 usec
K38: 1.00 usec
K39: 1.00 usec
K40: 1.00 usec
K41: 1.00 usec
K42: 1.00 usec
K43: 1.00 usec
K44: 1.00 usec
K45: 1.00 usec
K46: 1.00 usec
K47: 1.00 usec
K48: 1.00 usec
K49: 1.00 usec
K50: 1.00 usec
K51: 1.00 usec
K52: 1.00 usec
K53: 1.00 usec
K54: 1.00 usec
K55: 1.00 usec
K56: 1.00 usec
K57: 1.00 usec
K58: 1.00 usec
K59: 1.00 usec
K60: 1.00 usec
K61: 1.00 usec
K62: 1.00 usec
K63: 1.00 usec
K64: 1.00 usec
K65: 1.00 usec
K66: 1.00 usec
K67: 1.00 usec
K68: 1.00 usec
K69: 1.00 usec
K70: 1.00 usec
K71: 1.00 usec
K72: 1.00 usec
K73: 1.00 usec
K74: 1.00 usec
K75: 1.00 usec
K76: 1.00 usec
K77: 1.00 usec
K78: 1.00 usec
K79: 1.00 usec
K80: 1.00 usec
K81: 1.00 usec
K82: 1.00 usec
K83: 1.00 usec
K84: 1.00 usec
K85: 1.00 usec
K86: 1.00 usec
K87: 1.00 usec
K88: 1.00 usec
K89: 1.00 usec
K90: 1.00 usec
K91: 1.00 usec
K92: 1.00 usec
K93: 1.00 usec
K94: 1.00 usec
K95: 1.00 usec
K96: 1.00 usec
K97: 1.00 usec
K98: 1.00 usec
K99: 1.00 usec
K100: 1.00 usec
=====

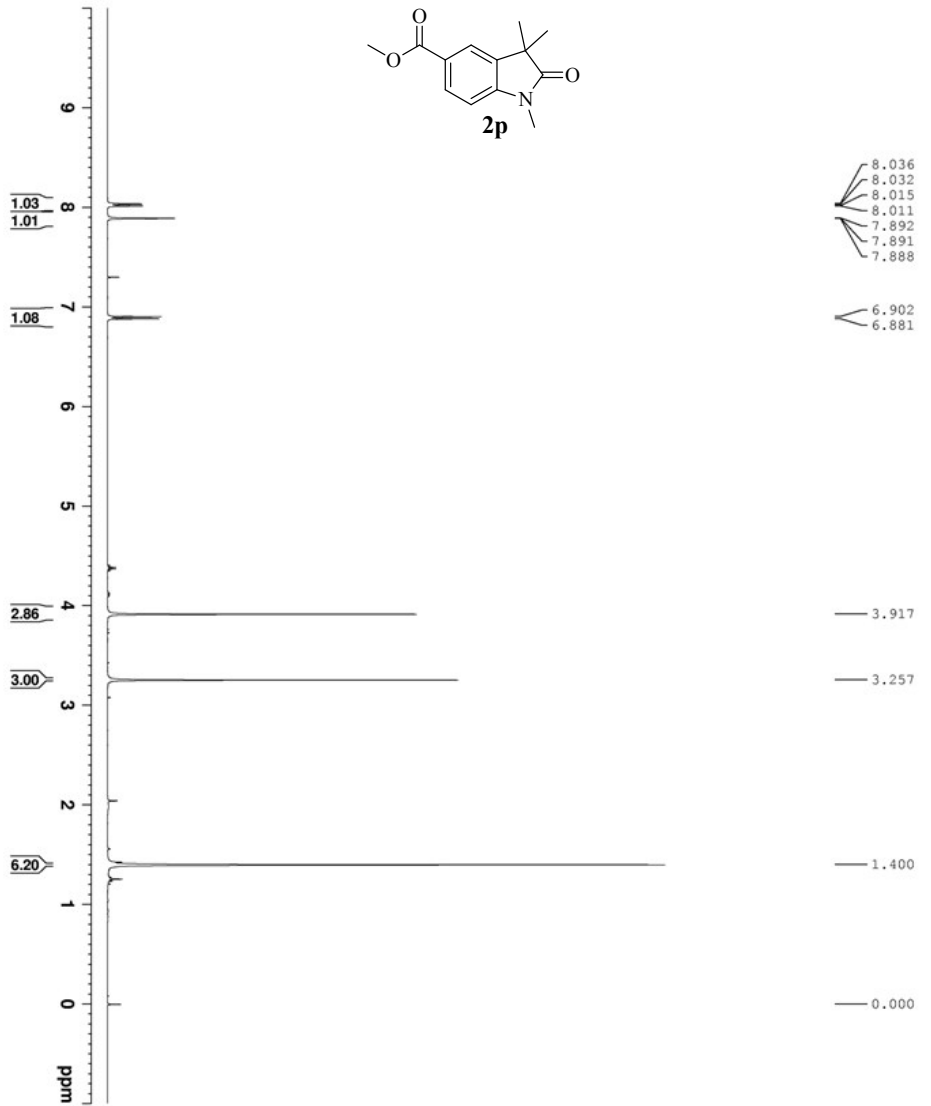
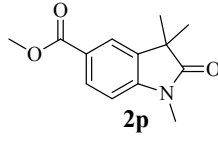
```

```

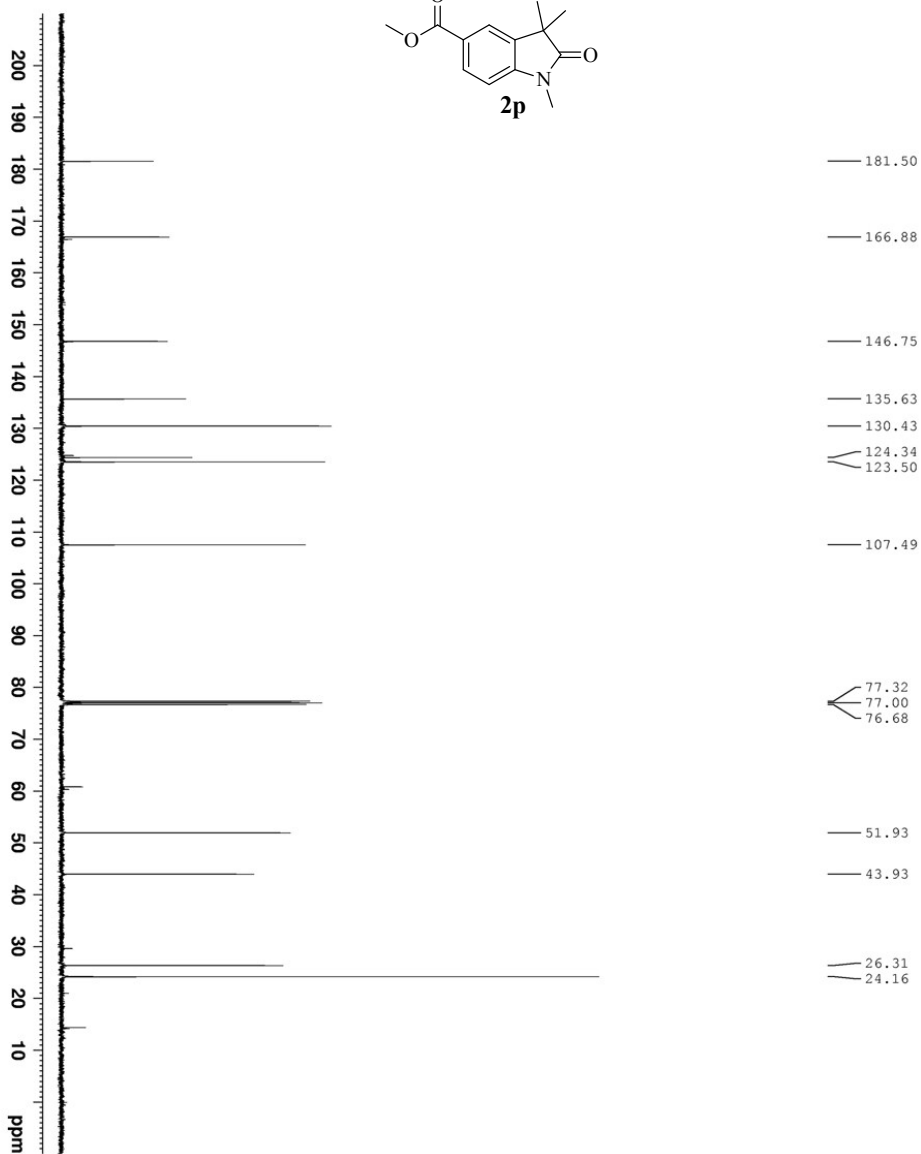
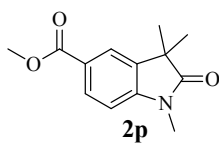
NAME          chem03f-4-p35-1-1
EXPNO         1
PROCNO        1
Date_         20150828
Time         12.21
INSTRUM       spect
PROBHD        5 mm PABBO BBI
PULPROG       zgpg30
TD            65536
SFO           100.6282998
NS            400
DS            2
SWH           24038.461 Hz
AQ            0.3241182 sec
RG            1.365198 sec
RC            2050
DM            20.800 usec
DE            297.0 usec
TE            300.2 K
D1            2.00000000 sec
D11           0.03000000 sec
D10           1
===== CHANNEL f1 =====
SFO1          100.6282998 MHz
NUC1          13C
P1            12.00 usec
SI            32768
SF            100.6127690 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

methyl 1,3,3-trimethyl-2-oxindoline-5-carboxylate (**2p**)



```

NAME: chemp3r-4-p40-1
EXPNO: 1
PROCNO: 1
Date_ : 20150919
Time: 21.13
INSTRUM: spect
PROBHD: 5 mm PABBO BB/
PULPROG: zgpg30
SOLVENT: DMSO
NS: 16
DS: 2
SWH: 8012.620 Hz
AQ: 0.1224617 sec
RG: 4.0894966 sec
DM: 62.101 usec
DE: 6.400 usec
TE: 294.3 K
D1: 1.00000000 sec
ID1: 1
===== CHANNEL f1 =====
SFO1: 400.1324710 MHz
NUC1: 13
SI: 10.78 usec
SF: 400.1299947 MHz
WIDEN: EX
LB: 0.30 Hz
GB: 0
PC: 1.00
    
```

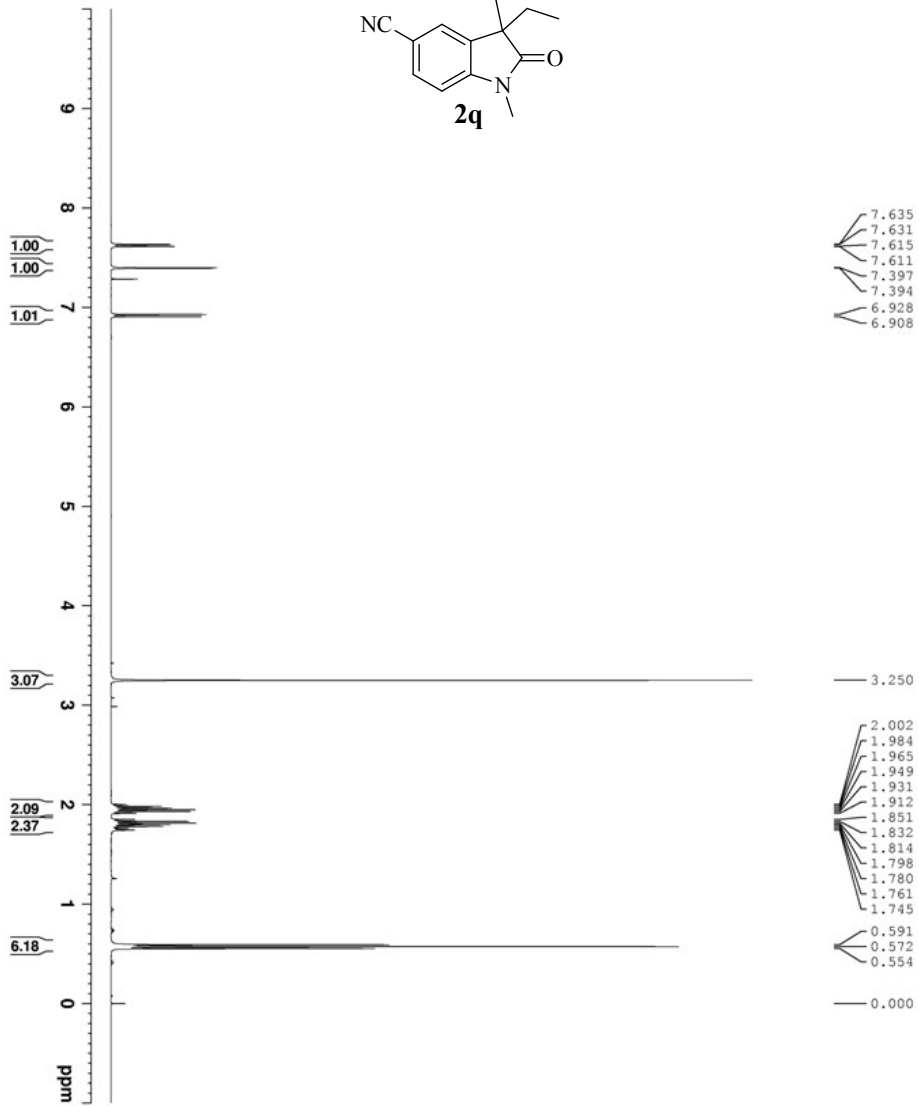
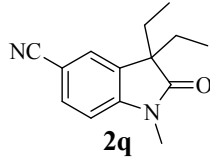


```

NAME: chen3q-4-pd-1
EXPNO: 12
PROCNO: 1
DATE_: 20150919
TIME: 12.43
INSTRUM: spect
PROBHD: 5 mm PABBO BB/
PULPROG: zgpg30
SOLVENT: CDCl3
NS: 320
DS: 2
SWH: 24038.461 Hz
F2: 101.6224298 MHz
AQ: 1.5613988 sec
RG: 2050
DM: 20.800 usec
DE: 0.0000000 sec
TE: 295.5 K
D1: 2.00000000 sec
D11: 0.03000000 sec
ID0: 1

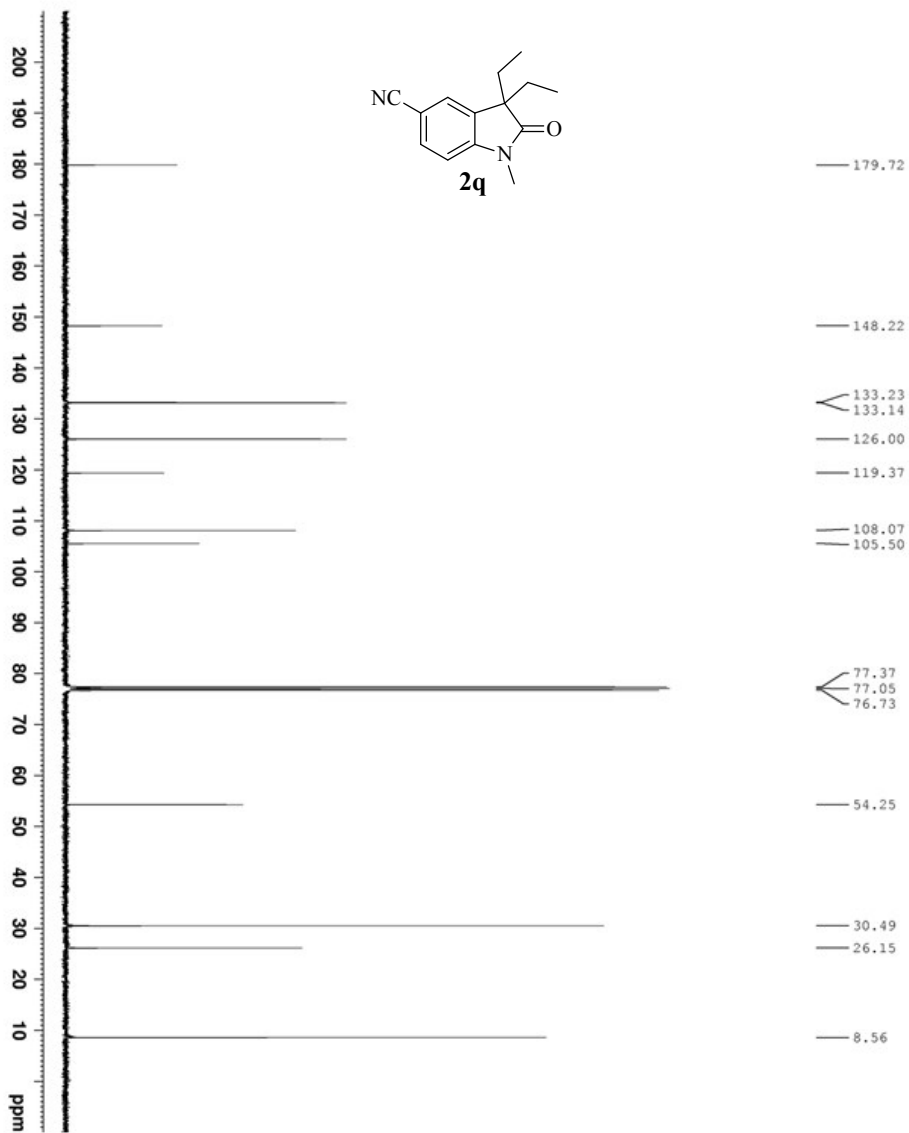
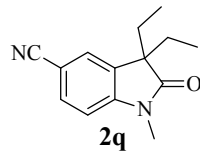
===== CHANNEL f1 =====
SF01: 100.6224298 MHz
NUC1: 13C
P1: 12.00 usec
SI: 32768
SF: 100.6127751 MHz
NAME: EX
NUC2: 13C
LB: 1.00 Hz
GB: 0
PC: 1.40
  
```

3,3-diethyl-1-methyl-2-oxindoline-5-carbonitrile (**2q**)



```

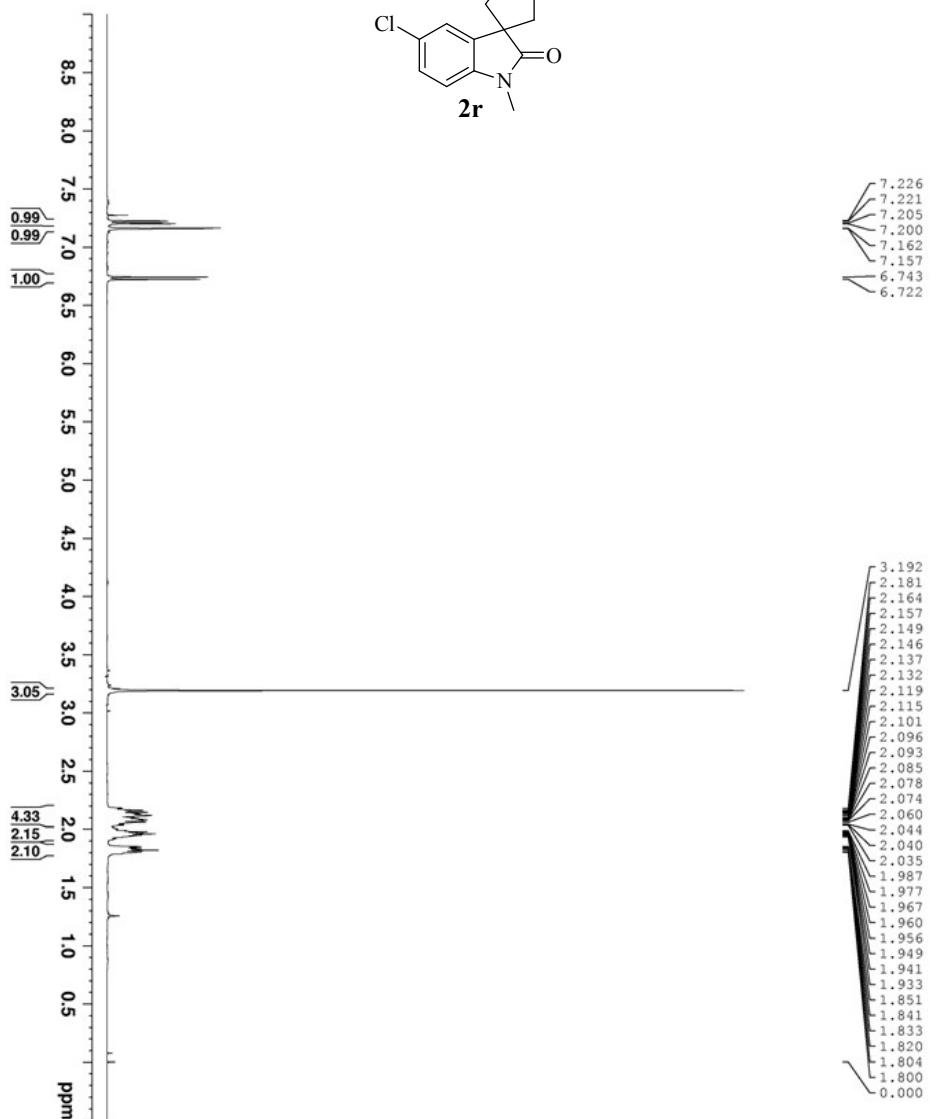
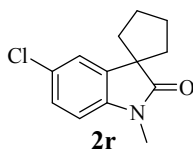
NAME      chem31-4-p38-4
EXPNO    1
PROCNO   1
Date_    20150907
Time     14.14
INSTRUM  spect
PROBHD   5 mm EBMQ HBT
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
AQ        0.122614
RG        4.094986
AQ1       42.400
AQ2       42.400
AQ3       42.400
TE        -1.9629
D1        1.00000000
D10       1
===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1     13
P1       10.78
SI       65536
SF       400.1299998 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



