Electronic Supplementary Material (ESI) for ChemComm.
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Synthesis of functionalized tryptamines by Brønsted acid catalyzed cascade reactions

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General Methods

^{1}H NMR spectra were recorded at 500, or 400 MHz spectrometer at ambient temperature with $CDCl_3$ as solvent. Data are reported as follows: chemical shifts (δ), multiplicity, coupling constants and integration. ^{13}C NMR spectra were recorded operating respectively at 126, or 101 MHz at 27°C with $CDCl_3$ as solvent. Infrared spectra were recorded on a FT-IR spectrophotometer. Low resolution mass spectral analyses were recorded in E.I. (70 eV) mode. High resolution mass spectra (HRMS) was recorded on a spectrometer using Positive Electro Ionization (ESI) mode. Analytical thin layer chromatography was performed using 0.25 mm silica gel 60-F plates. Flash chromatography was performed using columns of 230-400 mesh silica gel 60 (0.040-0.063 mm). Yields refer to chromatographically pure materials.

General procedure for the synthesis of tryptamines 3

A mixture of aryl amine **2** (0.930 mmol), freshly distilled 2-hydroxy cyclobutanone **1** (0.465 mmol), and PTSA (0.093 mmol) was stirred at room temperature for 6 days. The crude reaction mixture was directly loaded on silica gel column without aqueous work-up and pure products were obtained by flash column chromatography (silica gel, mixture of hexane/ether, 10:1→1:1).

3a: Yield 67% (82 mg); yellow oil. IR (neat): 3057, 3027, 2937, 2882, 2822, 1601, 1508, 1474, 1377, 1328 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.9 Hz, 1H), 7.33 – 7.26 (m, 2H), 7.26 – 7.19 (m, 2H), 7.17 – 7.08 (m, 1H), 6.85 (s, 1H), 6.78 (d, *J* = 8.0 Hz, 2H), 6.70 (t, *J* = 7.2 Hz, 1H), 3.73 (s, 3H), 3.67 – 3.60 (m, 2H), 3.04 – 2.97 (m, 2H), 2.94 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.19, 137.18, 129.38, 128.06, 126.67, 121.73, 118.98, 118.93, 116.16, 112.45, 112.32, 109.37, 53.89, 38.53, 32.71, 22.34. MS m/z: 264 (M^+ (19)), 144 (11), 120 (100), 105 (3). HRMS (ESI) Calcd. For $C_{18}H_{20}N_2$ (M+1) m/z 265,1699, found 265.1693.

3b: Yield 51% (69 mg); yellow oil. IR (neat): 3015, 2916, 2863, 1620, 1522, 1494, 1378 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (s, 1H), 7.18 (d, *J* = 8.4 Hz, 1H), 7.06 (t, *J* = 8.7 Hz, 3H), 6.81 (s, 1H), 6.72 (d, *J* = 8.5 Hz, 2H), 3.71 (s, 3H), 3.59 (dd, *J* = 9.1, 6.6 Hz, 2H), 2.96 (d, *J* = 8.1 Hz, 2H), 2.93 (s, 3H), 2.47 (s, 3H), 2.27 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.25, 135.62, 129.90, 128.27, 128.08, 126.72, 125.45, 123.32, 118.68, 112.80, 111.97, 109.07, 54.18, 38.72, 32.76, 22.12, 21.66, 20.38. MS m/z: 292 (M^+ (18)), 158 (9), 134 (100), 119 (5). HRMS (ESI) Calcd. For $C_{20}H_{24}N_2$(M+1) m/z 293,2012, found 293.2005.

3c: Yield 59% (88 mg); yellow oil. IR (neat): 3015, 2962, 2926, 2868, 1617, 1522, 1491, 1453, 1377 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (d, *J* = 0.8 Hz, 1H), 7.26 – 7.17 (m, 1H), 7.13 – 7.05 (m, 3H), 6.82 (s, 1H), 6.78 – 6.71 (m, 2H), 3.71 (s, 3H), 3.63 – 3.57 (m, 2H), 3.00 – 2.95 (m, 2H), 2.94 (s, 3H), 2.76 (q, *J* = 7.6 Hz, 2H), 2.57 (q, *J* = 7.6 Hz, 2H), 1.30 (t, *J* = 7.6 Hz, 3H), 1.22 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl3_3$) δ 147.45, 135.78, 134.88, 132.05, 128.70, 128.26, 126.69, 122.32, 117.45, 112.71, 112.18, 109.16, 54.14, 38.68, 32.75, 29.21, 27.94, 22.25, 16.71, 16.09. HRMS (ESI) Calcd. For $C_{22}H_{28}N_2$(M+1) m/z 321,2325, found 321.2317.

3d: Yield 45% (73 mg); yellow oil. IR (neat): 2957, 2926, 2868, 1615, 1519, 1491, 1453, 1378 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (s, 1H), 7.24 – 7.16 (m, 1H), 7.11 – 7.02 (m, 3H), 6.82 (s, 1H), 6.73 (d, *J* = 8.6 Hz, 2H), 3.71 (s, 3H), 3.67 – 3.54 (m, 2H), 2.97 (dd, *J* = 9.0, 6.7 Hz, 2H), 2.93 (s, 3H), 2.76 – 2.62 (m, 2H), 2.58 – 2.42 (m, 2H), 1.69 (dd, *J* = 15.0, 7.5 Hz, 2H), 1.61 (dd, *J* = 15.1, 7.4 Hz, 2H), 0.97 (t, *J* = 5.6 Hz, 3H), 0.94 (t, *J* = 5.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.42, 135.78, 133.21, 130.46, 129.30, 128.18, 126.63, 122.82, 118.19, 112.58, 112.14, 109.03,

54.13, 38.65, 38.44, 37.21, 32.74, 25.54, 25.03, 22.27, 14.09, 14.05. HRMS (ESI) Calcd. For $C_{24}H_{32}N_2$(M+1) m/z 349,2638, found 349.2621.

3e: Yield 59% (104 mg); yellow oil. IR (neat): 3015, 2954, 2924, 2858, 1615, 1522, 1491, 1456, 1373, 1355 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 0.8 Hz, 1H), 7.23 – 7.15 (m, 1H), 7.11 – 7.00 (m, 3H), 6.81 (s, 1H), 6.76 – 6.67 (m, 2H), 3.70 (s, 3H), 3.64 – 3.56 (m, 2H), 3.01 – 2.94 (m, 2H), 2.93 (s, 3H), 2.76 – 2.69 (m, 2H), 2.57 – 2.49 (m, 2H), 1.70 – 1.62 (m, 2H), 1.61 – 1.53 (m, 2H), 1.38 (tq, *J* = 14.6, 7.3 Hz, 4H), 0.95 (t, *J* = 5.1 Hz, 3H), 0.92 (t, *J* = 5.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.41, 135.77, 133.41, 130.65, 129.25, 128.21, 126.62, 122.79, 118.12, 112.61, 112.15, 109.03, 54.14, 38.64, 35.98, 34.74, 34.70, 34.17, 32.73, 22.60, 22.55, 22.29, 14.19, 14.16. HRMS (ESI) Calcd. For $C_{26}H_{36}N_2$(M+1) m/z 377,2951, found 377.2935.

3f: Yield 55% (94 mg); orange oil. IR (neat): 2954, 2906, 2870, 1615, 1523, 1489, 1362, 1297, 1254 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.63 – 7.57 (m, 1H), 7.31 (ddd, *J* = 9.0, 7.7, 2.0 Hz, 3H), 7.23 (d, *J* = 8.6 Hz, 1H), 6.83 (s, 1H), 6.80 – 6.73 (m, 2H), 3.70 (s, 3H), 3.66 – 3.57 (m, 2H), 3.05 – 2.97 (m, 2H), 2.95 (s, 3H), 1.41 (s, 9H), 1.32 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.00, 141.79, 138.80, 135.40, 127.81, 126.54, 126.16, 120.03, 114.72, 112.53, 112.16, 108.88, 54.03, 38.53, 34.72, 33.86, 32.70, 32.13, 31.72, 22.27. HRMS (ESI) Calcd. For $C_{26}H_{36}N_2$(M+1) m/z 377,2951, found 377.2942.

3g: Yield 60% (91 mg); yellow oil. IR (neat): 2989, 2939, 2904, 2833, 1622, 1575, 1511, 1494, 1459, 1426, 1247, 1226, 1176 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, *J* = 8.8 Hz, 1H), 7.01 (d, *J* = 2.4 Hz, 1H), 6.93 – 6.81 (m, 4H), 6.77 (d, *J* = 9.1 Hz, 2H), 3.85 (s, 3H), 3.77 (s, 3H), 3.71 (s, 3H), 3.56 (dd, *J* = 8.9, 6.6 Hz, 2H), 2.97 – 2.91 (m, 2H), 2.90 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.88, 151.71, 144.28, 132.61, 128.37, 127.20, 115.05, 114.58, 112.10, 111.89, 110.11, 101.03, 56.15, 56.01, 54.86, 39.21, 32.92, 22.16. HRMS (ESI) Calcd. For $C_{20}H_{24}N_2O_2$(M+1) m/z 325,1911, found 325.1900.

3h+3h'[1]: Inseparable 85:15 mixture of two regioisomers. Yield 81% (111 mg); yellow oil. IR (neat): 3040, 3030, 2914, 2858, 2815, 1602, 1580, 1499, 1475, 1378, 1327, 1247, 1226, 1176 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.1 Hz, 1H), 7.26 (s, 1H), 7.17 – 7.10 (m, 3H), 7.08 (dd, *J* = 3.0, 2.4 Hz, 2H), 6.95 (dd, *J* = 8.0, 0.9 Hz, 1H), 6.86 – 6.82 (m, 1H), 6.81 (s, 1H), 6.77 (s, 1H), 6.63 – 6.56 (m, 3H), 6.55 – 6.50 (m, 2H), 3.70 (s, 3H), 3.69 (s, 3H), 3.65 – 3.57 (m, 4H), 3.15 (dd, *J* = 8.7, 6.9 Hz, 2H), 2.99 – 2.95 (m, 2H), 2.94 (s, 3H), 2.93 (s, 3H), 2.73 (s, 3H), 2.50 (s, 3H), 2.32 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.36, 149.29, 139.03, 138.98, 137.60, 137.53, 131.55, 130.91, 129.23, 126.94, 126.47, 126.04, 125.96, 121.74, 120.72, 120.68, 118.67, 117.18, 117.12, 113.15, 113.11, 112.36, 109.58, 109.37, 107.45, 107.29, 55.11, 53.92, 38.55, 38.52, 32.79, 32.61, 24.19, 22.44, 22.12, 22.09, 22.00, 20.48. HRMS (ESI) Calcd. For $C_{20}H_{24}N_2$(M+1) m/z 293,2012, found 293.2007.

3i: Yield 0%. **Ai**: Yield 35% (34 mg); colourless oil. The spectroscopic data are in accordance with those presented in literature[1]. ^1H NMR (400 MHz, $CDCl_3$) δ 6.98 (ddd, *J* = 15.2, 7.6, 1.7 Hz, 2H), 6.93 – 6.80 (m, 2H), 4.92 (tt, *J* = 10.7, 2.2 Hz, 1H), 3.84 (s, 3H), 2.83 (s, 3H), 2.81 – 2.71 (m, 1H), 2.65 (dddd, *J* = 17.3, 9.9, 4.8, 2.4 Hz, 1H), 2.29 – 2.09 (m, 2H).

3j: Yield 23% (33 mg); white solid; m. p. 154-158°C. IR (nujol): 3015, 2934, 2906, 2851, 2218, 1605, 1522, 1486, 1388, 1350, 1174 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.87 (d, *J* = 0.8 Hz, 1H), 7.45 (ddd, *J* = 5.2, 4.2, 1.8 Hz, 3H), 7.34 (d, *J* = 8.5 Hz, 1H), 6.95 (s, 1H), 6.62 (d, *J* = 9.0 Hz, 2H), 3.77 (s, 3H), 3.71 – 3.64 (m, 2H), 3.05 – 2.99 (m, 2H), 2.94 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.34, 138.59, 133.68, 129.16, 127.62, 124.83, 124.37, 112.92, 111.44, 110.40, 102.22, 97.60, 53.13, 38.74, 33.03, 22.42. HRMS (ESI) Calcd. For $C_{20}H_{18}N_4$. (M+1) m/z 315,1604, found

315.1593. **Aj:** Yield 45% (42 mg); yellow oil. IR (neat): 3047, 2961, 2928, 2831, 2213, 1785, 1605, 1519, 1399, 1384, 1320, 1179, 1123, 1077 cm-1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.42 (m, 2H), 6.73 (d, *J* = 9.0 Hz, 2H), 5.14 (dd, *J* = 12.8, 5.9 Hz, 1H), 3.03 – 2.91 (m, 1H), 2.95 (s, 3H), 2.83 (dddd, *J* = 17.7, 10.1, 4.6, 2.4 Hz, 1H), 2.47 (ddd, *J* = 14.7, 10.6, 4.4 Hz, 1H), 2.14 (dt, *J* = 19.8, 9.8 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 204.98, 151.36, 133.62, 120.12, 112.80, 99.82, 73.06, 41.10, 34.00, 16.89. Calcd. For $C_{12}H_{12}N_2O$(M+Na) m/z 223,0842, found 223.0839.

3k: Yield 48% (59 mg); yellow oil. IR (neat): 3058, 2939, 2823, 1628, 1612, 1580, 1511, 1489, 1426, 1355, 1179 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 – 7.13 (m, 2H), 6.95 (ddd, *J* = 14.3, 6.8, 2.4 Hz, 3H), 6.86 (s, 1H), 6.69 – 6.60 (m, 2H), 3.71 (s, 3H), 3.54 (dd, *J* = 8.7, 6.6 Hz, 2H), 2.91 (d, *J* = 7.9 Hz, 2H), 2.88 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.94, 156.60, 154.26, 145.98, 133.81, 128.36, 115.69 (d, *J* = 22.0 Hz), 113.61 (d, *J* = 7.1 Hz), 112.31 (d, *J* = 4.5 Hz), 110.15 (d, *J* = 12.3 Hz), 109.97 (d, *J* = 3.7 Hz), 103.75 (d, *J* = 23.2 Hz), 54.47, 39.02, 32.99, 22.20. HRMS (ESI) Calcd. For $C_{18}H_{18}F_2N_2$(M+1) m/z 301,1511, found 301.1506.

3l: Yield 73% (137 mg); yellow oil. IR (neat): 3055, 3025, 2954, 2926, 2856, 1597, 1504, 1469, 1368, 1191 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 7.9 Hz, 1H), 7.27 – 7.19 (m, 3H), 7.11 (t, *J* = 7.4 Hz, 1H), 6.91 (s, 1H), 6.74 (d, *J* = 8.6 Hz, 2H), 6.65 (t, *J* = 7.2 Hz, 1H), 4.05 (t, *J* = 7.2 Hz, 2H), 3.63 – 3.54 (m, 2H), 3.31 – 3.22 (m, 2H), 3.05 – 2.97 (m, 2H), 1.79 (dd, *J* = 14.0, 7.0 Hz, 2H), 1.62 – 1.52 (m, 2H), 1.29 (d, *J* = 3.6 Hz, 12H), 0.88 (dd, *J* = 6.7, 4.2 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.09, 136.45, 129.44, 128.11, 125.56, 121.54 (s), 119.04, 118.80, 115.41, 112.37, 111.88, 109.57, 52.06 , 51.34, 46.36, 31.89, 31.60, 30.44, 27.52, 27.02, 26.86, 23.06, 22.83, 22.69, 14.20, 14.16. MS m/z: 404 (M^+ (11)), 331 (2), 228 (2), 214 (4), 190 (100), 120 (16), 106 (8). HRMS (ESI) Calcd. For $C_{28}H_{40}N_2$(M+1) m/z 405,3264, found 405.3246.

3m: Yield 37% (68 mg); yellow oil. IR (neat): 3058, 2931, 2856, 1597, 1504, 1461, 1448, 1360, 1343, 1300, 1214, 1156 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.7 Hz, 1H), 7.36 (d, *J* = 8.3 Hz, 1H), 7.29 – 7.22 (m, 2H), 7.20 (ddd, *J* = 8.2, 7.0, 1.1 Hz, 1H), 7.11 (ddd, *J* = 7.9, 7.0, 0.9 Hz, 1H), 7.07 (s, 1H), 6.85 (d, *J* = 8.1 Hz, 2H), 6.68 (t, *J* = 7.2 Hz, 1H), 4.20 (tt, *J* = 11.9, 3.7 Hz, 1H), 3.61 (tt, *J* = 11.5, 3.4 Hz, 1H), 3.53 – 3.46 (m, 2H), 3.03 – 2.96 (m, 2H), 2.16 – 2.09 (m, 2H), 1.98 – 1.66 (m, 10H), 1.56 – 1.23 (m, 7H), 1.15 (qt, *J* = 12.6, 3.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.69, 135.96, 129.43, 128.04, 121.62, 121.36, 119.20, 118.89, 115.94, 112.94, 112.73, 109.61, 57.60, 55.14, 46.08, 33.73, 31.02, 26.48, 26.17, 25.85, 25.67. HRMS (ESI) Calcd. For $C_{28}H_{36}N_2$(M+1) m/z 401,2951, found 401.2939.

3n: Yield 60% (104 mg); yellow oil. IR (neat): 3080, 3012, 2959, 2924, 2868, 1617, 1519, 1486, 1451, 1377, 1189 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (s, 1H), 7.24 – 7.17 (m, 1H), 7.07 (t, *J* = 6.9 Hz, 3H), 6.87 (s, 1H), 6.72 (d, *J* = 8.6 Hz, 2H), 5.95 (ddd, *J* = 22.4, 10.5, 5.4 Hz, 1H), 5.85 (ddt, *J* = 17.0, 10.1, 5.0 Hz, 1H), 5.25 – 4.98 (m, 4H), 4.63 (d, *J* = 5.4 Hz, 2H), 3.90 (d, *J* = 4.9 Hz, 2H), 3.68 – 3.53 (m, 2H), 3.02 (dd, *J* = 9.2, 6.6 Hz, 2H), 2.76 (q, *J* = 7.6 Hz, 2H), 2.56 (q, *J* = 7.6 Hz, 2H), 1.30 (t, *J* = 7.6 Hz, 3H), 1.21 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 146.46, 135.15, 135.01, 134.85, 133.91, 131.84, 128.67, 128.46, 125.58, 122.36, 117.54, 117.19, 115.97, 112.50, 109.52, 53.65, 51.81, 48.85, 29.16, 27.89, 23.16, 16.59, 16.05. HRMS (ESI) Calcd. For $C_{26}H_{32}N_2$(M+1) m/z 373,2638, found 373.2626.

3o: Yield 32% (60 mg); yellow oil. IR (neat): 3060, 2979, 2934, 1749, 1602, 1506, 1464, 1368, 1262, 1194, 1027 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.9 Hz, 1H), 7.25 – 7.21 (m, 4H), 7.17 – 7.09 (m, 1H), 6.92 (s, 1H), 6.76 – 6.68 (m, 3H), 4.79 (s, 2H), 4.21 (q, *J* = 7.1 Hz, 2H), 4.19 – 4.13 (m, 3H), 3.95 (s, 2H), 3.74 (dd, *J* = 8.5, 6.8 Hz, 2H), 3.15 – 3.05 (m, 2H), 1.26 (dd, *J* = 7.8, 5.5 Hz, 3H), 1.23 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.45, 168.78, 147.85,

137.03, 129.48, 128.30, 126.28, 122.36, 119.66, 119.24, 117.06, 113.70, 112.10, 109.17 , 61.79, 61.06, 53.36, 52.93, 47.87, 23.40, 14.37, 14.31. HRMS (ESI) Calcd. For $C_{24}H_{28}N_2O_4$(M+Na) m/z 431,1941, found 431.1926.

3p: Yield 80% (118 mg); orange oil. IR (neat): 3040, 2931, 2881, 2851, 2841, 1602, 1575, 1504, 1476, 1453, 1345, 1247, 1211, 1194 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.43 (dd, *J* = 8.0, 0.7 Hz, 1H), 7.08 (td, *J* = 8.2, 1.7 Hz, 1H), 7.02 (dd, *J* = 7.9, 7.0 Hz, 1H), 6.98 – 6.93 (m, 1H), 6.90 (dd, *J* = 7.1, 0.8 Hz, 1H), 6.89 (s, 1H), 6.72 (d, *J* = 8.1 Hz, 1H), 6.56 (td, *J* = 7.3, 1.0 Hz, 1H), 4.17 – 3.98 (m, 2H), 3.63 – 3.46 (m, 2H), 3.39 – 3.20 (m, 2H), 3.04 – 2.99 (m, 2H), 2.97 (t, *J* = 6.1 Hz, 2H), 2.75 (t, *J* = 6.4 Hz, 2H), 2.28 – 2.15 (m, 2H), 1.99 – 1.79 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 145.17, 134.68, 129.32, 127.29, 125.52, 123.89, 122.42, 121.86, 119.34, 118.58, 116.57, 115.50, 112.60, 110.66, 77.42, 77.16, 76.91, 52.64, 49.59, 44.00, 28.37, 24.85, 23.07, 22.39, 22.23. MS m/z: 316 (M^+ (20)), 170 (9), 146 (100), 130 (4). HRMS (ESI) Calcd. For $C_{22}H_{24}N_2$(M+1) m/z 317,2012, found 317.2006.

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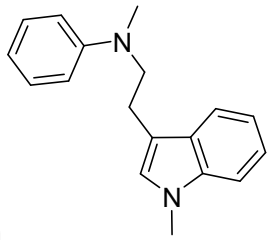
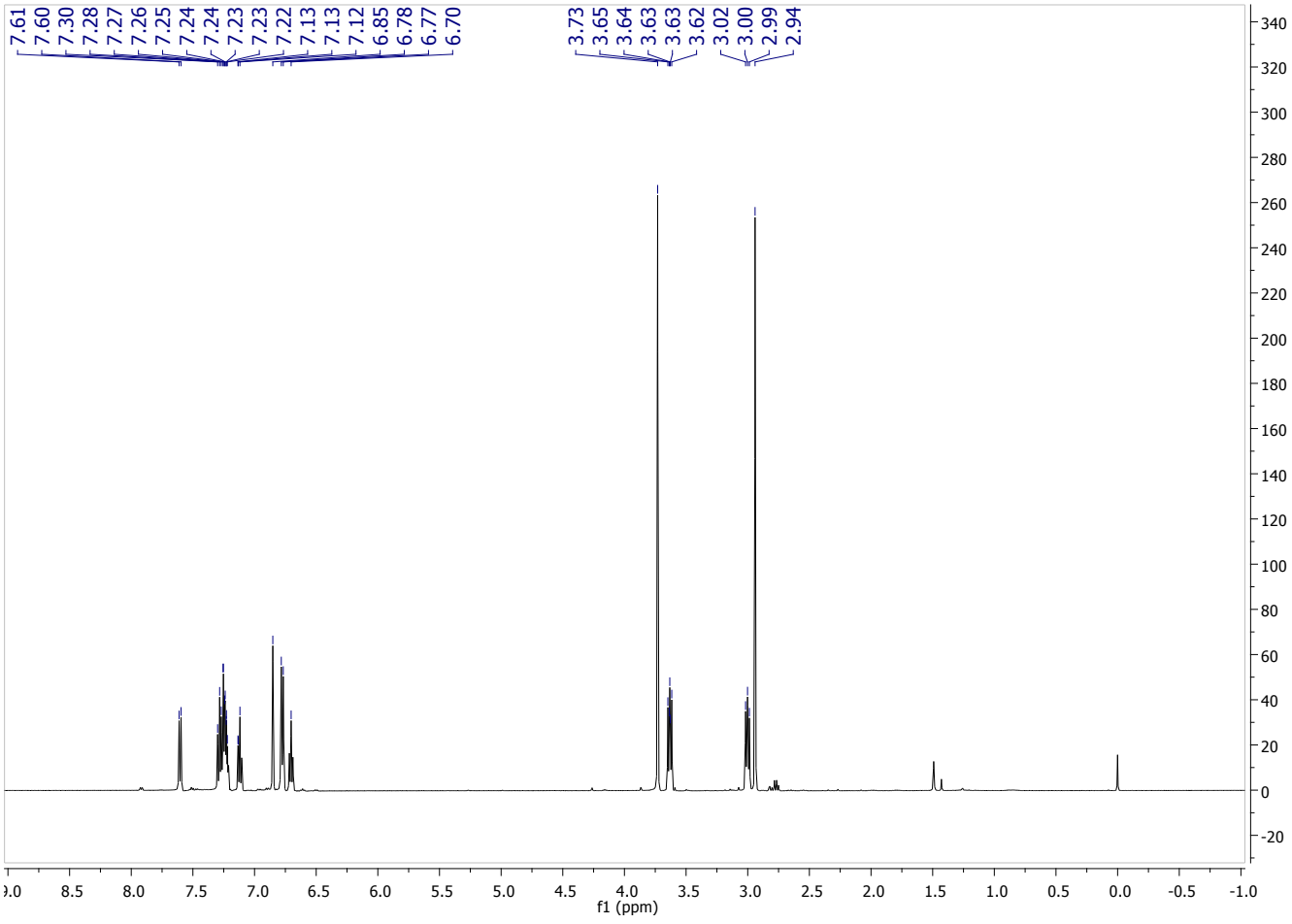
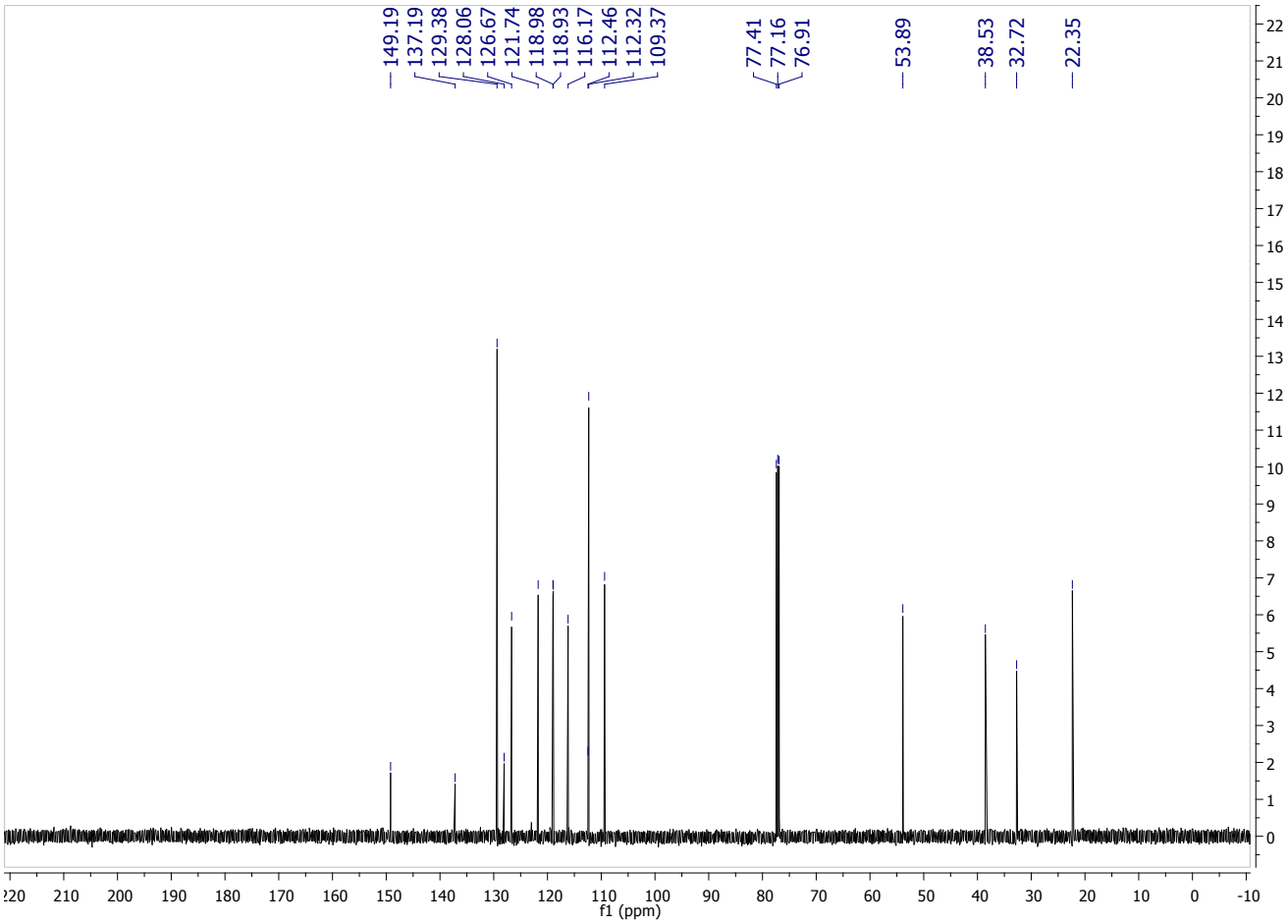
1) D. J. Aitken, P. Caboni, H. Eijsberg, A. Frongia, R. Guillot, J. Ollivier, P. P. Piras, F. Secci, *Adv. Synth. Catal.* 2014, **356**, 941.

Copies of NMR spectra



3a

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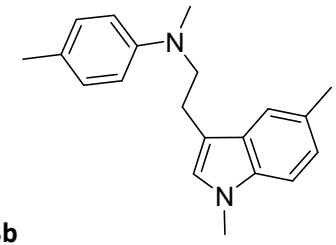
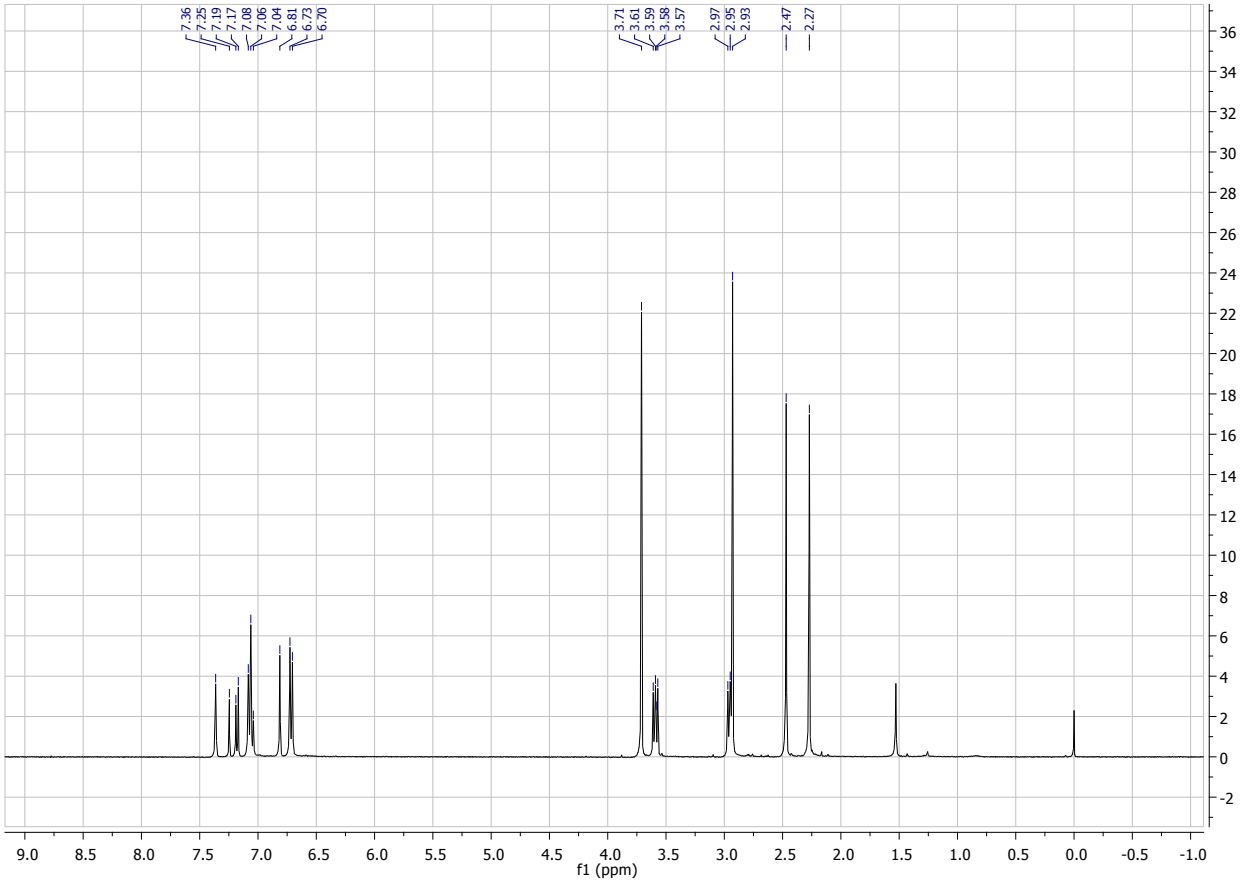


3b


7.36
7.25
7.19
7.17
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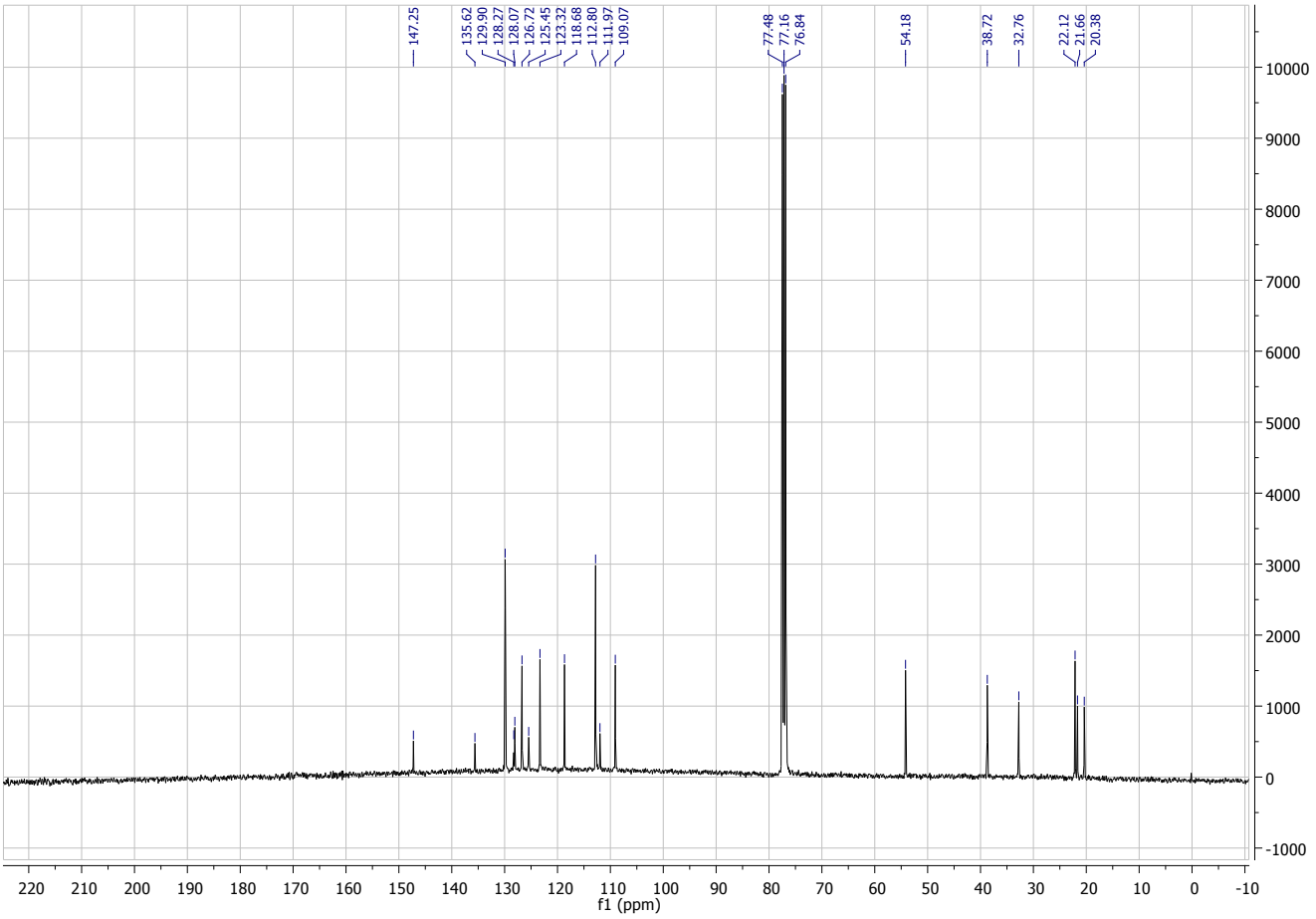
7.06
7.04
6.81
6.73
6.70
3.71
3.61
3.59
3.58
3.57
2.97
2.95
2.93
2.47
2.27
f1 (ppm)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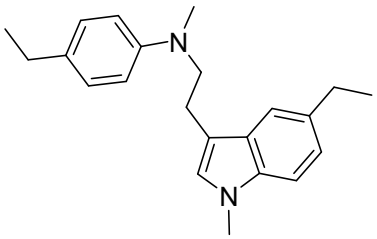
129.90
128.27
128.07
126.72
125.45
123.32
118.68
112.80
111.97
109.07
77.48
77.16
76.84
54.18
38.72
32.76
22.12
21.66
20.38
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



