Supporting information

Intramolecular Redox Reaction for the Synthesis of N-ArylPyrroles Catalyzed by Lewis Acids

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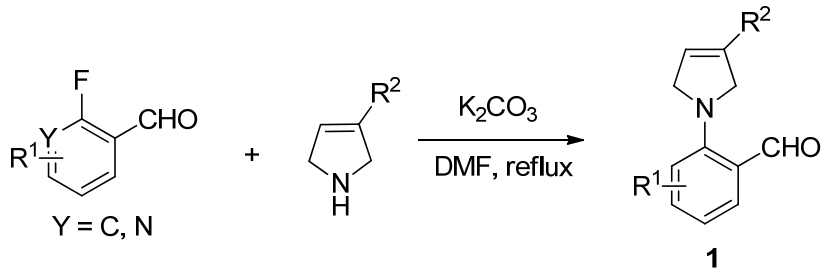
General Information.

^{1}H and ^{13}C NMR spectra were recorded on an ACF* 300Q Bruker or ACF* 500Q Bruker spectrometer. Low- and high-resolution mass spectra (LRMS and HRMS) were recorded in electron impact mode. The mass analyzer type used for the HRMS measurements was TOF. Reactions were monitored by TLC on silica gel 60 F254 plates (Qingdao Ocean Chemical Company, China). Column chromatography was carried out on silica gel (200-300 mesh, Qingdao Ocean Chemical Company, China). Data for ^{1}H NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, br = broad singlet, coupling constant(s) in Hz, integration). Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm).

General procedure for synthesis of substituted 3-pyrroline 1.



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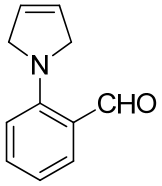
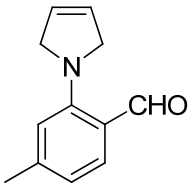
To a solution of 2-haloaldehyde (1.0 equiv.) and potassium carbonate (1.0-1.2 equiv.) in DMF was added the 3-pyrroline (1.0-1.2 equiv). The mixture was stirredat reflux until complete consumption monitored by TLC. The reaction was cooled to room temperature, diluted with water, and extracted with EtOAc (3 × 10 mL). The combined organic layers were washed with saturated NaCl solution and dried over Na_2SO_4. The solvents were removed under reduced pressure, purified by flash chromatography to give the desired product **1**.

2-(2,5-dihydro-1H-pyrrol-1-yl)benzaldehyde (1a)



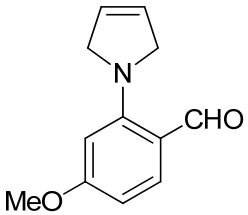
General procedure was performed with 2-flurobenzaldehyde (898 mg, 7.24 mmol), 3-pyrroline (600 mg, 8.69 mmol) and potassium carbonate (1.20 g, 8.69 mmol) in DMF (15 mL). Purified by flash chromatography (petroleum ether / ethyl acetate = 80 / 1) to give the compound **1a** (1.04 g, 83% yield) as yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.14 (s, 1H), 7.74 (d, *J* = 7.8 Hz, 1H), 7.43 (t, *J* = 7.8 Hz, 1H), 6.84-6.82 (m, 2H), 5.96 (s, 2H), 4,24 (s, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.2, 148.8, 134.4, 133.6, 125.7, 122.9, 116.3, 114.9, 58.6; HRMS (ESI): Exact mass calcd. for $C_{11}H_{11}NONa$ $[M+Na]^+$ 196.0738, found 196.0743.

2-(2,5-dihydro-1H-pyrrol-1-yl)-4-methylbenzaldehyde (1b)



General procedure was performed with 2-fluro-4-methylbenzaldehyde (200 mg, 1.45 mmol), 3-pyrroline (100 mg, 1.45 mmol) and potassium carbonate (220 mg, 1.60 mmol) in DMF (5 mL). Purified by flash chromatography (petroleum ether / ethyl acetate = 50 / 1) to give the compound **1b** (106 mg, 39% yield) as yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.62 (d, *J* = 7.8 Hz, 1H), 6.65 (d, *J* = 7.8 Hz, 1H), 6.59 (s, 1H), 5.93 (s, 2H), 4,23 (s, 4H), 2,36 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ1 89.5, 148.9, 145.4, 133.8, 125.7, 121.0, 117.9, 115.2, 58.5, 22.1; HRMS (ESI): Exact mass calcd. for $C_{12}H_{13}NONa$ $[M+Na]^+$ 210.0895, found 210.0890.

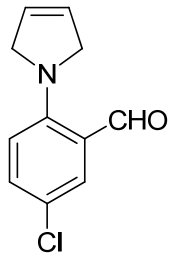
2-(2,5-dihydro-1H-pyrrol-1-yl)-4-methoxybenzaldehyde (1c)



General procedure was performed with 2-fluro-4-methoxybenzaldehyde (433 mg, 2.81 mmol), 3-pyrroline (194 mg, 2.81 mmol), potassium carbonate (427 mg, 3.10 mmol) in DMF (15 mL). Purified by flash chromatography (petroleum ether / ethyl acetate = 40 / 1) to give the compound **1c** (436 mg, 76% yield) as yellow solid. m.p.

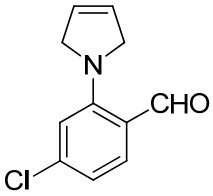
66-68°C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.97 (s, 1H), 7.69 (d, *J* = 8.8 Hz, 1H), 6.41 (dd, *J* = 8.7, 1.8 Hz, 1H), 6.19 (s, 1H), 5.92 (s, 2H), 4.23 (s, 4H), 3.85 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.2, 164.7, 150.6, 136.2, 125.6, 117.6, 103.6, 99.0, 58.5, 55.2; HRMS (ESI): Exact mass calcd. for $C_{12}H_{13}NO_2Na$ $[M+Na]^+$ 226.0844, found 226.0851.

5-chloro-2-(2,5-dihydro-1H-pyrrol-1-yl)benzaldehyde (1d)



General procedure was performed with 2-fluro-5-chlorobenzaldehyde (500 mg, 3.24 mmol), 3-pyrroline (224 mg, 3.24 mmol), potassium carbonate (491 mg, 3.56 mmol) in DMF (15 mL). Purified by flash chromatography (petroleum ether / ethyl acetate = 40 / 1) to give the compound **1d** (403 mg, 60% yield) as yellow solid. m.p. 82-85°C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.10 (s, 1H), 7.69 (d, *J* = 2.6 Hz, 1H), 7.33 (dd, *J* = 9.1, 2.6 Hz, 1H), 6.73 (d, *J* = 9.1 Hz, 1H),5.95 (s, 2H), 4.22 (s, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.4, 146.9, 133.7, 131.3, 125.2, 122.9, 120.9, 116.1, 58.4; HRMS (ESI): Exact mass calcd. for $C_{11}H_{10}NONaCl$ $[M+Na]^+$ 230.0349, found 230.0356.

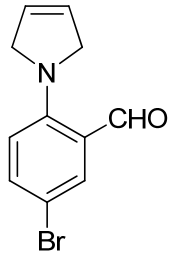
4-chloro-2-(2,5-dihydro-1H-pyrrol-1-yl)benzaldehyde (1e)



General procedure was performed with 2-fluro-4-chlorobenzaldehyde (200 mg, 1.26 mmol), 3-pyrroline (87 mg, 1.26 mmol), potassium carbonate (193 mg, 1.40 mmol) in DMF (5 mL). Purified by flash chromatography (petroleum ether / ethyl acetate = 40 / 1) to give the compound **1e** (196 mg, 75% yield) as yellow solid. m.p. 64-66°C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.08 (s, 1H), 7.65 (d, *J* = 8.8 Hz, 1H), 6.79-6.77 (m, 2H), 5.94 (s, 2H), 4.22 (s, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.0, 149.2, 140.7, 134.8, 125.6, 121.3, 116.6, 114.5, 58.7; HRMS (ESI): Exact mass calcd. for $C_{11}H_{10}NONaCl$

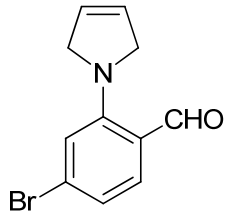
$[M+Na]^+$ 230.0349, found 230.0347.

5-bromo-2-(2,5-dihydro-1H-pyrrol-1-yl)benzaldehyde (1f)



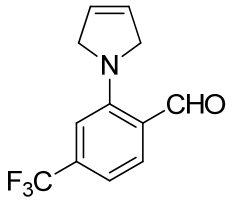
General procedure was performed with 2-fluro-5-bromobenzaldehyde (300 mg, 1.49 mmol), 3-pyrroline (103 mg, 1.49 mmol), potassium carbonate (226 mg, 1.64 mmol) in DMF (10 mL). Purified by flash chromatography (petroleum ether / ethyl acetate = 40 / 1) to give the compound **1f** (245 mg, 66% yield) as yellow solid. m.p. 81-84 ^oC; ^1H NMR (300 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.56 (d, *J* = 8.1 Hz, 1H), 6.92 (d, *J* = 8.2 Hz, 2H), 5.93 (s, 2H), 4.21 (s, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.7, 148.6, 134.3, 129.2, 125.1, 121.2, 119.1, 117.2, 58.2; HRMS (ESI): Exact mass calcd. for $C_{11}H_{10}NONaBr$ $[M+Na]^+$ 273.9843, found 273.9851.

4-bromo-2-(2,5-dihydro-1H-pyrrol-1-yl)benzaldehyde (1g)



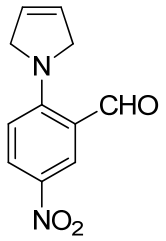
General procedure was performed with 2-fluro-4-bromobenzaldehyde (300 mg, 1.49 mmol), 3-pyrroline (103 mg, 1.49 mmol), potassium carbonate (226 mg, 1.64 mmol) in DMF (10 mL). Purified by flash chromatography (petroleum ether / ethyl acetate = 40 / 1) to give the compound **1g** (250 mg, 67% yield) as yellow solid. m.p. 86-88 ^oC; ^1H NMR (300 MHz, $CDCl_3$) δ 10.07 (s, 1H), 7.81 (d, *J* = 2.3 Hz, 1H), 7.44 (dd, *J* = 9.1, 2.4 Hz, 1H), 6.67 (d, *J* = 9.1 Hz, 1H), 5.94 (s, 2H), 4.20 (s, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.3, 147.2, 136.4, 134.4, 125.1, 123.4, 116.4, 107.6, 58.3; HRMS (ESI): Exact mass calcd. for $C_{11}H_{10}NONaBr$ $[M+Na]^+$ 273.9843, found 273.9853.

2-(2,5-dihydro-1H-pyrrol-1-yl)-4-(trifluoromethyl)benzaldehyde (1h)



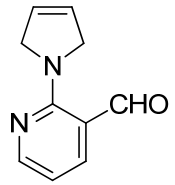
General procedure was performed with 2-fluro-4-(trifluoromethyl)benzaldehyde (200 mg, 1.04 mmol), 3-pyrroline (72 mg, 1.04 mmol), potassium carbonate (158 mg, 1.14 mmol) in DMF (5 mL). Purified by flash chromatography (petroleum ether / ethyl acetate = 50 / 1) to give the compound **1h** (134 mg, 53% yield) as yellow solid. m.p. 68-70 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.19 (s, 1H), 7.80 (d, *J* = 8.4 Hz, 1H), 7.02 (d, *J* = 7.1 Hz, 2H), 5.96 (s, 2H), 4.25 (s, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.4, 148.1, 135.3 (q, *J* = 31.8 Hz), 133.9, 125.5, 125.0, 123.7 (q, *J* = 270.0 Hz), 112.2 (q, *J* = 3.3 Hz), 111.8 (q, *J* = 4.1 Hz), 58.7; HRMS (ESI): Exact mass calcd. for $C_{12}H_{10}F_3NONa$ $[M+Na]^+$ 264.0612, found 264.0.619.

2-(2,5-dihydro-1H-pyrrol-1-yl)-5-nitrobenzaldehyde (1i)



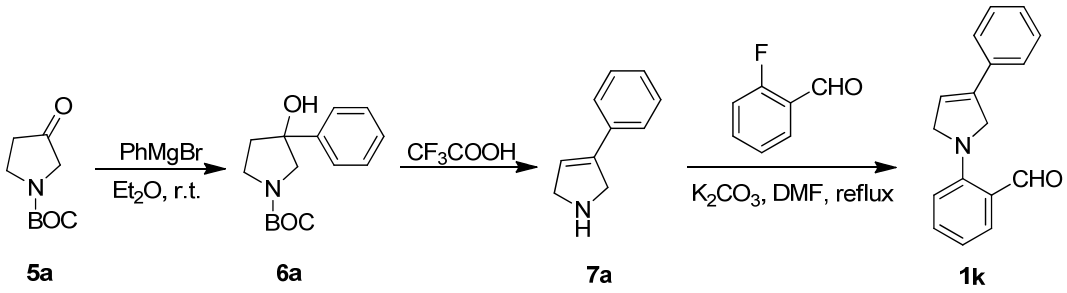
General procedure was performed with 2-fluro-5-nitrobenzaldehyde (200 mg, 1.18 mmol), 3-pyrroline (81 mg, 1.18 mmol), potassium carbonate (179 mg, 1.30 mmol) in DMF (5 mL). Purified by flash chromatography (petroleum ether / ethyl acetate = 10 / 1) to give the compound **1i** (250 mg, 97% yield) as yellow solid. m.p. 176-178 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.06 (s, 1H), 8.63 (d, *J* = 2.7 Hz, 1H), 8.22 (dd, *J* = 9.5, 2.7 Hz, 1H), 6.76 (d, *J* = 2.7 Hz, 1H), 5.98 (s, 2H), 4.31 (s, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.9, 151.4, 136.3, 130.0, 128.6, 125.7, 121.2, 116.4, 59.3; HRMS (ESI): Exact mass calcd. for $C_{11}H_{10}N_2O_3NaBr$ $[M+Na]^+$ 241.0589, found 241.0595.

2-(2,5-dihydro-1H-pyrrol-1-yl)nicotinaldehyde (1j)



General procedure was performed with 2-chloronicotinaldehyde (443 mg, 3.14 mmol), 3-pyrroline (217 mg, 3.14 mmol), potassium carbonate (476 mg, 3.45 mmol) in DMF (15 mL). Purified by flash chromatography (petroleum ether / ethyl acetate = 40 / 1) to give the compound **1j** (333 mg, 61% yield) as yellow solid. m.p. 36-38 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.05 (s, 1H), 8.34 (d, *J* = 2.9 Hz, 1H), 7.96 (d, *J* = 6.6 Hz, 1H), 6.73 (dd, *J* = 7.4, 4.6 Hz, 1H), 5.92 (s, 2H), 4.39 (s, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.3, 156.0, 152.8, 142.0, 125.5, 116.5, 112.1, 56.9; HRMS (ESI): Exact mass calcd. for $C_{10}H_{11}N_2O$ $[M+H]^+$ 175.0871, found 175.0867.

2-(3-phenyl-2,5-dihydro-1H-pyrrol-1-yl)benzaldehyde (1k)



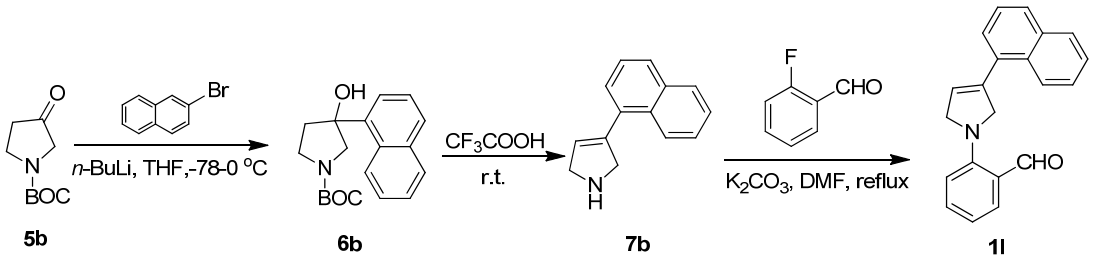
5a **6a** **7a** **1k**

To a solution of **5a** (500 mg, 2.70 mmol) in anhydrous ether (10 mL) was added PhMgBr (3.3 mL, 3.24 mmol, 1.0 M in THF), the mixture was stirred at room temperature until complete consumption, then purified by flash chromatography (petroleum ether / ethyl acetate = 10 / 1) to give the compound **6a** as white solid (340 mg, 48% yield).[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49-7.26 (m, 5H), 3.83-3.51 (m, 4H), 2.43-1.93 (m, 3H), 1.46 (s, 9H).

6a (183 mg, 1.03 mmol) was added to CF_3COOH (0.3 mL), the mixture was stirred at room temperature overnight, then concentrated in vacua to get the crude product **7a**, the crude product **7a** was dissolved in DMF (5 mL), 2-flurobenzaldehyde (128 mg, 1.03 mmol) and potassium carbonate (284 mg, 2.06 mmol) was added to the solution. The mixture was stirred at reflux until complete consumption monitored by TLC. The mixture was cooled to room temperature, added into water (15 mL), then extracted with EtOAc (3×10 mL), the organic layer was concentrated, purified by flash chromatography (petroleum ether / ethyl acetate = 100/1) to get the desired compound **1k** (92 mg, 39% yield over two steps) as yellow solid. m.p. 118-122 ^{o}C; ^{1}H NMR

(300 MHz, $CDCl_3$) δ 10.18 (s, 1H), 7.76 (d, *J* = 7.8 Hz, 1H), 7.53-7.29 (m, 6H), 6.92-6.86 (m, 1H), 6.29 (d, *J* = 1.7 Hz, 1H), 4.59 (s, 2H), 4.43 (s, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 190.2, 148.7, 137.6, 134.4, 133.1, 128.6, 128.1, 125.6, 123.0, 119.4, 116.6, 114.8, 59.6, 58.9; HRMS (ESI): Exact mass calcd. for $C_{17}H_{15}NONa$ $[M+Na]^+$ 272.1051, found 272.1055.

2-(3-(naphthalen-1-yl)-2,5-dihydro-1H-pyrrol-1-yl)benzaldehyde (1l)



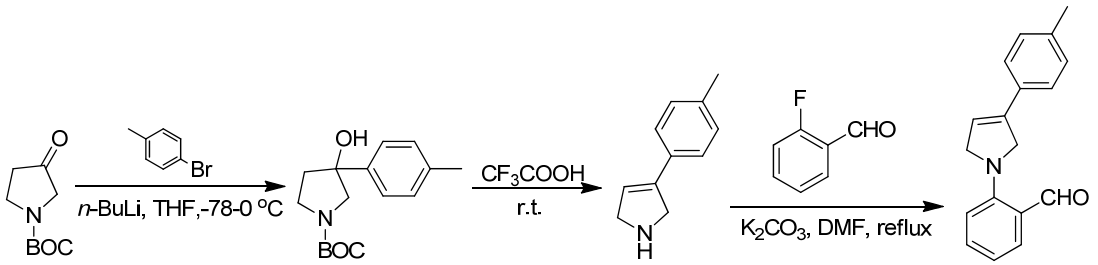
5b **6b** **7b** **1l**

To a solution of bromonaphthalene (496 mg, 2.4 mmol) in anhydrous THF (10 mL) was added *n*-BuLi (0.48 mL, 2.4 mmol, 2.5 M in THF) at -78 °C, the mixture was stirred for 1 h, then was added **5b** (270 mg, 2.0 mmol) in THF (5 mL), the mixture was allowed to warm slowly to room temperature until complete consumption. The reaction was added ice water, then extracted with EtOAc (3×10 mL), the organic layer was concentrated, purified by flash chromatography (petroleum ether / ethyl acetate = 10 / 1) to get the desired compound **6b** (240 mg, 55% yield) as white solid. [2] ^1H NMR (300 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.85-7.83 (m, 3H), 7.60-7.43 (m, 3H), 3.76-3.69 (m, 4H), 2.66 (br s, 1H), 2.48-2.15 (m, 2H), 1.49 (s, 9H).

6b (240 mg, 1.0 mmol) was added to CF_3COOH (1.22 g, 10 mmol) in CH_2Cl_2 (3 mL), the mixture was stirred at room temperature overnight, then concentrated in vacua to get the crude product **7b**, the crude product **7b** was dissolved in DMF (2 mL), then 2-flurobenzaldehyde (149 mg, 1.2 mmol) and potassium carbonate (345 mg, 2.5 mmol)was added to the solution, the mixture was stirred at reflux until complete consumption monitored by TLC. The mixture was cooled to room temperature, added into water (10 mL), then extracted with EtOAc (3×5 mL), the organic layer was concentrated, purified by flash chromatography (petroleum ether / ethyl acetate = 100 / 1) to get the desired compound **1l** (60 mg, 22% yield over two steps) as yellow solid. m.p. 114-116 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 10.23 (s, 1H), 7.83-7.64 (m, 7H),

7.52-7.40 (m, 4H), 6.39 (s, 1H), 4.69 (s, 2H), 4.45 (s, 2H); ^{13}C NMR (75 MHz, d_6-DMSO) δ 191.6, 150.1, 139.0, 135.9, 135.7, 135.1, 134.8, 134.4, 131.8, 129.7, 129.5, 129.1, 127.9, 127.7, 125.8, 124.8, 121.6, 118.1, 116.3, 61.0, 60.4; HRMS (ESI): Exact mass calcd. for $C_{21}H_{17}NONa$ $[M+Na]^+$ 322.1208, found 322.1202.

2-(3-phenyl-2,5-dihydro-1H-pyrrol-1-yl)benzaldehyde (1m)



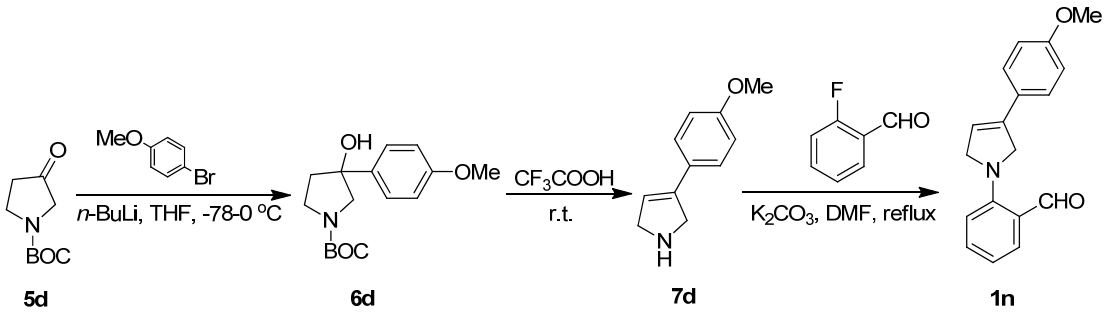
5c **6c** **7c** **1m**

To a solution of 4-methylbromobenzene (1.03 g, 6.0 mmol) in anhydrous THF (10 mL) was added *n*-BuLi (2.4 mL, 6.0 mmol, 2.5M in THF) at -78 °C, the mixture was stirred for 1 h, then was added **5c** (925 mg, 5.0 mmol) in THF (5 mL), the mixture was allowed to warm slowly to room temperature until complete consumption. The reaction was added ice water, then extracted with EtOAc (3×10 mL), the organic layer was concentrated, purified by flash chromatography (petroleum ether / ethyl acetate = 10 / 1) to get the desired compound **6c** (740 mg, 53% yield) as white solid. ^{1}H NMR (300 MHz, d_6-DMSO) δ 7.36 (d, *J* = 7.6 Hz, 2H), 7.18 (d, *J* = 7.9 Hz, 2H), 4.00-3.31 (m, 4H), 2.35 (s, 3H), 2.31-2.01 (m, 2H), 1.46 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ154.6, 140.0, 137.2, 129.0, 125.1, 80.3, 79.3, 59.5, 58.7, 45.0, 44.6, 39.5, 38.7, 28.4, 20.9.

6c (277 mg, 1.0 mmol) was added to CF_3COOH (1.14 g, 10 mmol) in CH_2Cl_2 (3 mL), the mixture was stirred at room temperature overnight, then concentrated in vacua to get the crude product **7c**, the crude product **7c** was dissolved in DMF (2 mL), then 2-flurobenzaldehyde (149 mg, 1.2 mmol) and potassium carbonate (345 mg, 2.5 mmol) was added to the solution, the mixture was stirred at reflux until complete consumption monitored by TLC. The mixture was cooled to room temperature, added to water (10 mL), then extracted with EtOAc (3×5 mL), the organic layer was concentrated, purified by flash chromatography (petroleum ether / ethyl acetate = 100

/ 1) to get the desired compound **7c** (65 mg, 25% yield over two steps) as yellow solid. m.p. 116-117 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.19 (s, 1H), 7.75 (d, *J* = 7.8 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 1H), 7.34 (d, *J* = 7.9 Hz, 2H), 7.18 (d, *J* = 7.9 Hz, 2H), 6.95-6.79 (m, 2H), 6.21 (s, 1H), 4.57 (s, 2H), 4.41 (s, 2H), 2.37 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ190.2, 148.7, 138.0, 137.4, 134.4, 133.6, 130.3, 129.3, 125.4, 122.9, 118.4, 116.5, 114.8, 59.6, 59.0, 21.2; HRMS (ESI): Exact masscalcd. for $C_{18}H_{17}NONa$ $[M+Na]^+$ 286.1208, found 286.1213.

2-(3-(4-methoxyphenyl)-2,5-dihydro-1H-pyrrol-1-yl)benzaldehyde (1n)

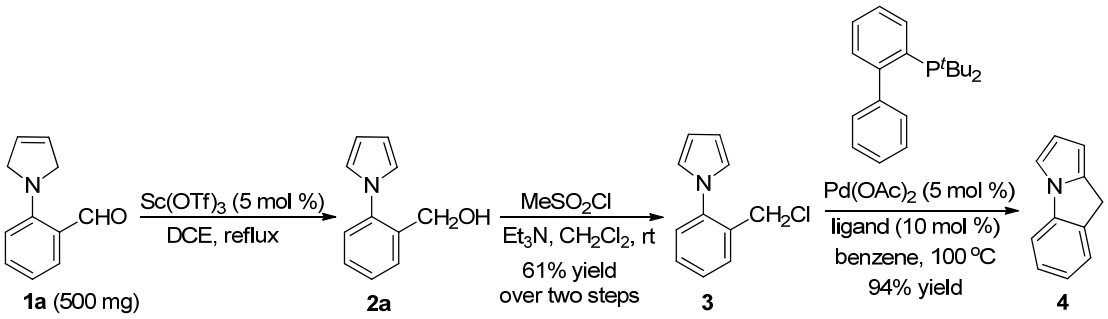


To a solution of 4-methoxybromobenzene (1.12 g, 6.0 mmol) in anhydrous THF (10 mL) was added *n*-BuLi (2.5 mL, 6.0 mmol, 2.5 M in THF) at -78^{o}C, the mixture was stirred for 1 h, then was added **5d** (925 mg, 5.0 mmol) in THF (5 mL), the mixture was allowed to warm slowly to room temperature until complete consumption. The reaction was added ice water, then extracted with EtOAc (3×10 mL), the organic layer was concentrated, purified by flash chromatography (petroleum ether / ethyl acetate = 10 / 1) to get the desired compound **6d** (800 mg, 55% yield) as white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.3 Hz, 1H), 6.92 (d, *J* = 8.6 Hz, 2H), 3.83 (s, 3H), 3.82-3.39 (m, 4H), 2.47-2.09 (m, 2H), 1.49 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ159.0, 154.7, 135.1, 126.4, 113.7, 80.1, 79.3, 59.4, 58.6, 45.0, 44.5, 39.4, 38.6, 28.4.

6d (290 mg, 1.0 mmol) was added to CF_3COOH (1.22 g, 10 mmol) in CH_2Cl_2 (3 mL), the mixture was stirred at room temperature overnight, then concentrated in vacua to get the crude product **7d**, the crude product**7d** was dissolved in DMF (2 mL), then 2-flurobenzaldehyde (149 mg, 1.2 mmol) and potassium carbonate (345 mg, 2.5 mmol) was added to the solution, the mixture was stirred at reflux until complete

consumption monitored by TLC. The mixture was cooled to room temperature, added to water (10 mL), then extracted with EtOAc (3×5 mL), the organic layer was concentrated, purified by flash chromatography (petroleum ether / ethyl acetate = 100 / 1) to get the desired compound **1n** (60 mg, 22% yield over two steps) as yellow solid; m.p. 115-116 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.19 (s, 1H), 7.75 (d, *J* = 7.7 Hz, 1H), 7.52-7.30 (m, 3H), 6.87 (dd, *J* = 18.0, 8.4 Hz, 4H), 6.12 (s, 1H), 4.54 (s, 2H), 4.39 (s, 2H), 3.82 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.2, 159.5 148.7, 137.0, 134.4, 133.6, 126.7, 125.9, 122.9, 117.2, 116.5, 114.8, 114.0, 59.6, 59.1, 55.3; HRMS (ESI): Exact mass calcd. for $C_{18}H_{17}NO_2Na$ $[M+Na]^+$ 302.1157, found 302.1149.

General procedure for synthesis of fluorazene 4.



1a (500 mg, 2.89 mmol) was dissolved in DCE (50 mL), then added the catalyst $Sc(OTf)_3$. The mixture was stirred at reflux for 15 min, concentrated in vacuo, added water (30 mL), then extracted with EtOAc (3 × 10 mL), the organic layers were washed with saturated NaCl solution, dried over Na_2SO_4, concentrated to get the crude product **2a**. The crude product **2a** was dissolved in CH_2Cl_2 (12 mL), Et_3N (0.6 mL, 3.47 mmol) was added, then $MeSO_2Cl$ (0.27 mL, 4.34 mmol) was added dropwise to the mixture at 0 ^{o}C, then stirred at room temperature until complete consumption. The mixture was concentrated, added water (30 mL), then extracted with EtOAc (3 × 10 mL), the organic layers were washed with saturated NaCl solution, dried over Na_2SO_4, then purified by flash chromatography (petroleum ether) to get the product **3** (340 mg, 61% yield over steps) as clear oil.[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60 (dd, *J* = 5.5, 3.6 Hz, 1H), 7.42 (dd, *J* = 5.8, 3.5 Hz, 2H), 7.33 (dd, *J* = 5.5, 3.6 Hz, 1H), 7.28 (s, 1H), 6.94 (s, 2H), 6.38 (s, 2H), 4.48 (s, 2H).