```

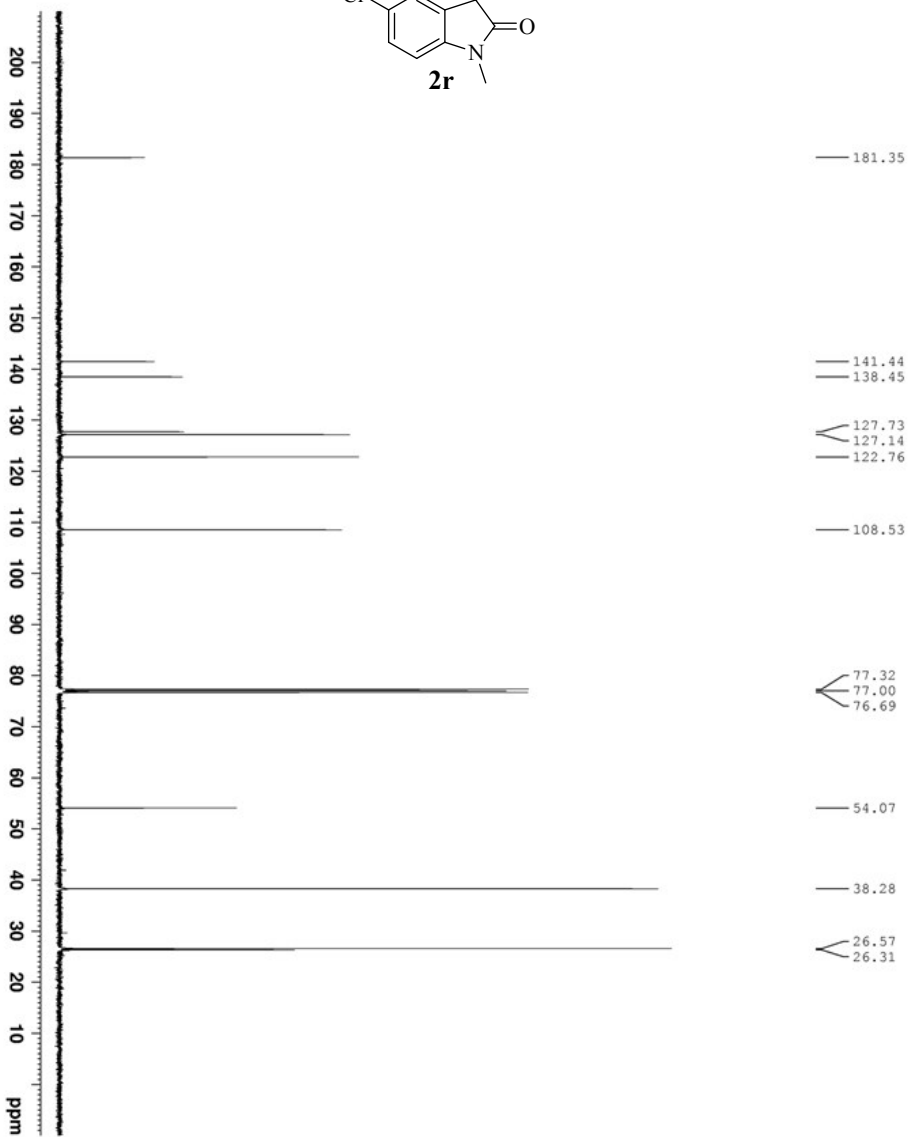
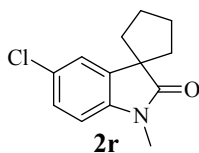
NAME          chen7q-4-p38-4
EXPNO         1
PROCNO        1
Date_         20150907
Time         14.44
PROBHD        5 mm PABBO HD/
PULPROG       zgpg30
TO            65536
SOLVENT       CDCl3
NS            2
DS            2
F2            24038.461 Hz
AQ            0.146198 sec
RG            1.46198 sec
BO            2050
DM            20.800 usec
DE            -23.50 usec
TE            300.2
D1            2.00000000 sec
D11           0.03000000 sec
D10           1
===== CHANNEL f1 =====
SFO1          100.6282298 MHz
NUC1          13C
P1            12.00 usec
SI            32768
SF            100.6276930 MHz
WIDOW         EX
SFO2          100.6276930 MHz
L3           1.00 Hz
GB            0
PC            1.40
  
```


5'-chloro-1'-methylspiro[cyclopentane-1,3'-indolin]-2'-one (**2r**)



```

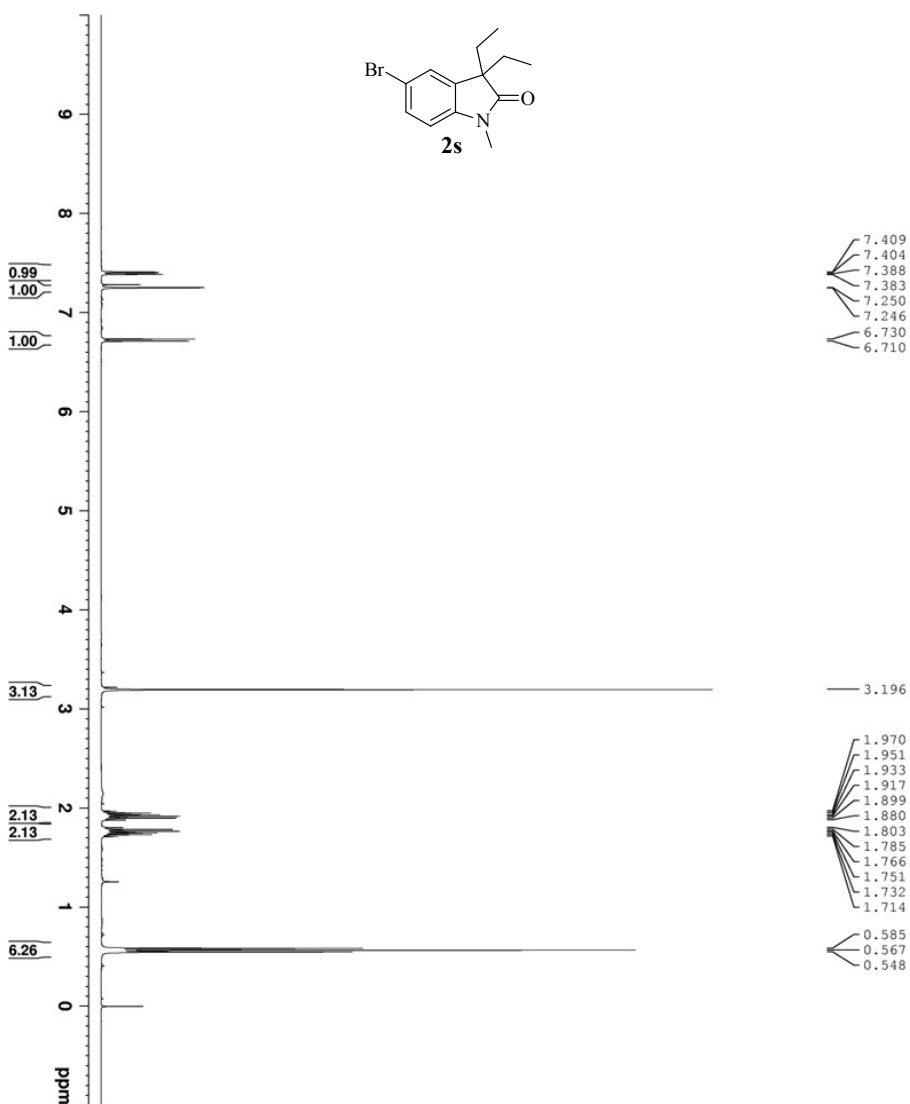
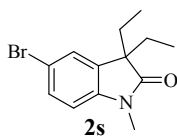
NAME: chem3r-4-p38-5-2
EXPNO: 10
PROCNO: 1
DATE_: 20150911
TIME: 11.11
INSTRUM: spect
PROBHD: 5 mm PABBO 90/
PULPROG: zgpg30
SOLVENT: CDCl3
NS: 8
DS: 2
AQ: 0.122266 Hz
RG: 62 114
FIDRES: 4.0893966 sec
AQ: 62 114
DE: 64.50 usec
TE: 294.4 K
D1: 1.0000000 sec
D10: 1
===== CHANNEL f1 =====
SFO1: 400.1324710 MHz
NUC1: 13
P1: 10.00 usec
SI: 65536
SF: 400.1300042 MHz
MNM: EM
MS: 40
LB: 0.30 Hz
GB: 0
FC: 1.00
    
```



```

NAME      chem3r-4-p38-5-2-C
EXPNO    1
PROCNO   1
Date_    20150911
Time     21.48
PROBHD   5 mm PABBO BB7
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
DS        2
SWH       24038.461 Hz
FIDRES    0.245928 Hz
AQ         1.469198 sec
RG         2050
DW         20.800 usec
DE         8.50 usec
TE         300.2 K
D1         2.00000000 sec
D11        0.03000000 sec
D12        1
D13        1
===== CHANNEL f1 =====
SFO1     100.628298 MHz
NUC1      13C
P1        12.00 usec
PL1       0 dB
SI         32768
SF        100.627736 MHz
KICK      EX
KICW      EX
LS         1.00 Hz
LN         0
GB         1.40
PC
  
```

5-bromo-3,3-diethyl-1-methylindolin-2-one (2s)



7.409
7.404
7.388
7.383
7.250
7.246
6.730
6.710

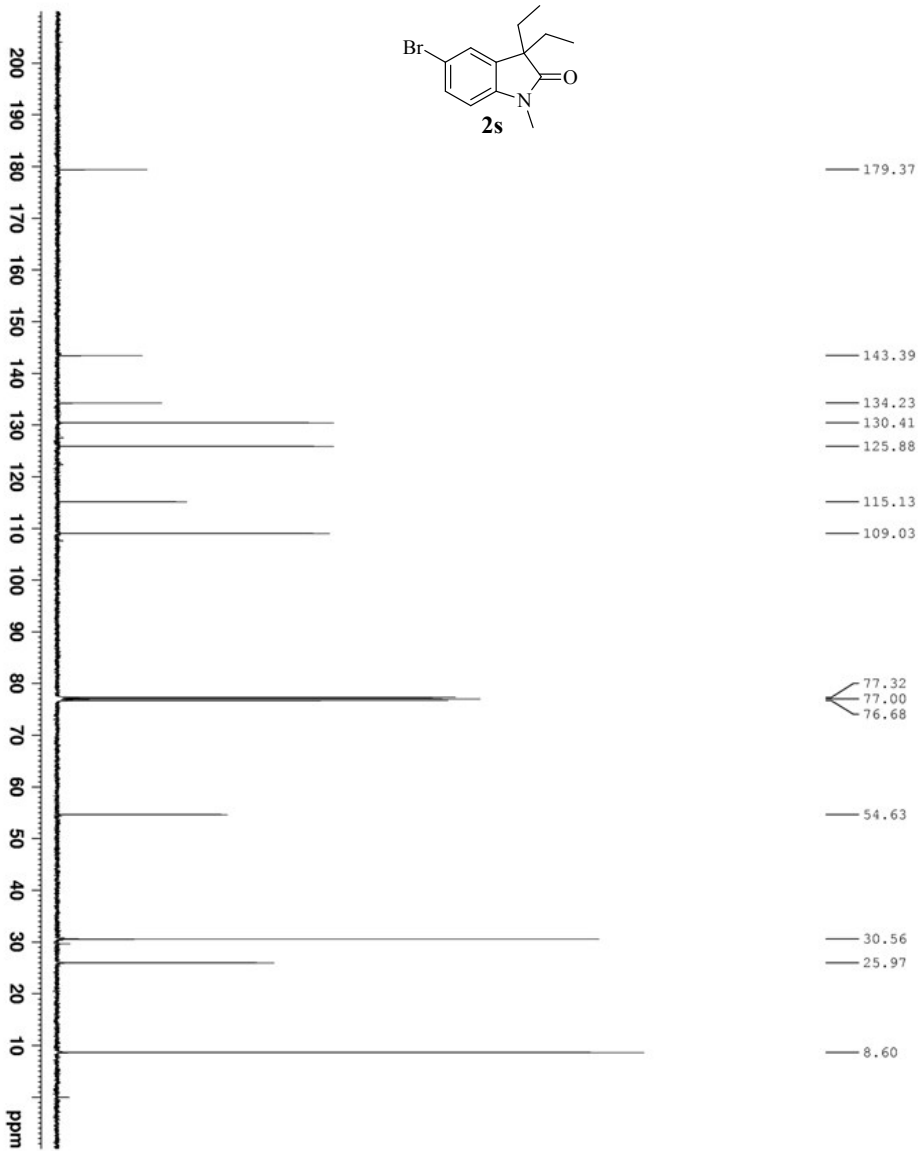
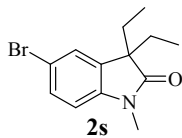
3.196

1.970
1.951
1.933
1.917
1.899
1.880
1.803
1.785
1.766
1.751
1.732
1.714

0.585
0.567
0.548

```

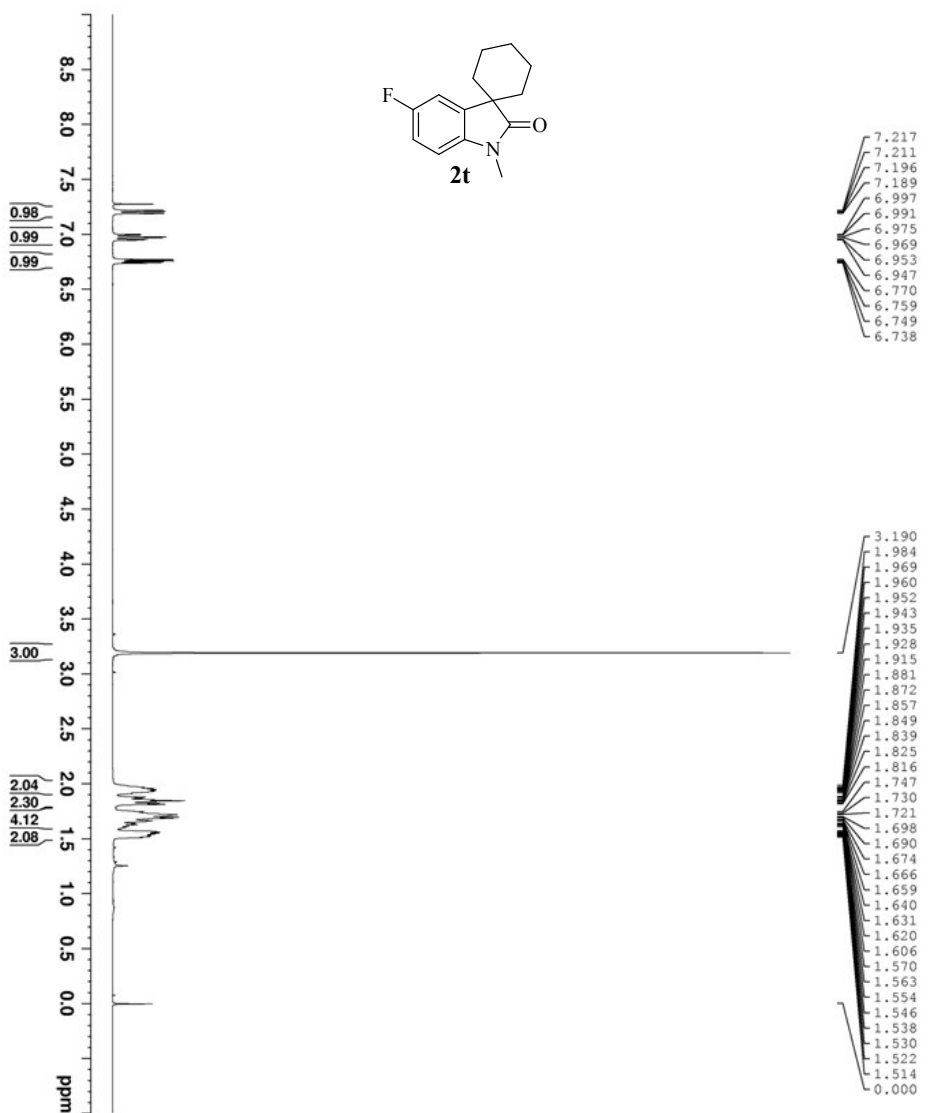
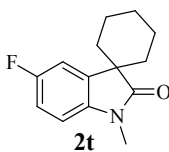
NAME      chemJg-4-p39-2
EXPNO    1
PROCNO   1
Date_    20150917
Time     11.56
INSTRUM  spect
PROBHD   5 mm PABBO SBI/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      8012.820 Hz
AQ       0.122440 sec
RG        409
AQ       4.089966 sec
RG        101
RG        62.400 usec
TE       294.4 K
D1       1.00000000 sec
D10      1
===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1     13
P1       10.00 usec
SI       65536
SF       400.1300019 MHz
WIDOW   EX
SFO2     0.30 Hz
GB       0
PC       1.00
    
```



```

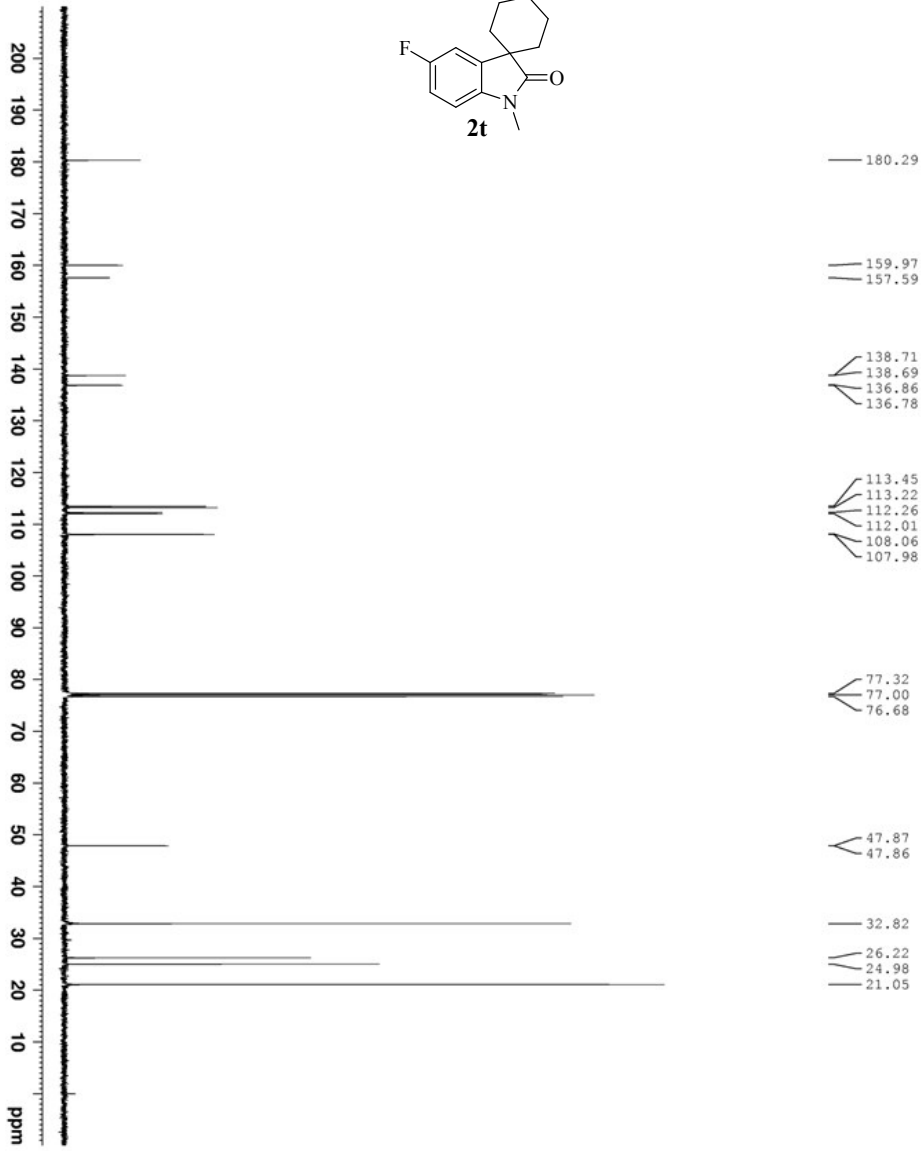
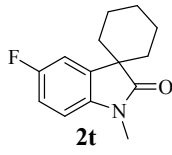
NAME: CHEN39-4-33-2
EXPNO: 11
PROCNO: 1
DATE_: 20150917
TIME: 11:42
INSTRUM: spect
PROBHD: 5 mm PABBO BBO
PULPROG: zgpg30
PCPDPR: 4
SOLVENT: CDCl3
NS: 400
DS: 4
SWH: 24038.441 Hz
F2: 0.347793 Hz
AQ: 1.5613988 sec
RG: 912
DM: 20.800 uS/RC
DE: 295.4 K
TE: 2.00000000 sec
D1: 0.03000000 sec
D11: 1
D12: 1
D13: 1
===== CHANNEL f1 =====
SFO1: 100.6282298 MHz
NUC1: 13C
P1: 14.00 uS/RC
SI: 32768
SF: 100.6127736 MHz
MVM: EX
LB: 0
GB: 0
PC: 1.40
  
```

5'-fluoro-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (**2t**)