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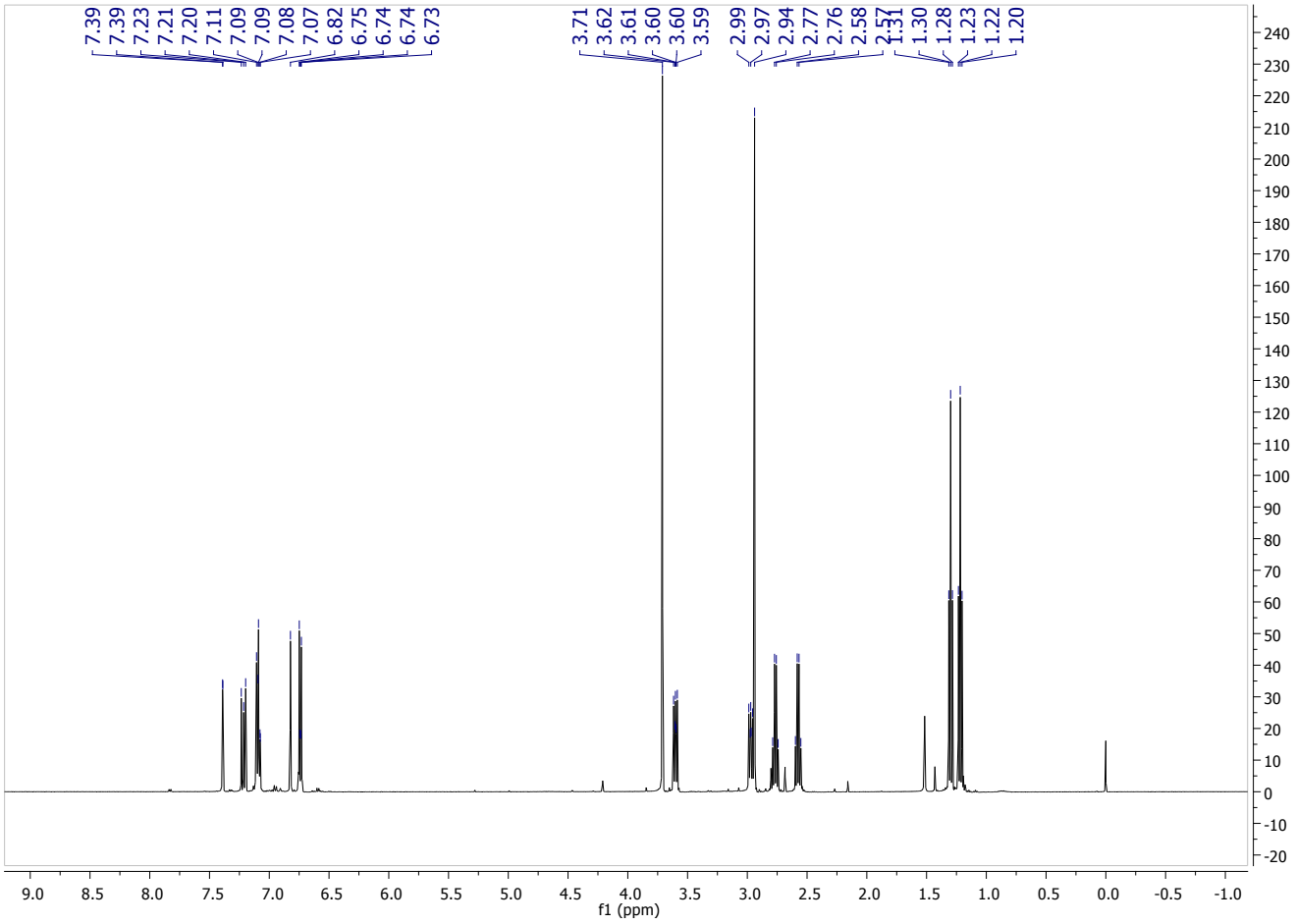
7.39
7.39
7.23
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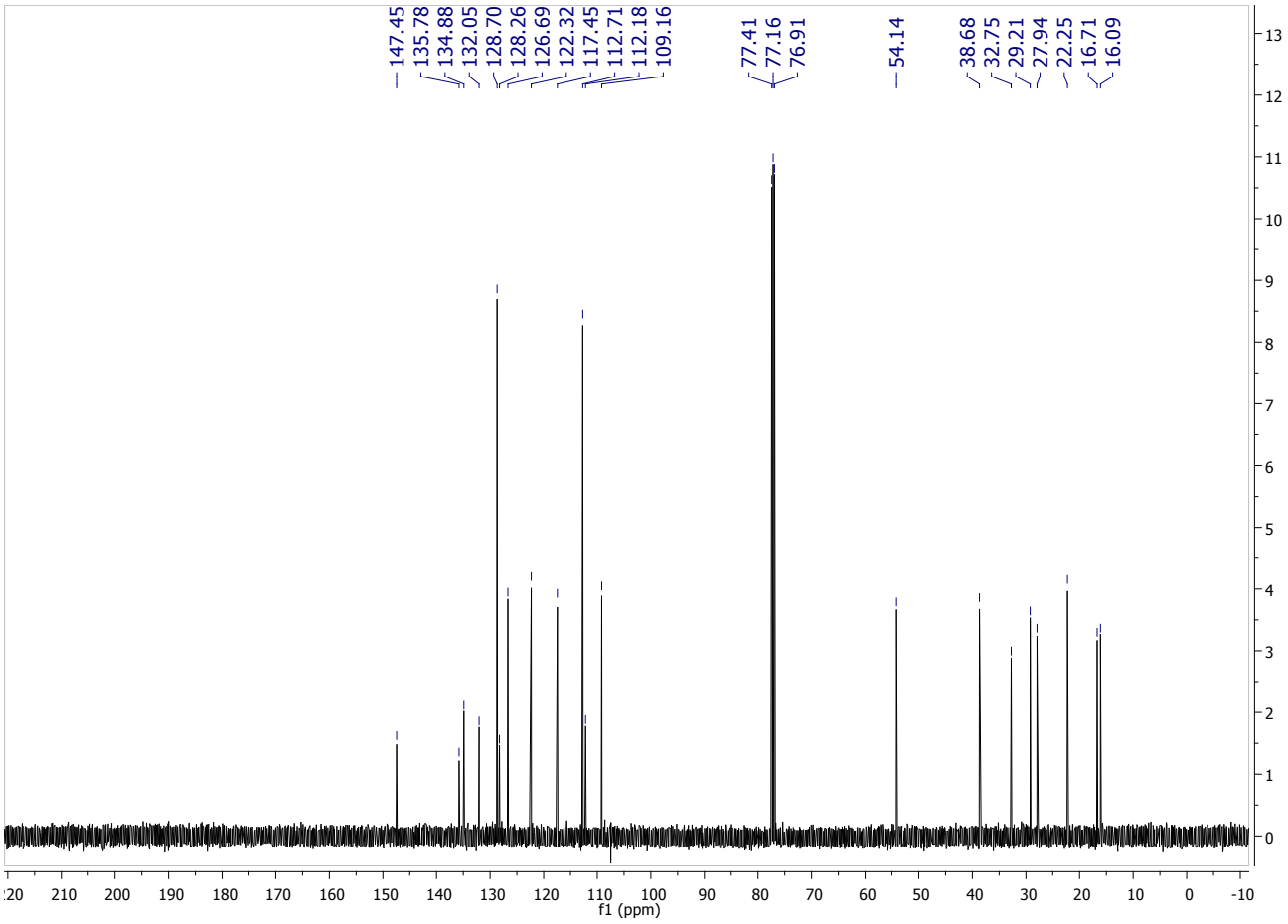
7.20
7.11
7.09
7.09
7.08
7.07
6.82
6.75
6.74
6.74
6.73
3.71
3.62
3.61
3.60
3.60
3.59
2.99
2.97
2.94
2.77
2.76
2.58
2.57
2.31
1.30
1.28
1.23
1.22
1.20
f1 (ppm)


147.45
135.78
134.88
132.05
128.70
128.26
126.69
122.32
117.45
112.71
112.18
109.16
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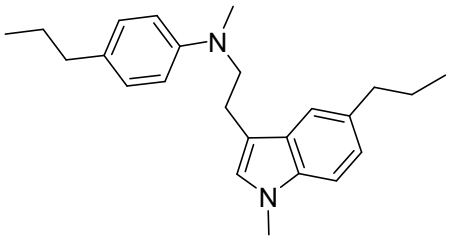
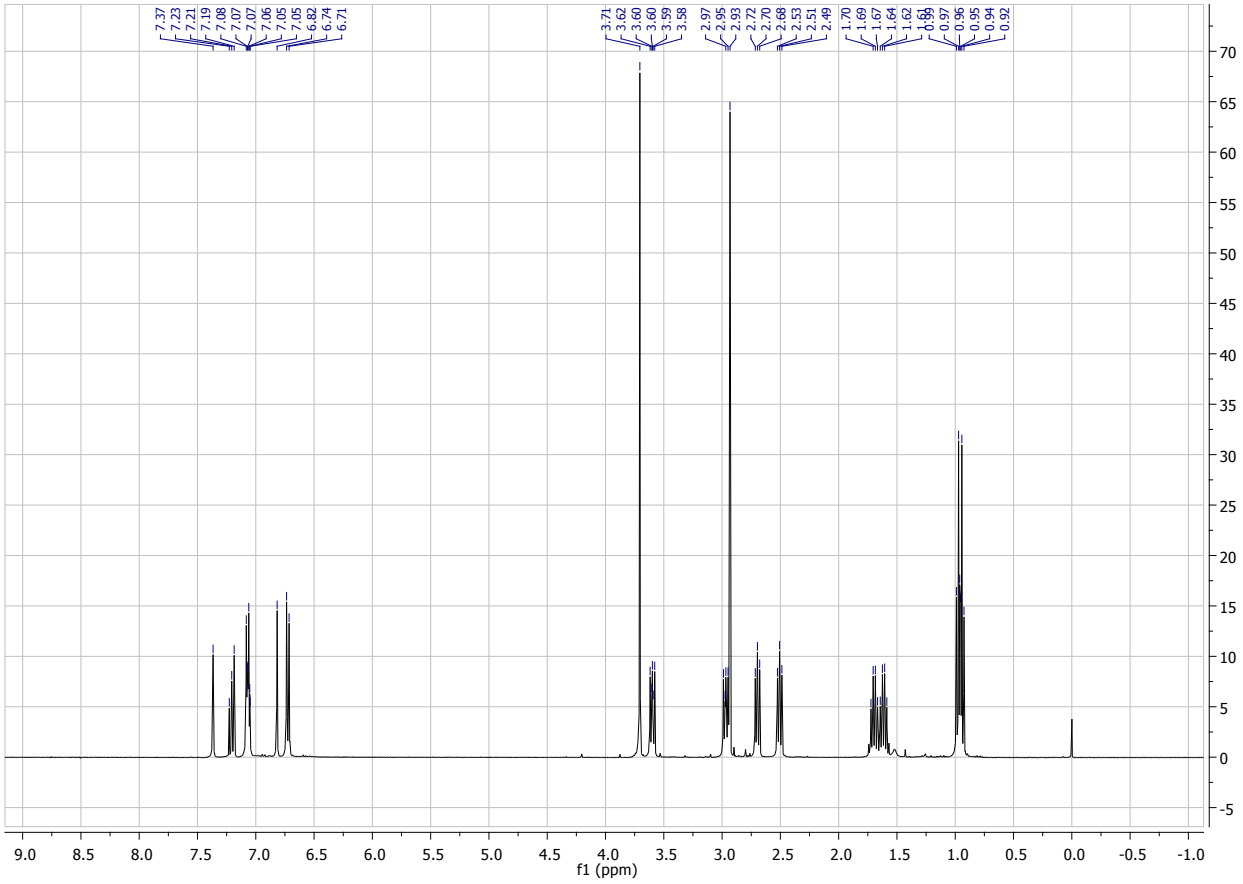
27.94
22.25
16.71
16.09
f1 (ppm)



3d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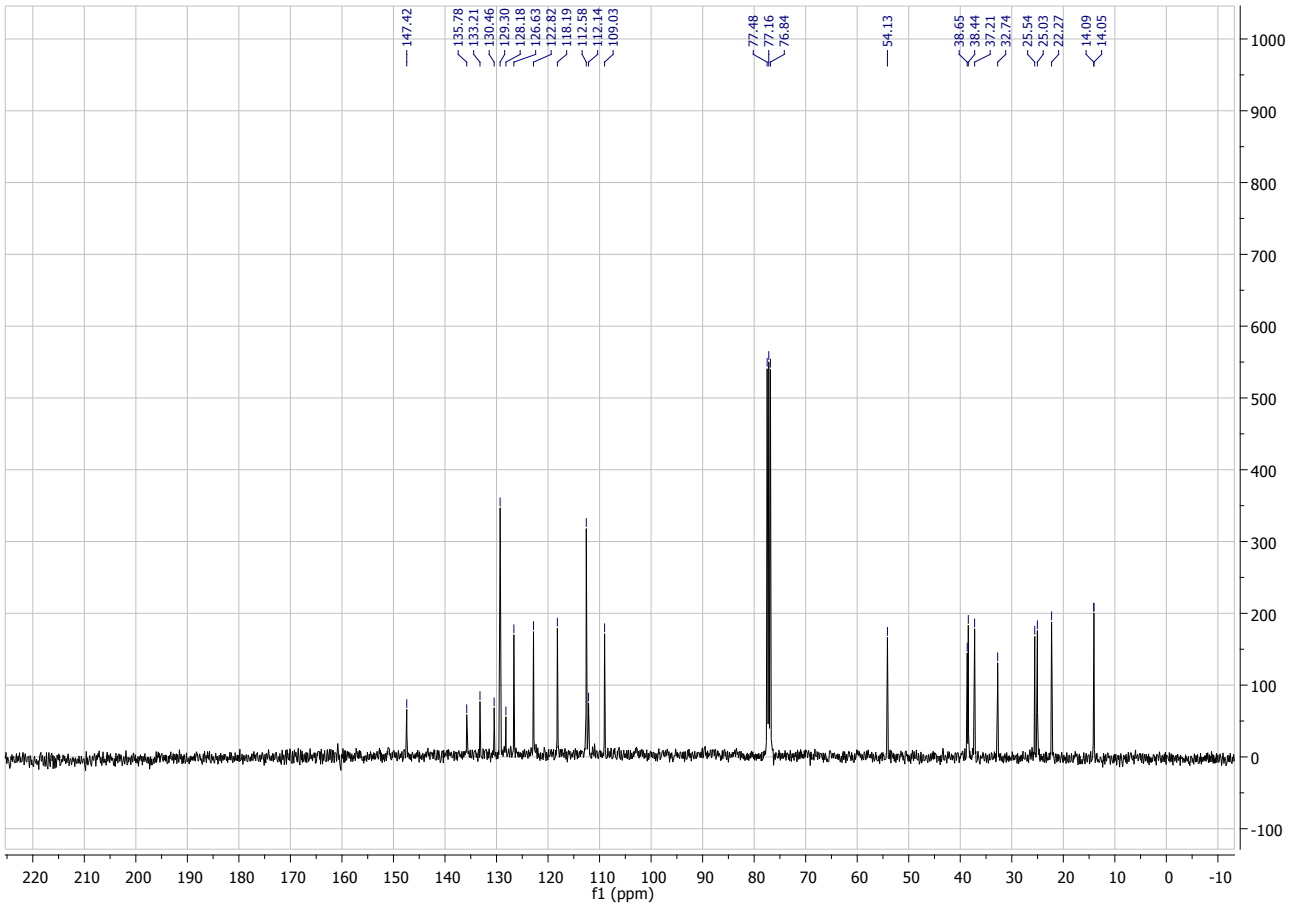
7.23
7.21
7.19
7.08
7.07
7.07
7.06
7.05
7.05
6.82
6.74
6.71
3.71
3.62
3.60
3.60
3.59
3.58
2.97
2.95
2.93
2.72
2.70
2.68
2.53
2.51
2.49
1.70
1.69
1.67
1.64
1.62
0.97
0.96
0.95
0.94
0.92
f1 (ppm)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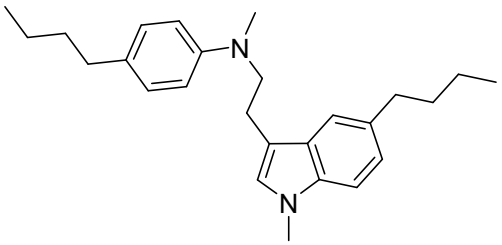
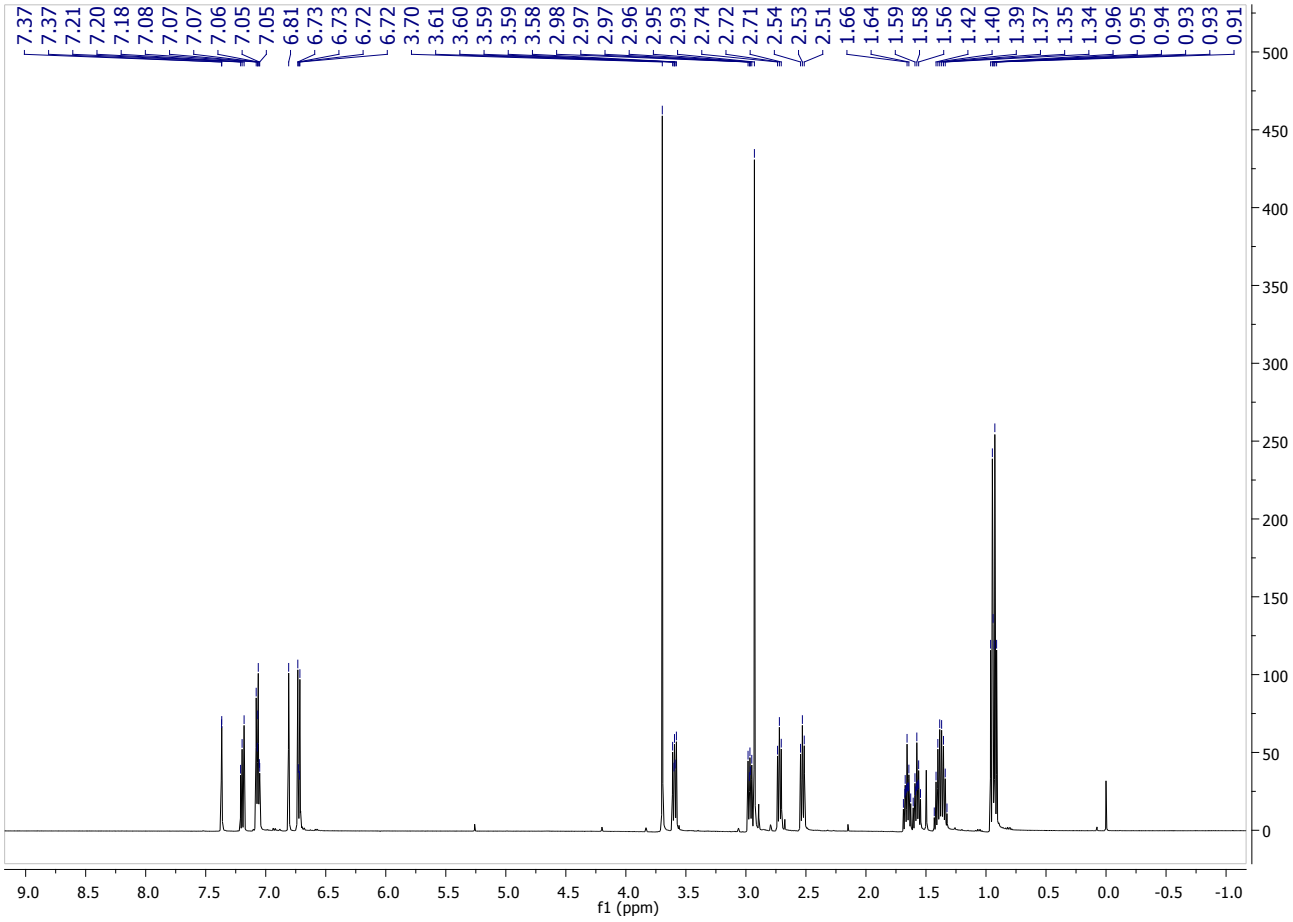
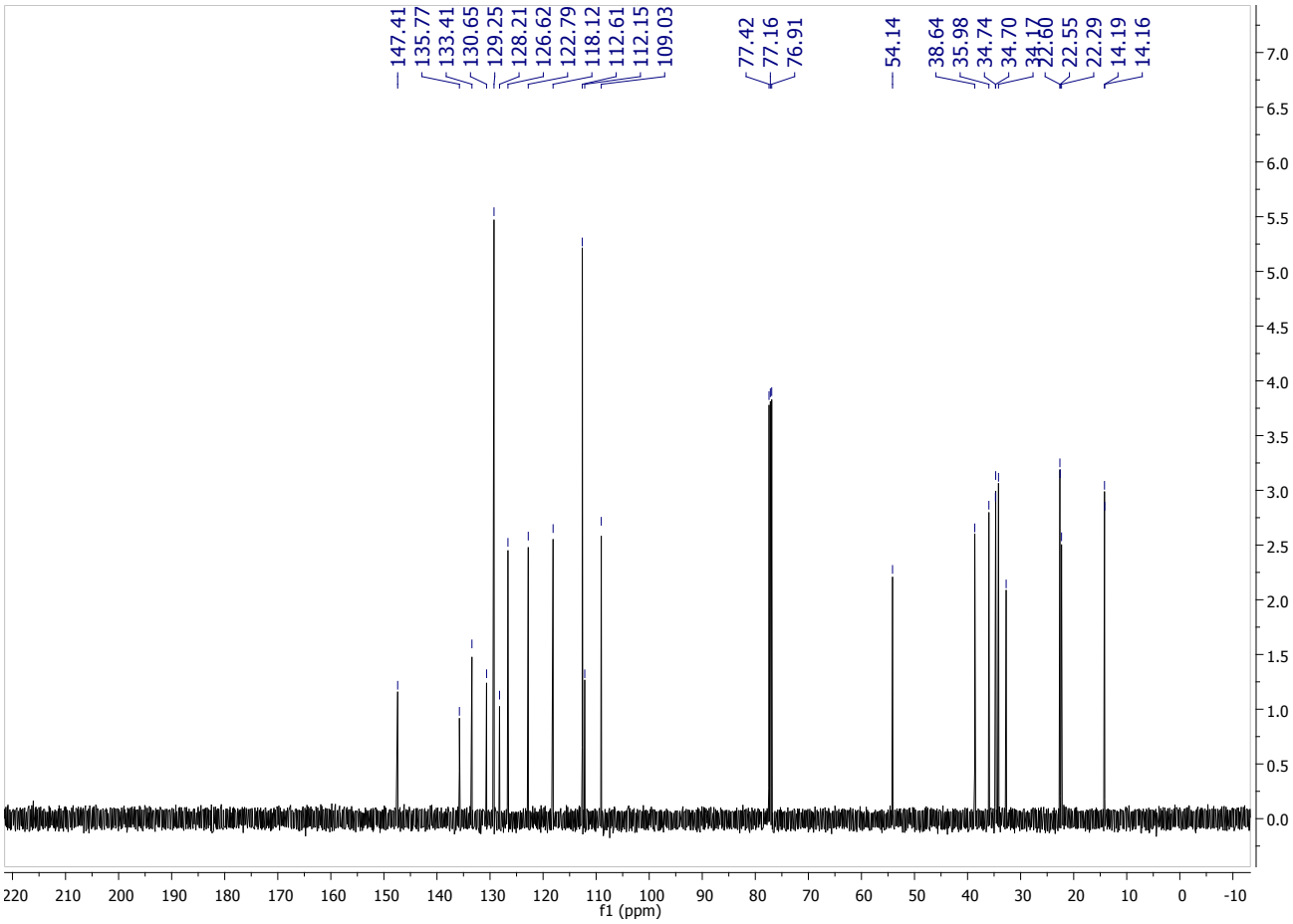
128.18
126.63
122.82
118.19
112.58
112.14
109.03
77.48
77.16
76.84
54.13
38.65
38.44
37.21
32.74
25.54
25.03
22.27
14.09
14.05
1000
900
800
700
600
500
400
300
200
100
0
-100
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



3e


7.37 7.37 7.21 7.20 7.18 7.08 7.07 7.07 7.06 7.05 7.05 6.81 6.73 6.73 6.72 6.72 3.70 3.61 3.60 3.59 3.59 3.58 2.98 2.97 2.97 2.96 2.95 2.93 2.74 2.72 2.71 2.54 2.53 2.51 1.66 1.64 1.59 1.58 1.56 1.42 1.40 1.39 1.37 1.35 1.34 0.96 0.95 0.94 0.93 0.93 0.91
500 450 400 350 300 250 200 150 100 50 0
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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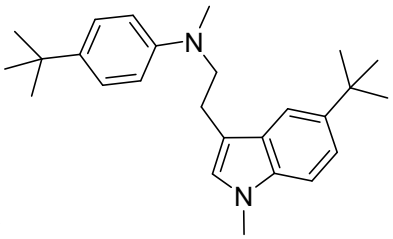
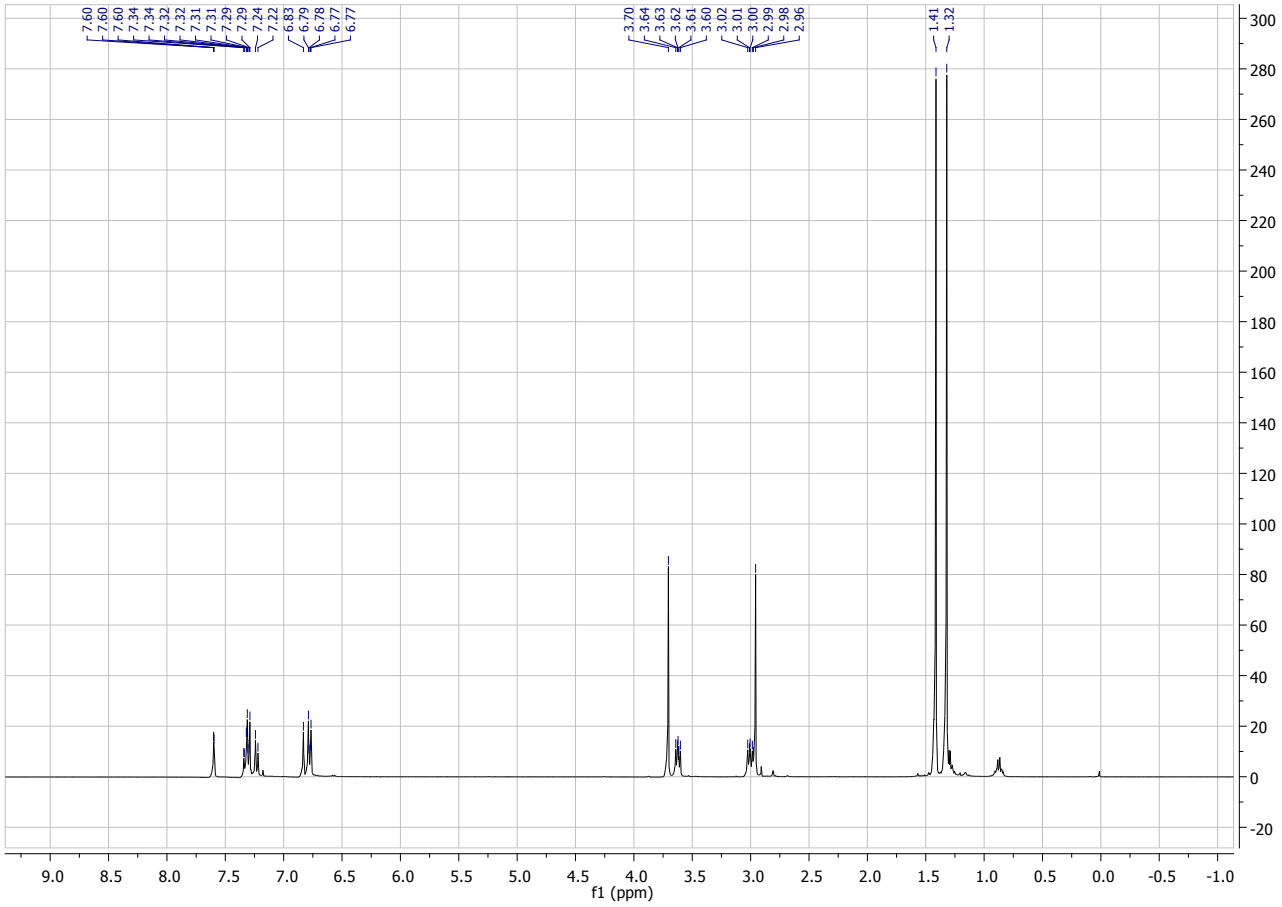
129.25
128.21
126.62
122.79
118.12
112.61
112.15
109.03
77.42
77.16
76.91
54.14
38.64
35.98
34.74
34.70
34.17
22.60
22.55
22.29
14.19
14.16
f1 (ppm)



3f


7.60
7.60
7.60
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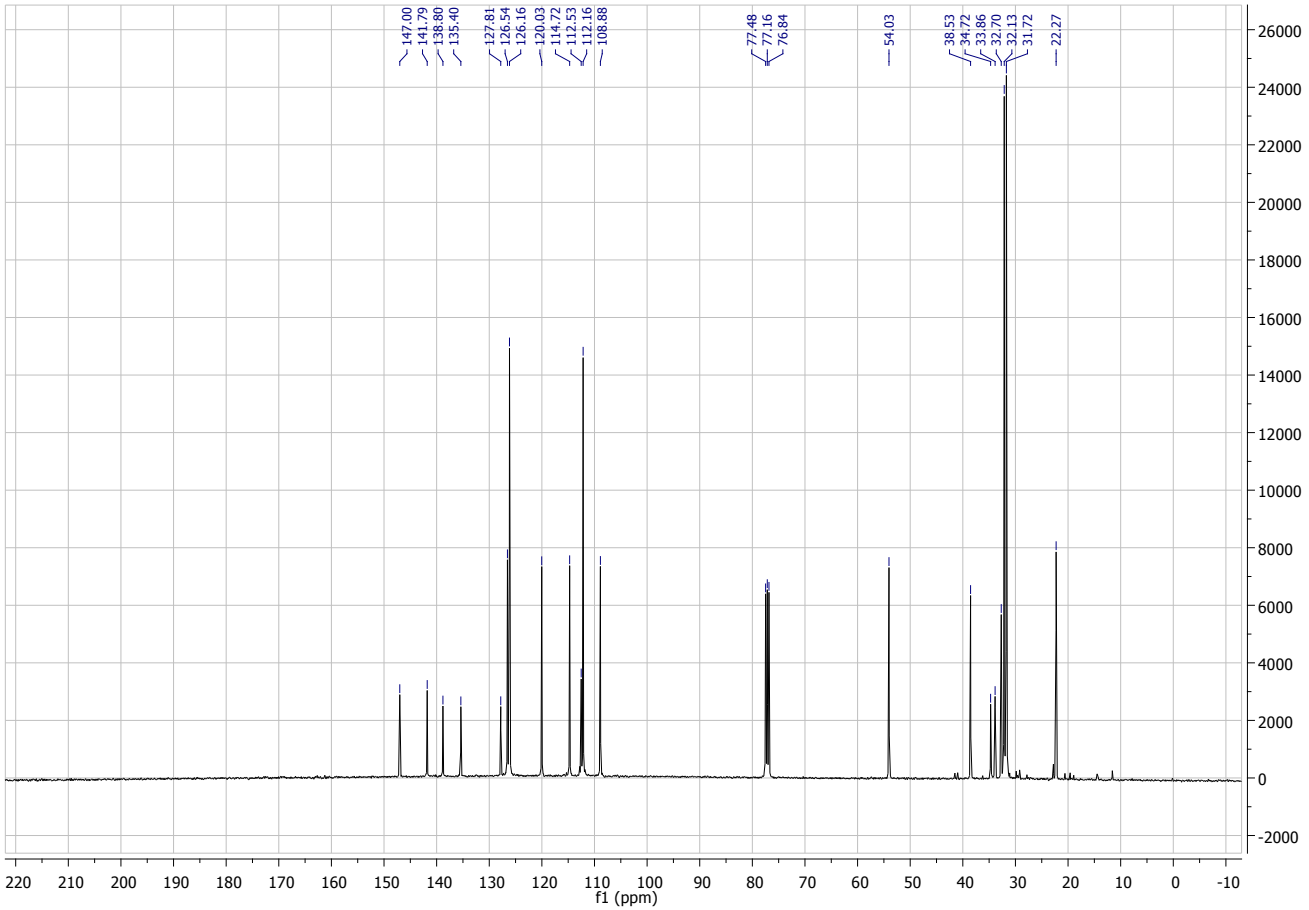
7.34
7.32
7.32
7.31
7.31
7.29
7.29
7.24
7.22
6.83
6.79
6.78
6.77
6.77
3.70
3.64
3.63
3.62
3.61
3.60
3.02
3.01
3.00
2.99
2.98
2.96
1.41
1.32
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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141.79
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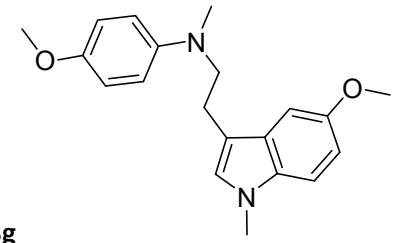
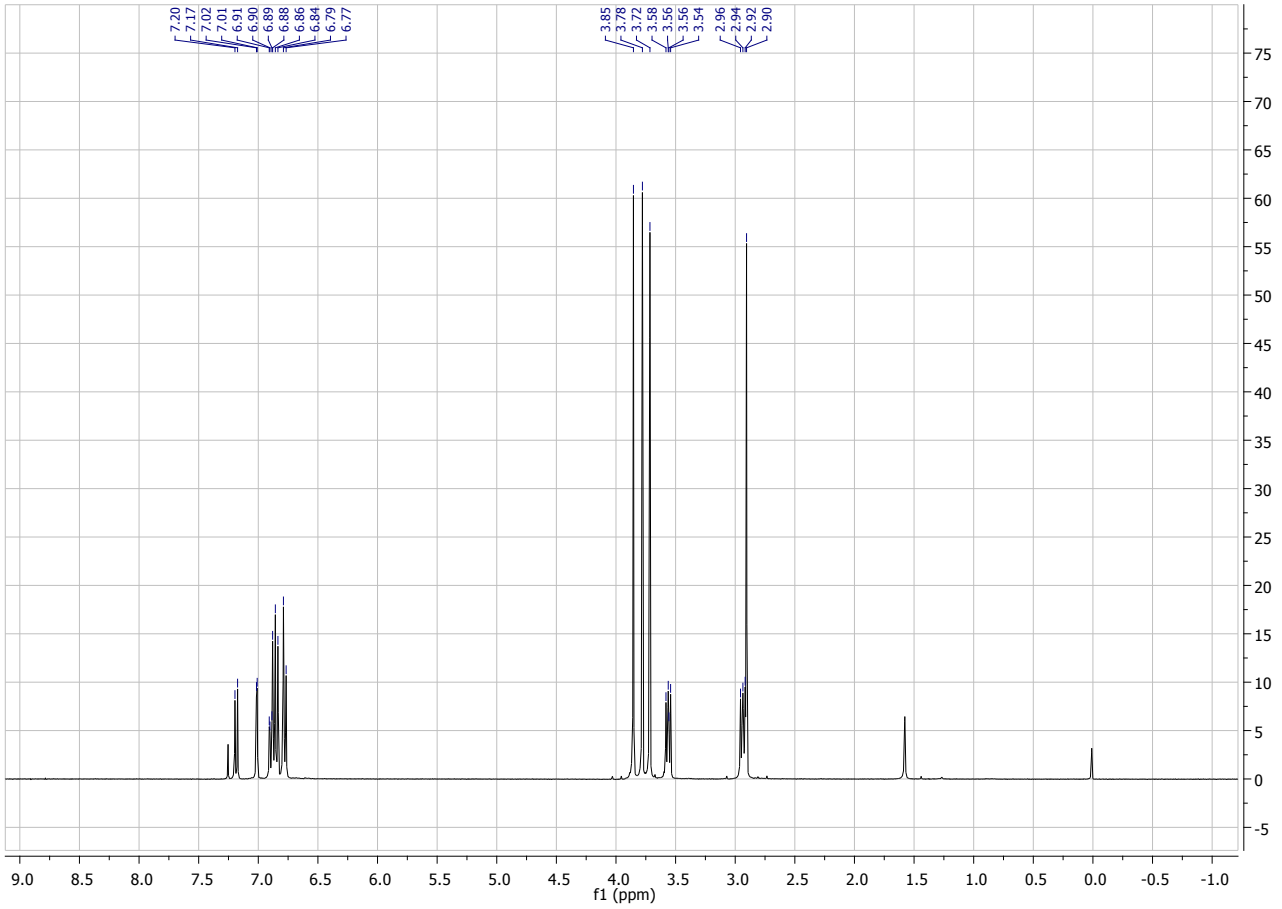
135.40
127.81
126.54
126.16
120.03
114.72
112.53
112.16
108.88
77.48
77.16
76.84
54.03
38.53
34.72
33.86
32.70
32.13
31.72
22.27
f1 (ppm)



3g


7.20
7.17
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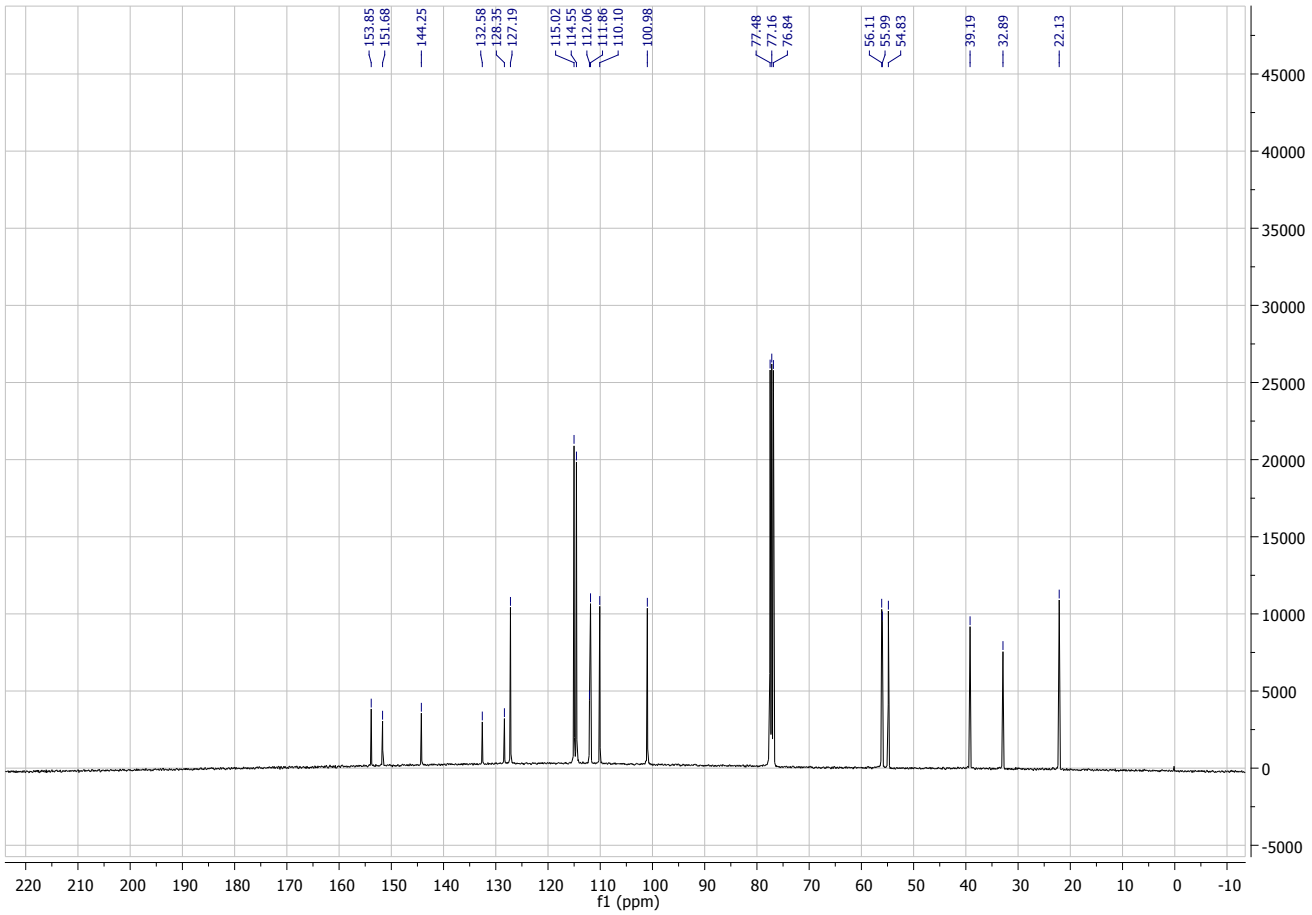
7.01
6.91
6.90
6.89
6.88
6.86
6.84
6.79
6.77
3.85
3.78
3.72
3.58
3.56
3.56
3.54
2.96
2.94
2.92
2.90
f1 (ppm)