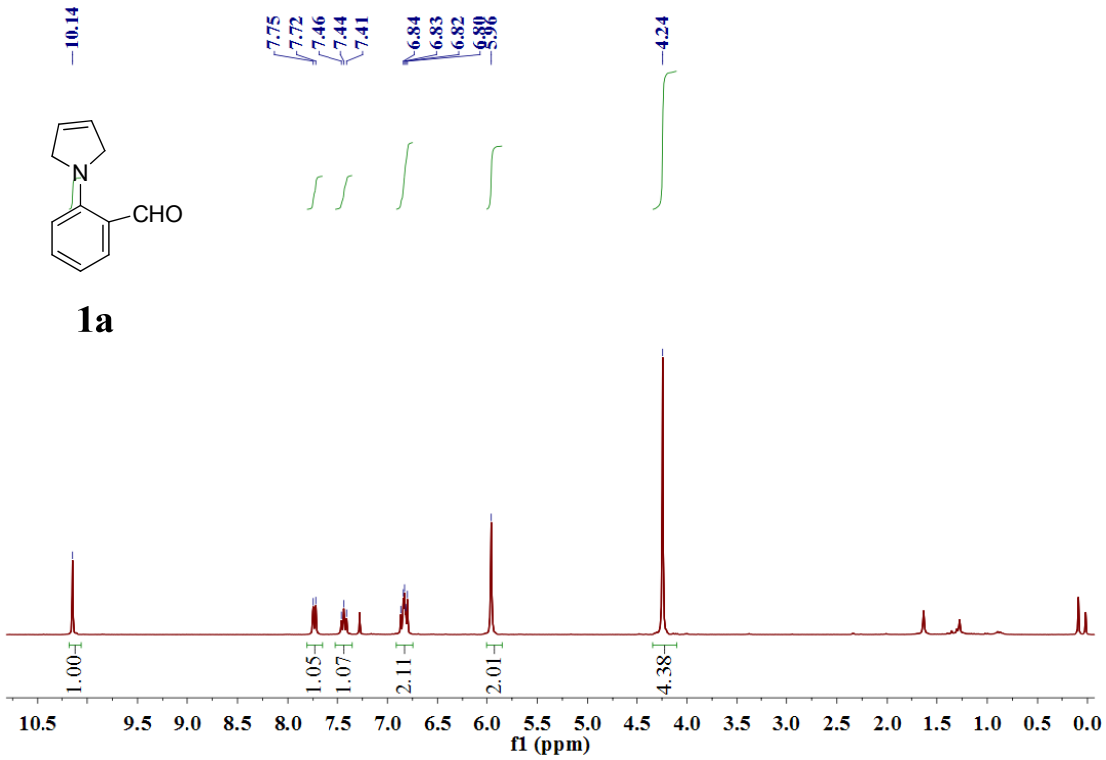
3 (340 mg, 1.76 mmol), $Pd(OAc)_2$ (20 mg, 0.088mmol), ligand (53 mg, 0.176 mmol) was added in benzene, the mixture was stirred under Ar at 100 ^oC for 30 min, then added EtOAc (20 mL), washed with water, saturated NaCl solution, concentrated, purified by flash chromatography (petroleum ether) to get the desired product **4** (256 mg, 94% yield).[3] ^1H NMR (300 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.4 Hz, 1H), 7.38-7.25 (m, 2H), 7.14 (m, 2H), 6.45 (t, *J* = 2.8 Hz, 1H), 6.17 (s, 1H).

References:

[1] K. C. Nicolaou, A. Krasovskiy, U. Majumder, V. É. Trépanier and D. Y.-K. Chen, *J. Am. Chem. Soc.*, 2009, **131**, 3690.

[2] G. Wu and J.-D. Chen, CN 102766080 A, 2012.

[3] S. J. Hwang, S. H. Cho and S. Chang, *J. Am. Chem. Soc.*, 2008, **130**, 16158.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2.11
2.01
4.38
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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2.5
2.0
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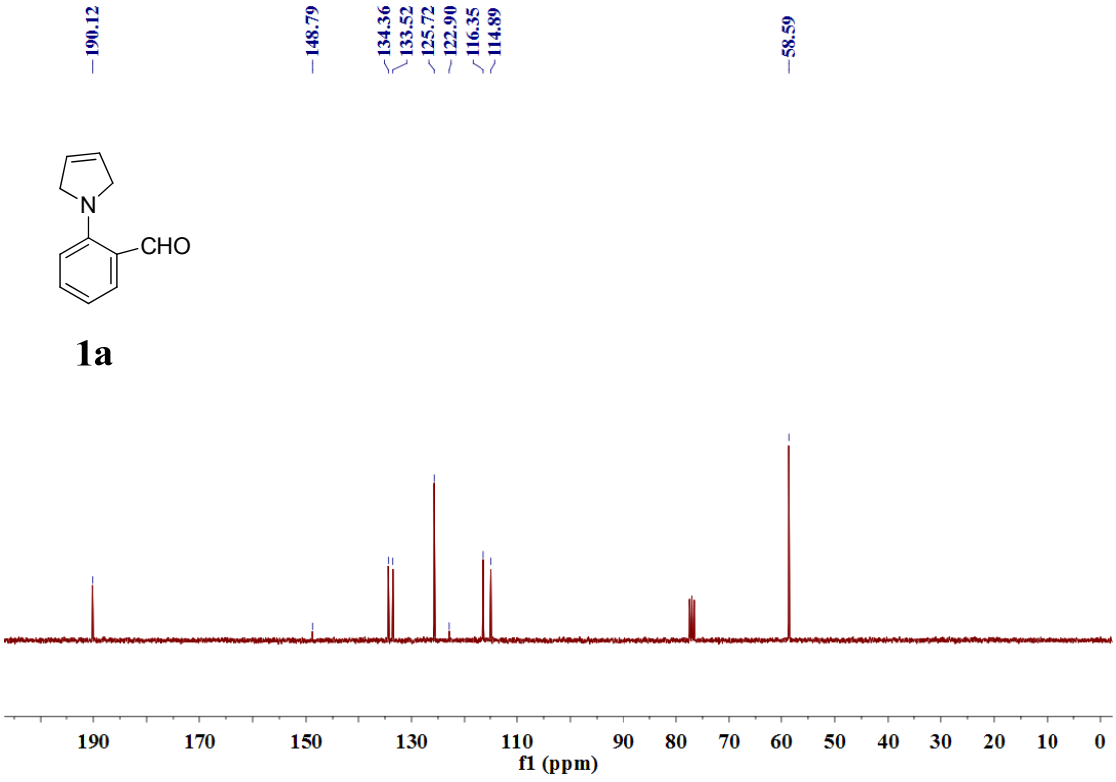
0.0
f1 (ppm)

1a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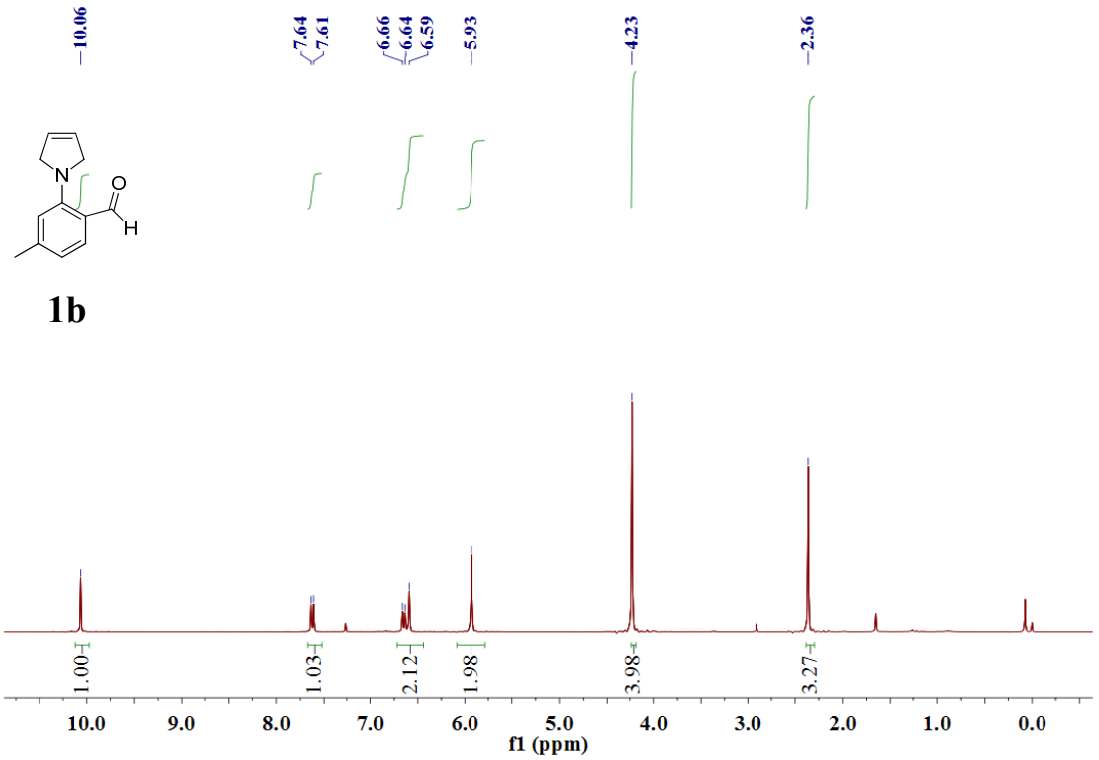
0
f1 (ppm)