```

NAME          chem3f-4-p10-4
EXPNO         10
PROCNO        1
Date_         20150923
Time          11.11
INSTRUM       spect
PROBHD        5 mm PABBO BH/
PULPROG       zgpg30
AQ            6.2930
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.822 Hz
FIDRES       0.1222266 Hz
AQ           4.0894966 sec
RG            114
AQ           62.430 uSAC
DS            4
TE            294.2 K
D1            1.00000000 sec
D11           1
===== CHANNEL f1 =====
SFO1          400.1324710 MHz
NUC1          13C
P1            10.70 uSAC
SI            65536
SF            400.1300038 MHz
MVM          EX
NUC2          13C
P2            0.30 Hz
LA            0
GB            1.00
PC            1.00
    
```

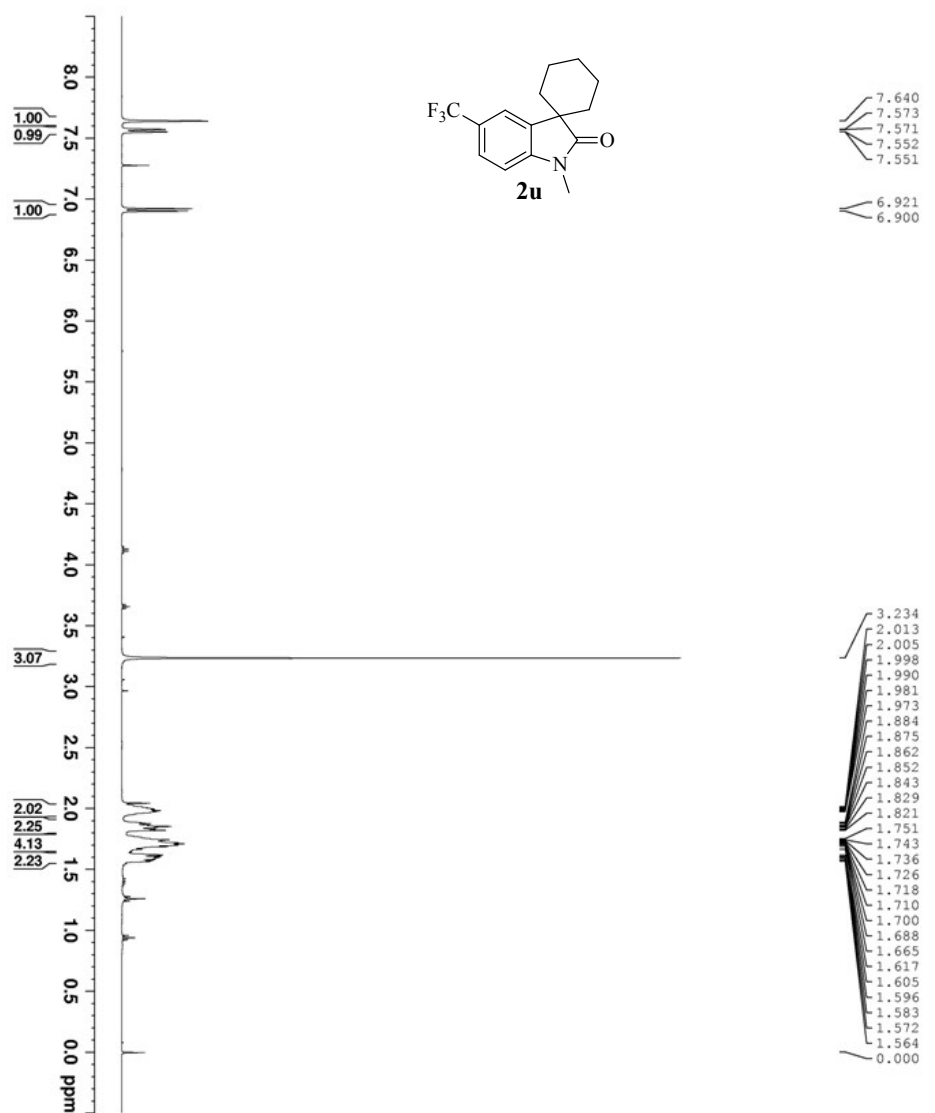


```

NAME      chem3g-4-pd-4
EXPNO    11
PROCNO   1
Date_    20150923
Time     17.17
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
SOLVENT  CDCl3
NS       350
DS       2
F2 -     24038.461 Hz
F3 -     0.366796 Hz
F4 -     1.3631968 sec
AQ       2050
RG       20.800 USRC
DE       4.000 USRC
TE       295.4 K
D1       2.00000000 sec
D11      0.03000000 sec
D10      1
===== CHANNEL f1 =====
SFO1     100.628228 MHz
NUC1     13C
P1       12.00 usec
SI       32768
SF       100.627736 MHz
RG       20.800
IC       EX
MS       1.00 Hz
LB       0
GB       0
PC       1.40

```

1'-methyl-5'-(trifluoromethyl)spiro[cyclohexane-1,3'-indolin]-2'-one (**2u**)

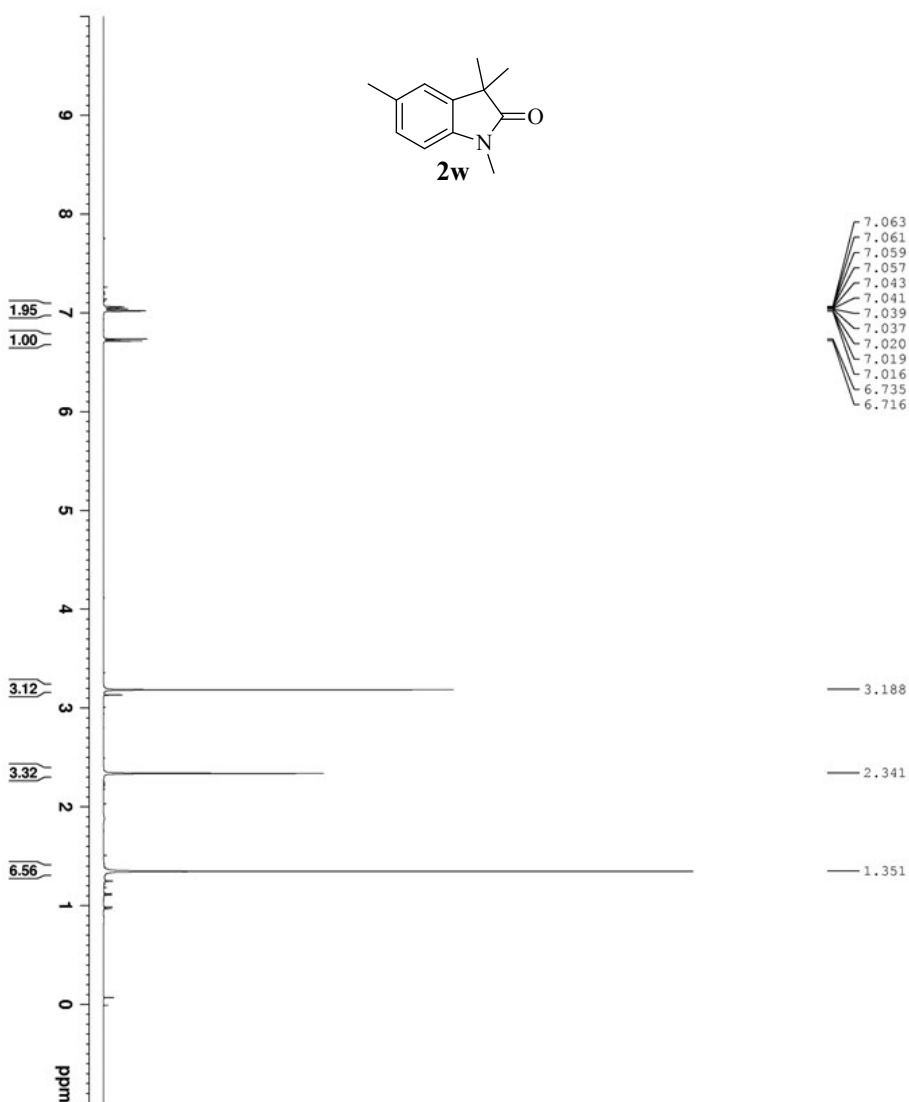
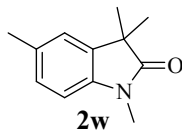


```

NAME          chm03f-4-p10-2
EXPNO        12
PROCNO       1
Date_         20150919
Time         21.29
INSTRUM      spect
PROBHD       5 mm PABBO BB/
PULPROG      zg30
TD           65536
SFO          400.1324710
F2           400.1300030
AQ           4.0894986
RG           90.5
DM           62.400
DE           234.4
TE           300.2
D1           1.00000000
D10          1

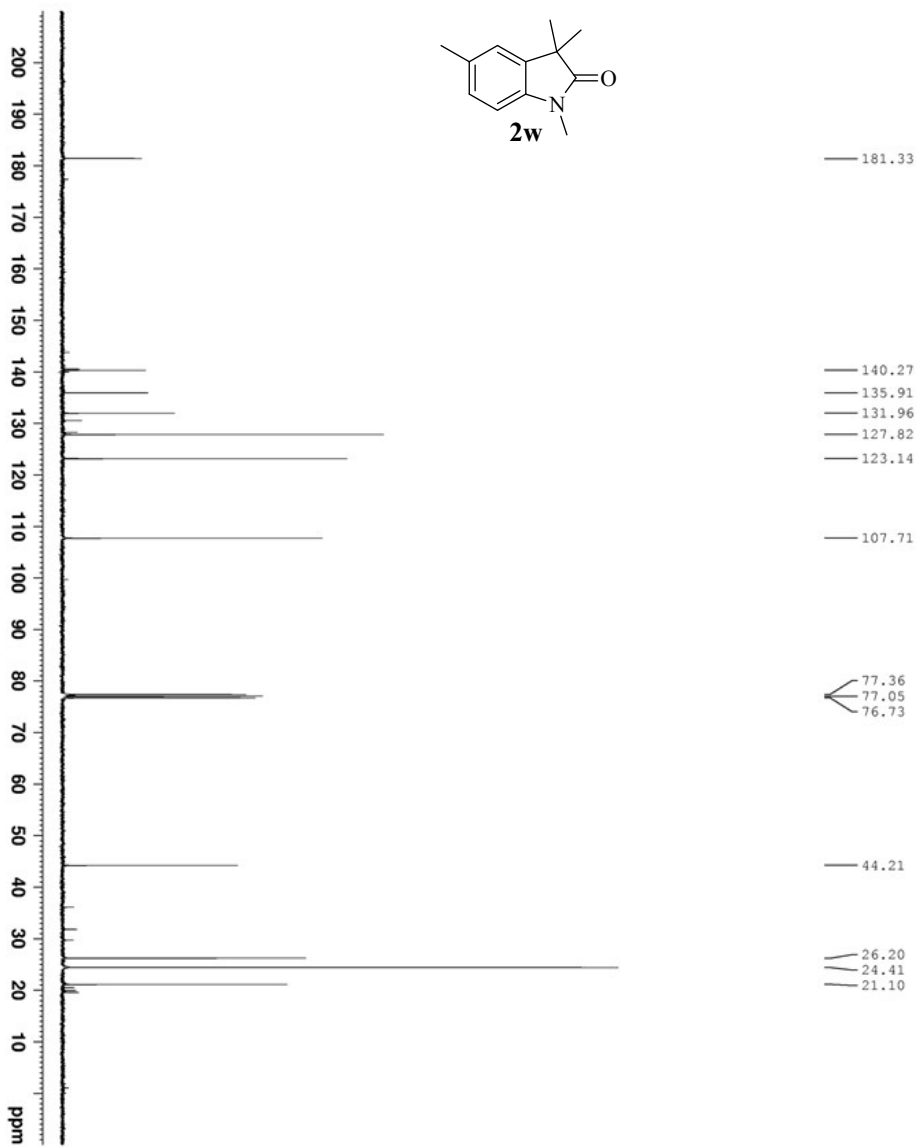
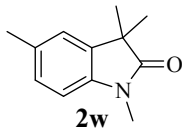
===== CHANNEL f1 =====
SFO1         400.1324710 MHz
NUC1         13
SI           10.78 usec
SF           400.1300030 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
  
```


1,3,3,5-tetramethylindolin-2-one (**2w**)



```

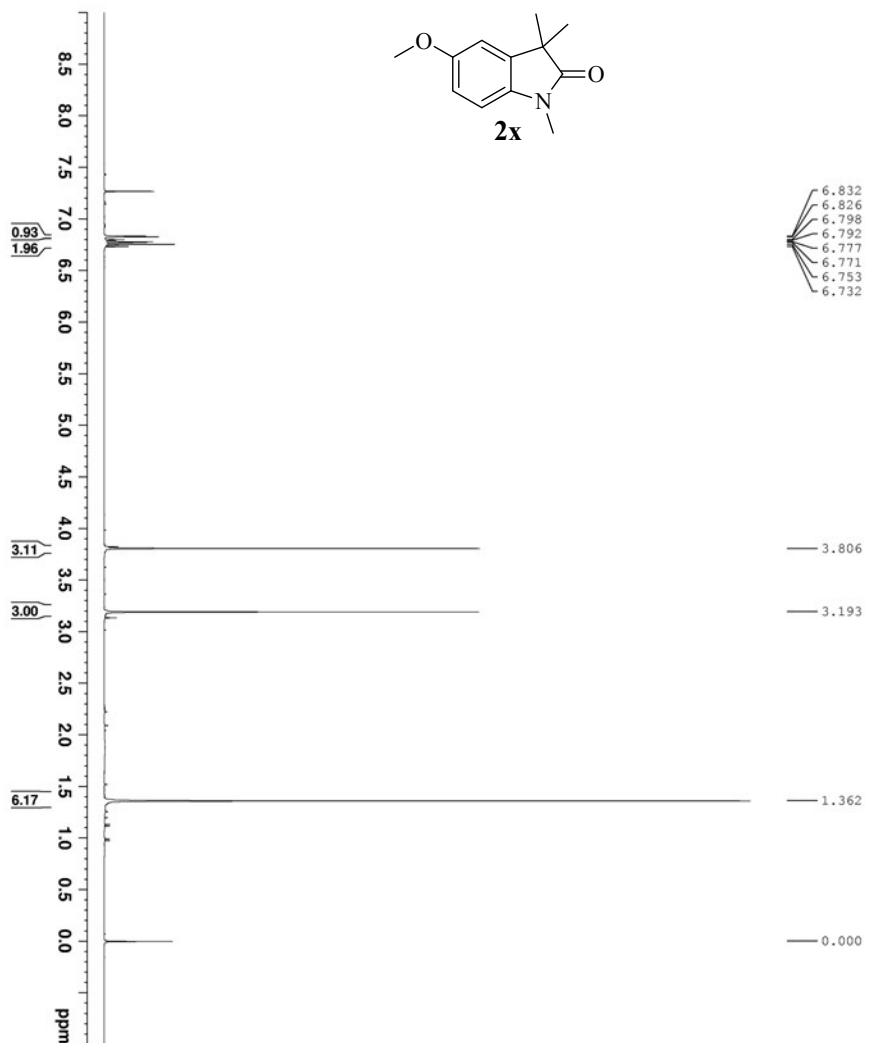
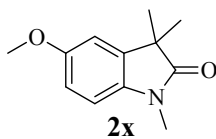
NAME: chen1g-4-p37-2
PROCNO: 1
Date_: 20150901
Time: 1.33
INSTRUM: spect
PROBHD: 5 mm PABBO 13B/
PULPROG: zg30
TD: 65536
SOLVENT: CDCl3
NS: 16
DS: 2
AQ: 0.12420000 sec
FIDRES: 0.12420000 Hz
AQ: 62.400 usec
RG: 101
INTEGR: 4.089996E sec
TE: 296.3 K
D1: 1.00000000 sec
===== CHANNEL f1 =====
SFO1: 400.1324710 MHz
NUC1: 13
P1: 10.00 usec
PT: 1.00 usec
SF: 400.1300095 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00
    
```



```

NAME: chm134-4-p37-2
EXPNO: 11
PROCNO: 1
Date_ : 20150901
Time: 12:42
INSTRUM: spect
PROBHD: 5 mm PABBO AB/
PULPROG: zgpg30
D0: 0.012
SFO: 125.761
NS: 500
DS: 2
SWH: 24038.463 Hz
AQ: 0.00000000 sec
RG: 1.3631368 sec
DM: 20.800 usec
TE: 297.4 K
D1: 2.00000000 sec
D11: 0.03000000 sec
D12: 1
D13: 1
===== CHANNEL f1 =====
SF01: 100.6282295 MHz
NUC1: 13C
PUL1: 14.00 usec
SI: 32768
SF: 100.627690 MHz
WGM: EX
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40
  
```

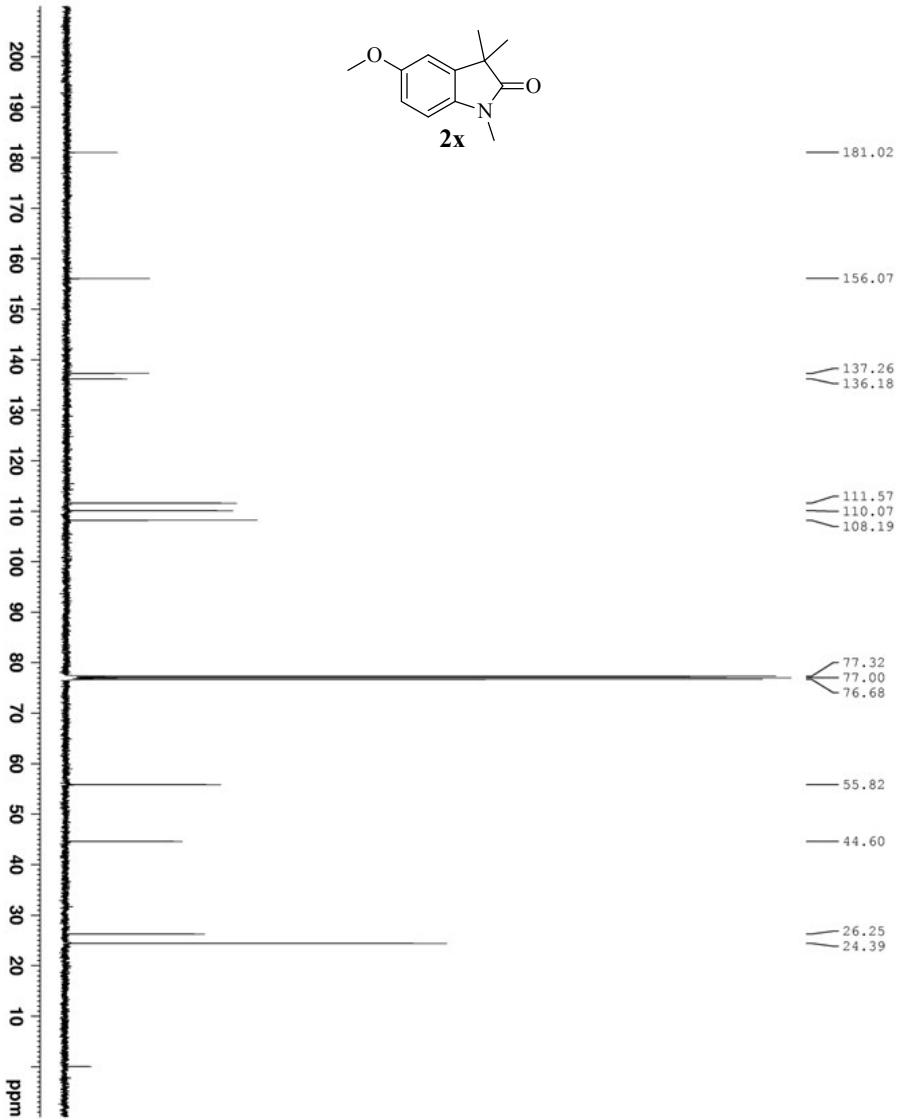
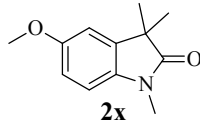
5-methoxy-1,3,3-trimethylindolin-2-one (**2x**)



```

NAME: chem34-4-p48
PROCNO: 1
DATE_: 20191024
INSTRUM: spect
PROBHD: 5 mm PABBO BBI/
PULPROG: zgpg30
SOLVENT: CDCl3
NS: 16
DS: 4
AQ: 0.122286 Hz
FIDRES: 4.0894916 sec
AQ: 4.0894916 sec
DE: 62.400 usec
TE: 300.2 K
D1: 6.50 usec
D11: 1.00000010 sec
TD0: 1