153.85
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144.25
132.58
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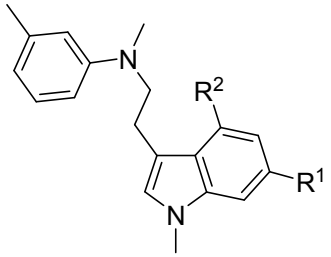
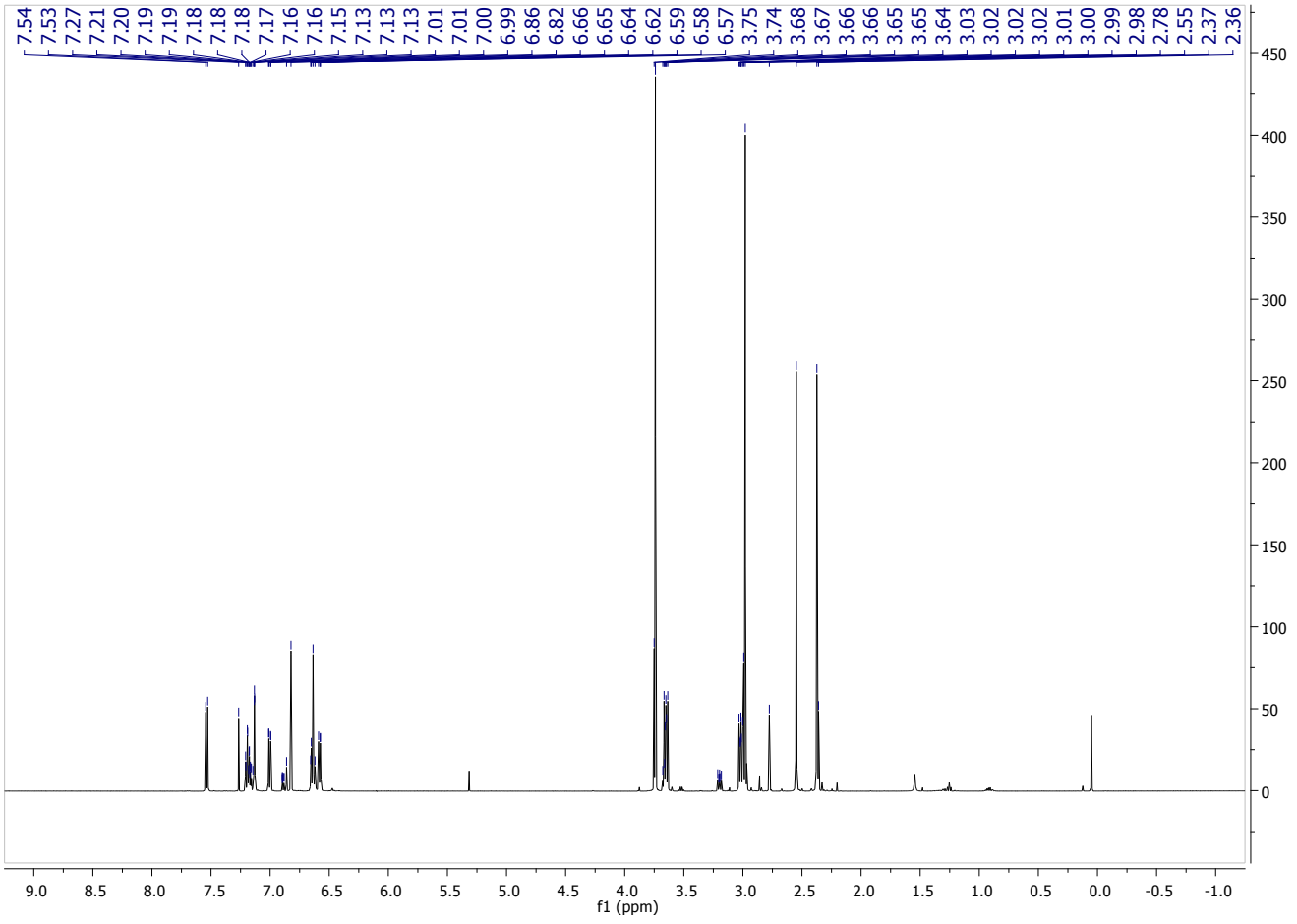
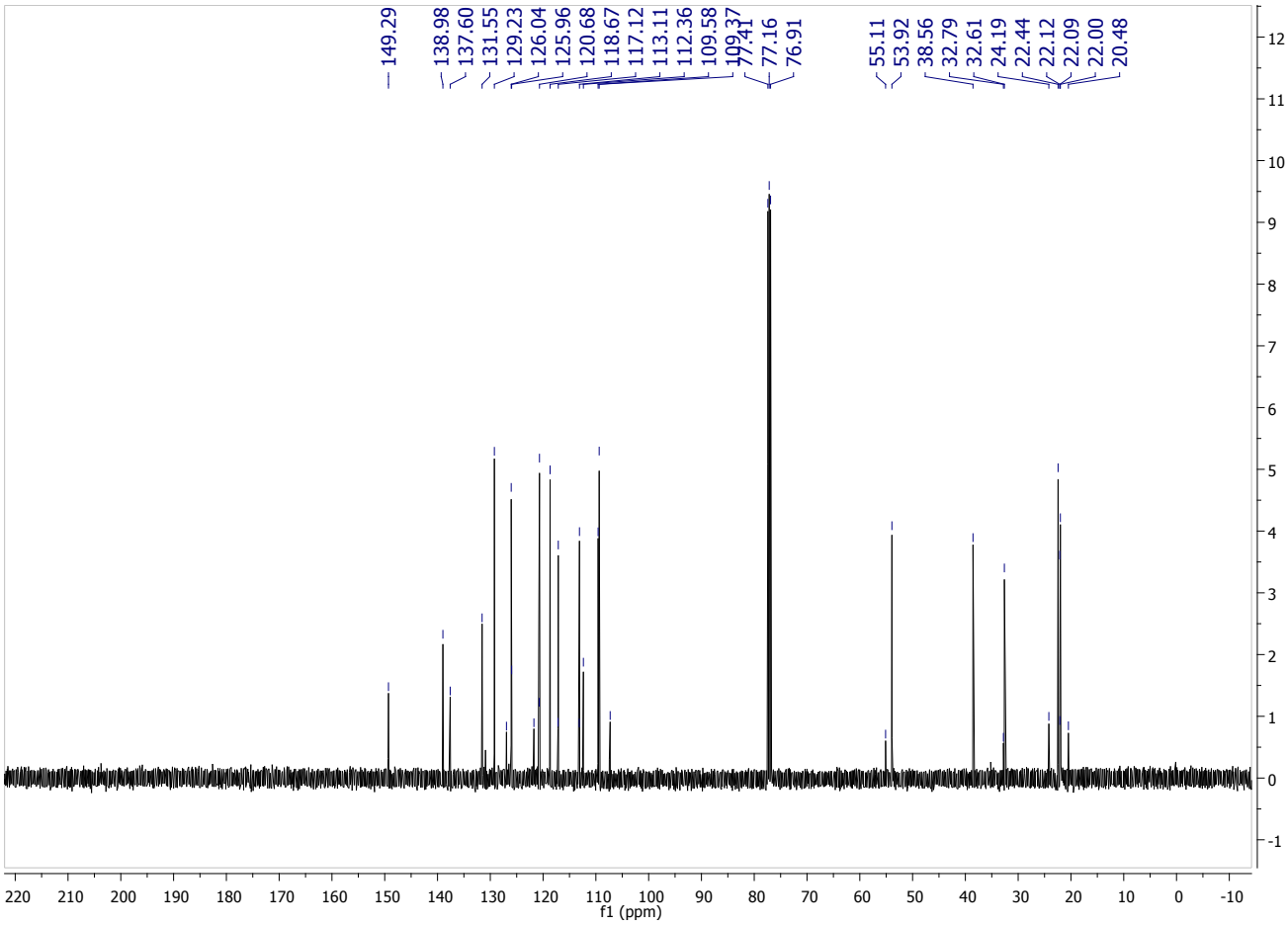
127.19
115.02
114.55
112.06
111.86
110.10
100.98
77.48
77.16
76.84
56.11
55.99
54.83
39.19
32.89
22.13
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



3h R^1=Me, R^2=H + 3h^1 R^1=H, R_2=Me

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131.55
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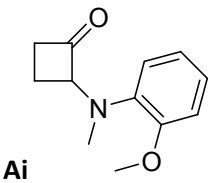
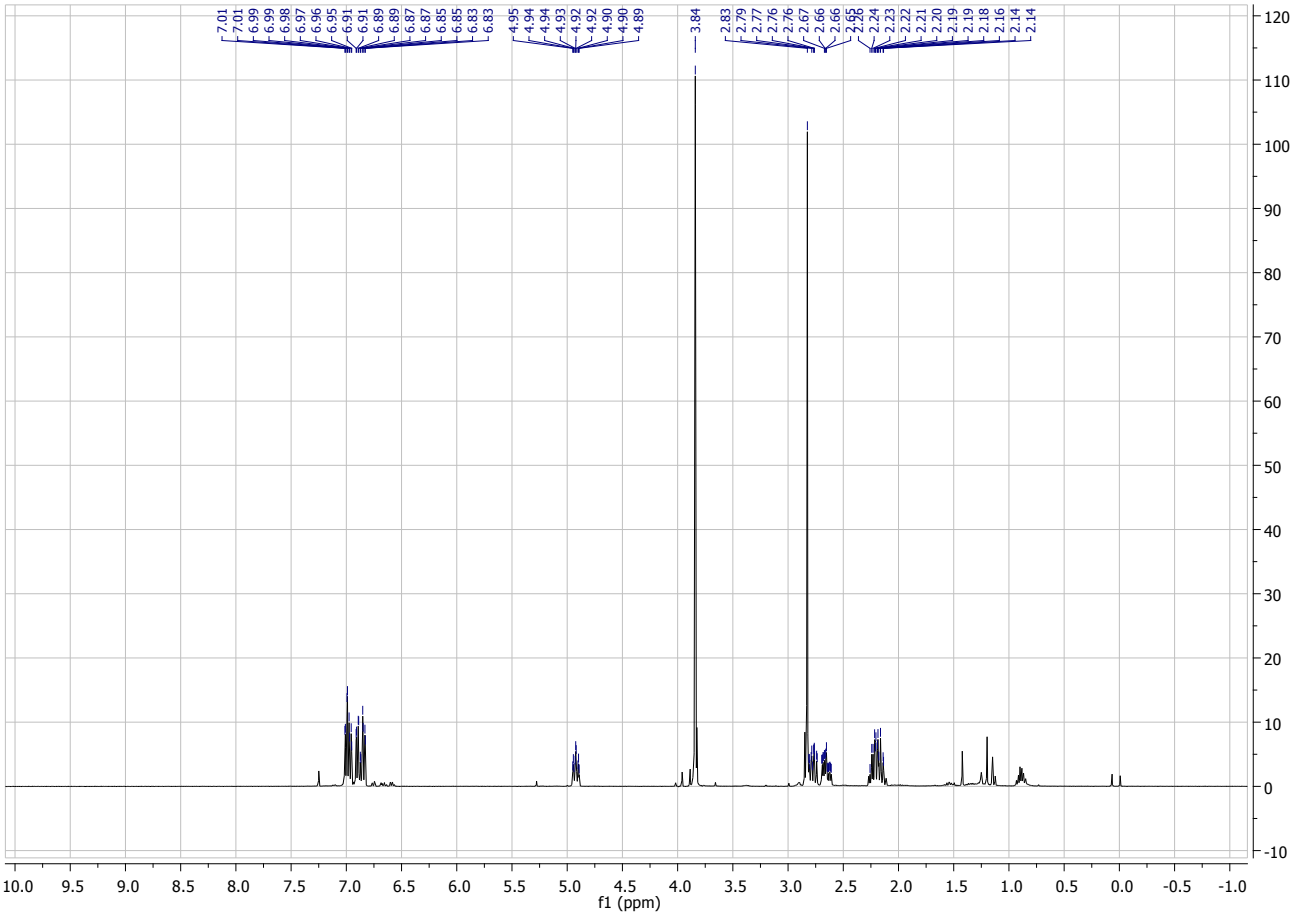
126.04
125.96
120.68
118.67
117.12
113.11
112.36
109.58
77.41
77.16
76.91
55.11
53.92
38.56
32.79
32.61
24.19
22.44
22.12
22.09
22.00
20.48
f1 (ppm)


O
N
O

Ai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7.01
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6.91
6.89
6.89
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6.85
6.85
6.83
6.83
4.95
4.94
4.94
4.93
4.92
4.92
4.90
4.90
4.89
3.84
2.83
2.79
2.77
2.76
2.76
2.67
2.66
2.66
2.24
2.23
2.22
2.21
2.20
2.19
2.19
2.18
2.16
2.14
2.14
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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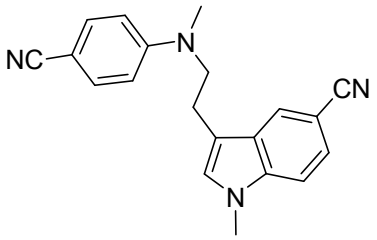
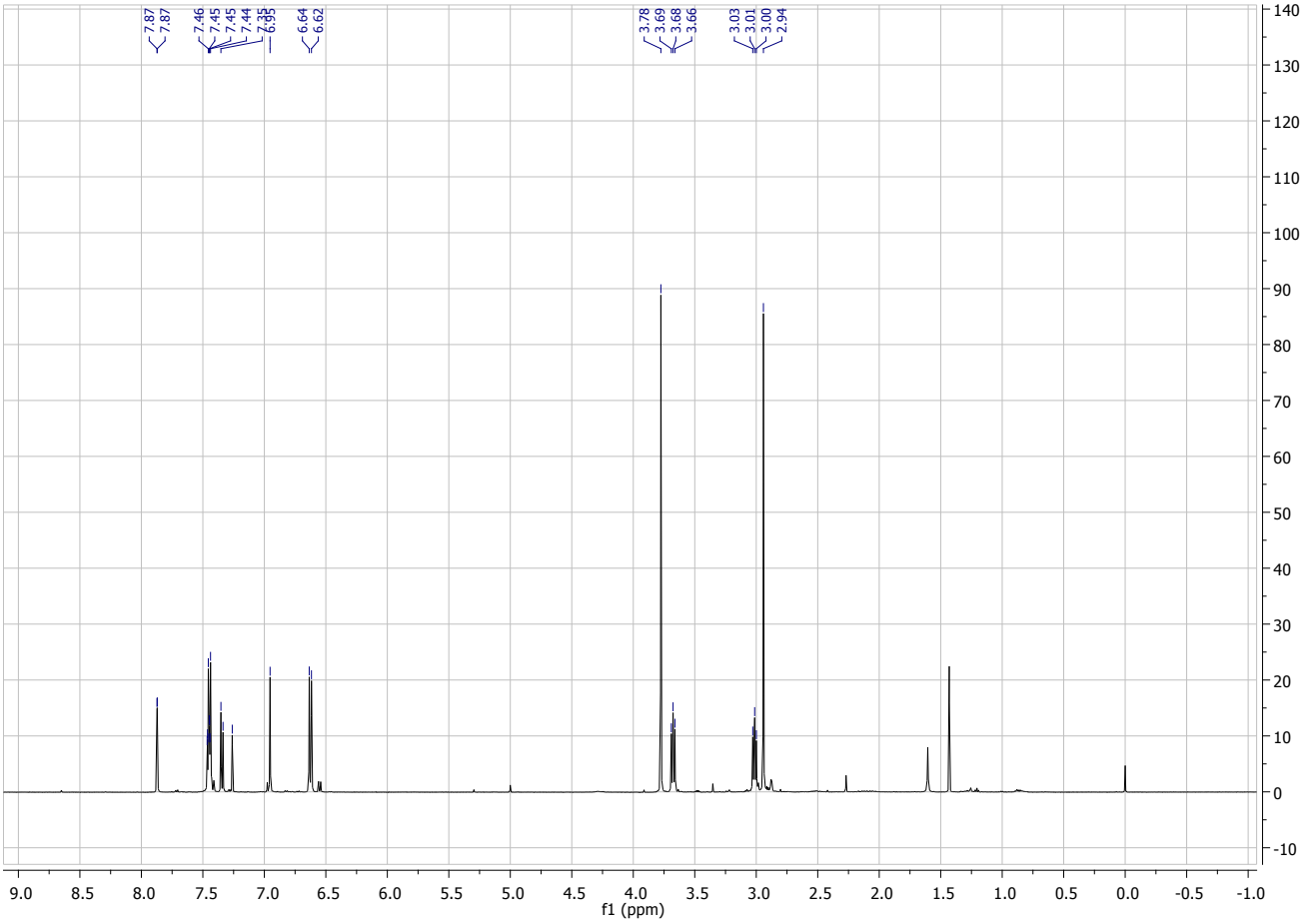
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


NC
N
CN
N

3j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7.87
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7.45
7.44
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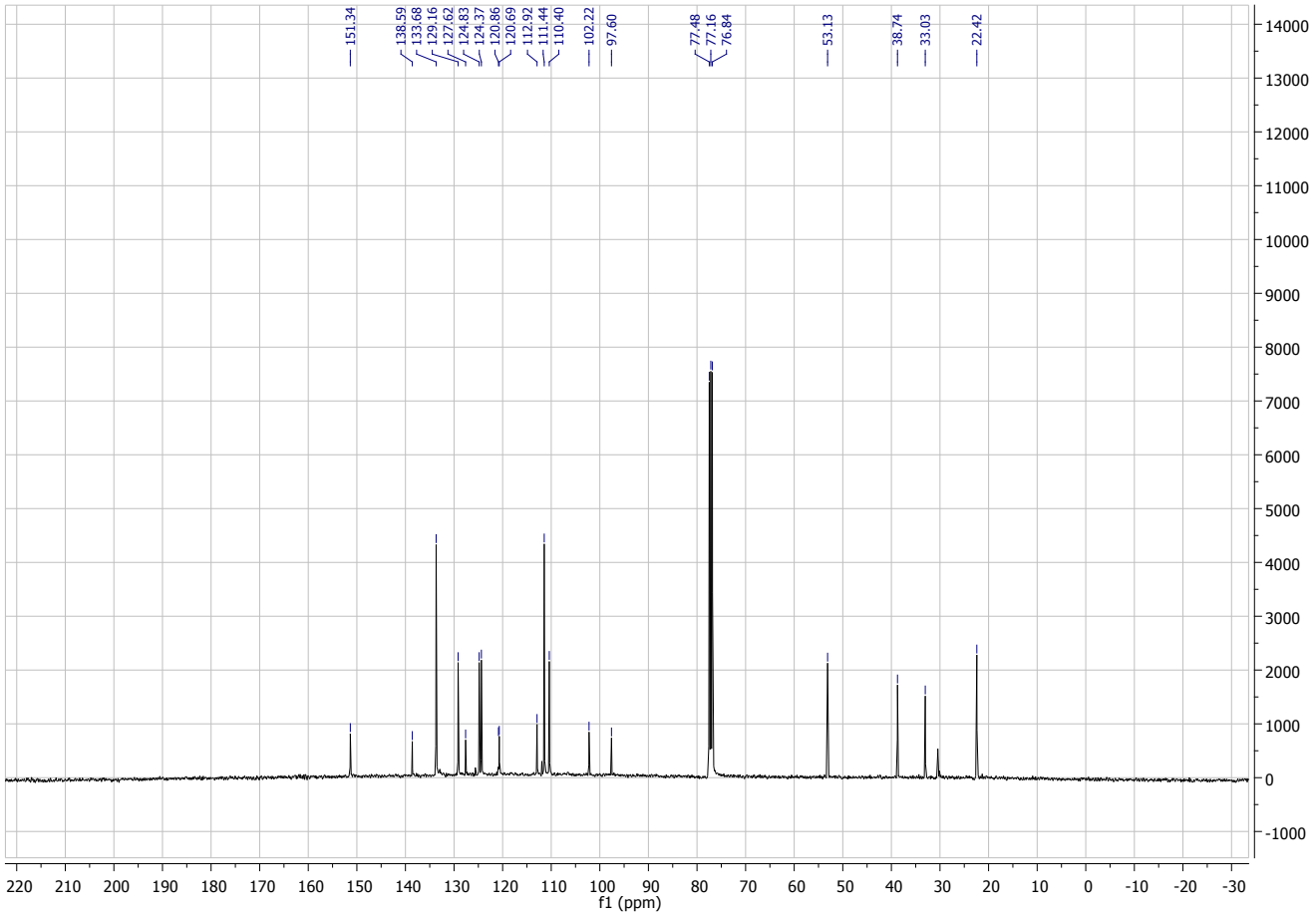
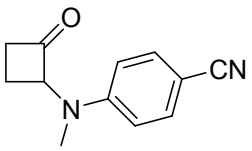
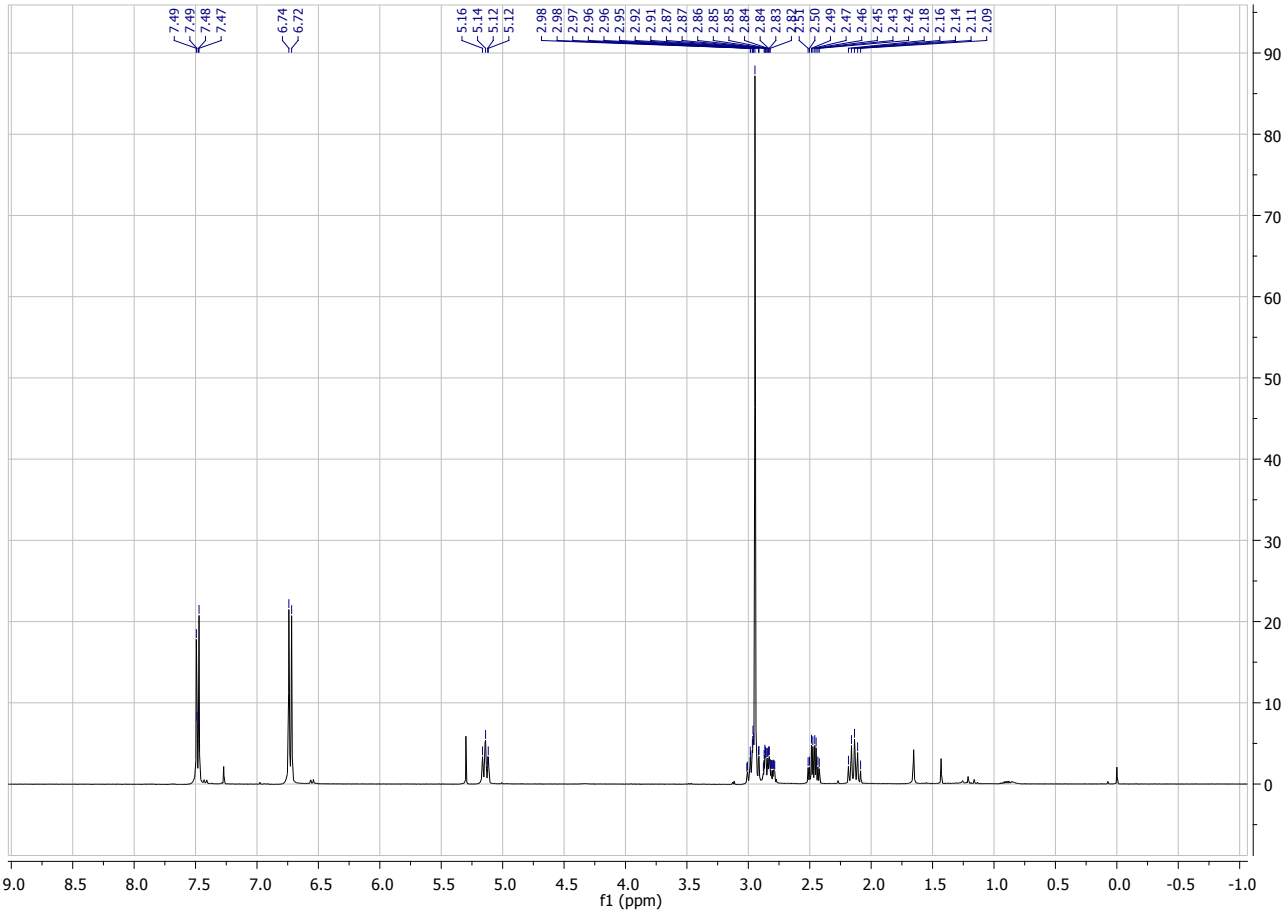
6.64
6.62
3.78
3.69
3.68
3.66
3.03
3.01
3.00
2.94
f1 (ppm)


151.34
138.59
133.68
129.16
127.62
124.83
124.37
120.86
120.69
112.92
111.44
110.40
102.22
97.60
77.48
77.16
76.84
53.13
38.74
33.03
22.42
f1 (ppm)


O
CN
N

Aj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6.72
5.16
5.14
5.12
5.12
2.98
2.98
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2.96
2.96
2.95
2.92
2.91
2.87
2.87
2.86
2.85
2.85
2.84
2.84
2.83
2.82
2.51
2.50
2.49
2.47
2.46
2.45
2.43
2.42
2.18
2.16
2.14
2.11
2.09
90
80
70
60
50
40
30
20
10
0
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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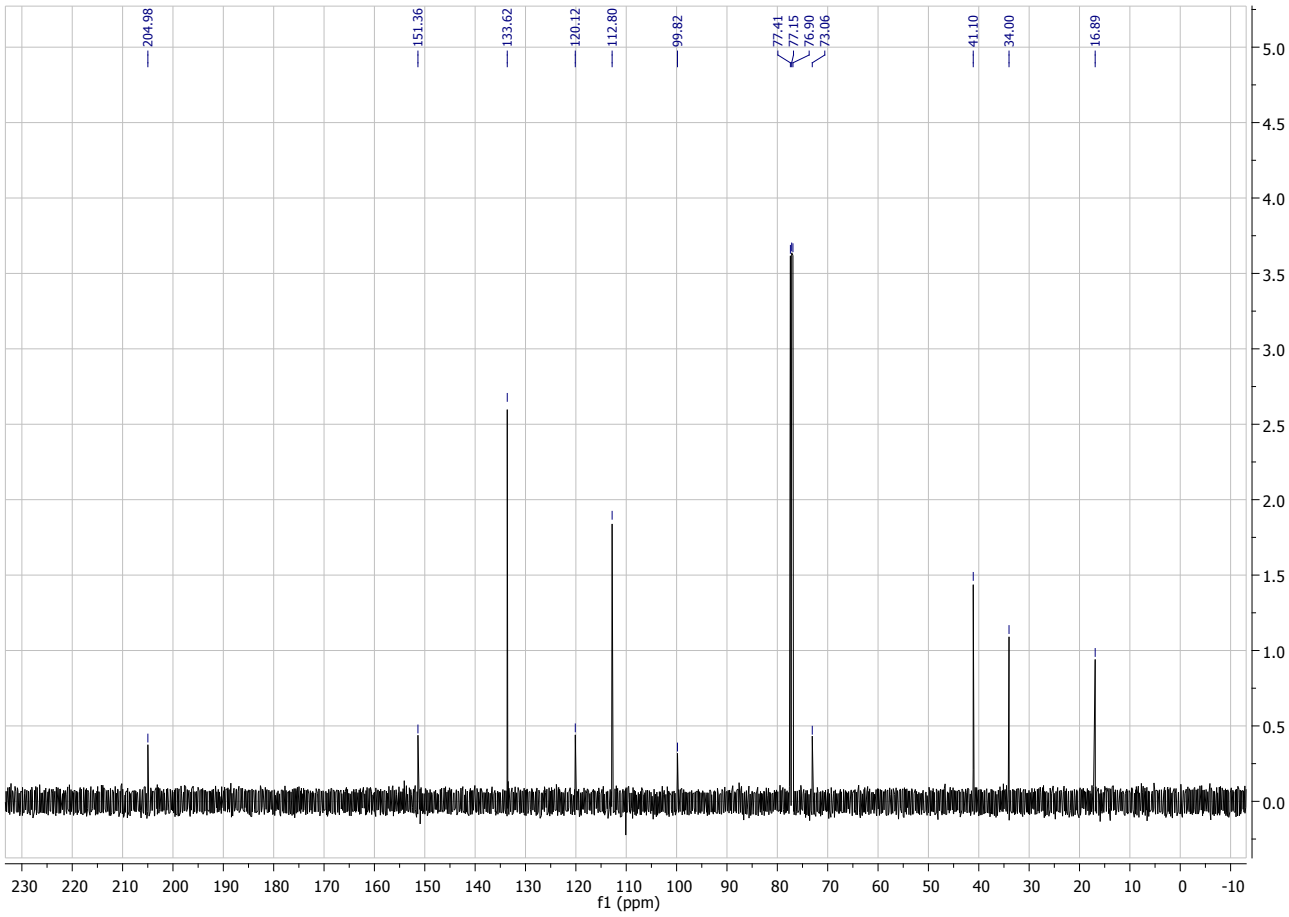
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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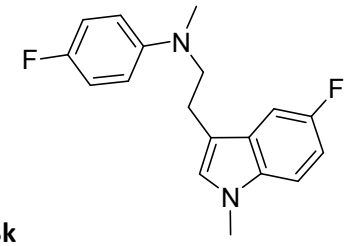
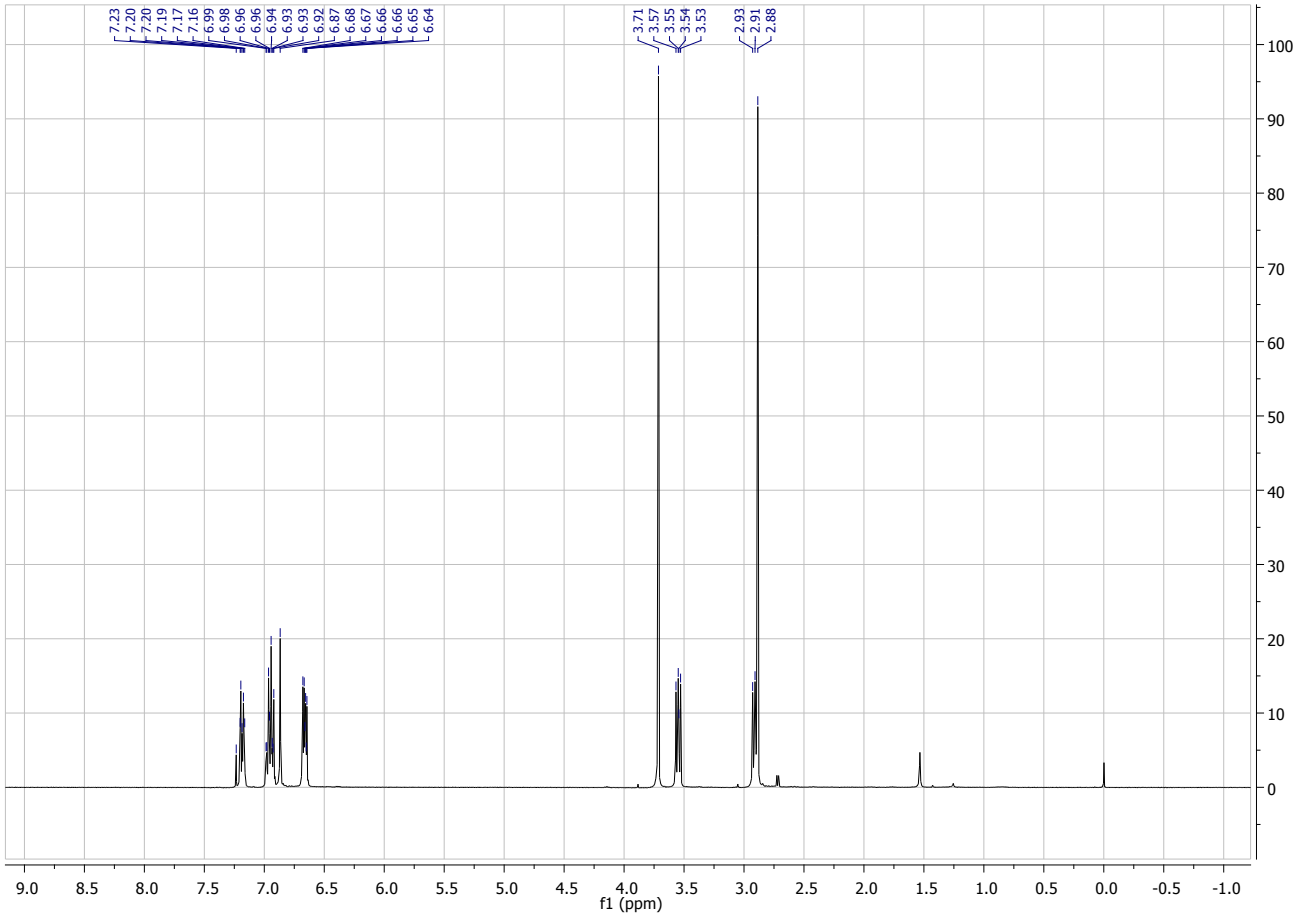
99.82
77.41
77.15
76.90
73.06
41.10
34.00
16.89
f1 (ppm)


F
N
F
N

3k


7.23
7.20
7.20
7.19
7.17
7.16
6.99
6.98
6.96
6.96
6.94
6.93
6.93
6.92
6.87
6.68
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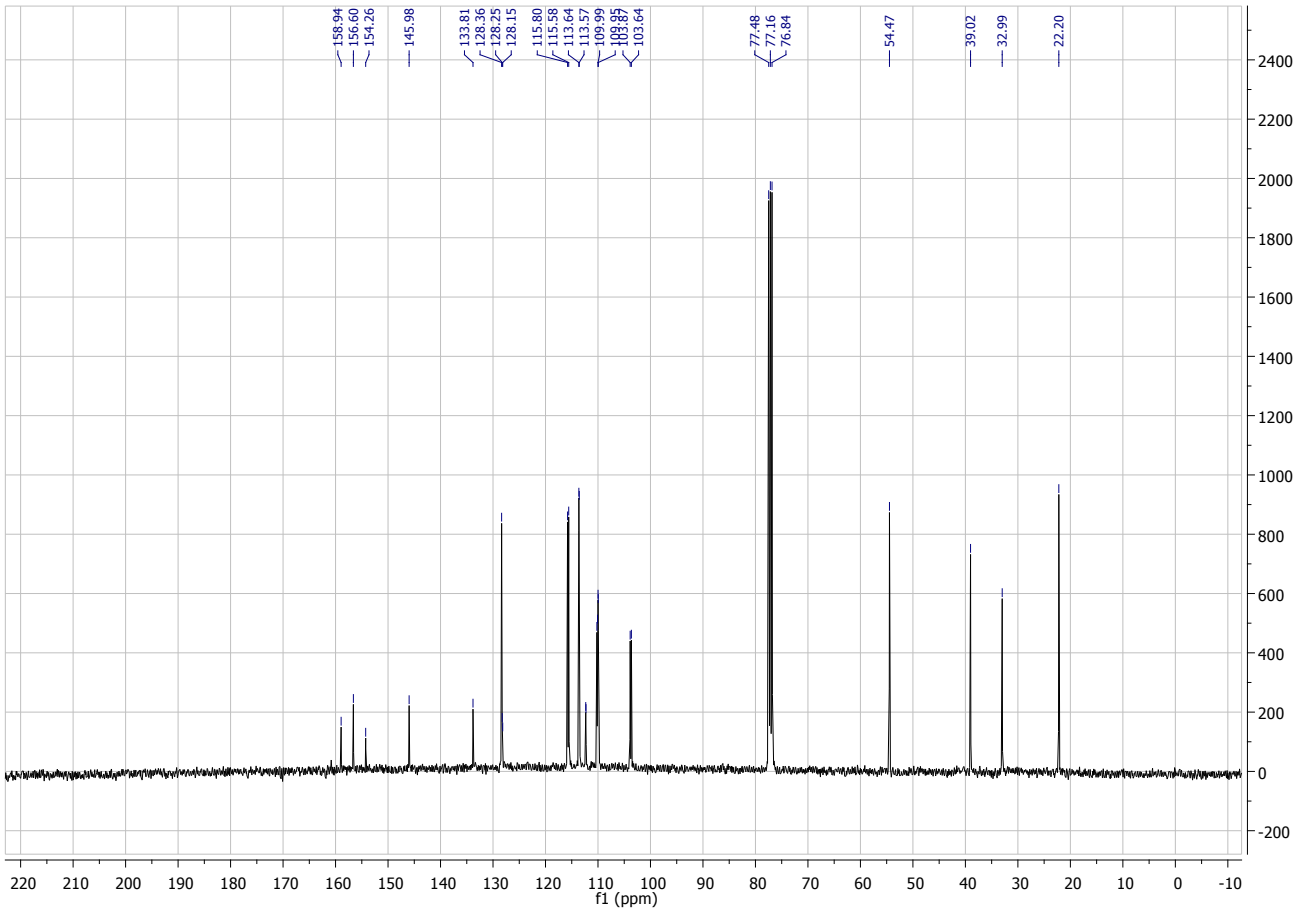
6.66
6.66
6.65
6.64
3.71
3.57
3.55
3.54
3.53
2.93
2.91
2.88
100
90
80
70
60
50
40
30
20
10
0
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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128.25
128.15
115.80
115.58
113.64
113.57
109.99
103.85
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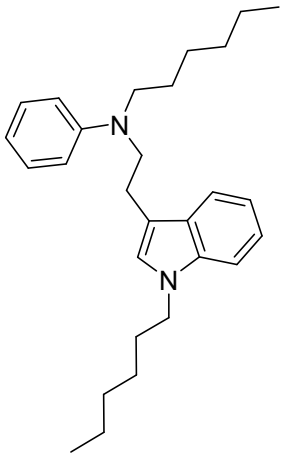
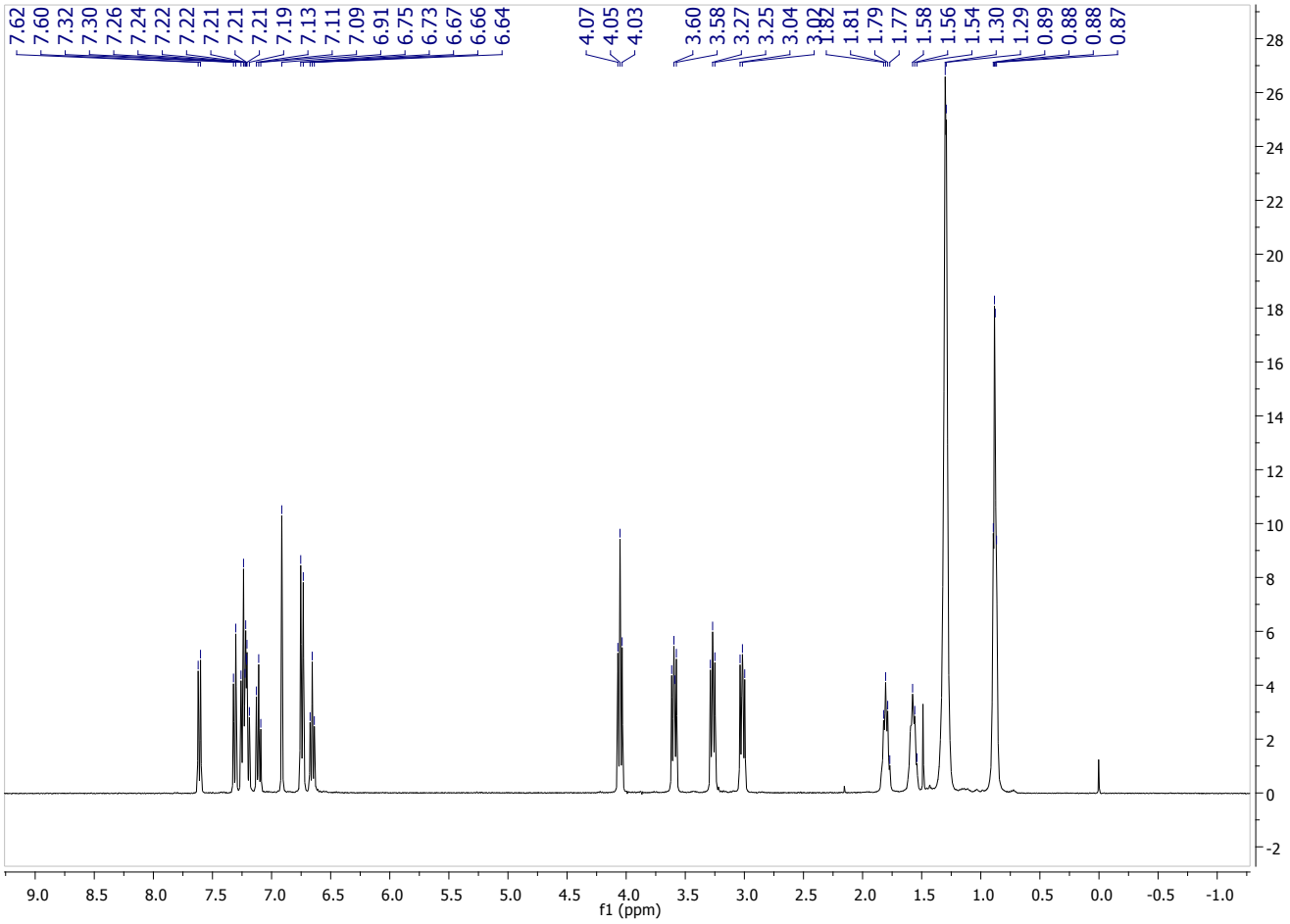
103.64
77.48
77.16
76.84
54.47
39.02
32.99
22.20
f1 (ppm)