1a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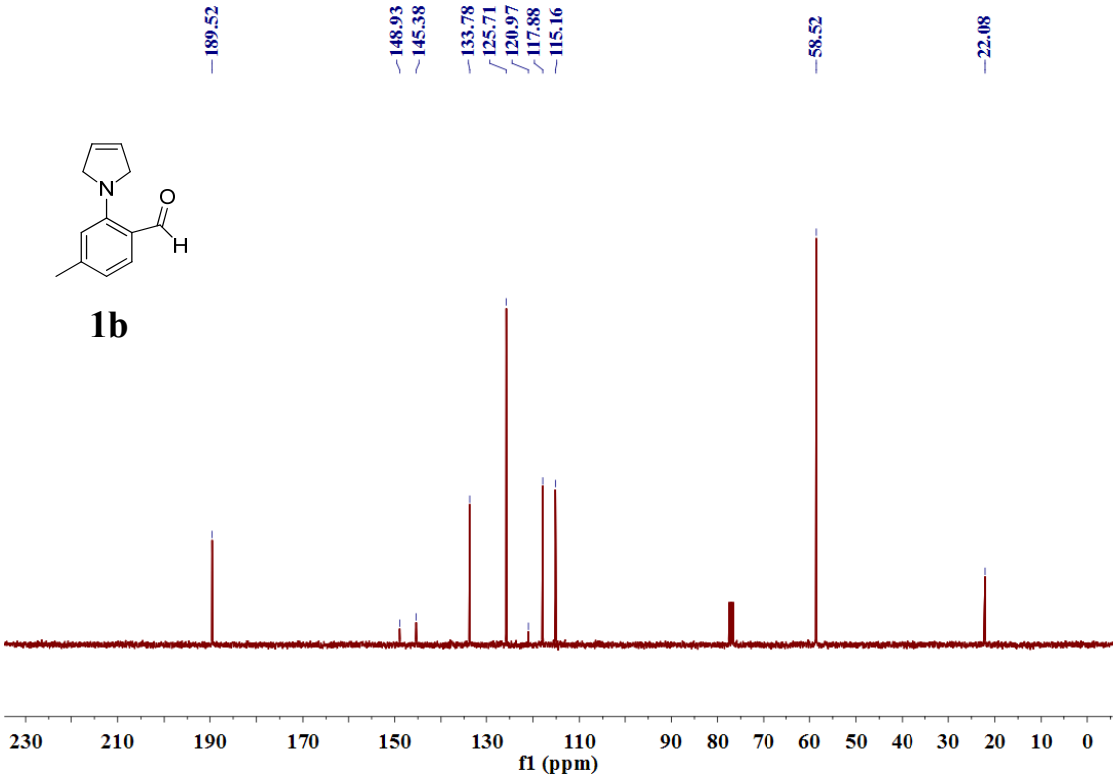
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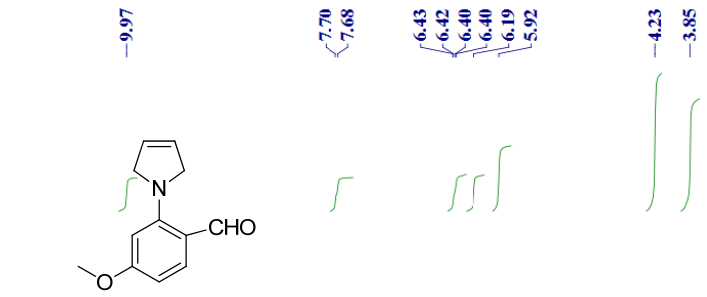
1b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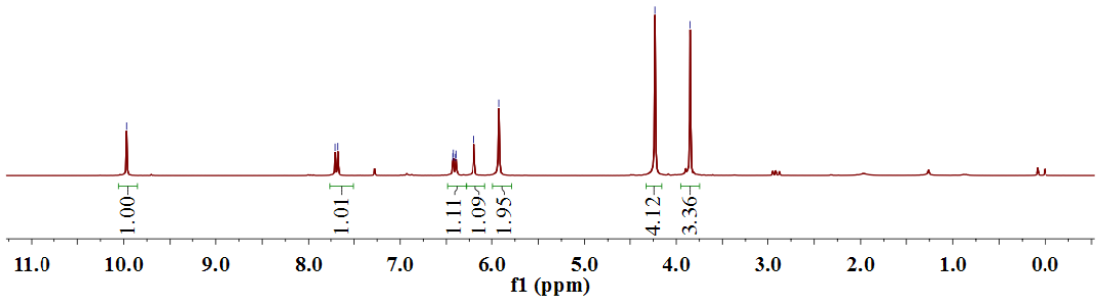
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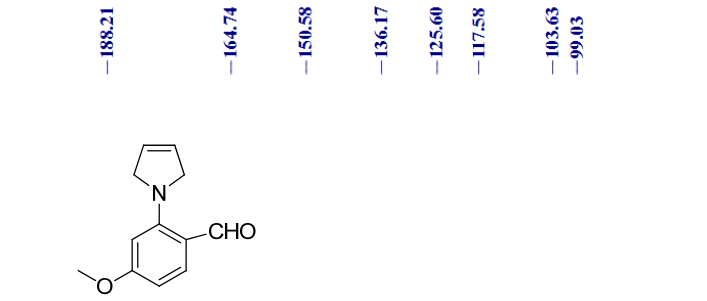
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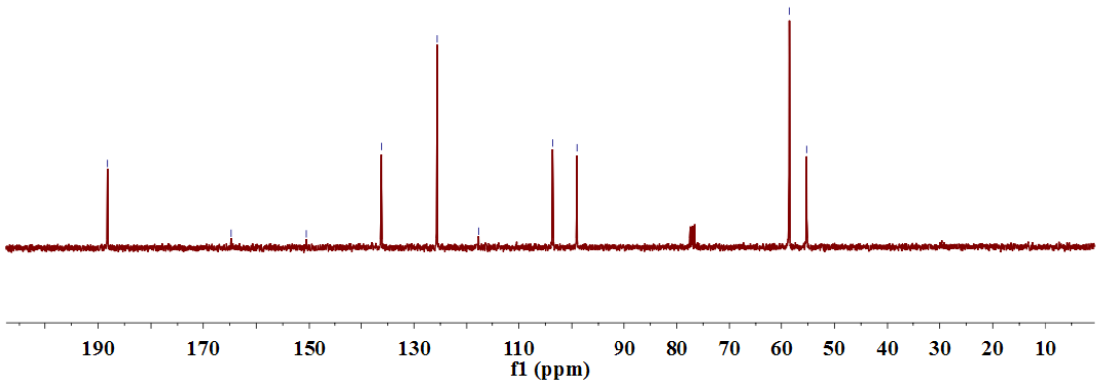
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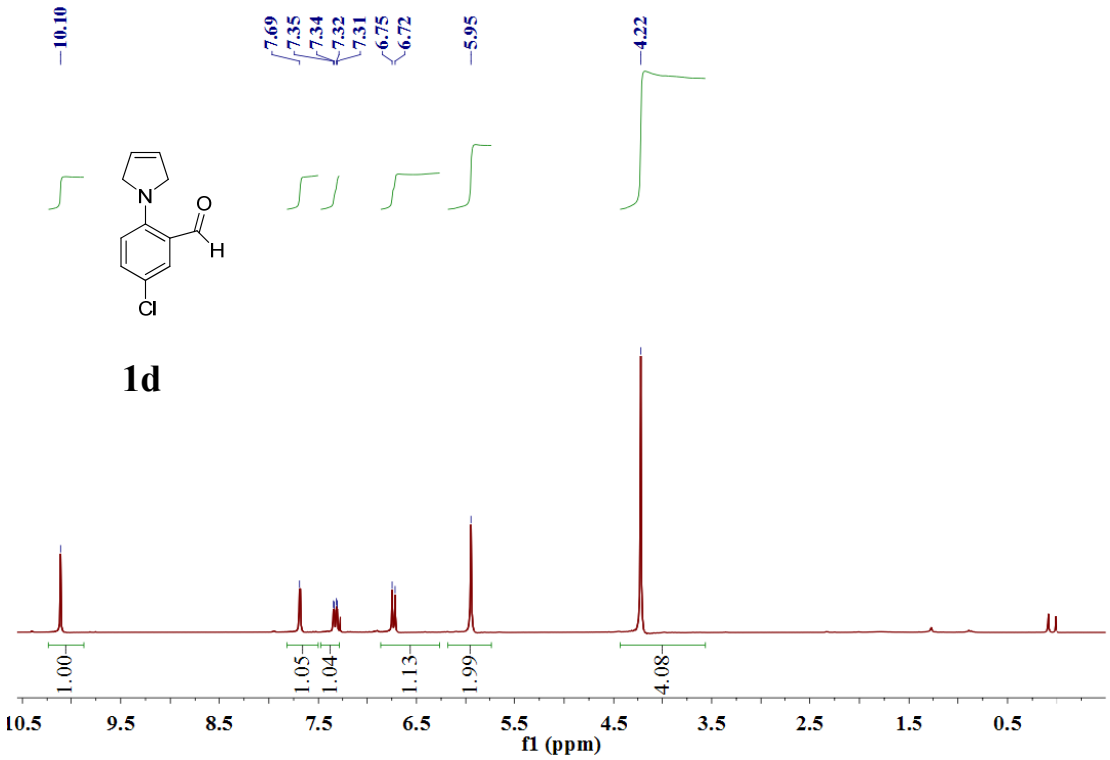
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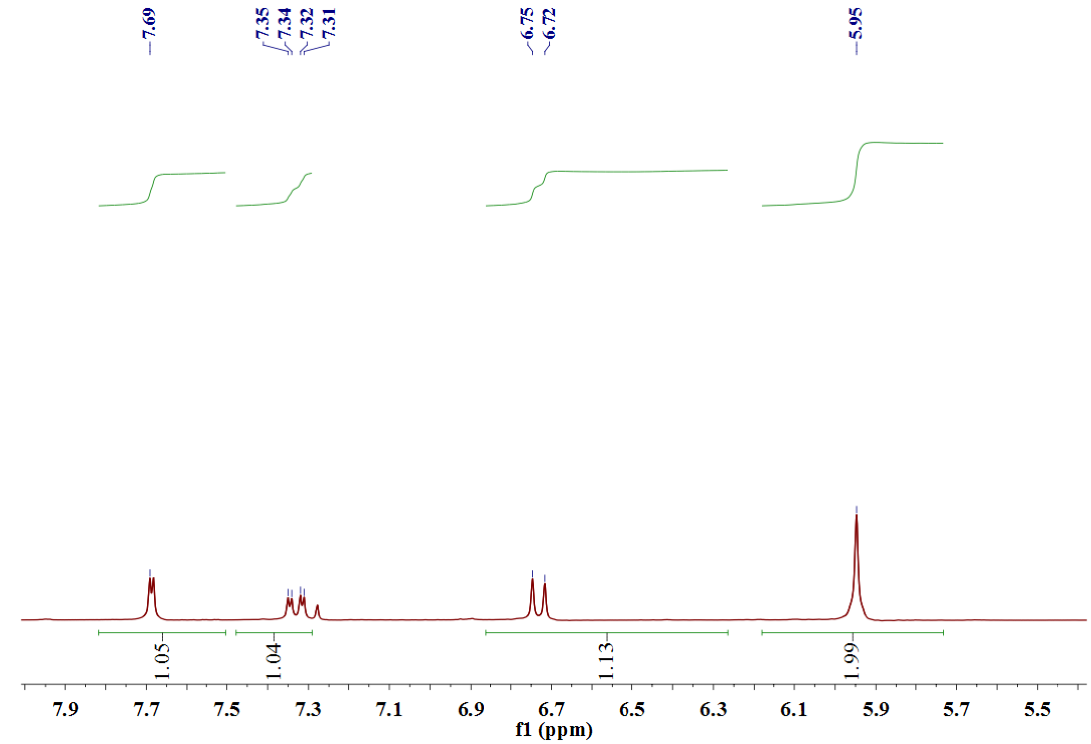
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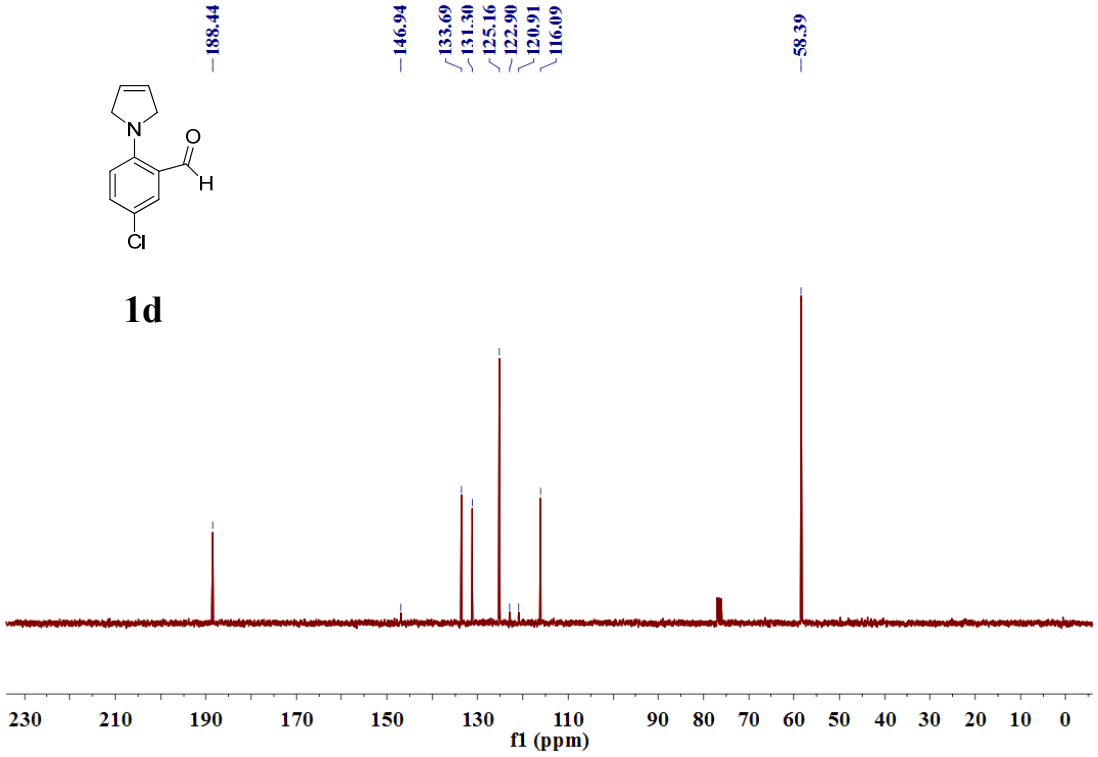
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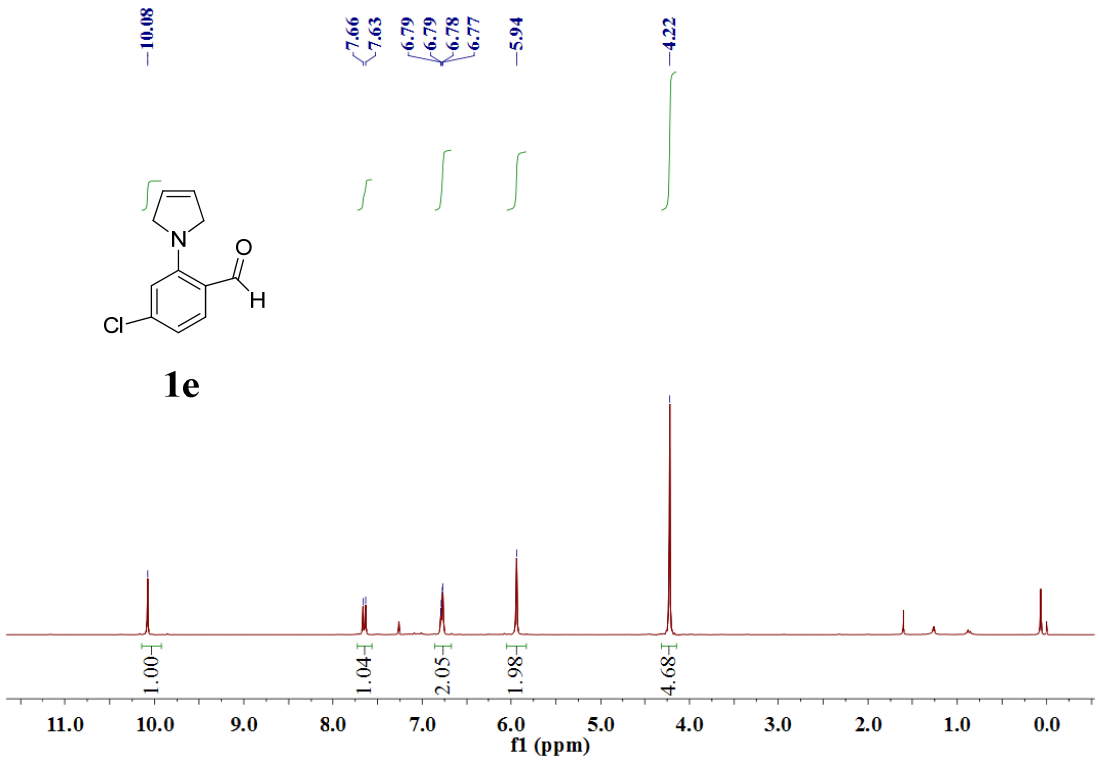
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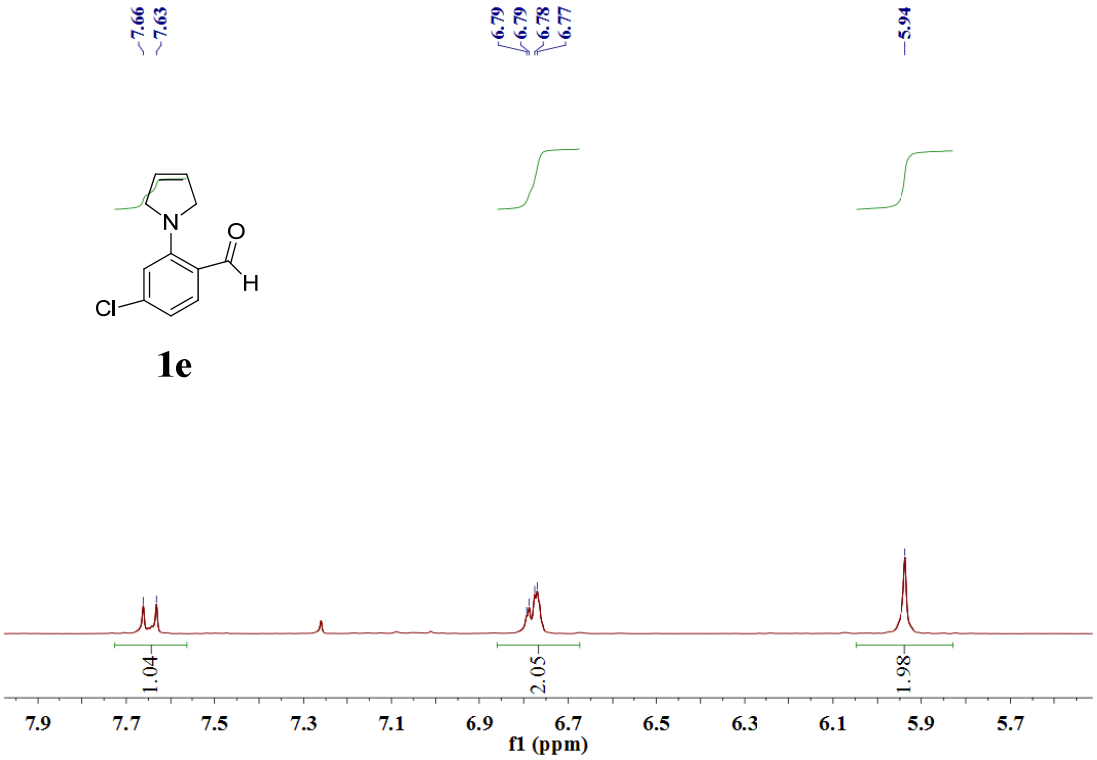
1e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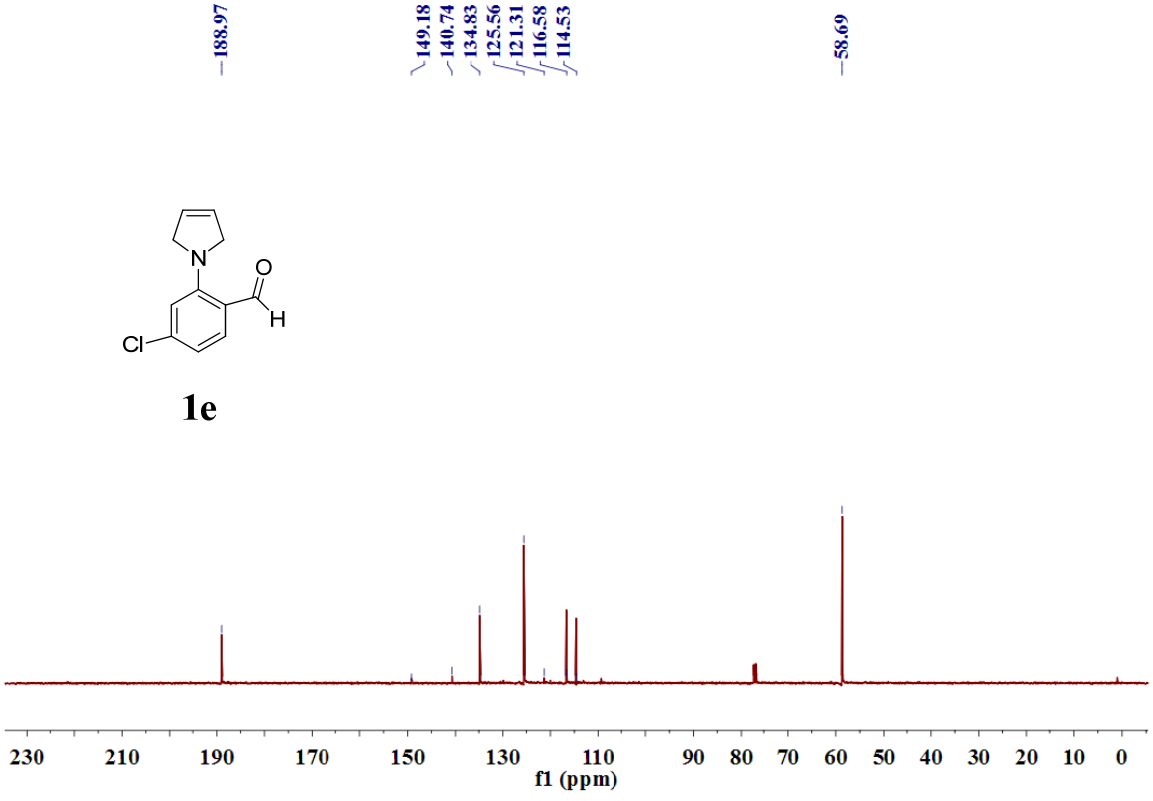
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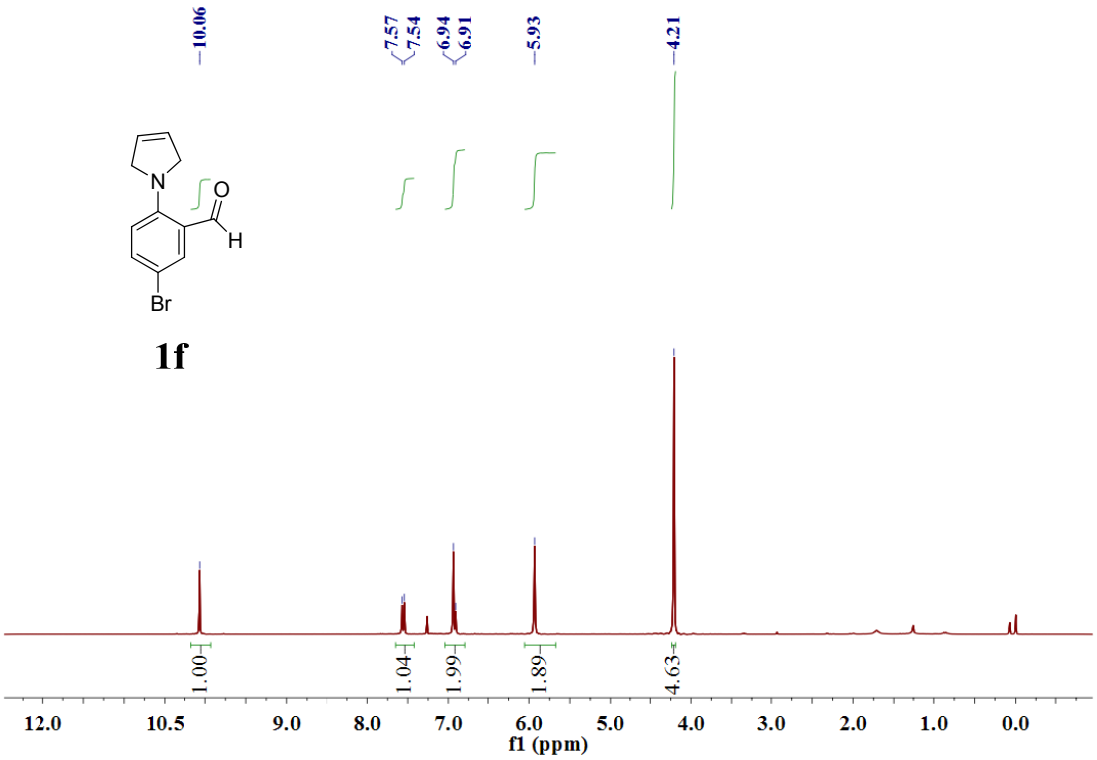
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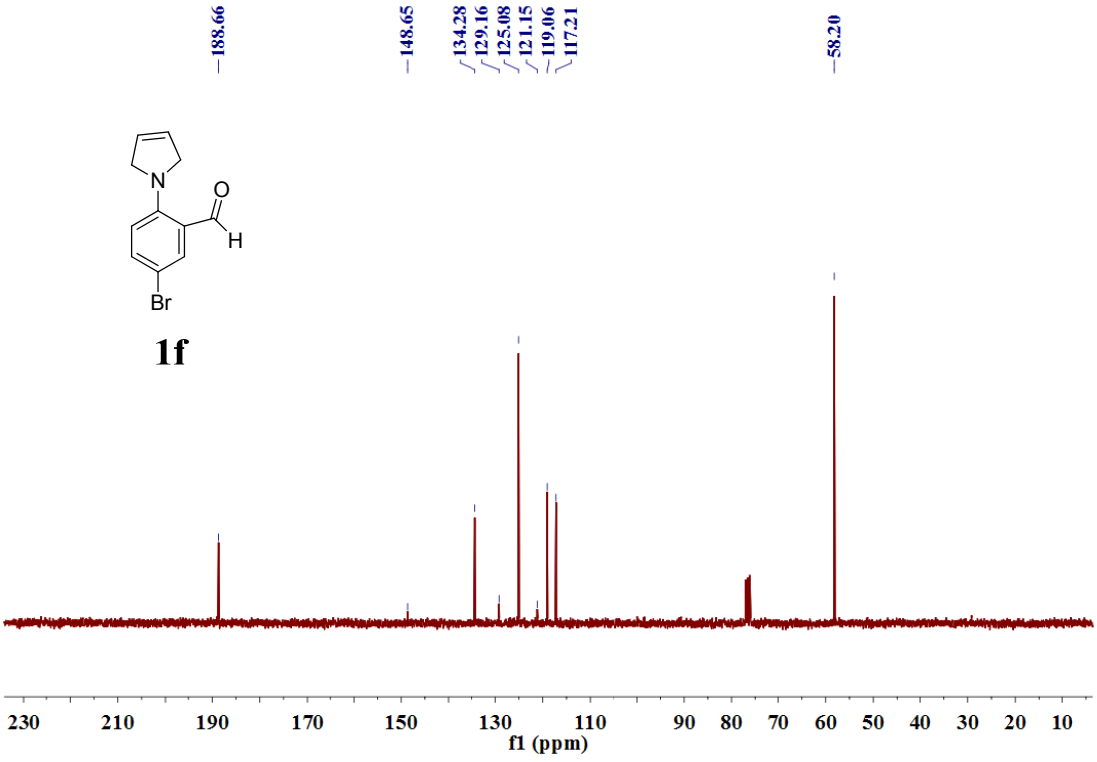
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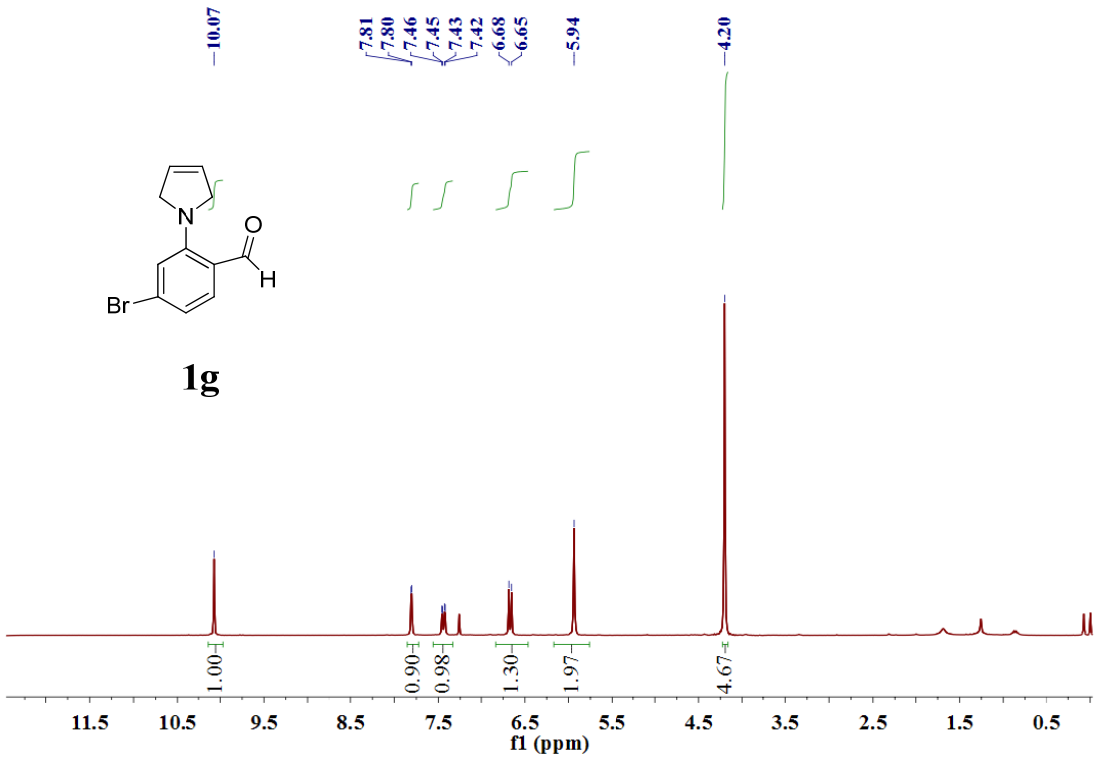
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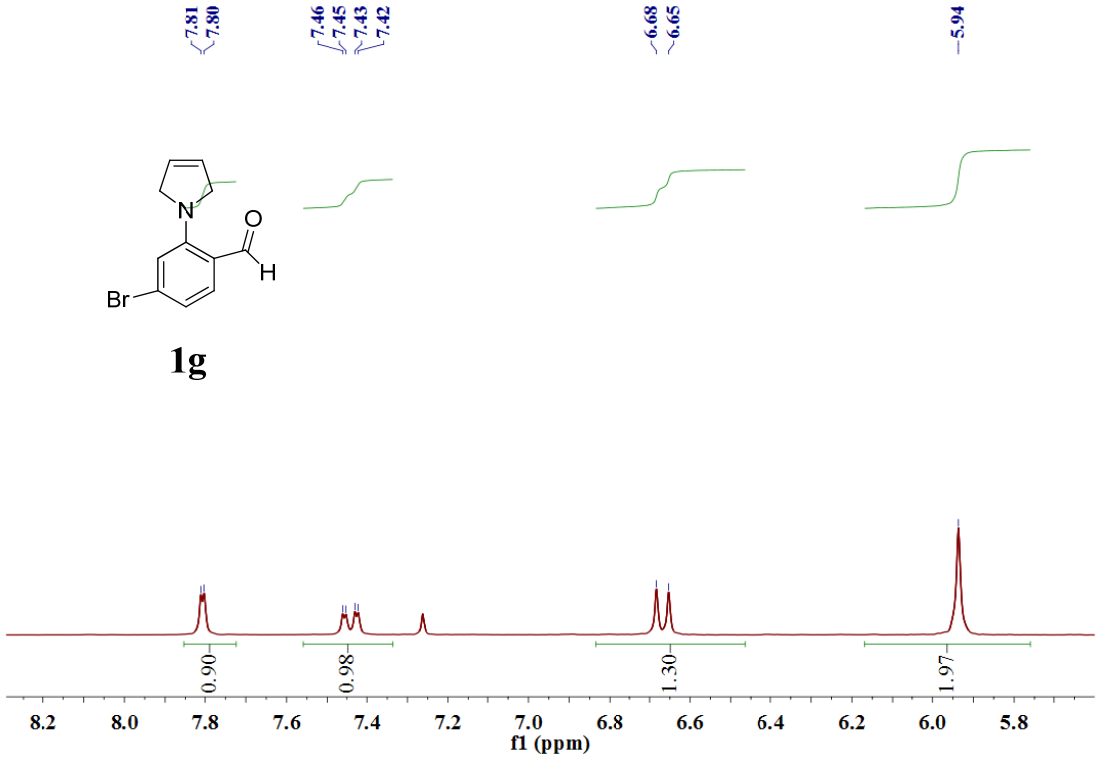
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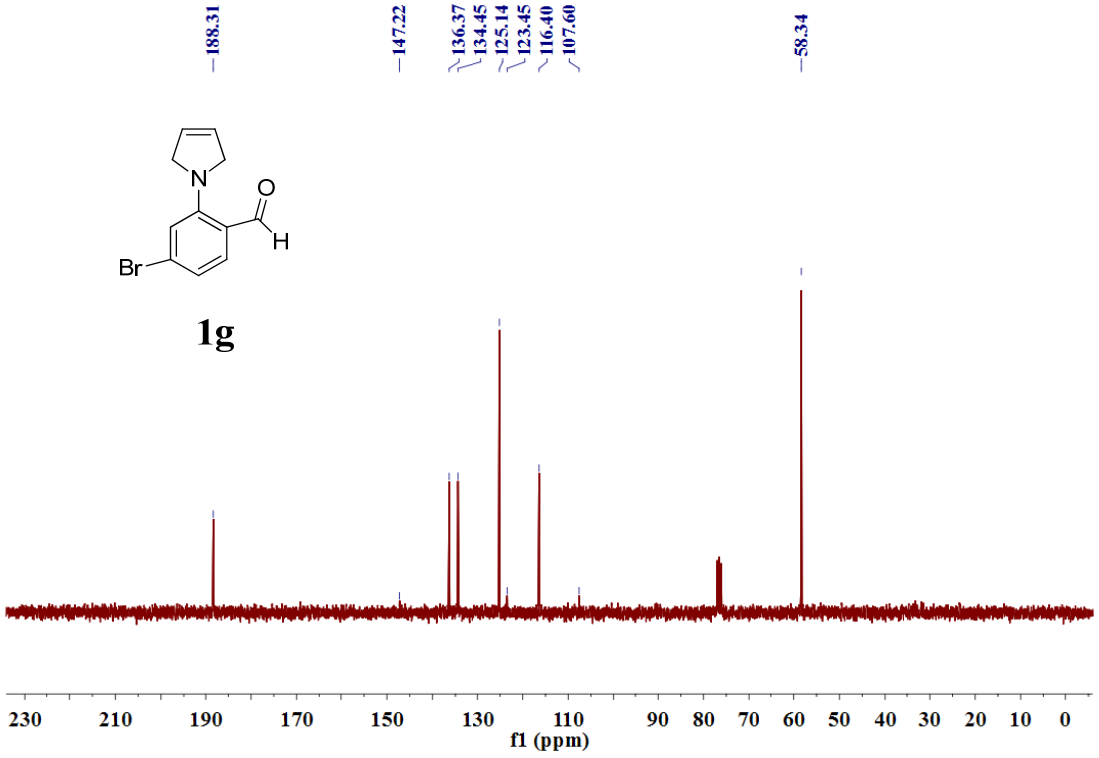
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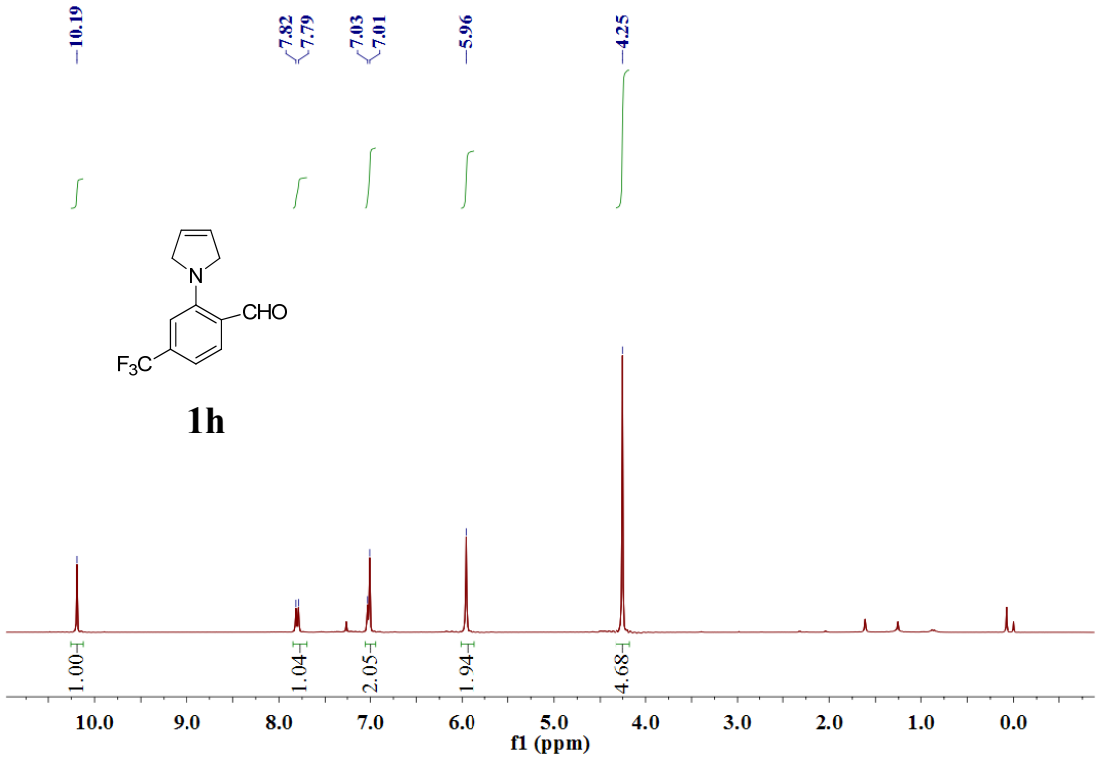
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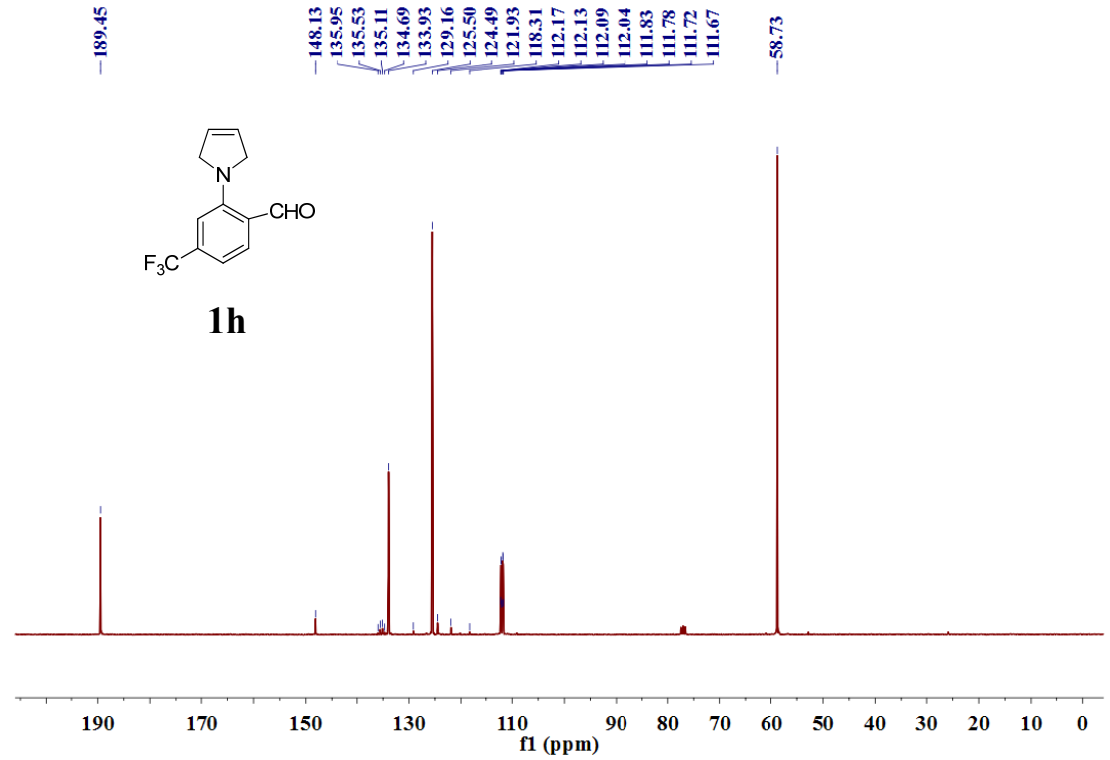
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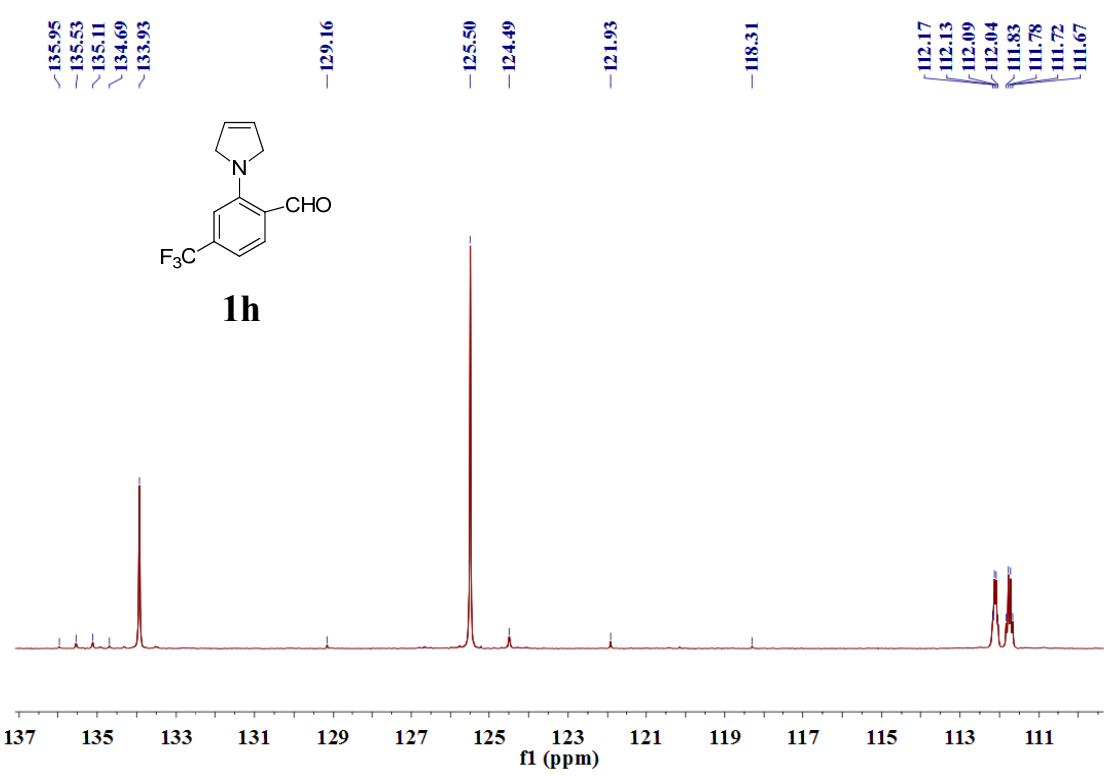
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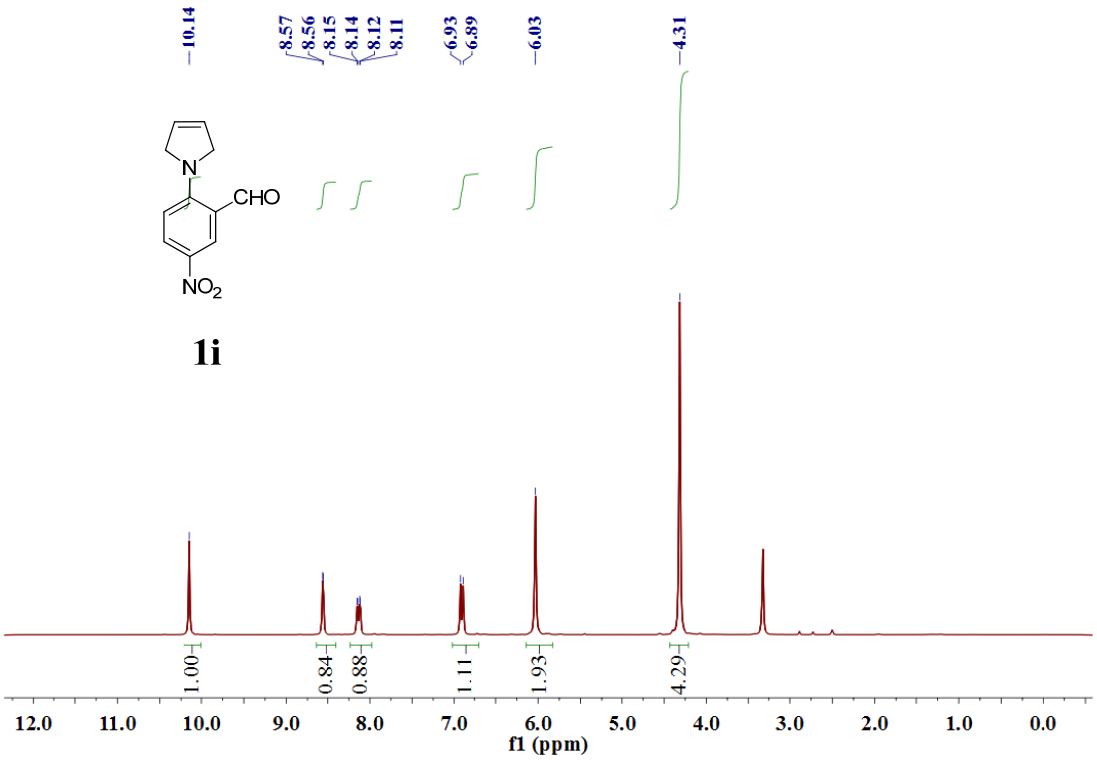
f1 (ppm)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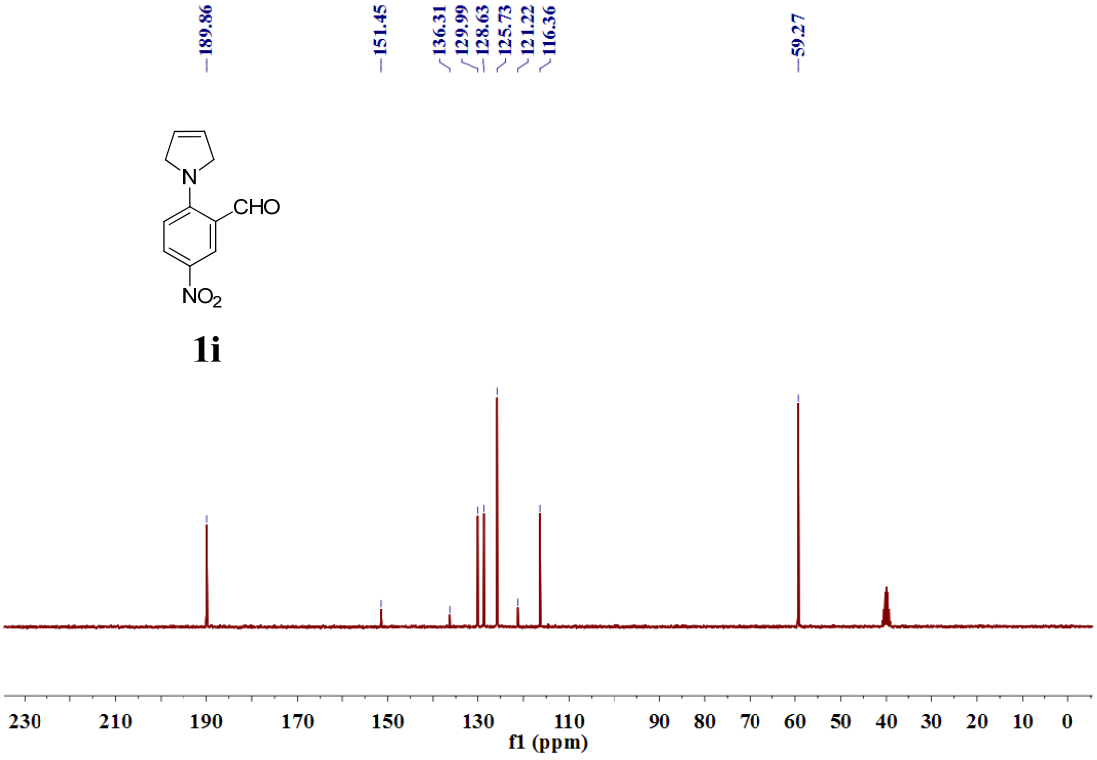
f1 (ppm)

1i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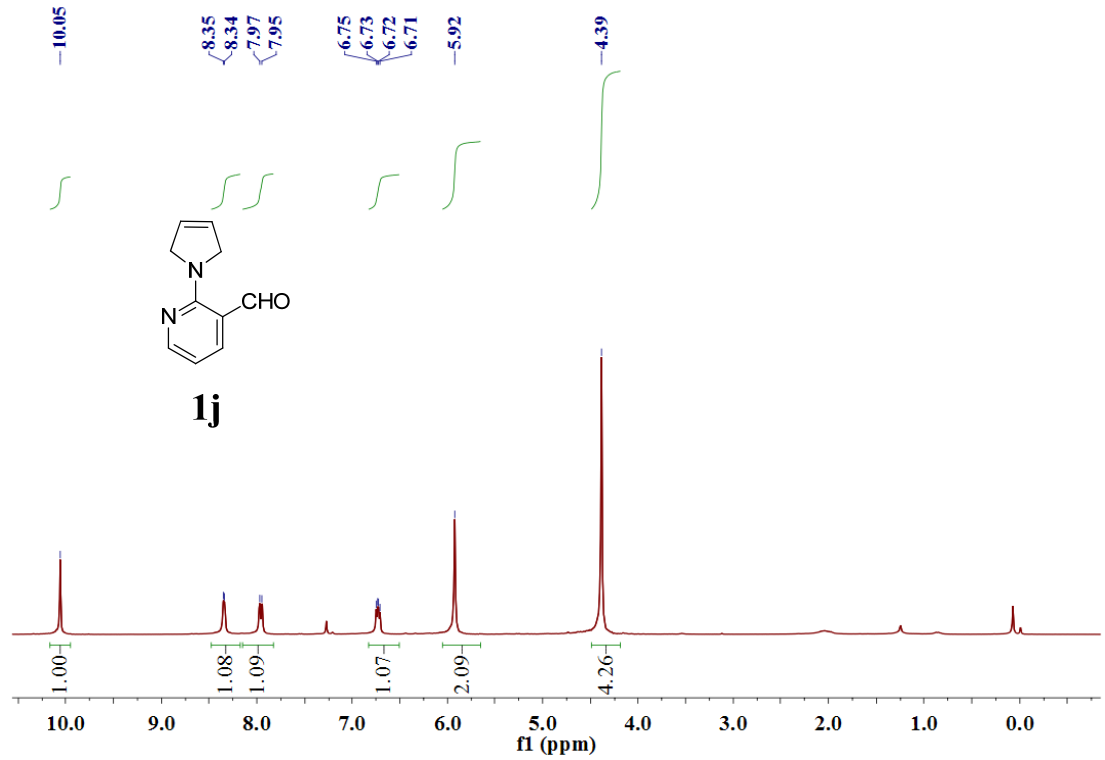
1i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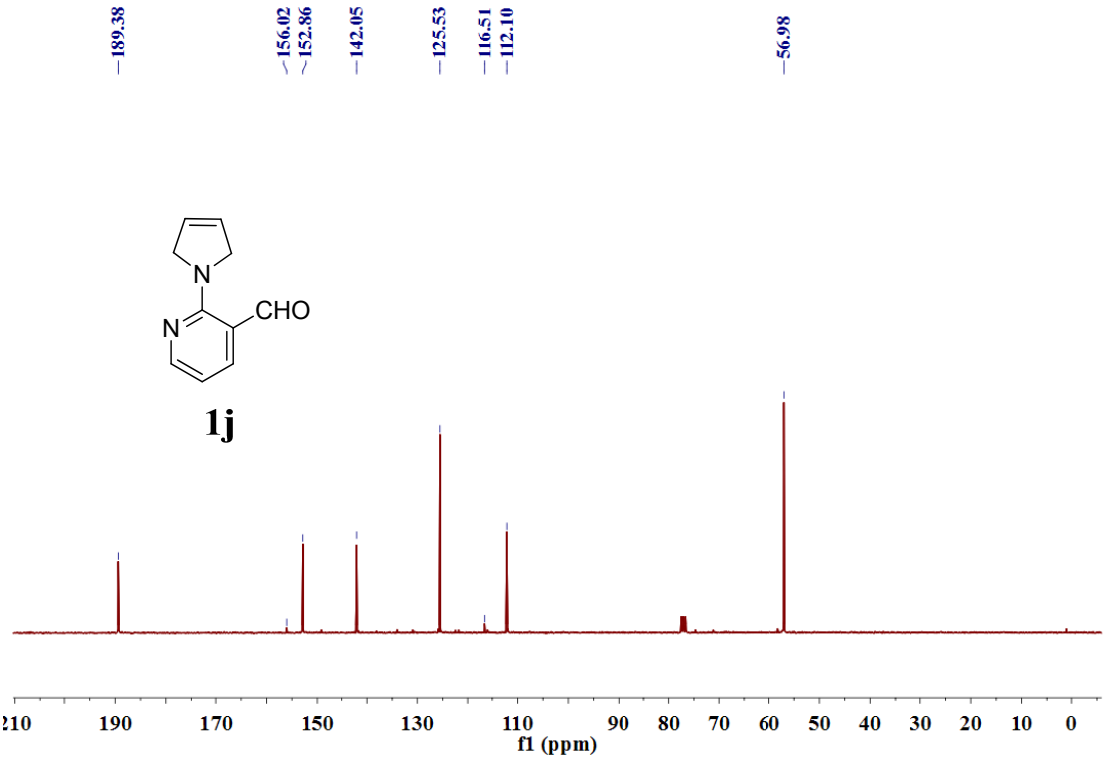
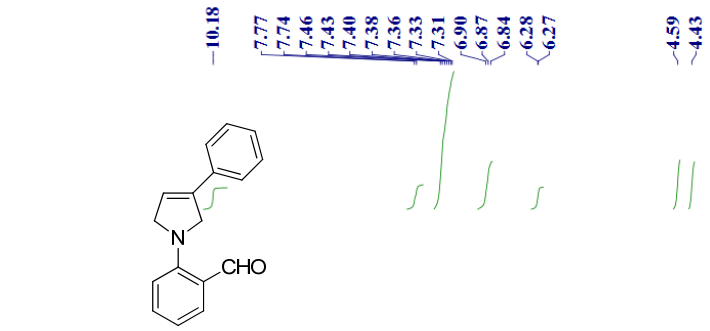
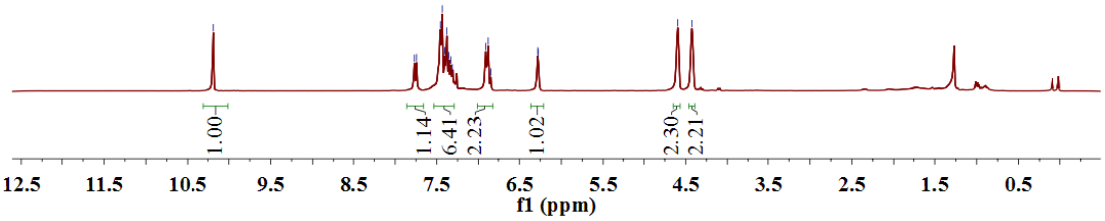
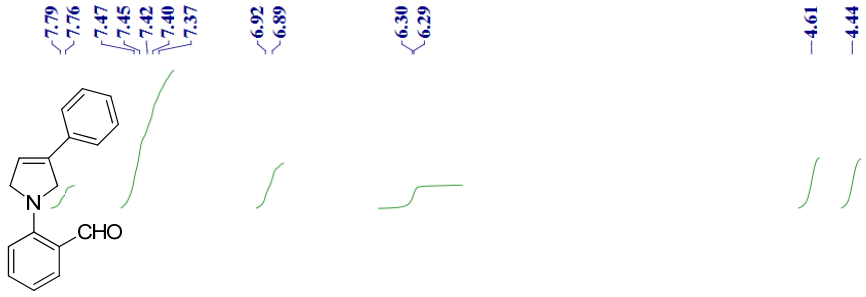
6.72
6.71
5.92
4.39
N
N
CHO
1j
1.00
1.08
1.09
1.07
2.09
4.26
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