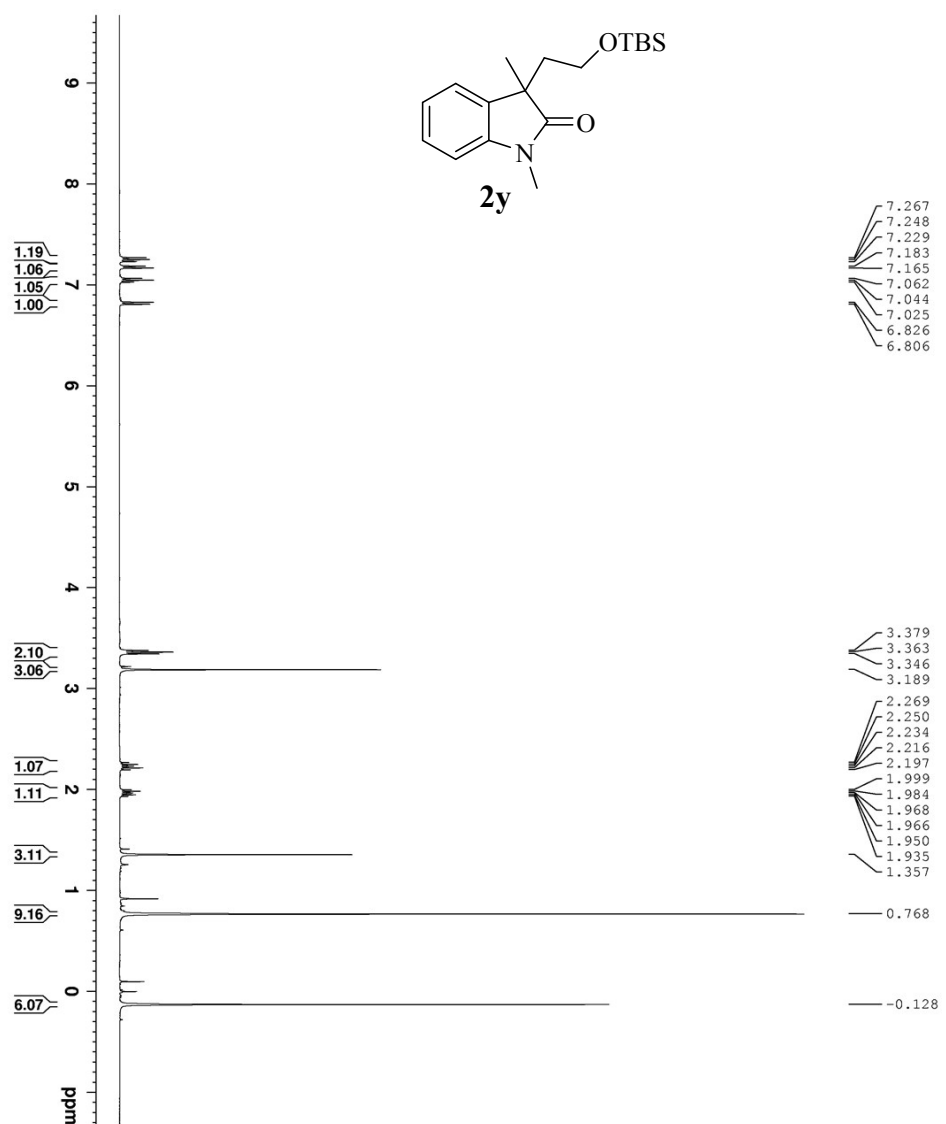
===== CHANNEL f1 =====
NUC1: 13C
P1: 1.00
SFO1: 400.1324710 MHz
SI: 10.24 usec
SF: 65536
SR: 400.1300070 MHz
K1: 20
L1: 0.30 Hz
GB: 0
PC: 1.00
    
```



```

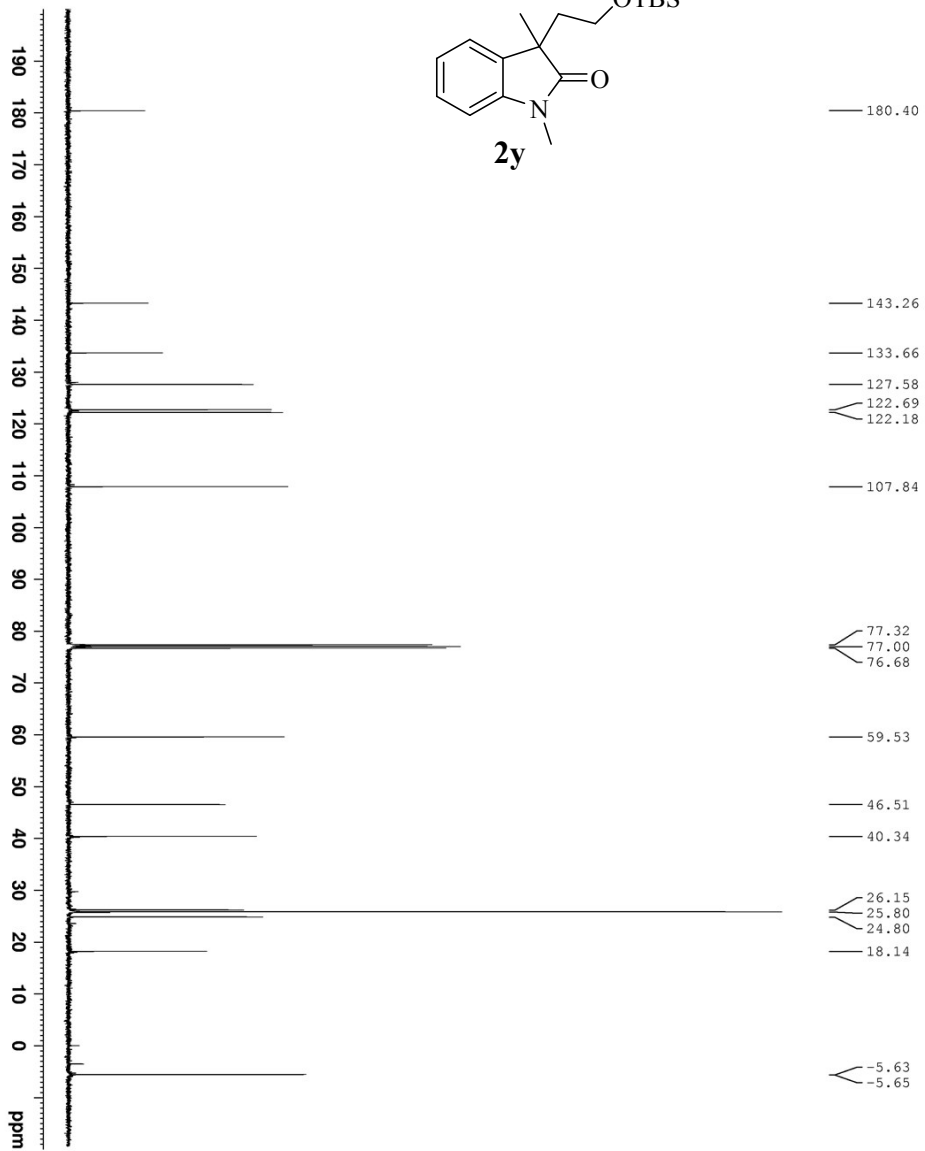
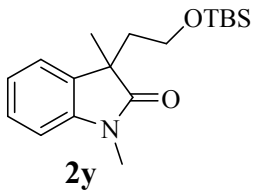
NAME          Chem[3]-1-P48
EXPNO         11
PROCNO        2011024
Date_         17-45
Time          17-45
INSTRUM       5 mm BBOHD 900
PROBHD        BBO
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
DS            397
DE            24038.461 Hz
SMH           0.366798 Hz
FIDRES        1.3611030
AQ            0.720
RG            20.800 usec
DE            6.50 usec
TE            300.2 K
D1            2.0000000 sec
D11           0.03000000 sec
D10           1
===== CHANNEL f1 =====
SFO1          100.628298 MHz
NUC1          13C
P1            1.30 usec
SFO2          100.628298 MHz
SFO3          100.628298 MHz
SF            100.628298 MHz
WDW           EM
SSB           0
GB            0
PC            1.40
  
```

3-(2-((tert-butyldimethylsilyl)oxy)ethyl)-1,3-dimethylindolin-2-one (**2y**)



```

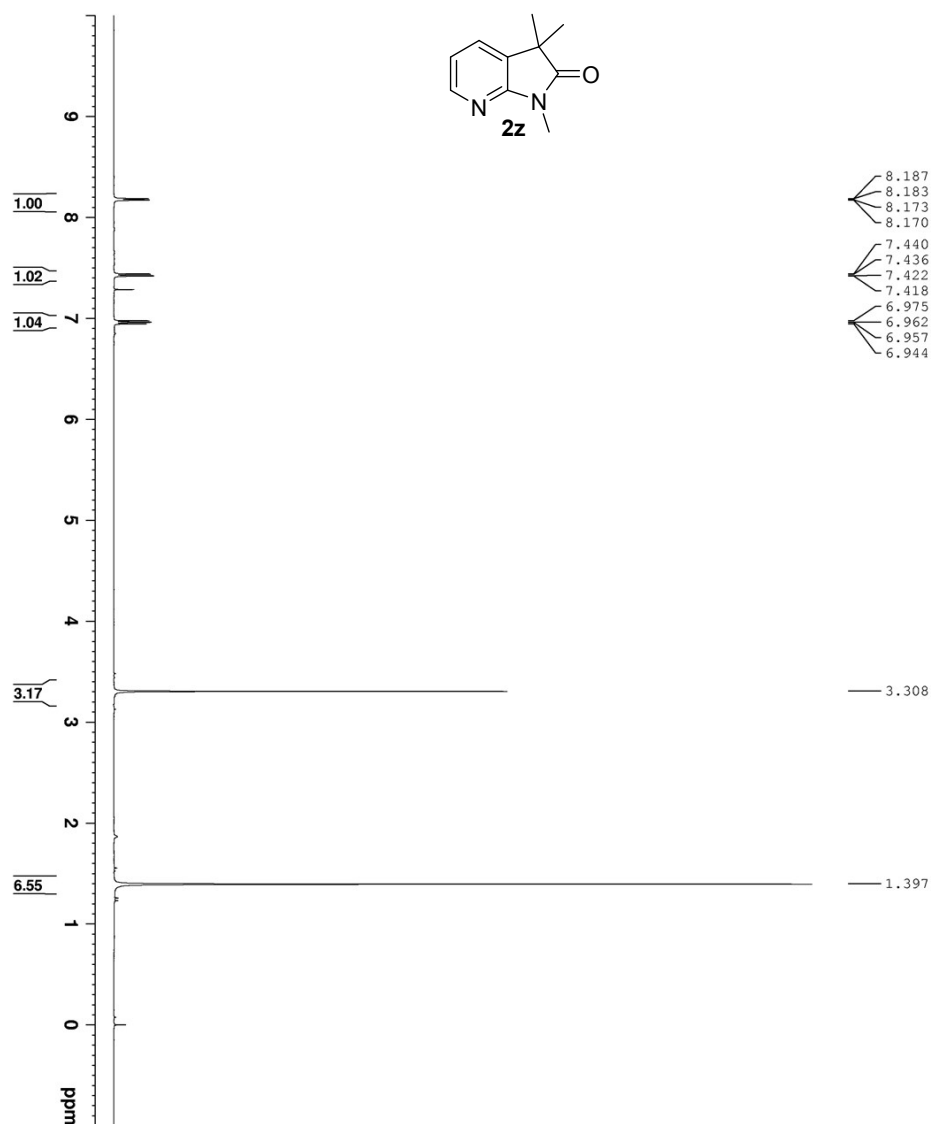
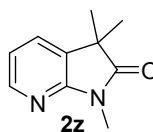
NAME          chemjg-4-p59
EXPNO         1
PROCNO        1
Date_         20160107
Time          11.15
PROBHD        5 mm PABBO BB7
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            1
DS            2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.092466 sec
RG            80.6
DM            62.400 usec
DE            9.50 usec
TE            29.50 usec
D1            1.00000000 sec
TD0           1
===== CHANNEL f1 =====
SFO1          400.1324710 MHz
NUC1          1H
P1            16.79 usec
SF            400.1300069 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



```

NAME: chen13-4-p89
EXPNO: 1
PROCNO: 1
Date_=: 2016107
Time: 11.39
INSTRUM: spect
PROBHD: 5 mm PABBO
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
DS: 402
AS:
SM: 24038.461 Hz
FIDRES: 0.366798 Hz
AQ: 1.363198 sec
RG: 140
DE: 20.800 usec
TE: 298.0 K
D1: 2.0000000 sec
D11: 0.03000000 sec
D10: 1
===== CHANNEL f1 =====
SFO1: 100.628298 MHz
NUC1: 13C
P1: 12.00 usec
SI: 32768
SF: 100.612774 MHz
WDW: EM
SSB: 0
GB: 1.00 Hz
PC: 1.40
  
```

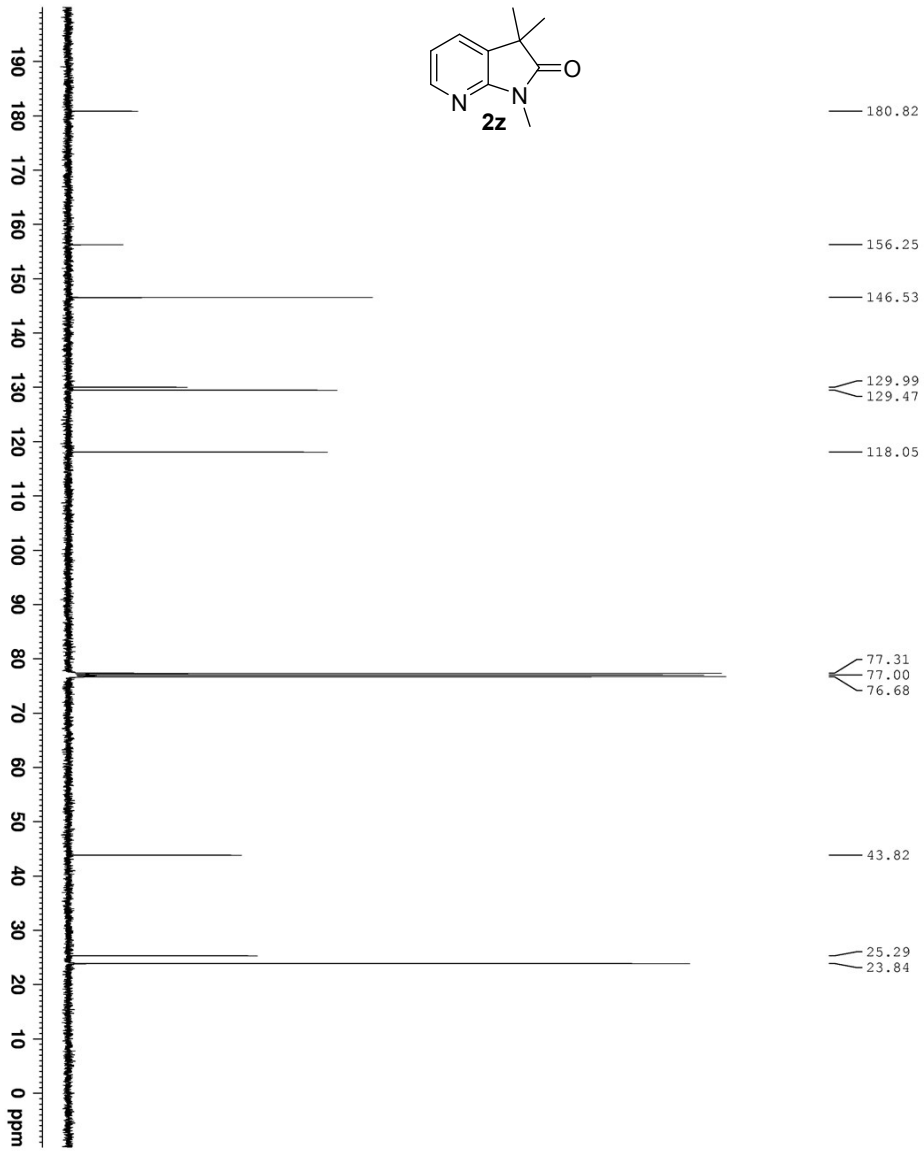
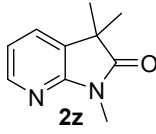

1,3,3-trimethyl-1,3-dihydro-2H-pyrrolo[2,3-b]pyridin-2-one (**2z**)



```

NAME          chen3g-6-p23
EXPNO         1
PROCNO        1
Date_         20160401
Time          11.30
INSTRUM       5 mm PABBO BB7
PROBHD        zg30
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1
DS            2
SWH           8012.820 Hz
FIDRES       0.122266 Hz
AQ           4.089161 sec
RG            161
DM           62.400 usec
DE           4.50 usec
TE           29.50 usec
D1           1.00000000 sec
TD0           1

===== CHANNEL f1 =====
SFO1          400.1324710 MHz
NUC1          1H
P1            10.92 usec
SP           400.1300000 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
    
```

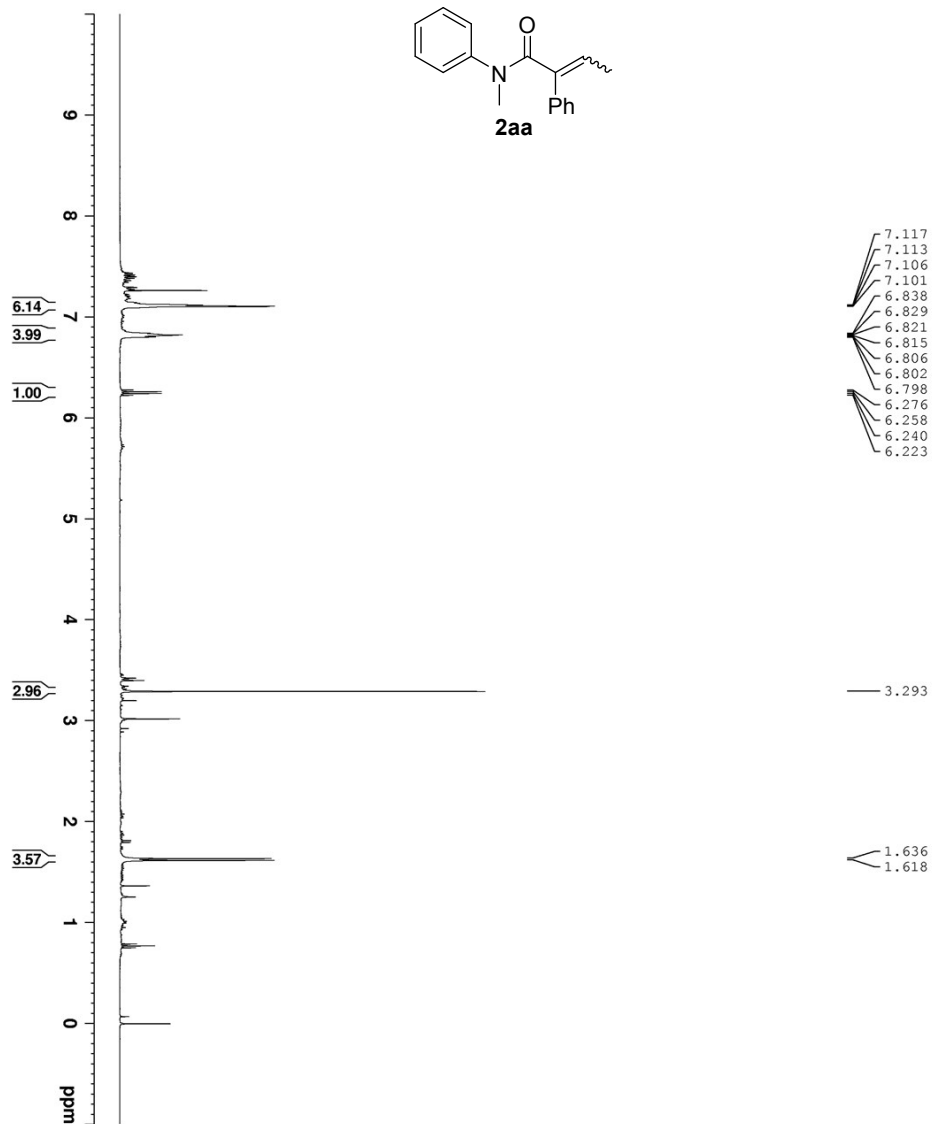
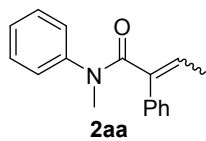


```

NAME          chem3f-6-p23-c
EXPNO         10
PROCNO        1
Date_         20160401
Time         13.26
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            6250
DS            2
SWH           24038.461 Hz
AQ            0.2475713 sec
RG            1.3631968 sec
RG            2050
DM            20.800 usec
DE            29.50 usec
TE            29.44 K
D1            2.00000000 sec
D11           0.03000000 sec
ID0           1

===== CHANNEL f1 =====
SF01          100.628296 MHz
NUC01         13C
P1            1.30 usec
SI            32768
SF            100.627722 MHz
WDW           EM
SSB           0
GB            0
PC            1.40
  
```

N-methyl-N,2-diphenylbut-2-enamide (**2aa**)