3l


7.62
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7.32
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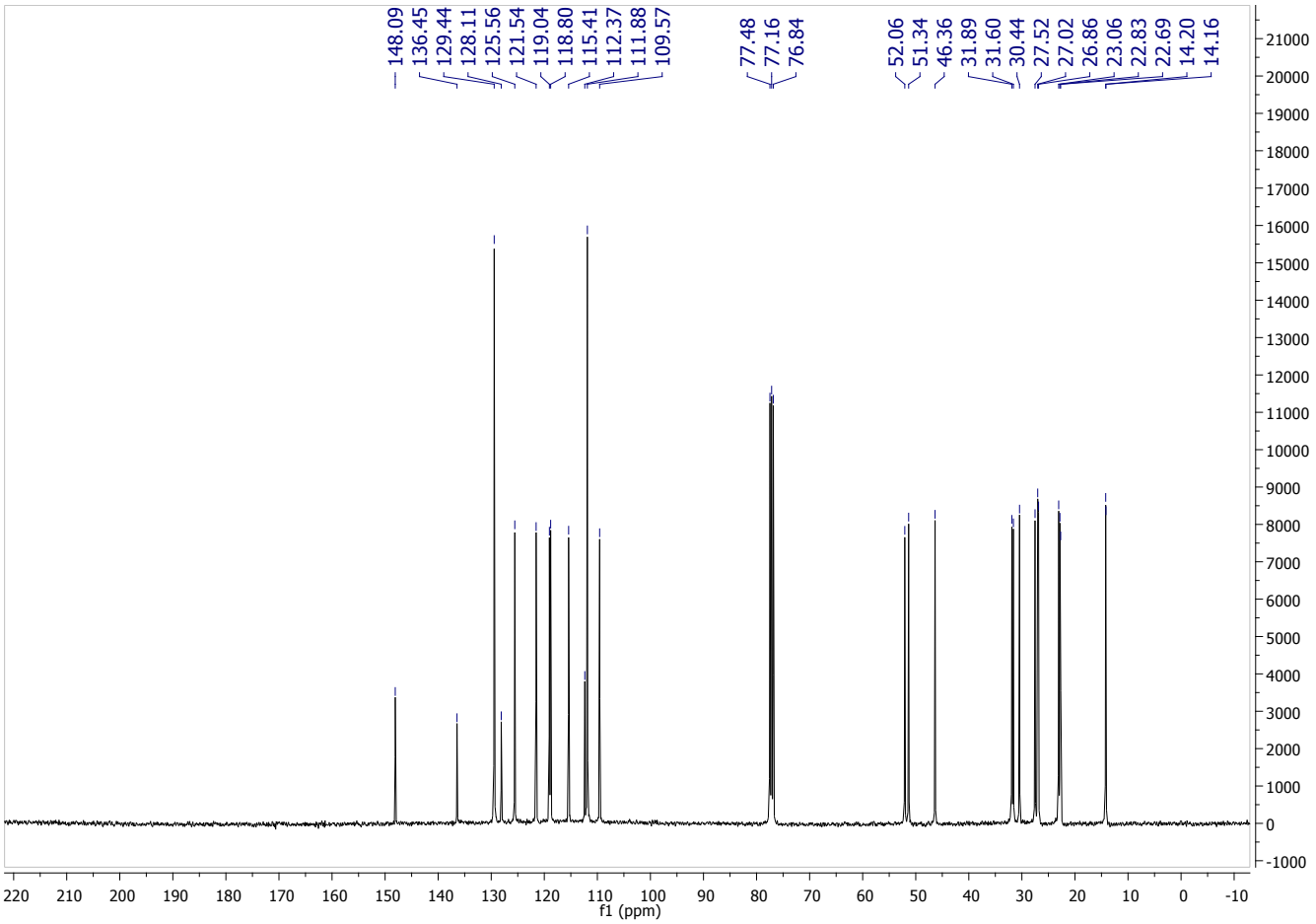
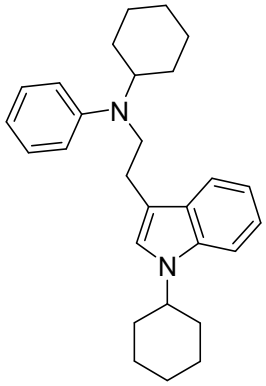
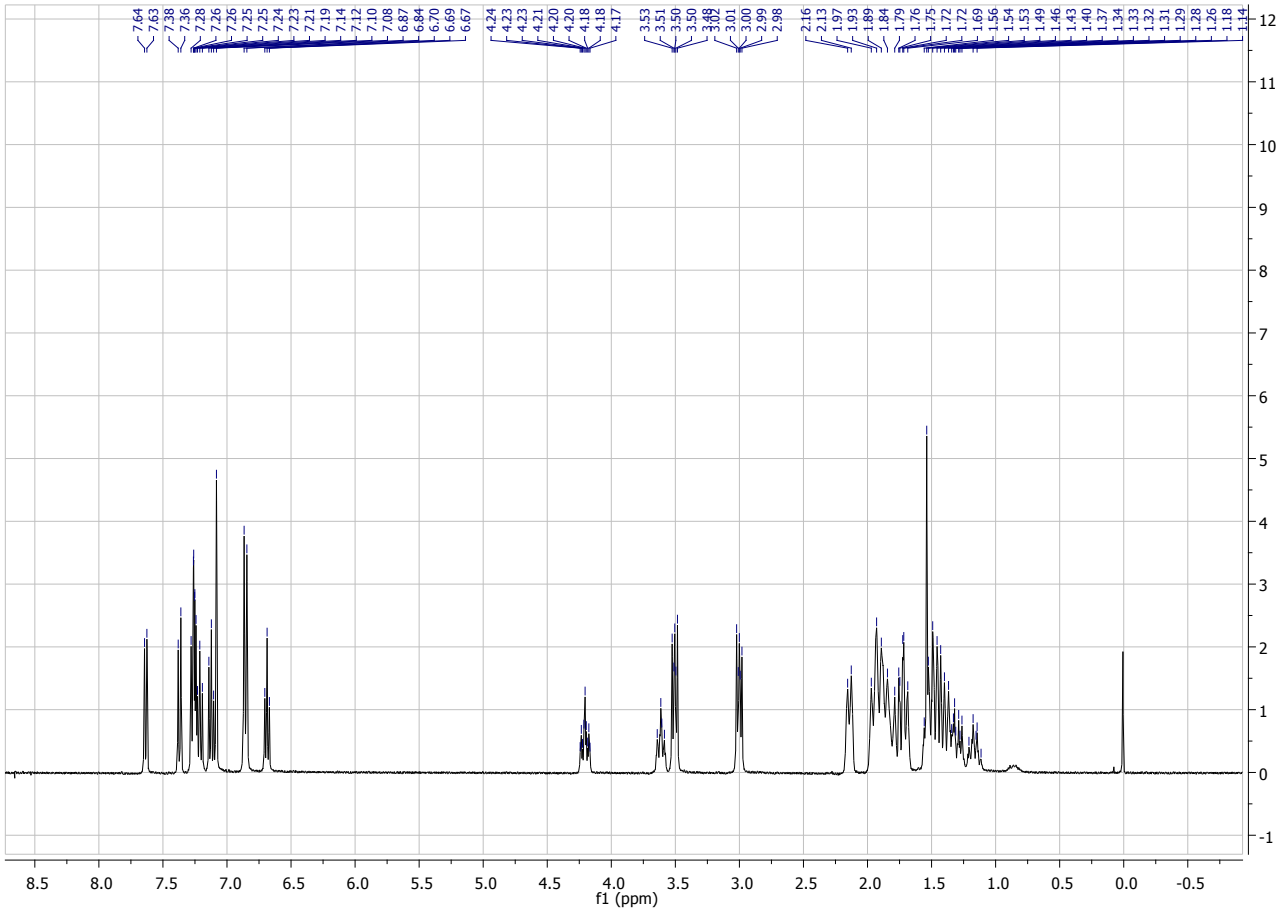
7.26
7.24
7.22
7.22
7.21
7.21
7.21
7.19
7.13
7.11
7.09
6.91
6.75
6.73
6.67
6.66
6.64
4.07
4.05
4.03
3.60
3.58
3.27
3.25
3.04
3.02
1.82
1.81
1.79
1.77
1.58
1.56
1.54
1.30
1.29
0.89
0.88
0.88
0.87
f1 (ppm)


148.09
136.45
129.44
128.11
125.56
121.54
119.04
118.80
115.41
112.37
111.88
109.57
77.48
77.16
76.84
52.06
51.34
46.36
31.89
31.60
30.44
27.52
27.02
26.86
23.06
22.83
22.69
14.20
14.16
f1 (ppm)



3m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7.63
7.38
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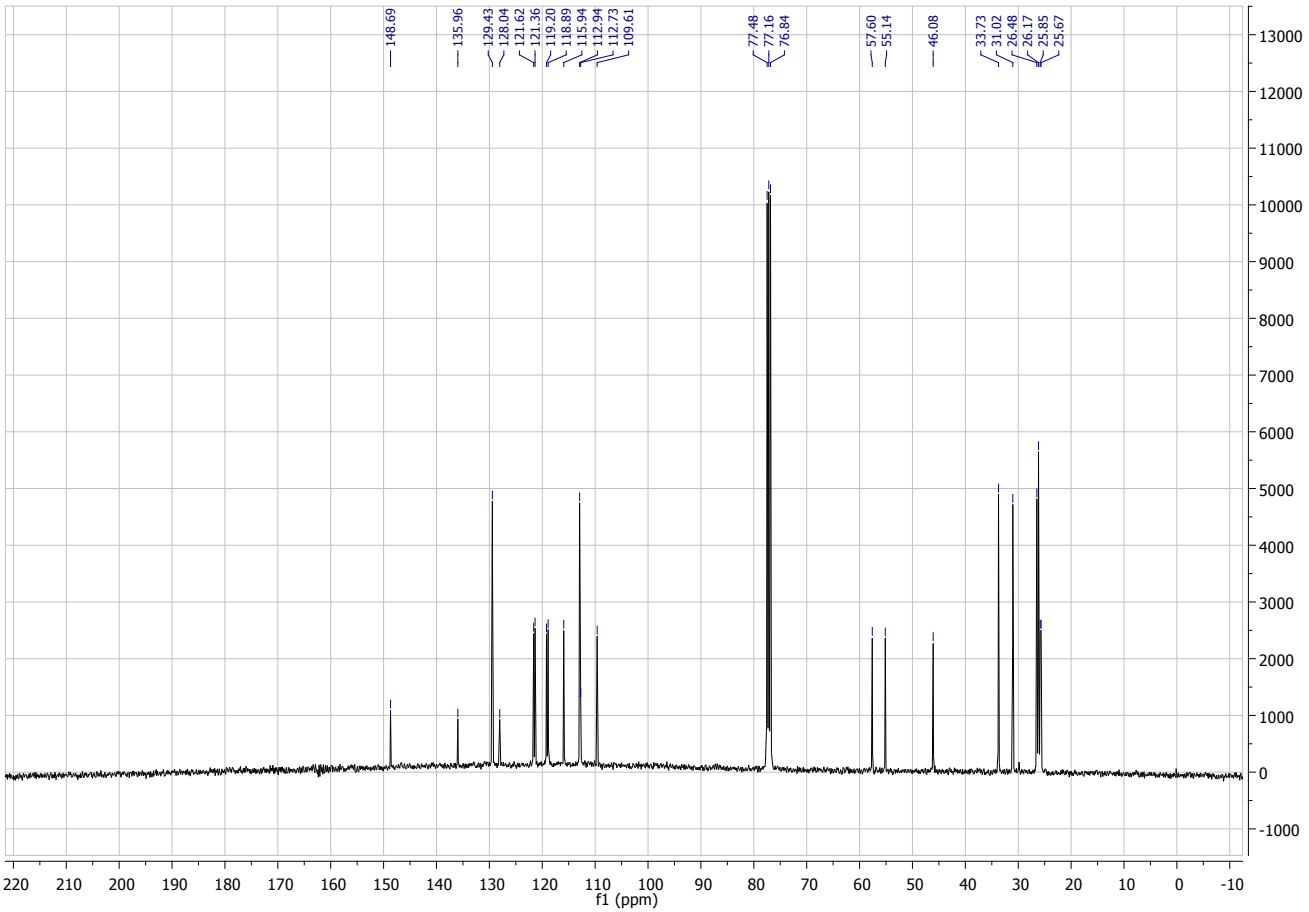
7.28
7.26
7.26
7.25
7.25
7.24
7.23
7.21
7.19
7.14
7.12
7.10
7.08
6.87
6.84
6.70
6.69
6.67
4.24
4.23
4.23
4.21
4.20
4.20
4.18
4.17
3.53
3.51
3.50
3.50
3.48
3.02
3.01
3.00
2.99
2.98
2.16
2.13
1.97
1.93
1.89
1.84
1.79
1.76
1.75
1.72
1.72
1.69
1.56
1.54
1.53
1.49
1.46
1.43
1.40
1.37
1.34
1.33
1.32
1.31
1.29
1.28
1.26
1.18
1.14
f1 (ppm)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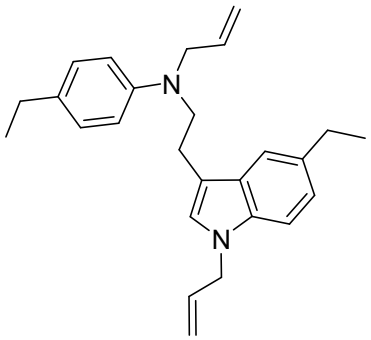
129.43
128.04
121.62
121.36
119.20
118.89
115.94
112.94
112.73
109.61
77.48
77.16
76.84
57.60
55.14
46.08
33.73
31.02
26.48
26.17
25.85
25.67
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



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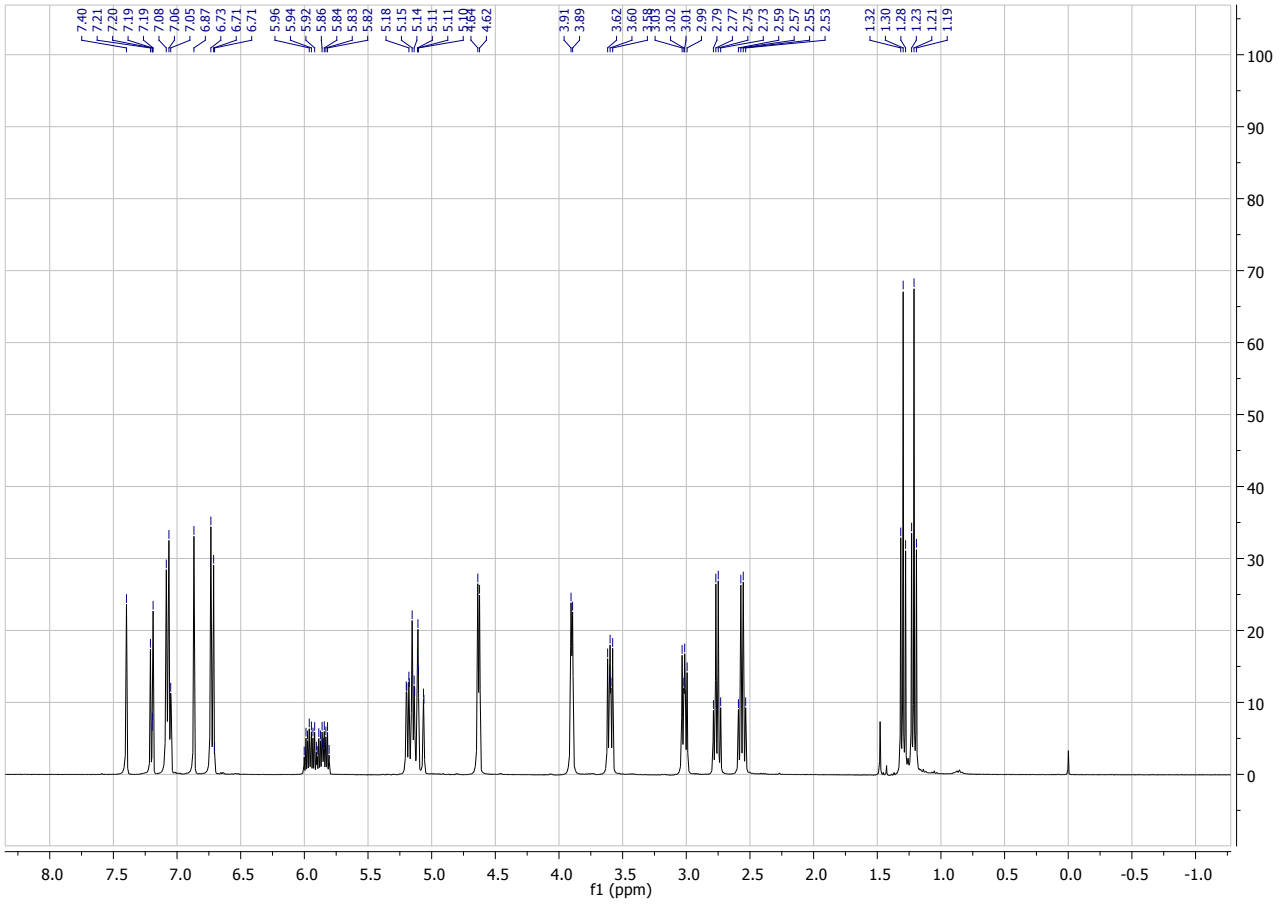
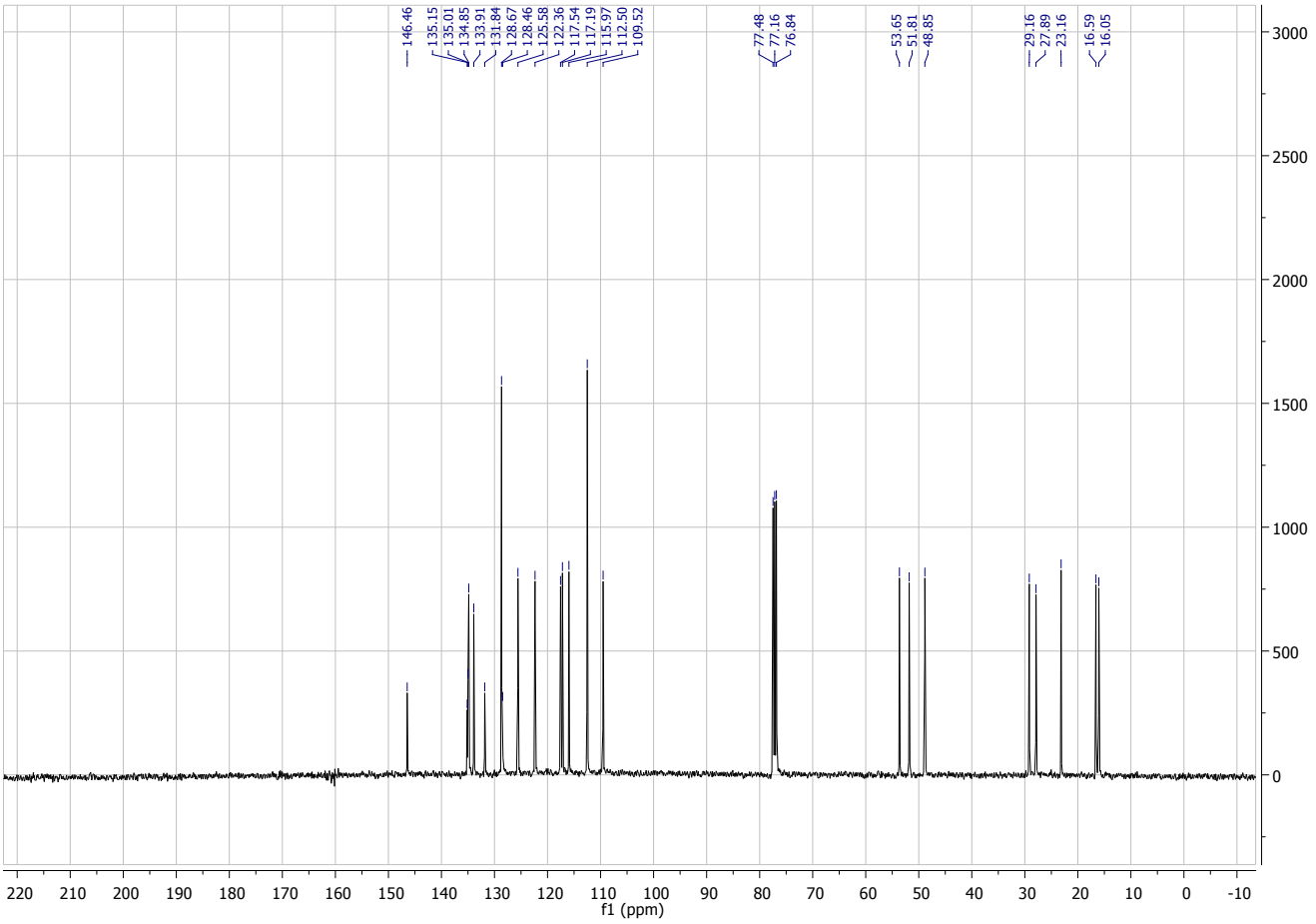

7.40
7.21
7.20
7.19
7.19
7.08
7.06
7.05
6.87
6.73
6.71
6.71
5.96
5.94
5.92
5.86
5.84
5.83
5.82
5.18
5.15
5.14
5.11
5.11
4.64
4.62
3.91
3.89
3.62
3.60
3.58
3.03
3.02
3.01
2.99
2.79
2.77
2.75
2.73
2.59
2.57
2.55
2.53
1.32
1.30
1.28
1.23
1.21
1.19
100
90
80
70
60
50
40
30
20
10
0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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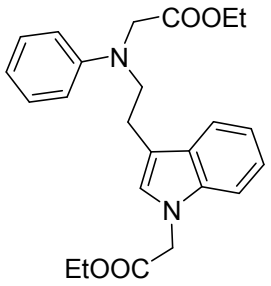
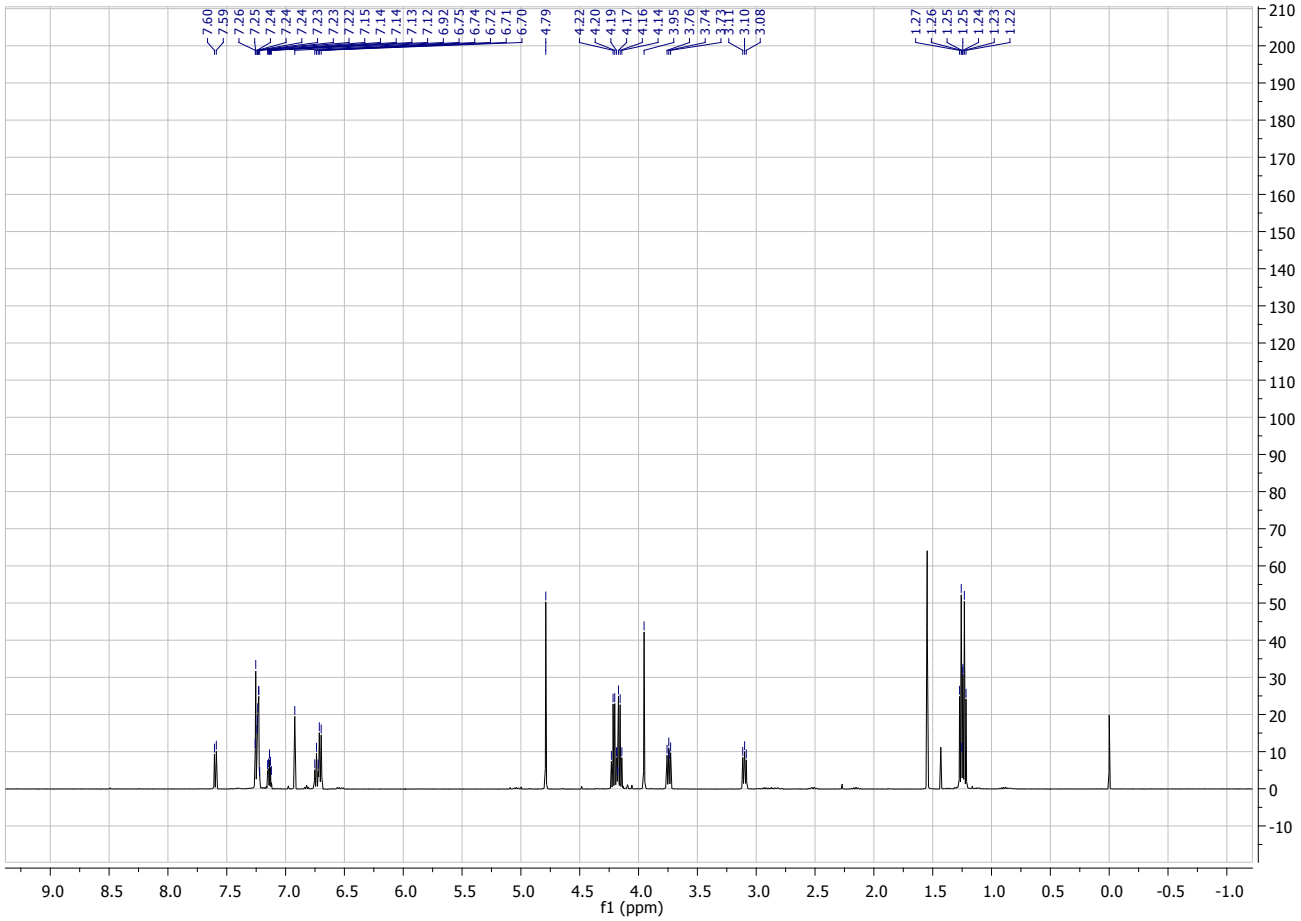
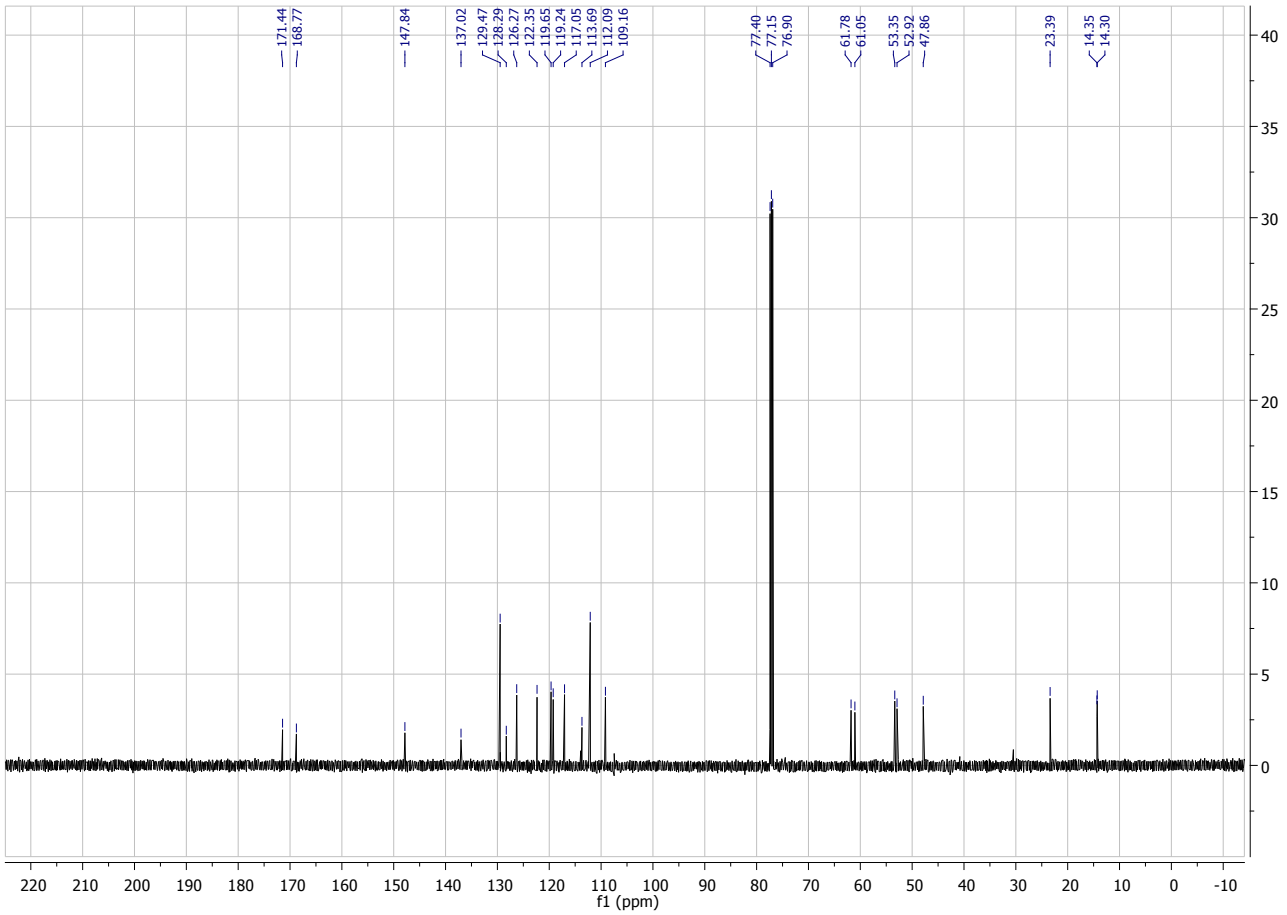
135.15
135.01
134.85
133.91
131.84
128.67
128.46
125.58
122.36
117.54
117.19
115.97
112.50
109.52
77.48
77.16
76.84
53.65
51.81
48.85
29.16
27.89
23.16
16.59
16.05
3000
2500
2000
1500
1000
500
0
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


COOEt
N
N
EtOOC

3o


7.60
7.59
7.26
7.25
7.24
7.24
7.24
7.23
7.23
7.22
7.15
7.14
7.14
7.13
7.12
6.92
6.75
6.74
6.72
6.71
6.70
4.79
4.22
4.20
4.19
4.17
4.16
4.14
3.95
3.76
3.74
3.73
3.11
3.10
3.08
1.27
1.26
1.25
1.25
1.24
1.23
1.22
f1 (ppm)


171.44
168.77
147.84
137.02
129.47
128.29
126.27
122.35
119.65
119.24
117.05
113.69
112.09
109.16
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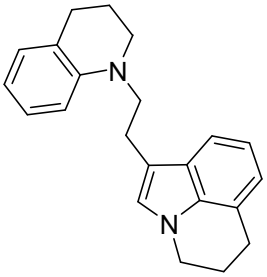
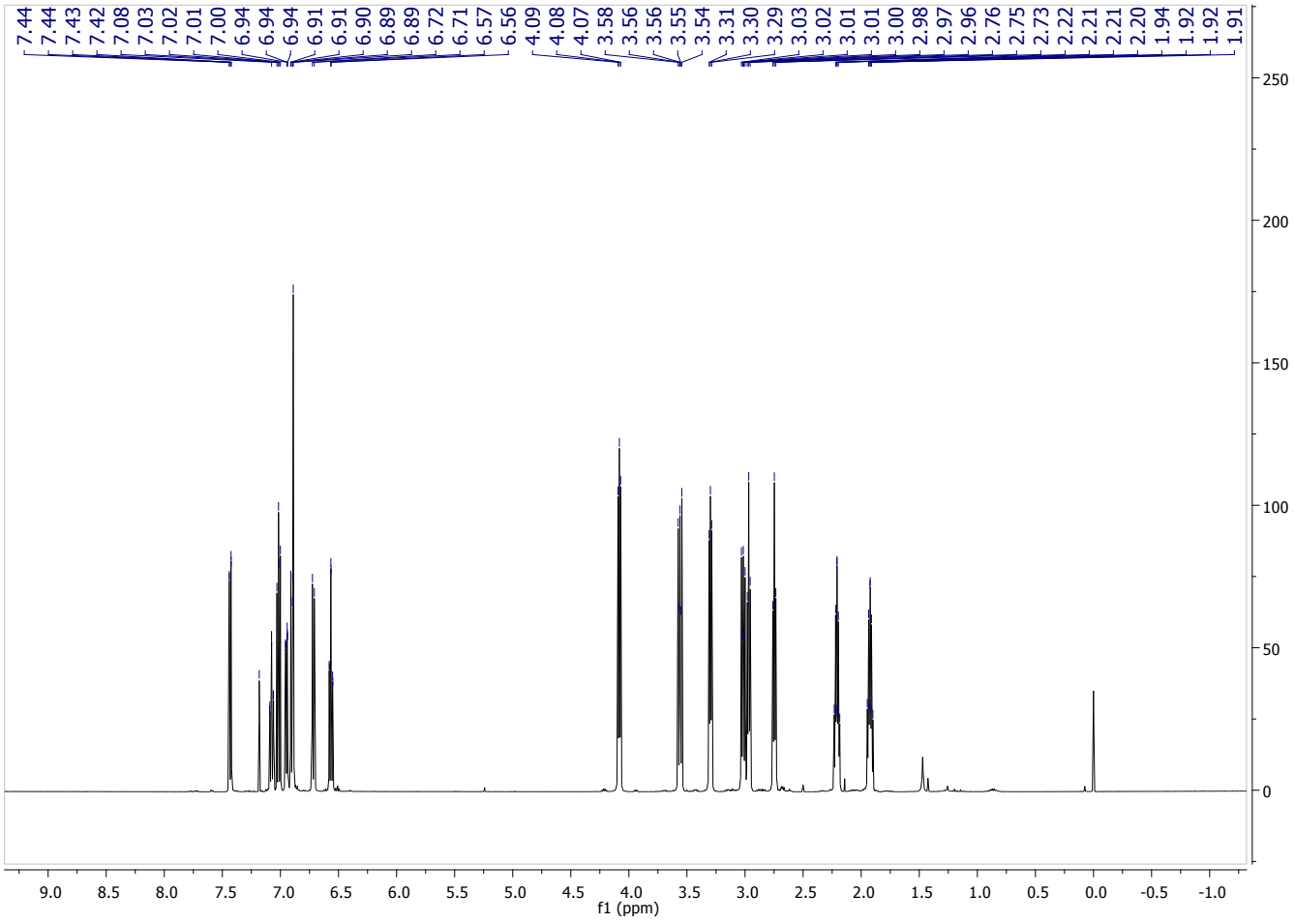
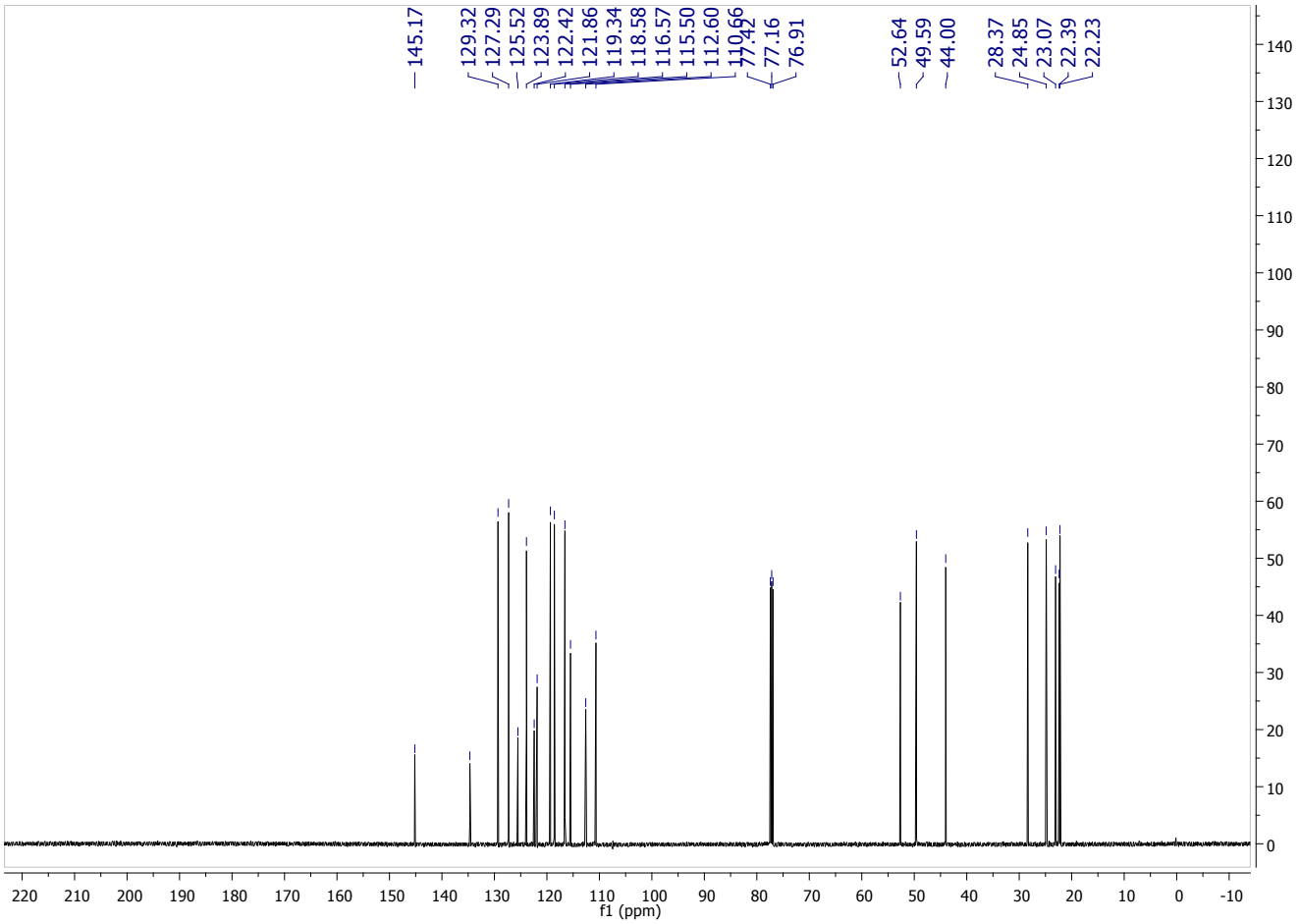
52.92
47.86
23.39
14.35
14.30
f1 (ppm)



3p


7.44 7.44 7.43 7.42 7.08 7.03 7.02 7.01 7.00 6.94 6.94 6.91 6.91 6.90 6.89 6.89 6.72 6.71 6.57 6.56 4.09 4.08 4.07 3.58 3.56 3.56 3.55 3.54 3.31 3.30 3.29 3.03 3.02 3.01 3.01 3.00 2.98 2.97 2.96 2.76 2.75 2.73 2.22 2.21 2.21 2.20 1.94 1.92 1.92 1.91
250
200
150
100
50
0
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


145.17
129.32
127.29
125.52
123.89
122.42
121.86
119.34
118.58
116.57
115.50
112.60
110.66
77.42
77.16
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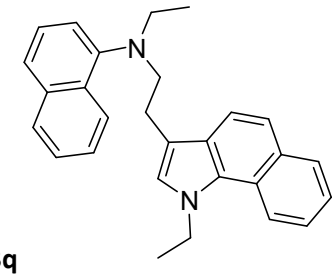
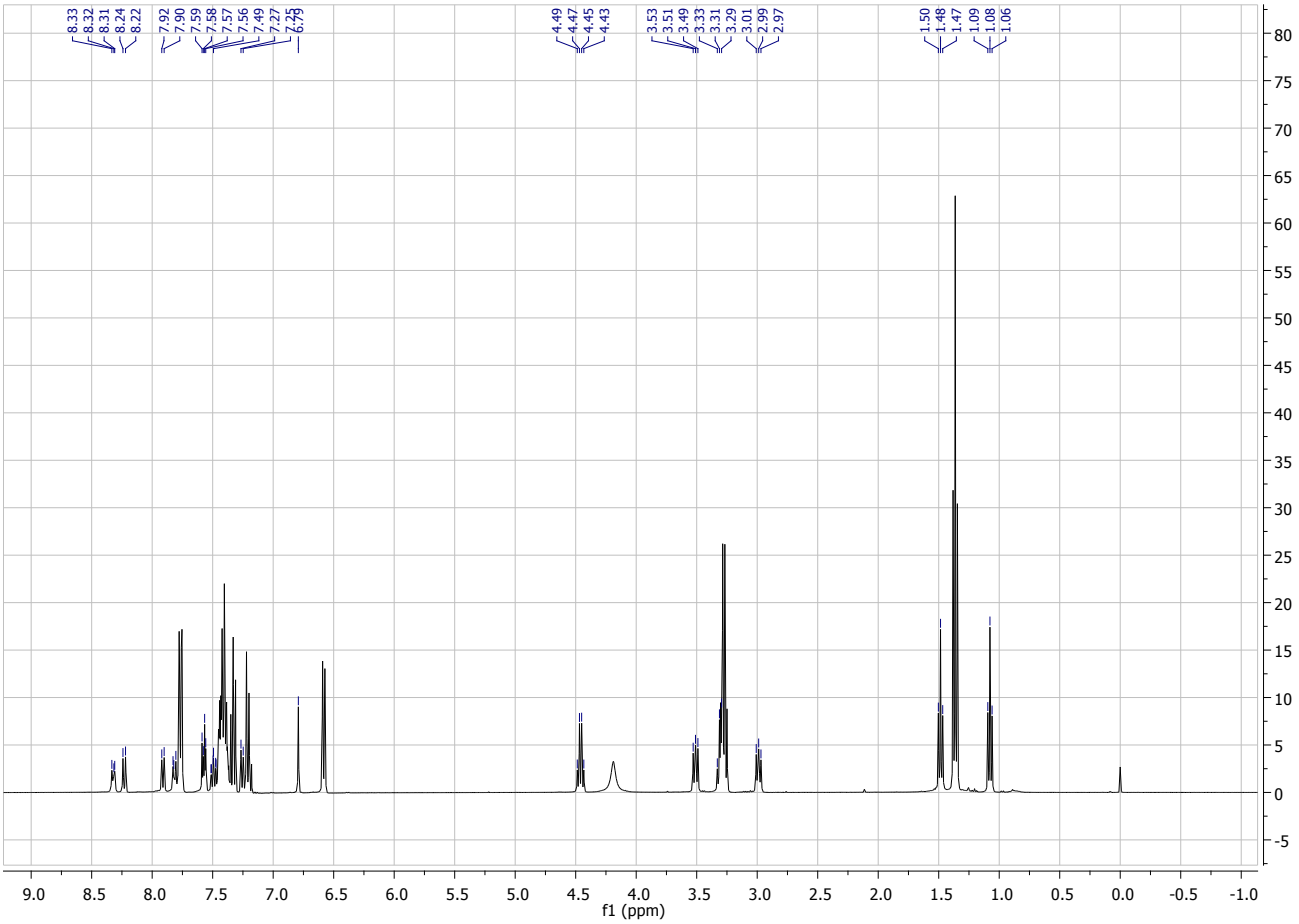
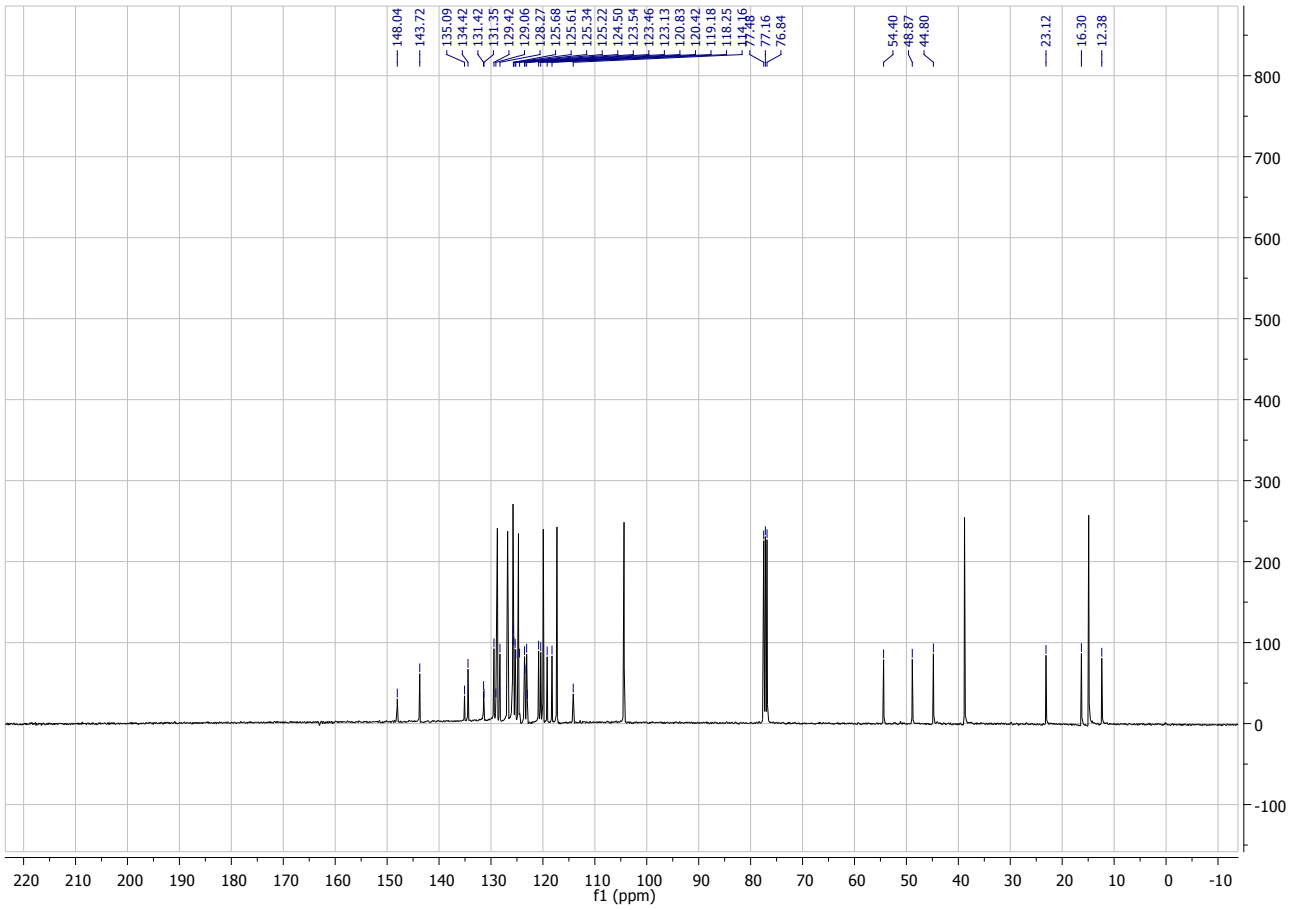
28.37
24.85
23.07
22.39
22.23
f1 (ppm)



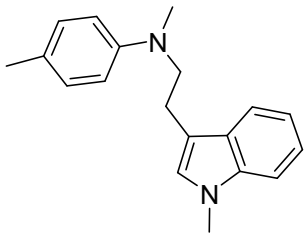
3q


8.33
8.32
8.31
8.24
8.22
7.92
7.90
7.59
7.58
7.57
7.56
7.49
7.27
4.49
4.47
4.45
4.43
3.53
3.51
3.49
3.33
3.31
3.29
3.01
2.99
2.97
1.50
1.48
1.47
1.09
1.08
1.06
f1 (ppm)

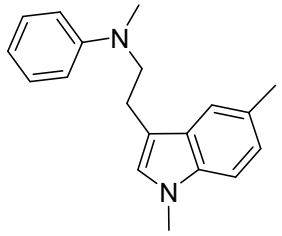

148.04
143.72
135.09
134.42
131.42
131.35
129.42
129.06
128.27
125.68
125.61
125.34
125.22
124.50
123.54
123.46
123.13
120.83
120.42
119.18
118.25
77.16
76.84
54.40
48.87
44.80
23.12
16.30
12.38
800
700
600
500
400
300
200
100
0
-100
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Synthesis of tryptamines derived from two different anilines by one pot sequential procedure (Scheme 4)

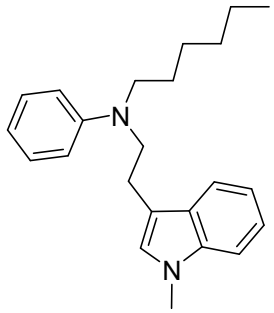
General procedure: 2-hydroxy cyclobutanone **1** (0.465 mmol), **2** (0.465 mmol) and PTSA (0.093 mmol) were made to react for 4h at room temperature and then, after addition of **2'**(0.465 mmol) the mixture was stirred for 4d. Yields were calculated by GC-MS analysis of the crude reaction mixture. Because of the closeness in R_f values, we were unable to separate tryptamines **3**, **3'**, **4** and **5** by silica gel column chromatography. Tryptamines **4** and **5** were characterized by mass spectrometry:



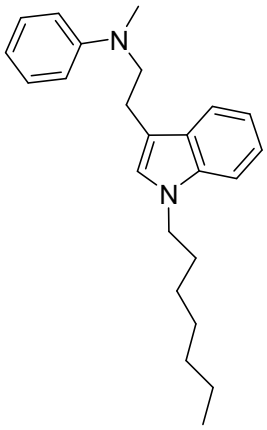
MS *m/z*: 278 (M^+ (20)), 158 (4), 134 (100), 120 (18), 91 (3).



