Supporting information

Palladium/Copper-Catalyzed Aerobic Oxidative C-H Carbonylation for the Synthesis of *o*-Aminobenzoates

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General Information

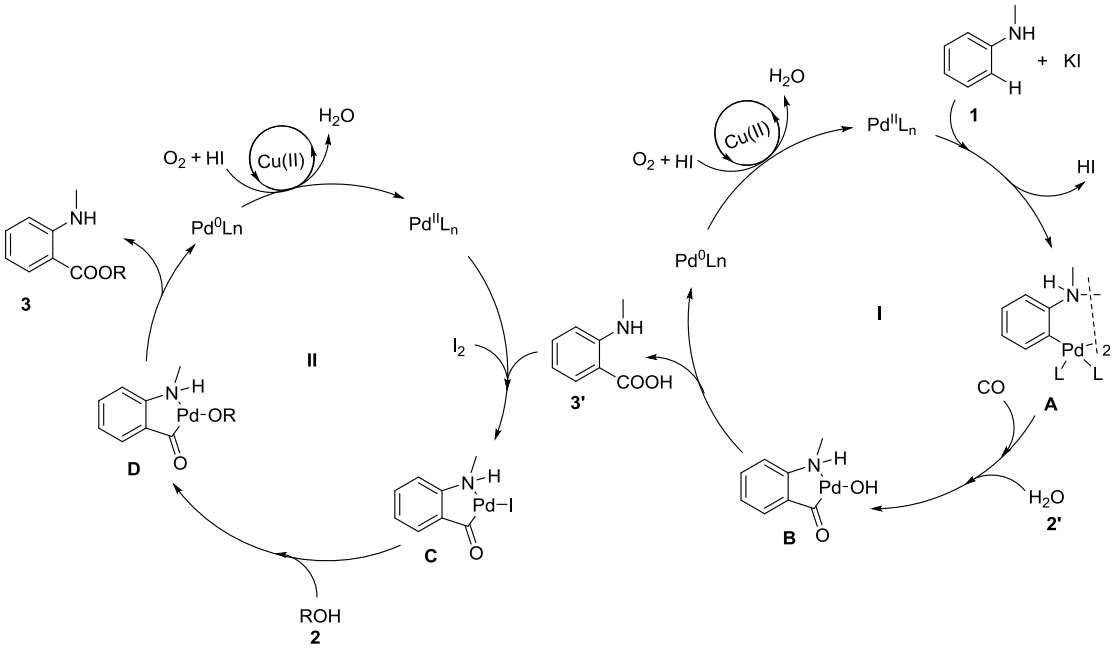
All reactions were carried out in oven-dried Schlenk tube under a carbon monoxide atmosphere with a balloon. *N*-Alkyl anilines were synthesized according to literature procedures.[1] DMSO was dried and distilled from CaH_2 under vacuum. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in hexane. All new compounds were characterized by ^{1}H NMR, ^{13}C NMR and HRMS. The known compounds were characterized by ^{1}H NMR, ^{13}C NMR. ^{1}H and ^{13}C NMR data were recorded with ADVANCE III 400 MHz with tetramethylsilane as an internal standard. High resolution mass spectra (HRMS) were measured with a Waters Micromass GCT instrument. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. All chemical shifts wererereported relative to tetramethylsilane (0 ppm for ^{1}H). All chemical shifts are reported relative to tetramethylsilane and *d*-solvent peaks (77.3 ppm, chloroform), respectively.

General Procedure for the Synthesis of *o*-Aminobenzoates:

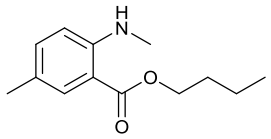
General procedure for the palladium/copper-catalyzed aerobic oxidative C-H carbonylation for the synthesis of *o*-aminobenzoates: In an oven-dried Schlenk tube equipped with a stir bar, $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), $Cu(OPiv)_2$ (31 mg, 0.12 mmol), and KI (17 mg, 0.10 mmol), a balloon filled with O_2/CO (1:10, 1 atm) was connected to the Schlenk tube by the side tube and purged three times. Then, DMSO (0.3 mL), toluene (0.3 mL) and MeCN (0.3 mL) were added to the tube through a syringe respectively, and *N*-alkyl anilienes (0.20 mmol) and ROH (0.40 mmol) were then injected into the tube by syringe. The Schlenk tube was heated at 90 °C for 12 h and then cooled to room temperature. After the balloon gas was released carefully, the reaction was quenched by saturated potassium carbonate solution and extracted with CH_2Cl_2 three times. The combined organic layer was dried over anhydrous Na_2SO_4 and evaporated in vacuum. The desired products were obtained in the corresponding yields after purification by flash chromatography on silica gel with hexane and ethyl acetate.

Proposed Mechanism:

Scheme S1. Proposed Mechanism



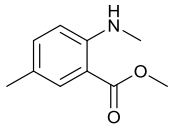
Characterization of Synthesized Compounds



3a

butyl 5-methyl-2-(methylamino)benzoate

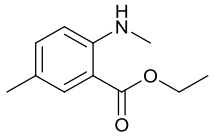
Yield: 82% (36.2 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 1.7 Hz, 1H), 7.50 (s, 1H), 7.24 (dd, *J* = 8.5, 2.0 Hz, 1H), 6.63 (d, *J* = 8.5 Hz, 1H), 4.28 (t, *J* = 6.6 Hz, 2H), 2.92 (s, 3H), 2.28 (s, 3H), 1.77 (dt, *J* = 14.6, 6.7 Hz, 2H), 1.50 (dq, *J* = 14.7, 7.4 Hz, 2H), 1.01 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8 , 150.2, 135.6, 131.2, 123.2, 110.8, 110.0, 64.0, 30.9, 29.7, 20.2, 19.4, 13.8. HRMS (ESI^+) calculated for $C_{13}H_{19}NO_2$ (M^+): 221.1416; found: 221.1418.



3b

methyl 5-methyl-2-(methylamino)benzoate

Yield: 68% (24.3 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 1.3 Hz, 1H), 7.56 (s, 1H), 7.31 (d, *J* = 1.9 Hz, 1H), 6.81 (d, *J* = 8.5 Hz, 1H), 3.90 (s, 3H), 2.96 (s, 3H), 2.30 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8, 148.0, 135.8, 131.5, 126.1, 113.1, 111.4, 51.7, 31.1, 20.3. HRMS (ESI^+) calculated for $C_{10}H_{13}NO_2$ (M^+): 179.0946; found: 179.0945.

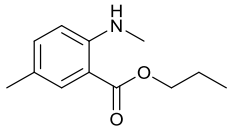


3c

ethyl 5-methyl-2-(methylamino)benzoate

Yield: 69% (26.6 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 1.2 Hz, 1H), 7.51 (s, 1H), 7.24 (dd, *J* = 8.5, 1.9 Hz, 1H), 6.63 (d, *J* = 8.5 Hz, 1H), 4.34 (q, *J* = 7.1 Hz, 2H), 2.92 (s, 3H), 2.27 (s, 3H), 1.41 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 150.1, 135.6, 131.3, 123.3,

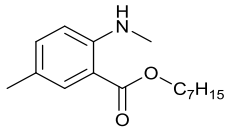
110.9, 110.0, 60.2, 29.8, 20.2, 14.4. HRMS (ESI$^+$) calculated for $C_{11}H_{15}NO_2$ (M^+): 193.1103; found: 193.1104.



3d

propyl 5-methyl-2-(methylamino)benzoate

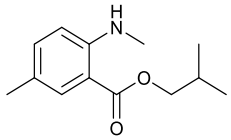
Yield: 67% (27.7 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 1.8 Hz, 1H), 7.50 (s, 1H), 7.24 (dd, *J* = 8.5, 1.9 Hz, 1H), 6.63 (d, *J* = 8.5 Hz, 1H), 4.24 (t, *J* = 6.7 Hz, 2H), 2.92 (s, 3H), 2.27 (s, 3H), 1.87 – 1.75 (m, 2H), 1.06 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8, 150.1, 143.5, 135.6, 131.2, 123.2, 110.9, 110.0, 65.8, 29.8, 22.2, 10.7. HRMS (ESI$^+$) calculated for $C_{12}H_{17}NO_2$ (M^+): 207.1259; found: 207.1256.



3e

heptyl 5-methyl-2-(methylamino)benzoate

Yield: 73% (38.4 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 1.7 Hz, 1H), 7.50 (s, 1H), 7.24 (dd, *J* = 8.5, 2.0 Hz, 1H), 6.63 (d, *J* = 8.5 Hz, 1H), 4.27 (t, *J* = 6.7 Hz, 2H), 2.92 (s, 3H), 2.28 (s, 3H), 1.84 – 1.73 (m, 2H), 1.41 (dddd, *J* = 18.6, 12.4, 7.3, 3.3 Hz, 8H), 0.93 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8, 150.1, 135.6, 131.3, 123.2, 110.9, 110.0, 64.4, 31.8, 29.8, 29.0, 28.8, 26.1, 22.6, 20.2, 14.1. HRMS (ESI$^+$) calculated for $C_{16}H_{25}NO_2$ (M^+): 263.1885; found: 263.1883.

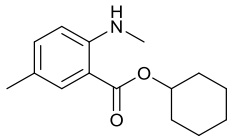


3f

isobutyl 5-methyl-2-(methylamino)benzoate

Yield: 76% (33.6 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 1.4 Hz, 1H), 7.51 (s, 1H), 7.25 (dd, *J* = 8.5, 2.0 Hz, 1H), 6.63 (d, *J* = 8.5 Hz, 1H), 4.07 (d, *J* = 6.6 Hz, 2H), 2.93 (s, 3H),

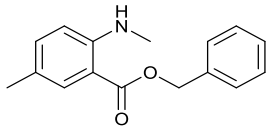
2.29 (s, 3H), 2.11 (dt, *J* = 13.4, 6.7 Hz, 1H), 1.06 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 150.2, 135.6, 131.2, 123.2, 110.9, 109.9, 70.3, 29.8, 27.9, 20.3, 19.3. HRMS (ESI^+) calculated for $C_{13}H_{19}NO_2$ (M^+): 221.1416; found: 221.1421.



3g

cyclohexyl 5-methyl-2-(methylamino)benzoate

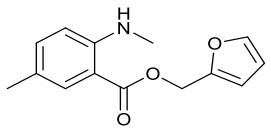
Yield: 60% (29.6 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 1.8 Hz, 1H), 7.53 (s, 1H), 7.24 (dd, *J* = 8.5, 2.0 Hz, 1H), 6.63 (d, *J* = 8.5 Hz, 1H), 5.06 – 4.89 (m, 1H), 2.92 (s, 3H), 2.28 (s, 3H), 2.02 – 1.92 (m, 2H), 1.83 (dd, *J* = 9.6, 3.3 Hz, 2H), 1.67 – 1.56 (m, 3H), 1.53 – 1.34 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.2, 150.2, 135.5, 131.3, 123.2, 110.8, 110.4, 72.3, 31.8, 29.8, 25.6, 23.8, 20.3. HRMS (ESI^+) calculated for $C_{15}H_{21}NO_2$ (M^+): 247.1572; found: 247.1574.



3h

benzyl 5-methyl-2-(methylamino)benzoate

Yield: 85% (43.4 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 1.7 Hz, 1H), 7.53 (s, 1H), 7.51 – 7.41 (m, 4H), 7.41 – 7.36 (m, 1H), 7.27 (dd, *J* = 8.6, 2.1 Hz, 1H), 6.65 (d, *J* = 8.6 Hz, 1H), 5.36 (s, 2H), 2.94 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.4, 150.3, 136.5, 135.9, 131.4, 128.6, 128.1, 128.0, 123.3, 110.9, 109.5, 65.9, 29.8, 20.2. HRMS (ESI^+) calculated for $C_{16}H_{17}NO_2$ (M^+): 255.1259; found: 255.1263.

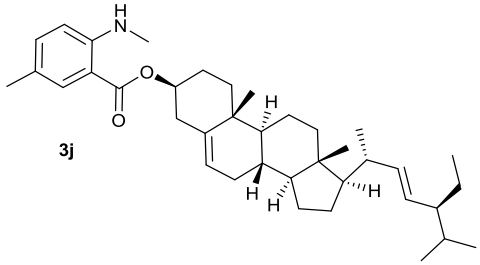


3i

furan-2-ylmethyl 5-methyl-2-(methylamino)benzoate

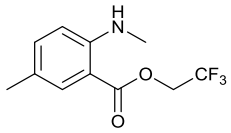
Yield: 88% (43.1 mg), white solid, mp. 47-50 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 1.6 Hz, 1H), 7.48 (d, *J* = 1.0 Hz, 2H), 7.24 (dd, *J* = 8.5, 2.0 Hz, 1H), 6.62 (d, *J* = 8.6 Hz, 1H), 6.50 (d, *J* = 3.2

Hz, 1H), 6.42 (dd, *J* = 3.1, 1.9 Hz, 1H), 5.28 (s, 2H), 2.93 (s, 3H), 2.25 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.2, 150.3, 150.0, 143.2, 136.0, 131.4, 123.3, 110.8, 110.6, 110.5, 109.3, 57.7, 29.7, 20.2. HRMS (ESI$^+$) calculated for $C_{14}H_{15}NO_3$ (M^+): 245.1052; found: 245.1053.



3j

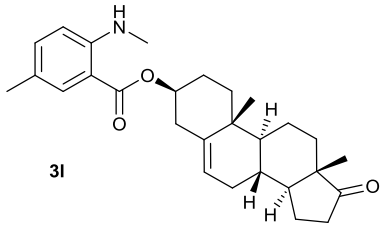
Yield: 55% (61.5 mg), white solid, mp. 169-170 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 1.5 Hz, 1H), 7.52 (s, 1H), 7.23 (dd, *J* = 8.5, 2.0 Hz, 1H), 6.62 (d, *J* = 8.5 Hz, 1H), 5.45 (d, *J* = 4.6 Hz, 1H), 5.20 (dd, *J* = 15.1, 8.6 Hz, 1H), 5.06 (dd, *J* = 15.1, 8.6 Hz, 1H), 4.89 – 4.73 (m, 1H), 2.92 (s, 3H), 2.48 (d, *J* = 7.0 Hz, 2H), 2.28 (s, 3H), 2.13 – 1.90 (m, 5H), 1.84 – 1.69 (m, 2H), 1.64 – 1.45 (m, 8H), 1.34 – 1.15 (m, 6H), 1.14 – 1.02 (m, 9H), 0.93 – 0.79 (m, 10H), 0.74 (d, *J* = 4.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.1, 150.2, 139.9, 138.3, 135.5, 131.3, 129.3, 123.2, 122.6, 110.8, 110.3, 73.8, 56.8, 56.0, 51.3, 50.1, 42.2, 40.5, 39.7, 38.4, 37.1, 36.7, 32.0, 31.9, 29.7, 28.9, 28.0, 25.4, 24.4, 21.3, 21.1, 21.1, 20.2, 19.4, 19.0, 12.3, 12.1. HRMS (ESI$^+$) calculated for $C_{38}H_{57}NO_2$ $[M^+H]^+$: 560.4468; found: 560.4450.



3k

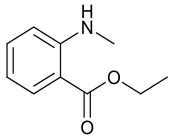
2,2,2-trifluoroethyl 5-methyl-2-(methylamino)benzoate

Yield: 77% (38.0 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 1.5 Hz, 1H), 7.36 (s, 1H), 7.31 – 7.26 (m, 1H), 6.65 (d, *J* = 8.6 Hz, 1H), 4.67 (q, *J* = 8.5 Hz, 2H), 2.94 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 150.7, 136.8, 131.3, 123.6, 111.0, 107.6, 60.5 (q, *J* = 36.3 Hz), 29.7, 20.2. ^{19}F NMR (377 MHz, $CDCl_3$) δ -73.56. HRMS (ESI$^+$) calculated for $C_{11}H_{12}F_3NO_2$ (M^+): 247.0820; found: 247.0810.