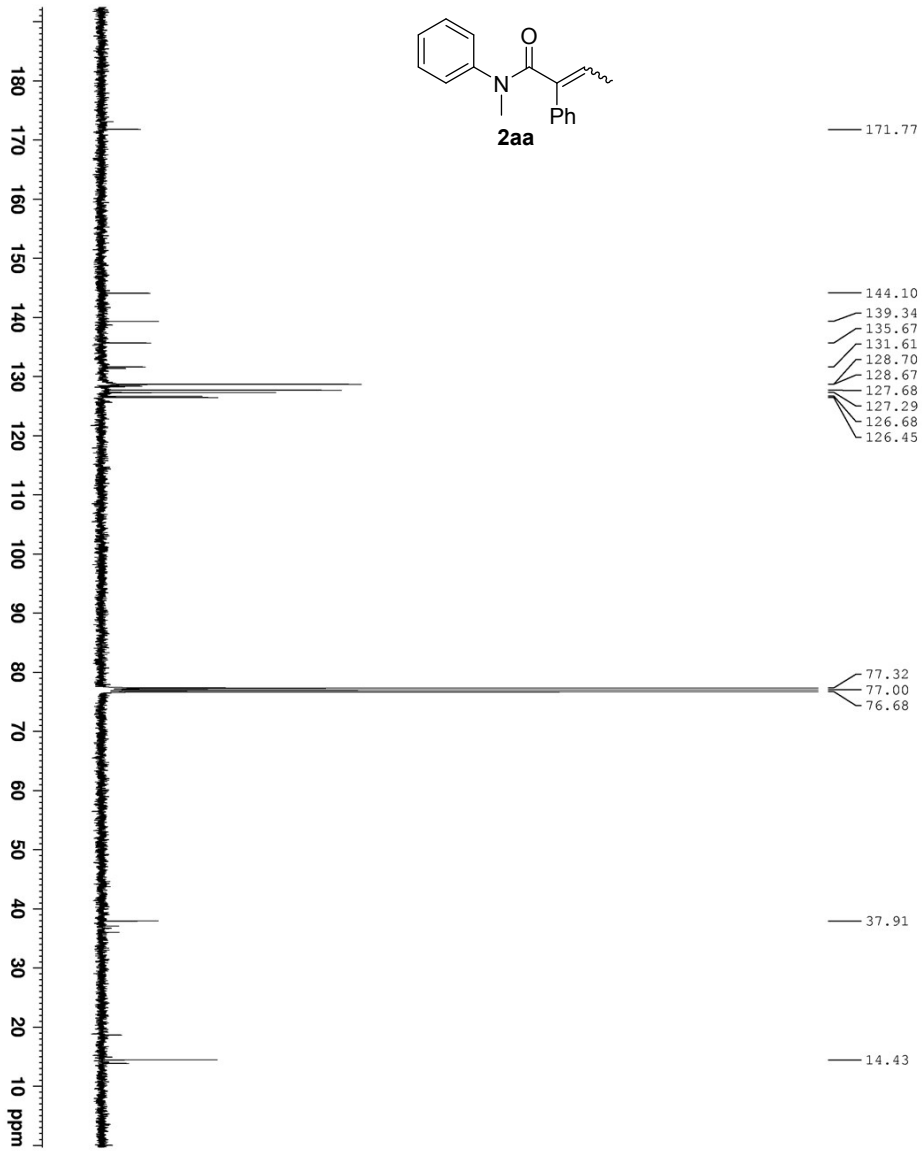
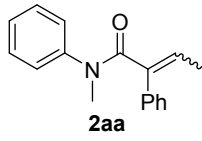


```

===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 10.79 usec
SP 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

=====
NAME chemj9-4-p34-3-k1a
EXPNO 2
PROCNO 1
Date_ 20150825
Time 16.52
PROBHD 5 mm PABBO BB7
PULPROG zg30
TD 65536
SOLVENT CHCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.059287 sec
RG 62.400 usec
DE 9.50 usec
TE 29.50
D1 1.00000000 sec
TDO 1
=====

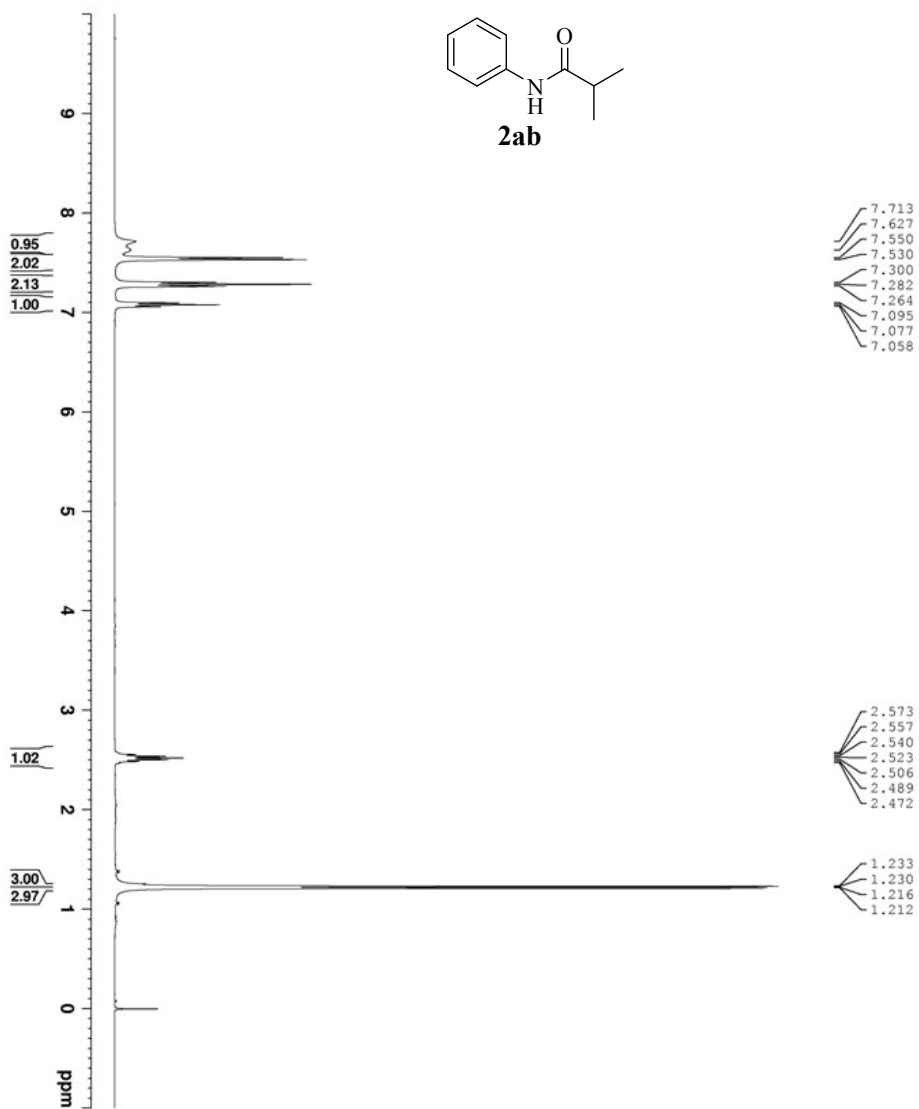
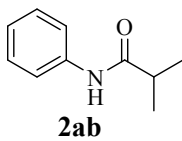
```



```

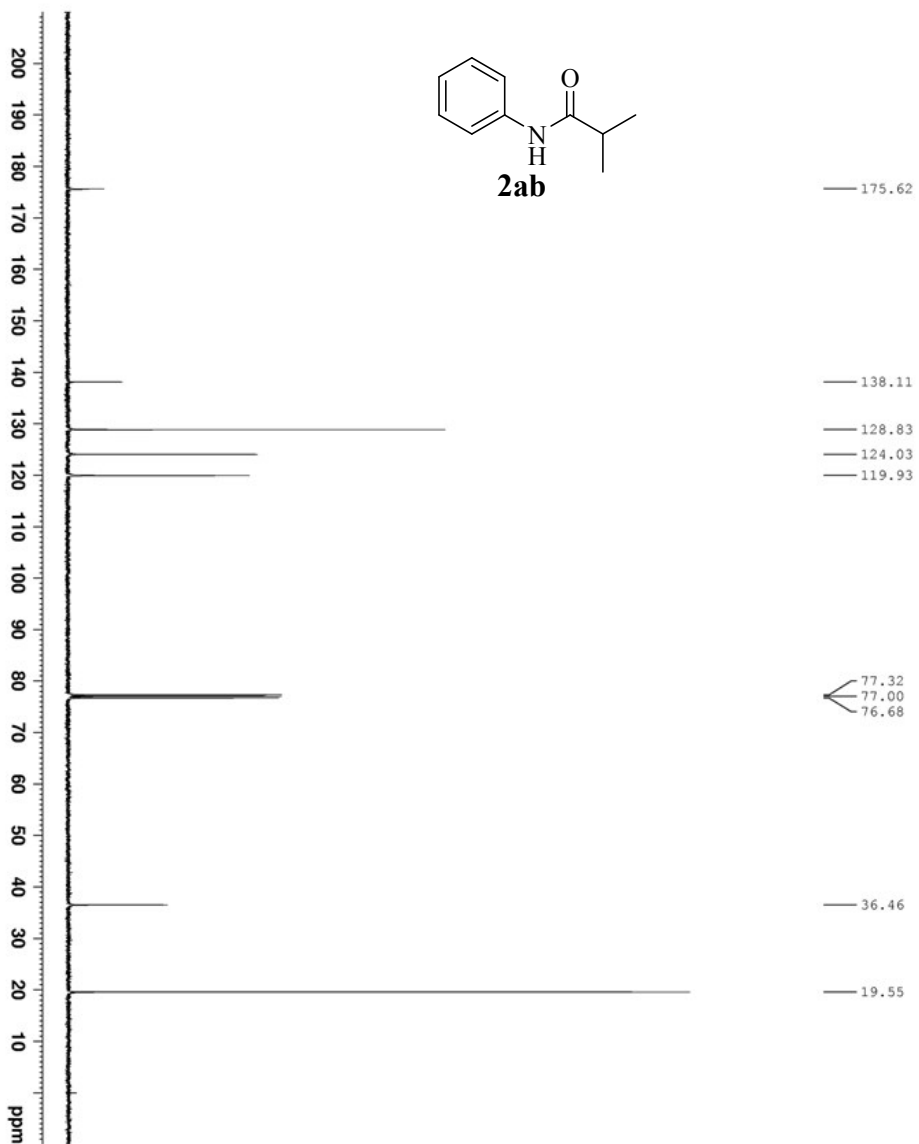
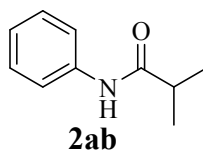
NAME: chenj4-4-p34-3-k1a
EXPNO: 30
PROCNO: 1
Date_=: 20150825
Time: 17.33
INSTRUM: spect
PROBHD: 5 mm PABBO
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
DS: 702
AQ: 24038.461 Hz
FIDRES: 0.366798 Hz
AQ: 1.363398 sec
RG: 240
DE: 20.800 usec
DE: 6.50 usec
TE: 300.4 K
D1: 2.0000000 sec
D11: 0.03000000 sec
TD0: 1
===== CHANNEL f1 =====
SFO1: 100.628238 MHz
NUC1: 13C
P1: 12.00 usec
SI: 32768
SF: 100.612189 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40
  
```

N-phenylisobutyramide (**2ab**)



```

NAME: chem31-4-p44-3
EXPNO: 1
PROCNO: 1
Date_: 20151008
Time: 11.23
INSTRUM: spect
PROBHD: 5 mm EBMQ HBT
PULPROG: zg30
TD: 65536
SOLVENT: CDCl3
NS: 16
DS: 2
SWH: 8012.820 Hz
AQ: 0.1122417 sec
RG: 409.5
AQ2: 4.089496 sec
DM: 62.400 usec
DE: 4.500 usec
TE: 293.2 K
D1: 1.00000000 sec
D11: 1
===== CHANNEL f1 =====
SFO1: 400.1324710 MHz
NUC1: 1H
P1: 10.00 usec
SI: 65536
SF: 400.1300112 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00
    
```

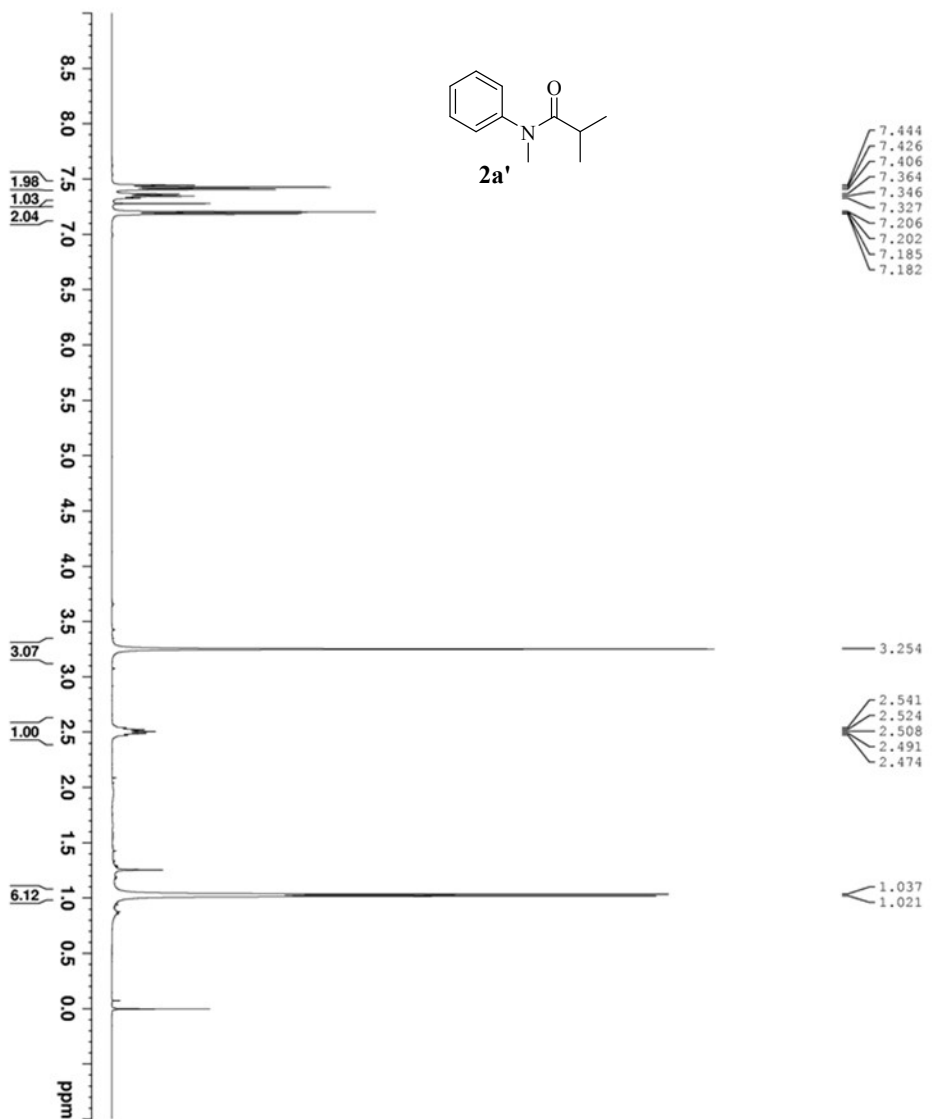


```

NAME          Chem14-4-P44-3
EXPNO         11
PROCNO        1
Date_         20151008
Time         11.21
INSTRUM       spect
PROBHD        5 mm PABD BBI/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS           300
DS            2
SWH           24038.461 Hz
FIDRES        0.344 Hz
AQ            1.361398 sec
RG            812
DM           20.800 usec
DE           294.00 usec
TE           294.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1

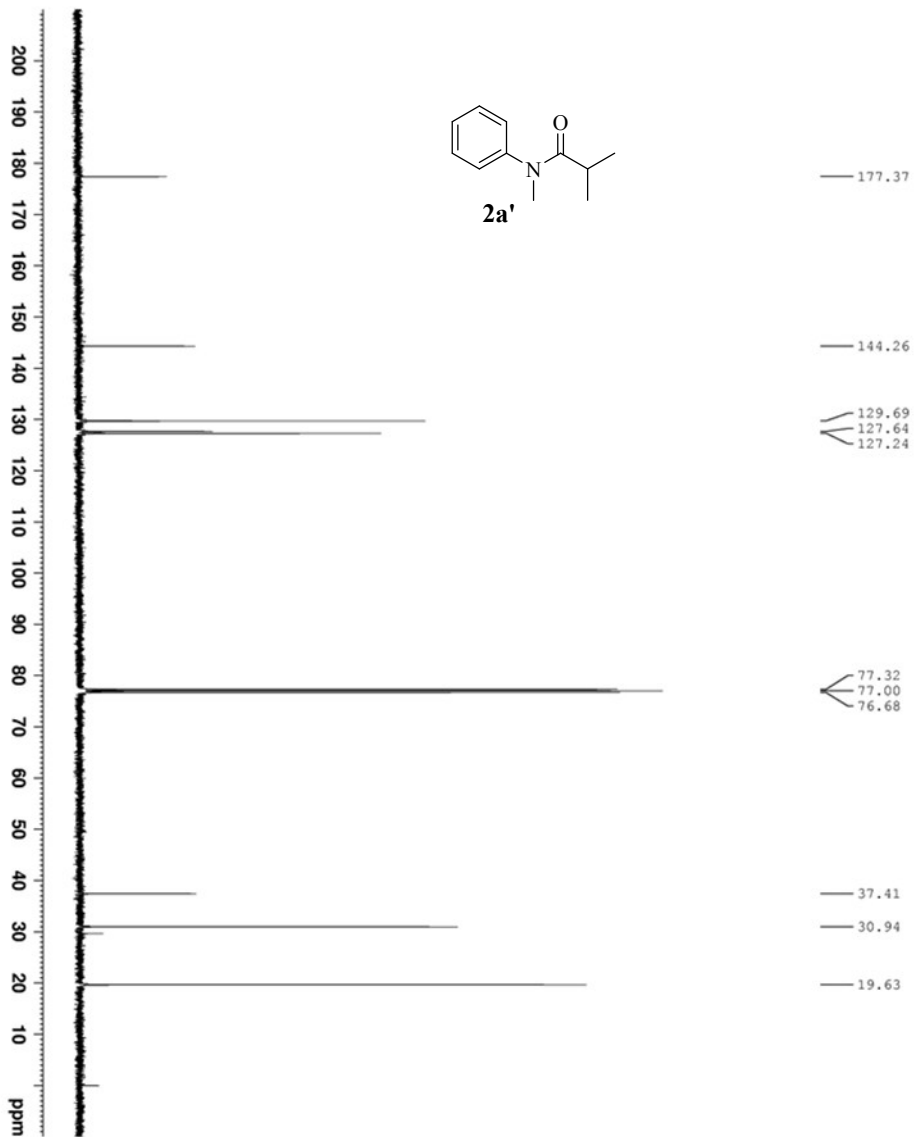
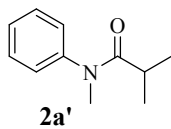
===== CHANNEL f1 =====
SFO1         100.628298 MHz
NUC1         13C
P1           14.00 usec
SI           32768
SF           100.627773 MHz
WDW          EM
SS           0
LB           1.00 Hz
GB           0
PC           1.40
  
```

N-methyl-N-phenylisobutyramide (**2a'**)