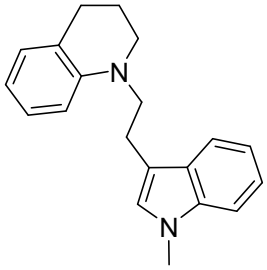
MS m/z: 278 (M^+ (19)), 158 (19), 120 (100), 105 (3).



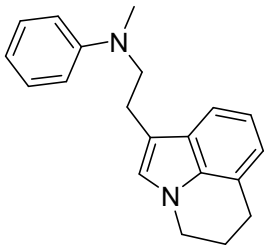
MS m/z: 334 (M^+ (14)), 190 (100), 158 (6), 144 (8), 120 (26), 106 (12)



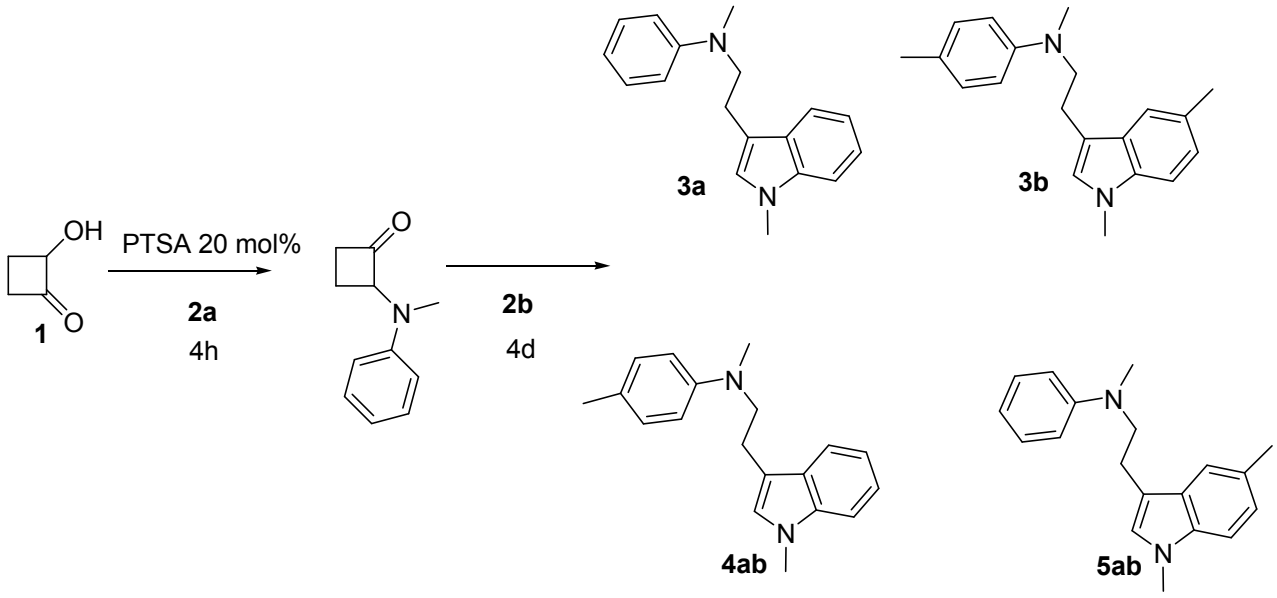
MS m/z: 334 (M^+ (18)), 214 (14), 190 (15), 120 (100).



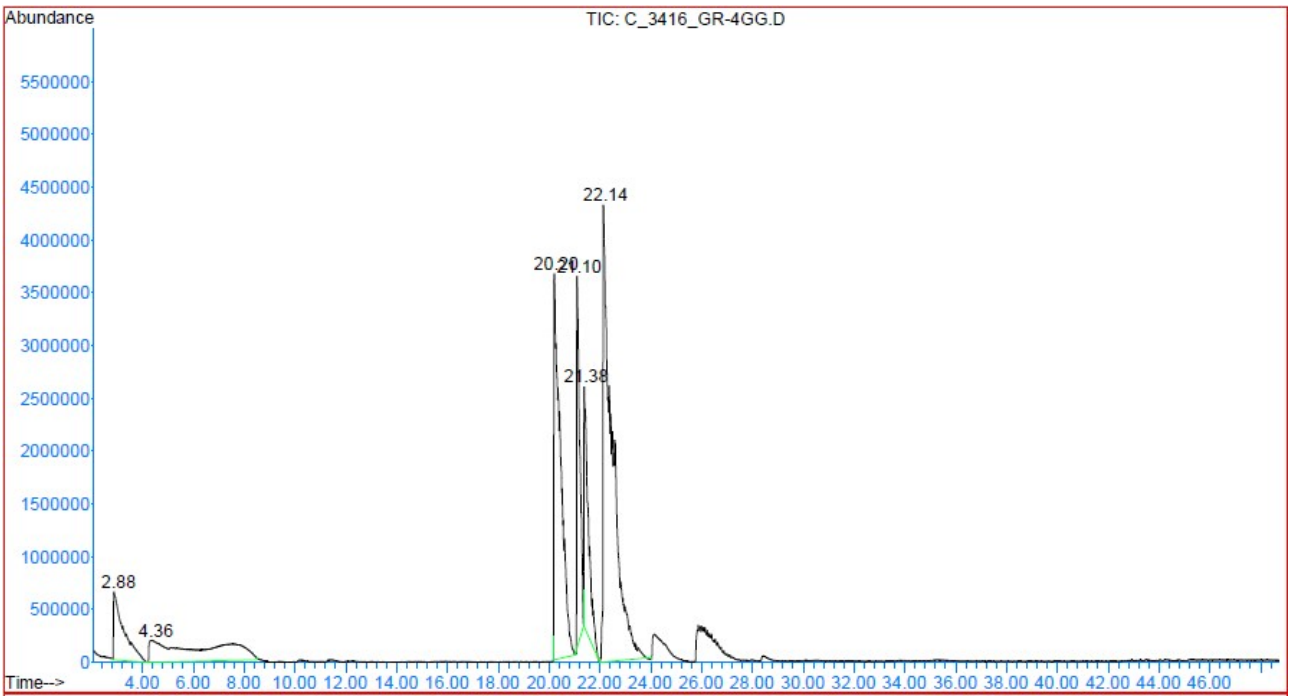
MS m/z: 290 (M^+ (18)), 146 (100), 131 (5), 118 (5), 91 (5).



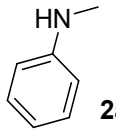
MS m/z: 290 (M^+ (24)), 170 (27), 142 (8), 120 (100), 105 (3).



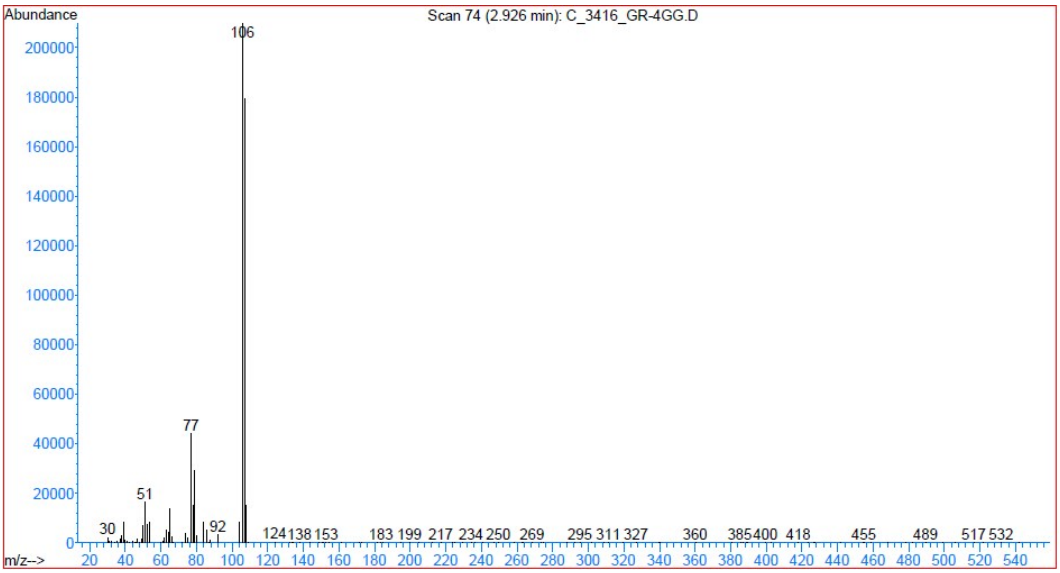
GC-MS analysis of the crude reaction mixture

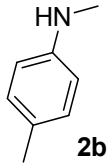


peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	2.880	65	70	185	M3	644722	177467477	16.43%	6.374%
2	4.355	185	198	557	M3	209029	329276190	30.49%	11.826%
3	20.200	1571	1573	1644	M	3698120	642252755	59.46%	23.067%
4	21.099	1644	1651	1674	M	3554848	286058255	26.48%	10.274%
5	21.376	1674	1675	1729	M2	2445472	269151296	24.92%	9.667%
6	22.136	1729	1741	1905	M2	4480440	1080105674	100.00%	38.793%

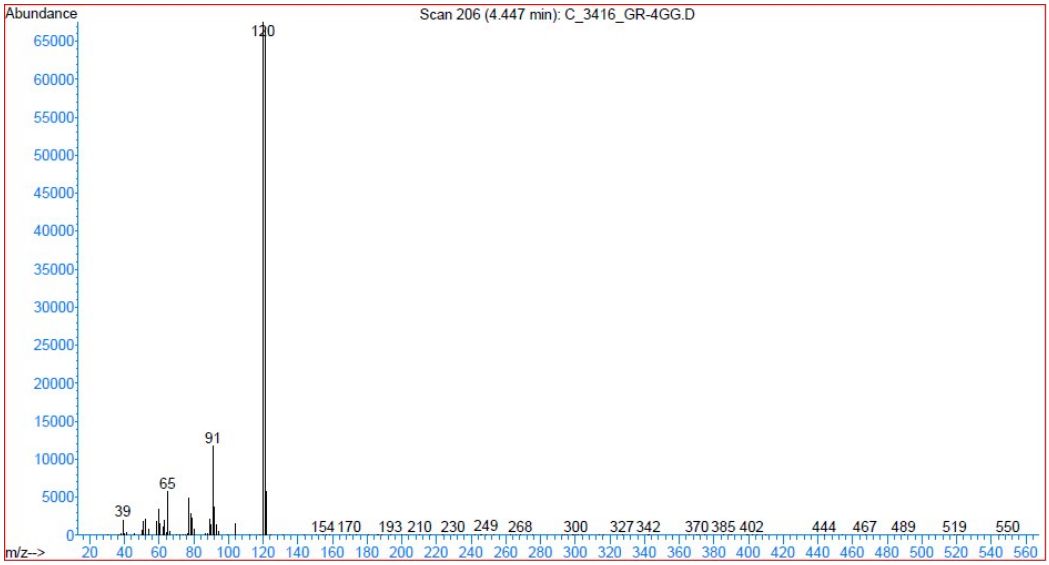

HN

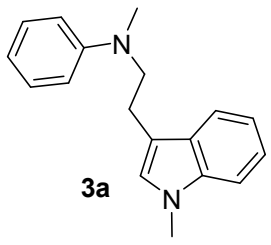
2a


Abundance
Scan 74 (2.926 min): C_3416_GR-4GG.D
106
77
51
92
30
m/z-->

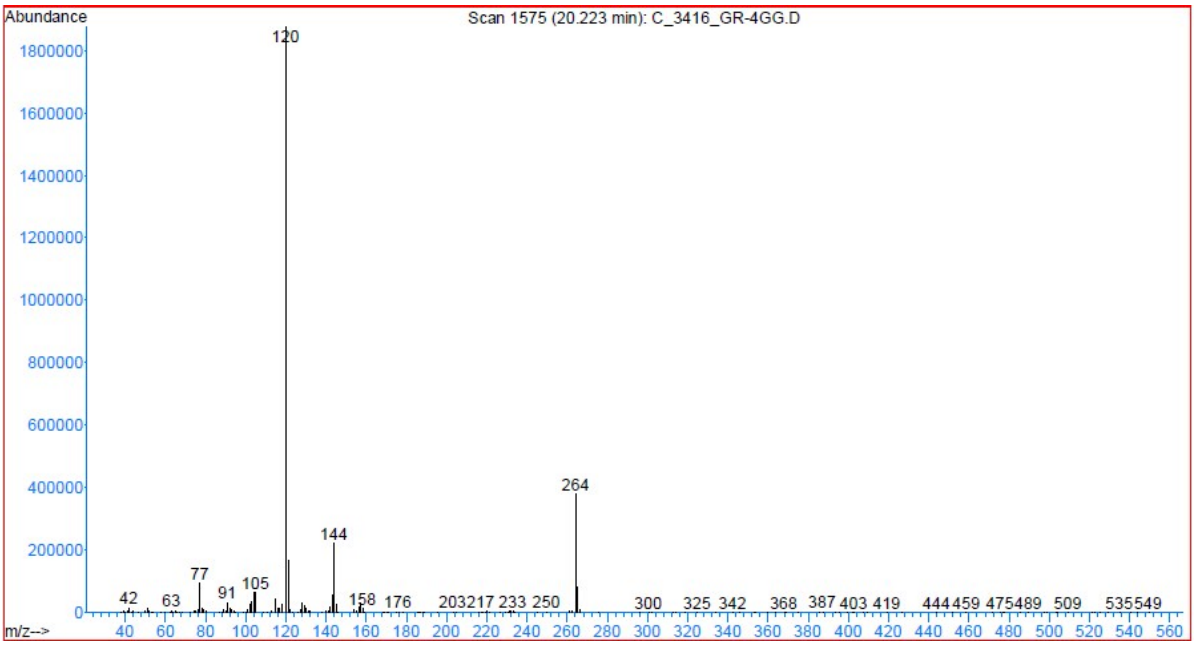

HN

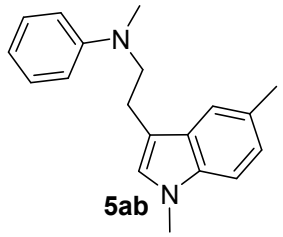
2b


Abundance
Scan 206 (4.447 min): C_3416_GR-4GG.D
120
91
65
39
m/z-->

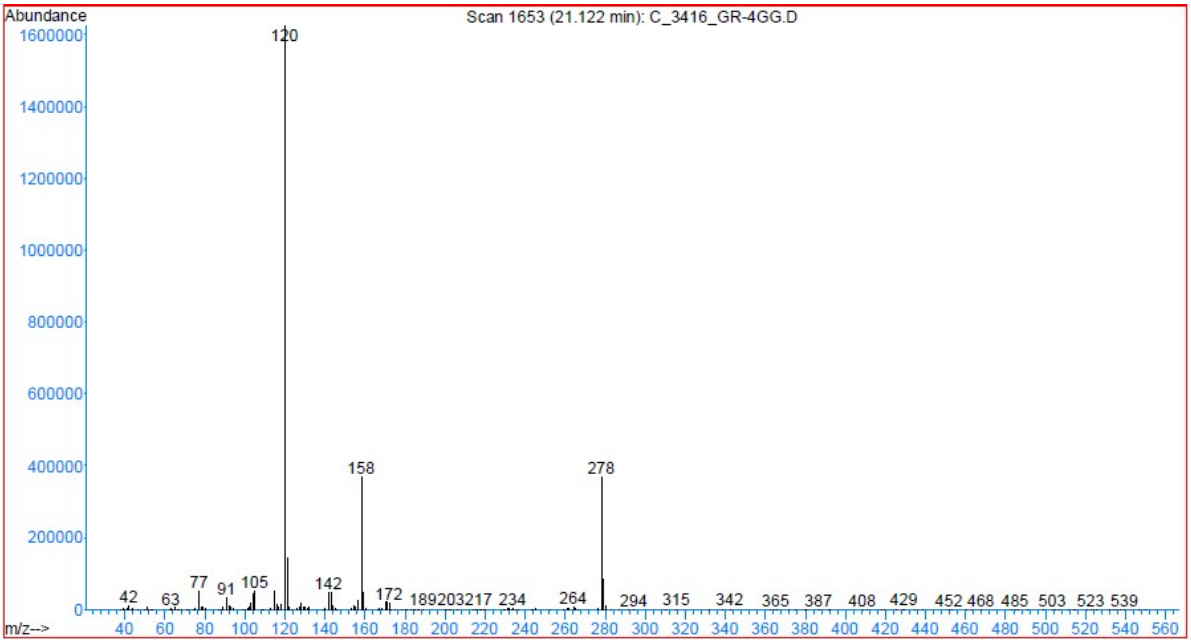

N
N

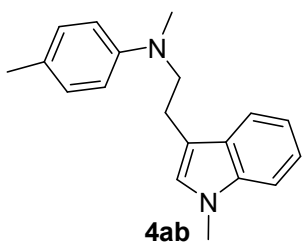
3a


Abundance
Scan 1575 (20.223 min): C_3416_GR-4GG.D
120
264
144
77
105
91
42
63
158
176
203217 233 250
300
325 342
368
387 403 419
444 459 475489
509
535549
m/z-->

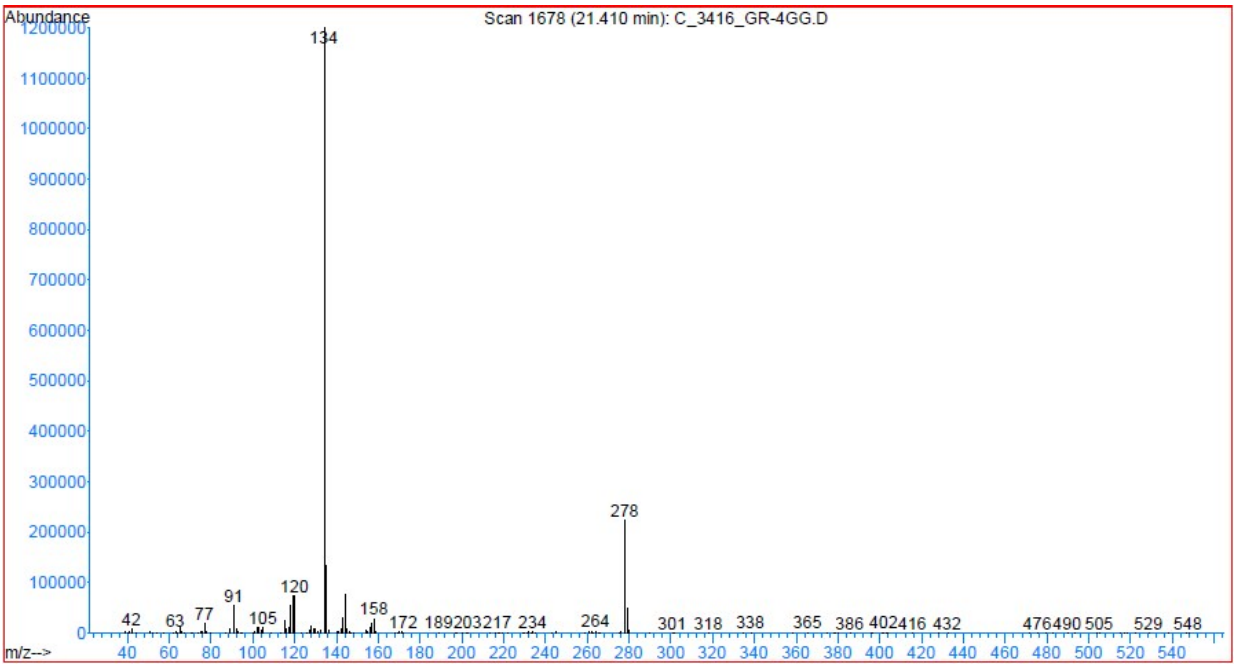

N
N

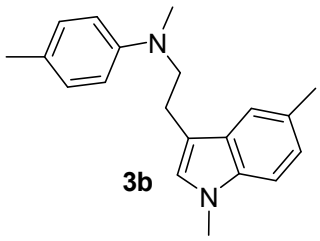
5ab

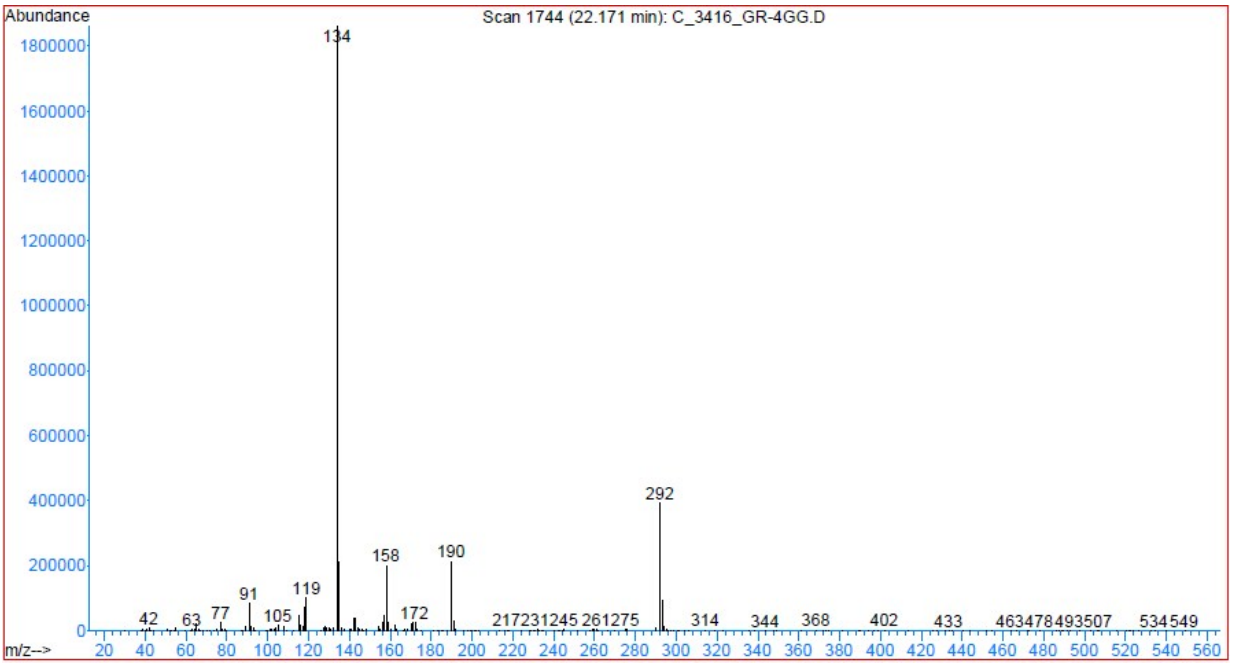

Abundance
Scan 1653 (21.122 min): C_3416_GR-4GG.D
120
158
278
77
105
91
142
42
63
172
189203217 234
264
294
315
342
365
387
408
429
452 468 485
503
523 539
m/z-->


N
N

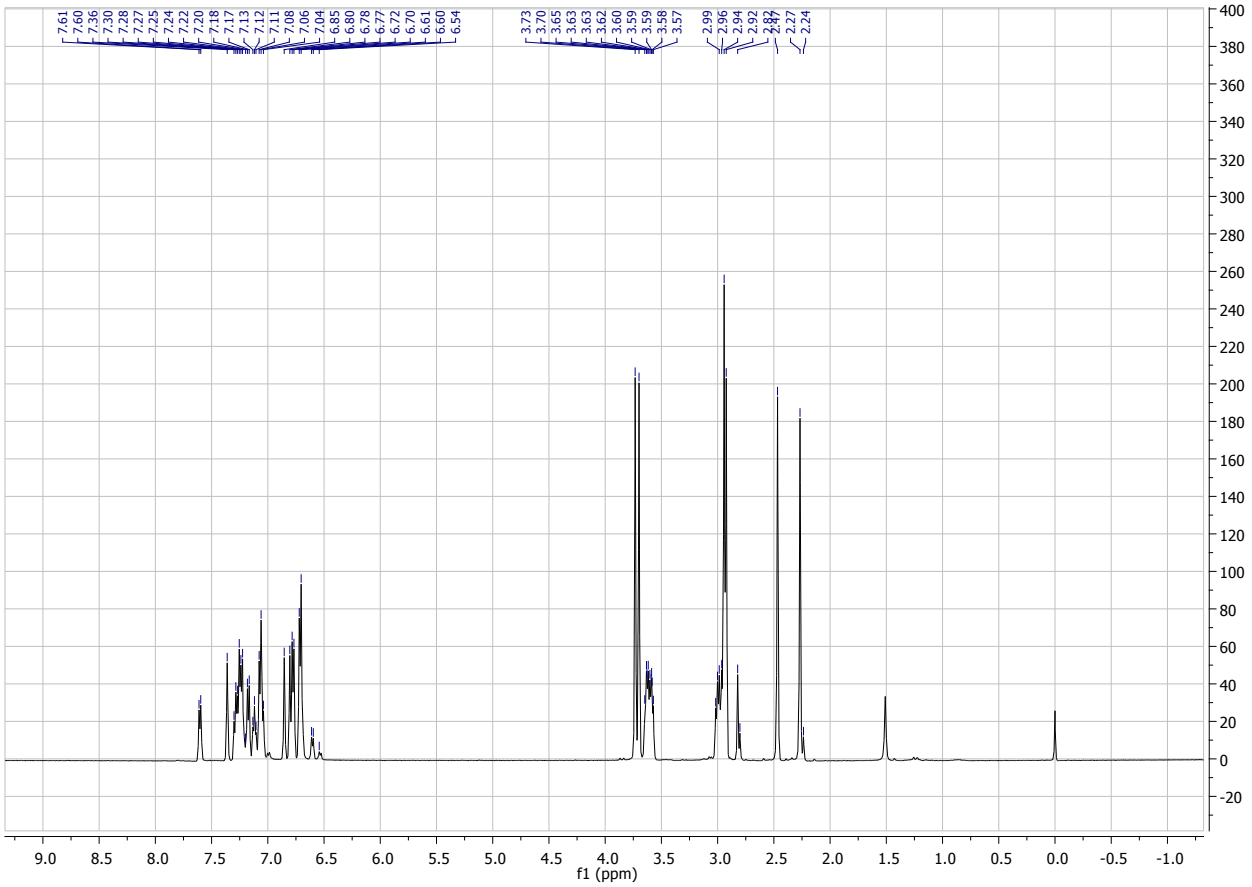
4ab

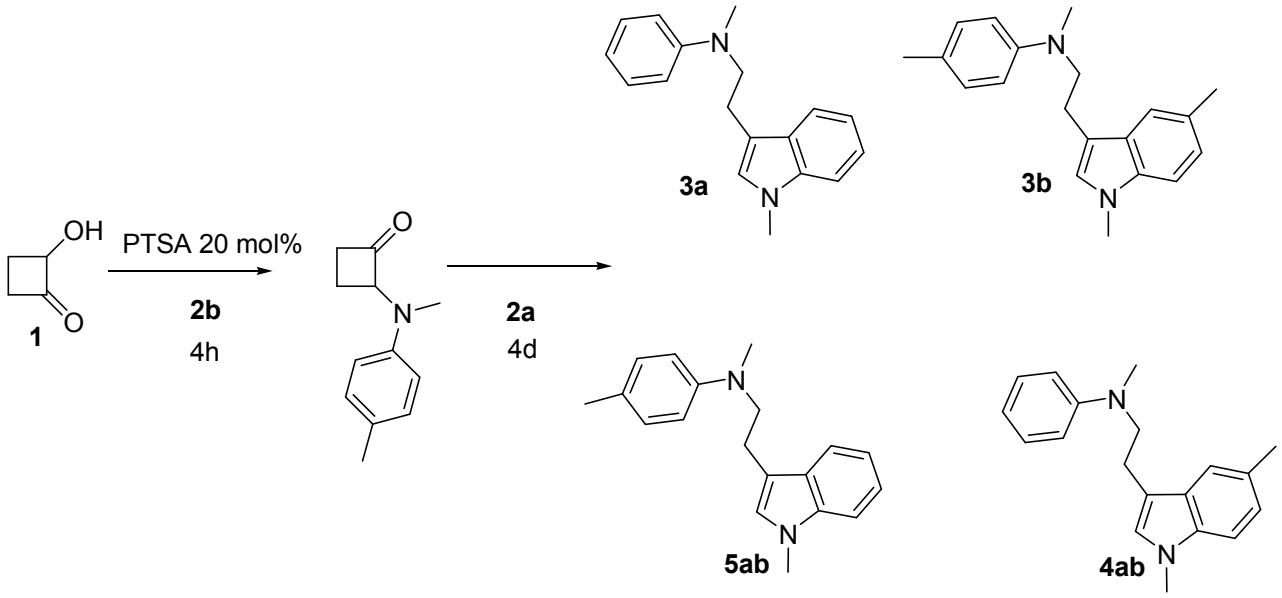




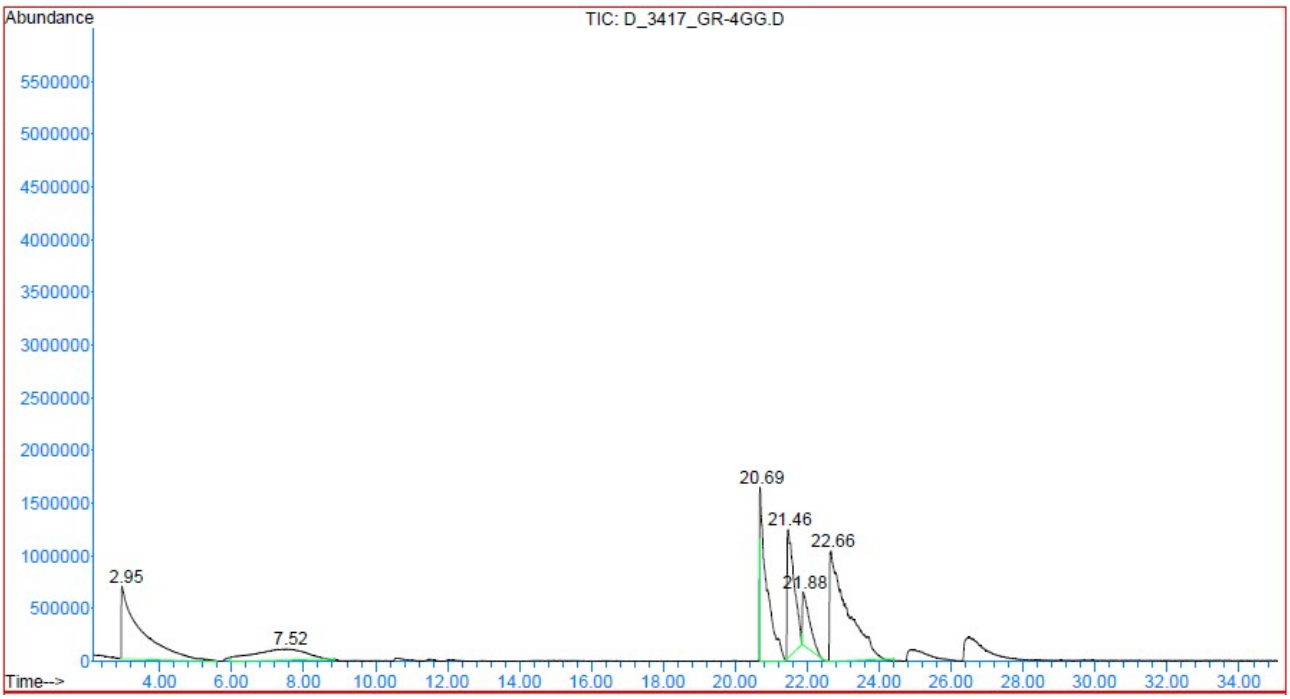


^{1}H NMR of the inseparable reaction products

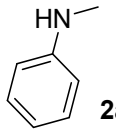

7.61
7.60
7.36
7.30
7.28
7.27
7.25
7.24
7.22
7.20
7.18
7.17
7.13
7.12
7.11
7.08
7.06
7.04
6.85
6.80
6.78
6.77
6.72
6.70
6.61
6.60
6.54
3.73
3.70
3.65
3.63
3.62
3.60
3.59
3.59
3.58
3.57
2.99
2.96
2.94
2.92
2.27
2.24
400
380
360
340
320
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



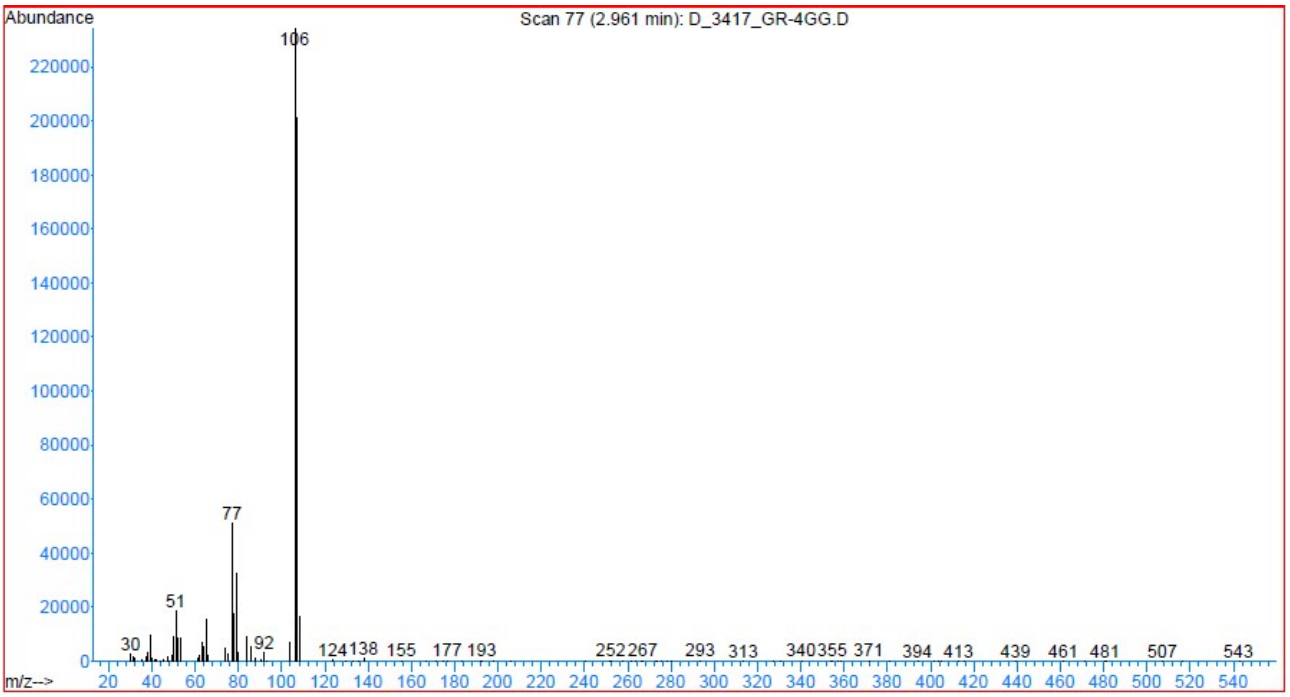
GC-MS analysis of the crude reaction mixture

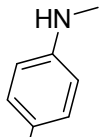


peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	2.949	71	76	304	M2	690395	255621750	72.51%	21.760%
2	7.524	337	473	591	M2	106588	107613422	30.53%	9.161%
3	20.684	1614	1615	1674	M	1654981	224806214	63.77%	19.137%
4	21.468	1674	1683	1716	M	1220175	163956053	46.51%	13.957%
5	21.883	1716	1719	1774	M	503818	70202975	19.92%	5.976%
6	22.655	1777	1786	1937	M2	1048403	352511736	100.00%	30.008%