3l

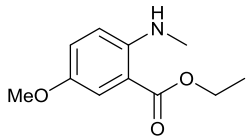
Yield: 57% (49.6 mg), white solid, mp. 189-191 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 1.6 Hz, 1H), 7.51 (s, 1H), 7.23 (dd, *J* = 8.5, 2.0 Hz, 1H), 6.62 (d, *J* = 8.5 Hz, 1H), 5.48 (d, *J* = 4.7 Hz, 1H), 4.87 – 4.76 (m, 1H), 2.92 (s, 3H), 2.56 – 2.43 (m, 3H), 2.28 (s, 3H), 2.22 – 2.07 (m, 2H), 1.95 (dddd, *J* = 16.4, 12.7, 9.3, 4.9 Hz, 4H), 1.76 (ddd, *J* = 16.2, 14.4, 6.4 Hz, 4H), 1.61 – 1.52 (m, 2H), 1.40 – 1.32 (m, 2H), 1.24 (dd, *J* = 13.5, 3.1 Hz, 1H), 1.16 – 1.06 (m, 4H), 0.96 – 0.90 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.1, 150.2, 140.2, 135.6, 131.2, 123.2, 121.8, 110.9, 110.1, 73.5, 51.7, 50.2, 47.6, 38.3, 37.1, 36.9, 35.9, 31.5, 31.4, 30.8, 29.7, 27.9, 21.9, 20.4, 20.2, 19.5, 13.6. HRMS (ESI$^+$) calculated for $C_{28}H_{37}NO_3$ $[M^+H]^+$: 436.2852; found: 436.2838.



3m

ethyl 2-(methylamino)benzoate

Yield: 72% (25.8 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.70 (s, 1H), 7.41 (ddd, *J* = 8.6, 7.1, 1.7 Hz, 1H), 6.70 (d, *J* = 8.5 Hz, 1H), 6.65 – 6.59 (m, 1H), 4.34 (q, *J* = 7.1 Hz, 2H), 2.94 (s, 3H), 1.41 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 152.0, 134.5, 131.5, 114.3, 110.7, 110.1, 60.2, 29.6, 14.4.

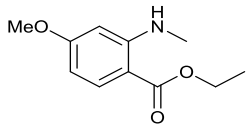


3n

ethyl 5-methoxy-2-(methylamino)benzoate

Yield: 63% (26.3 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, *J* = 3.1 Hz, 1H), 7.32 (s, 1H), 7.09 (dd, *J* = 9.1, 3.1 Hz, 1H), 6.67 (d, *J* = 9.1 Hz, 1H), 4.35 (q, *J* = 7.1 Hz, 2H), 3.80 (s, 3H), 2.92 (s, 3H), 1.41 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.3, 149.1, 147.4, 123.1, 114.7,

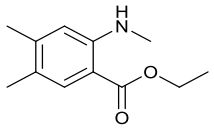
112.1, 110.0, 60.3, 56.1, 30.0, 14.4. HRMS (ESI$^+$) calculated for $C_{11}H_{15}NO_3$ (M^+): 209.1052; found: 209.1047.



3o

ethyl 4-methoxy-2-(methylamino)benzoate

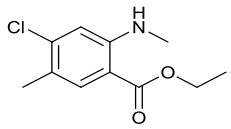
Yield: 37% (15.5 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.9 Hz, 1H), 7.81 (s, 1H), 6.20 (dd, *J* = 8.9, 2.4 Hz, 1H), 6.11 (d, *J* = 2.3 Hz, 1H), 4.31 (q, *J* = 7.1 Hz, 2H), 3.87 (s, 3H), 2.92 (d, *J* = 4.8 Hz, 3H), 1.39 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.4, 164.8, 154.0, 133.5, 103.9, 101.5, 94.5, 59.9, 55.1, 29.5, 14.4. HRMS (ESI$^+$) calculated for $C_{11}H_{15}NO_3$ (M^+): 209.1052; found: 209.1056.



3p

ethyl 4,5-dimethyl-2-(methylamino)benzoate

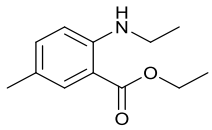
Yield: 53% (21.9 mg), white solid, mp. 50-52 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (s, 1H), 7.46 (s, 1H), 6.51 (s, 1H), 4.33 (q, *J* = 7.1 Hz, 2H), 2.92 (s, 3H), 2.29 (s, 3H), 2.19 (s, 3H), 1.40 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 150.5, 144.3, 131.7, 122.5, 111.9, 107.8, 60.0, 29.8, 20.7, 18.6, 14.5. HRMS (ESI$^+$) calculated for $C_{12}H_{17}NO_2$ (M^+): 207.1259; found: 207.1266.



3q

ethyl 4-chloro-5-methyl-2-(methylamino)benzoate

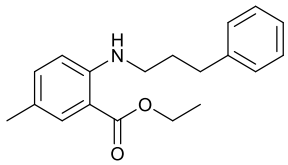
Yield: 52% (23.6 mg), white solid, mp. 44-46 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (s, 1H), 7.53 (s, 1H), 6.69 (s, 1H), 4.33 (q, *J* = 7.1 Hz, 2H), 2.90 (s, 3H), 2.28 (s, 3H), 1.41 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.1, 150.9, 141.1, 133.2, 121.4, 111.0, 108.9, 60.4, 29.7, 18.8, 14.4. HRMS (ESI$^+$) calculated for $C_{11}H_{14}ClNO_2$ (M^+): 227.0713; found: 227.0714.



3r

ethyl 2-(ethylamino)-5-methylbenzoate

Yield: 58% (24.0 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 1.5 Hz, 1H), 7.45 (s, 1H), 7.21 (dd, *J* = 8.5, 1.9 Hz, 1H), 6.64 (d, *J* = 8.6 Hz, 1H), 4.34 (q, *J* = 7.1 Hz, 2H), 3.24 (dd, *J* = 7.0, 4.4 Hz, 2H), 2.27 (s, 3H), 1.42 (t, *J* = 7.1 Hz, 3H), 1.34 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 149.3, 135.6, 131.3, 123.1, 111.3, 109.7, 60.1, 37.5, 20.2, 14.6, 14.4. HRMS (ESI^+) calculated for $C_{12}H_{17}NO_2$ (M^+): 207.1259; found: 207.1264.

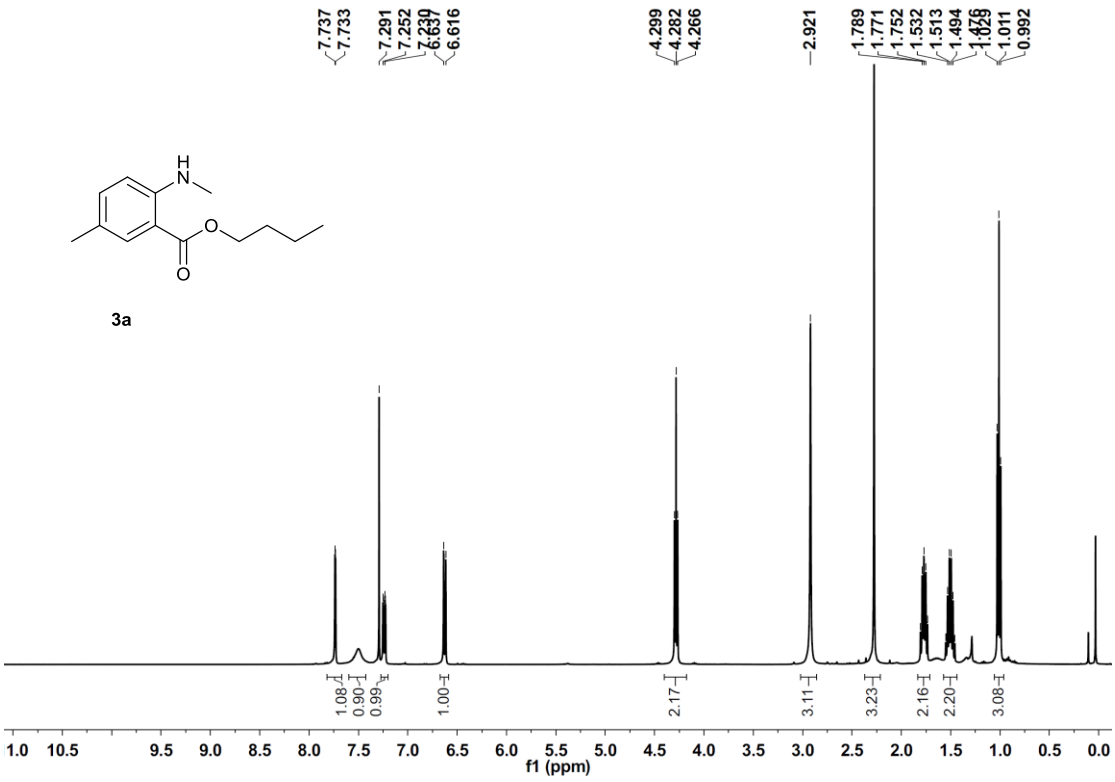


3s

ethyl 5-methyl-2-((3-phenylpropyl)amino)benzoate

Yield: 61% (36.2 mg), colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 1.8 Hz, 1H), 7.65 (s, 1H), 7.37 – 7.31 (m, 2H), 7.28 – 7.18 (m, 4H), 6.62 (d, *J* = 8.6 Hz, 1H), 4.37 (q, *J* = 7.1 Hz, 2H), 3.24 (dd, *J* = 12.2, 6.8 Hz, 2H), 2.86 – 2.74 (m, 2H), 2.29 (s, 3H), 2.12 – 1.99 (m, 2H), 1.44 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8, 149.3, 141.6, 135.6, 131.4, 128.5, 128.4, 125.9, 123.2, 111.4, 109.9, 60.2, 42.2, 33.3, 30.8, 20.2, 14.5. HRMS (ESI^+) calculated for $C_{19}H_{23}NO_2$ (M^+): 297.1729; found: 297.1728.