189.38
156.02
152.86
142.05
125.53
116.51
112.10
56.98
N
N
CHO
1j
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

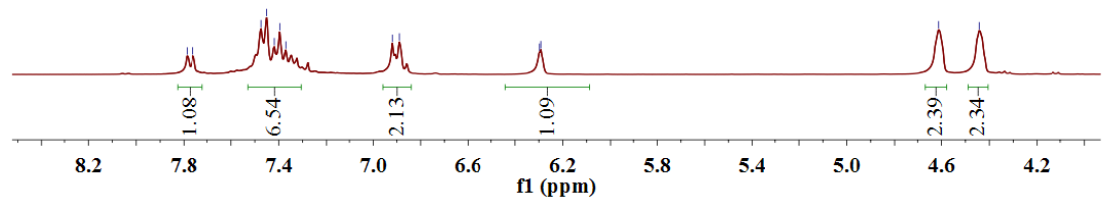

10.18
7.77
7.74
7.46
7.43
7.40
7.38
7.36
7.33
7.31
6.90
6.87
6.84
6.28
6.27
4.59
4.43
N
CHO

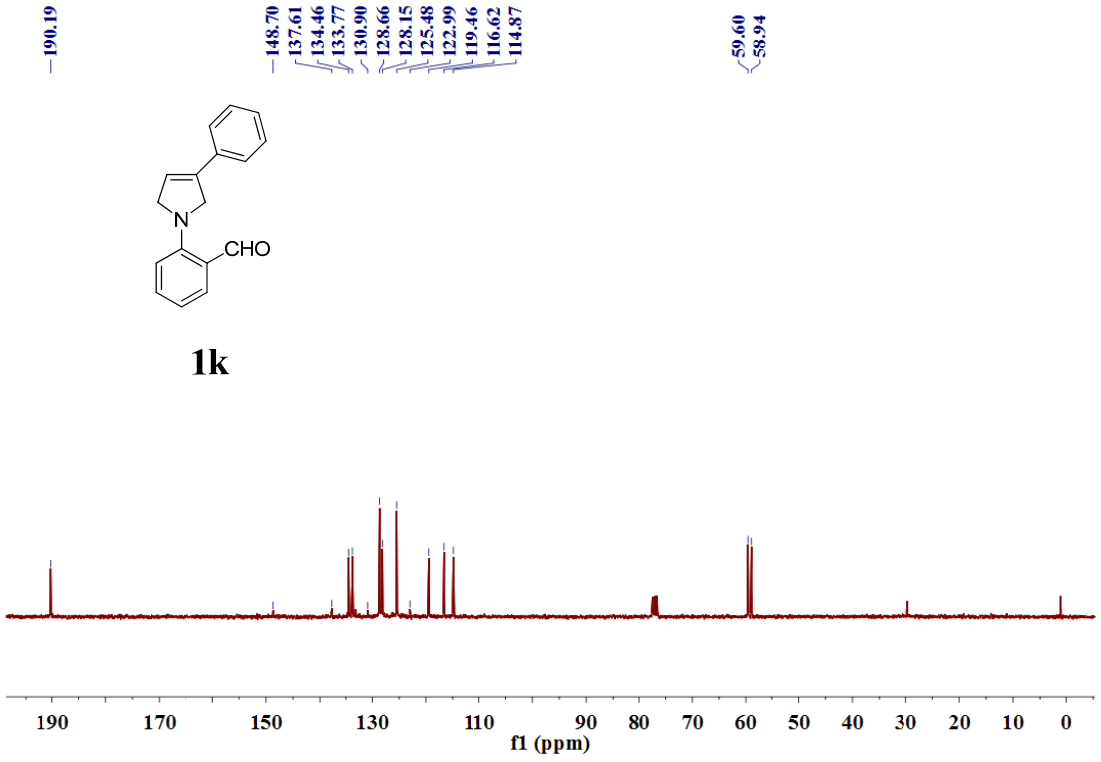
1k


1.00
1.14
6.41
2.23
1.02
2.30
2.21
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

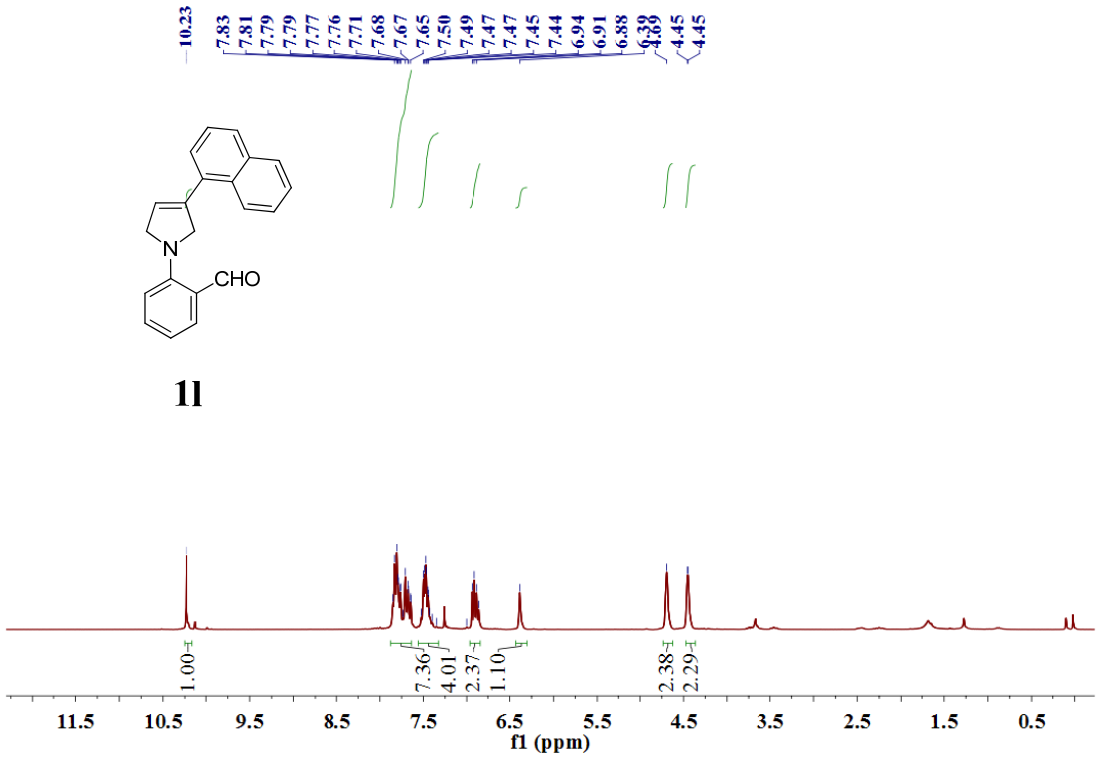

7.79
7.76
7.47
7.45
7.42
7.40
7.37
6.92
6.89
6.30
6.29
4.61
4.44
N
CHO

1k

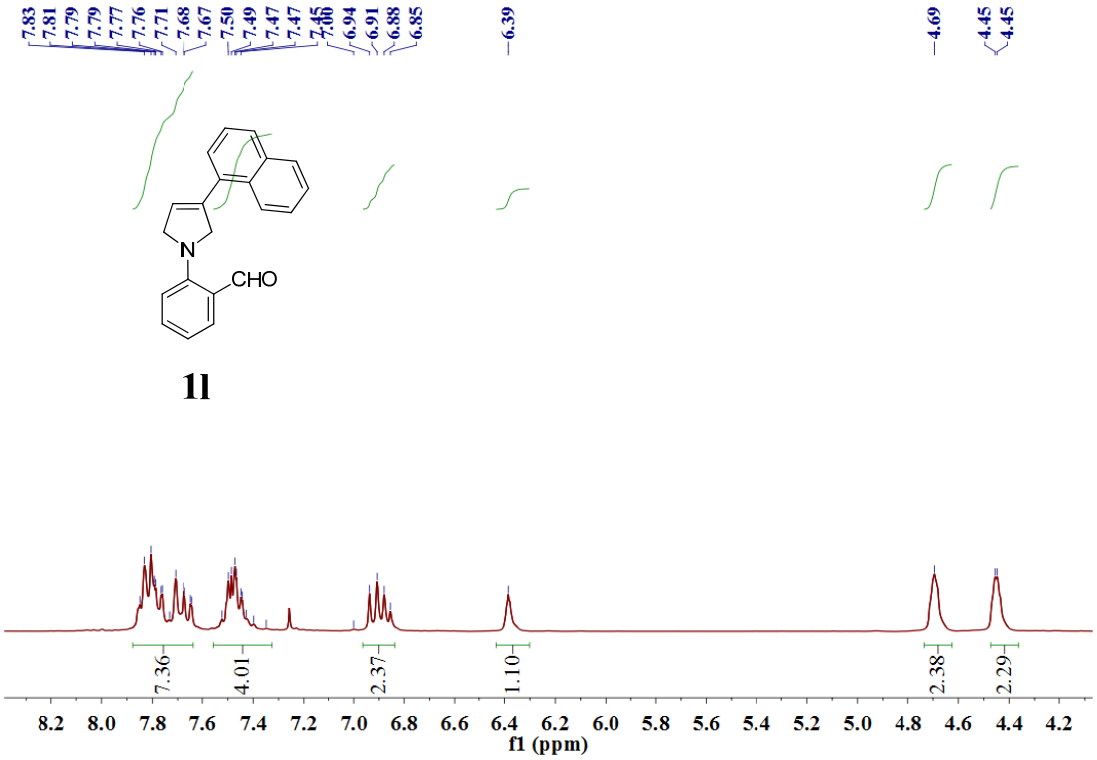

1.08
6.54
2.13
1.09
2.39
2.34
8.2
7.8
7.4
7.0
6.6
6.2
5.8
5.4
5.0
4.6
4.2
f1 (ppm)


190.19
148.70
137.61
134.46
133.77
130.90
128.66
128.15
125.48
122.99
119.46
116.62
114.87
59.60
58.94
N
CHO
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

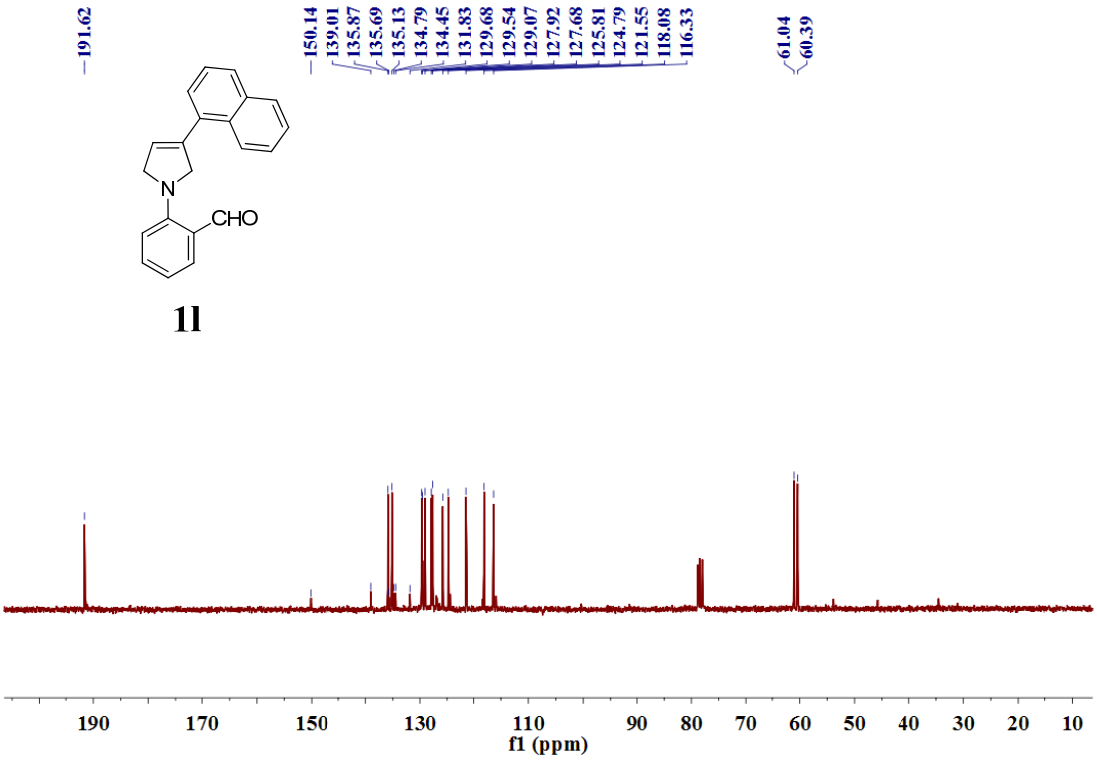
1k


10.23
7.83
7.81
7.79
7.79
7.77
7.76
7.71
7.68
7.67
7.65
7.50
7.49
7.47
7.47
7.45
7.44
6.94
6.91
6.88
4.45
4.45
N
CHO
1.00
7.36
4.01
2.37
1.10
2.38
2.29
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

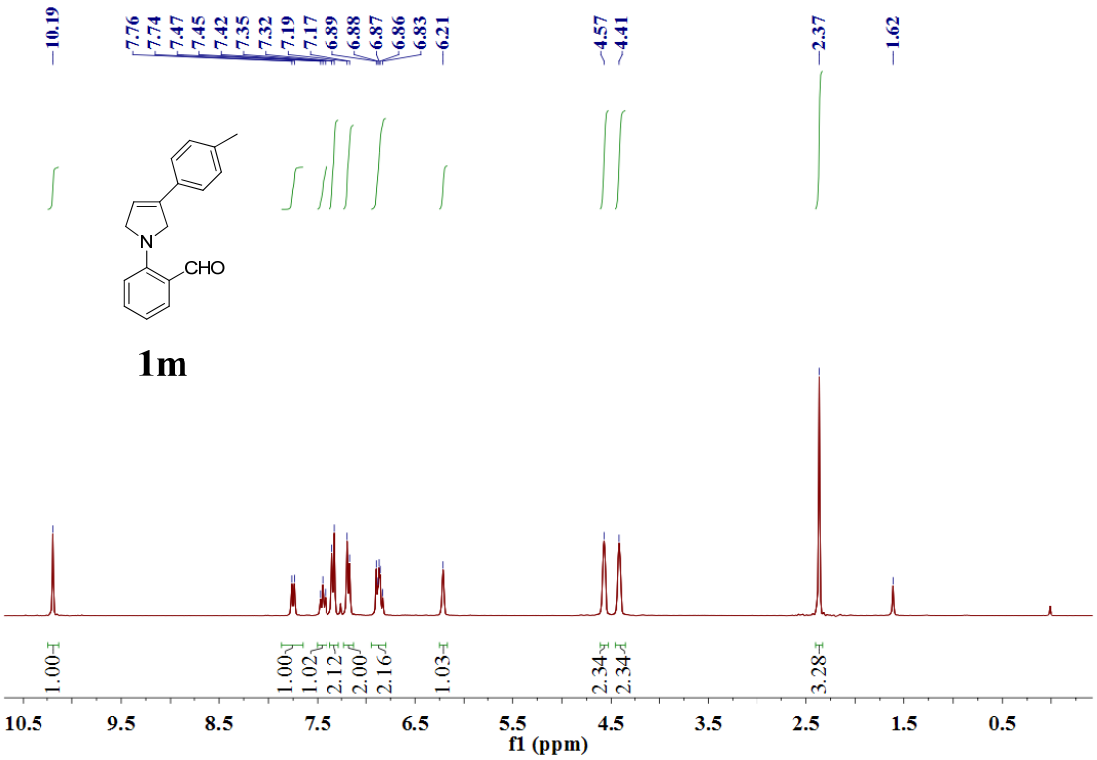
1l


7.83
7.81
7.79
7.79
7.77
7.76
7.71
7.68
7.67
7.50
7.49
7.47
7.47
7.45
7.40
6.94
6.91
6.88
6.85
6.39
4.69
4.45
4.45
N
CHO
7.36
4.01
2.37
1.10
2.38
2.29
8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2
f1 (ppm)

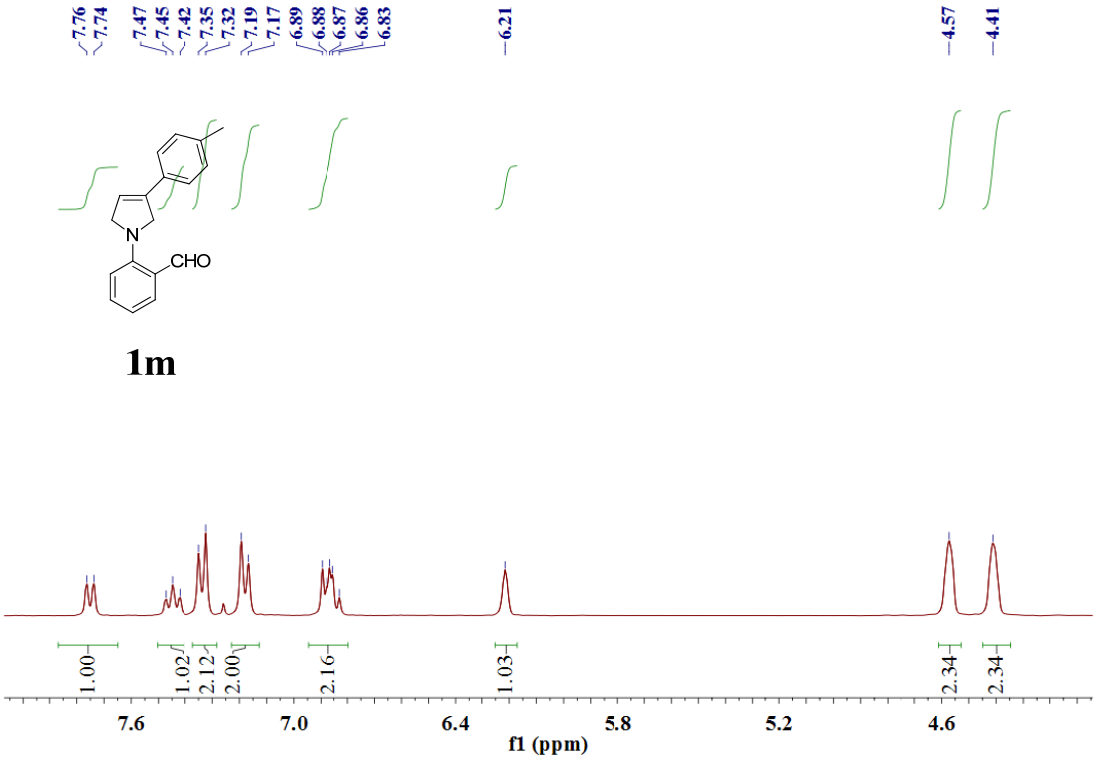
1l


191.62
150.14
139.01
135.87
135.69
135.13
134.79
134.45
131.83
129.68
129.54
129.07
127.92
127.68
125.81
124.79
121.55
118.08
116.33
61.04
60.39
N
CHO
190 170 150 130 110 90 80 70 60 50 40 30 20 10
f1 (ppm)

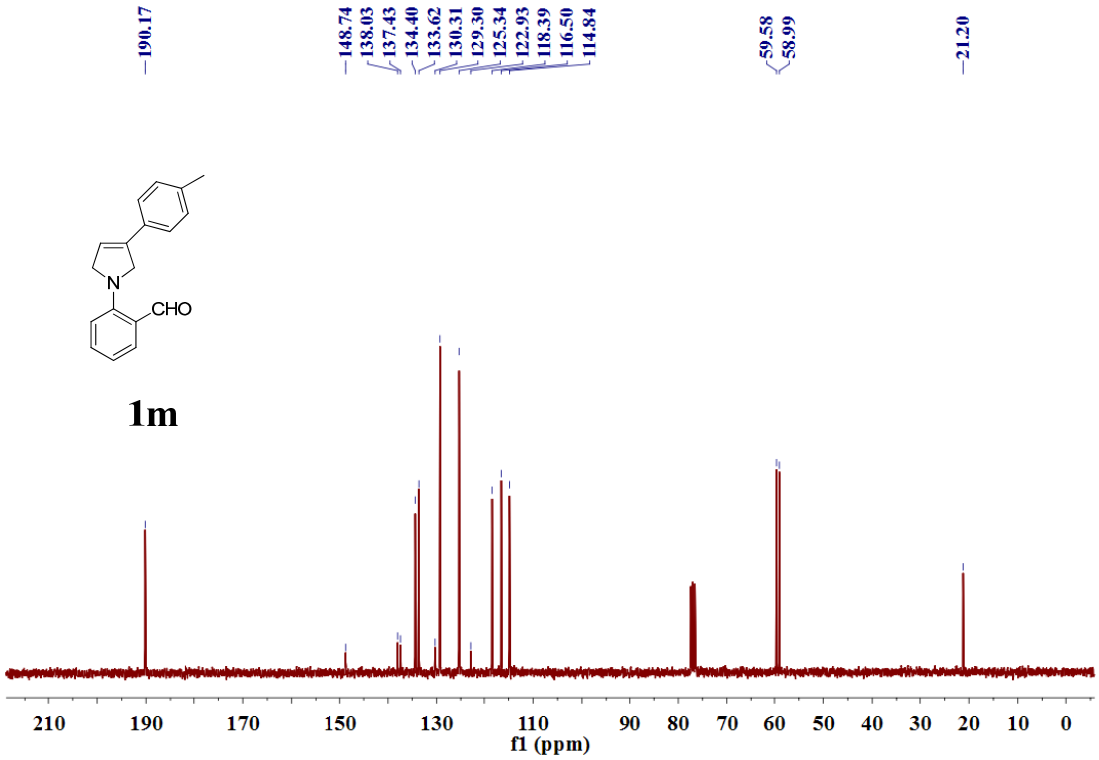
1l

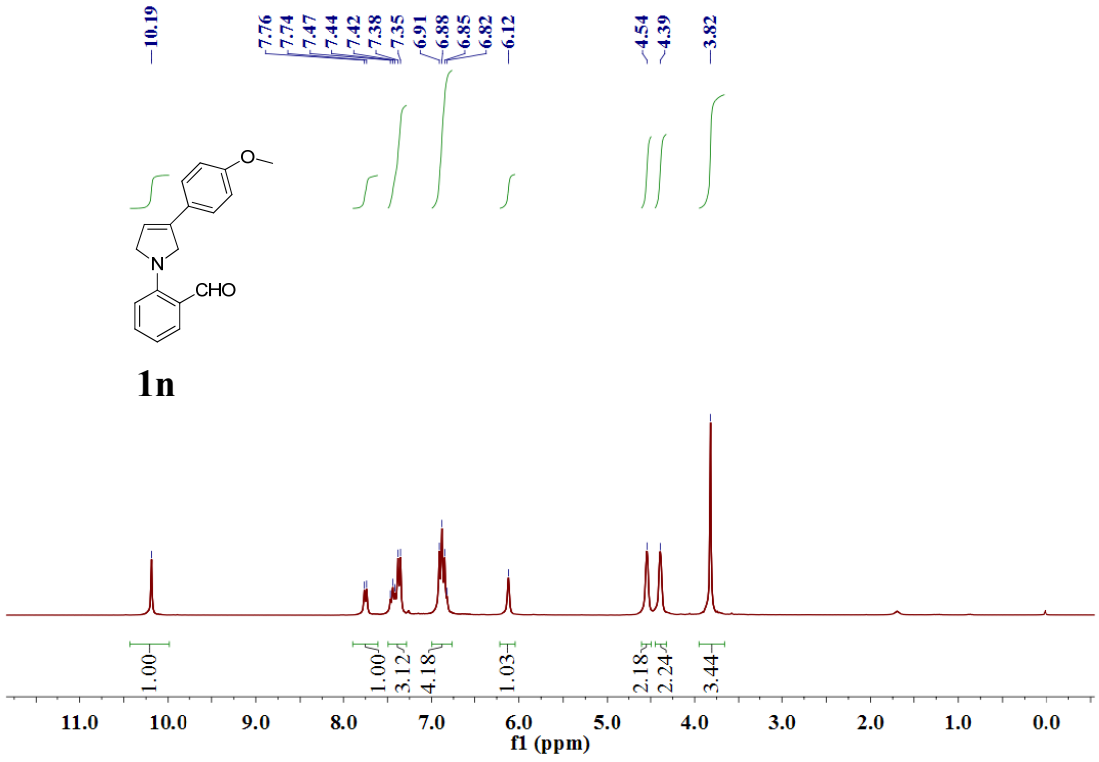

10.19
7.76
7.74
7.47
7.45
7.42
7.35
7.32
7.19
7.17
6.89
6.88
6.87
6.86
6.83
6.21
4.57
4.41
2.37
1.62
N
CHO
1.00
1.00
1.02
2.12
2.00
2.16
1.03
2.34
2.34
3.28
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

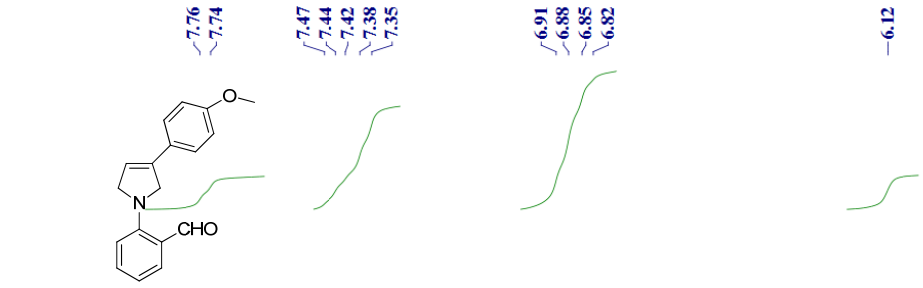
1m


7.76
7.74
7.47
7.45
7.42
7.35
7.32
7.19
7.17
6.89
6.88
6.87
6.86
6.83
6.21
4.57
4.41
N
CHO
1.00
1.02
2.12
2.00
2.16
1.03
2.34
2.34
7.6
7.0
6.4
5.8
5.2
4.6
f1 (ppm)

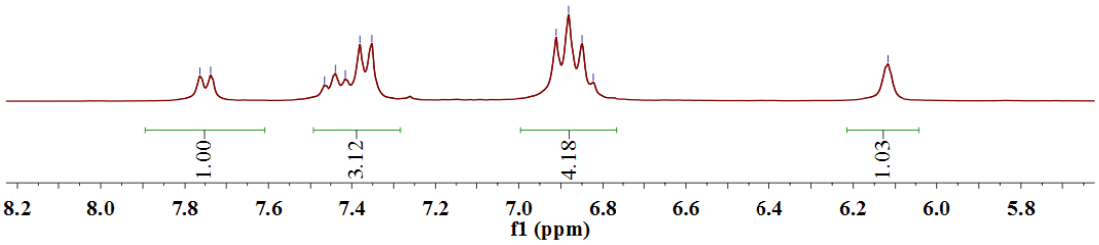
1m

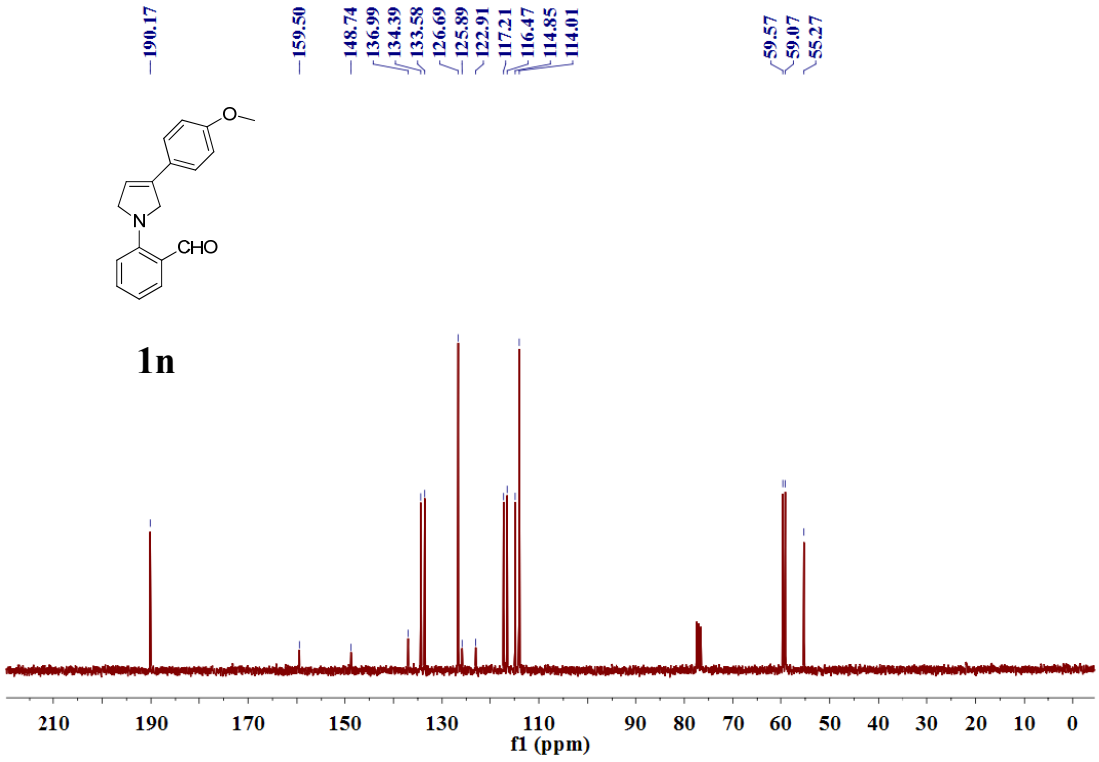

190.17
148.74
138.03
137.43
134.40
133.62
130.31
129.30
125.34
122.93
118.39
116.50
114.84
59.58
58.99
21.20
N
CHO
1m
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