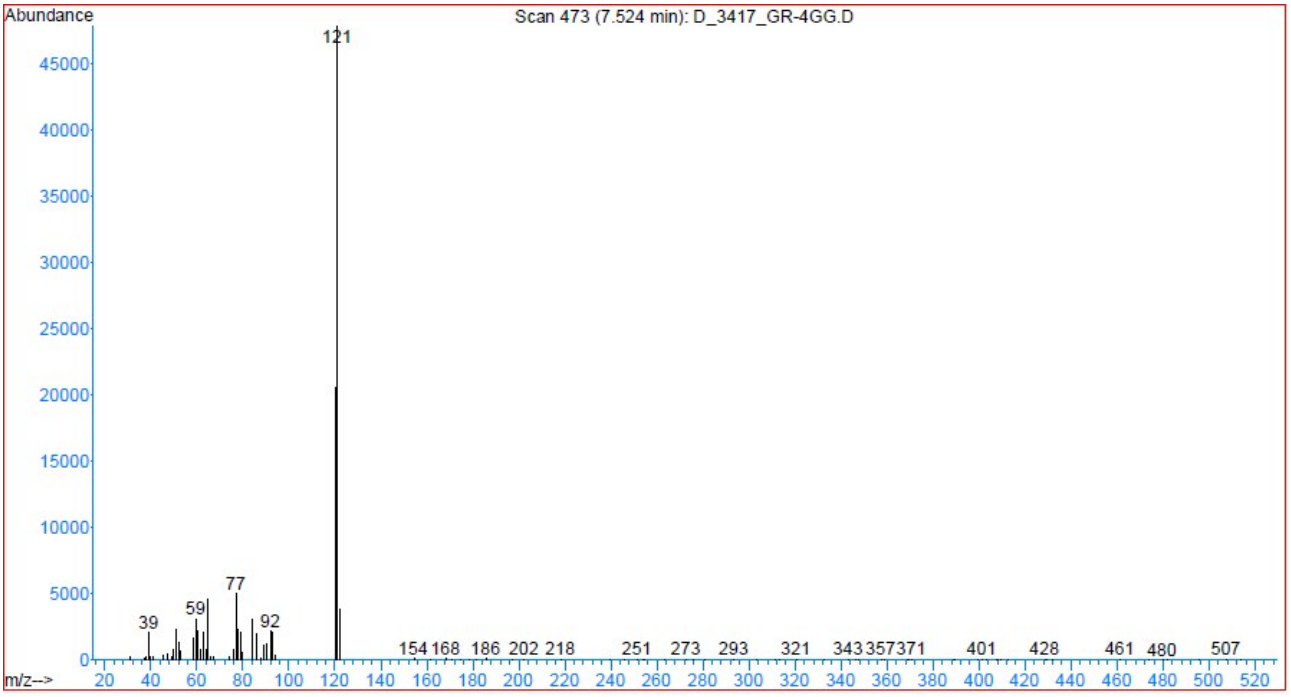

HN

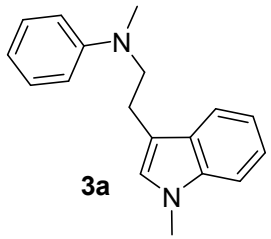
2a


Abundance
Scan 77 (2.961 min): D_3417_GR-4GG.D
106
77
51
92
30
m/z-->

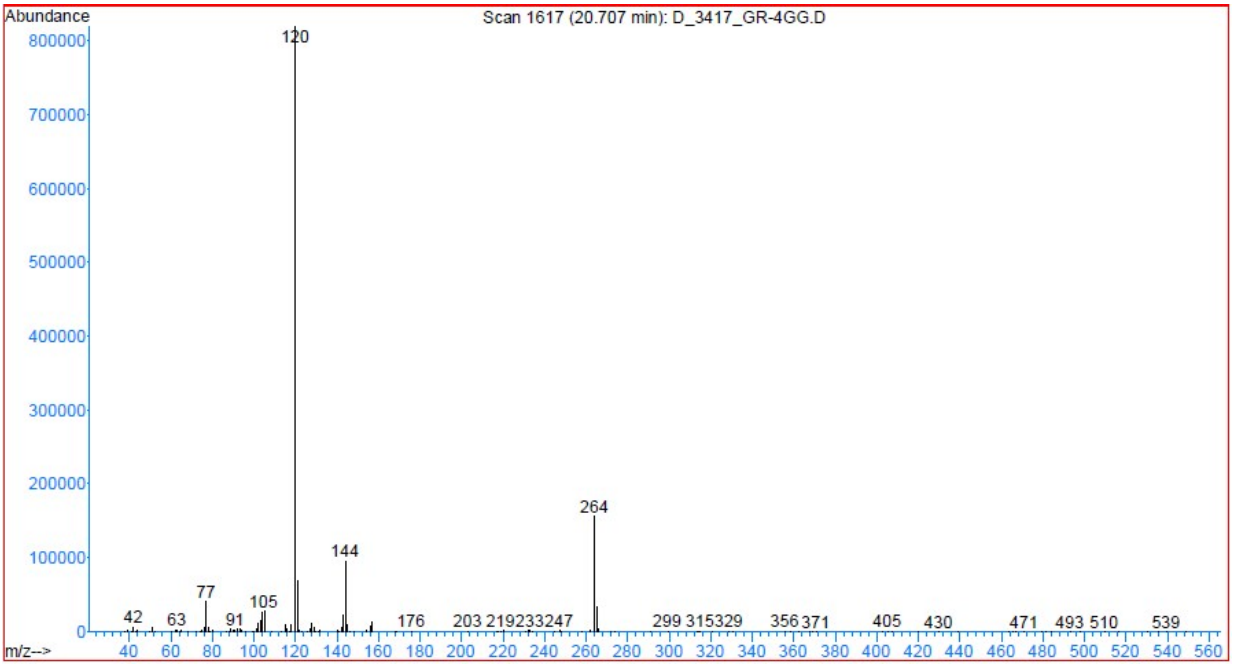

HN

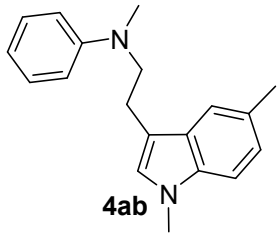
2b


Abundance
Scan 473 (7.524 min): D_3417_GR-4GG.D
121
77
59
39
92
m/z-->

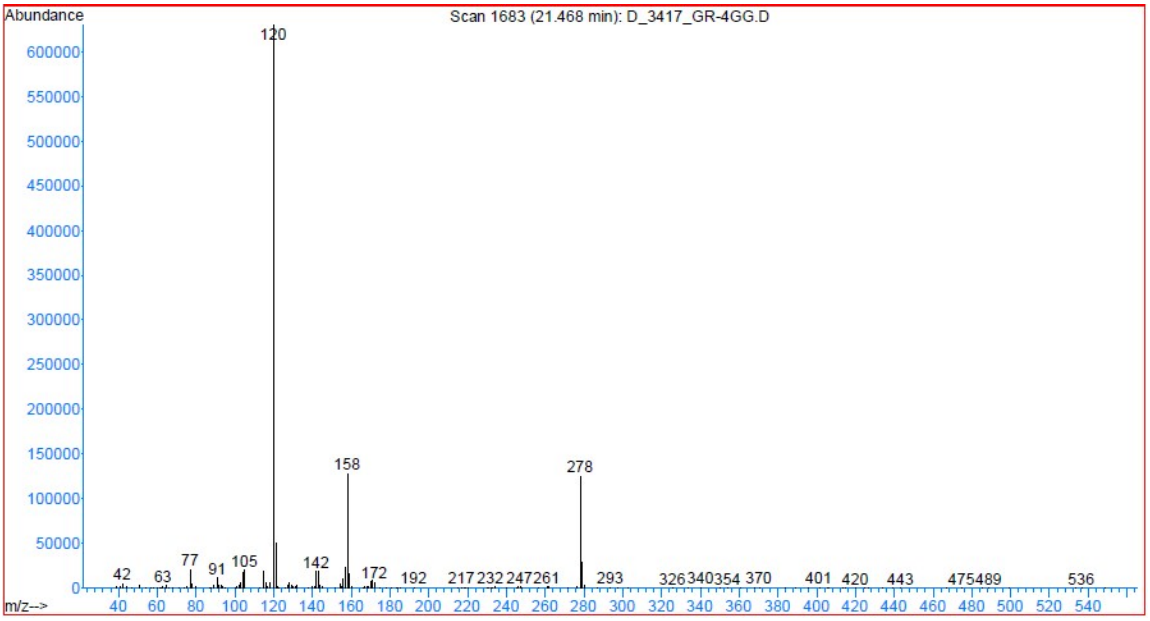


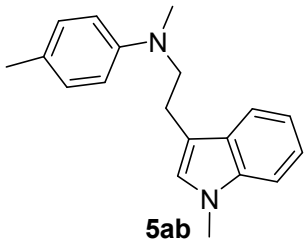
3a


Abundance
Scan 1617 (20.707 min): D_3417_GR-4GG.D
m/z-->

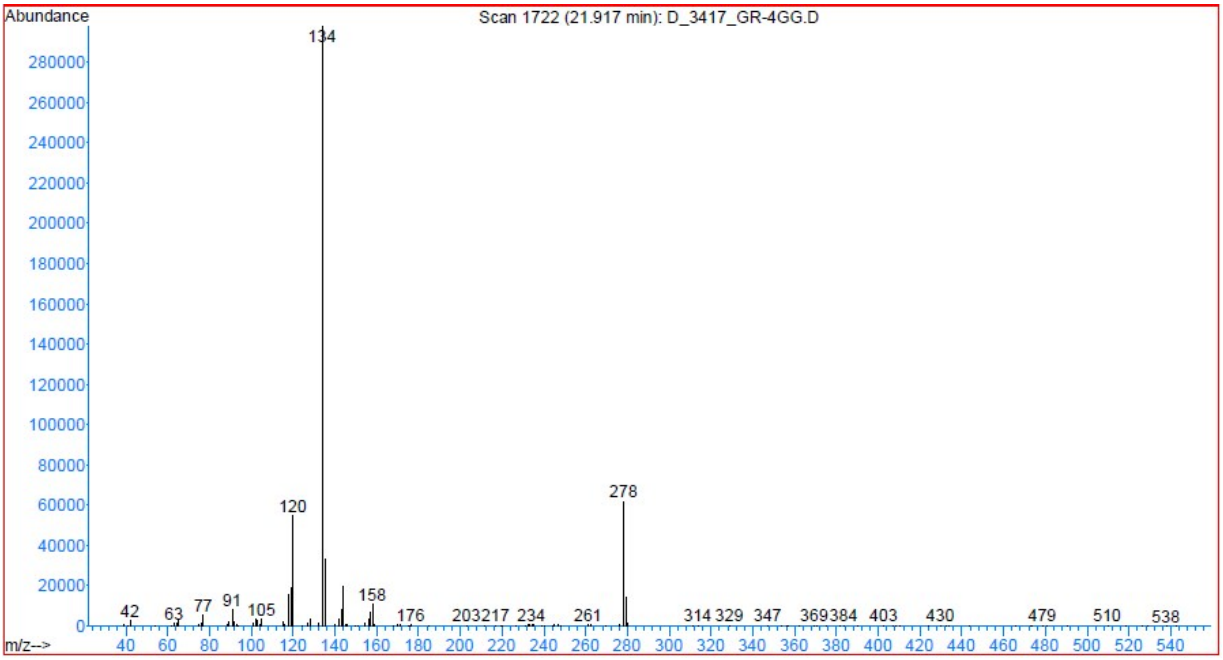


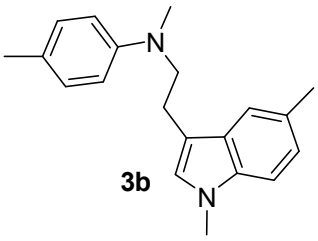
4ab

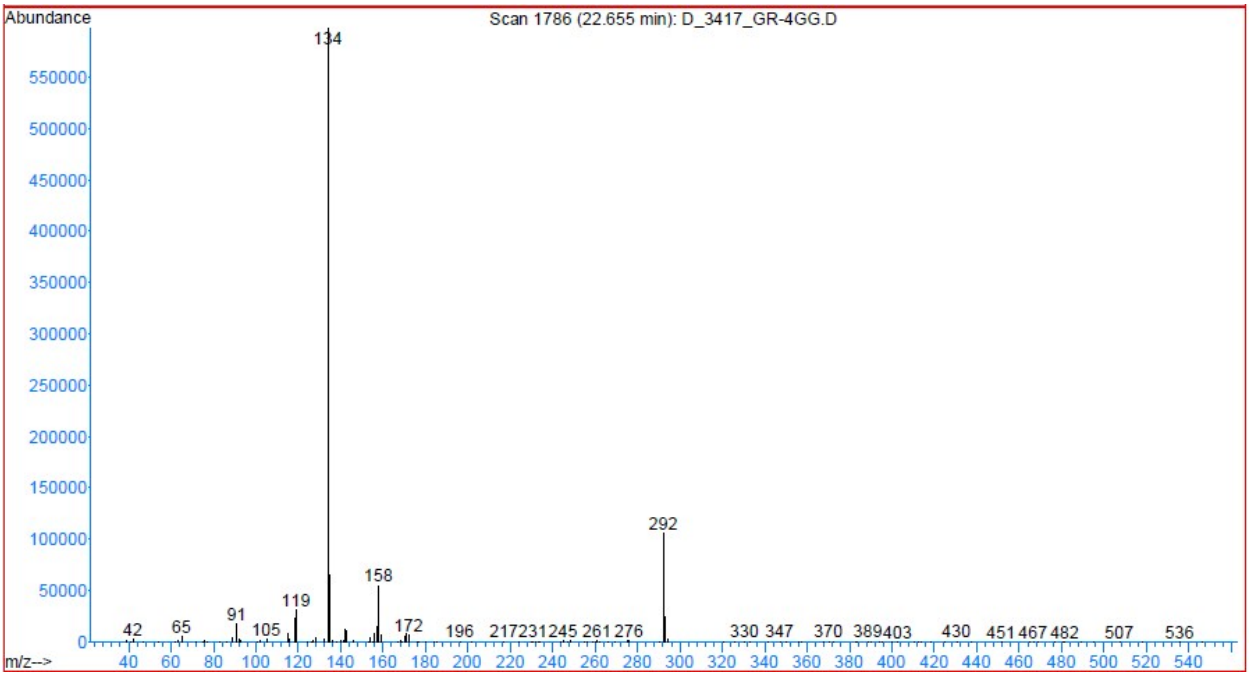

Abundance
Scan 1683 (21.468 min): D_3417_GR-4GG.D
m/z-->



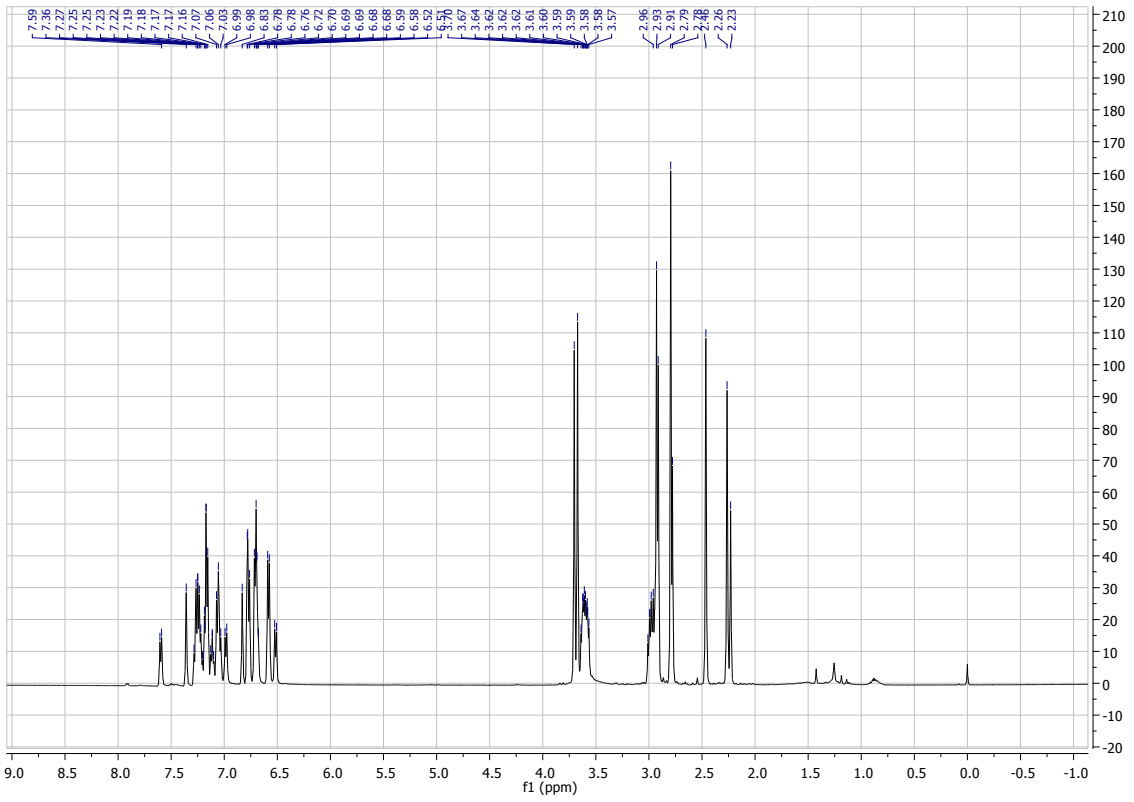
5ab

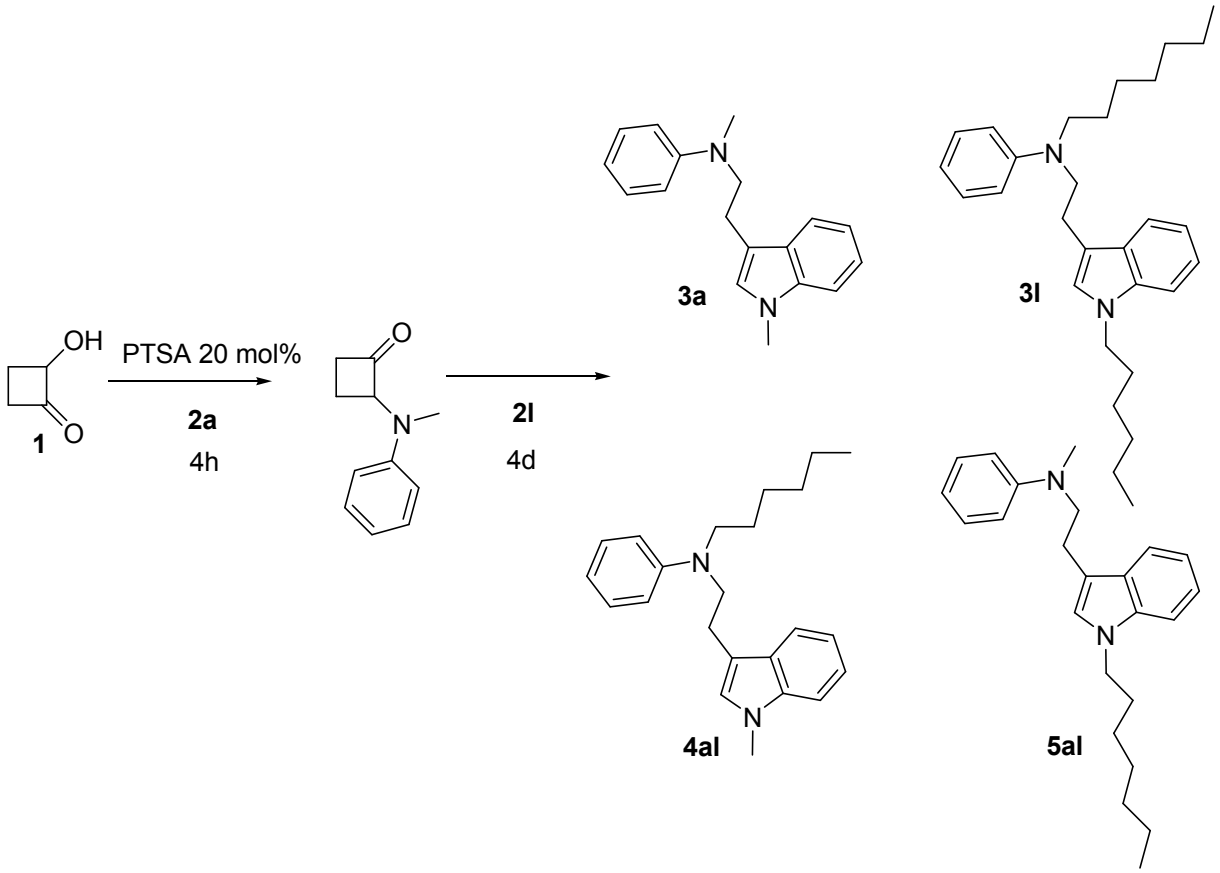

Abundance
Scan 1722 (21.917 min): D_3417_GR-4GG.D
134
278
120
158
m/z-->



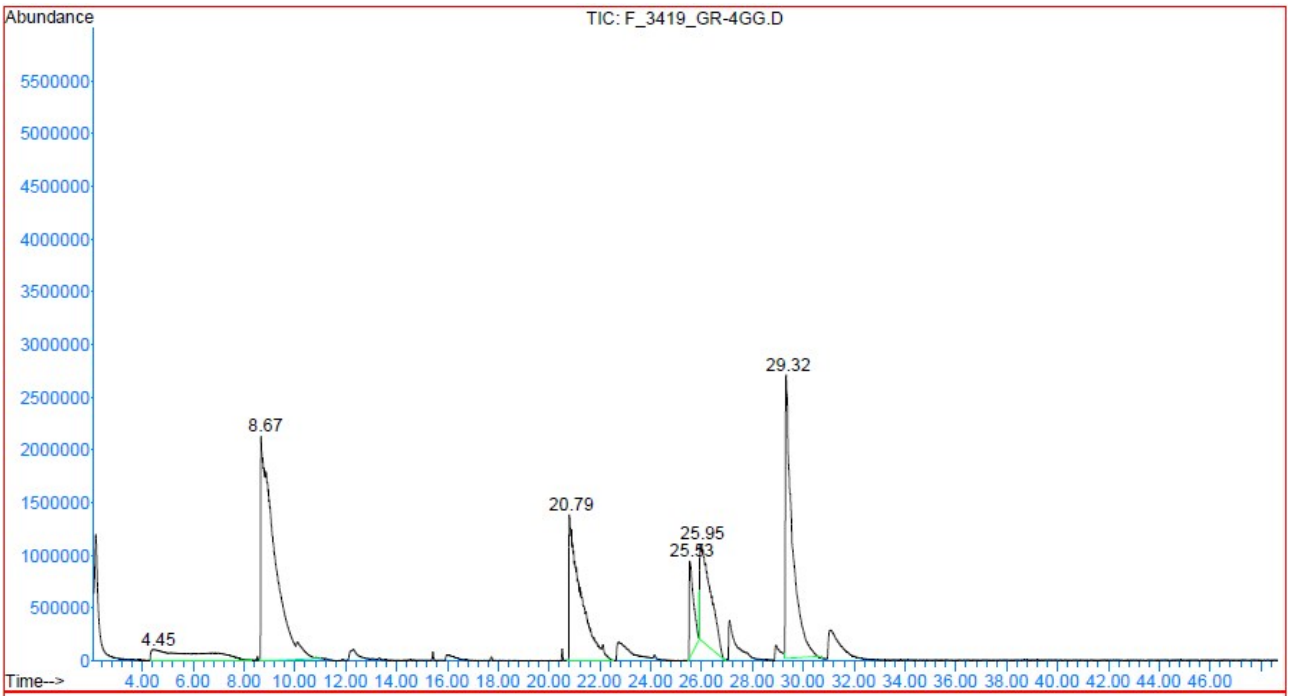


^{1}H NMR of the inseparable reaction products

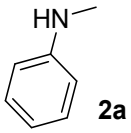




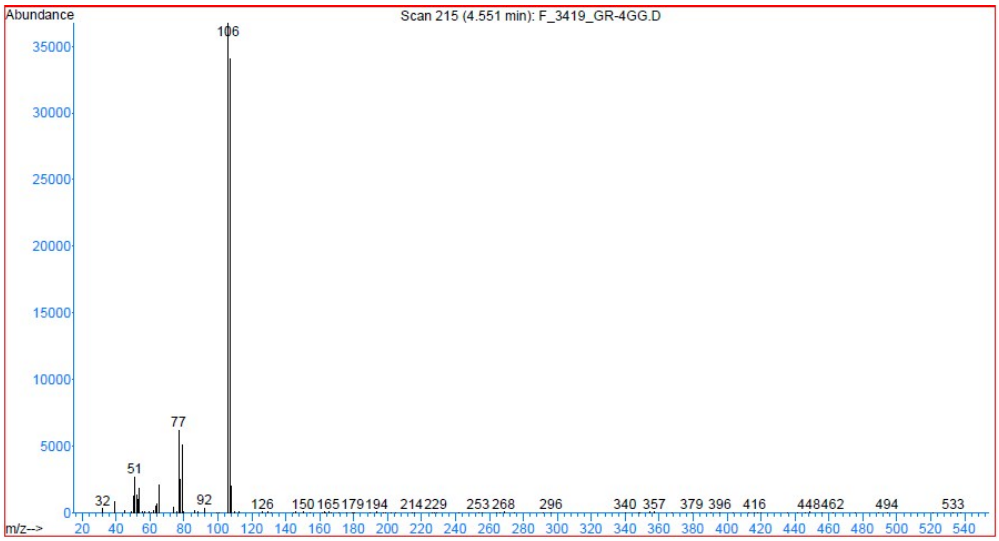
GC-MS analysis of the crude reaction mixture

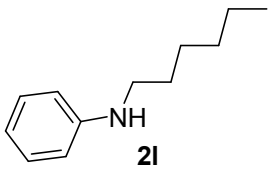


peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	4.447	193	206	539	M4	108428	151724041	19.24%	6.708%
2	8.665	569	572	774	M	2139673	788691545	100.00%	34.869%
3	20.788	1620	1624	1774	M2	1415815	437929147	55.53%	19.361%
4	25.536	2030	2036	2069	M	919366	106436526	13.50%	4.706%
5	25.950	2069	2072	2158	M2	910921	275952017	34.99%	12.200%
6	29.315	2359	2364	2487	M	2706289	501146389	63.54%	22.156%

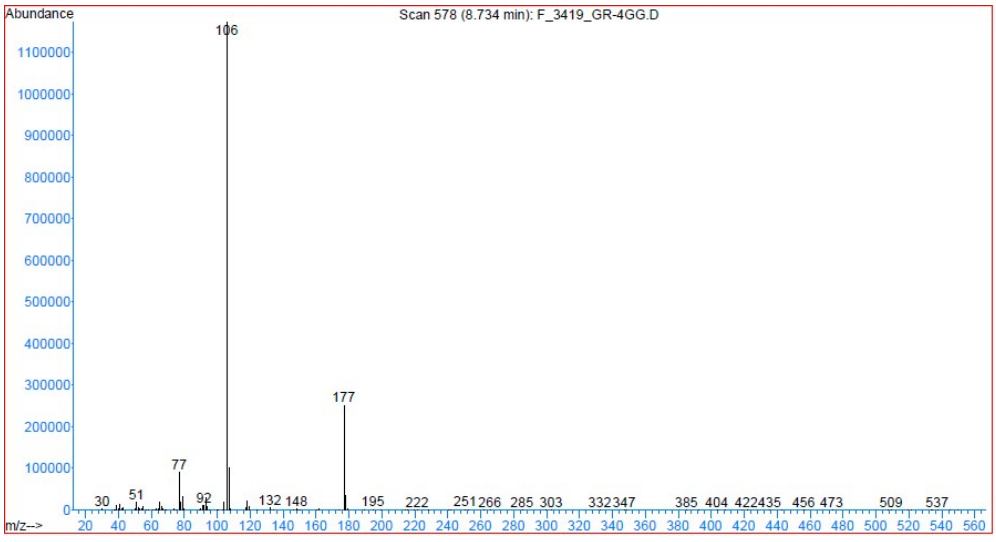


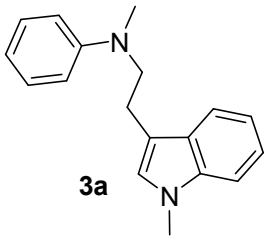
2a



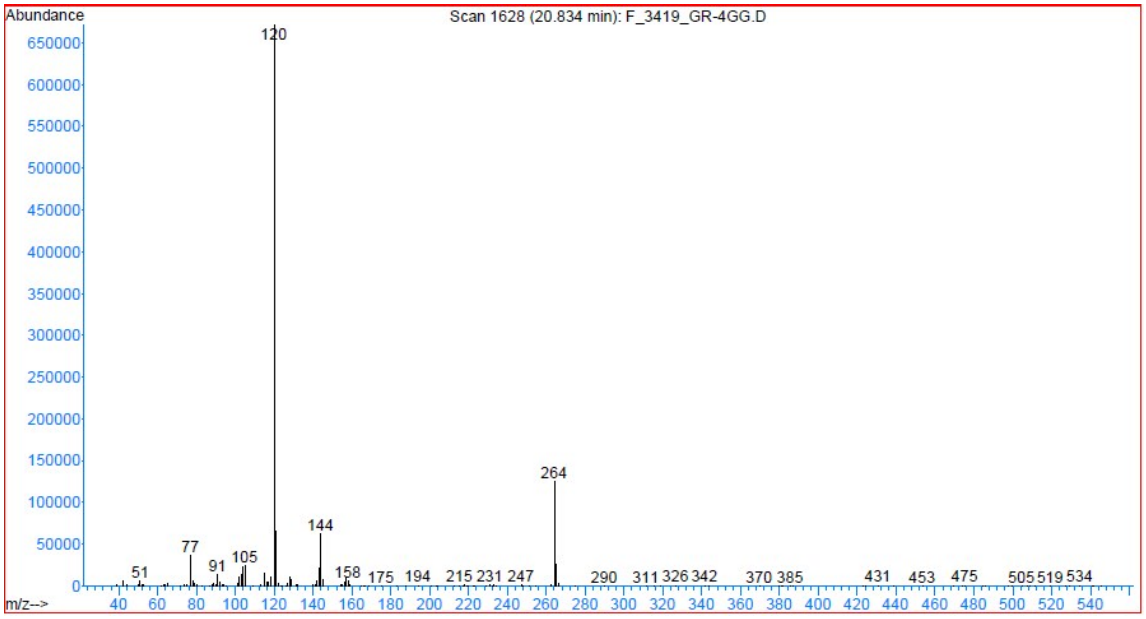


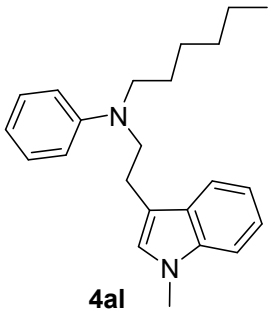
2l



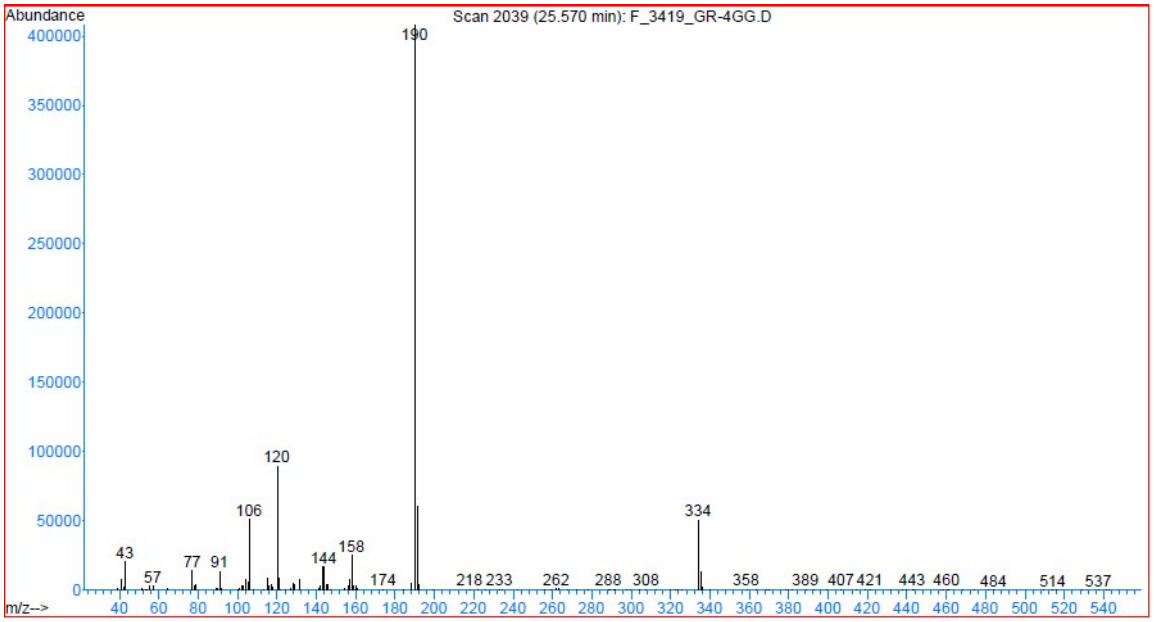


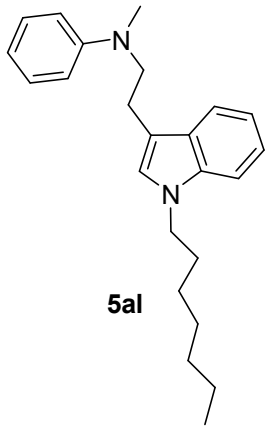
3a


Abundance
Scan 1628 (20.834 min): F_3419_GR-4GG.D
m/z-->

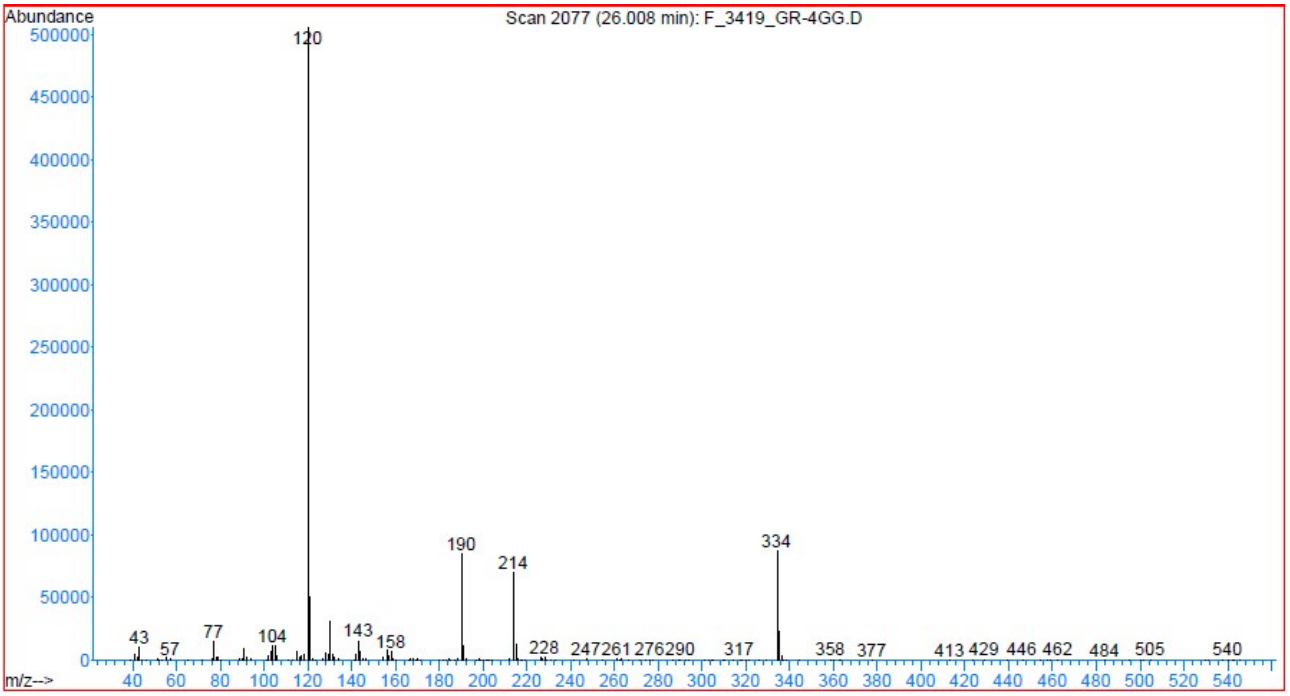


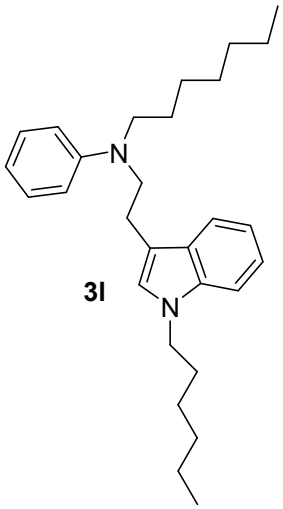
4al


Abundance
Scan 2039 (25.570 min): F_3419_GR-4GG.D
m/z-->

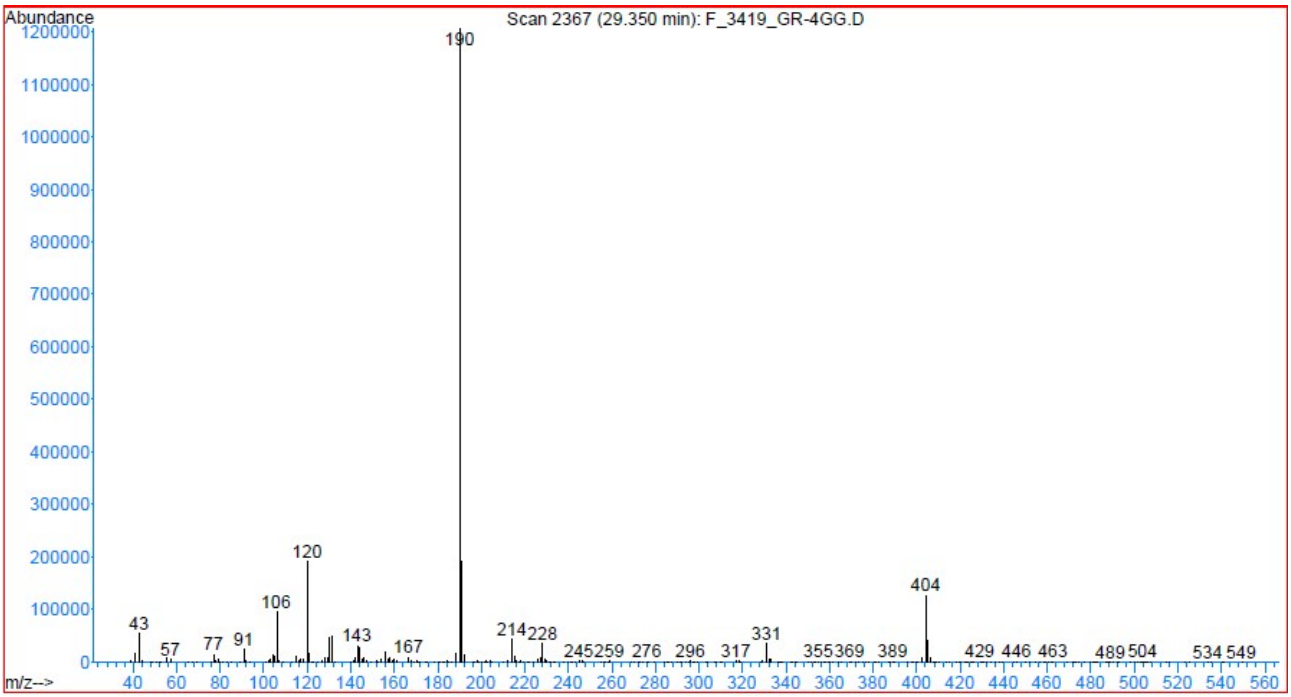


5al

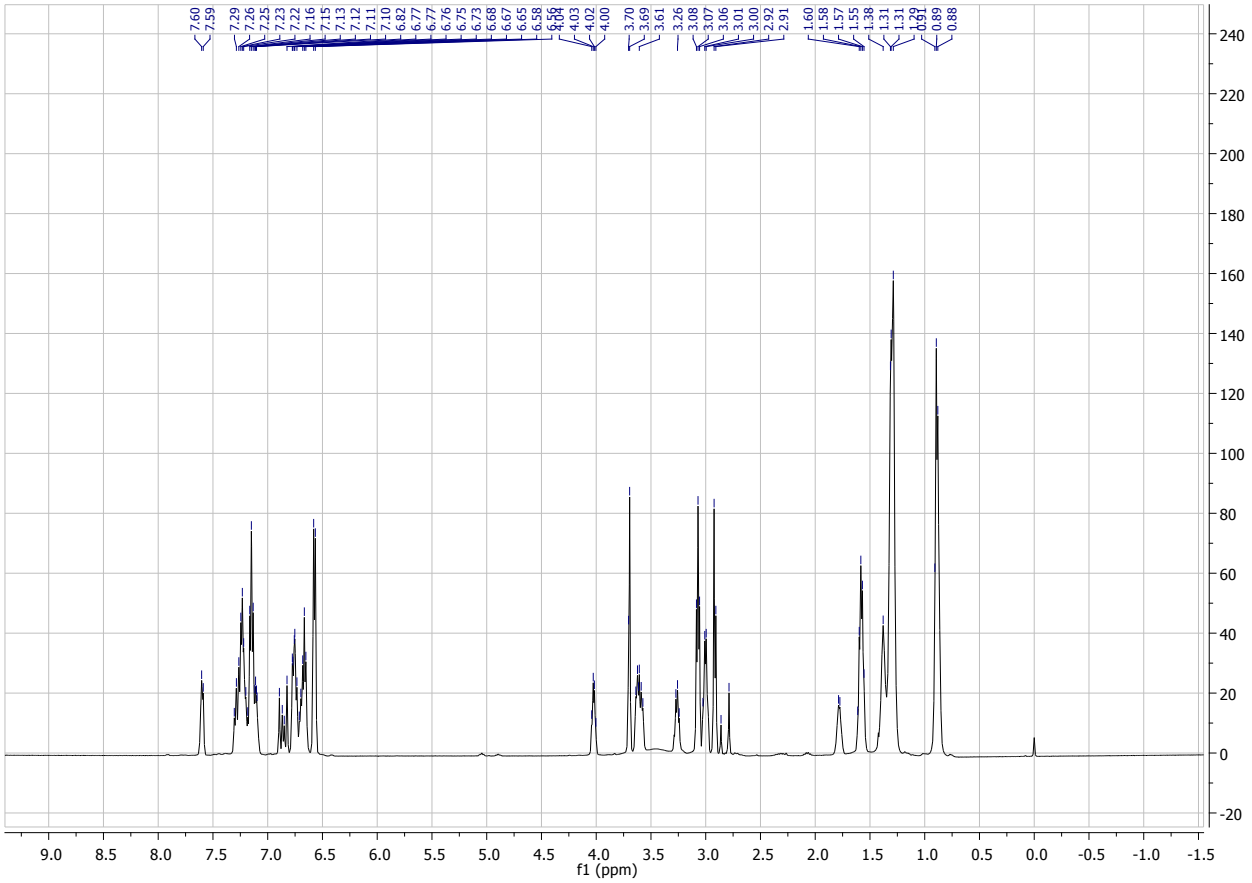

Abundance
Scan 2077 (26.008 min): F_3419_GR-4GG.D
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
m/z-->
120
190
214
334
43
57
77
104
143
158
228
247
261
276
290
317
358
377
413
429
446
462
484
505
540
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500 520 540