NMR Spectra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1.771
1.752
1.532
1.513
1.494
1.011
0.992
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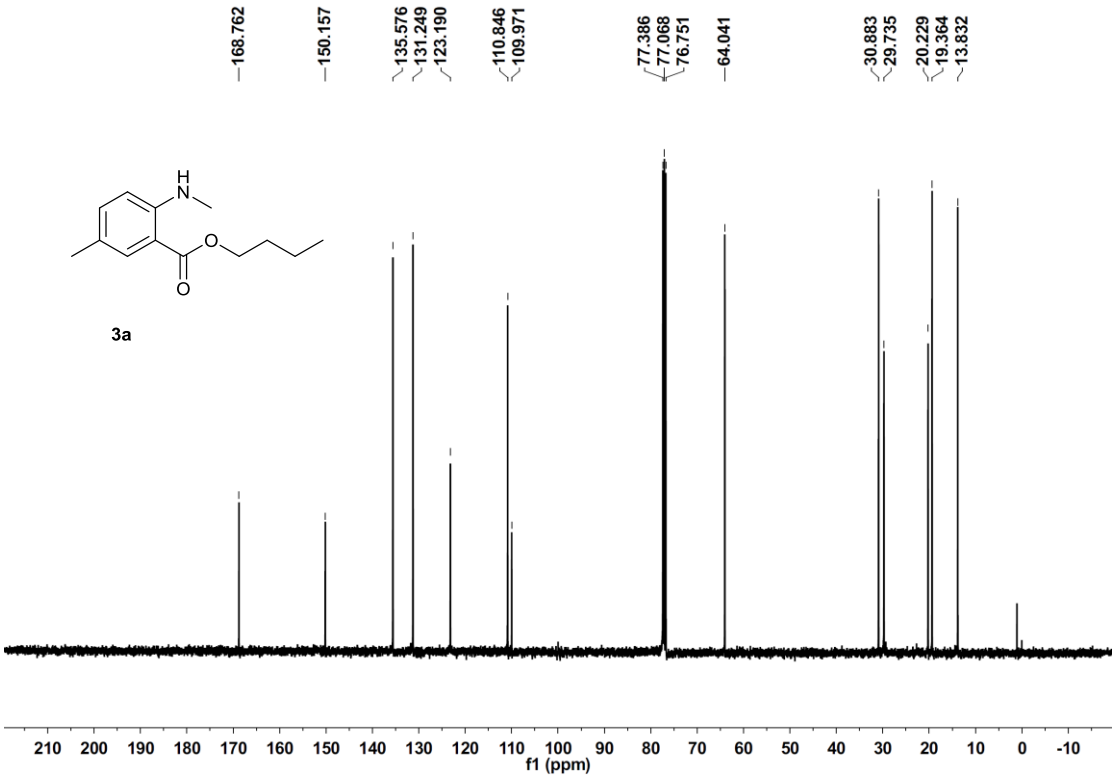
1.08
0.90
0.99
1.00
2.17
3.11
3.23
2.16
2.20
3.08
f1 (ppm)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19.364
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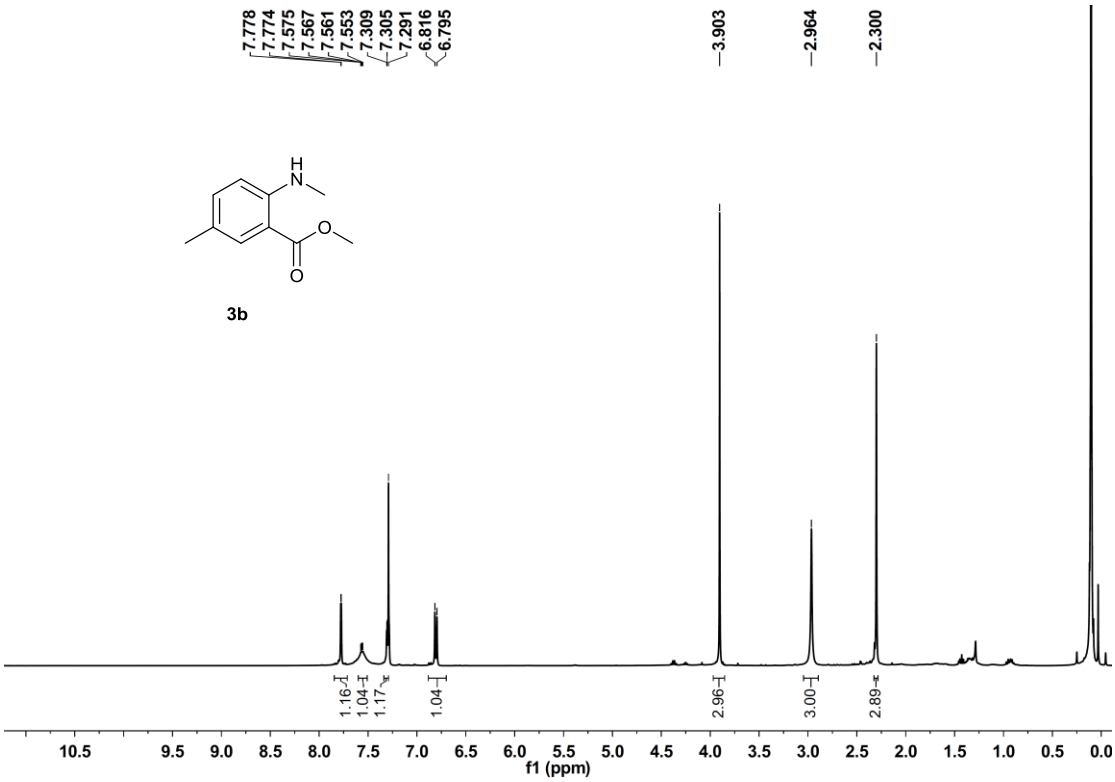
f1 (ppm)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7.305
7.291
6.816
6.795
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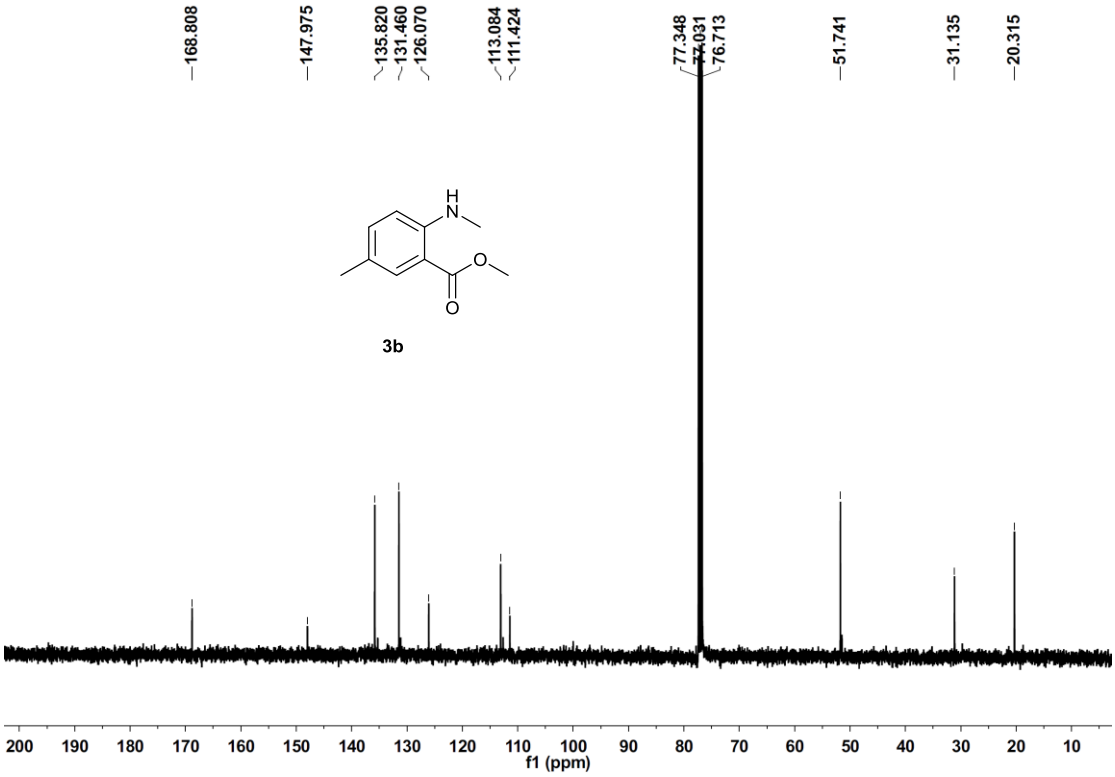
1.04
2.96
3.00
2.89
f1 (ppm)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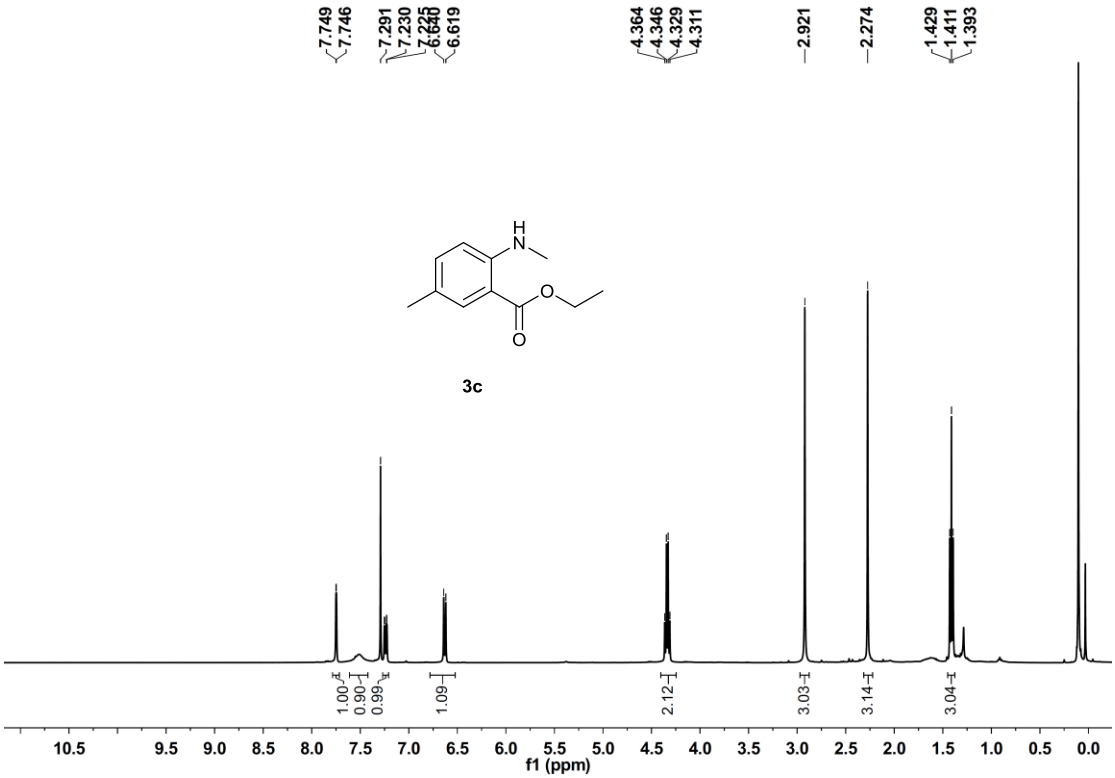
f1 (ppm)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4.346
4.329
4.311
2.921
2.274
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1.393
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0.99
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2.12
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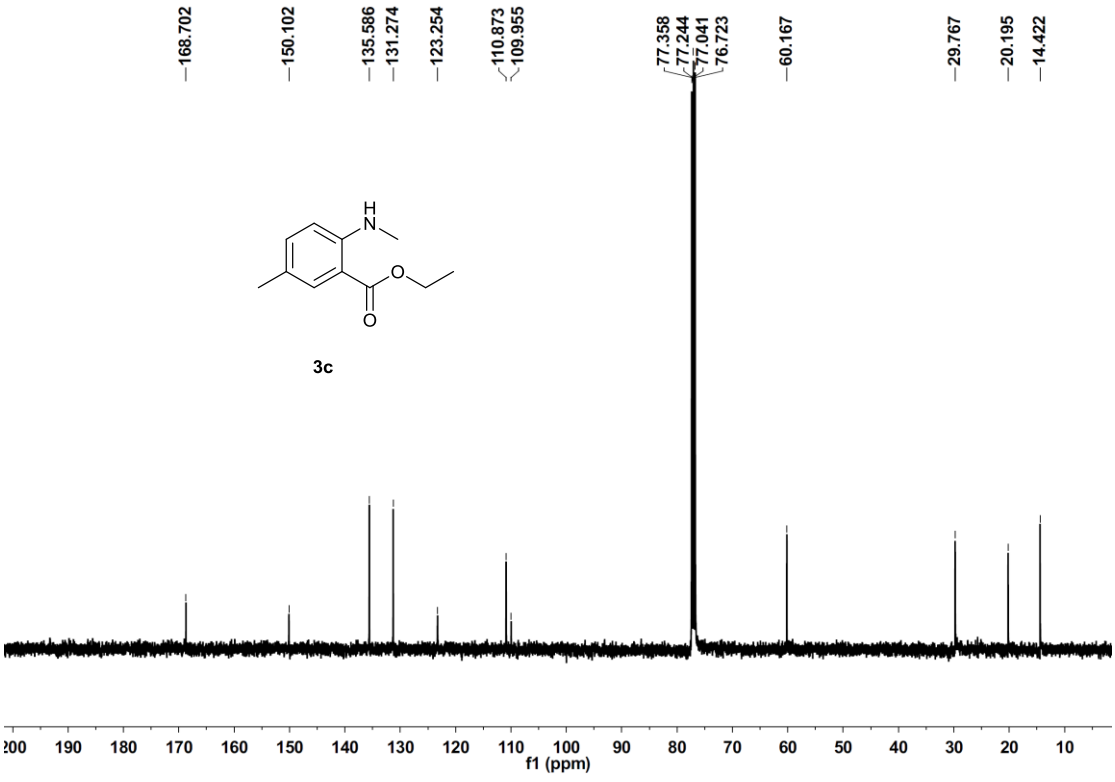