10.19
7.76
7.74
7.47
7.44
7.42
7.38
7.35
6.91
6.88
6.85
6.82
6.12
4.54
4.39
3.82
O
N
CHO
1n
1.00
1.00
3.12
4.18
1.03
2.18
2.24
3.44
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

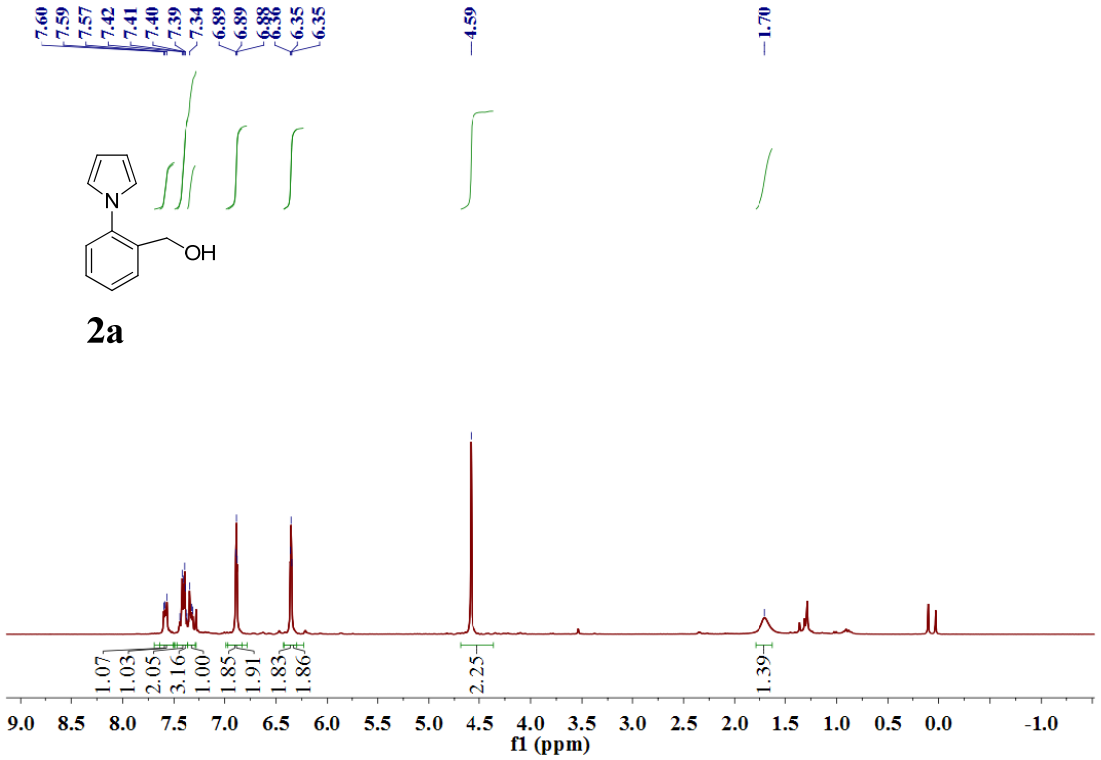

7.76
7.74
7.47
7.44
7.42
7.38
7.35
6.91
6.88
6.85
6.82
6.12
O
N
CHO

1n

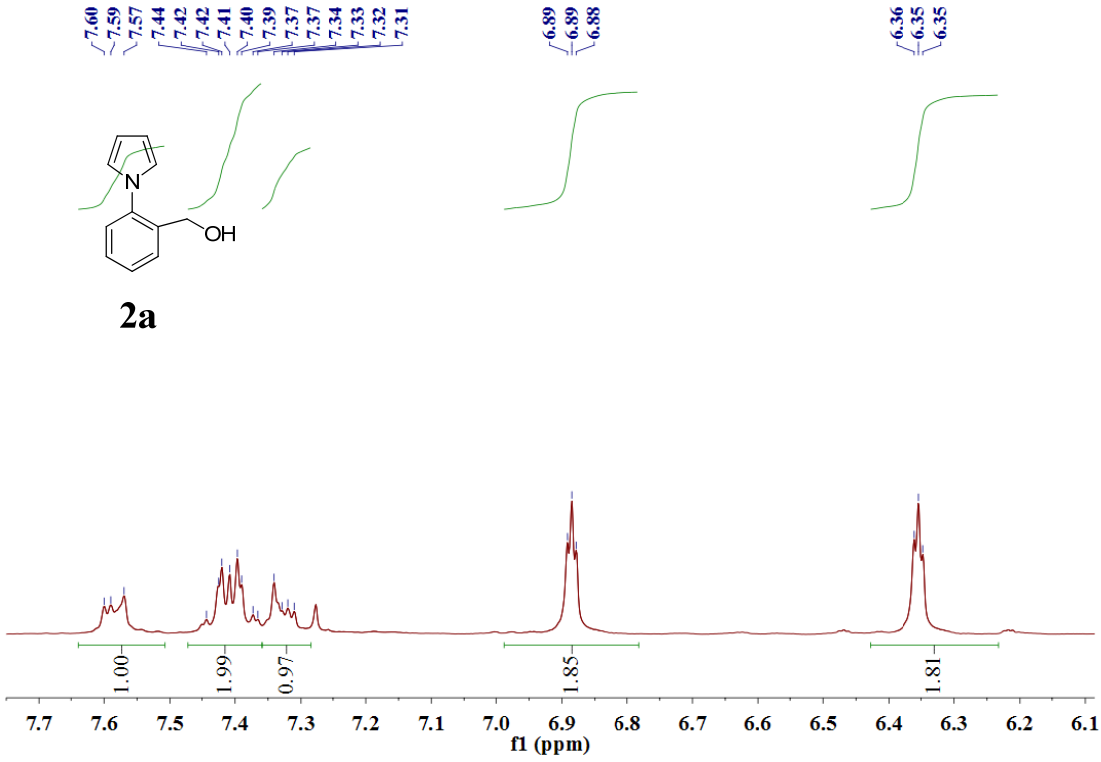

1.00
3.12
4.18
1.03
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
6.0
5.8
f1 (ppm)


190.17
159.50
148.74
136.99
134.39
133.58
126.69
125.89
122.91
117.21
116.47
114.85
114.01
59.57
59.07
55.27
O
N
CHO
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

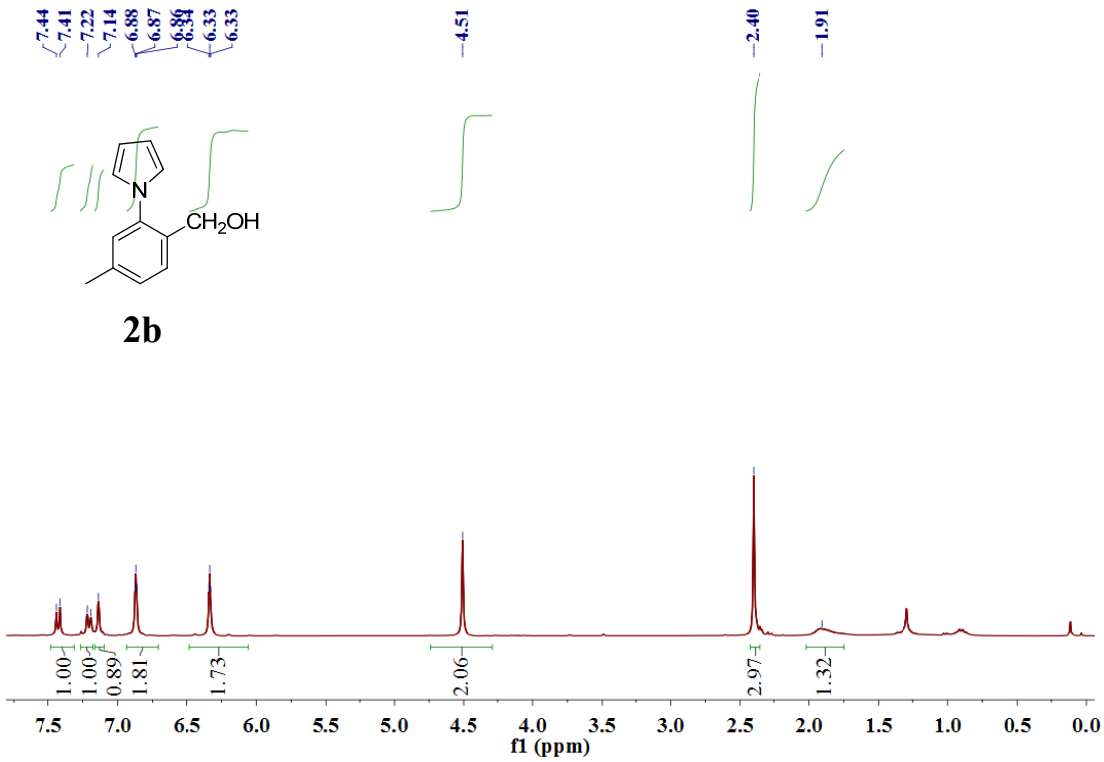
1n


7.60
7.59
7.57
7.42
7.41
7.40
7.39
7.34
6.89
6.89
6.88
6.36
6.35
6.35
4.59
1.70
N
OH
1.07
1.03
2.05
3.16
1.00
1.85
1.91
1.83
1.86
2.25
1.39
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 -1.0
f1 (ppm)

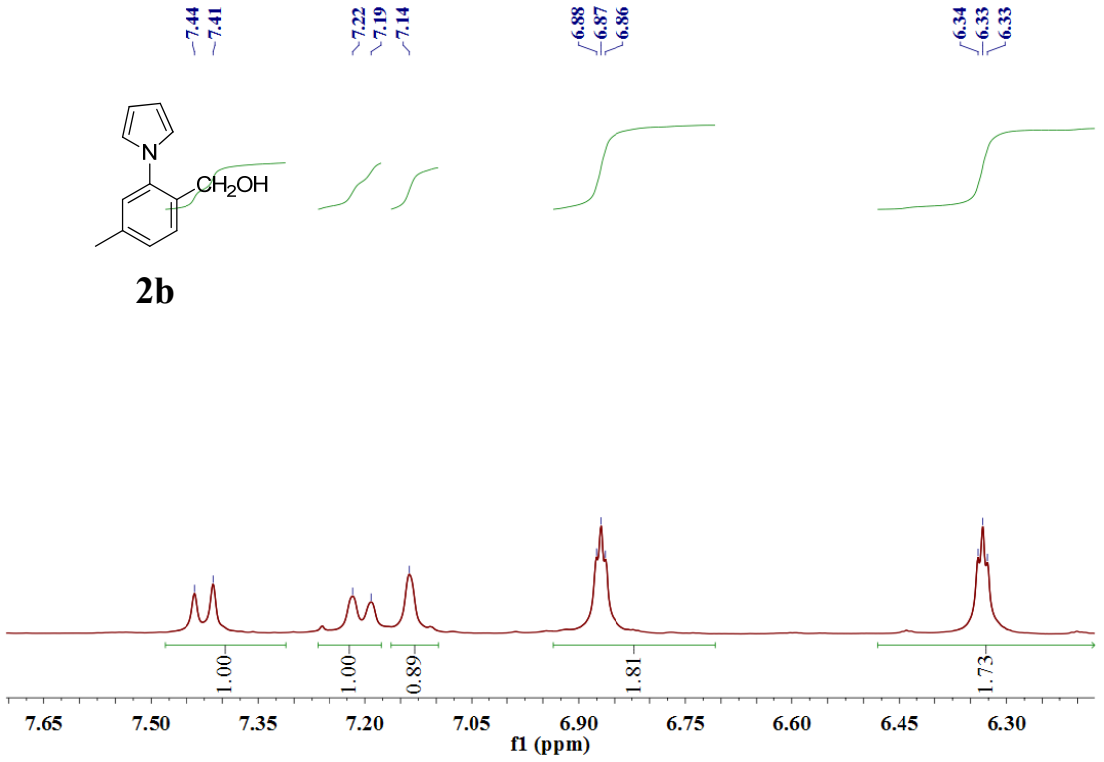
2a


7.60
7.59
7.57
7.44
7.42
7.42
7.41
7.40
7.39
7.37
7.37
7.34
7.33
7.32
7.31
6.89
6.89
6.88
6.36
6.35
6.35
N
OH
1.00
1.99
0.97
1.85
1.81
7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1
f1 (ppm)

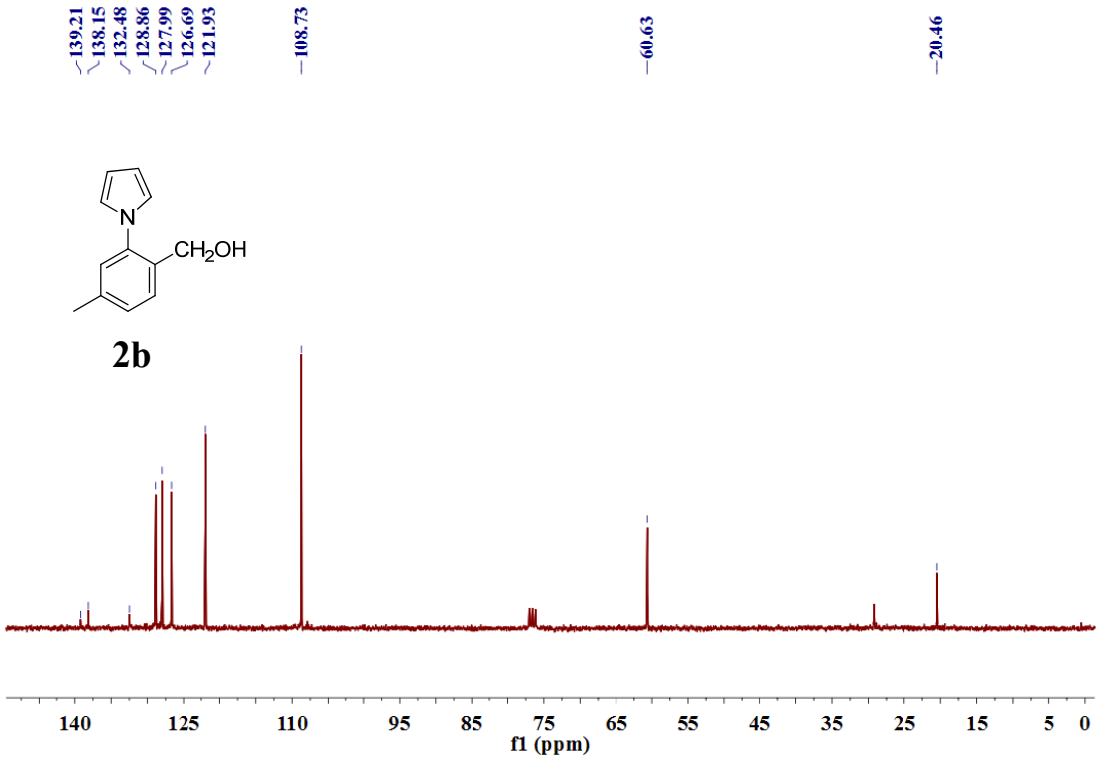
2a


7.44
7.41
7.22
7.14
6.88
6.87
6.86
6.34
6.33
6.33
4.51
2.40
1.91
N
CH2OH
1.00
1.00
0.89
1.81
1.73
2.06
2.97
1.32
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
f1 (ppm)

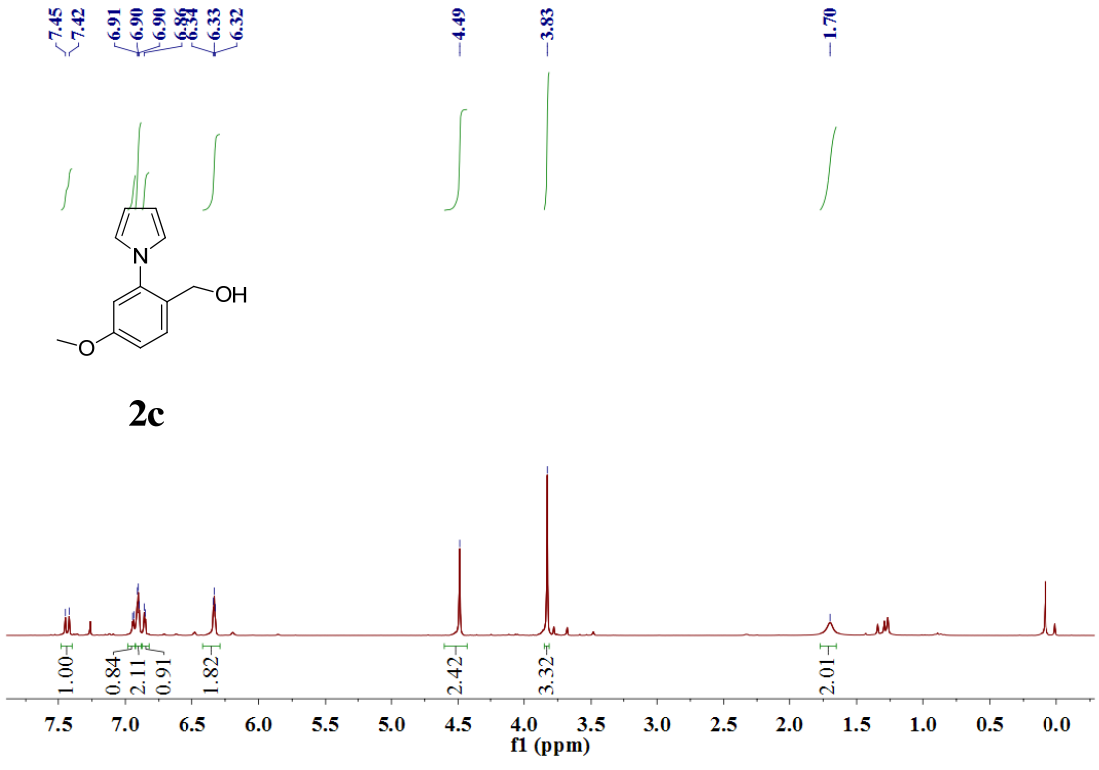
2b


7.44
7.41
7.22
7.19
7.14
6.88
6.87
6.86
6.34
6.33
6.33
N
CH2OH
1.00
1.00
0.89
1.81
1.73
7.65
7.50
7.35
7.20
7.05
6.90
6.75
6.60
6.45
6.30
f1 (ppm)

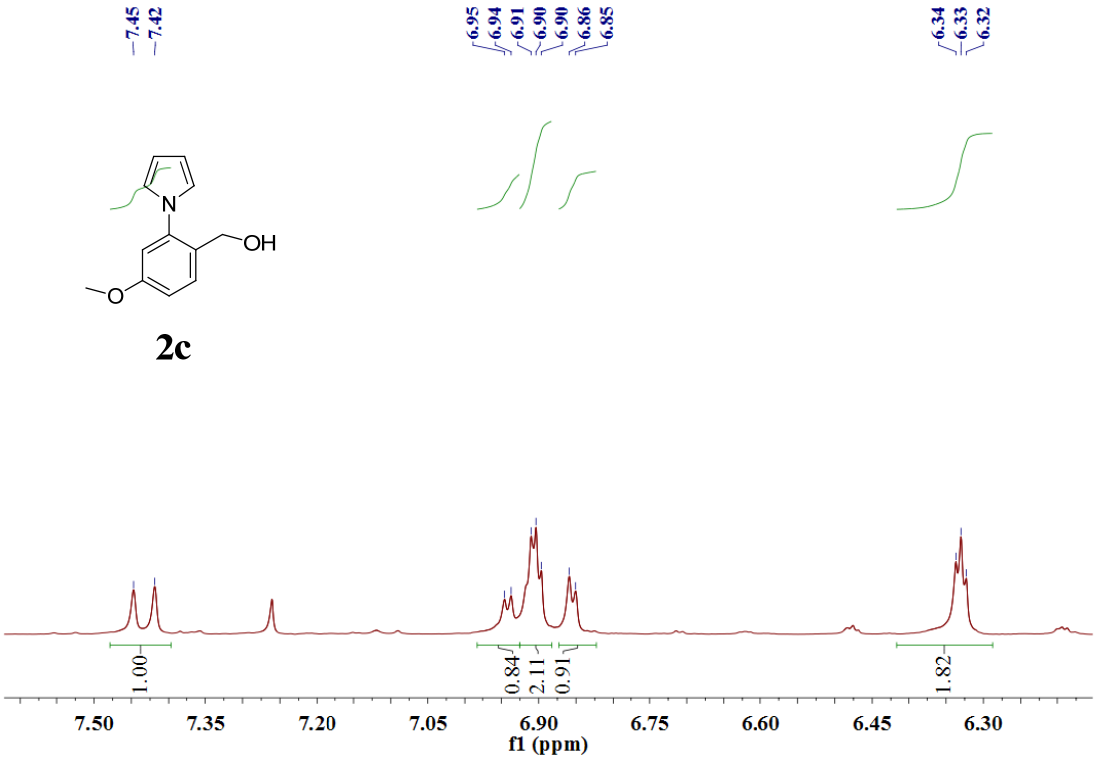
2b


139.21
138.15
132.48
128.86
127.99
126.69
121.93
108.73
60.63
20.46
N
CH2OH
140
125
110
95
85
75
65
55
45
35
25
15
5
0
f1 (ppm)

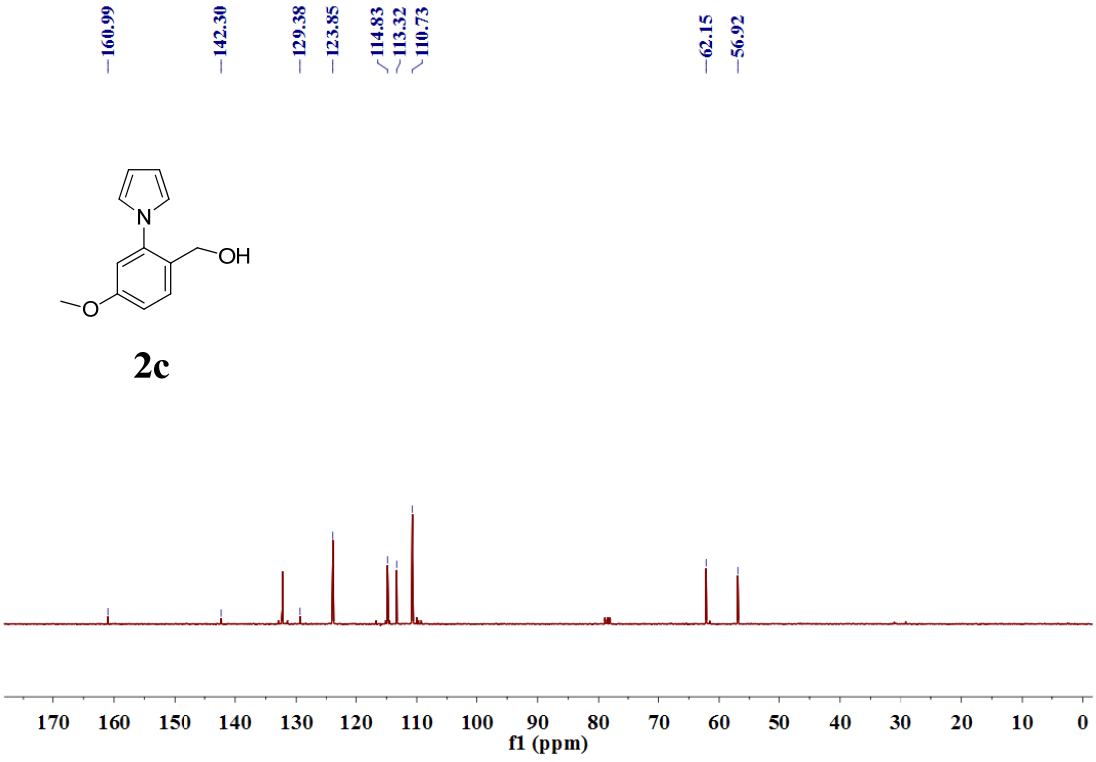
2b


7.45
7.42
6.91
6.90
6.90
6.86
6.34
6.33
6.32
4.49
3.83
1.70
N
OH
O
1.00
0.84
2.11
0.91
1.82
2.42
3.32
2.01
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
f1 (ppm)

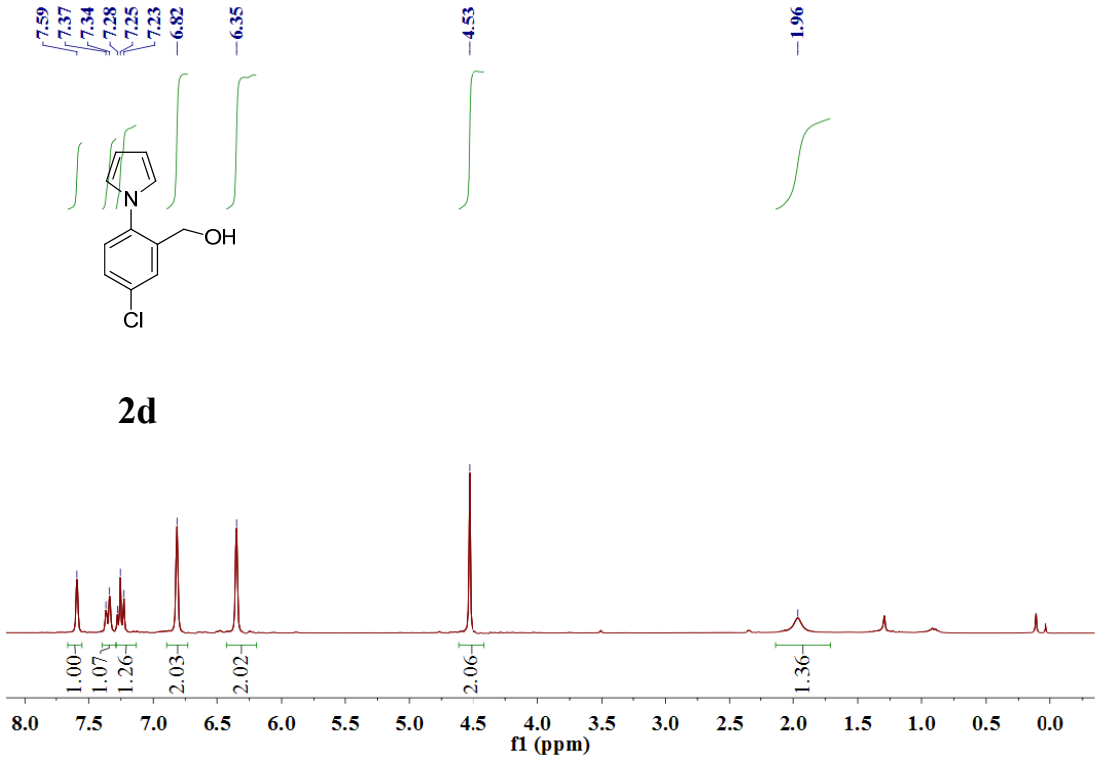
2c


7.45
7.42
6.95
6.94
6.91
6.90
6.90
6.86
6.85
6.34
6.33
6.32
N
OH
O
1.00
0.84
2.11
0.91
1.82
7.50
7.35
7.20
7.05
6.90
6.75
6.60
6.45
6.30
f1 (ppm)

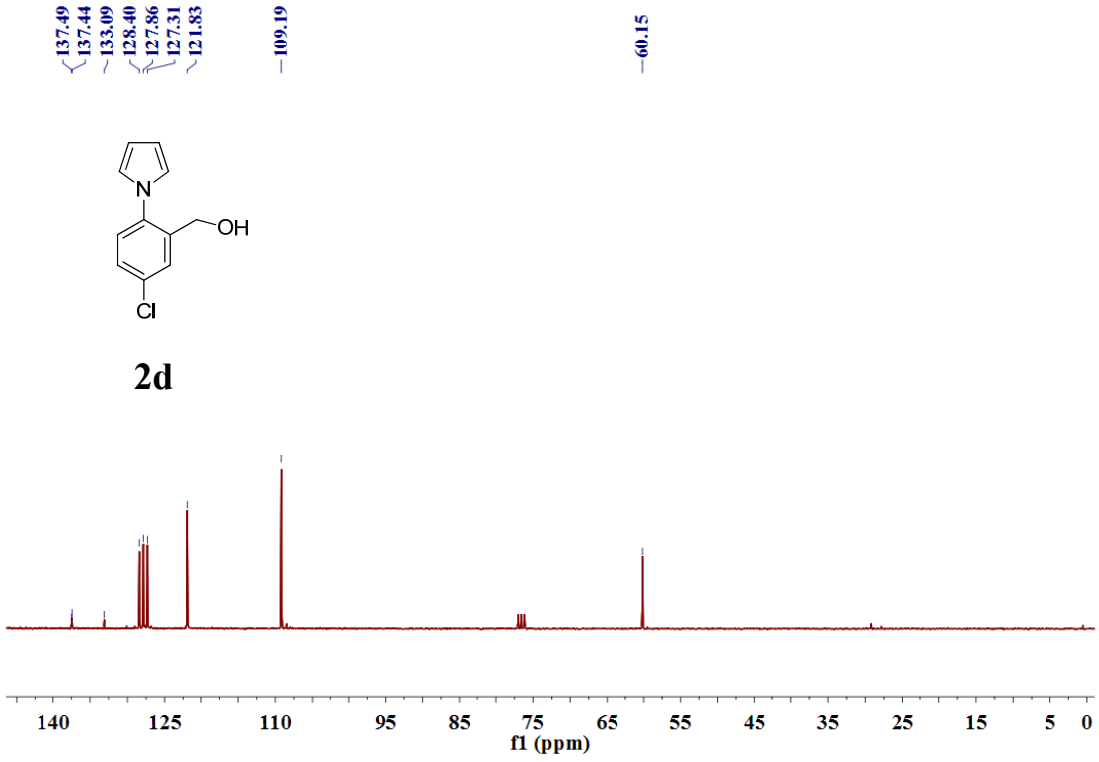
2c


160.99
142.30
129.38
123.85
114.83
113.32
110.73
62.15
56.92
N
OH
O
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

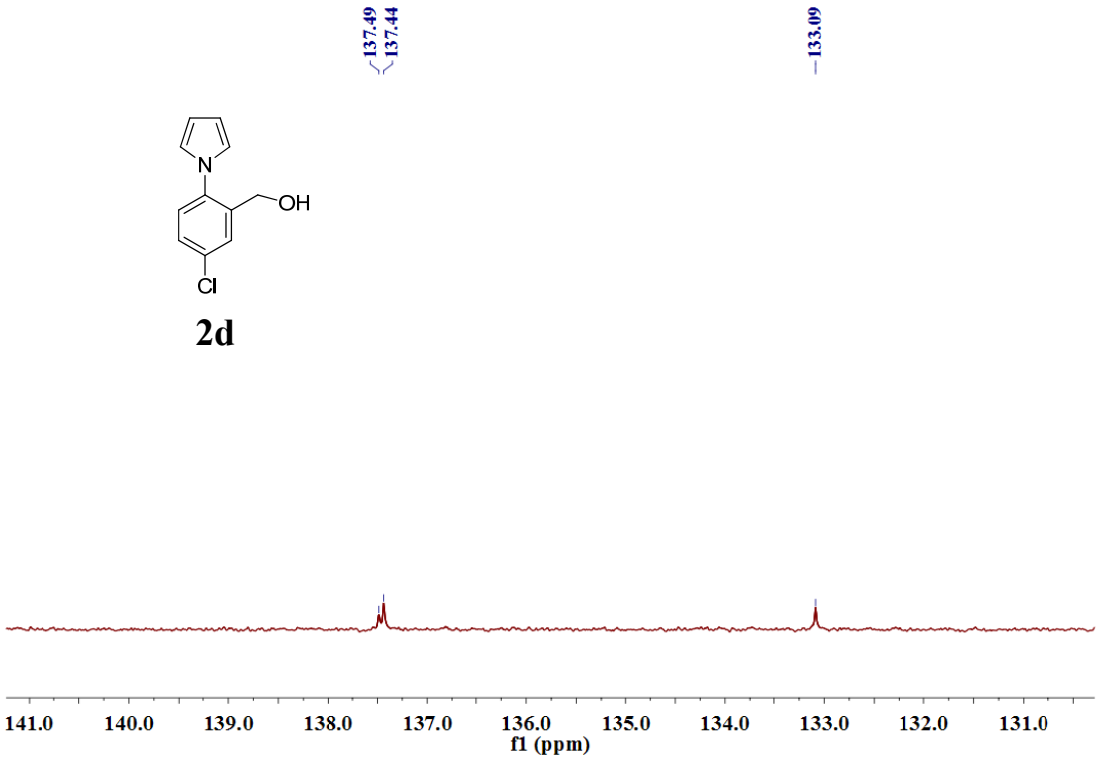
2c


7.59
7.37
7.34
7.28
7.25
7.23
6.82
6.35
4.53
1.96
N
OH
Cl
1.00
1.07
1.26
2.03
2.02
2.06
1.36
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
f1 (ppm)