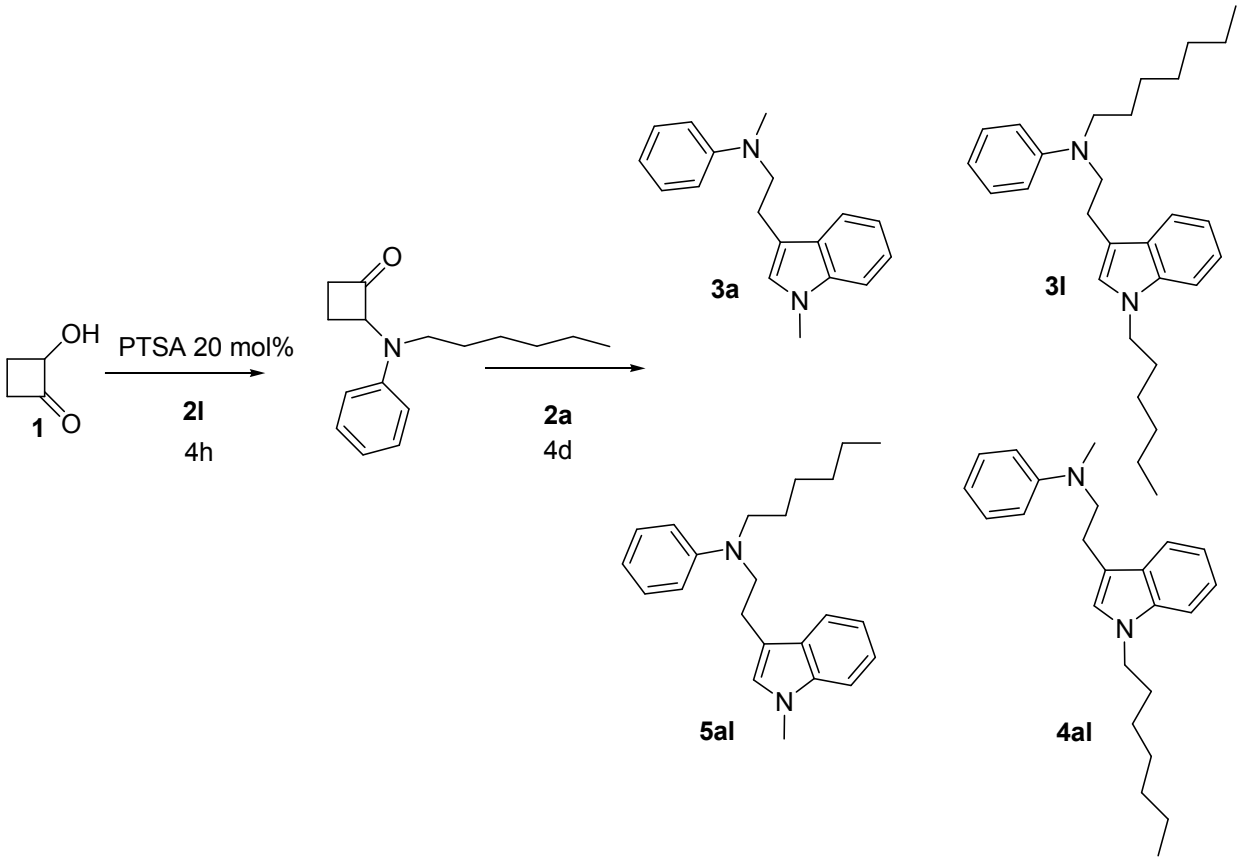


3l

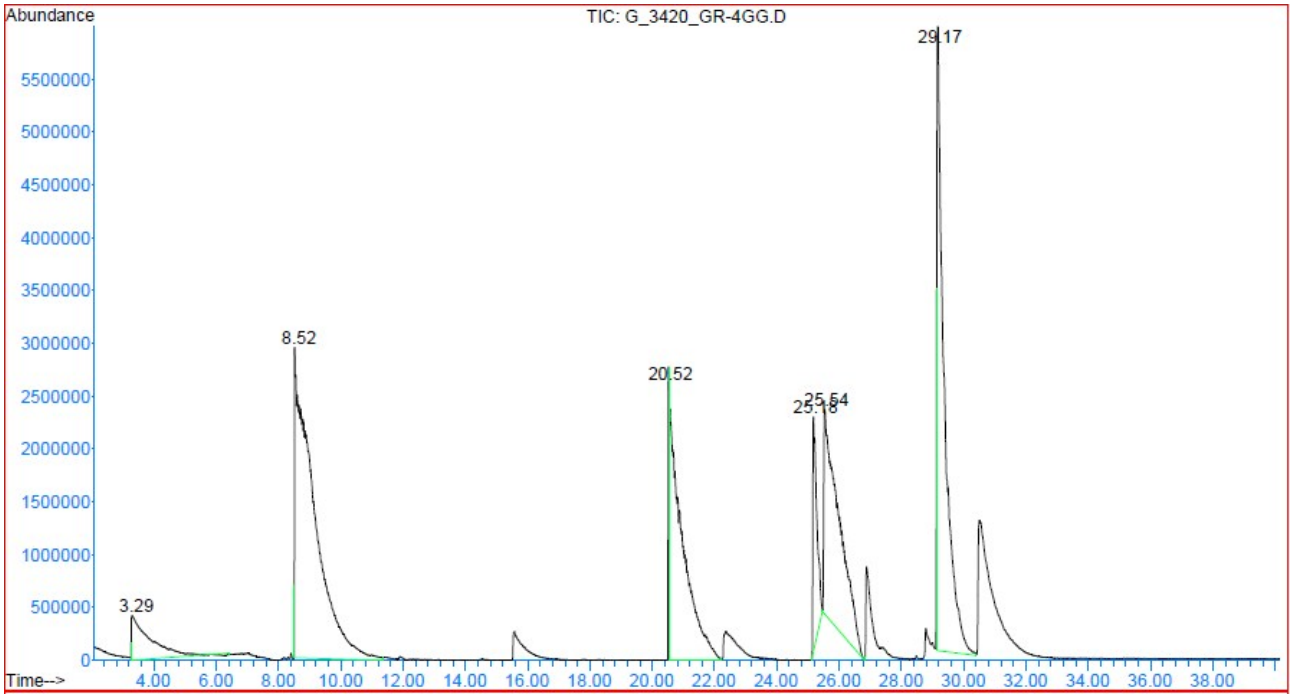

Abundance
Scan 2367 (29.350 min): F_3419_GR-4GG.D
1200000
1100000
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
m/z-->
190
120
106
404
43
57
77
91
143
167
214
228
245
259
276
296
317
331
355
369
389
429
446
463
489
504
534
549
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500 520 540 560

^{1}H NMR of the inseparable reaction products

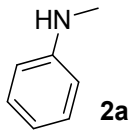




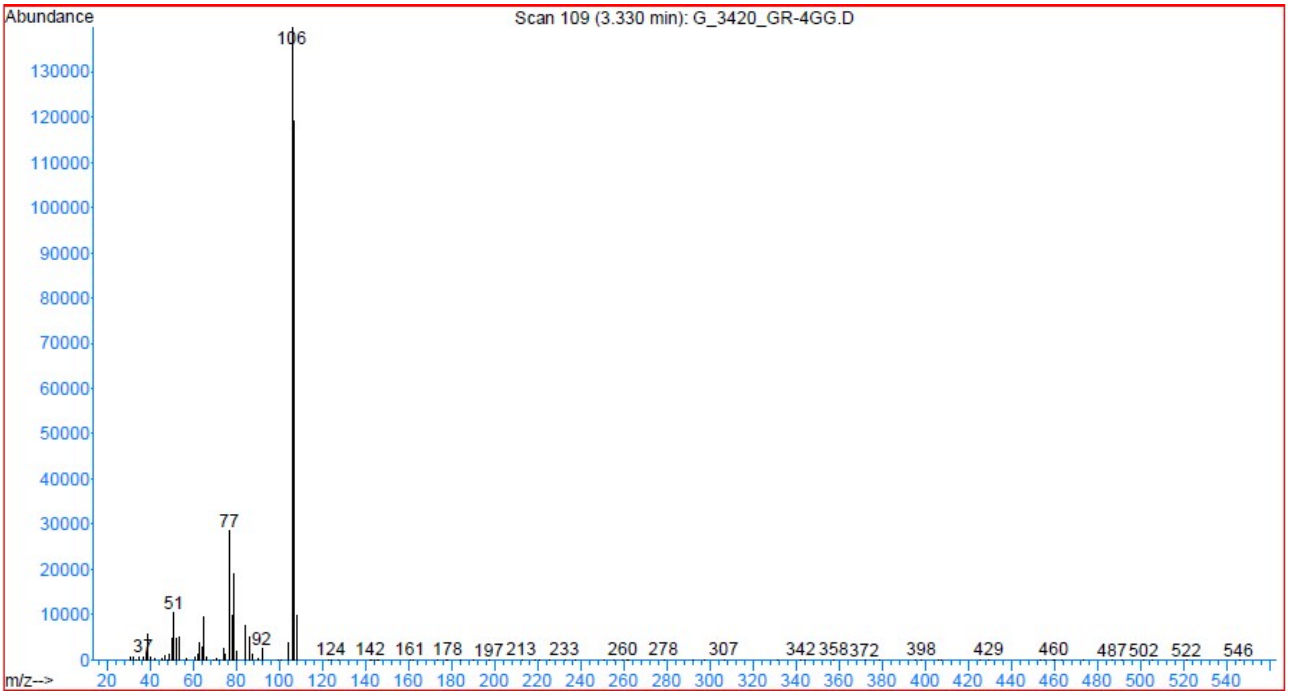
GC-MS analysis of the crude reaction mixture

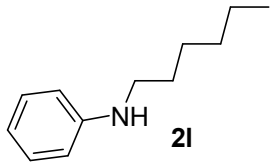


peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	3.295	103	106	376	M3	422761	173983926	14.05%	4.365%
2	8.515	558	559	805	M	2937771	1238217843	100.00%	31.062%
3	20.523	1601	1601	1749	M	2768541	751396797	60.68%	18.850%
4	25.178	1999	2005	2029	M	2226220	195303486	15.77%	4.899%
5	25.535	2029	2036	2147	M	1942798	673479764	54.39%	16.895%
6	29.165	2348	2351	2458	M	5985083	953846271	77.03%	23.929%

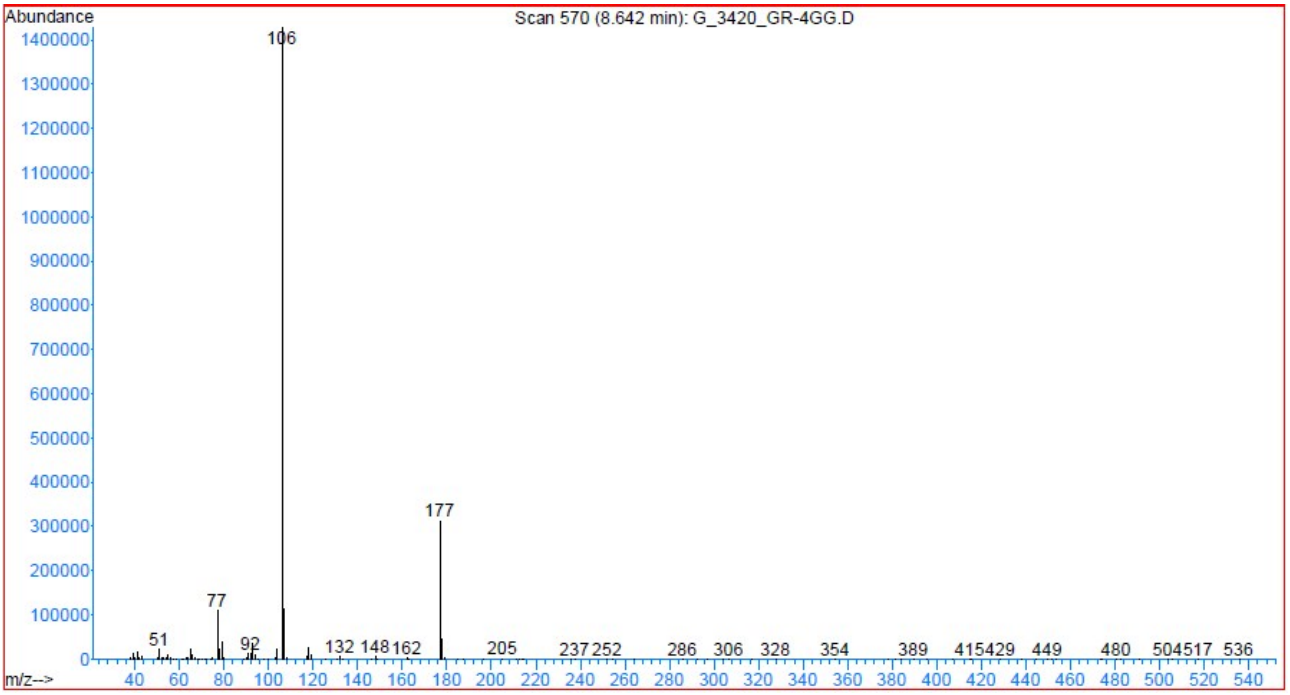


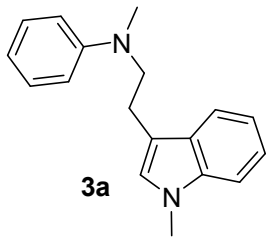
2a



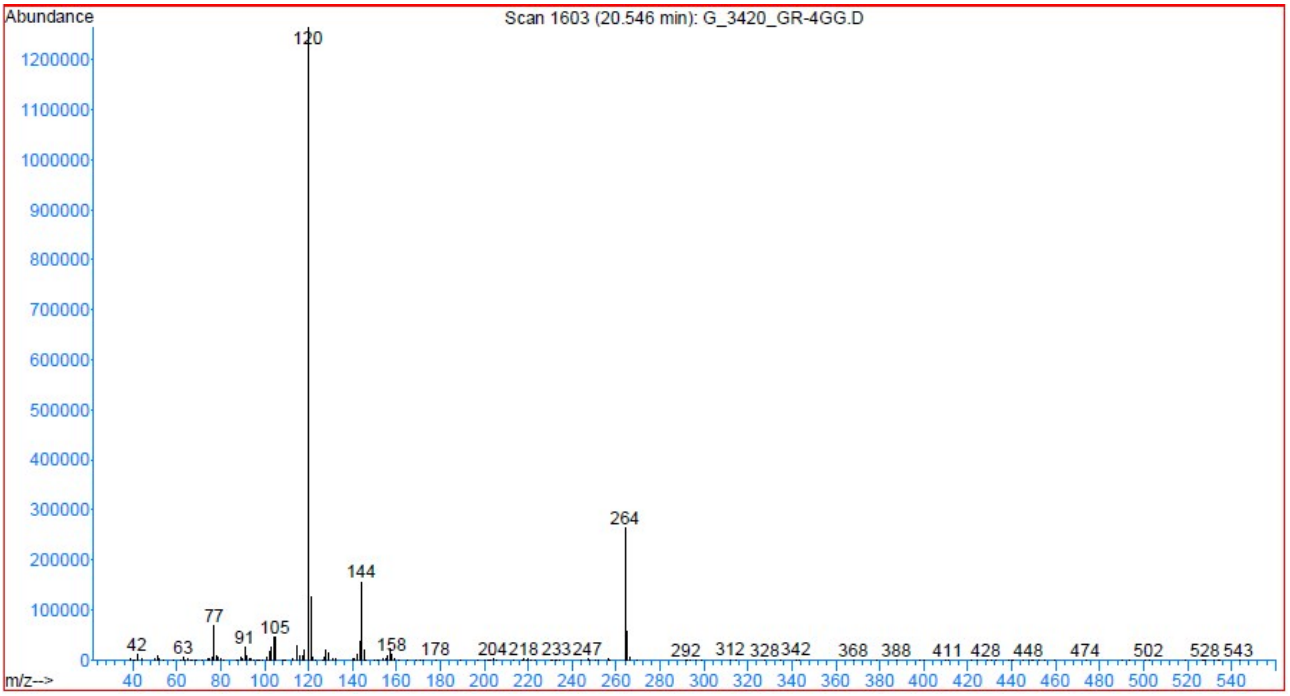

NH

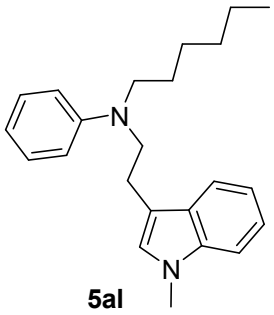
2l


Abundance
Scan 570 (8.642 min): G_3420_GR-4GG.D
106
177
77
51
92
m/z-->

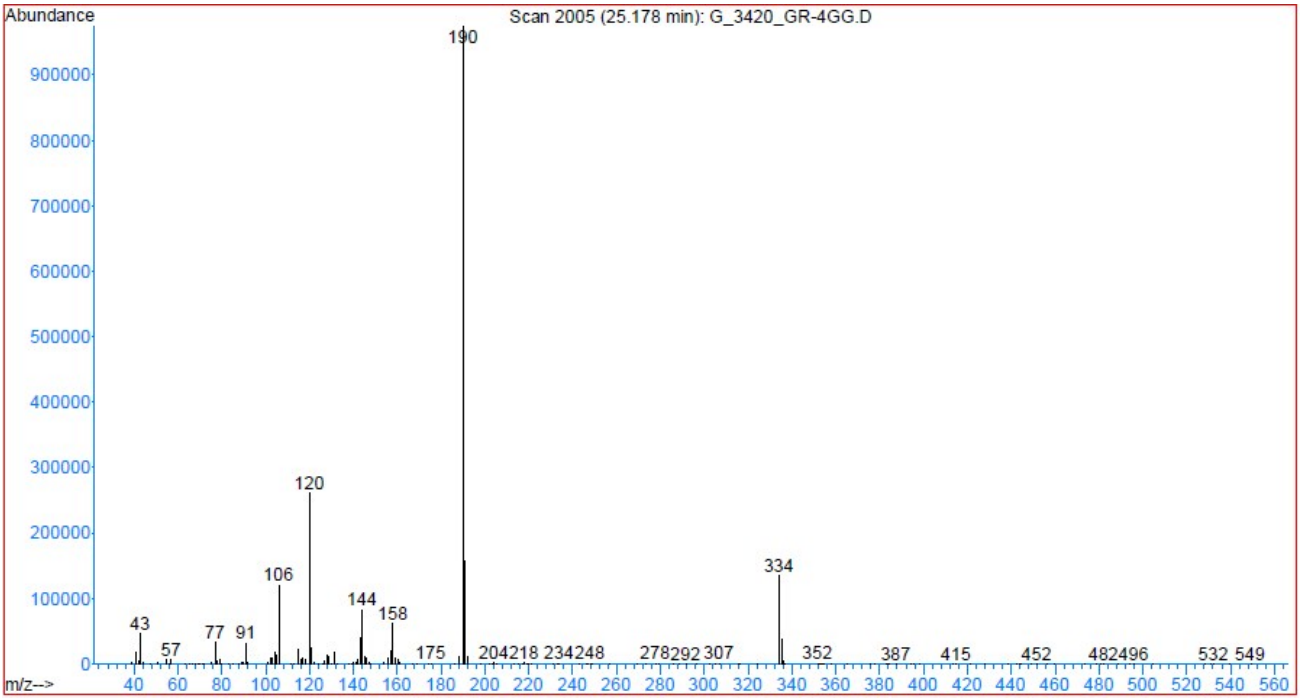

N
N

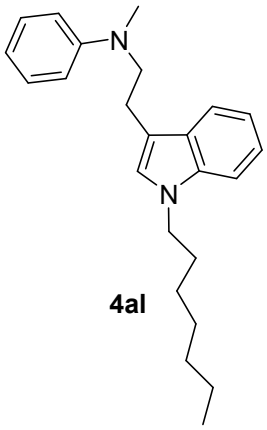
3a


Abundance
Scan 1603 (20.546 min): G_3420_GR-4GG.D
120
264
144
77
105
91
42
63
158
178
m/z-->

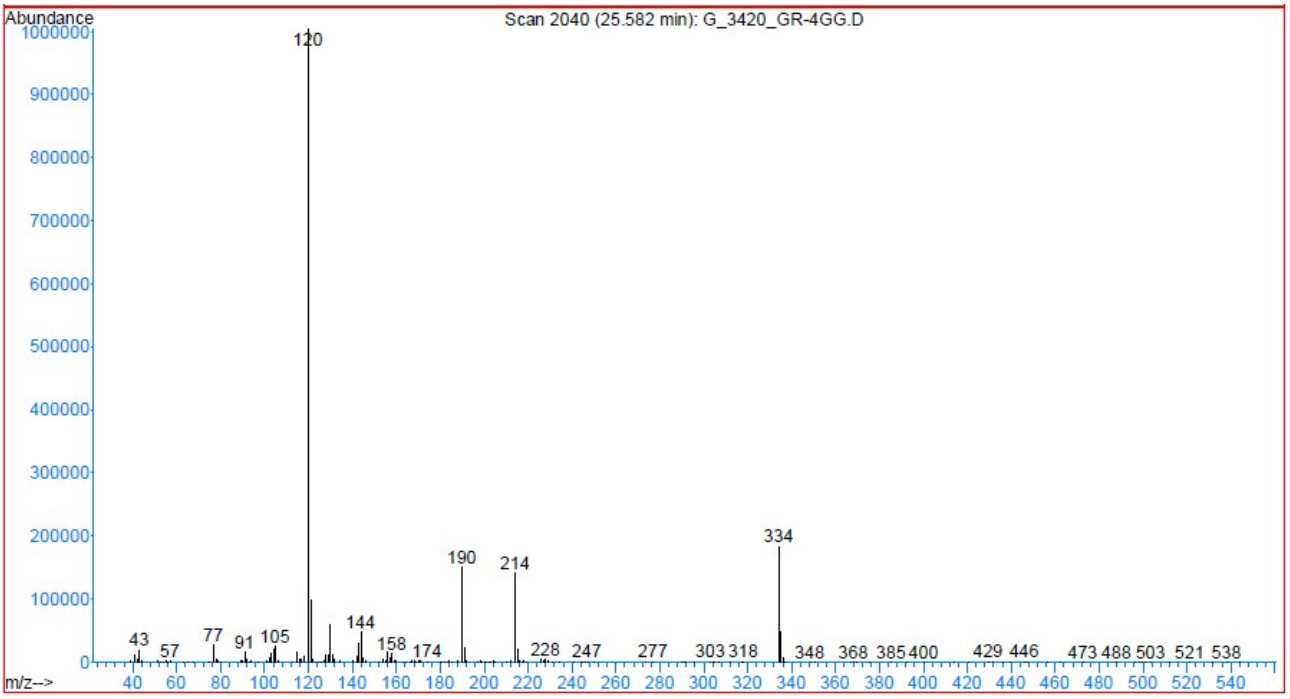


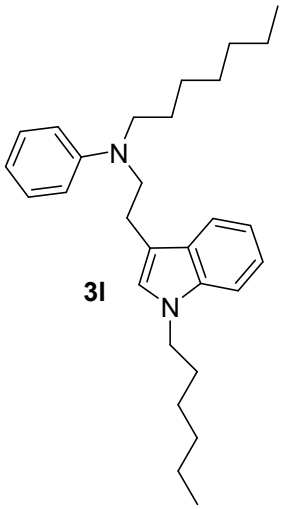
5al


Abundance
Scan 2005 (25.178 min): G_3420_GR-4GG.D
190
120
106
334
144
158
43
57
77
91
175
204218
234248
278292
307
352
387
415
452
482496
532
549
m/z-->

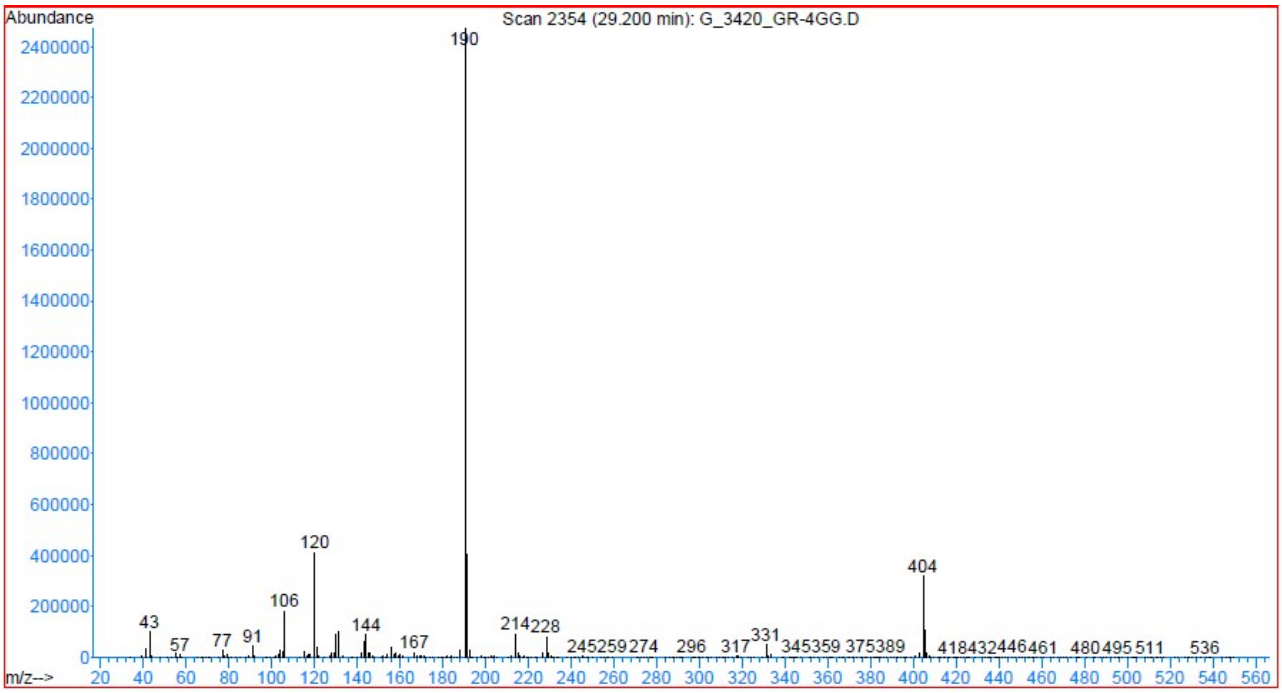


4al

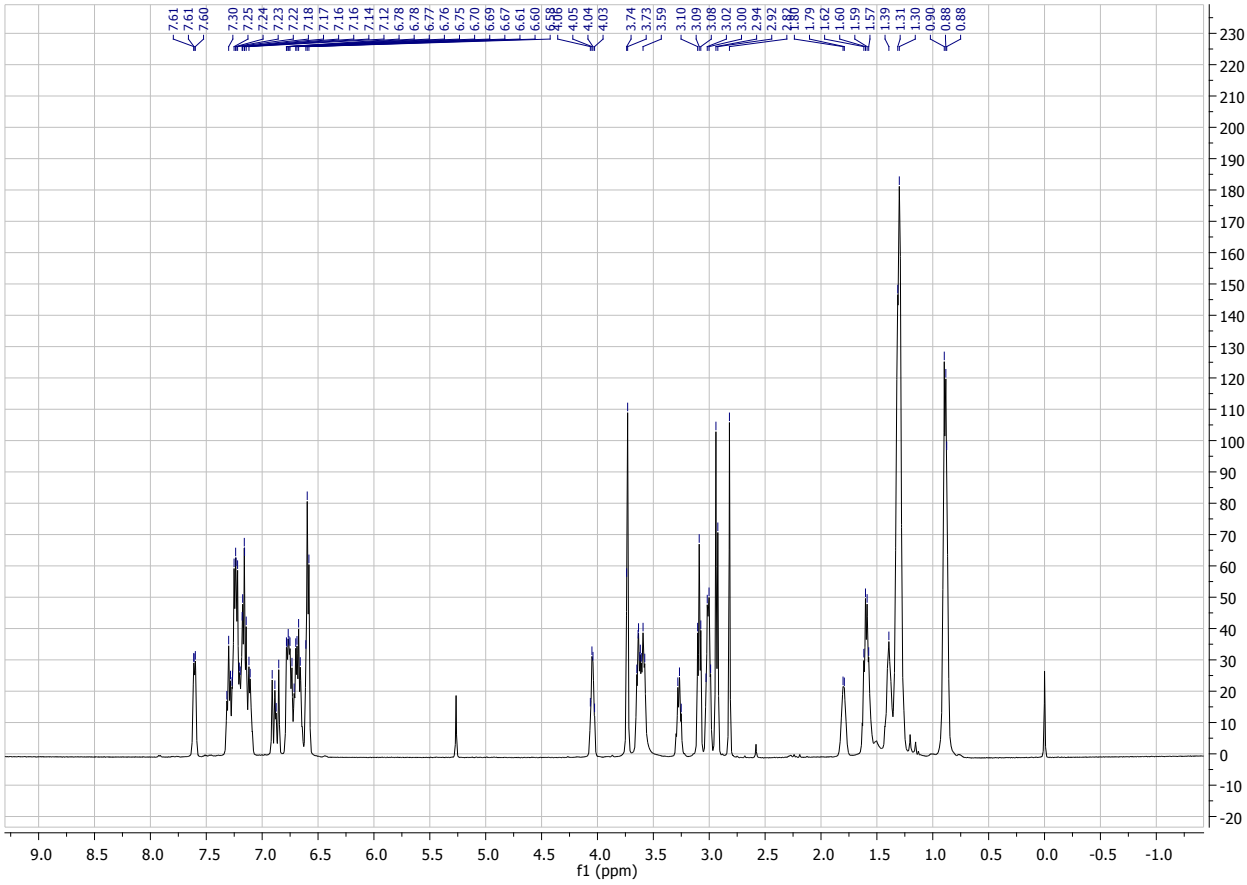

Abundance
Scan 2040 (25.582 min): G_3420_GR-4GG.D
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
m/z-->
120
334
190
214
144
43
57
77
91
105
158
174
228
247
277
303
318
348
368
385
400
429
446
473
488
503
521
538
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500 520 540

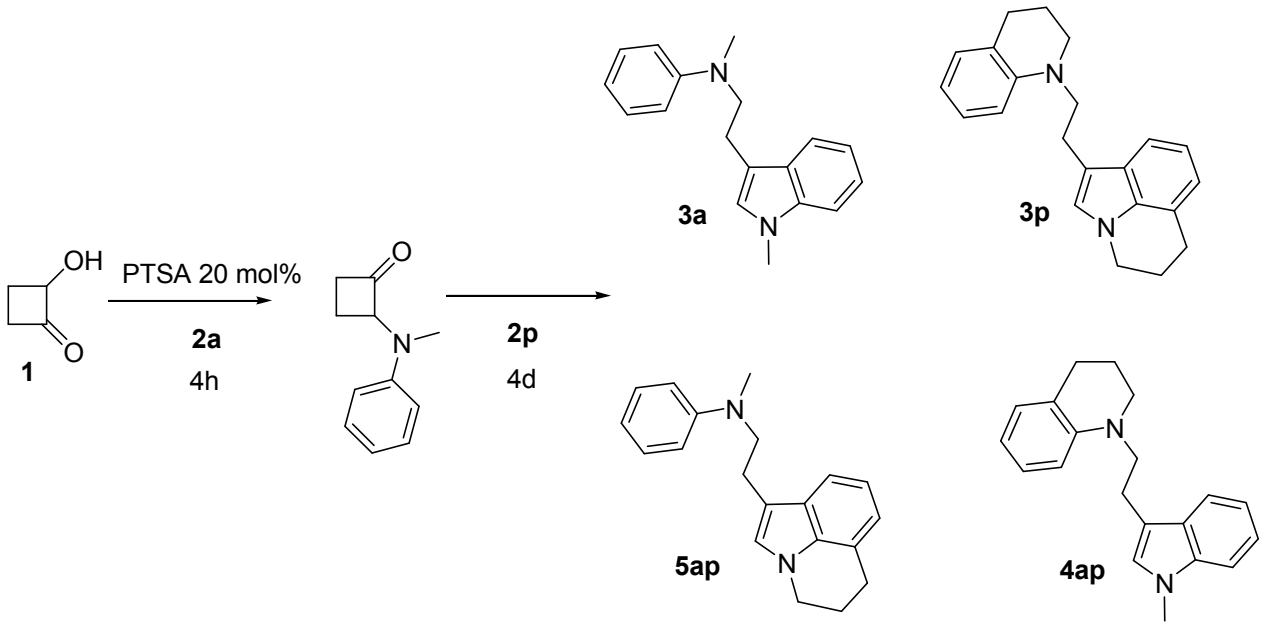


3l

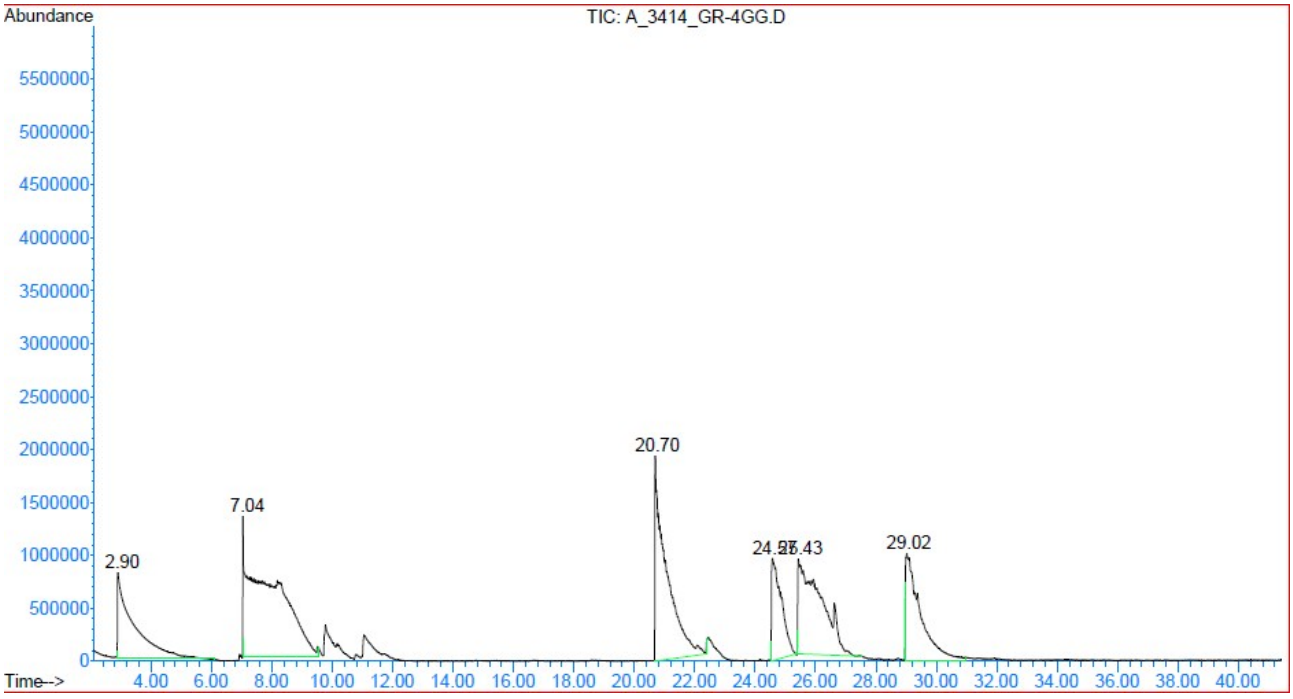

Abundance
Scan 2354 (29.200 min): G_3420_GR-4GG.D
190
120
404
106
43
57
77
91
144
167
214
228
245
259
274
296
317
331
345
359
375
389
418
432
446
461
480
495
511
536
m/z-->

^{1}H NMR of the inseparable reaction products

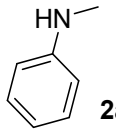




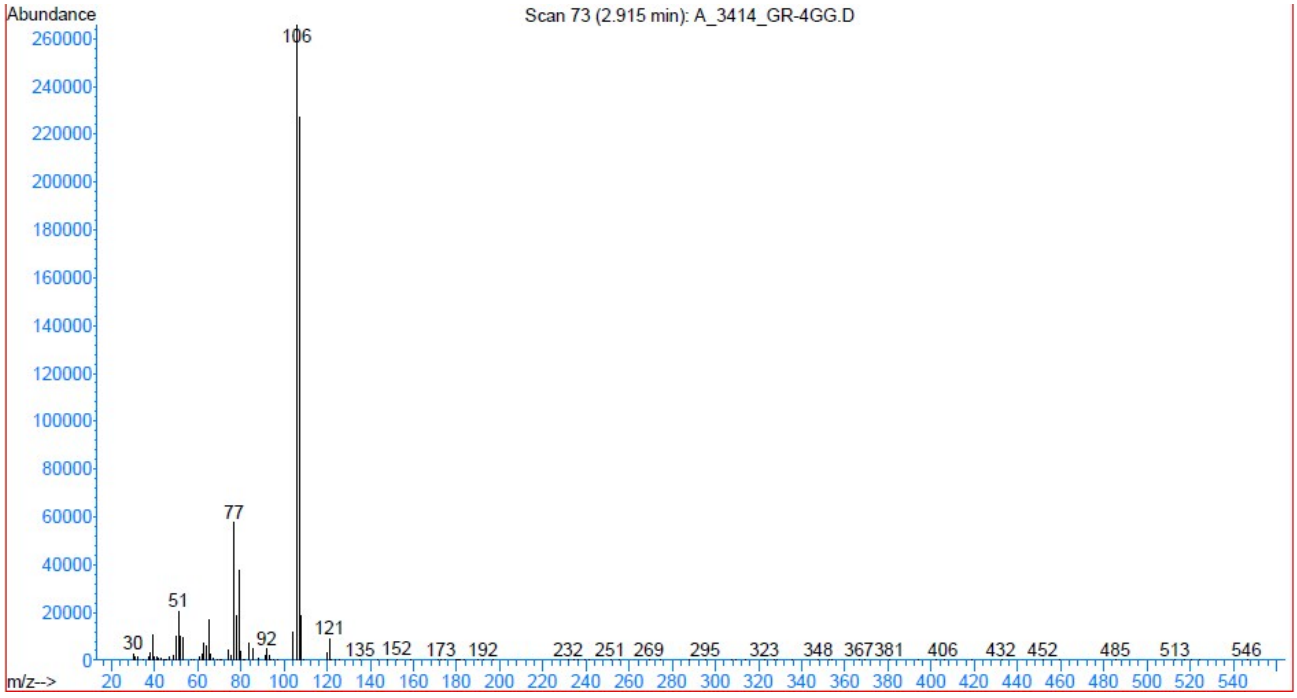
GC-MS analysis of the crude reaction mixture

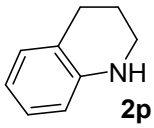


peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	2.903	69	72	307	M2	792365	273333012	35.76%	10.605%
2	7.040	430	431	647	M5	1325940	764335321	100.00%	29.656%
3	20.696	1613	1616	1765	M	1939018	503342442	65.85%	19.529%
4	24.579	1949	1953	2021	M2	970010	217151534	28.41%	8.425%
5	25.432	2024	2027	2208	M2	899382	473278110	61.92%	18.363%
6	29.027	2334	2339	2508	M2	1020174	345934134	45.26%	13.422%