3.14
3.04
f1 (ppm)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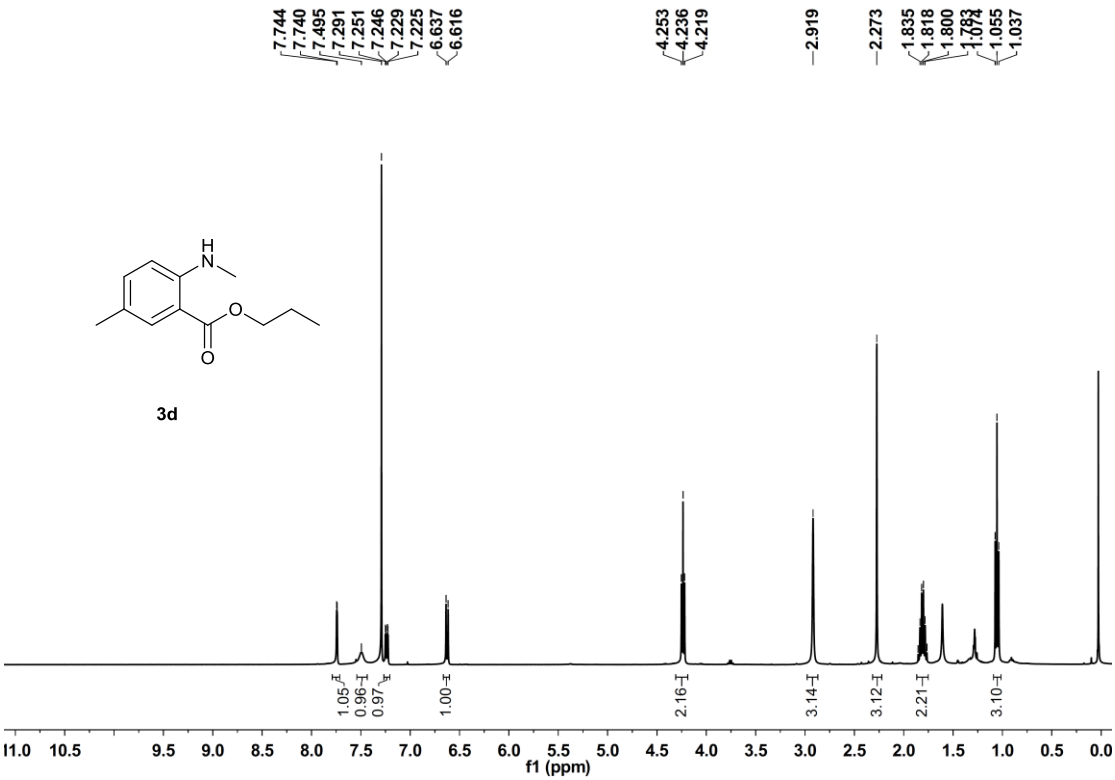
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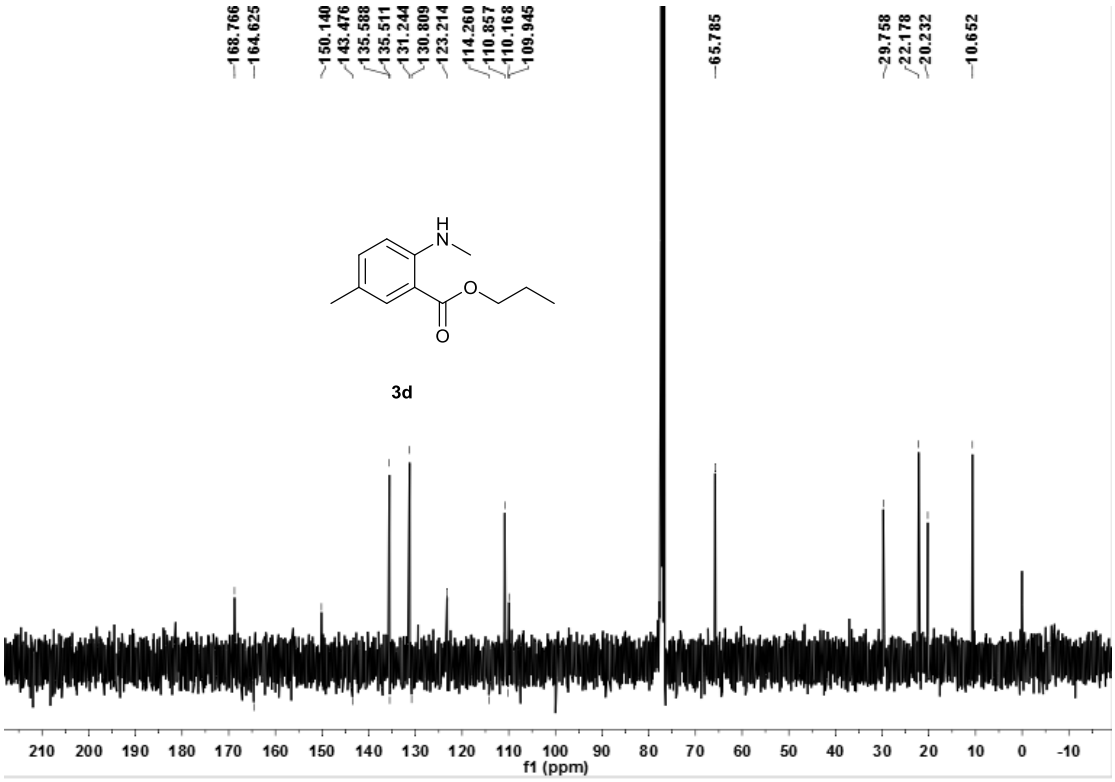
f1 (ppm)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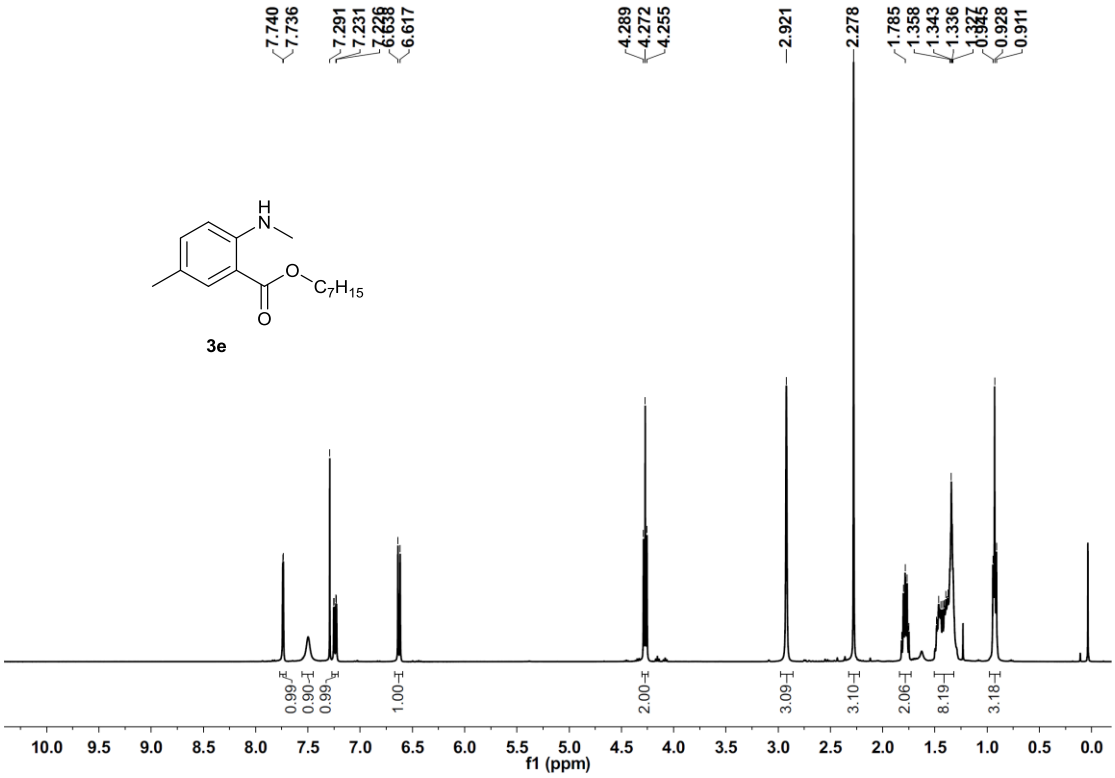
f1 (ppm)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0.911
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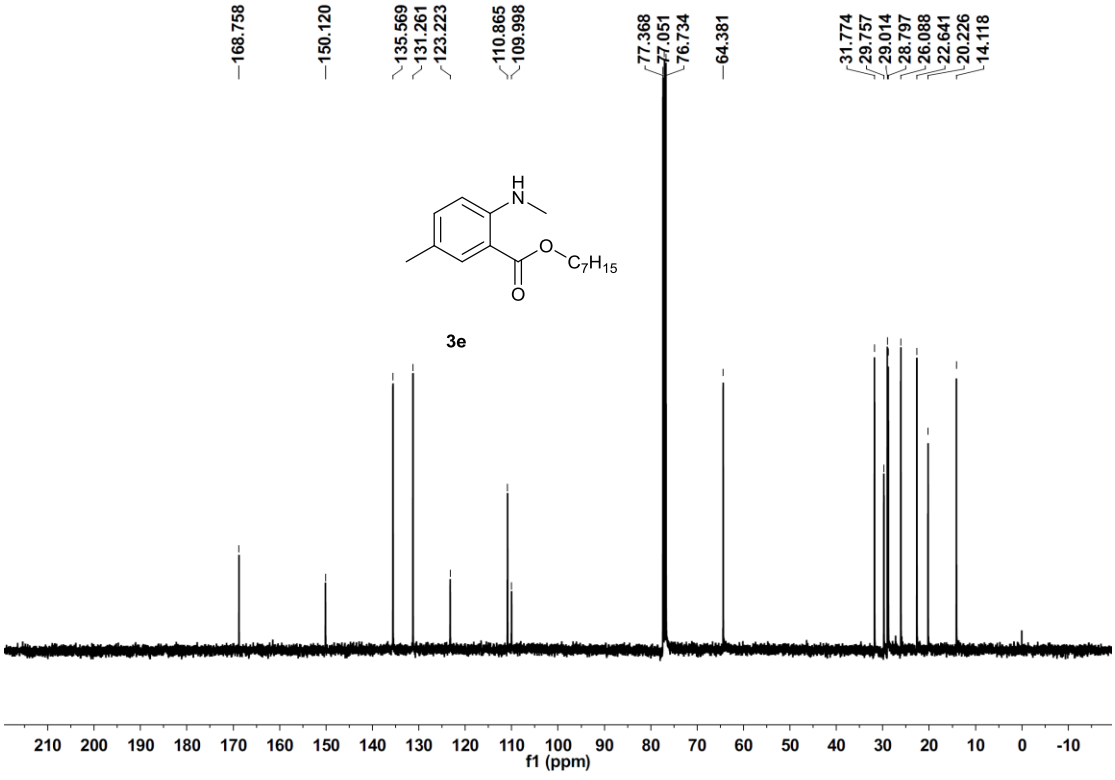
f1 (ppm)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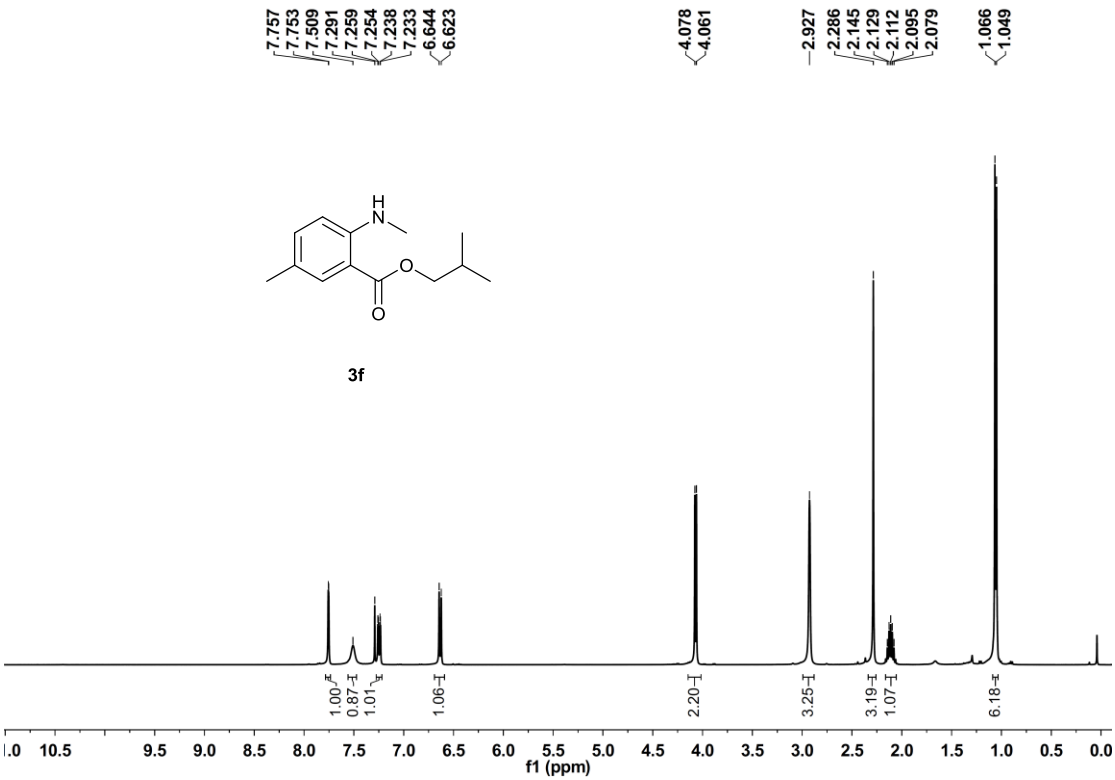
f1 (ppm)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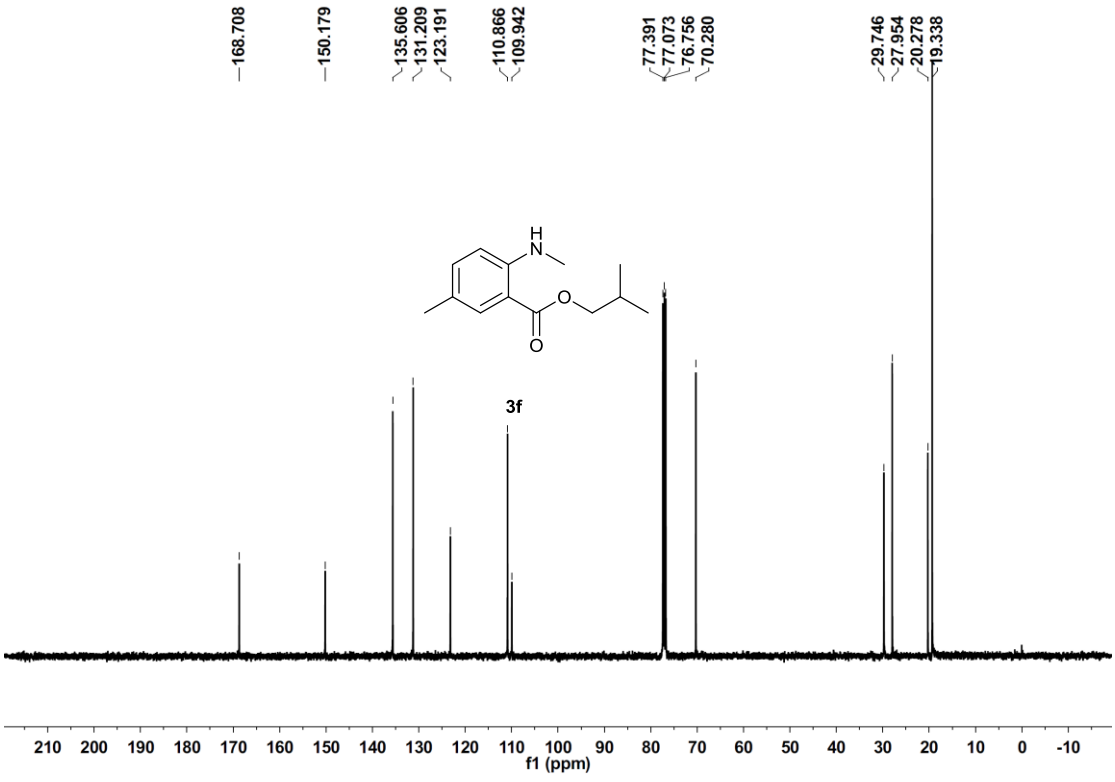
f1 (ppm)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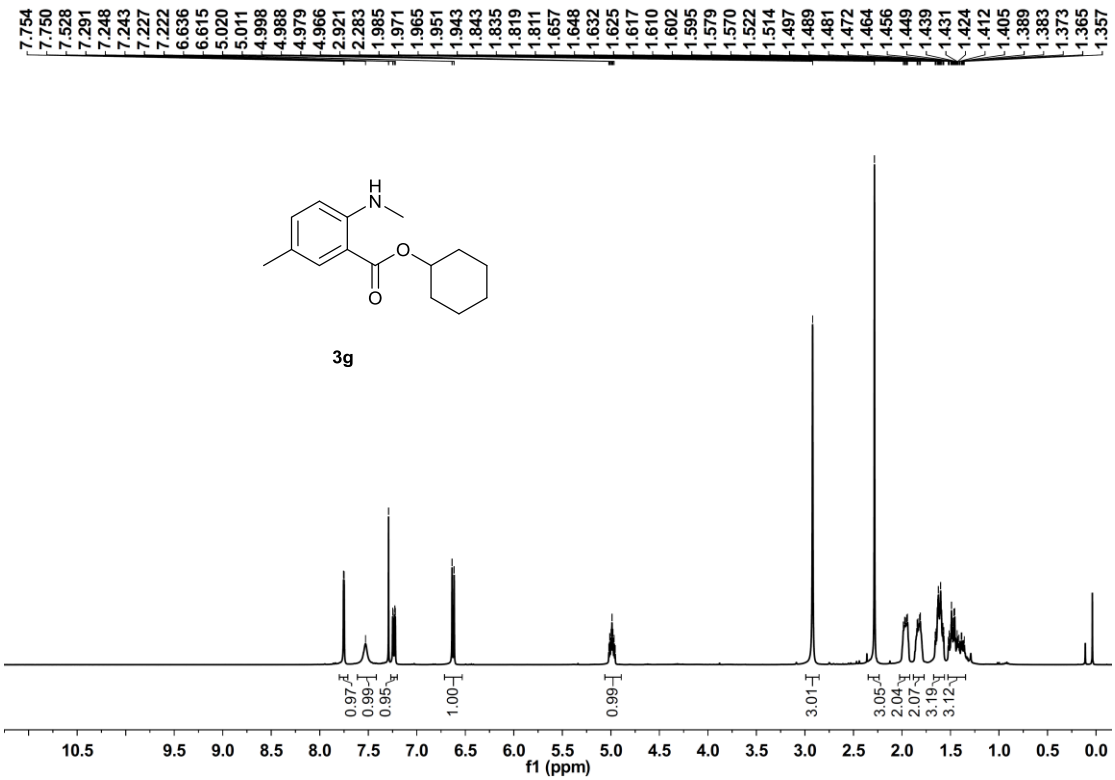
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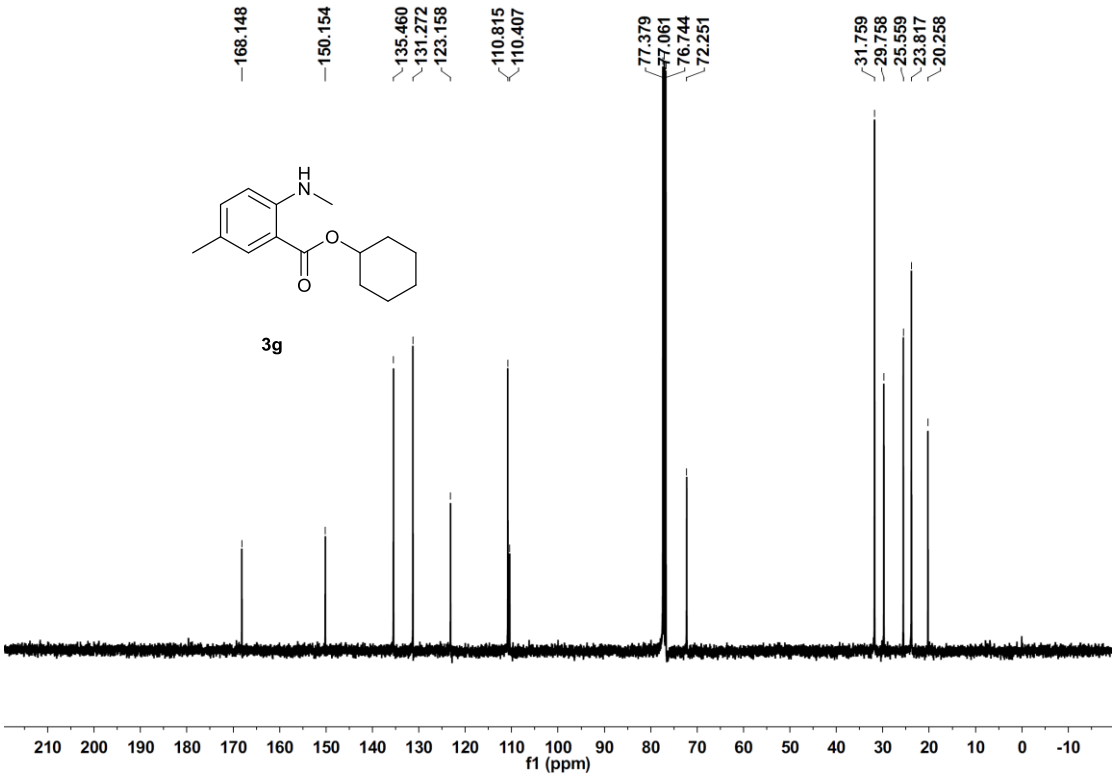
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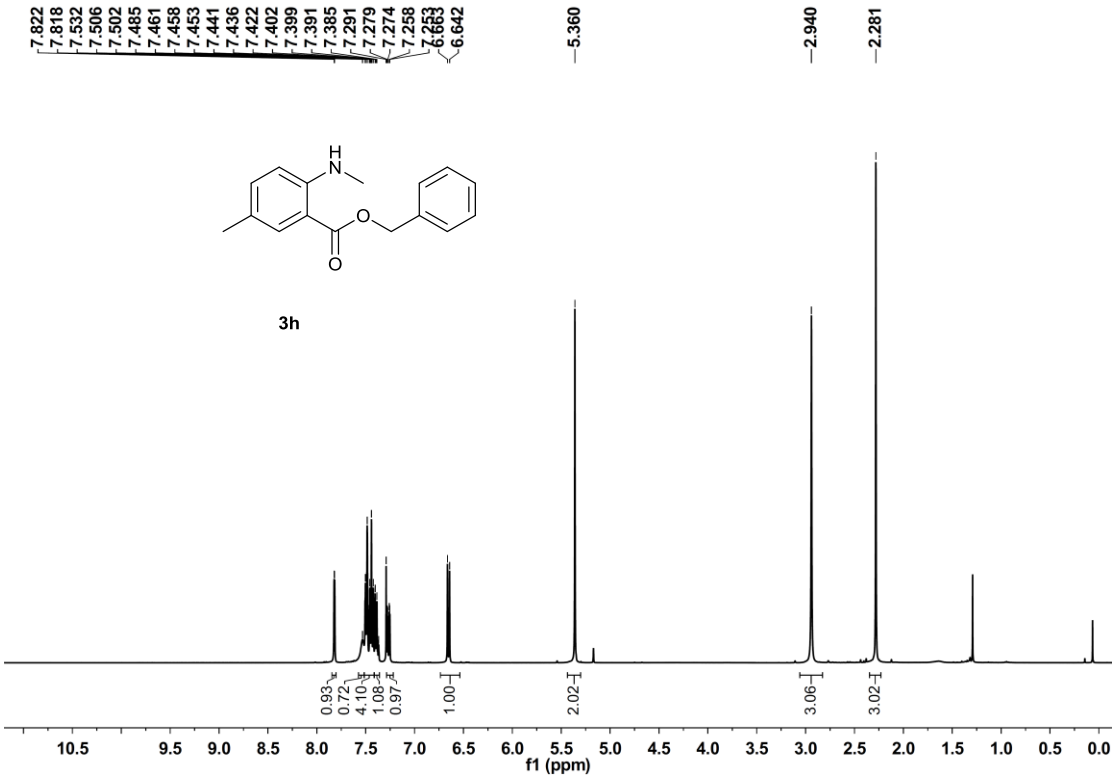
f1 (ppm)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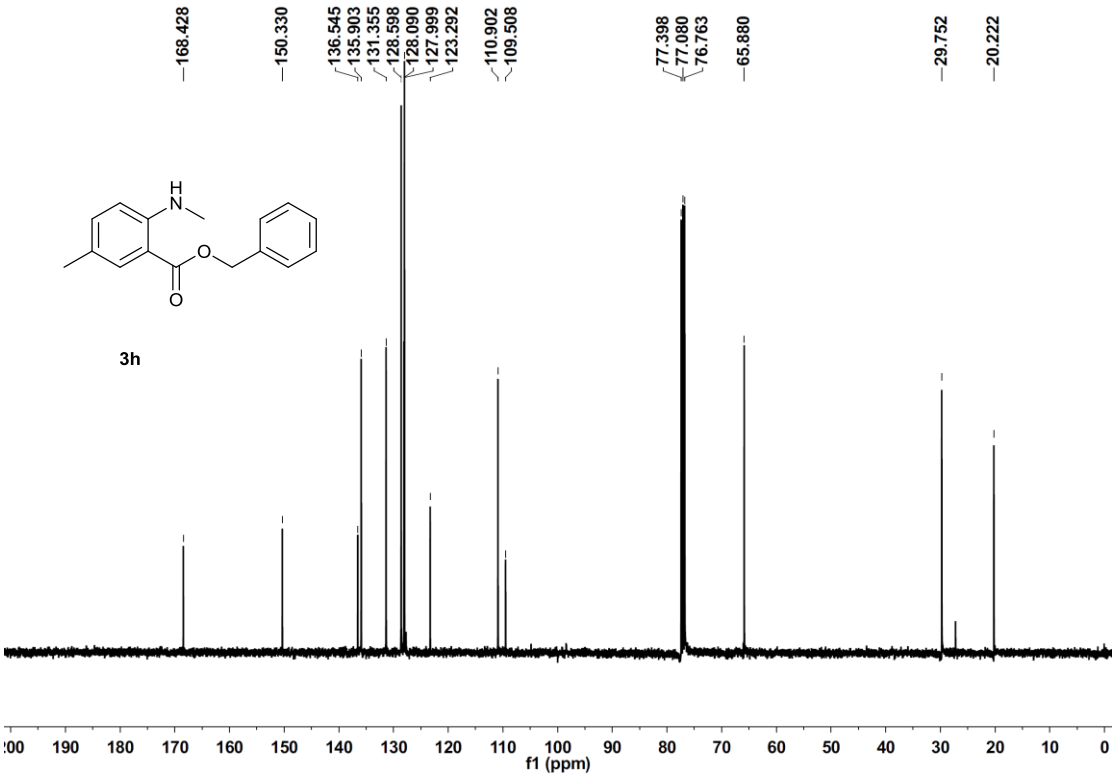