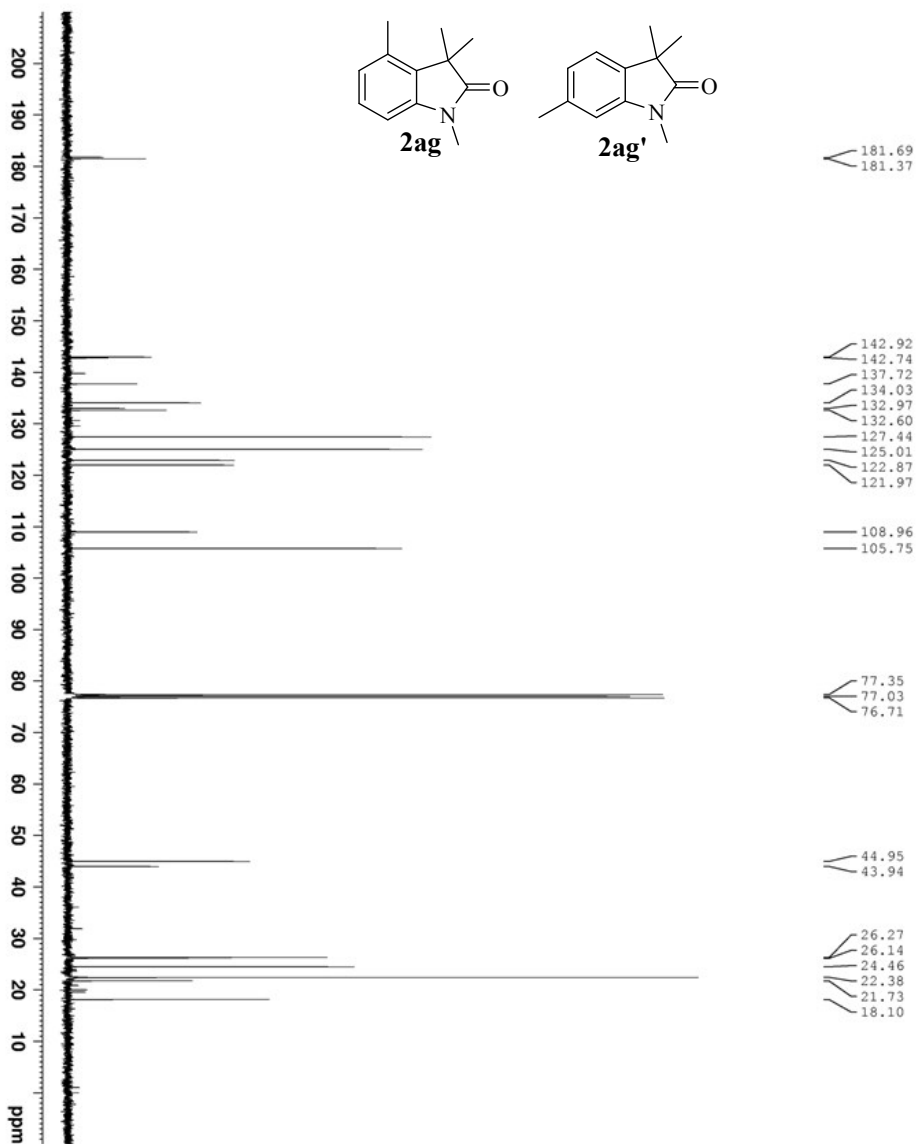
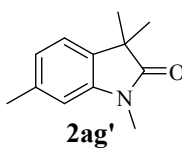
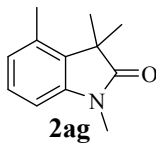
```

NAME: phenyl-4-45-5-2
EXPNO: 10
PROCNO: 1
PULPROG: zgpg30
DPRG: spect
INSTRUM: spect
PROBHD: 5 mm PABBO 1H/
PULPROG: zgpg30
SOLVENT: CDCl3
NS: 16
DS: 4
AQ: 0.122266 Hz
RG: 114
AQ: 4.0899966 sec
TE: 293.7 K
D1: 1.0000000 sec
===== CHANNEL f1 =====
SF01: 400.1324710 MHz
NUC1: 13
P1: 10.00 usec
SI: 65336
SF2: 400.1300025 MHz
NUC2: 1H
P2: 0.30 usec
SI: 0
PC: 1.00
  
```



```

NAME: chem34-4-45_1-2
PROCNO: 1
Date_: 2015109
Time: 11.06
PROBHD: 5 mm PABBO
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
DS: 2
SWH: 24038.461 HZ
FIDRES: 0.246728 HZ
AQ: 1.46912 sec
RG: 1.691912
DM: 20.880 usec
DE: 6.50 usec
TE: 29.21
D1: 2.0000000 sec
D11: 0.0900000 sec
T0: 1
===== CHANNEL f1 =====
SFO1: 100.6228238 MHz
NUC1: 13C
P1: 14.00 usec
PL1: 0.00 dB
SFO2: 100.6127236 MHz
NUC2: 13C
P2: 14.00 usec
PL2: 0.00 dB
=====
  
```

181.69
181.37

142.92
142.74
137.72
134.03
132.97
132.60
127.44
125.01
122.87
121.97

108.96
105.75

77.35
77.03
76.71

44.95
43.94

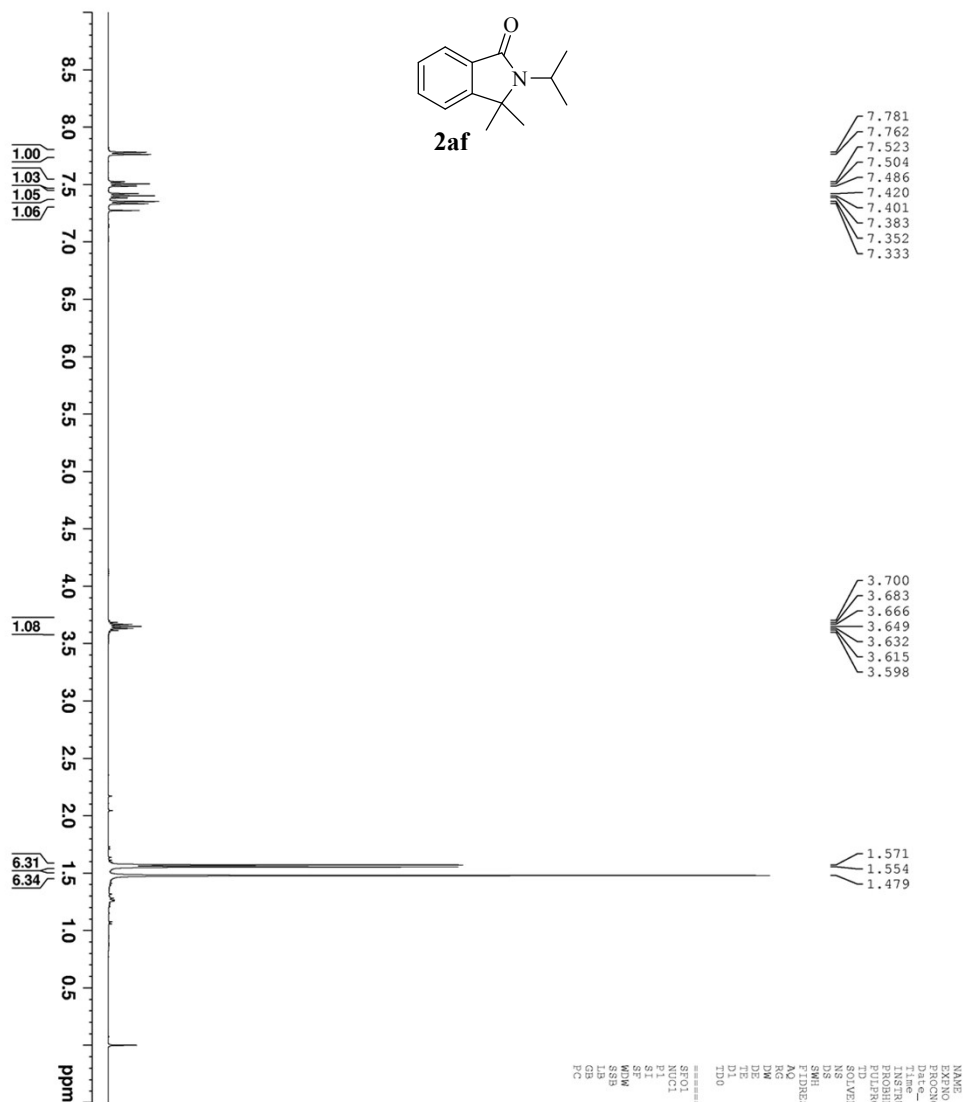
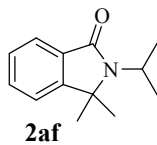
26.27
26.14
24.46
22.38
21.73
18.10

```

NAME          Chem14-4-p37-2
EXPNO         1
PROCNO        1
Date_         20150901
Time         22:27
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS           500
DS           2
SWH           24038.461 Hz
AQ           1.361368 sec
RG           1050
DM           20.800 usec
DE           297.9 K
TE           297.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1
===== CHANNEL f1 =====
SFO1          100.628298 MHz
NUC1          13C
P1           14.00 usec
SI           32768
SF           100.627690 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40

```

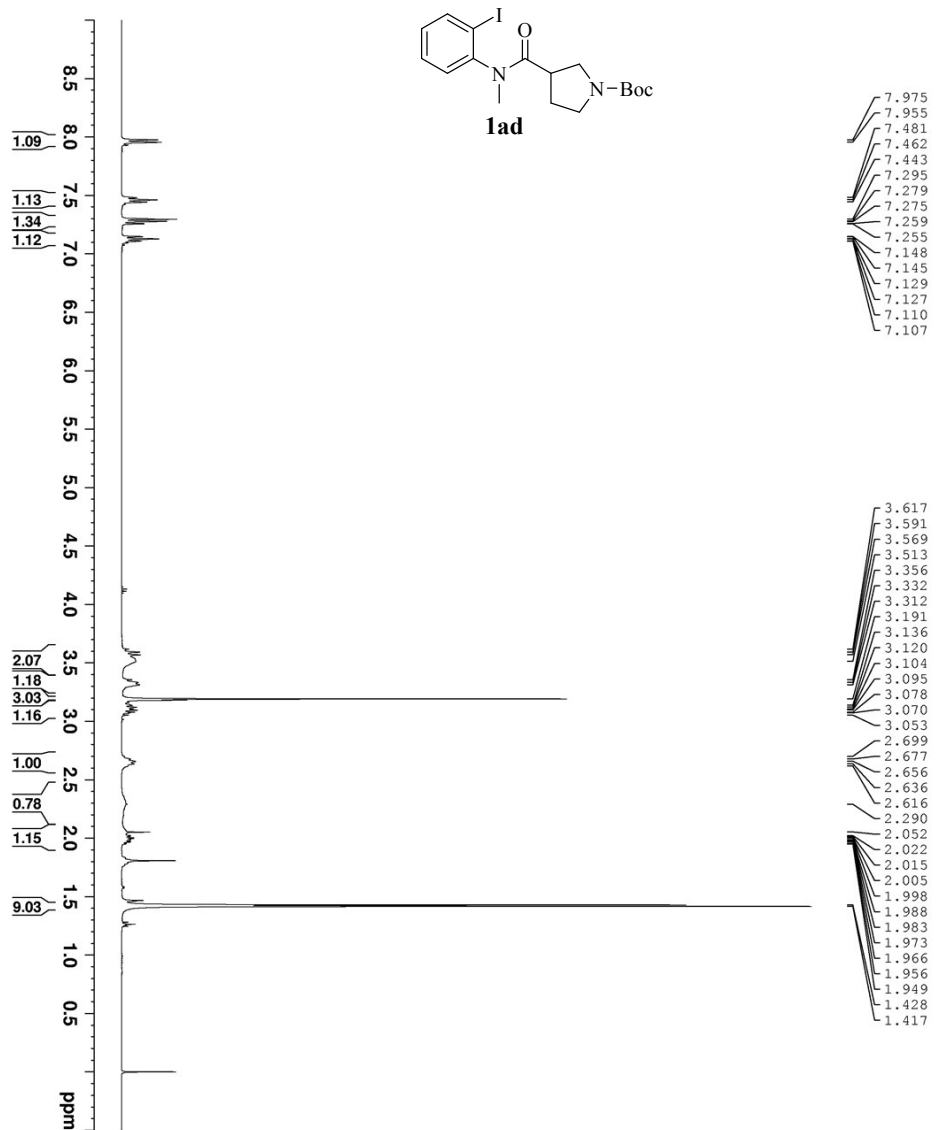
2-isopropyl-3,3-dimethylisoindolin-1-one (**2af**)



```

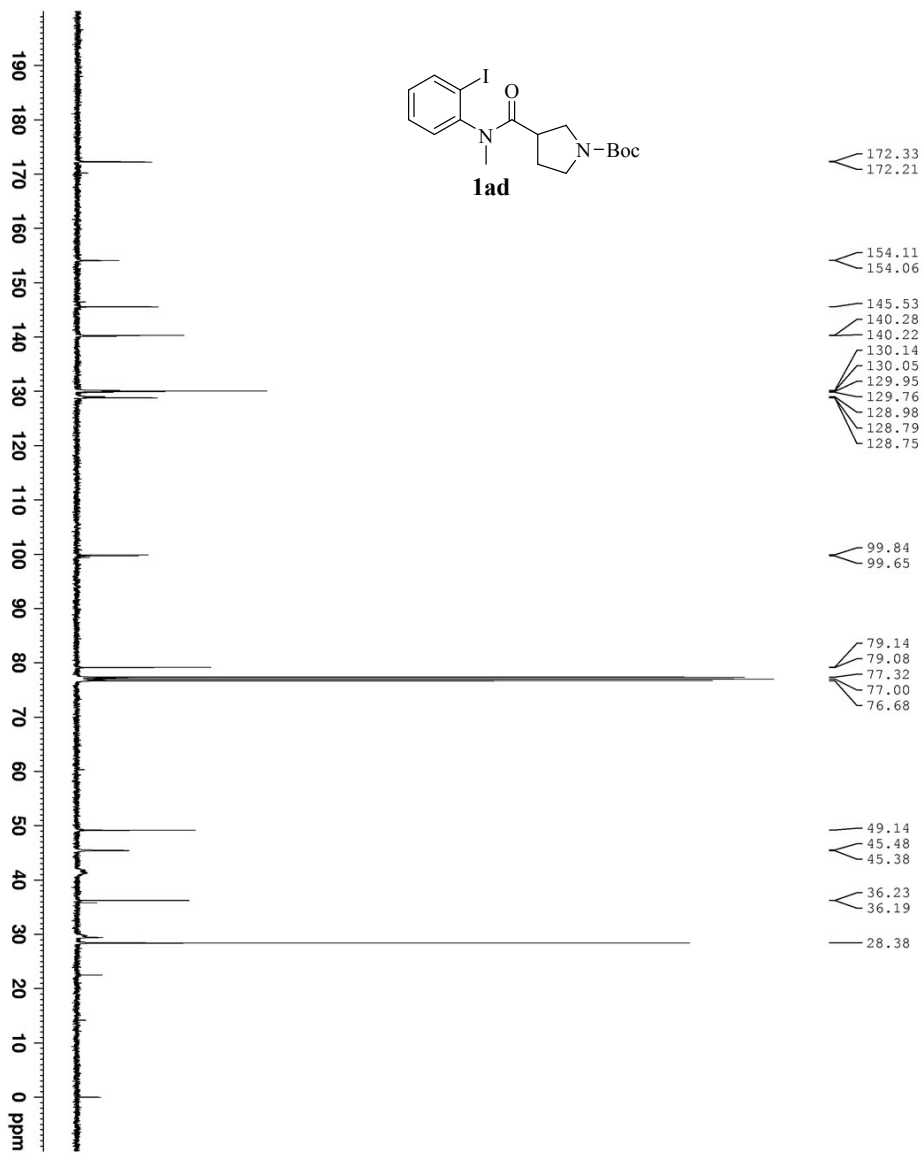
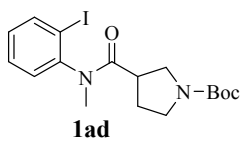
NAME      chem19-4-p62-2
EXPNO    12
PROCNO   1
PRF1     20151211
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
F2       62.50
SOLVENT  CDCl3
NS       16
DS       2
AQ       8012.827 Hz
FIDRES   0.122266 Hz
RG       4.0894966 sec
RG2      62.128
RG3      4.0894966 sec
DE       6.50 usec
TE       292.1 K
D1       1.00000000 sec
D10      1
===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1     13
P1       10.02 usec
SI       65536
SF       400.1300043 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
FS       1.00
    
```


tert-butyl 3-((2-iodophenyl)(methyl)carbamoyl)pyrrolidine-1-carboxylate (**1ad**)



```

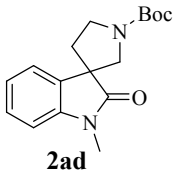
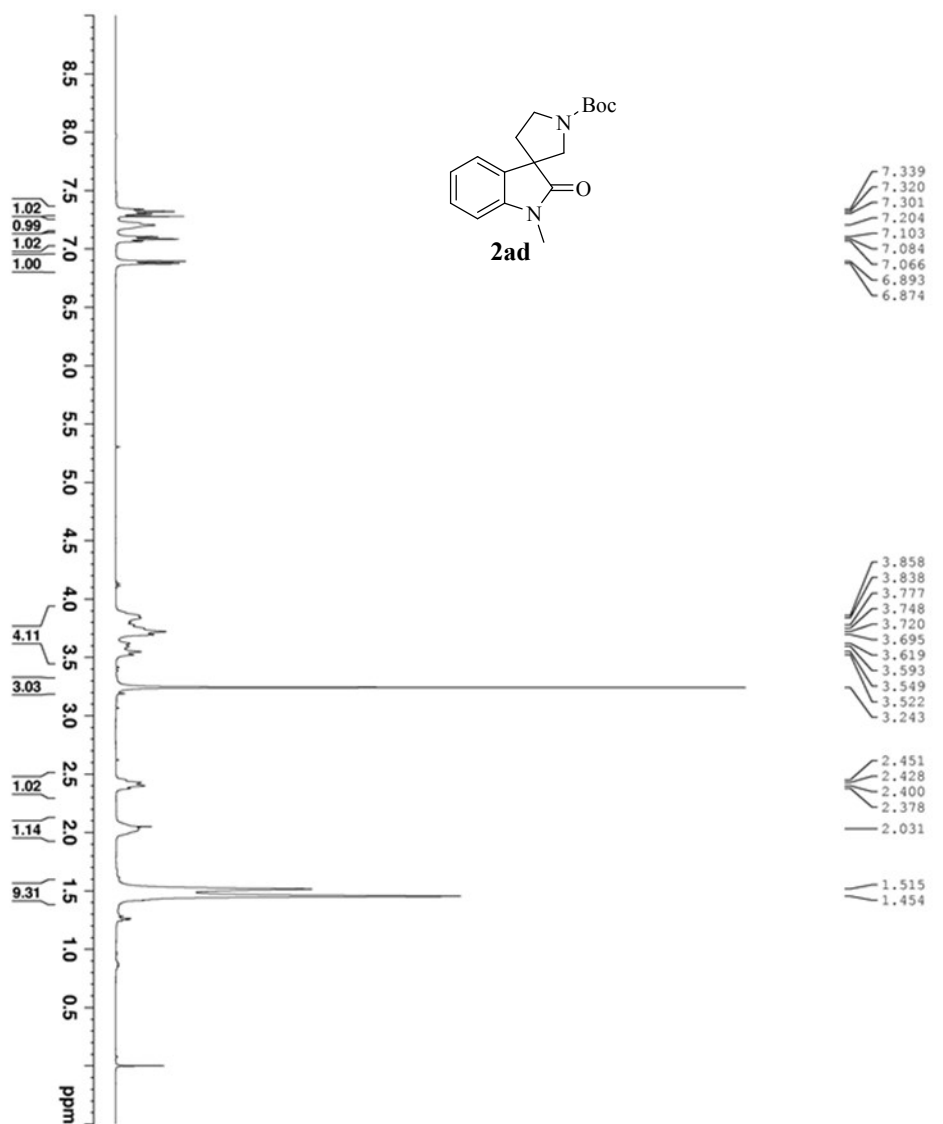
NAME          chen1g-5-p21
EXPNO         2
PROCNO        1
Date_         20160103
Time          14.48
TimeUnit      sec
PROBHD        5 mm PABBO BB7
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            1
DS            2
SWH           8012.820 Hz
FIDRES       0.122266 Hz
AQ           4.06926 sec
RG            80.6
DM           62.400 usec
DE           28.50 usec
TE           29.50 usec
D1           1.00000000 sec
TD0          1
===== CHANNEL f1 =====
SFO1          400.1324710 MHz
NUC1          1H
P1           10.92 usec
SF           400.129960 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
    
```



```

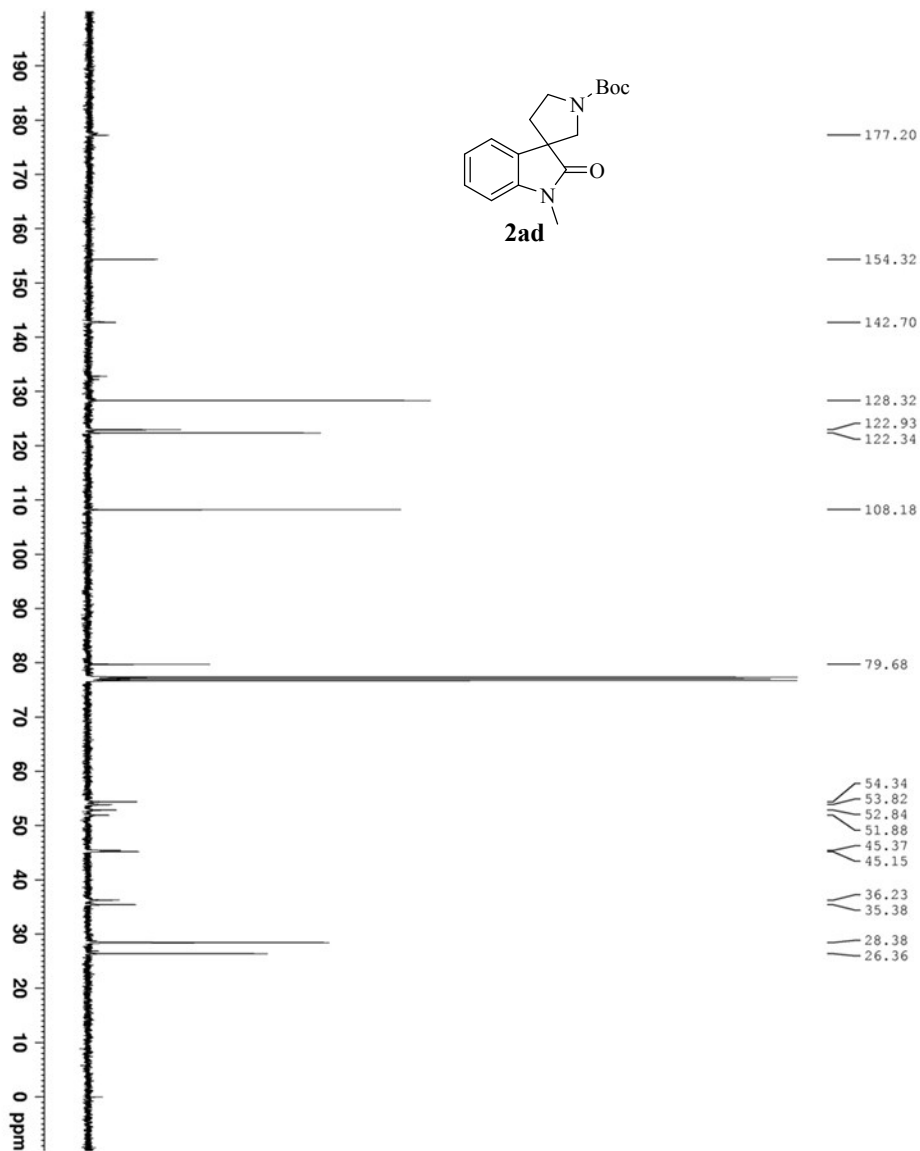
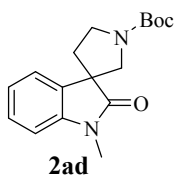
NAME: chen19-5-f21
EXPNO: 21
PROCNO: 1
Date_: 20160103
Time: 15.15
INSTRUM: spect
PROBHD: 5 mm PABBO
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
DS: 400
AS:
SM: 24038.461 Hz
FIDRES: 0.366798 Hz
AQ: 1.363198 sec
RG: 240
DE: 20.800 usec
TE: 281.7 K
D1: 2.0000000 sec
D11: 0.03000000 sec
TD0: 1
===== CHANNEL f1 =====
SFO1: 100.628238 MHz
NUC1: 13C
P1: 14.70 usec
SI: 32768
WDW: EM
SSB: 0
GB: 1.00 Hz
PC: 1.40
  