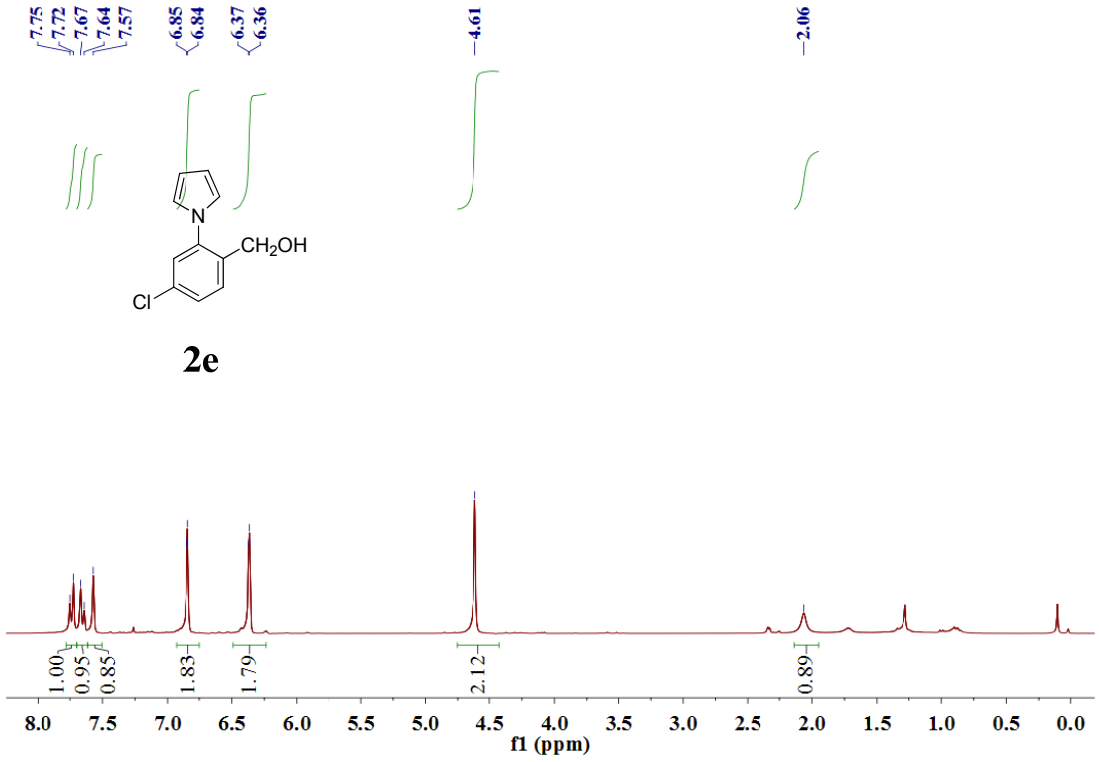
2d


137.49
137.44
133.09
128.40
127.86
127.31
121.83
109.19
60.15
N
OH
Cl
140
125
110
95
85
75
65
55
45
35
25
15
5
0
f1 (ppm)

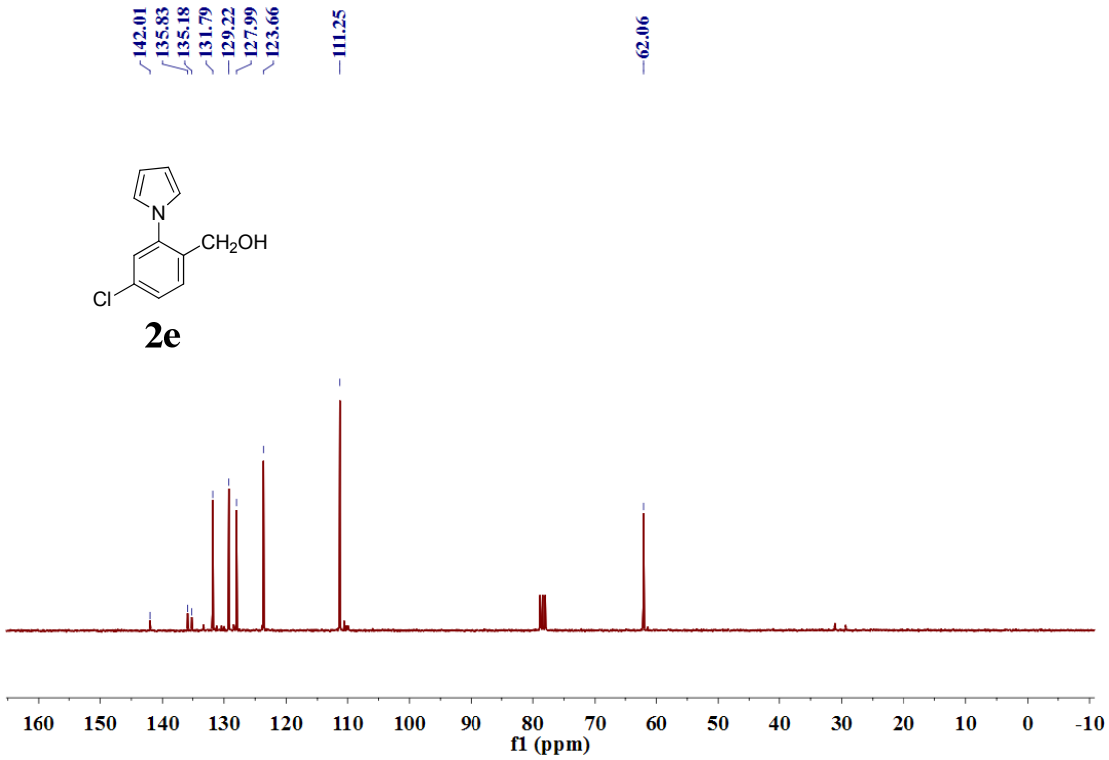
2d


137.49
137.44
133.09
N
OH
Cl
141.0
140.0
139.0
138.0
137.0
136.0
135.0
134.0
133.0
132.0
131.0
f1 (ppm)

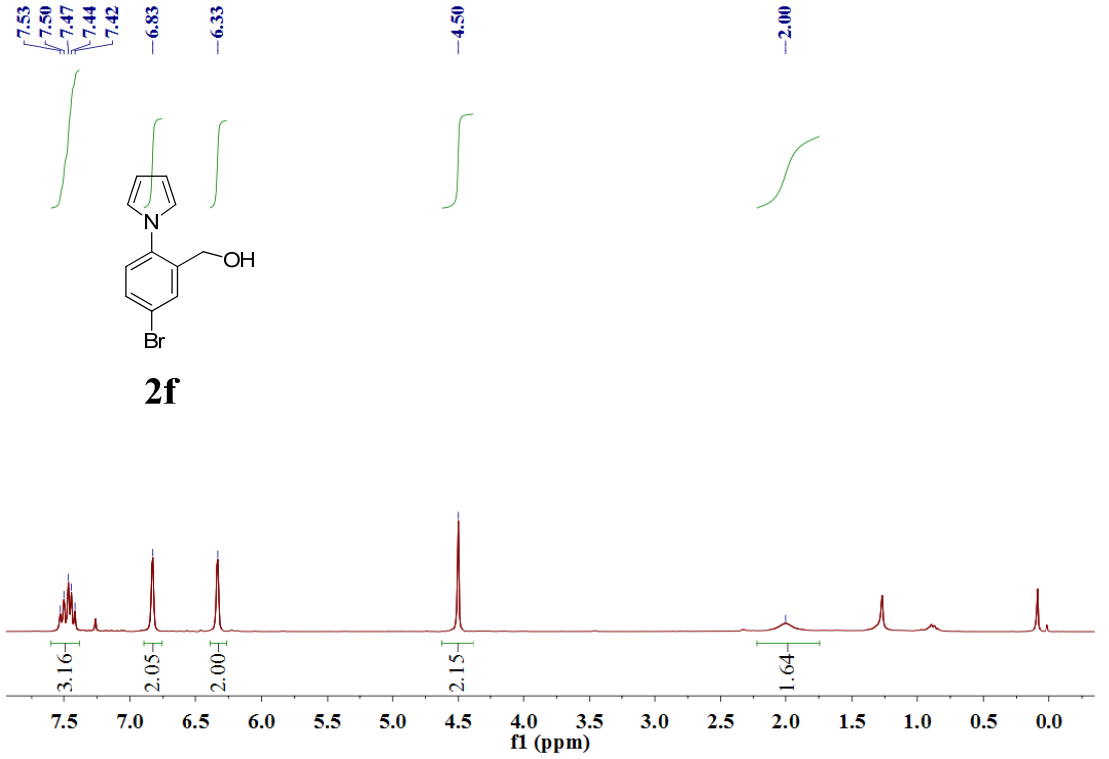
2d


7.75
7.72
7.67
7.64
7.57
6.85
6.84
6.37
6.36
4.61
2.06
N
CH2OH
Cl
1.00
0.95
0.85
1.83
1.79
2.12
0.89
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
f1 (ppm)

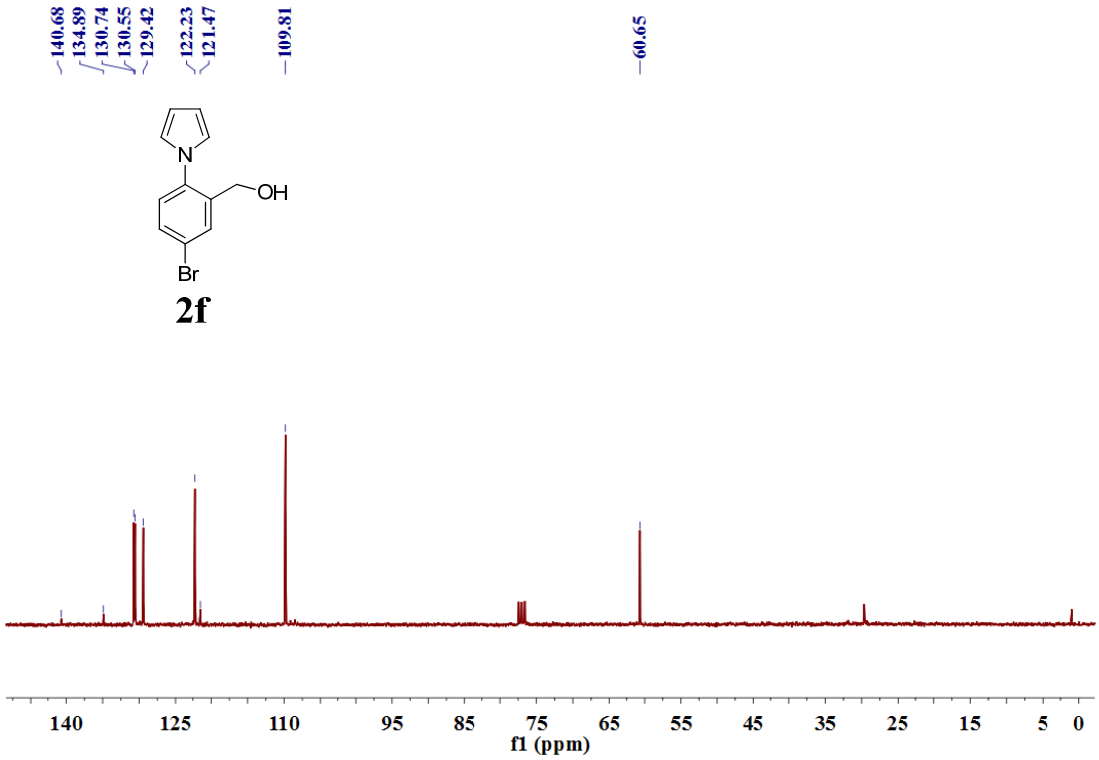
2e


142.01
135.83
135.18
131.79
129.22
127.99
123.66
111.25
62.06
N
CH2OH
Cl
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

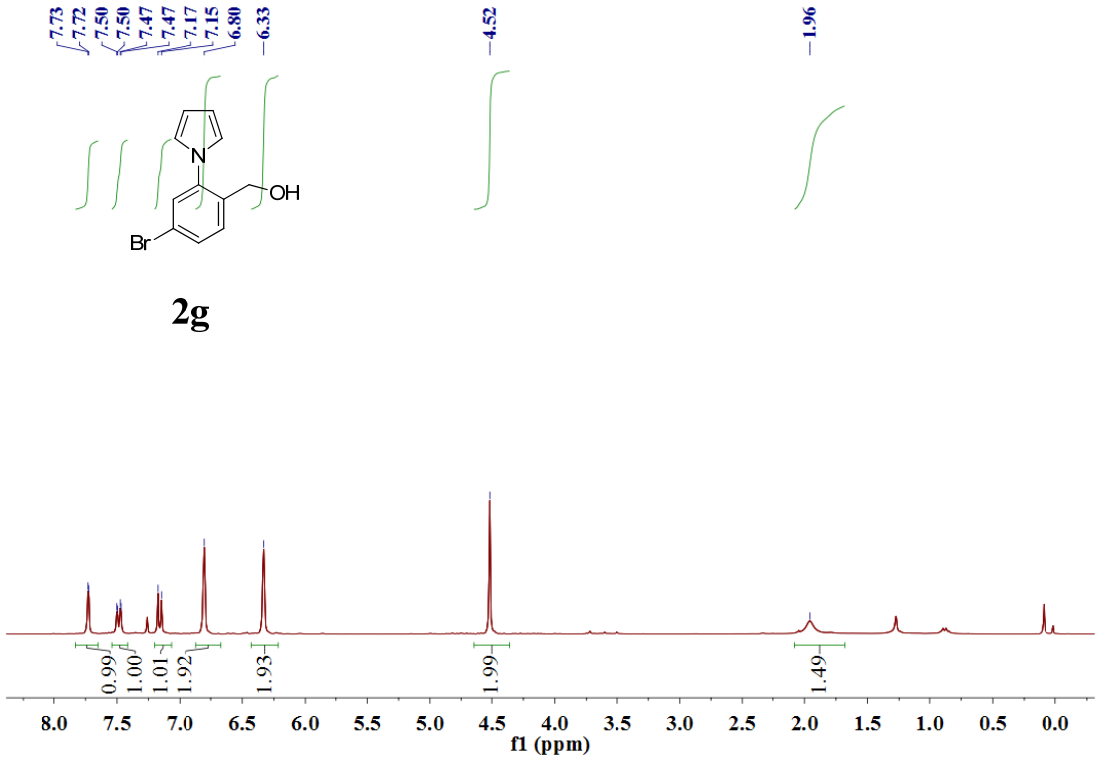
2e


7.53
7.50
7.47
7.44
7.42
6.83
6.33
4.50
2.00
N
OH
Br
3.16
2.05
2.00
2.15
1.64
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
f1 (ppm)

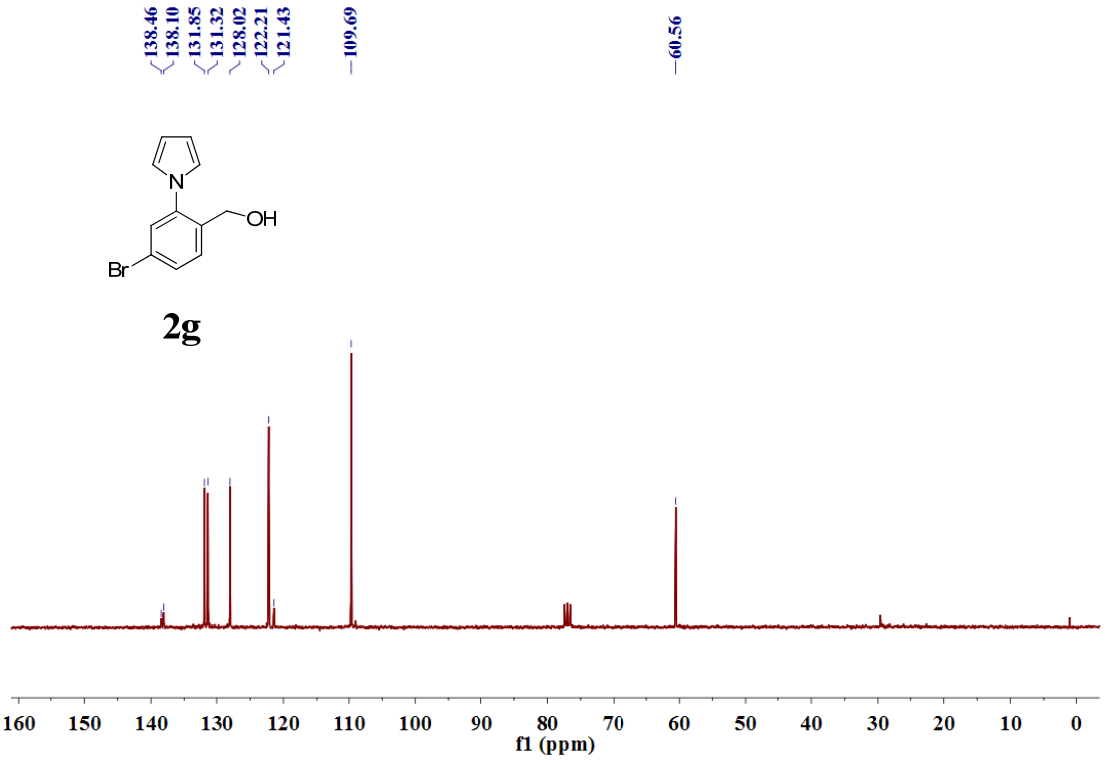
2f


140.68
134.89
130.74
130.55
129.42
122.23
121.47
109.81
60.65
N
OH
Br
140
125
110
95
85
75
65
55
45
35
25
15
5
0
f1 (ppm)

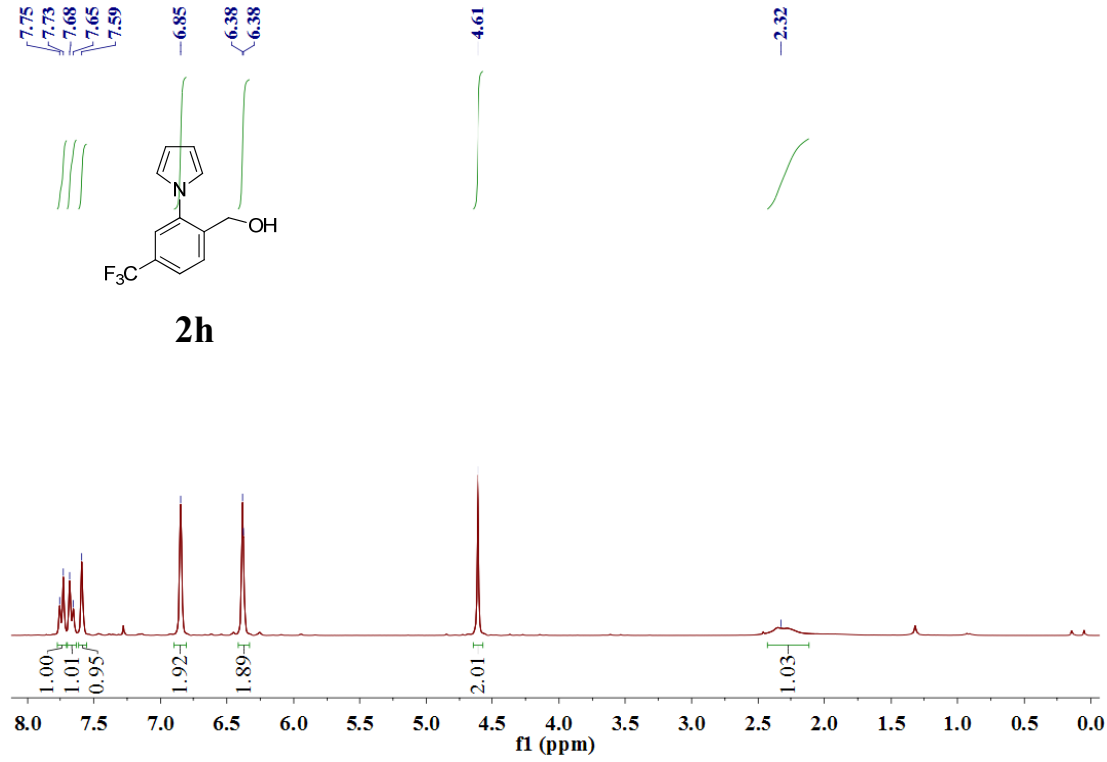
2f


7.73
7.72
7.50
7.50
7.47
7.47
7.17
7.15
6.80
6.33
4.52
1.96
N
OH
Br
0.99
1.00
1.01
1.92
1.93
1.99
1.49
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
f1 (ppm)

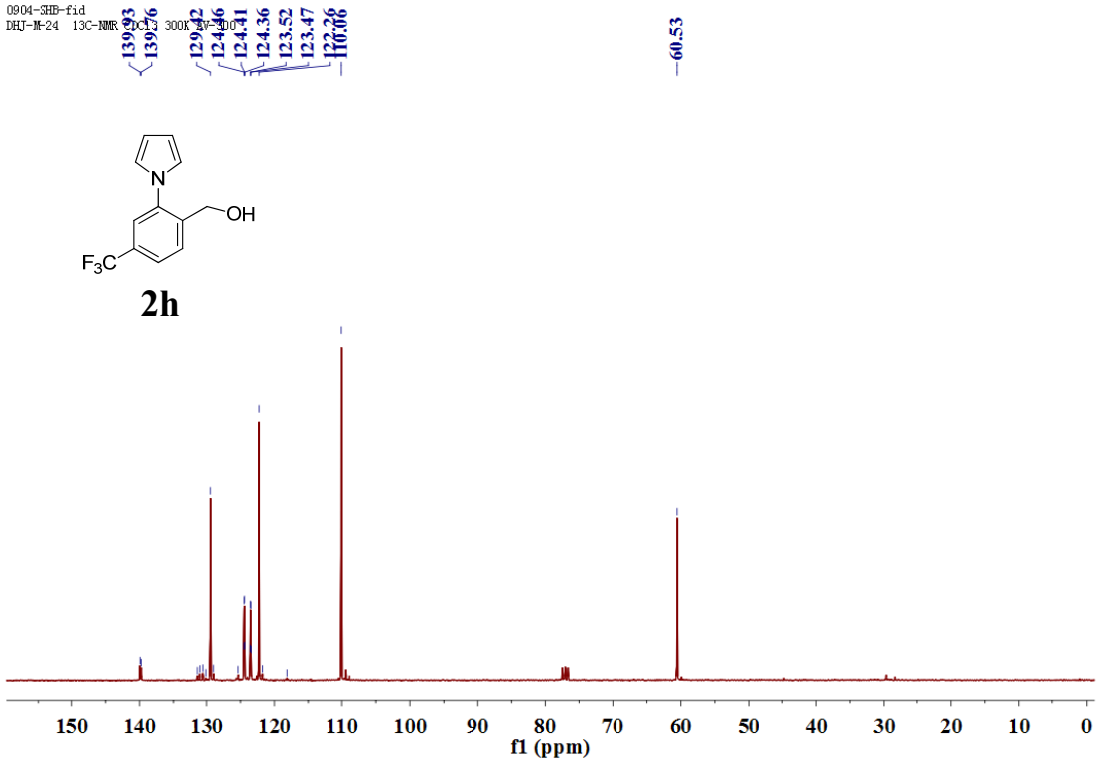
2g


138.46
138.10
131.85
131.32
128.02
122.21
121.43
109.69
60.56
N
OH
Br
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

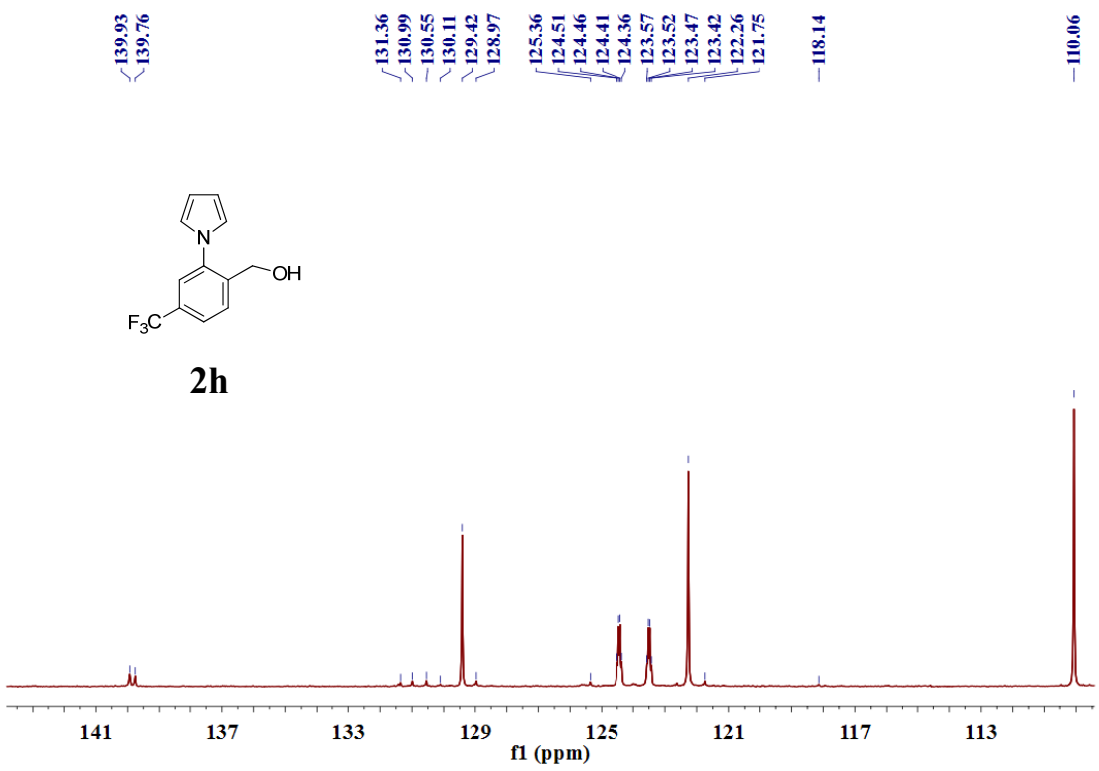
2g


7.75
7.73
7.68
7.65
7.59
6.85
6.38
6.38
4.61
2.32
N
OH
F3C
1.00
1.01
0.95
1.92
1.89
2.01
1.03
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
f1 (ppm)

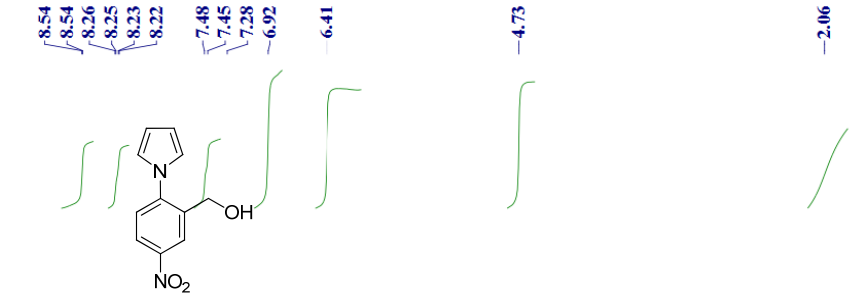
2h


0904-SHB-fid
DHJ-M-24 13C-NMR CDCl3 300K
139.93
139.76
129.42
124.46
124.41
124.36
123.52
123.47
122.26
110.06
60.53
N
OH
F3C
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

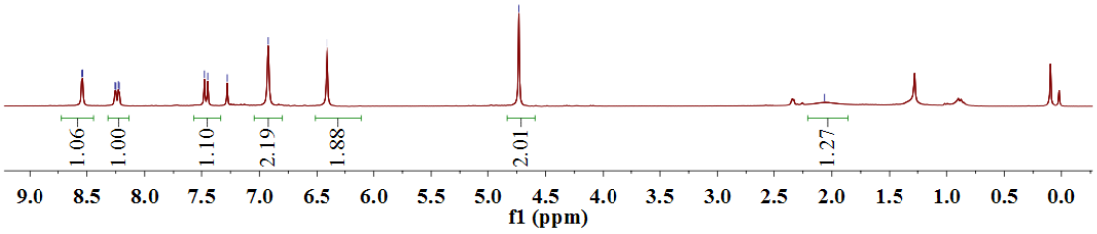
2h


139.93
139.76
131.36
130.99
130.55
130.11
129.42
128.97
125.36
124.51
124.46
124.41
124.36
123.57
123.52
123.47
123.42
122.26
121.75
118.14
110.06
N
OH
F3C
141 137 133 129 125 121 117 113
f1 (ppm)