f1 (ppm)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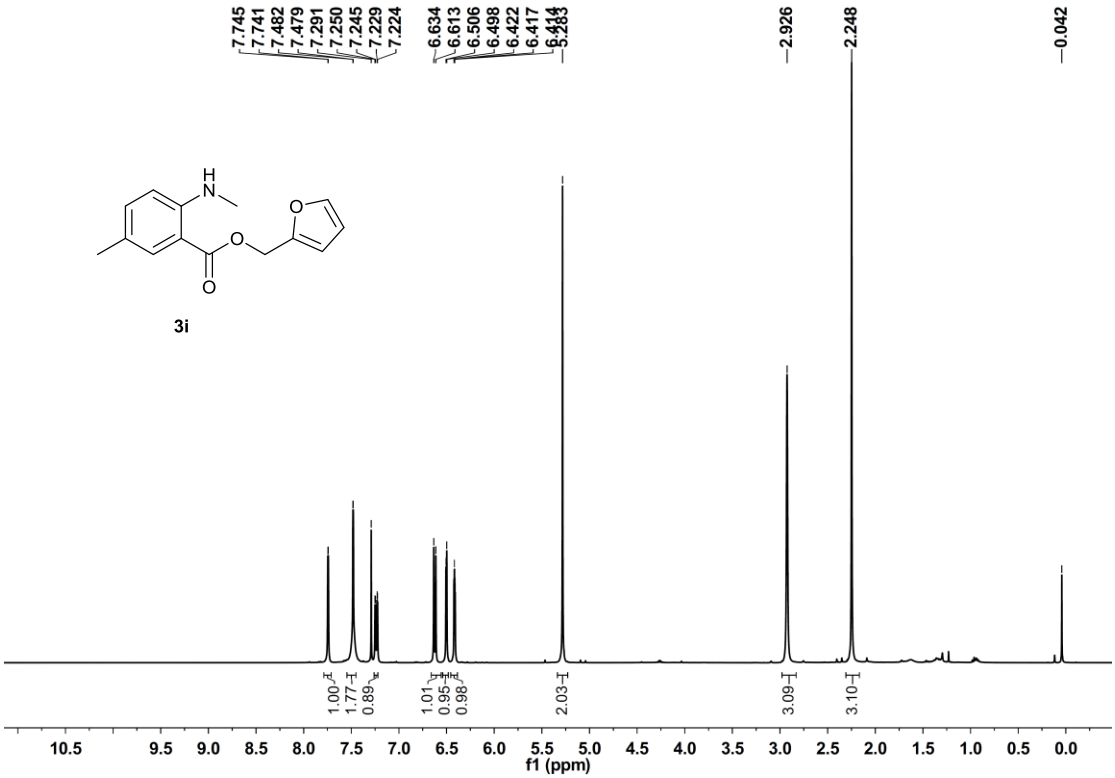
f1 (ppm)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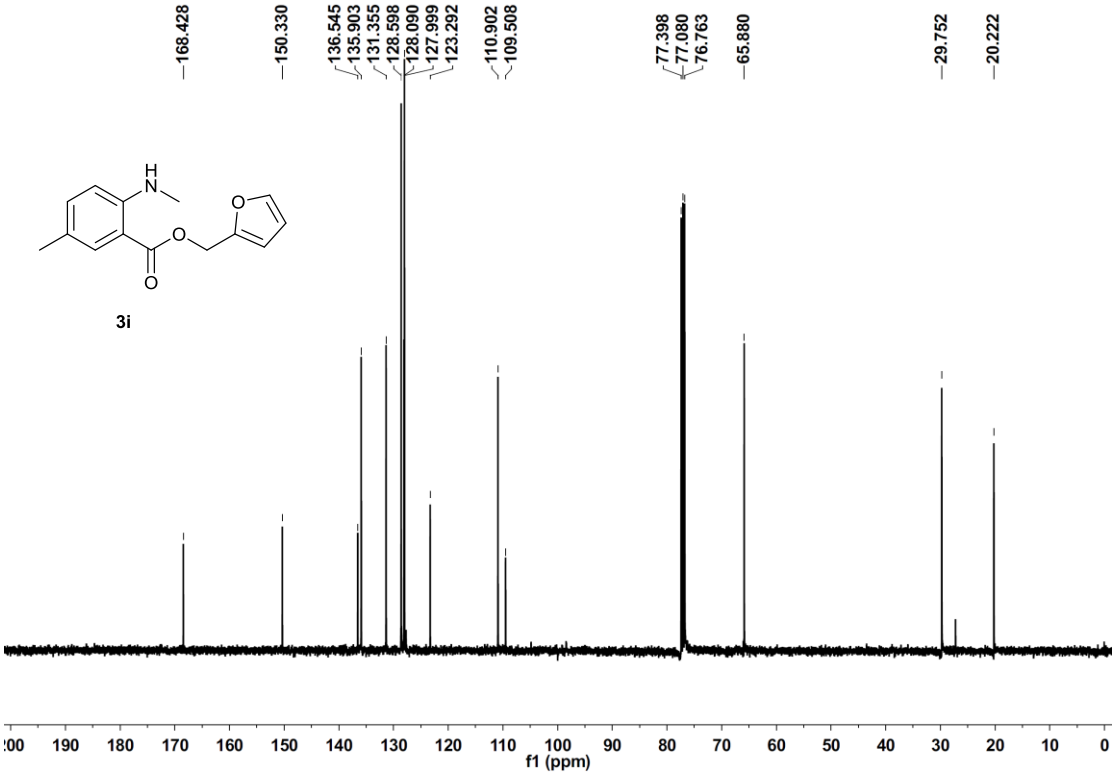
f1 (ppm)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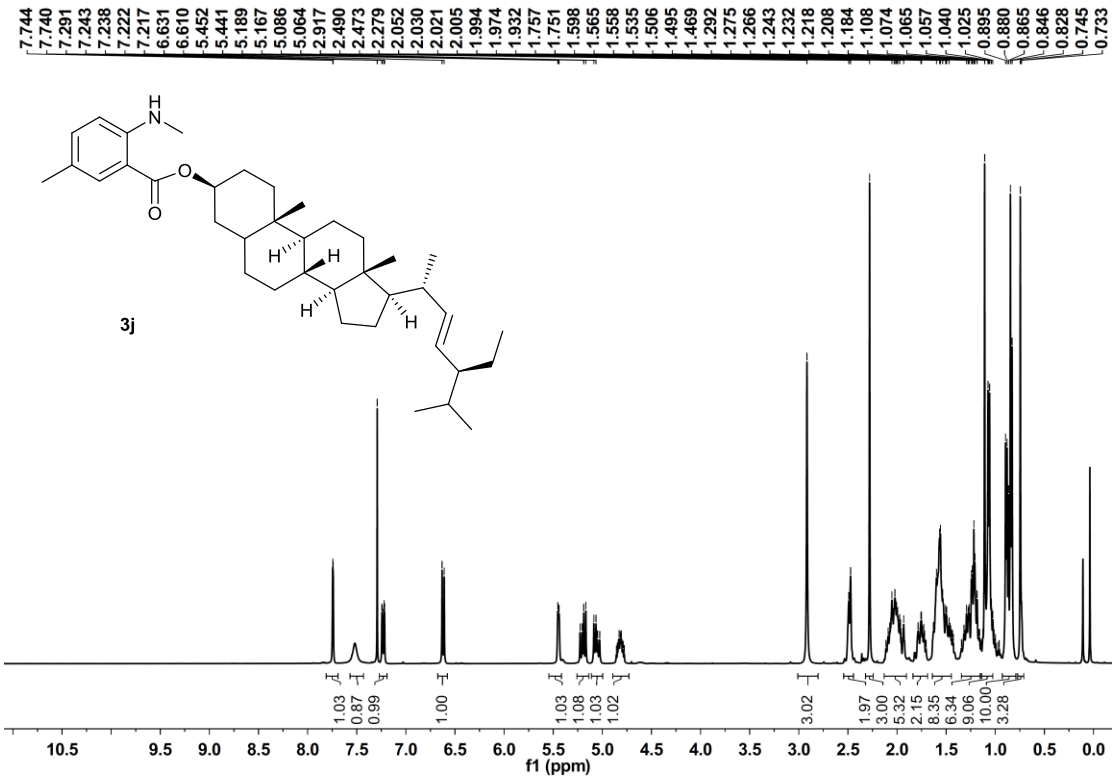
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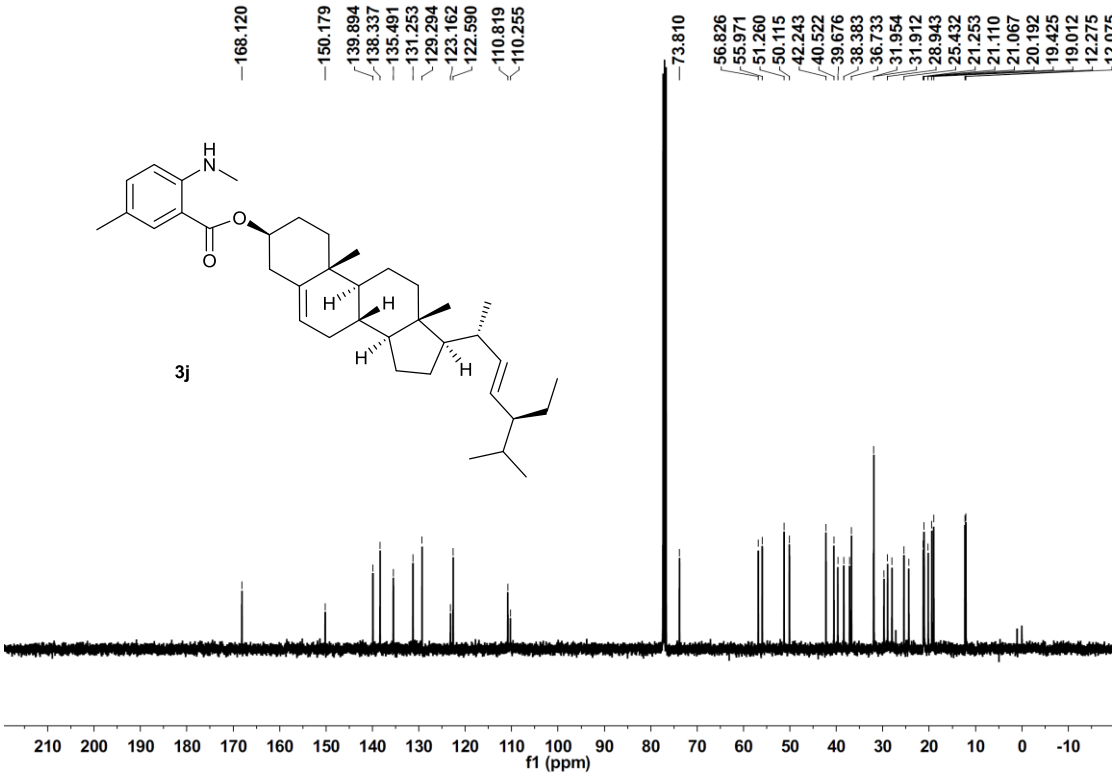
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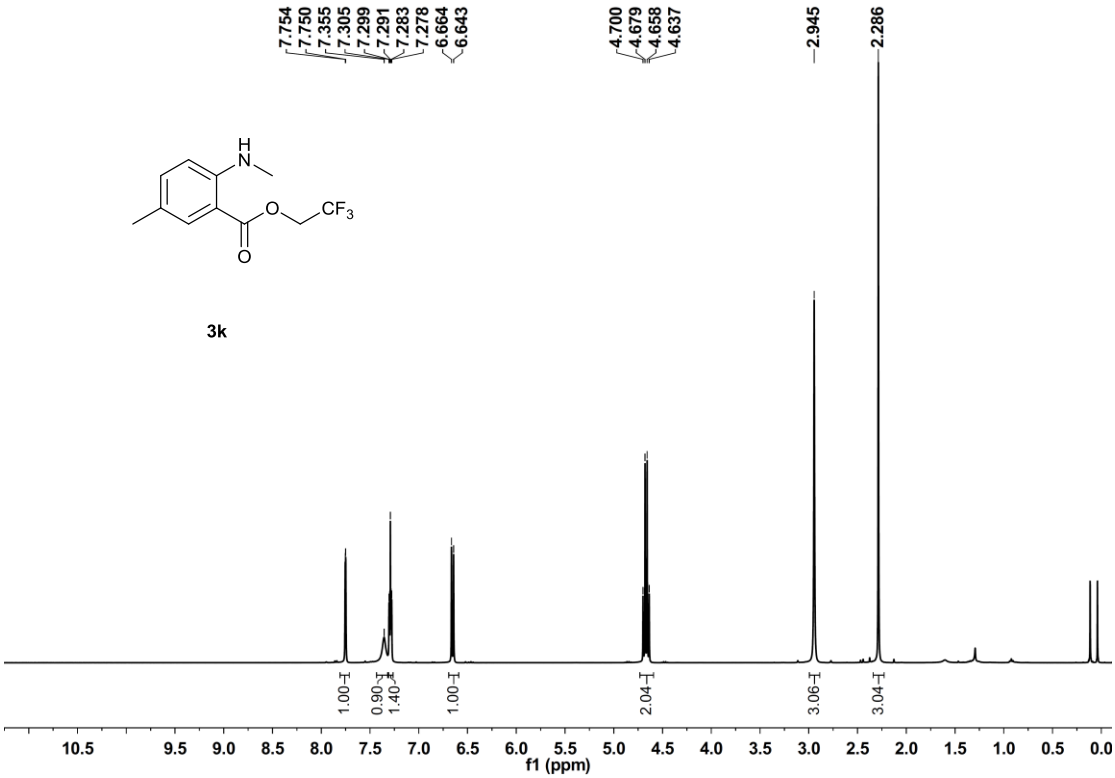
f1 (ppm)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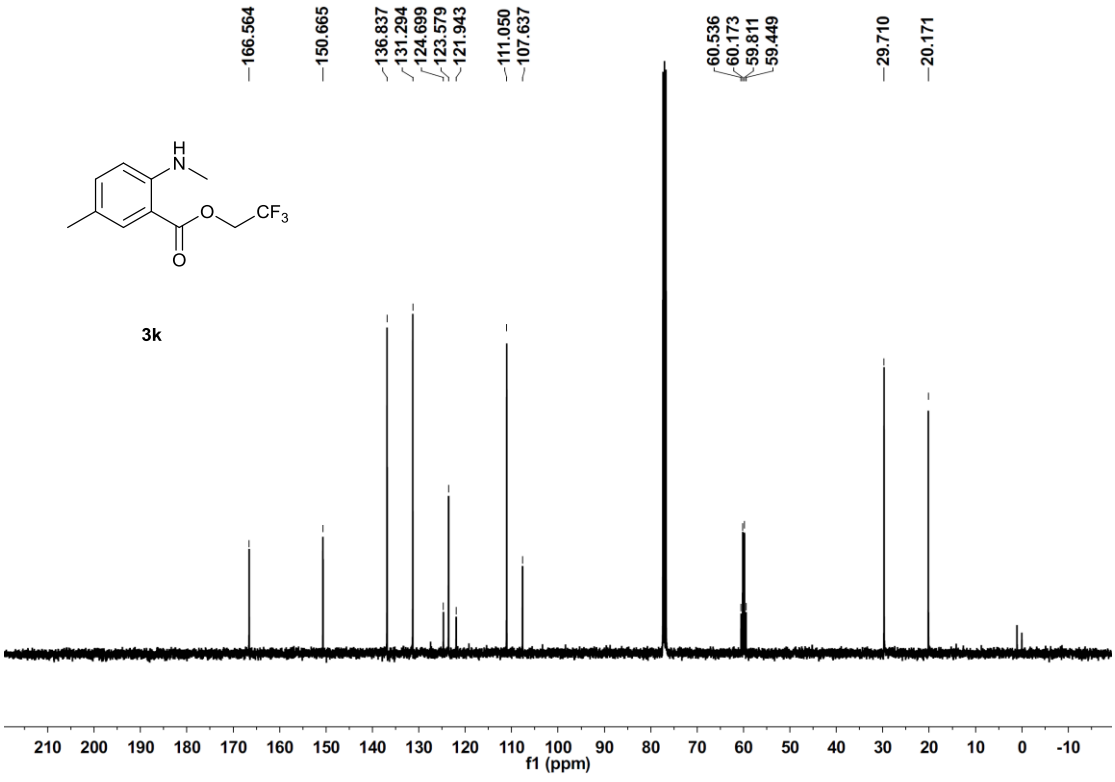
3.04
f1 (ppm)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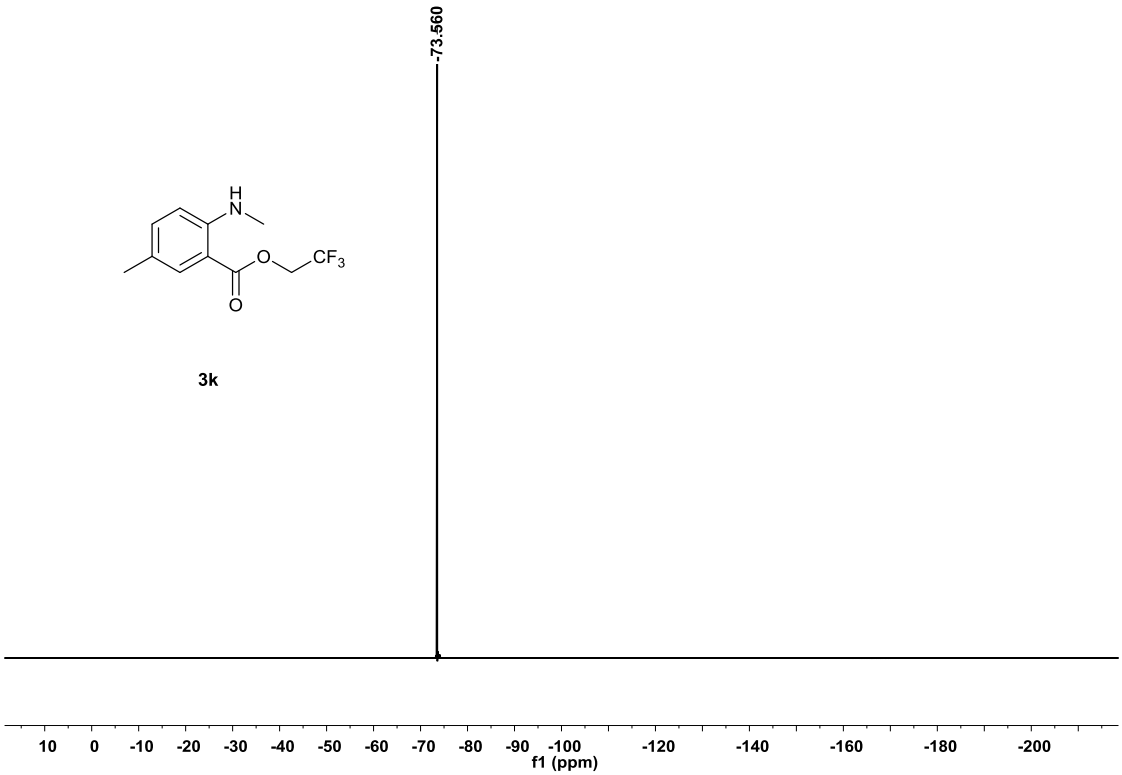
f1 (ppm)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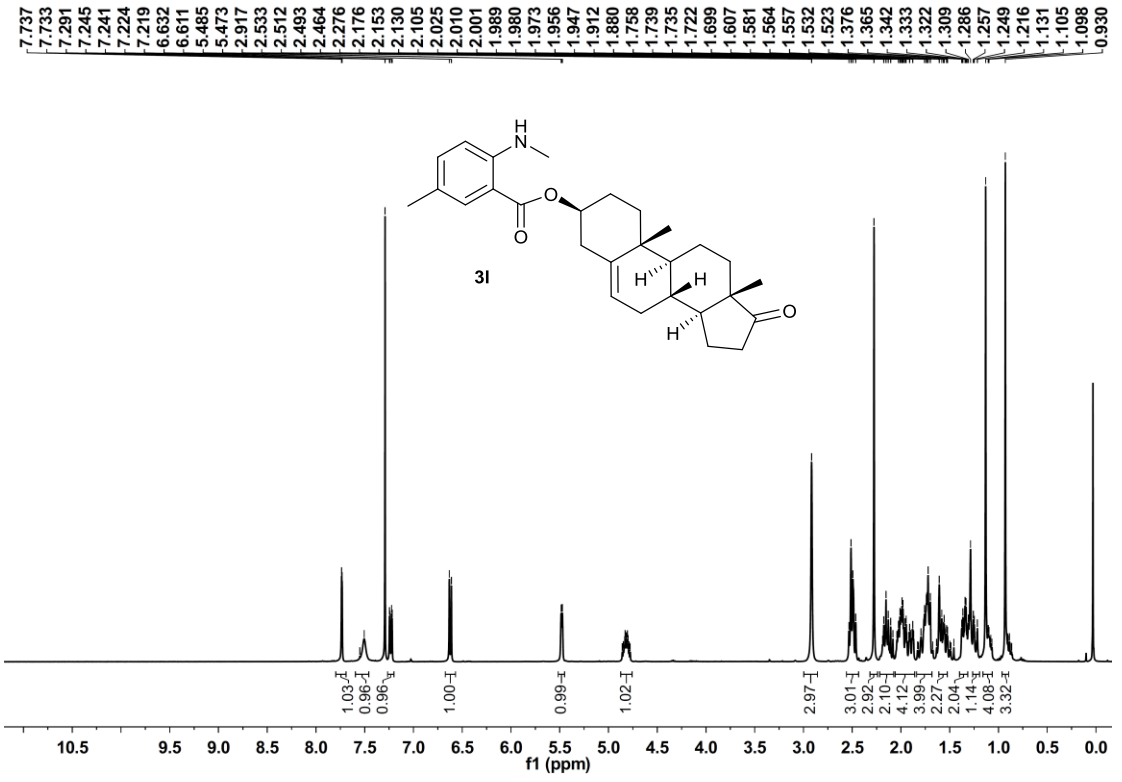
3k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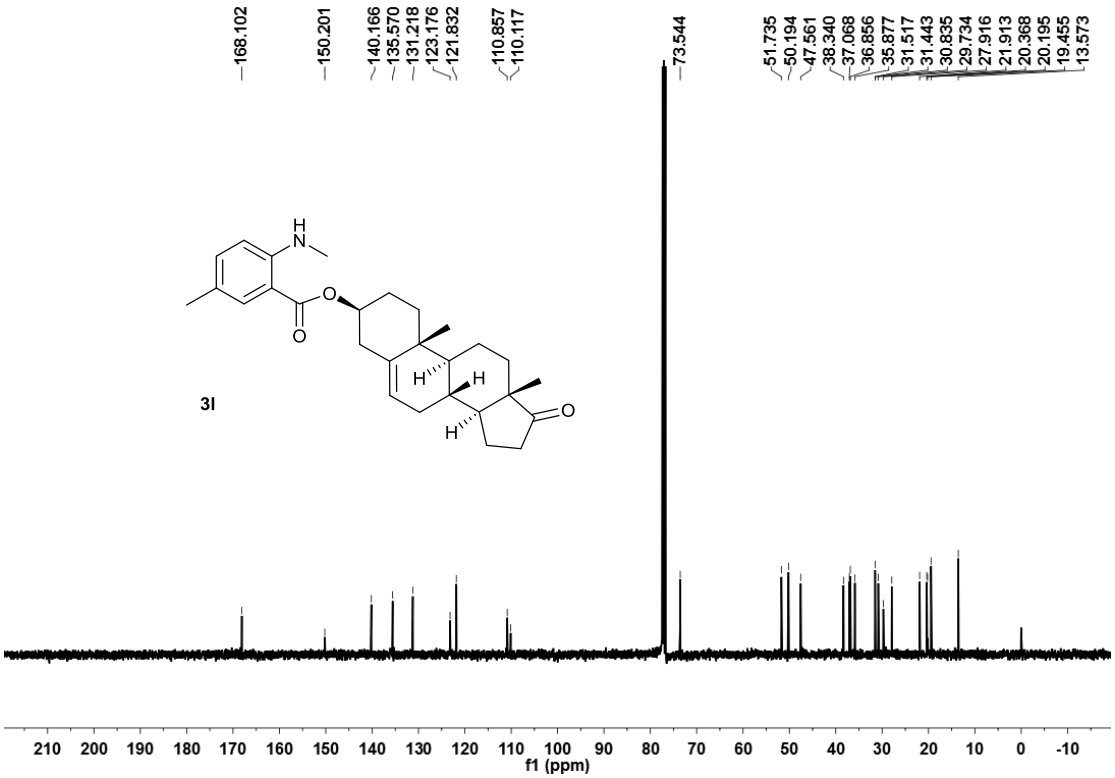
1.03 0.96 0.96 1.00 0.99 1.02 2.97 3.01 2.92 2.10 4.12 3.99 2.27 2.04 1.14 4.08 3.32
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