```

tert-butyl 1-methyl-2-oxospiro[indoline-3,3'-pyrrolidine]-1'-carboxylate (**2ad**)



```

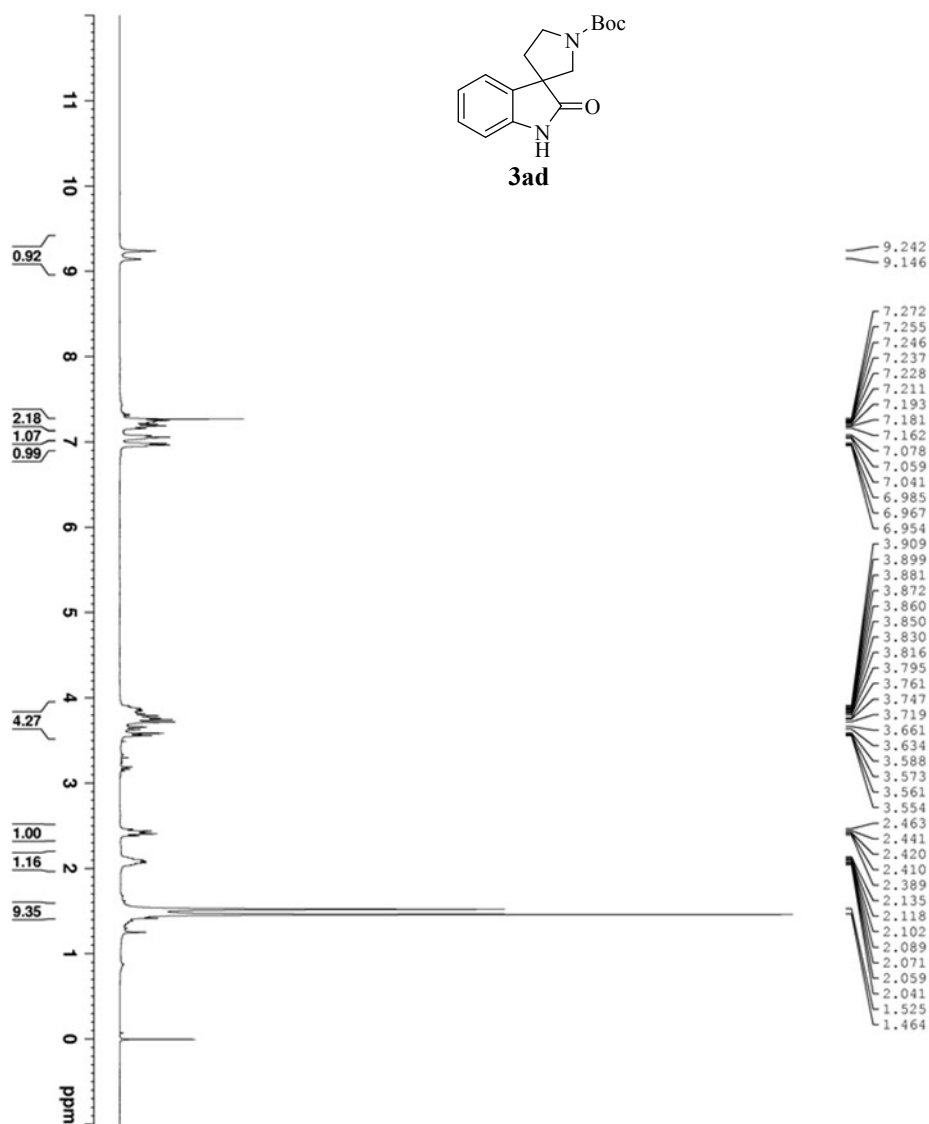
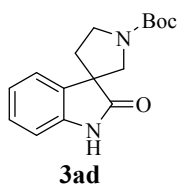
NAME: chen|q-4-p67.5
EXPNO: 1
PROCNO: 1
Date_ : 20161013
Time: 16:29
INSTRUM: spect
PROBHD: 5 mm PABBO 95/
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 2
DS: 2
SWH: 8012.820 Hz
AQ: 0.122566 Hz
RG: 41.97266 Hz
AQ: 4.199796 sec
RG: 90.5 Hz
DM: 62.400 usec
DE: 2.420 usec
TE: 29.50 Hz
D1: 1.00000000 sec
TDO: 1
===== CHANNEL f1 =====
SFO1: 400.1324710 MHz
NUC1: 13C
P1: 10.18 usec
PL1: 0.00 dB
SFO2: 400.1300019 MHz
NUC2: 1H
P2: 0.30 Hz
PL2: 1.00
    
```



```

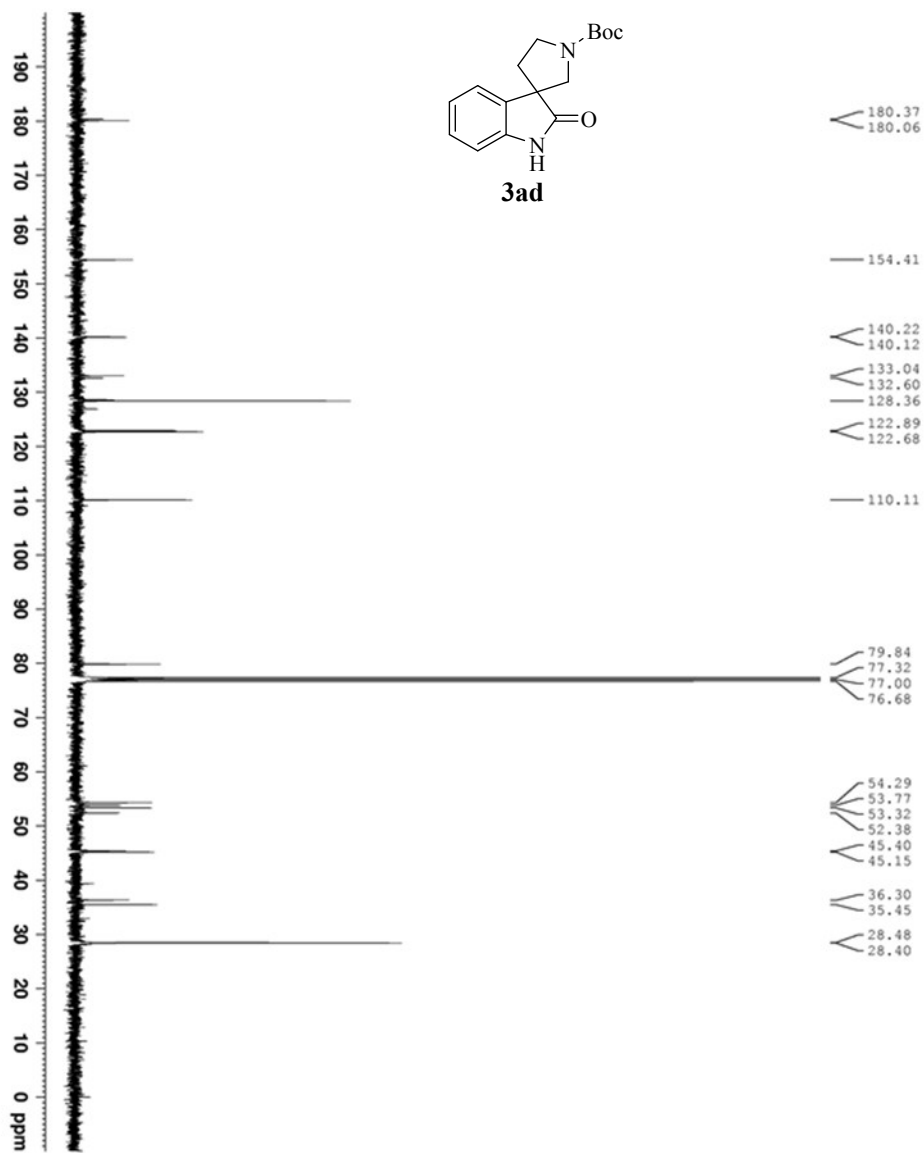
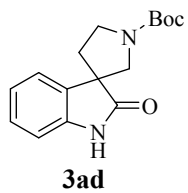
NAME          chenjq-4-pf7.5
EXPNO         11
PROCNO        1
DATE_         20160103
Time          16.55
INSTRUM       5 mm PABBO 300/
PROBHD        zgpg30
TD            65536
SOLVENT       CDCl3
NS            430
DS            2
AQ            24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.363188 sec
SFO            300.136200 MHz
DE            20.800 usec
DE            6.50 usec
TE            287.2 K
D11           0.0300000 sec
D11           0.0300000 sec
TDO           1
===== CHANNEL f1 =====
SFO1          300.136200 MHz
NUC1          13C
P1            14.70 usec
SI            32768
SF            100.612773 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```


tert-butyl 2-oxospiro[indoline-3,3'-pyrrolidine]-1'-carboxylate (**3ad**)



```

NAME: cben[3'-4'-p42,5-(6),C]
EXPNO: 1
PROCNO: 1
Date_: 20160107
Time: 11.39
INSTR: spect
PROBHD: 5 mm PABBO BH/
PULPROG: zgpg30
SOLVENT: CDCl3
NS: 16
DS: 2
AQ: 8012.823 Hz
RG: 0.122266 Hz
AQ: 4.0894966 sec
RG: 62.114 usec
DE: 6.50 usec
TE: 288.4 K
D1: 1.00000000 sec
TD: 1
===== CHANNEL f1 =====
NUC1: 13C
P1: 400.126410 MHz
PC: 10.92 usec
SI: 65536
SF: 400.135000 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00
    
```

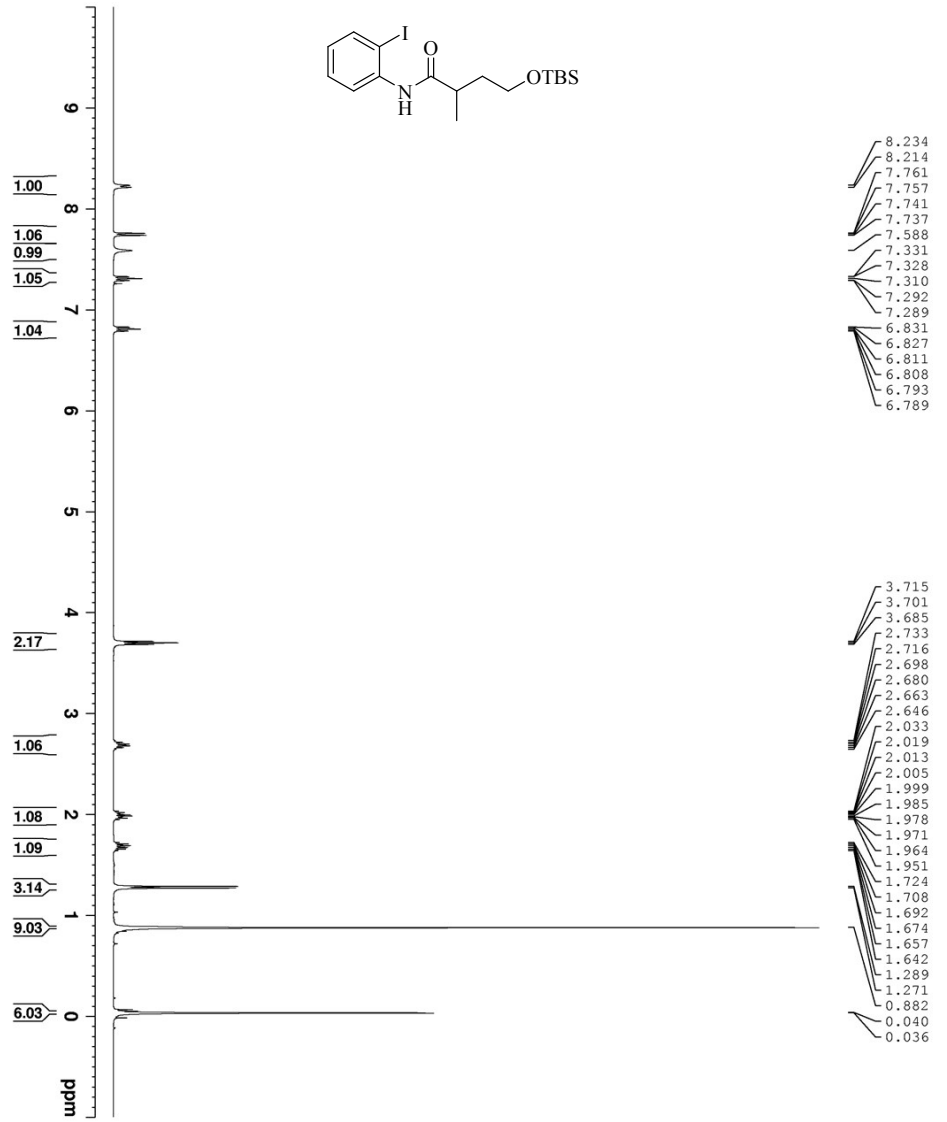
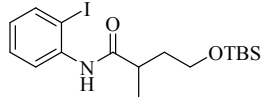


```

NAME: cben[9-4-pd].1-(B), C1
EXPNO: 1
PROCNO: 1
Date_: 20140107
Time: 13.56
INSTRUM: spect
PROBHD: 5 mm PABBO 5B/
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 280
DS: 2
AQ: 2.0319461 Hz
RG: 0.364341 Hz
FREQ: 125.7603000 MHz
NUC1: 13C
NUC2: 13C
PCPD: 13C
P1: 14.70 usec
SI: 32768
SF: 100.621718 MHz
WCM: EM
SSB: 0
LB: 1.00 Hz
GB:
PC: 1.40
===== CHANNEL f1 =====
NUC1: 13C
NUC2: 13C
PCPD: 13C
P1: 14.70 usec
SI: 32768
SF: 100.621718 MHz
WCM: EM
SSB: 0
LB: 1.00 Hz
GB:
PC: 1.40

```

4-((tert-butyldimethylsilyloxy)-N-(2-iodophenyl)-2-methylbutanamide

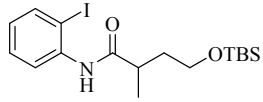


```

===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 10.92 usec
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

=====
NAME chemjg-5-p28-15
EXPNO 1
PROCNO 1
Date_ 20160103
Time 15.21
F2 -
PROBHD 5 mm PABBO BB7
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.089746 sec
RG 32
DM 62.400 usec
DE 8.50 usec
TE 29.50
D1 1.00000000 sec
TDO 1
=====

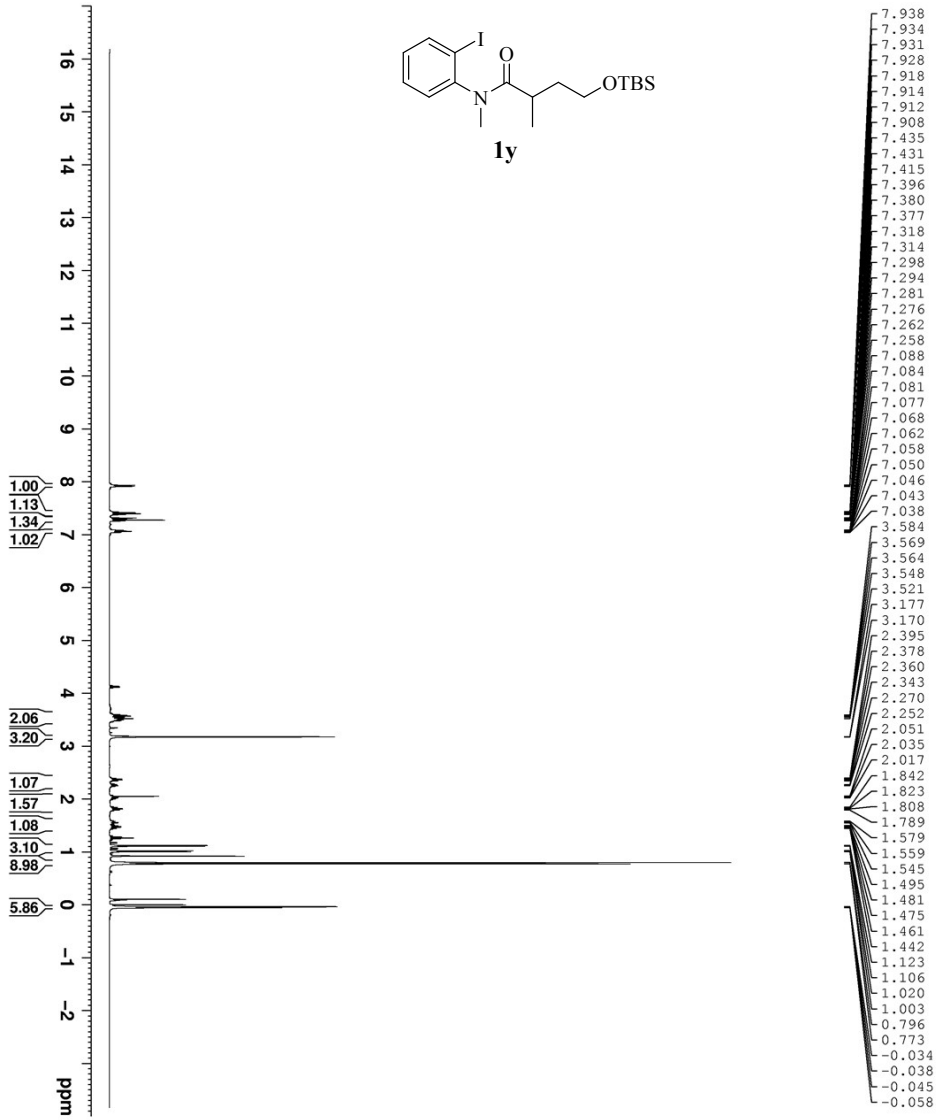
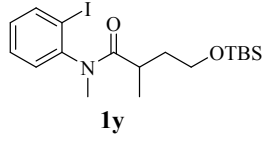
```