2h

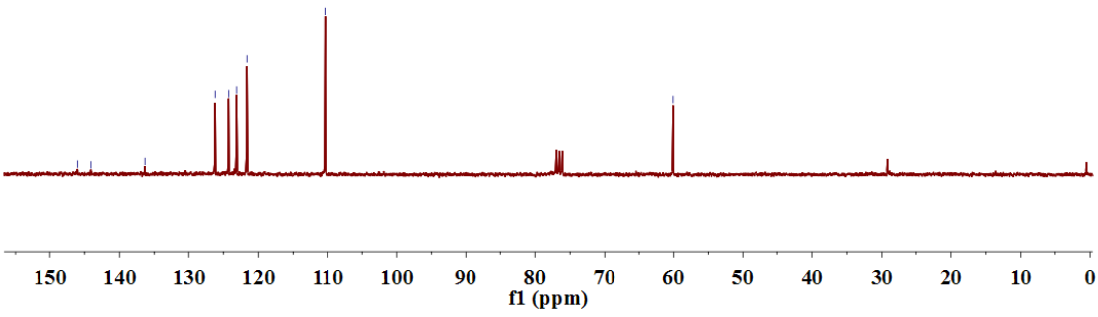

8.54
8.54
8.26
8.25
8.23
8.22
7.48
7.45
7.28
6.92
6.41
4.73
2.06
N
OH
NO2

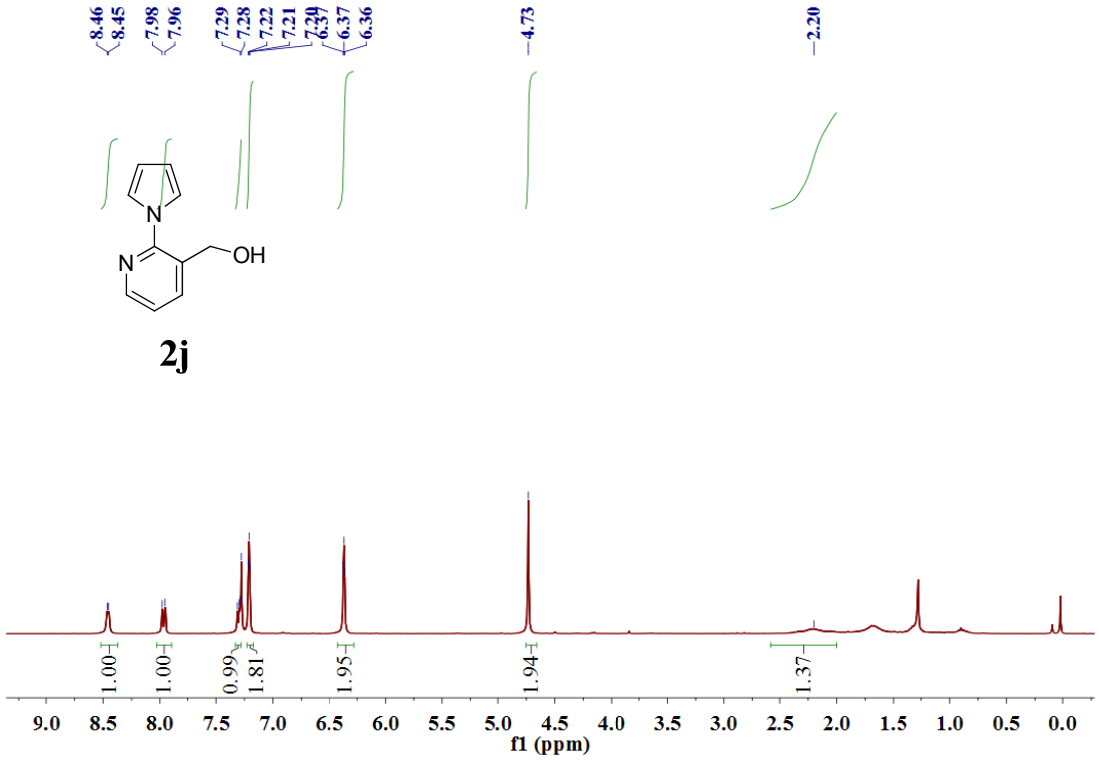
2i


1.06
1.00
1.10
2.19
1.88
2.01
1.27
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
f1 (ppm)

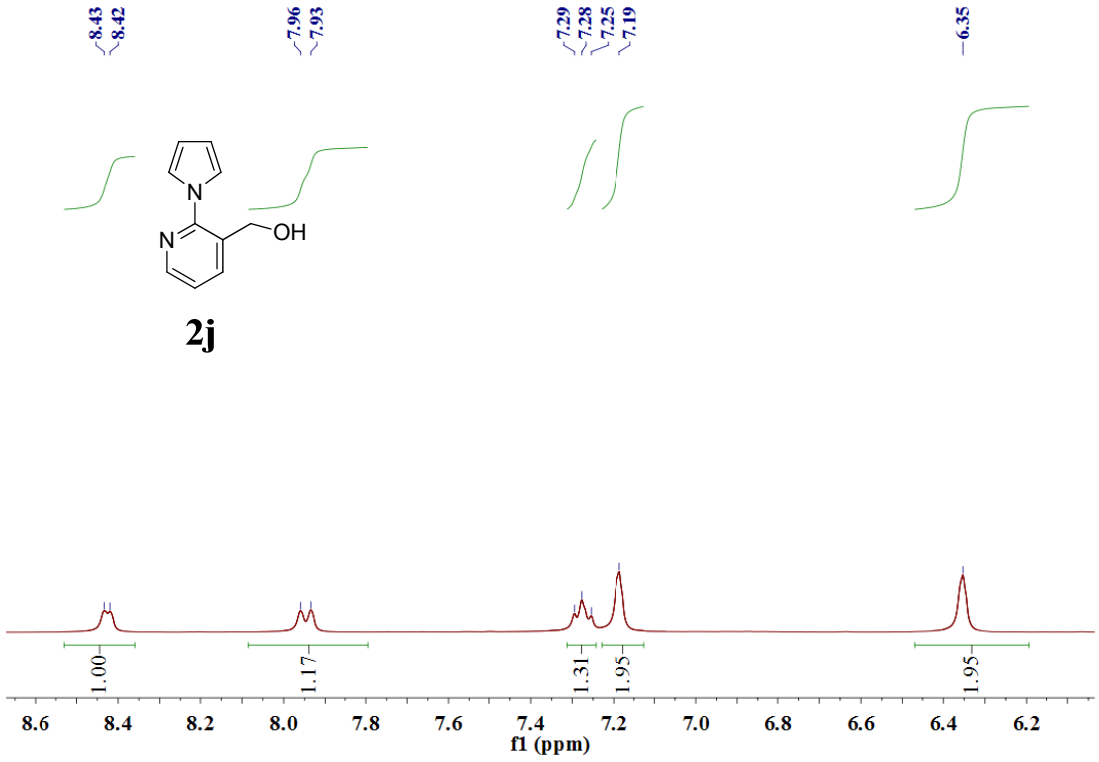

146.01
144.07
136.33
126.17
124.24
123.10
121.61
110.39
60.07
N
OH
NO2

2i

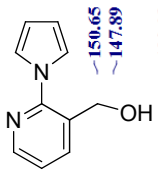

150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.46
8.45
7.98
7.96
7.29
7.28
7.22
7.21
7.20
6.37
6.36
4.73
2.20
N
N
OH
1.00
1.00
0.99
1.81
1.95
1.94
1.37
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
f1 (ppm)

2j

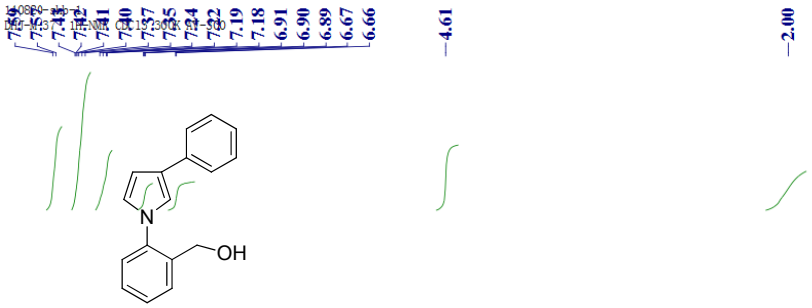

8.43
8.42
7.96
7.93
7.29
7.28
7.25
7.19
6.35
N
N
OH
1.00
1.17
1.31
1.95
1.95
8.6 8.4 8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2
f1 (ppm)

2j

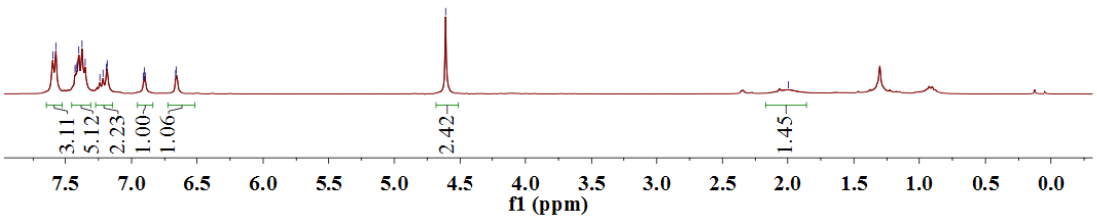

N
N
OH

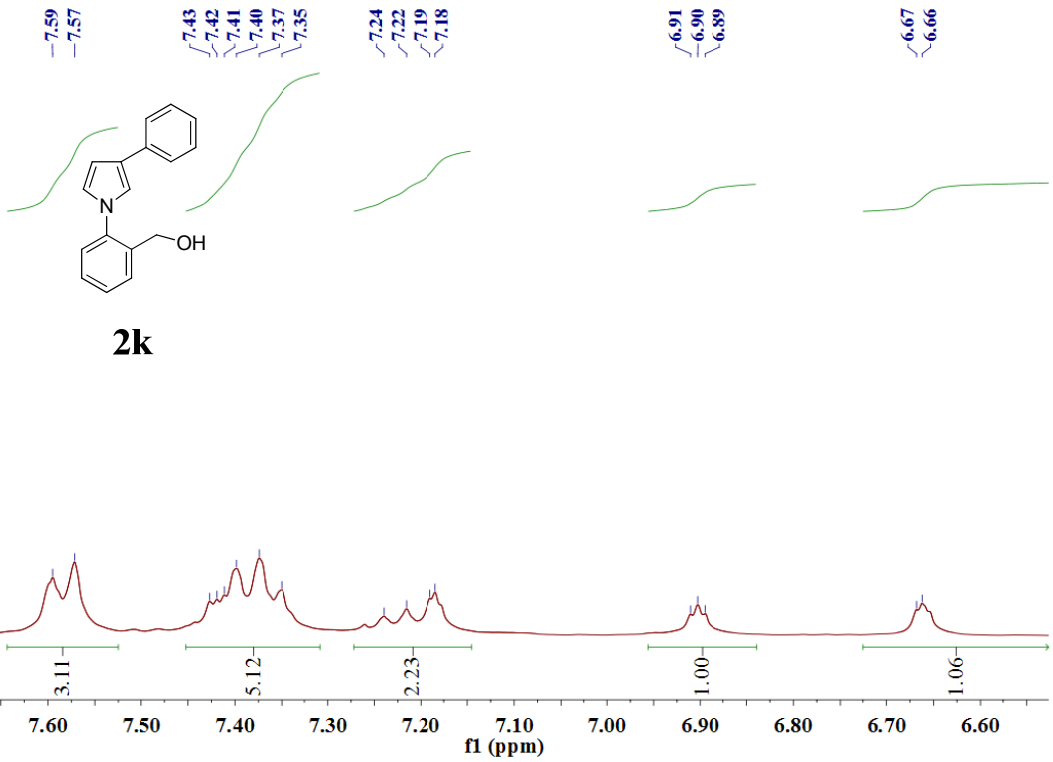
2j

150.65
147.89
139.08
128.43
122.05
121.24
110.23
60.71
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

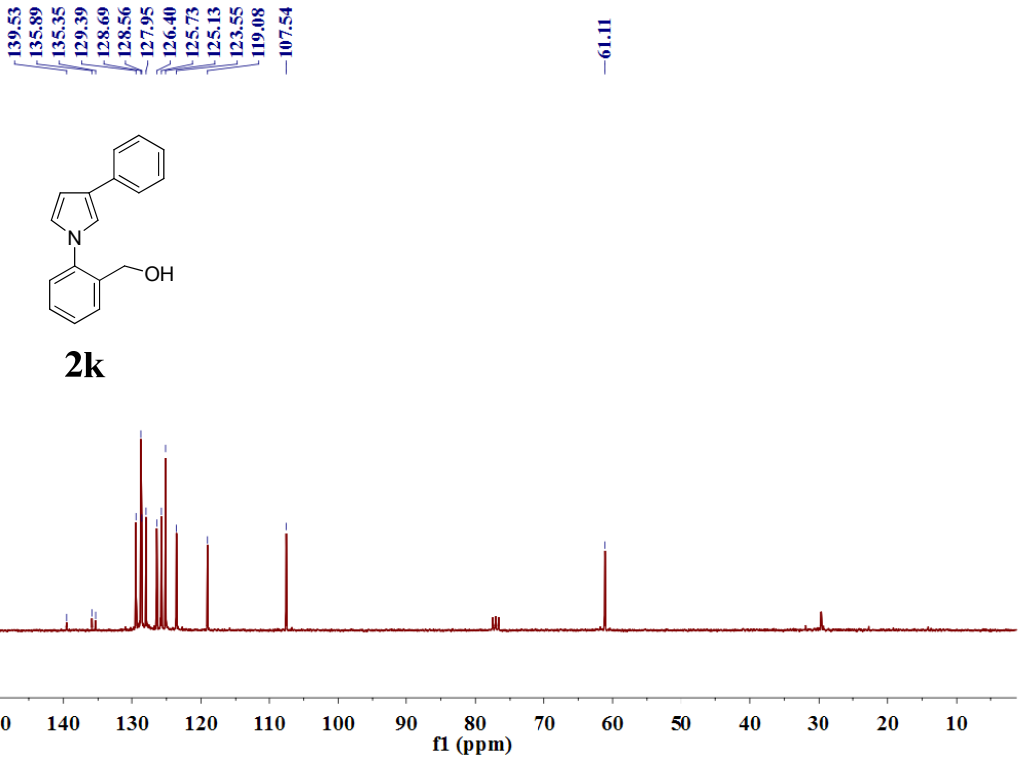

7.43
7.42
7.41
7.40
7.37
7.24
7.22
7.19
7.18
6.91
6.90
6.89
6.67
6.66
4.61
2.00
N
OH

2k

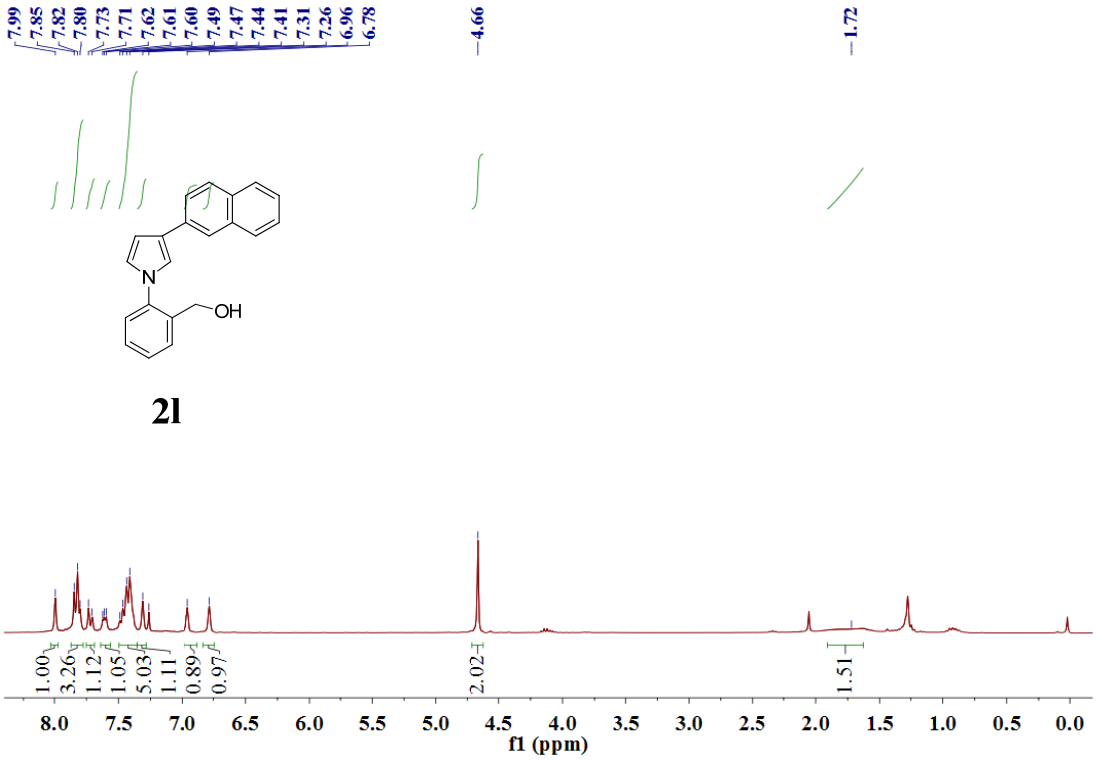

3.11
5.12
2.23
1.00
1.06
2.42
1.45
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


7.59
7.57
7.43
7.42
7.41
7.40
7.37
7.35
7.24
7.22
7.19
7.18
6.91
6.90
6.89
6.67
6.66
N
OH
3.11
5.12
2.23
1.00
1.06
7.60
7.50
7.40
7.30
7.20
7.10
7.00
6.90
6.80
6.70
6.60
f1 (ppm)

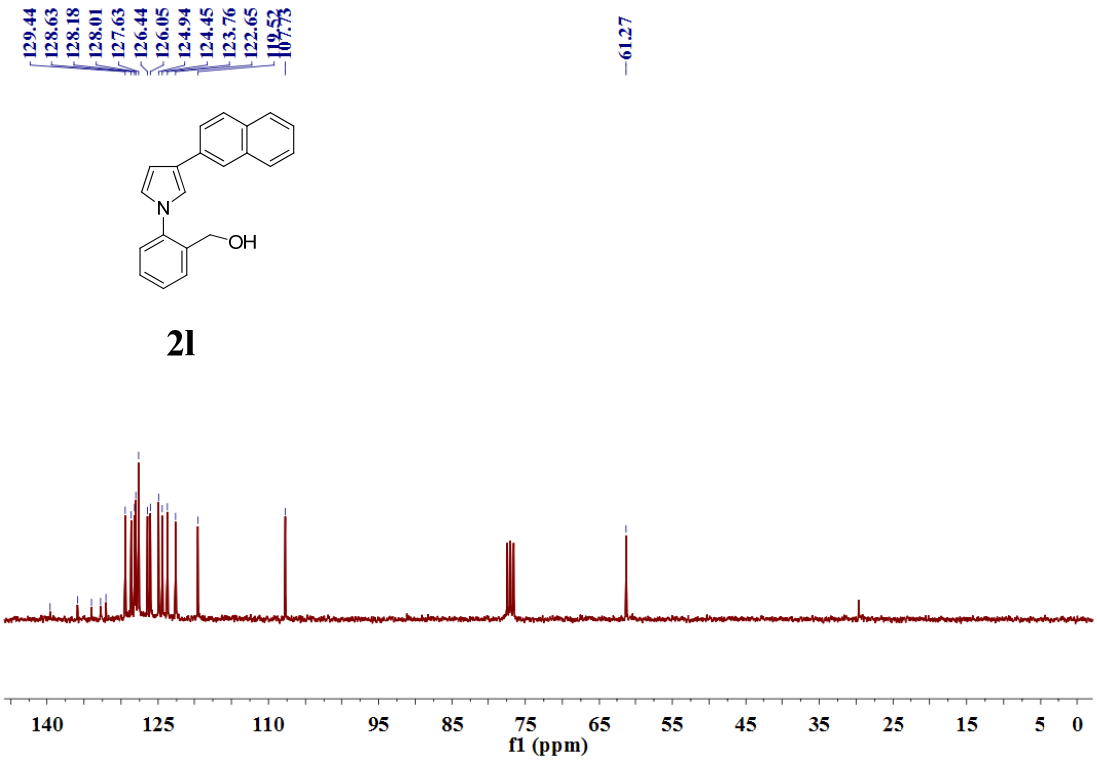
2k


139.53
135.89
135.35
129.39
128.69
128.56
127.95
126.40
125.73
125.13
123.55
119.08
107.54
61.11
N
OH
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

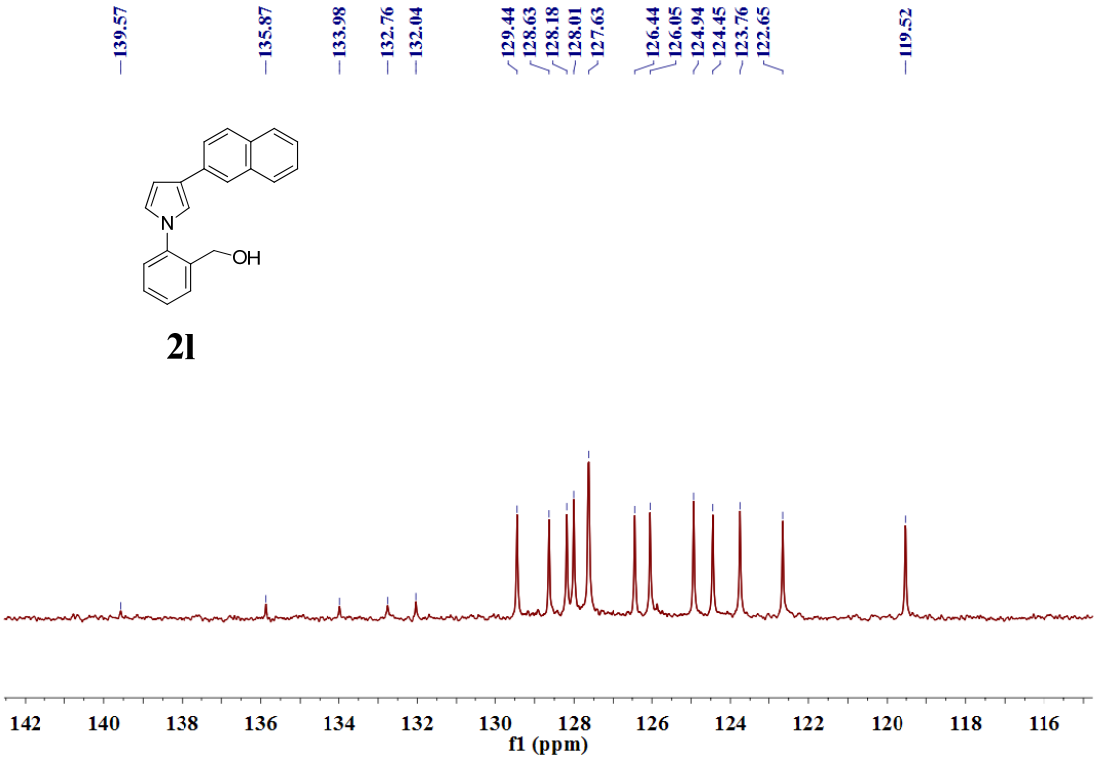
2k


7.99
7.85
7.82
7.80
7.73
7.71
7.62
7.61
7.60
7.49
7.47
7.44
7.41
7.31
7.26
6.96
6.78
4.66
1.72
N
OH
1.00
3.26
1.12
1.05
5.03
1.11
0.89
0.97
2.02
1.51
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
f1 (ppm)

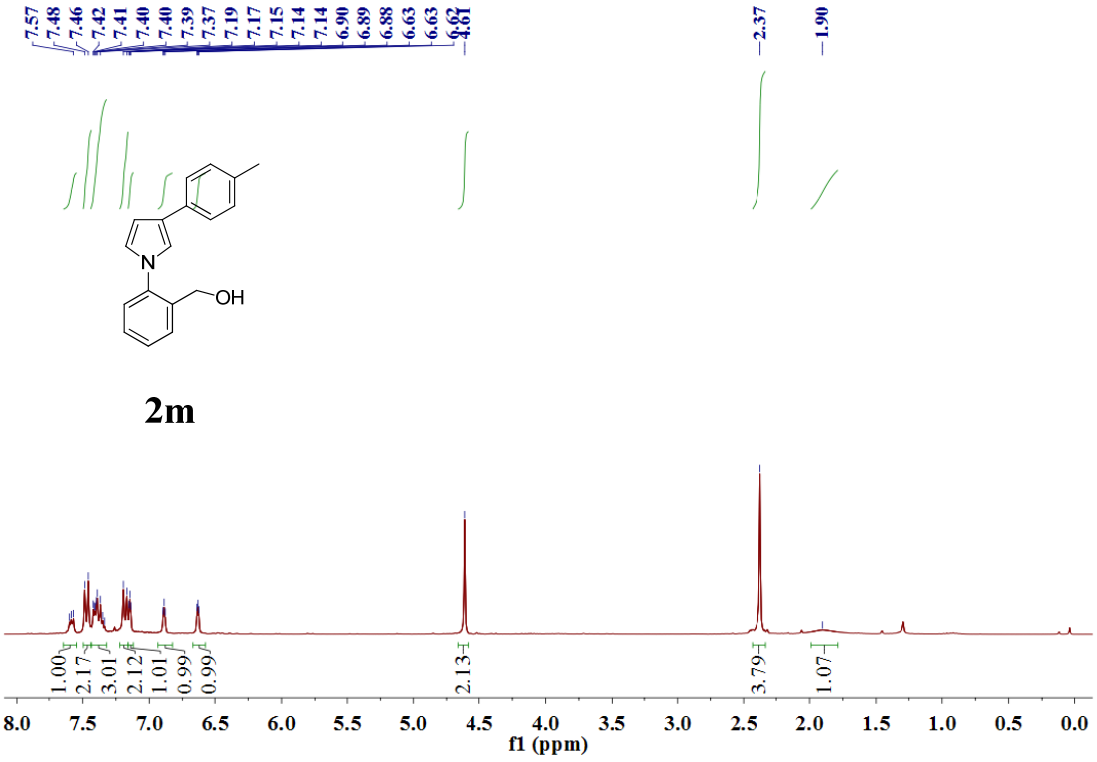
2l


129.44
128.63
128.18
128.01
127.63
126.44
126.05
124.94
124.45
123.76
122.65
119.52
107.73
61.27
N
OH
140
125
110
95
85
75
65
55
45
35
25
15
5
0
f1 (ppm)

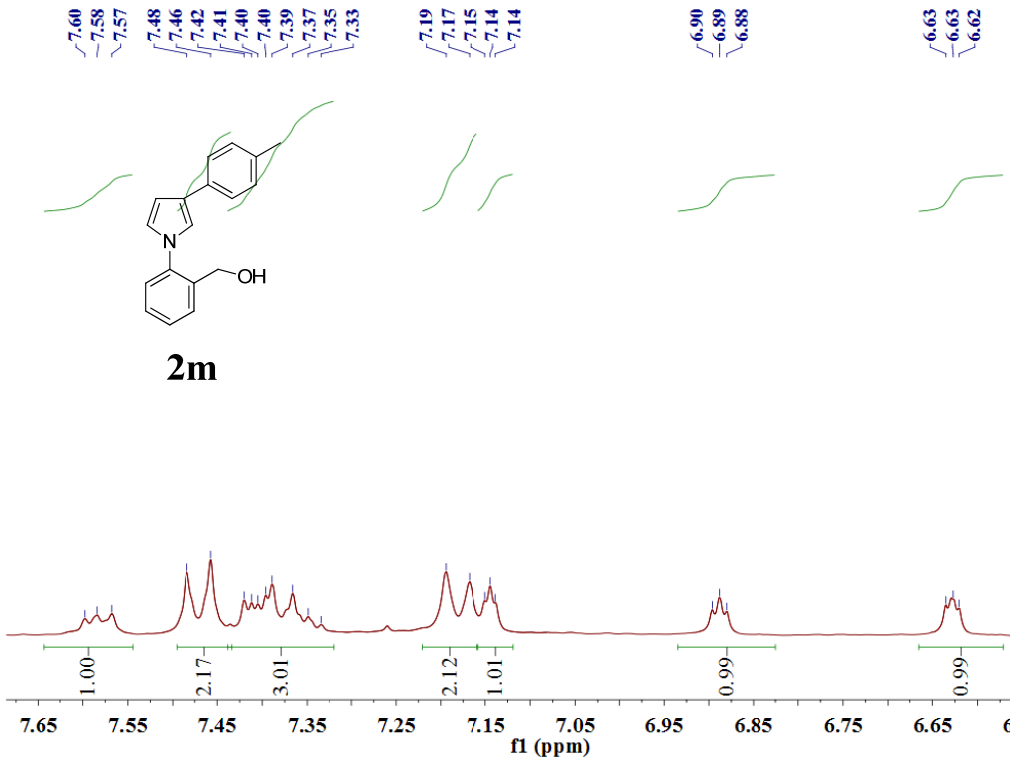
2l


139.57
135.87
133.98
132.76
132.04
129.44
128.63
128.18
128.01
127.63
126.44
126.05
124.94
124.45
123.76
122.65
119.52
N
OH
142
140
138
136
134
132
130
128
126
124
122
120
118
116
f1 (ppm)

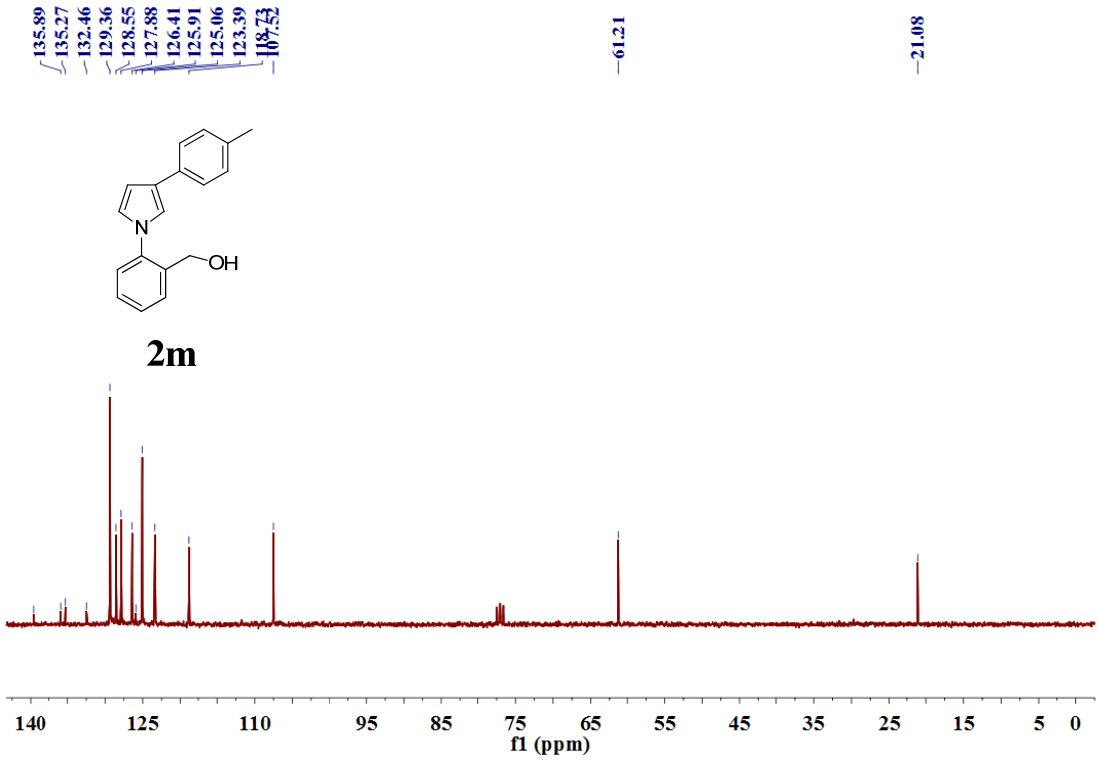
2l


7.57
7.48
7.46
7.42
7.41
7.40
7.40
7.39
7.37
7.19
7.17
7.15
7.14
7.14
6.90
6.89
6.88
6.63
6.63
6.62
2.37
1.90
N
OH
1.00
2.17
3.01
2.12
1.01
0.99
0.99
2.13
3.79
1.07
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
f1 (ppm)