3l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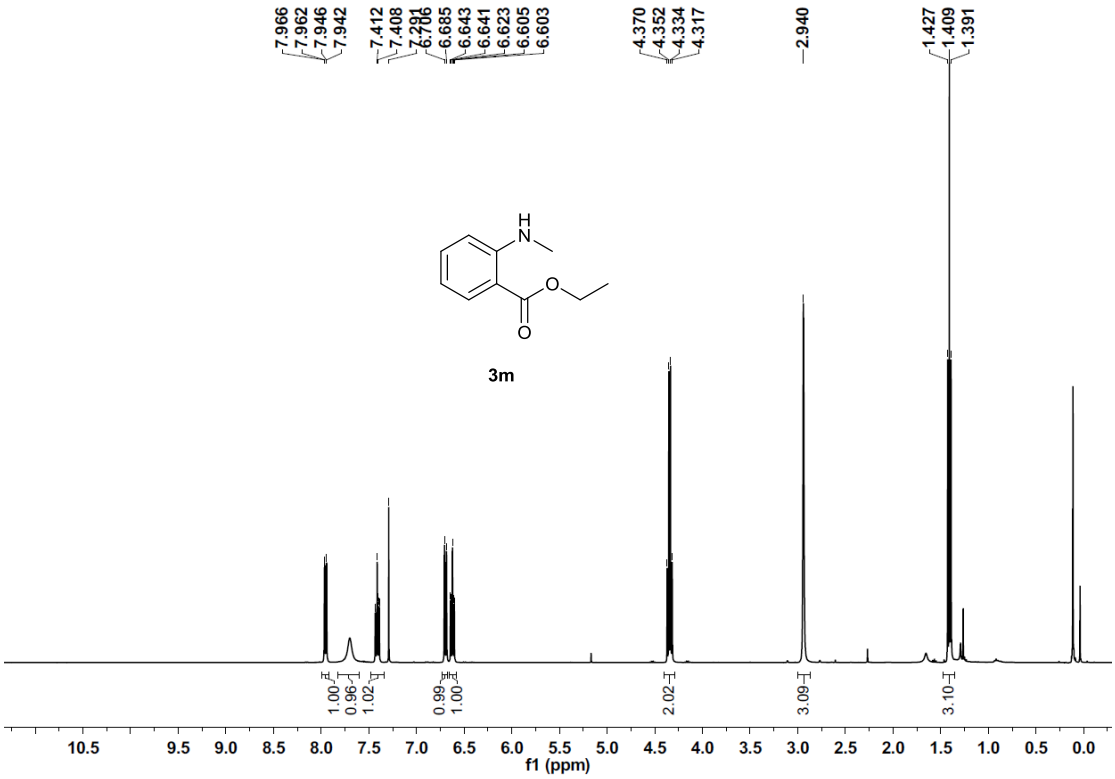
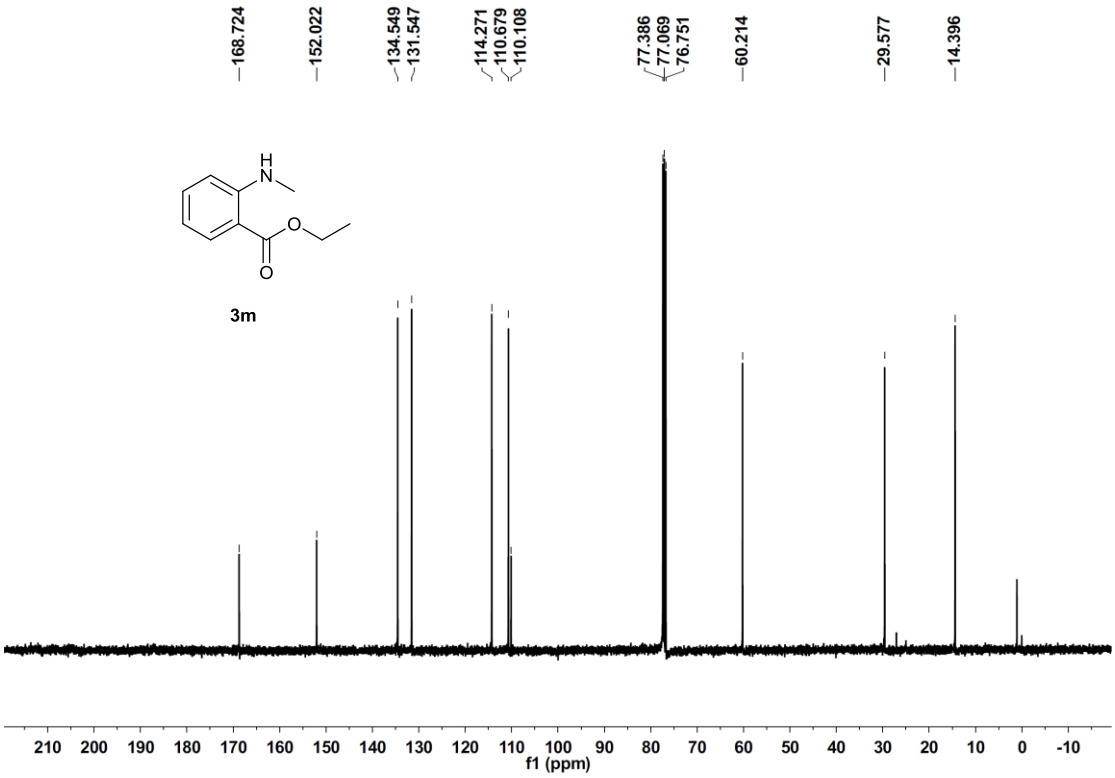
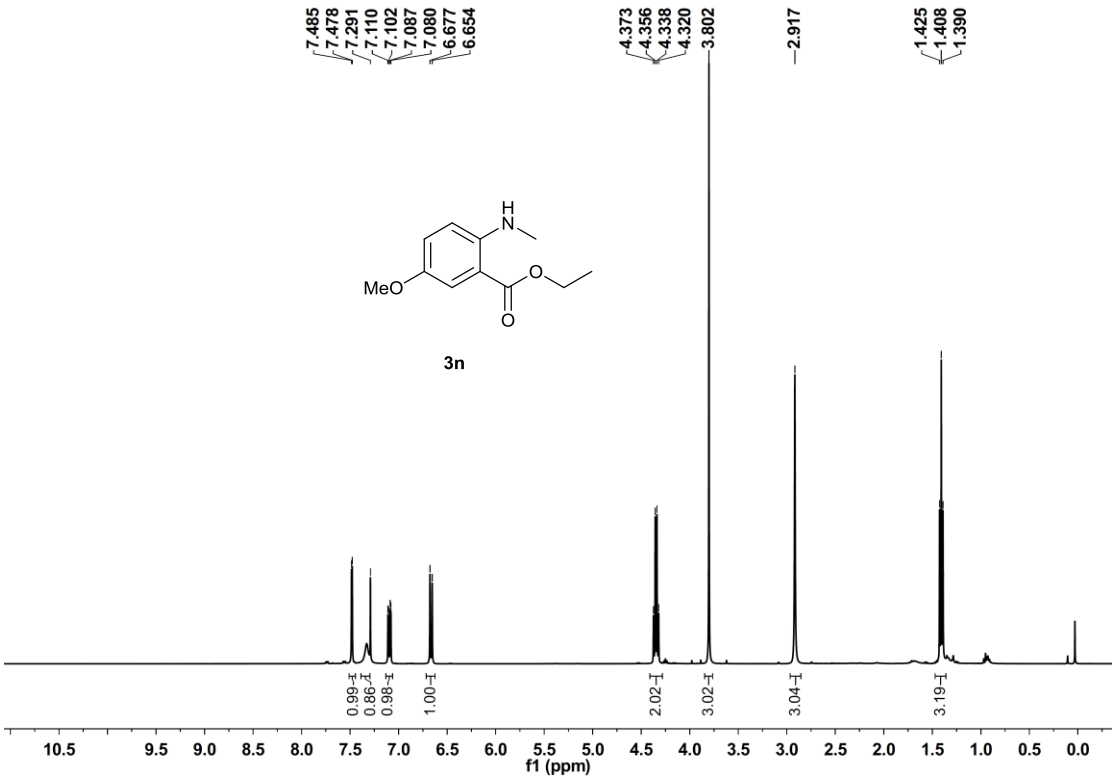
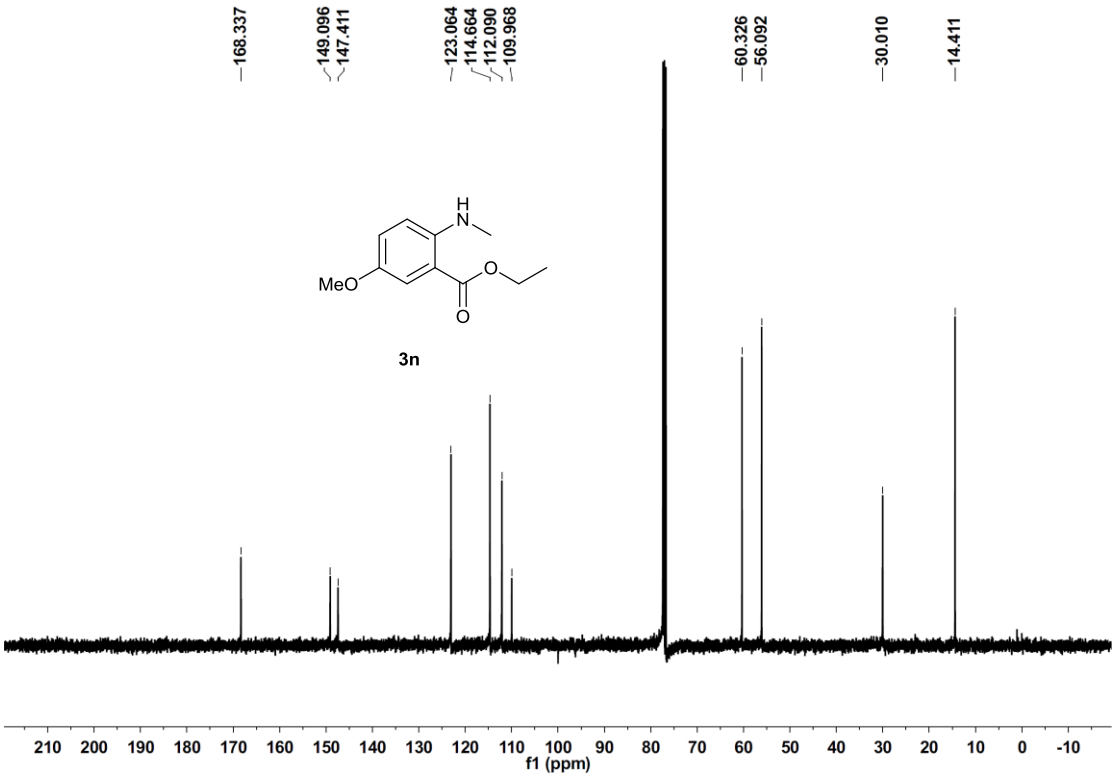
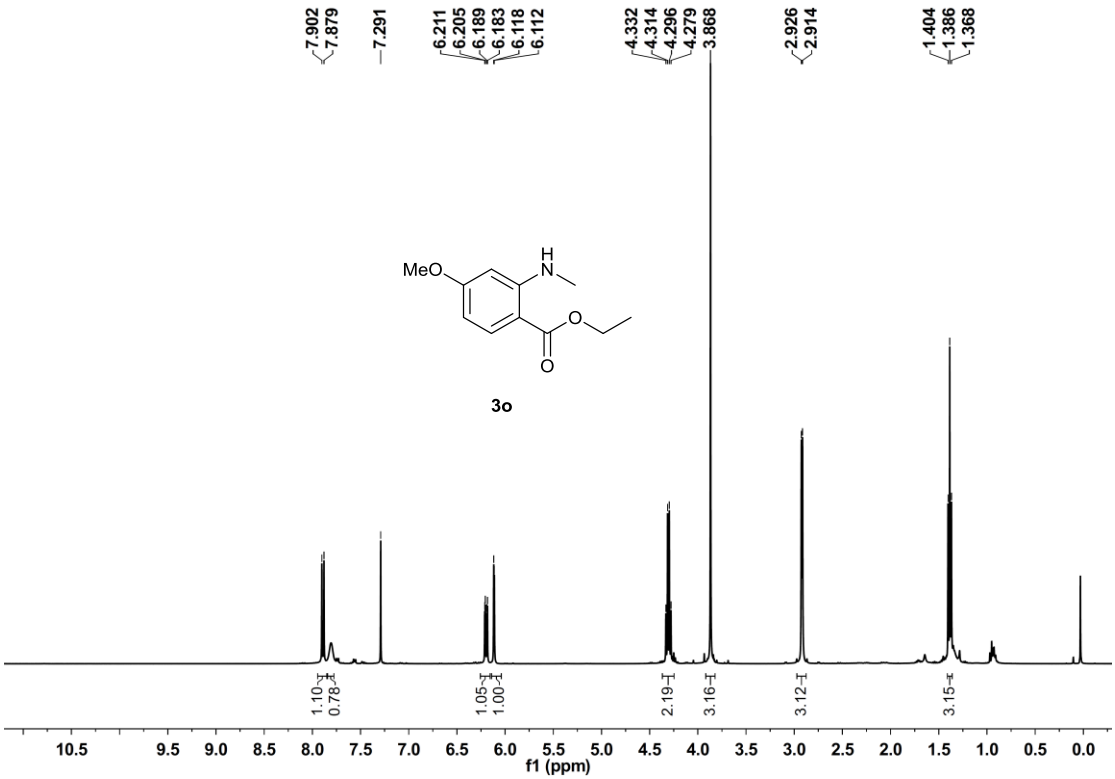
150.201
140.166
135.570
131.218
123.176
121.832
110.857
110.117
73.544
51.735
50.194
47.561
38.340
37.068
36.856
35.877
31.517
31.443
30.835
29.734
27.916
21.913
20.368
20.195
19.455
13.573
3l
f1 (ppm)