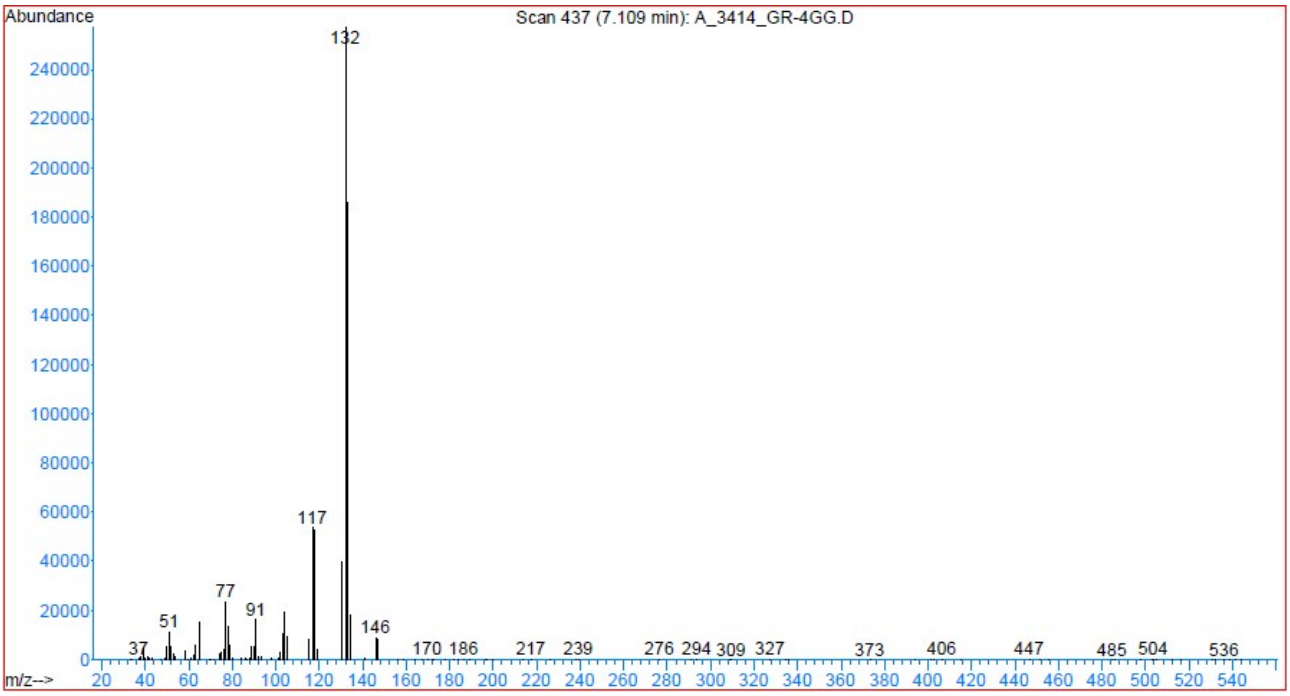


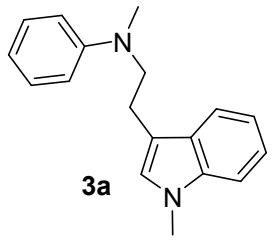
2a



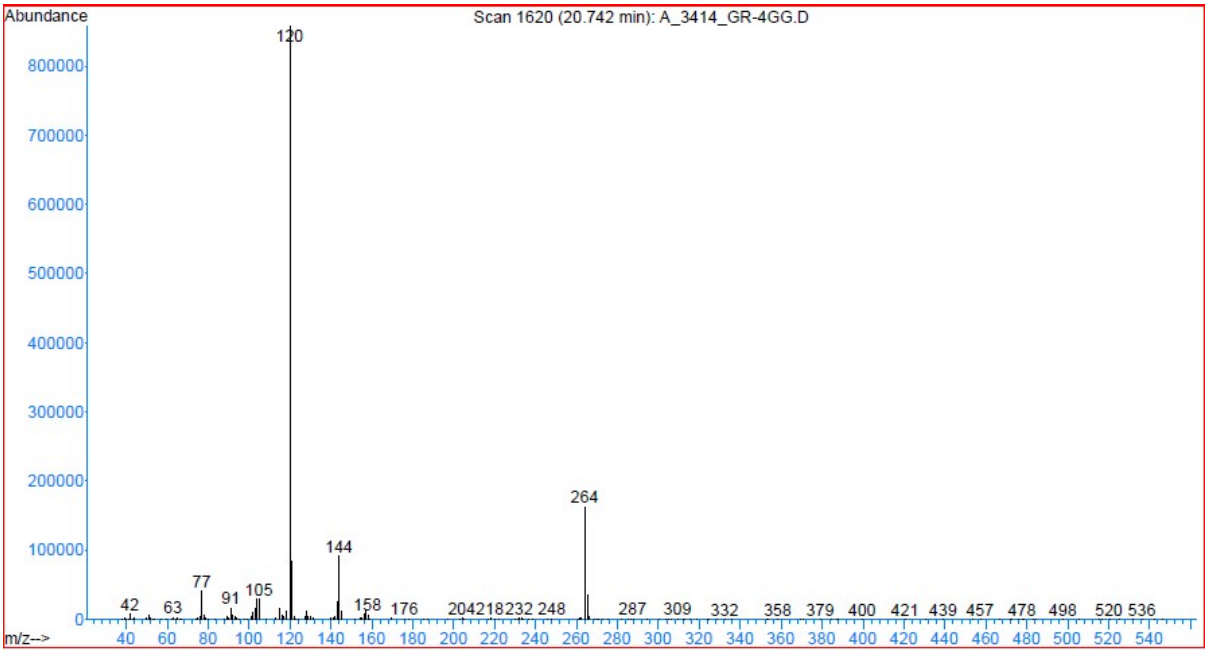


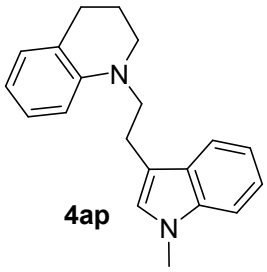
2p


Abundance
Scan 437 (7.109 min): A_3414_GR-4GG.D
132
117
77
91
51
146
37
170 186
217 239
276 294 309 327
373
406
447
485 504
536
m/z-->

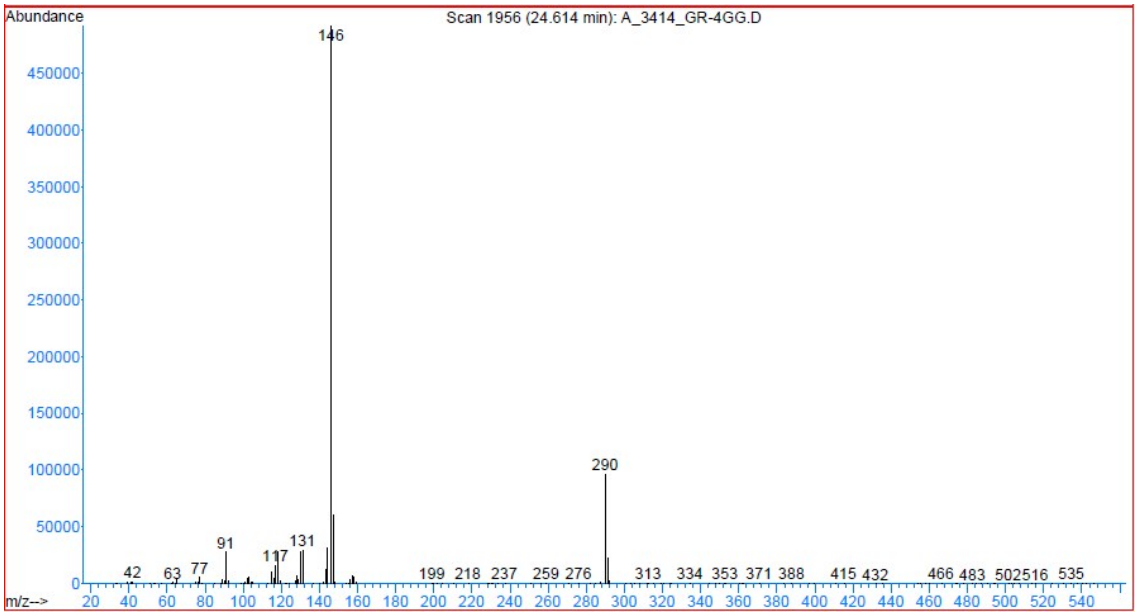

N
N

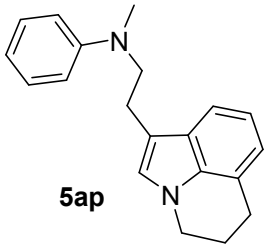
3a


Abundance
Scan 1620 (20.742 min): A_3414_GR-4GG.D
120
264
144
77
91
105
42
63
158
176
204218232 248
287
309
332
358
379
400
421
439 457
478
498
520 536
m/z-->

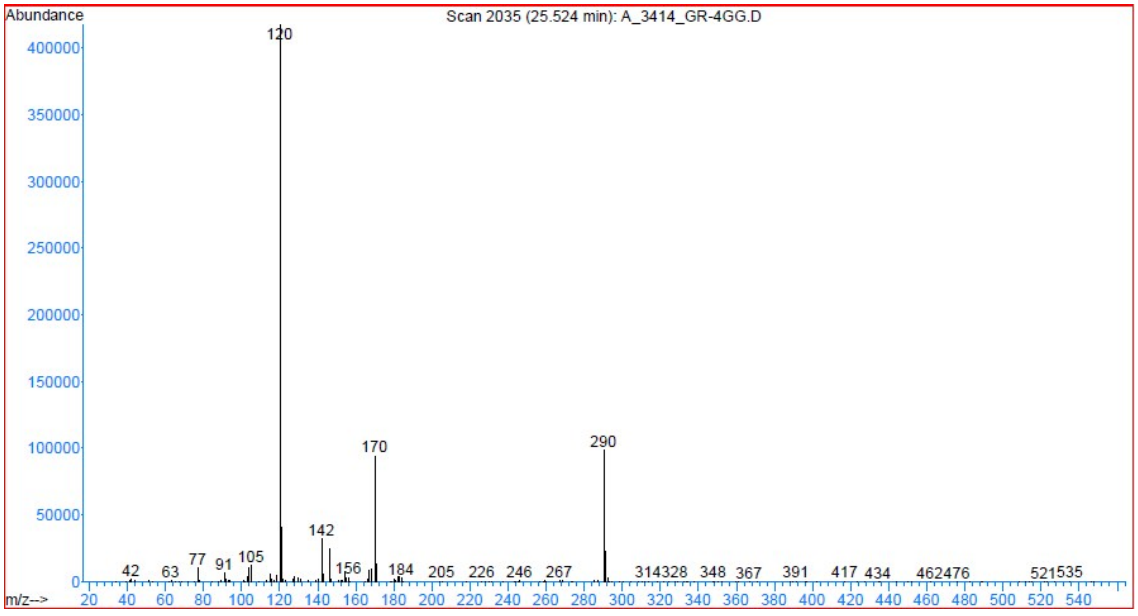


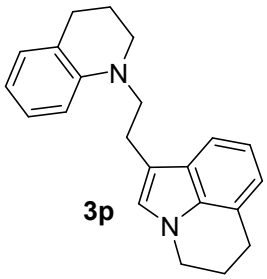
4ap


Abundance
Scan 1956 (24.614 min): A_3414_GR-4GG.D
146
290
91
131
117
42
63
77
199
218
237
259
276
313
334
353
371
388
415
432
466
483
502
516
535
m/z-->

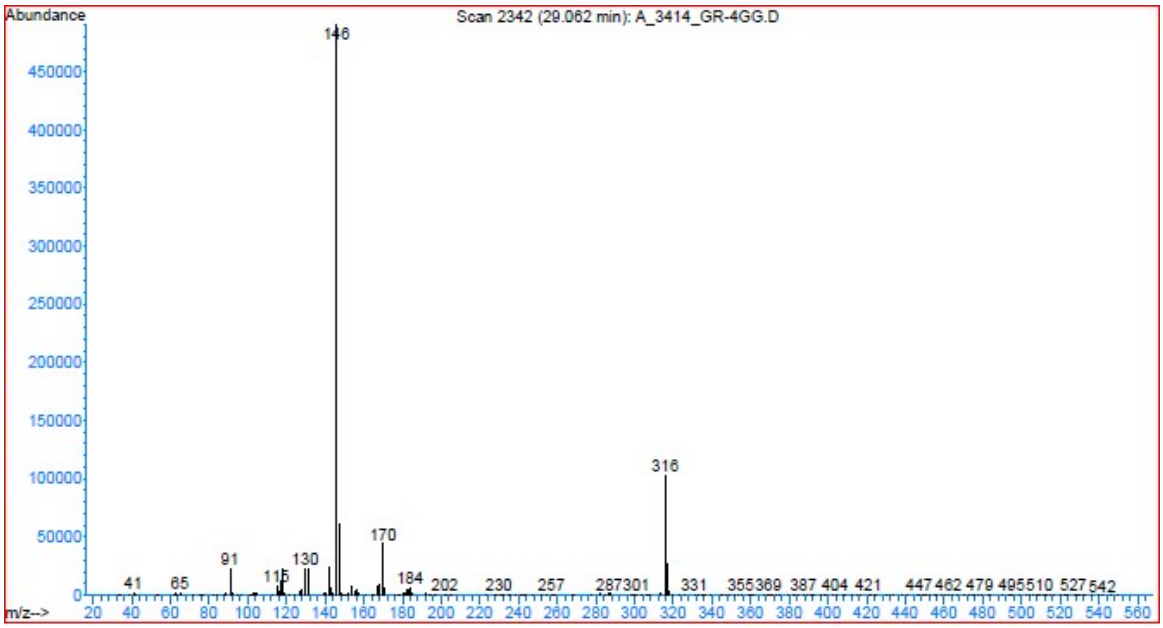


5ap

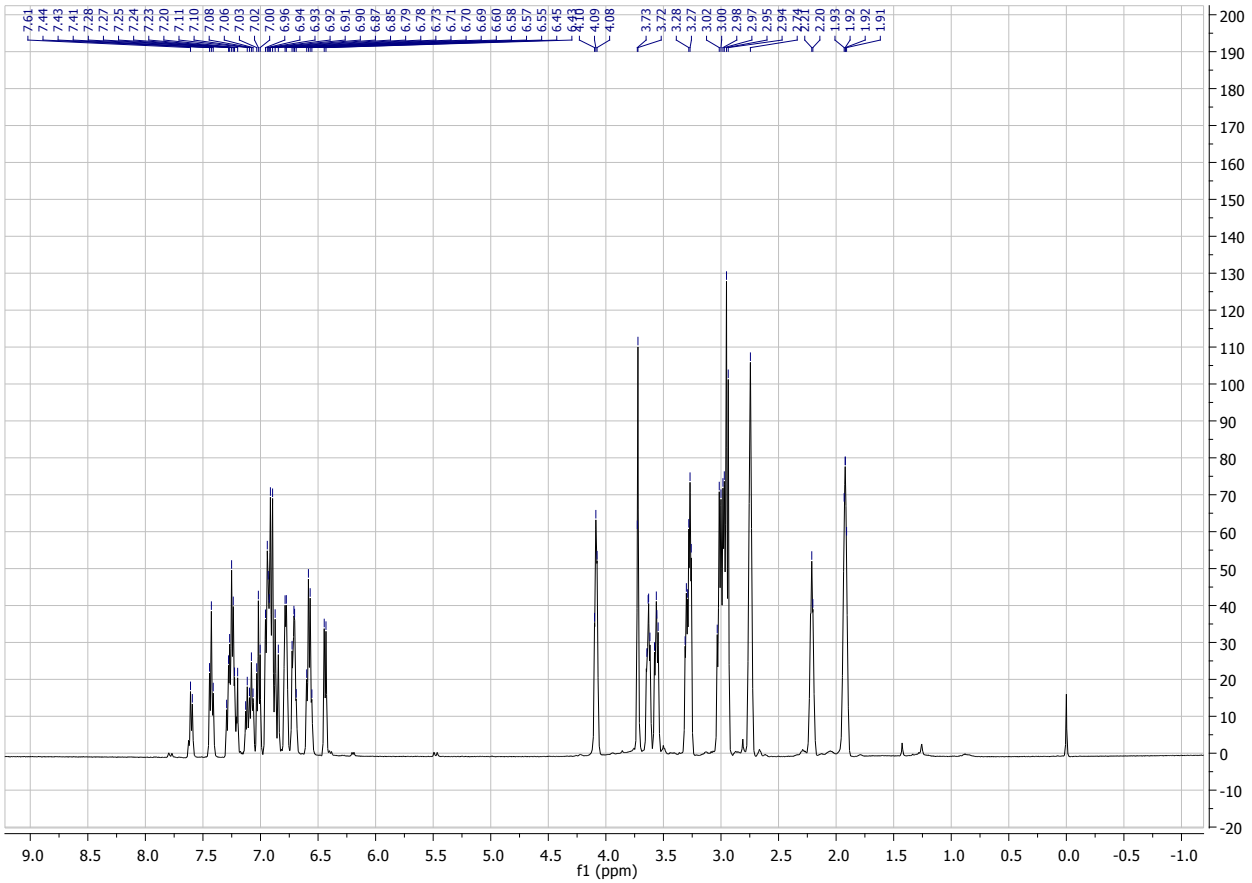

Abundance
Scan 2035 (25.524 min): A_3414_GR-4GG.D
120
170
290
142
42
63
77
91
105
156
184
205
226
246
267
314
328
348
367
391
417
434
462
476
521
535
m/z-->

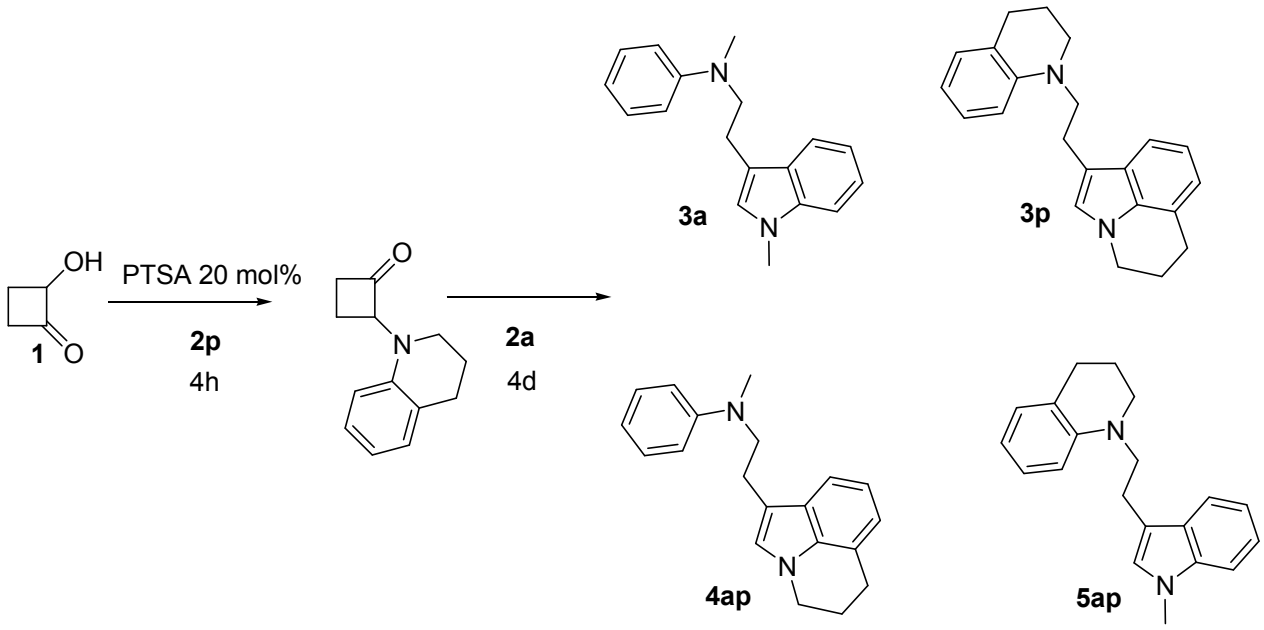


3p

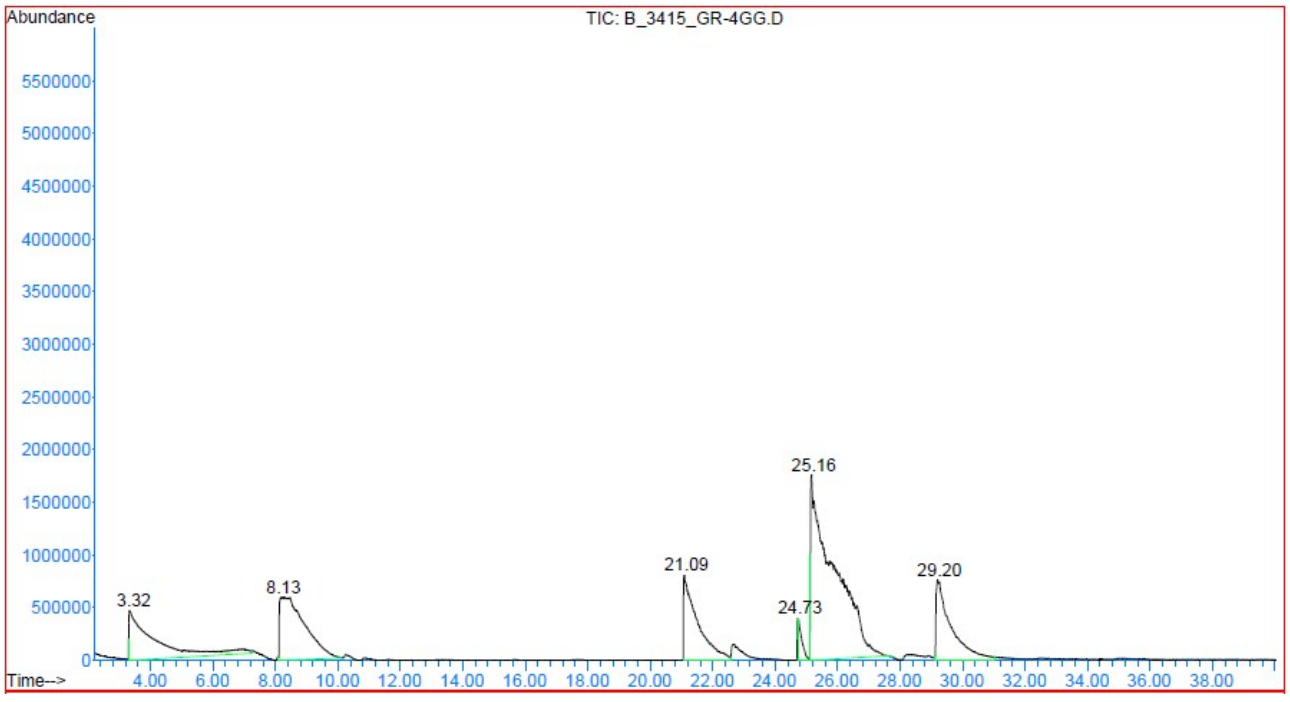


^{1}H NMR of the inseparable reaction products

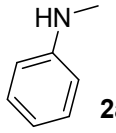

7.61
7.44
7.43
7.41
7.28
7.27
7.25
7.24
7.23
7.20
7.11
7.10
7.08
7.06
7.03
7.02
7.00
6.96
6.94
6.93
6.92
6.91
6.90
6.87
6.85
6.79
6.78
6.73
6.71
6.70
6.69
6.60
6.58
6.57
6.55
6.45
4.10
4.09
4.08
3.73
3.72
3.28
3.27
3.02
3.00
2.98
2.97
2.95
2.94
2.24
2.21
2.20
1.93
1.92
1.92
1.91
f1 (ppm)



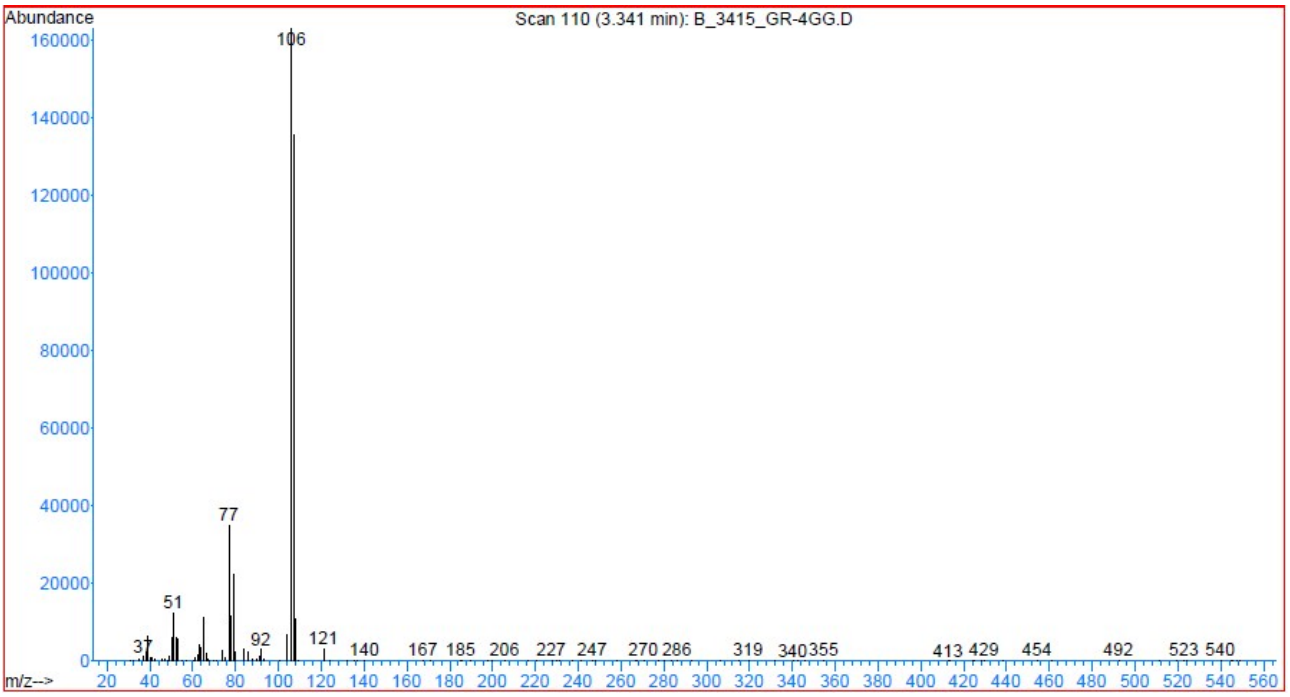
GC-MS analysis of the crude reaction mixture

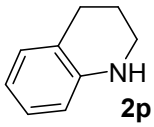


peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	3.318	107	108	450	M2	486701	263649548	29.51%	12.977%
2	8.123	523	525	696	M6	617000	336696452	37.69%	16.572%
3	21.099	1647	1651	1779	M	814946	249110163	27.89%	12.261%
4	24.729	1966	1966	1997	M4	400517	33792420	3.78%	1.663%
5	25.167	2001	2004	2226	M	1770074	893302144	100.00%	43.969%
6	29.200	2347	2354	2514	M	769010	255132934	28.56%	12.558%

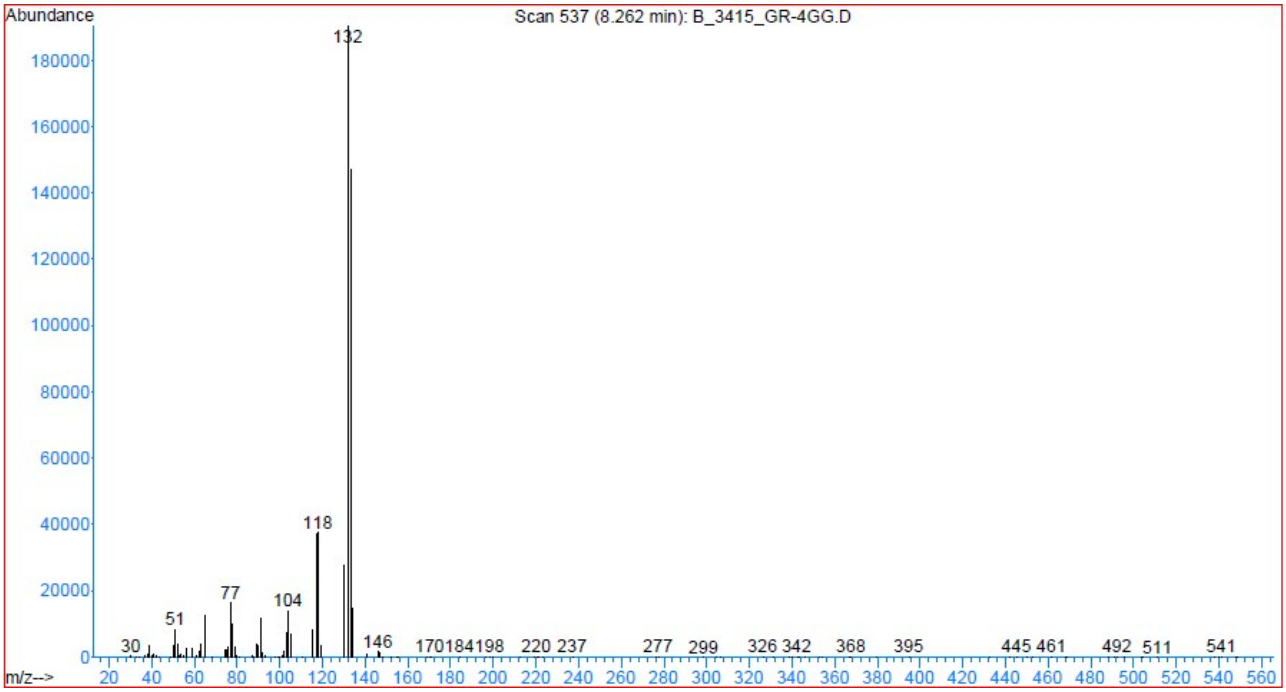


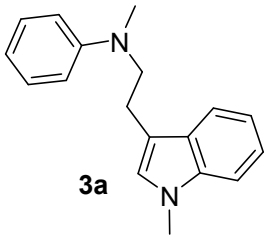
2a



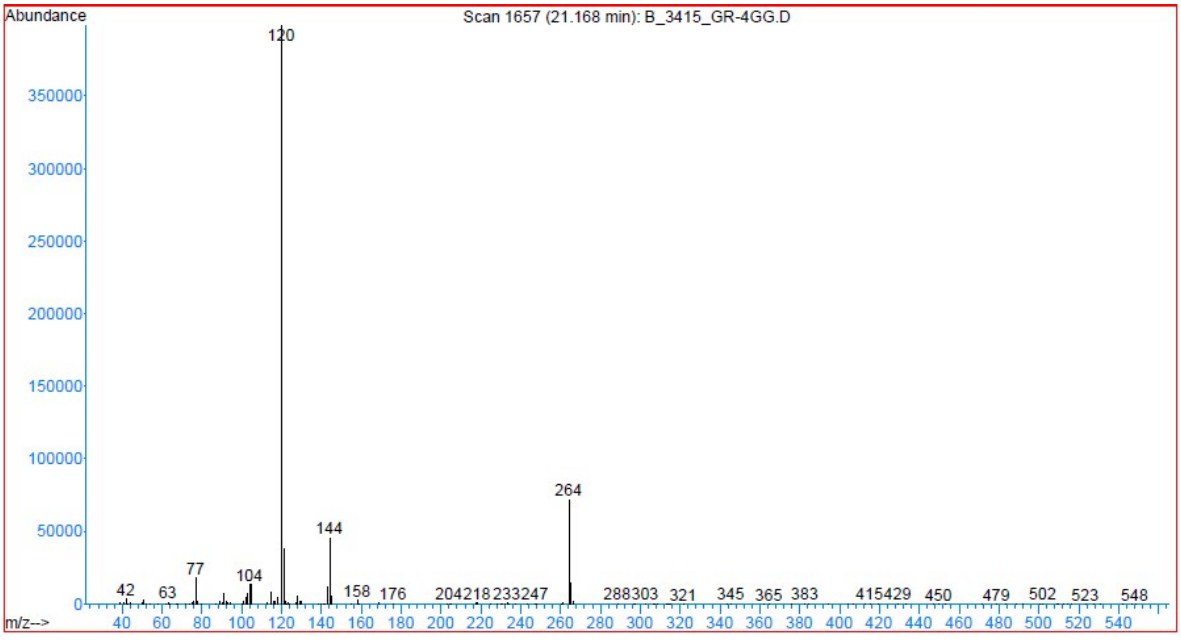

NH

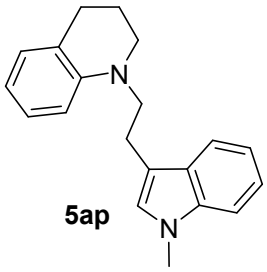
2p


Abundance
Scan 537 (8.262 min): B_3415_GR-4GG.D
132
118
77
104
51
146
m/z-->

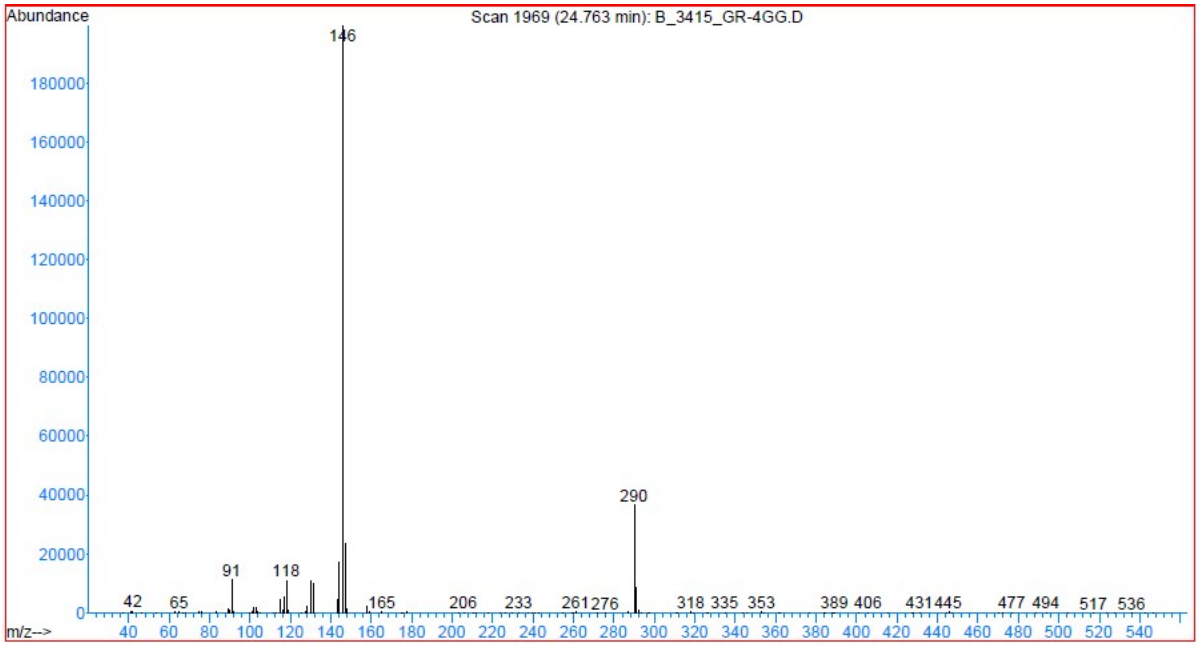

N
N

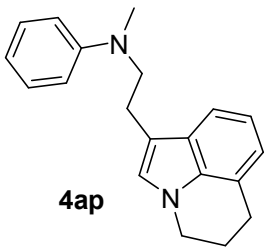
3a


Abundance
Scan 1657 (21.168 min): B_3415_GR-4GG.D
120
264
144
77
104
42
63
158
176
m/z-->

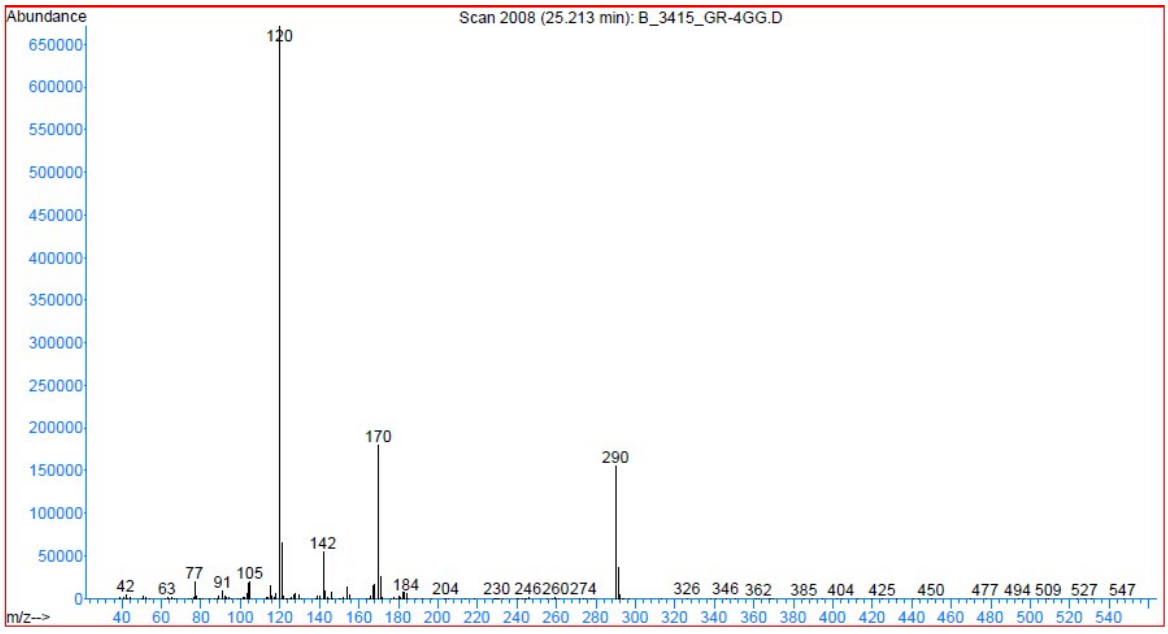


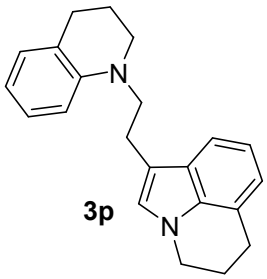
5ap


Abundance
Scan 1969 (24.763 min): B_3415_GR-4GG.D
146
290
91
118
m/z-->

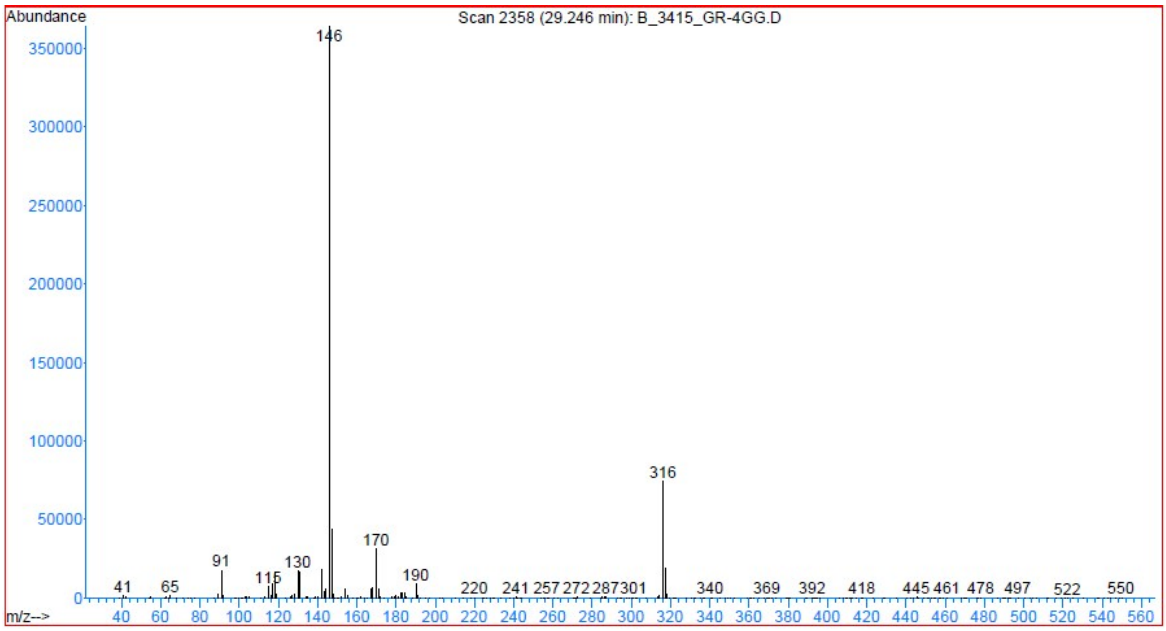


4ap


Abundance
Scan 2008 (25.213 min): B_3415_GR-4GG.D
120
170
290
142
m/z-->



3p



^{1}H NMR of the inseparable reaction products