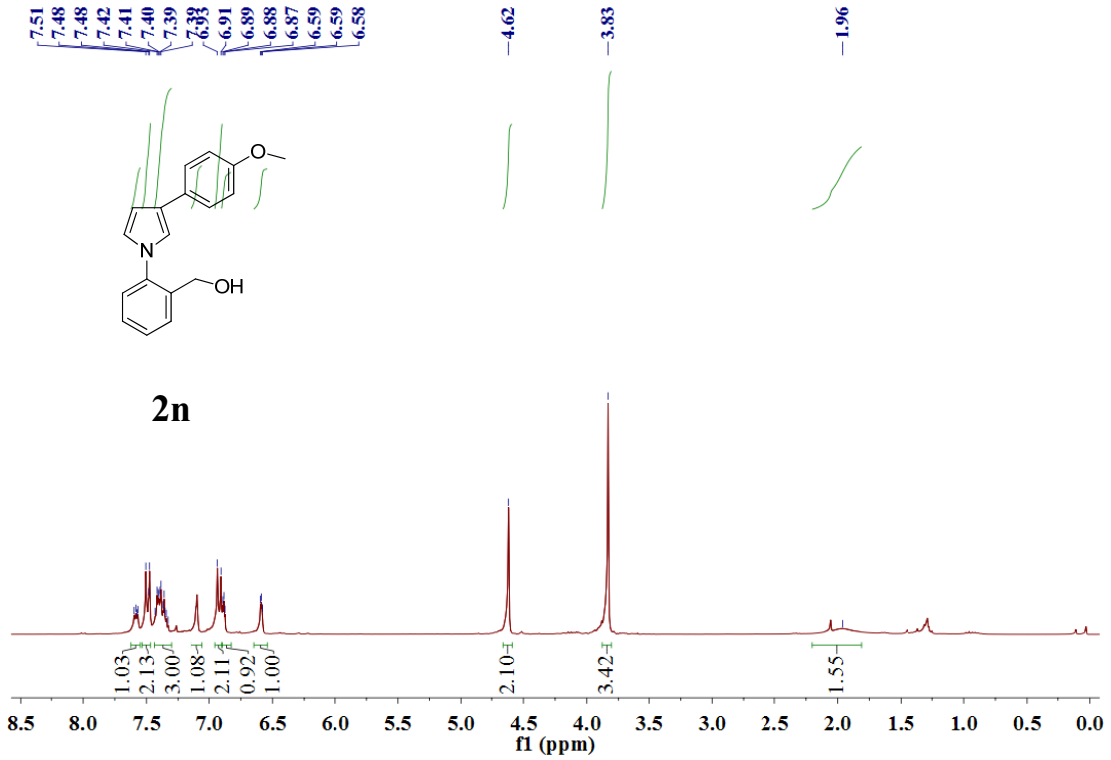
2m


7.60
7.58
7.57
7.48
7.46
7.42
7.41
7.40
7.40
7.39
7.37
7.35
7.33
7.19
7.17
7.15
7.14
7.14
6.90
6.89
6.88
6.63
6.63
6.62
N
OH
1.00
2.17
3.01
2.12
1.01
0.99
0.99
7.65
7.55
7.45
7.35
7.25
7.15
7.05
6.95
6.85
6.75
6.65
6.55
f1 (ppm)

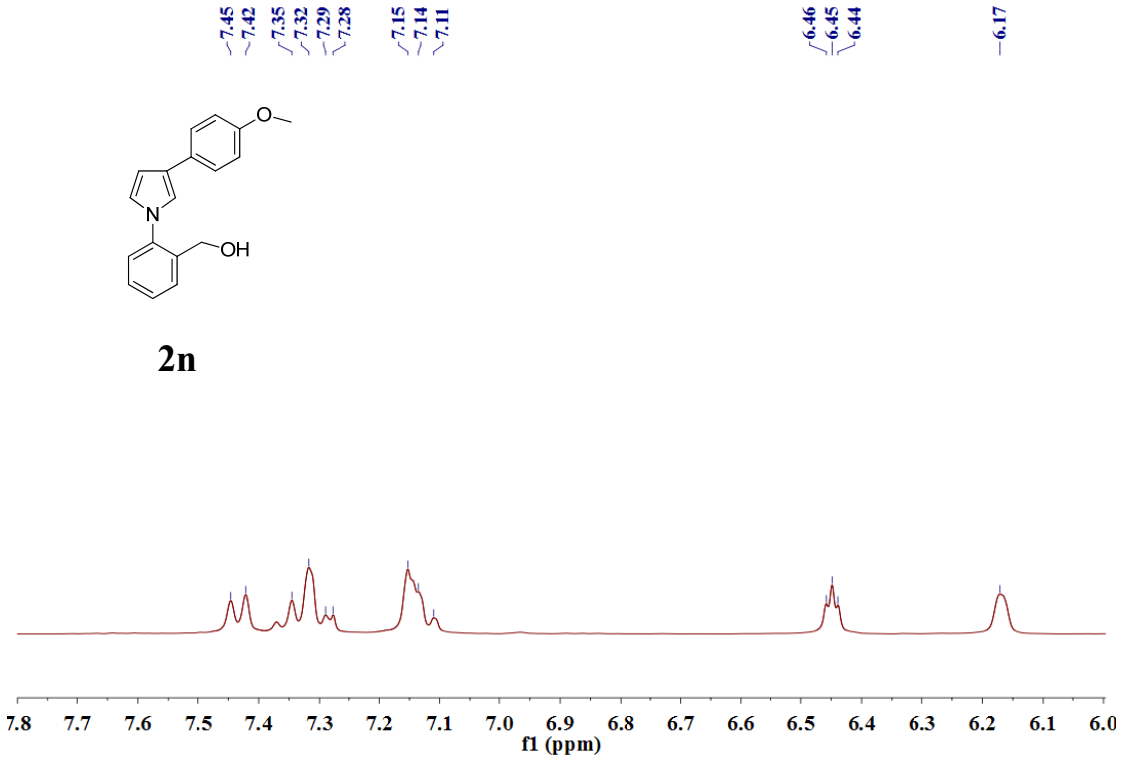
2m


135.89
135.27
132.46
129.36
128.55
127.88
126.41
125.91
125.06
123.39
61.21
21.08
N
OH
140
125
110
95
85
75
65
55
45
35
25
15
5
0
f1 (ppm)

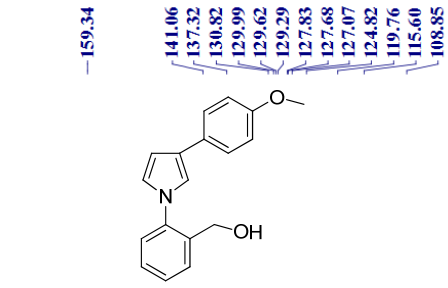
2m


7.51
7.48
7.48
7.42
7.41
7.40
7.39
6.91
6.89
6.88
6.87
6.59
6.59
6.58
4.62
3.83
1.96
O
N
OH
1.03
2.13
3.00
1.08
2.11
0.92
1.00
2.10
3.42
1.55
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
f1 (ppm)

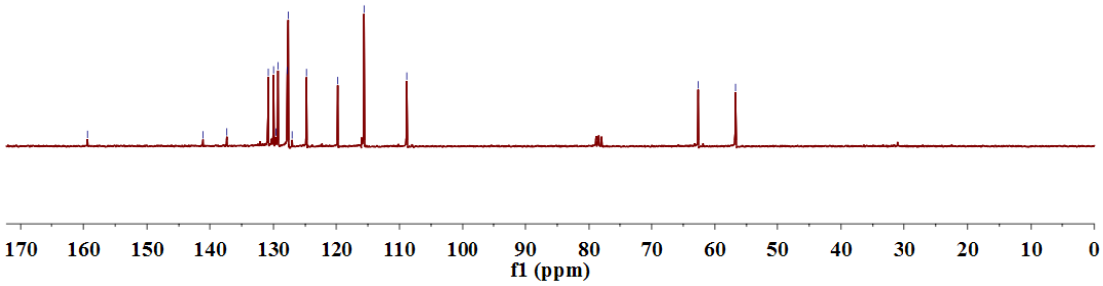
2n

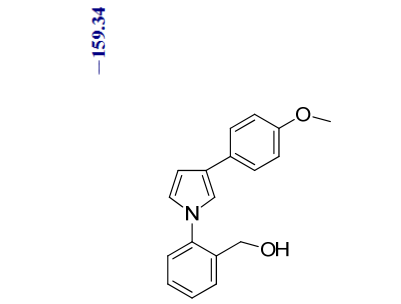

7.45
7.42
7.35
7.32
7.29
7.28
7.15
7.14
7.11
6.46
6.45
6.44
6.17
O
N
OH
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1 6.0
f1 (ppm)

2n

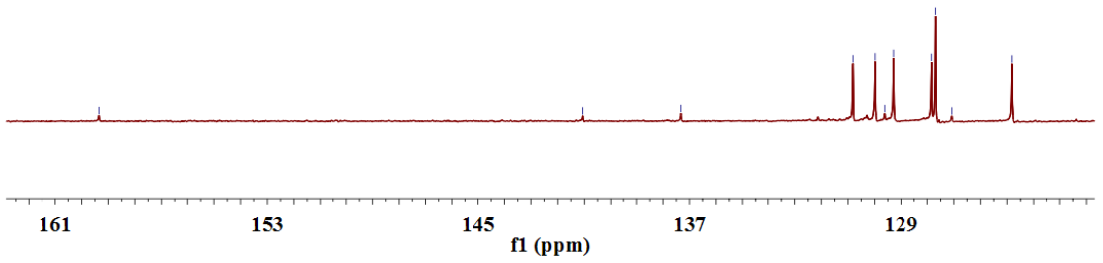

159.34
141.06
137.32
130.82
129.99
129.62
129.29
127.83
127.68
127.07
124.82
119.76
115.60
108.85
62.64
56.74
O
N
OH

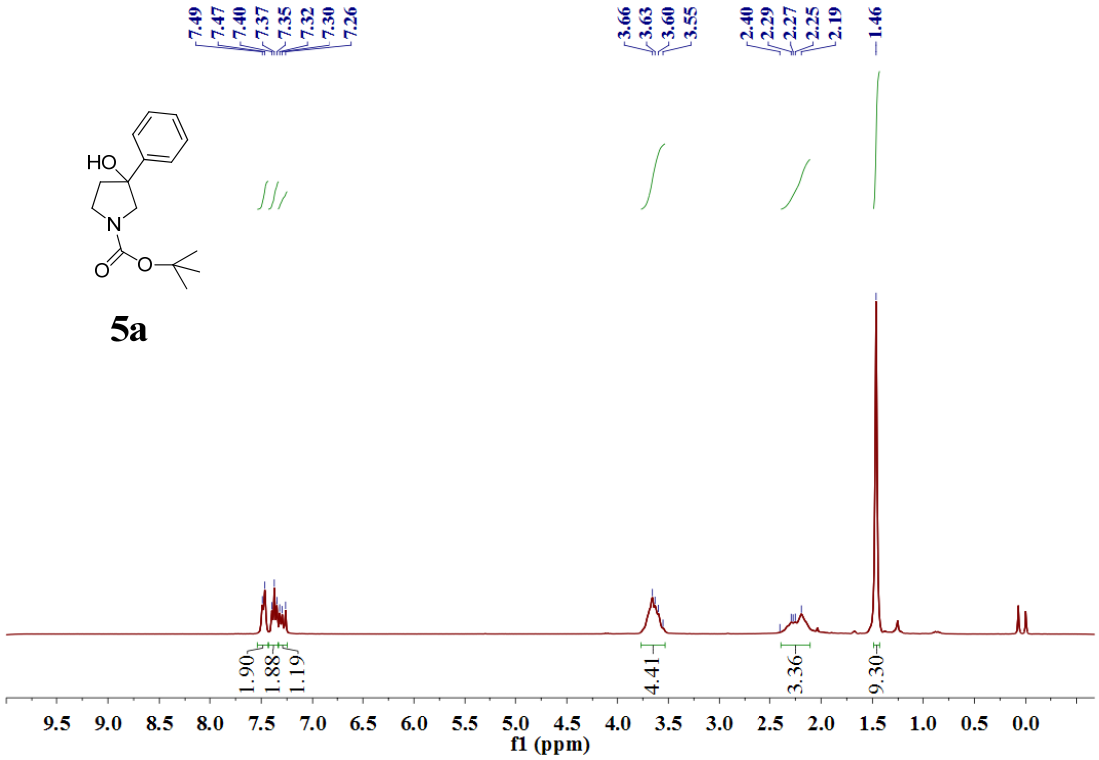
2n


170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

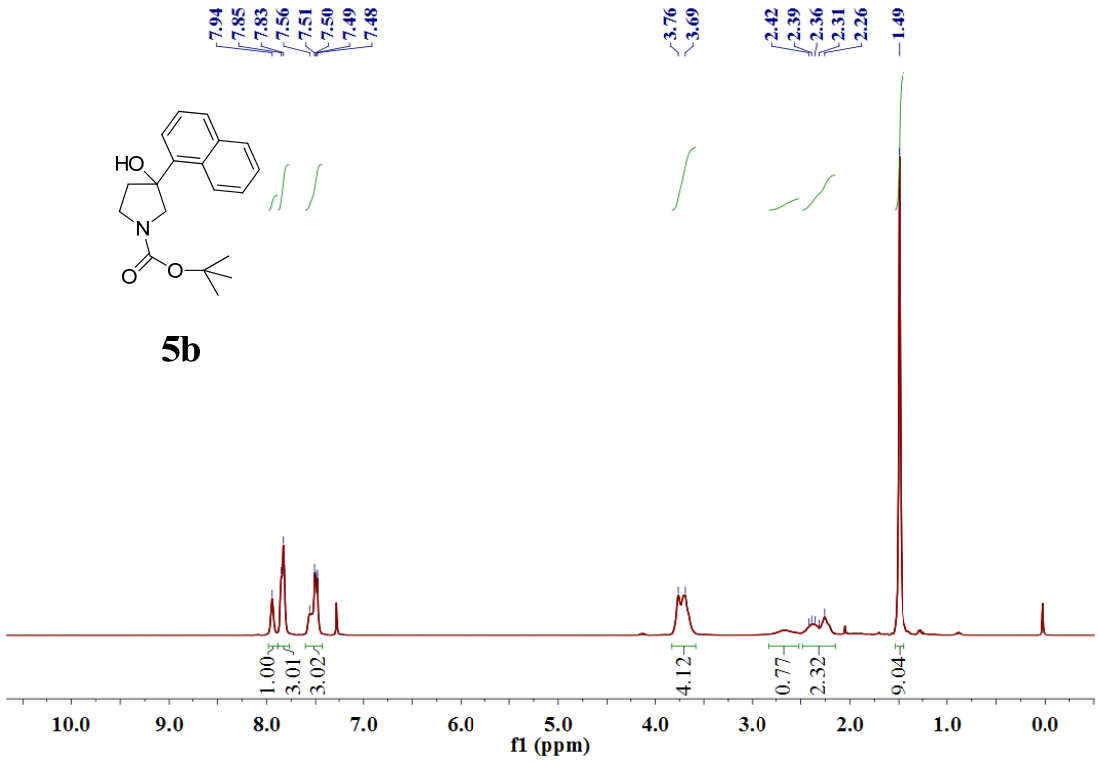

159.34
141.06
137.32
130.82
129.99
129.62
129.29
127.83
127.68
127.07
124.82
O
N
OH

2n

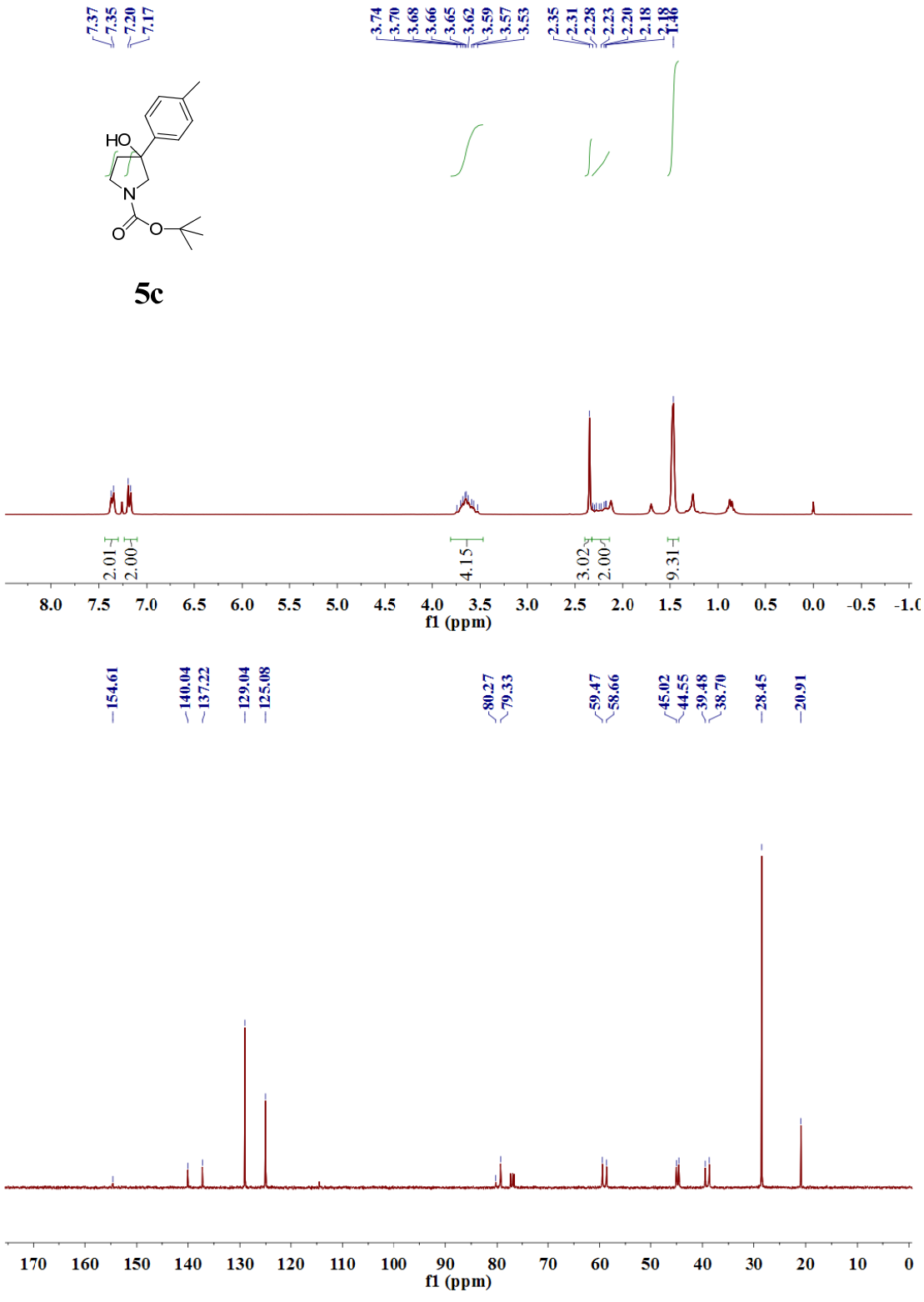

161
153
145
137
129
f1 (ppm)


7.49
7.47
7.40
7.37
7.35
7.32
7.30
7.26
3.66
3.63
3.60
3.55
2.40
2.29
2.27
2.25
2.19
1.46
HO
N
O
O
1.90
1.88
1.19
4.41
3.36
9.30
9.5 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
f1 (ppm)

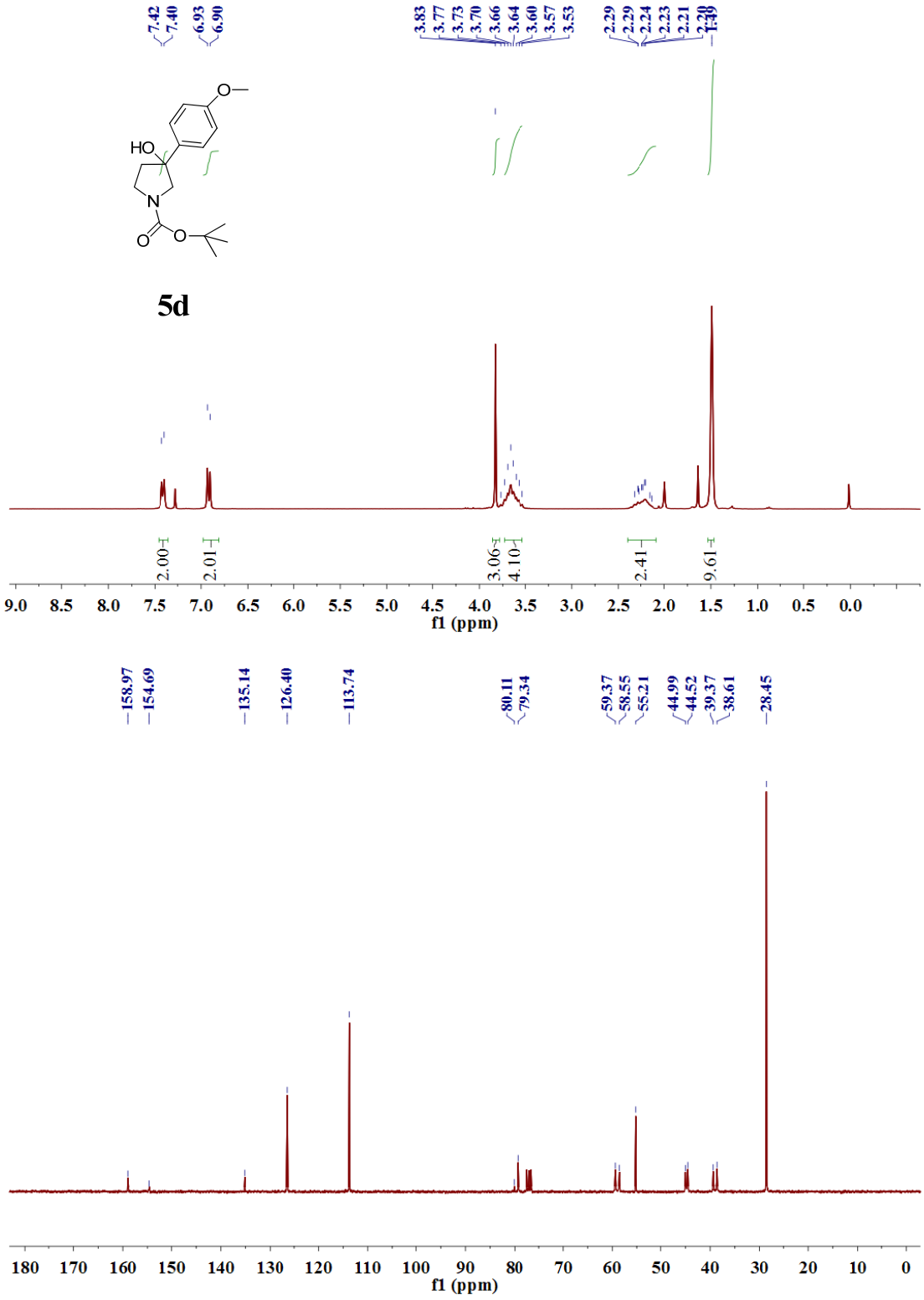
5a

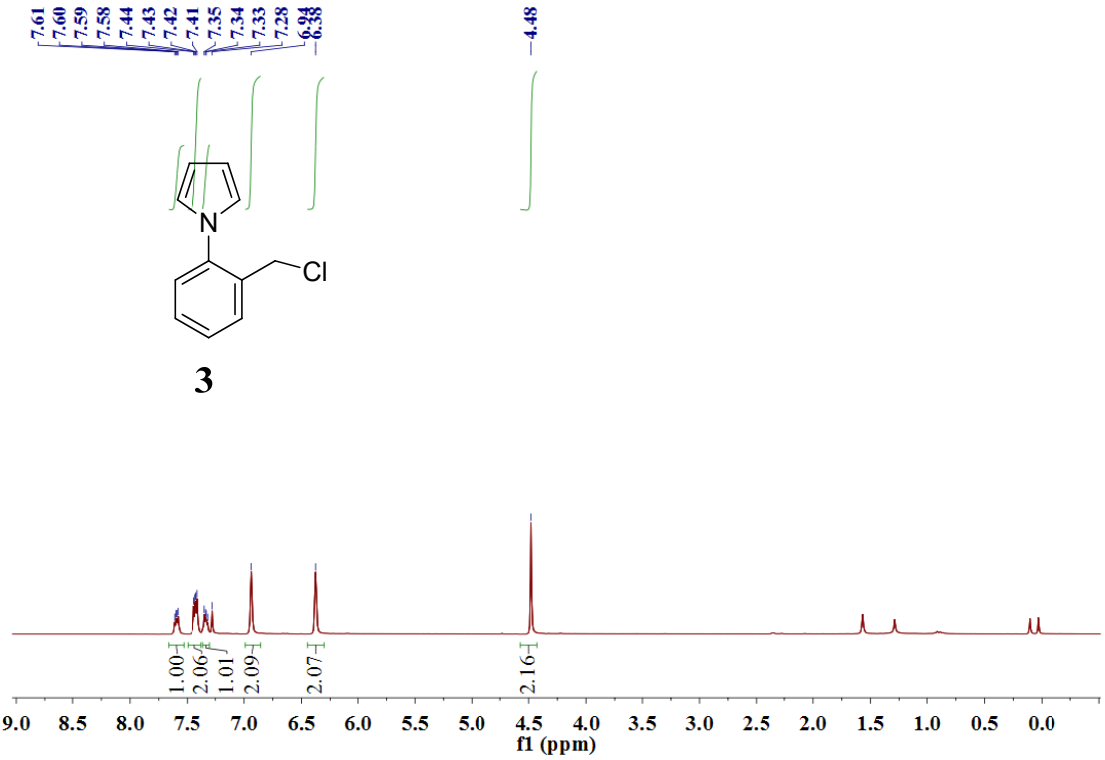

7.94
7.85
7.83
7.56
7.51
7.50
7.49
7.48
3.76
3.69
2.42
2.39
2.36
2.31
2.26
1.49
HO
N
O
O
1.00
3.01
3.02
4.12
0.77
2.32
9.04
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

5b

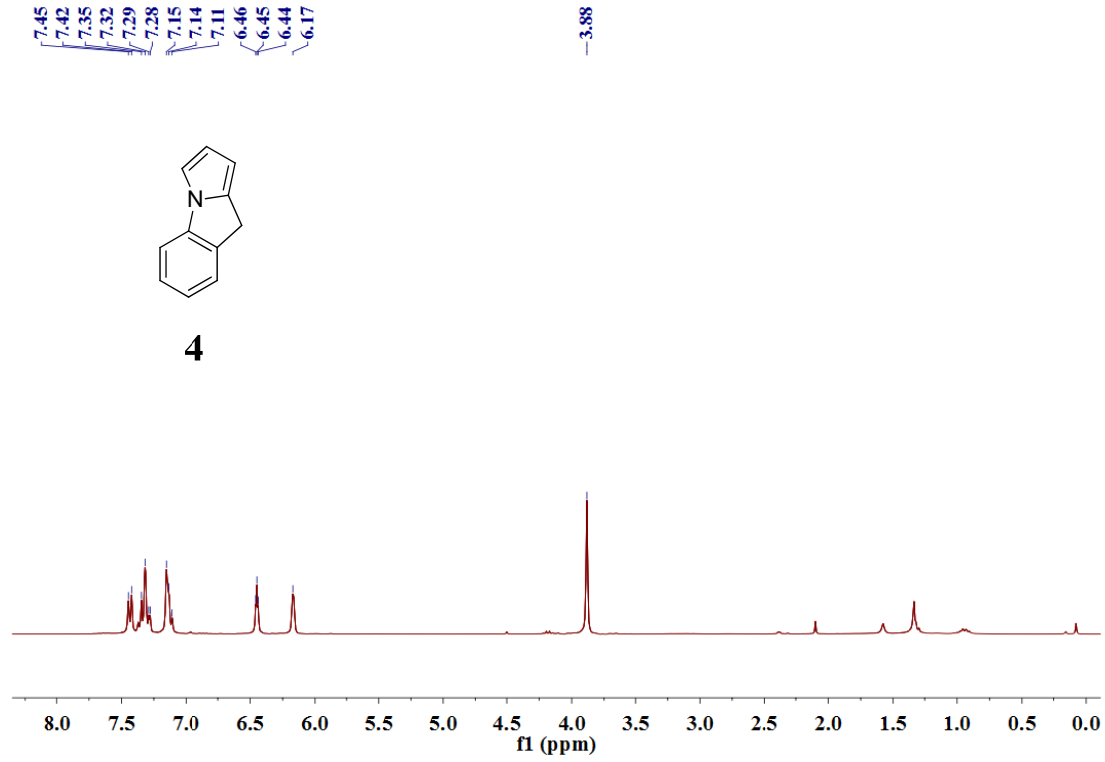

7.37
7.35
7.20
7.17
3.74
3.70
3.68
3.66
3.65
3.62
3.59
3.57
3.53
2.35
2.31
2.28
2.23
2.20
2.18
1.48
HO
N
O
O
2.01
2.00
4.15
3.02
2.00
9.31
f1 (ppm)
154.61
140.04
137.22
129.04
125.08
80.27
79.33
59.47
58.66
45.02
44.55
39.48
38.70
28.45
20.91
f1 (ppm)

5c

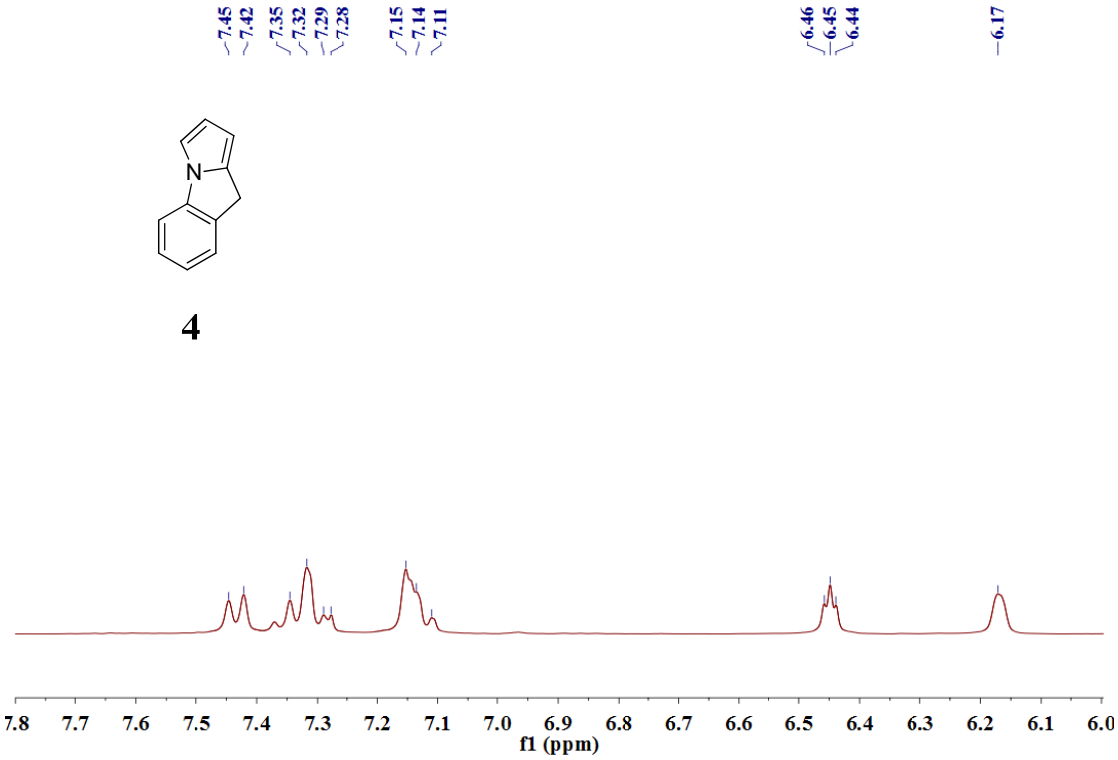

7.42
7.40
6.93
6.90
3.83
3.77
3.73
3.70
3.66
3.64
3.60
3.57
3.53
2.29
2.29
2.24
2.23
2.21
2.20
1.49
HO
O
N
O
O
5d
2.00
2.01
3.06
4.10
2.41
9.61
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
158.97
154.69
135.14
126.40
113.74
80.11
79.34
59.37
58.55
55.21
44.99
44.52
39.37
38.61
28.45
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.61
7.60
7.59
7.58
7.44
7.43
7.42
7.41
7.35
7.34
7.33
7.28
6.94
6.38
4.48
N
Cl
1.00
2.06
1.01
2.09
2.07
2.16
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
f1 (ppm)

3


7.45
7.42
7.35
7.32
7.29
7.28
7.15
7.14
7.11
6.46
6.45
6.44
6.17
3.88
N
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
f1 (ppm)

4


7.45
7.42
7.35
7.32
7.29
7.28
7.15
7.14
7.11
6.46
6.45
6.44
6.17
N
4
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1 6.0
f1 (ppm)