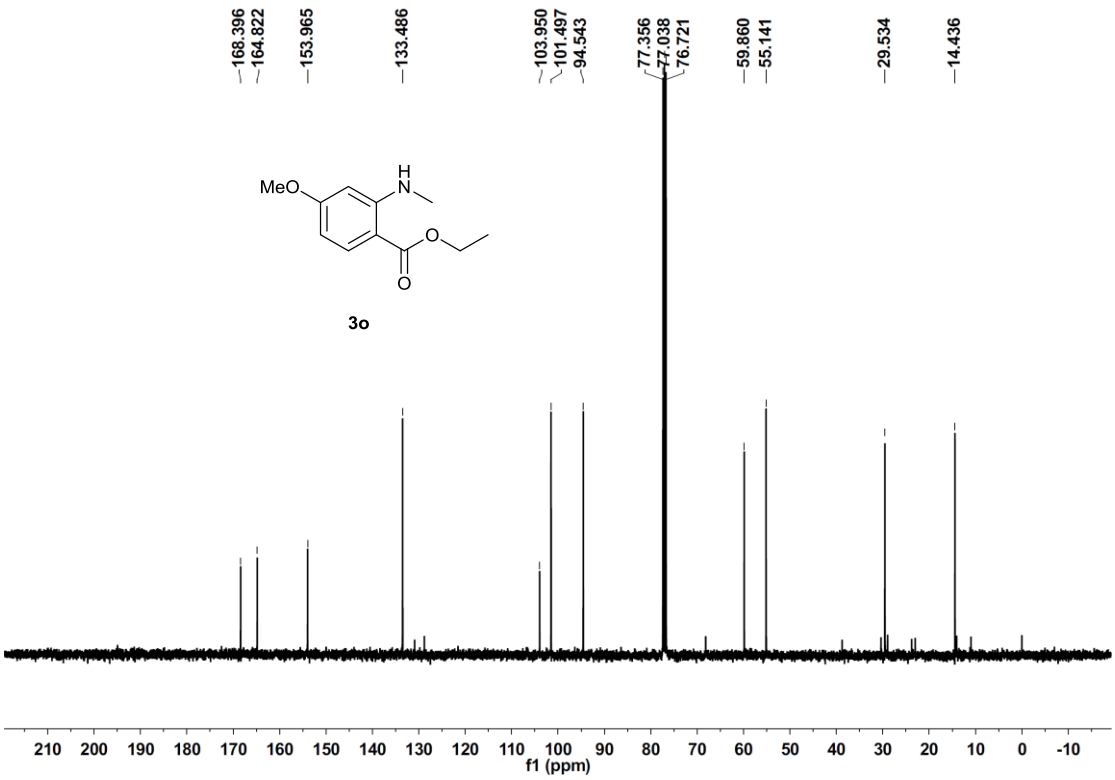

7.966
7.962
7.946
7.942
7.412
7.408
7.281
6.706
6.685
6.643
6.641
6.623
6.605
6.603
4.370
4.352
4.334
4.317
2.940
1.427
1.409
1.391
3m
1.00
0.96
1.02
0.99
1.00
2.02
3.09
3.10
f1 (ppm)

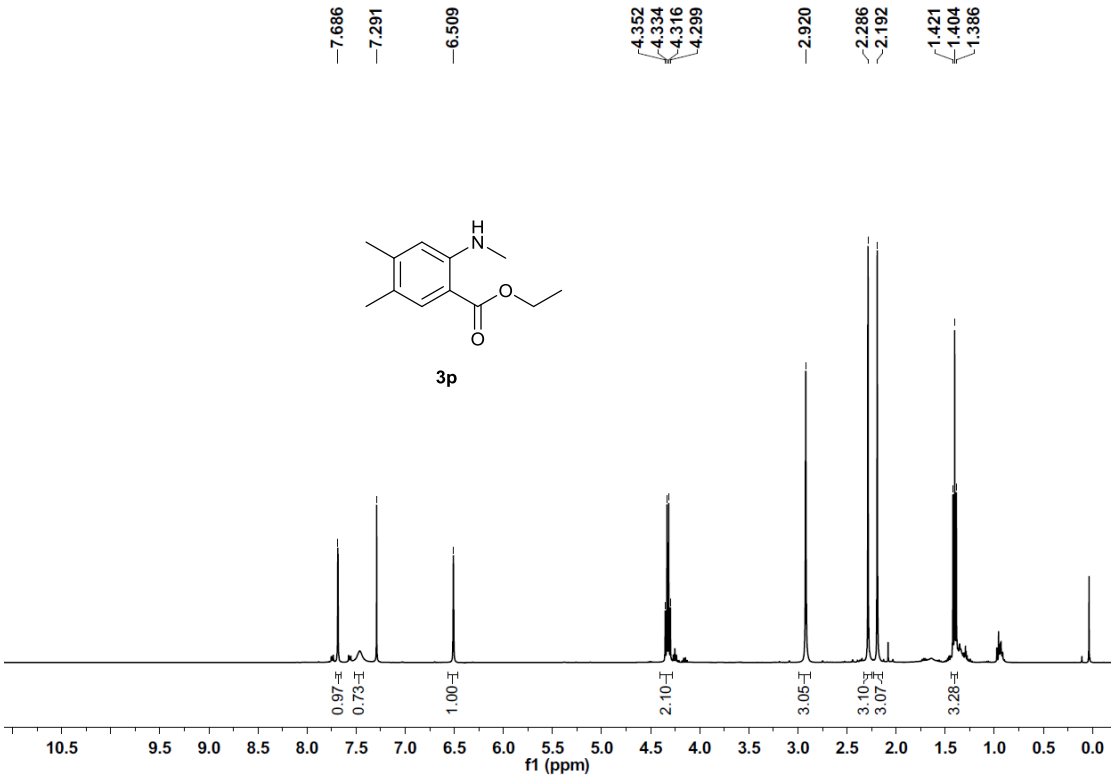

168.724
152.022
134.549
131.547
114.271
110.679
110.108
77.386
77.069
76.751
60.214
29.577
14.396
3m
f1 (ppm)

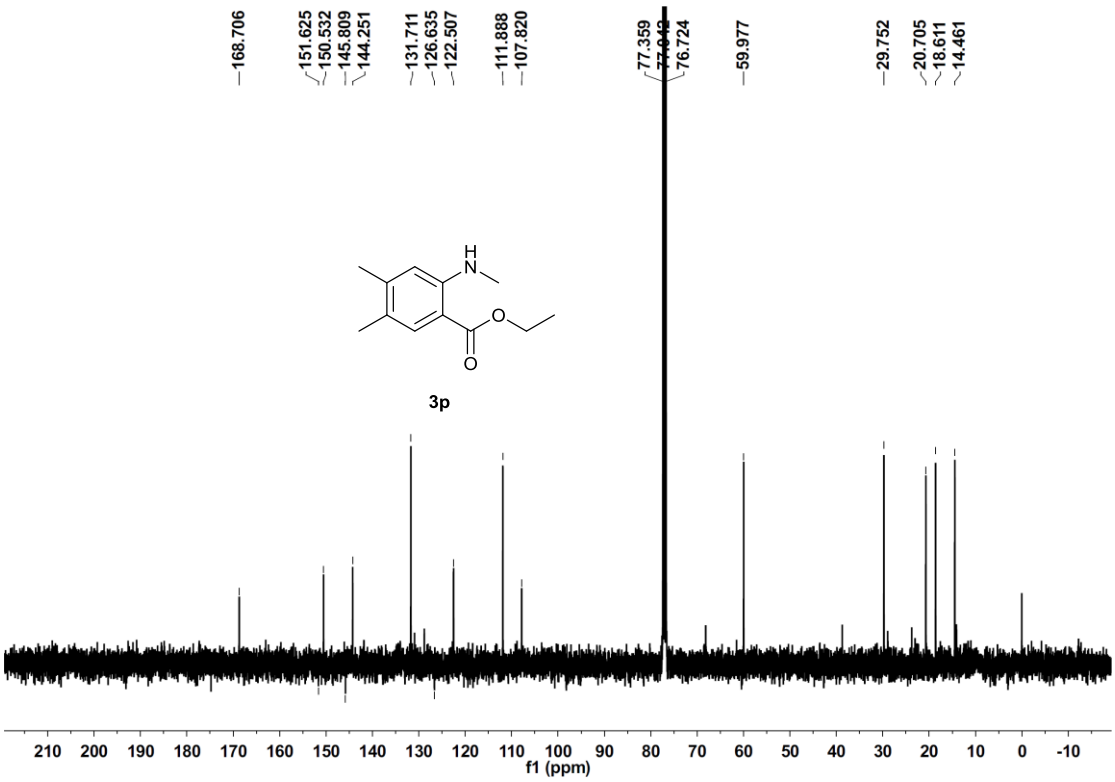

7.485
7.478
7.291
7.110
7.102
7.087
7.080
6.677
6.654
4.373
4.356
4.338
4.320
3.802
2.917
1.425
1.408
1.390
MeO
3n
0.99
0.86
0.98
1.00
2.02
3.02
3.04
3.19
f1 (ppm)