```

NAME: chen34-5-p28.5
EXPNO: 1
PROCNO: 1
DATE_ : 20160103
Time: 19.47
INSTRUM: spect
PROBHD: 5 mm PABBO
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
DS: 4
AQ: 24038.461 Hz
FIDRES: 0.366798 Hz
AQ: 1.363288 sec
RG: 240
DE: 20.800 usec
TE: 288.0 K
D1: 2.0000000 sec
D11: 0.03000000 sec
TD0: 1
===== CHANNEL f1 =====
SFO1: 100.628238 MHz
NUC1: 13C
P1: 14.70 usec
SI: 32768
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40
  
```

4-((tert-butyldimethylsilyl)oxy)-N-(2-iodophenyl)-N,2-dimethylbutanamide (**1y**)

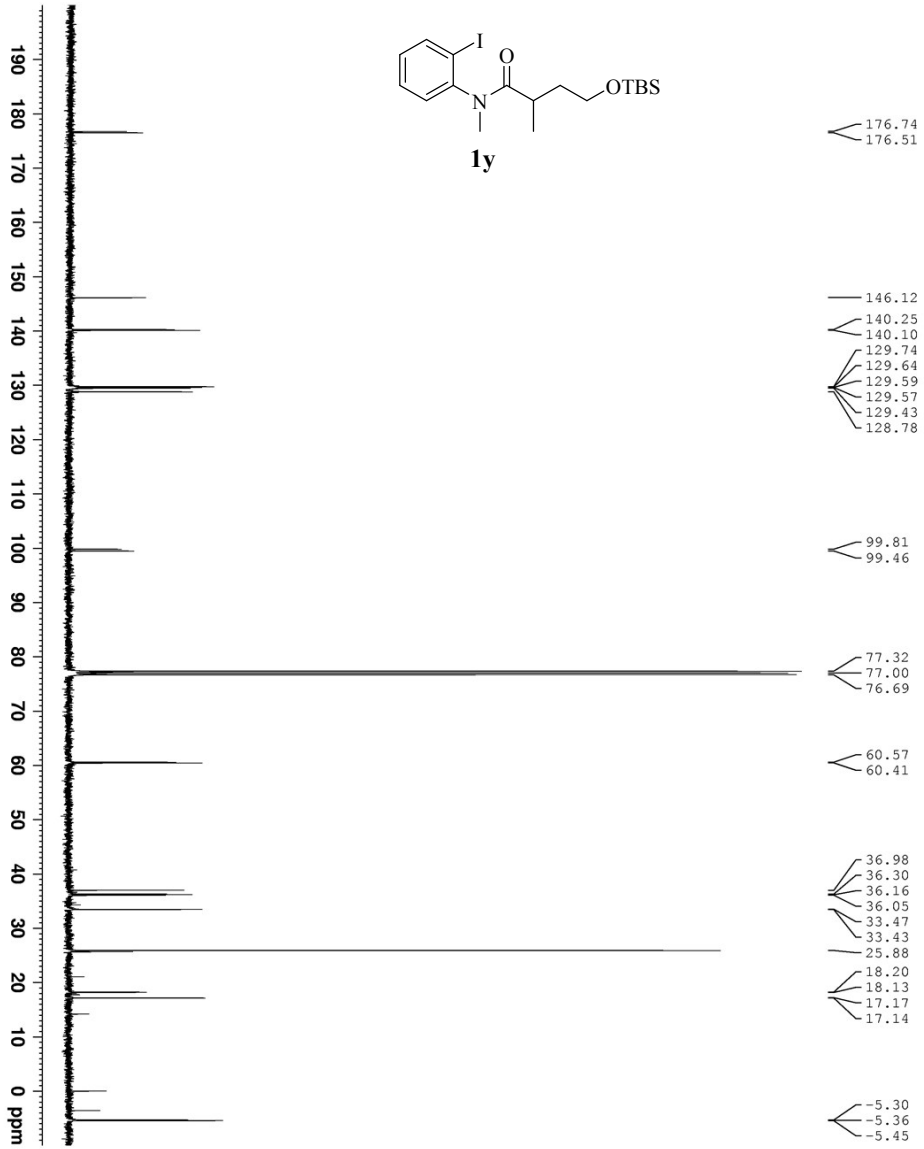
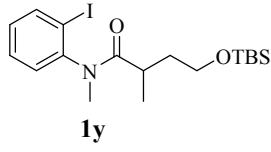


```

===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 10.92 usec
SP 400.1300036 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

=====
NAME chemjg-5-p30-15
EXPNO 1
PROCNO 1
Date_ 20160103
Time 17.01
INSTRUM spect
PROBHD 5 mm PABBO BB7
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.060266 sec
RG 90.5
DM 62.400 usec
DE 8.50 usec
TE 29.50
D1 1.00000000 sec
TDO 1
=====

```



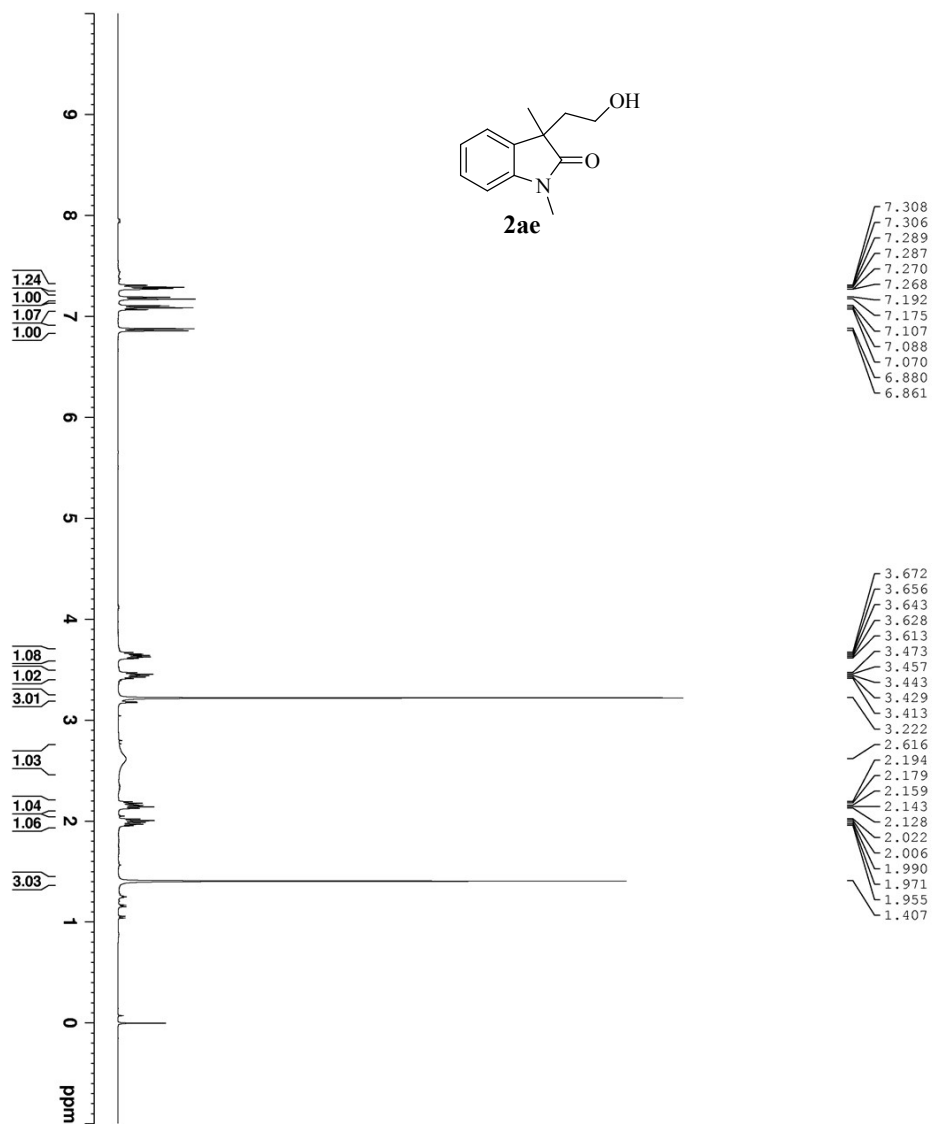
- 176.74
- 176.51
- 146.12
- 140.25
- 140.10
- 129.74
- 129.64
- 129.59
- 129.57
- 129.43
- 128.78
- 99.81
- 99.46
- 77.32
- 77.00
- 76.69
- 60.57
- 60.41
- 36.98
- 36.30
- 36.16
- 36.06
- 33.47
- 33.43
- 25.88
- 18.20
- 18.13
- 17.17
- 17.14
- 5.30
- 5.36
- 5.45

```

NAME: chen34-5-p30.5
EXPNO: 13
PROCNO: 1
Date_: 20160103
Time: 17.27
INSTRUM: spect
PROBHD: 5 mm PABBO
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
DS: 402
SWH: 24038.461 Hz
FIDRES: 0.366798 Hz
AQ: 1.363398 sec
RG: 240
DE: 20.800 usec
TE: 287.8 K
D1: 2.0000000 sec
D11: 0.0300000 sec
TD0: 1
===== CHANNEL f1 =====
SF01: 100.628238 MHz
NUC1: 13C
P1: 14.70 usec
SI: 32768
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

```

3-(2-hydroxyethyl)-1,3-dimethylindolin-2-one (**2ae**)



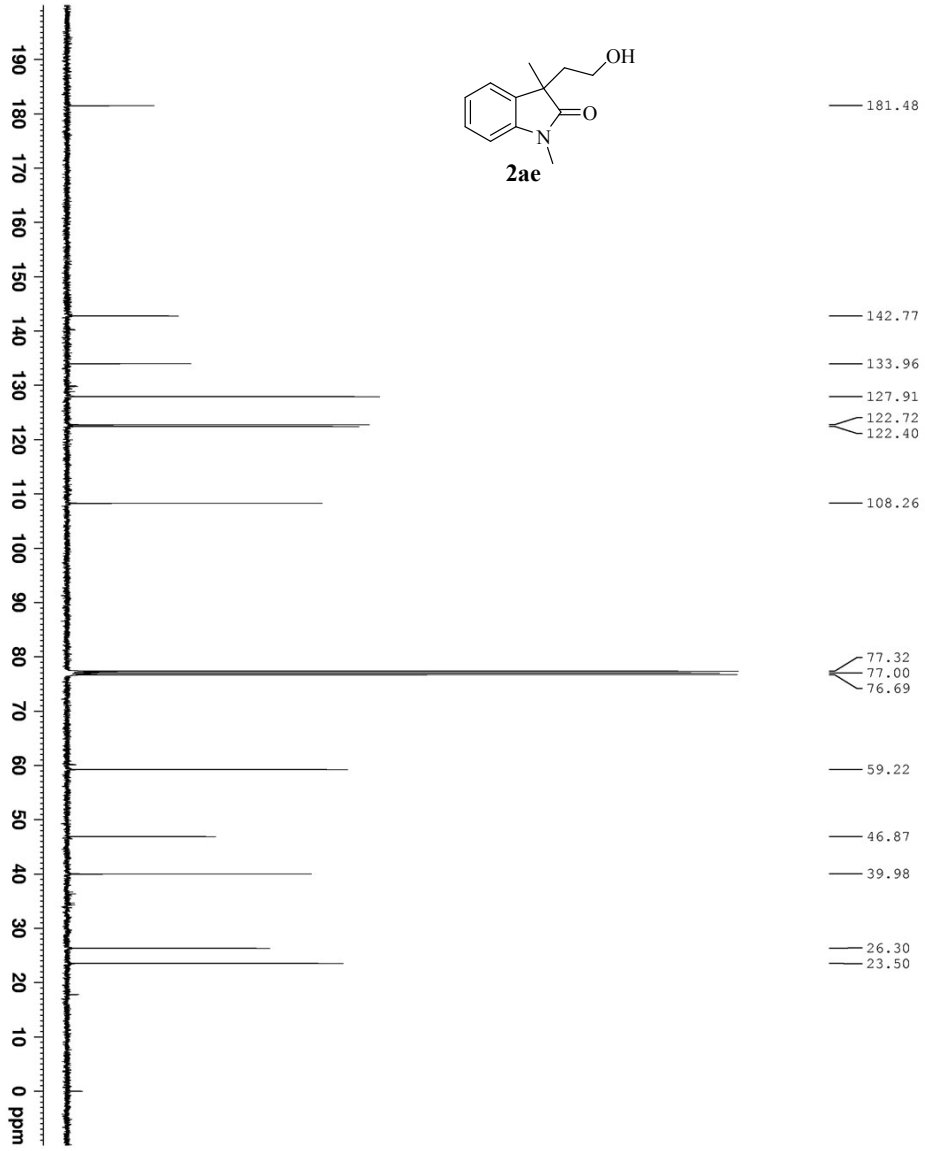
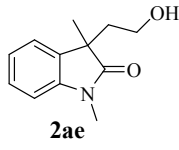
7.308
7.306
7.289
7.287
7.270
7.268
7.192
7.175
7.107
7.088
7.070
6.880
6.861

3.672
3.656
3.643
3.628
3.613
3.473
3.457
3.443
3.429
3.413
3.222
2.616
2.194
2.179
2.159
2.143
2.128
2.022
2.006
1.990
1.971
1.955
1.407

```

NAME          chemj9-4-p/3-1-H
EXPNO         1
PROCNO        1
Date_         20160126
Time          12.24
INSTRUM       spect
PROBHD        5 mm PABBO BB
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            1
DS            2
SWH           8012.820 Hz
FIDRES       0.122266 Hz
AQ           4.09246 sec
RG            90.5
DM           62.400 usec
DE           4.50 usec
TE           29.50
D1           1.00000000 sec
TD0           1

===== CHANNEL f1 =====
SFO1          400.1324710 MHz
NUC1          1H
P1            10.92 usec
SF            400.1300033 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

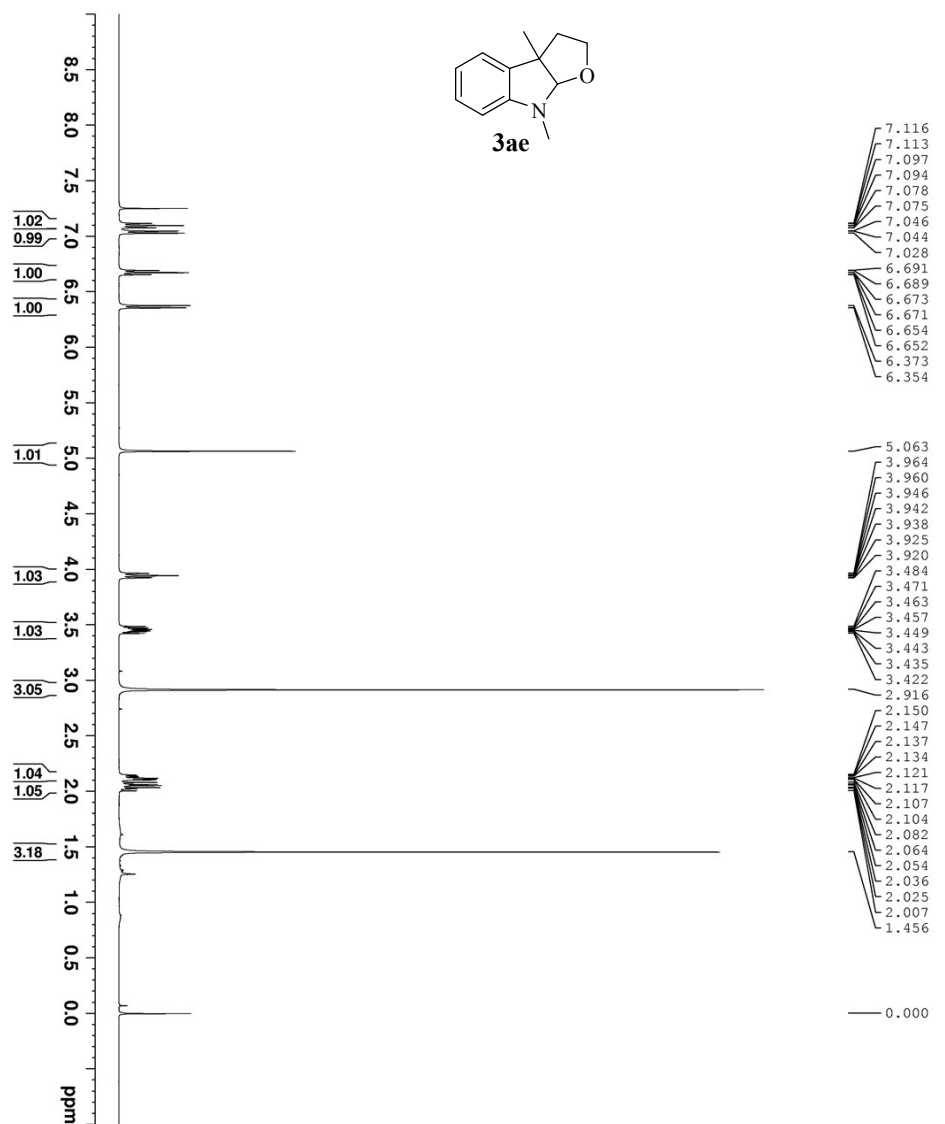
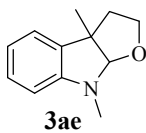


```

NAME: chen34-p/3-1-c
EXPNO: 1
PROCNO: 1
Date_: 20160126
Time: 20.29
INSTRUM: spect
PROBHD: 5 mm PABBO
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
DS: 402
NS: 24038.461 Hz
FIDRES: 0.366798 Hz
AQ: 1.363398 sec
RG: 240
DE: 20.800 usec
TE: 285.5 K
D1: 2.0000000 sec
D11: 0.03000000 sec
TD0: 1

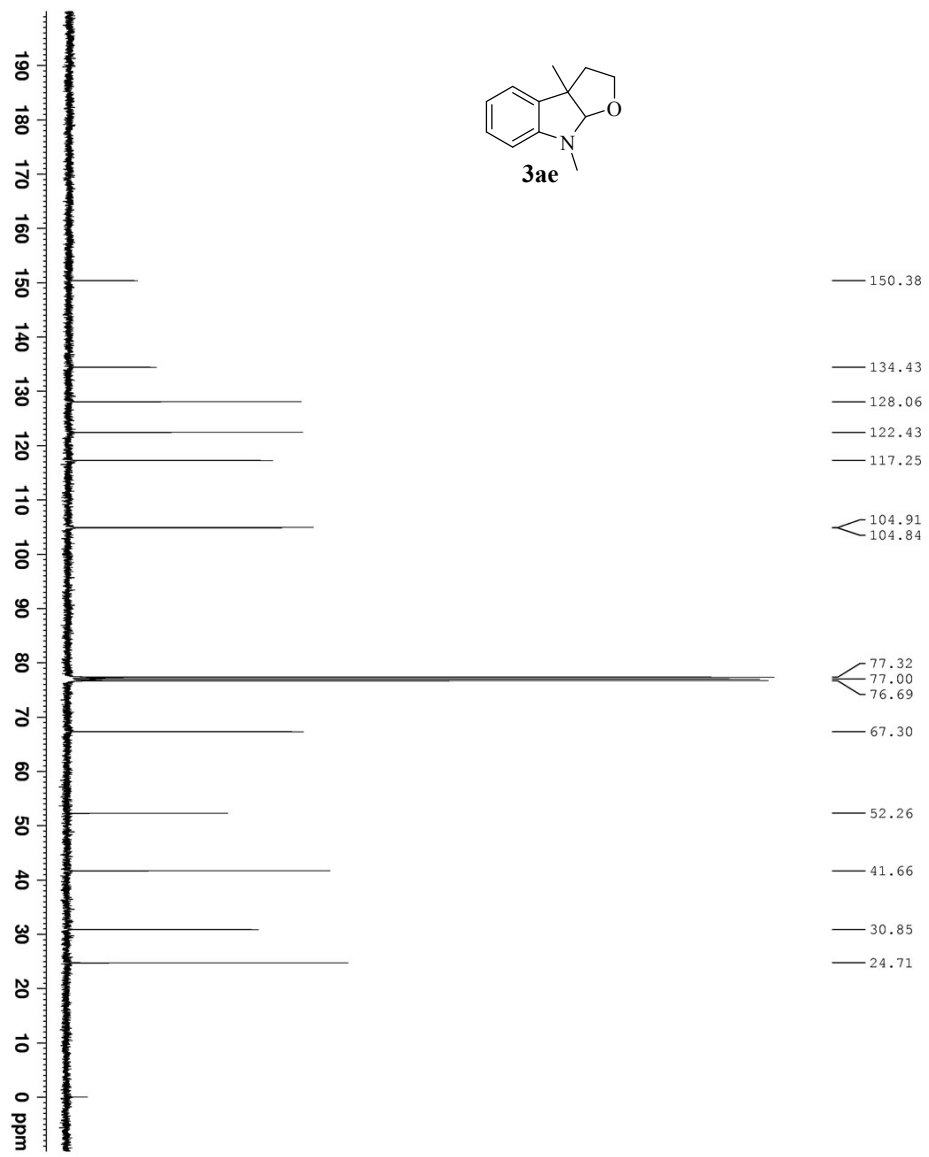
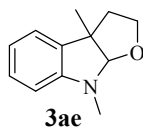
===== CHANNEL f1 =====
SFO1: 100.628238 MHz
NUC1: 13C
P1: 14.70 usec
SI: 32768
SF: 100.612778 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40
  
```


3a,8-dimethyl-3,3a,8,8a-tetrahydro-2H-furo[2,3-b]indole (**3ae**)



```

NAME          chemj1-4-f710
EXPNO         1
PROCNO        1
Date_         20160108
Time          21.09
F2 - F1       14.4
PROBHD        5 mm PABBO BB7
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            1
DS            2
SWH           8012.820 Hz
FIDRES       0.122266 Hz
AQ           4.0897161 sec
RG            161
DM           62.400 usec
DE           29.50 usec
TE           300.2 K
D1           1.00000000 sec
TD0          1
===== CHANNEL f1 =====
SFO1          400.1324710 MHz
NUC1          1H
P1           16.79 usec
SF           400.1300140 MHz
WDW          EM
SSB          0
GB           0.30 Hz
PC           1.00
    
```



```

NAME: chen34-4-p10-C
EXPNO: 1
PROCNO: 1
Date_: 20160111
Time: 12.29
INSTRUM: spect
PROBHD: 5 mm PABBO
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
DS: 329
AQ: 24038.461 Hz
FIDRES: 0.366798 Hz
AQ: 1.363198 sec
RG: 132
DE: 20.800 usec
TE: 288.5 K
D1: 2.0000000 sec
D11: 0.03000000 sec
TD0: 1
===== CHANNEL f1 =====
SFO1: 100.628238 MHz
NUC1: 13C
P1: 14.70 usec
SI: 32768
SF: 100.612771 MHz
WDW: EM
SSB: 0
GB: 1.00 Hz
PC: 1.40
  
```