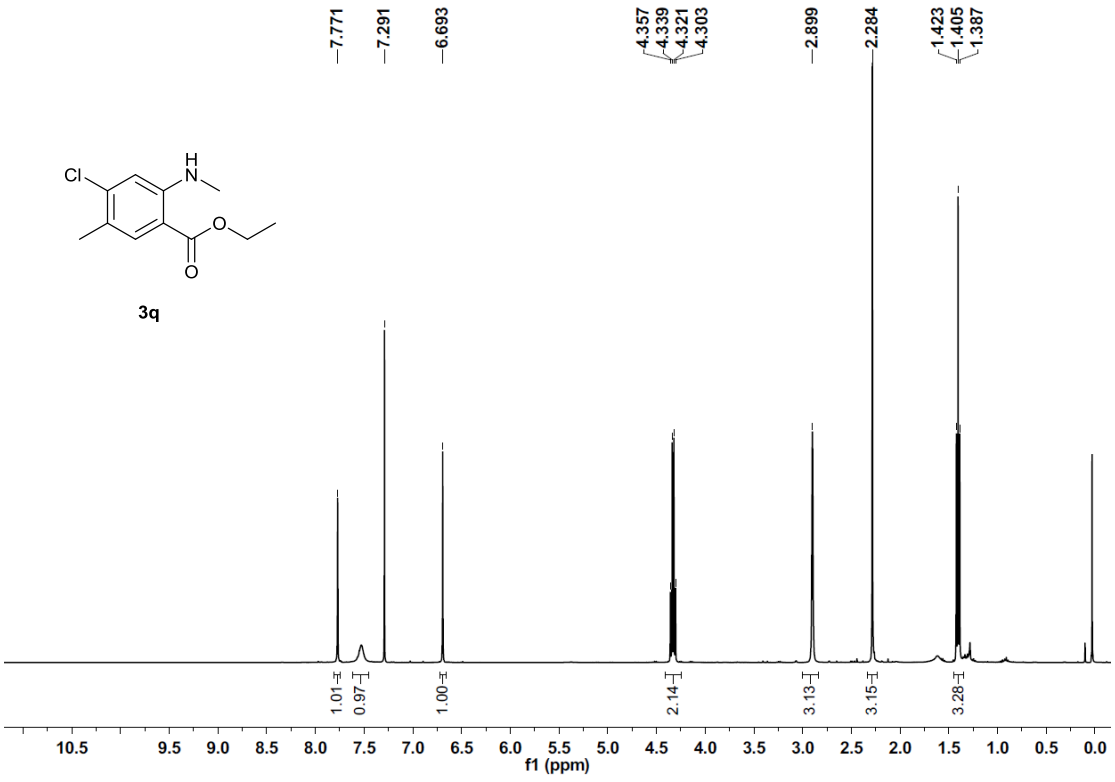

168.337
149.096
147.411
123.064
114.664
112.090
109.968
60.326
56.092
30.010
14.411
MeO
H
N
O
O
3n
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

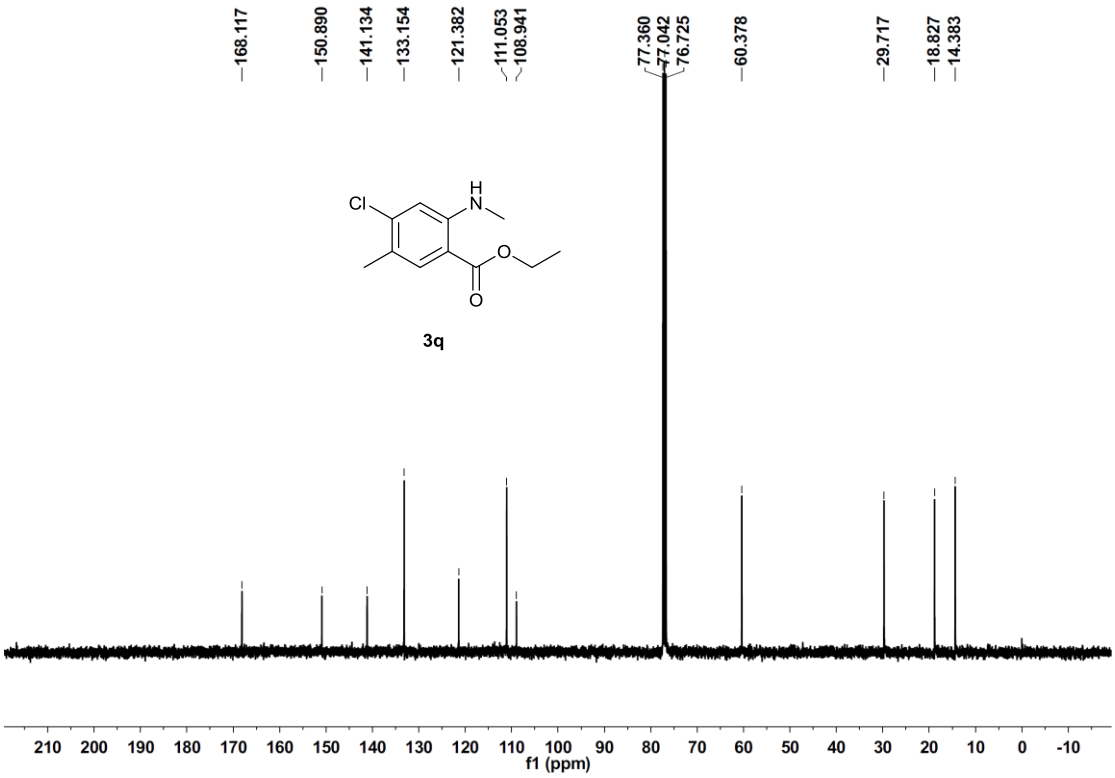

7.902
7.879
7.291
6.211
6.205
6.189
6.183
6.118
6.112
4.332
4.314
4.296
4.279
3.868
2.926
2.914
1.404
1.386
1.368
MeO
H
N
O
O
3o
1.10
0.78
1.05
1.00
2.19
3.16
3.12
3.15
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


168.396
164.822
153.965
133.486
103.950
101.497
94.543
77.356
77.038
76.721
59.860
55.141
29.534
14.436
MeO
H
N
O
O
3o
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)

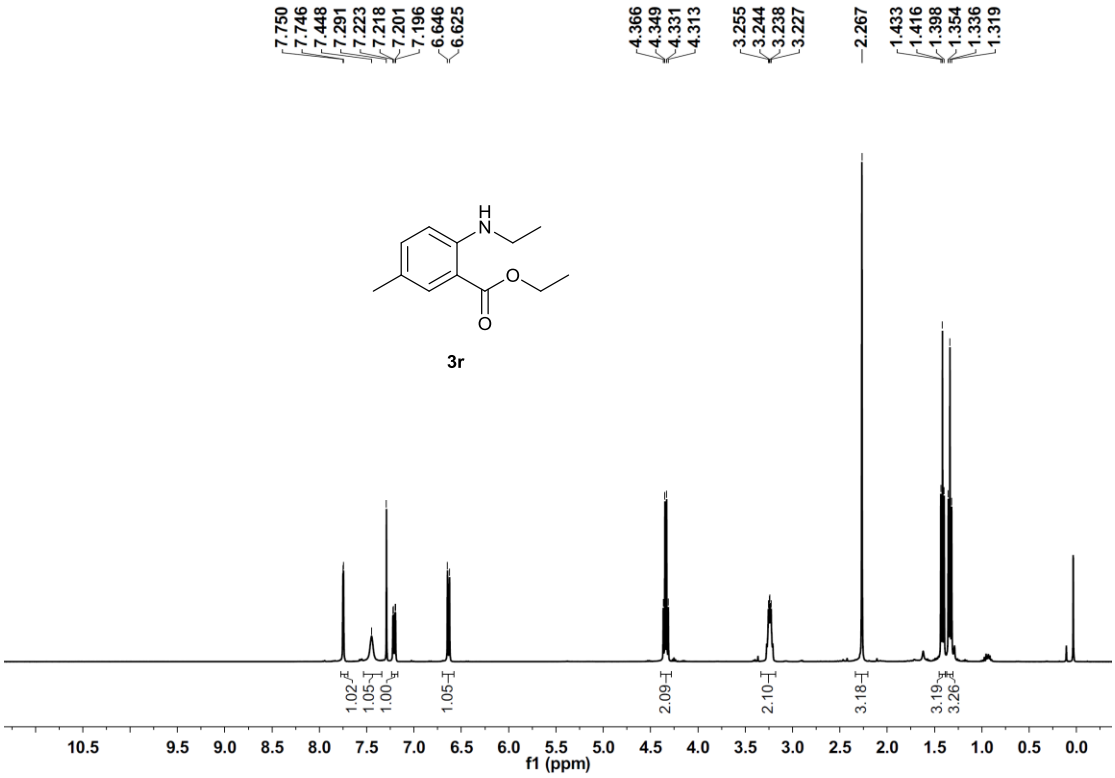

7.686
7.291
6.509
4.352
4.334
4.316
4.299
2.920
2.286
2.192
1.421
1.404
1.386
H
N
O
O
3p
0.97
0.73
1.00
2.10
3.05
3.10
3.07
3.28
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


168.706
151.625
150.532
145.809
144.251
131.711
126.635
122.507
111.888
107.820
77.359
76.724
59.977
29.752
20.705
18.611
14.461
3p
f1 (ppm)

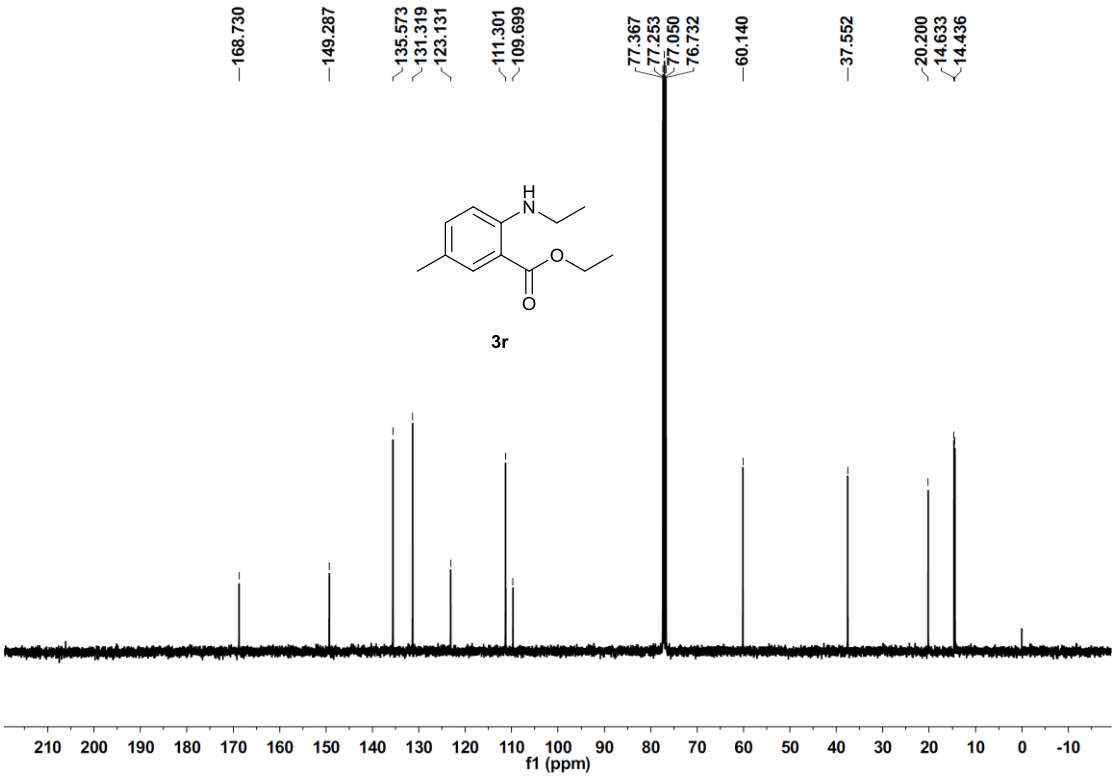

7.771
7.291
6.693
4.357
4.339
4.321
4.303
2.899
2.284
1.423
1.405
1.387
3q
1.01
0.97
1.00
2.14
3.13
3.15
3.28
f1 (ppm)

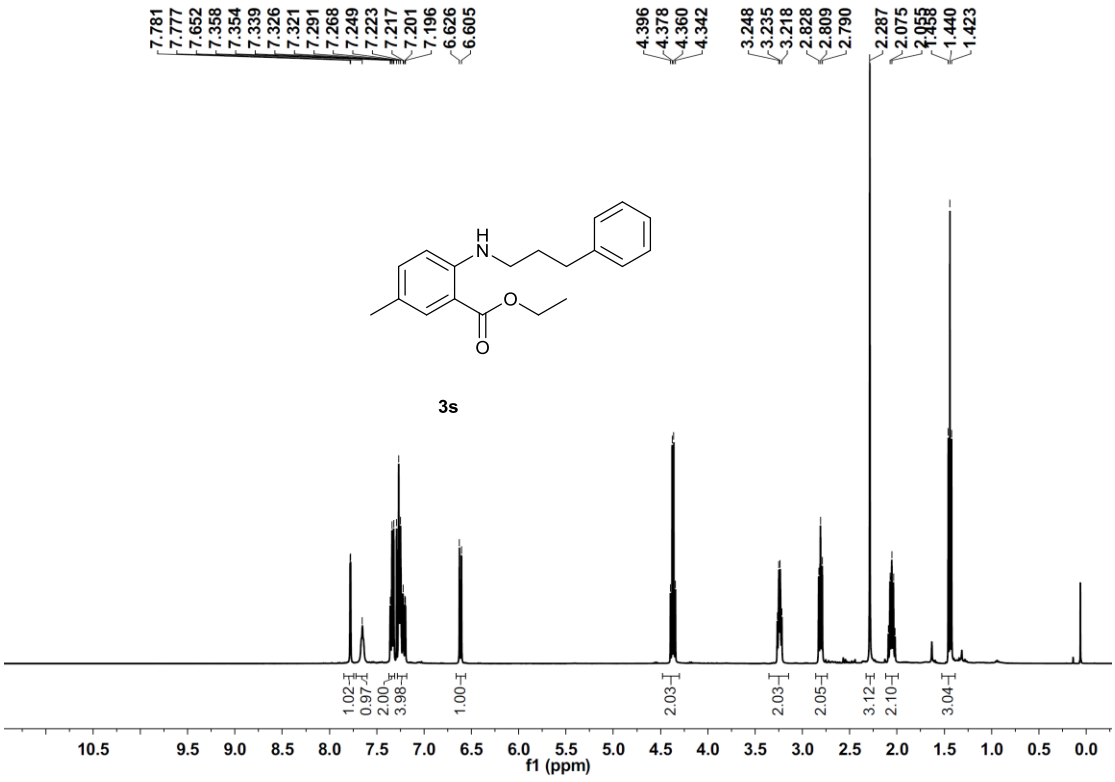

168.117
150.890
141.134
133.154
121.382
111.053
108.941
77.360
77.042
76.725
60.378
29.717
18.827
14.383
Cl
H
N
O
O
f1 (ppm)

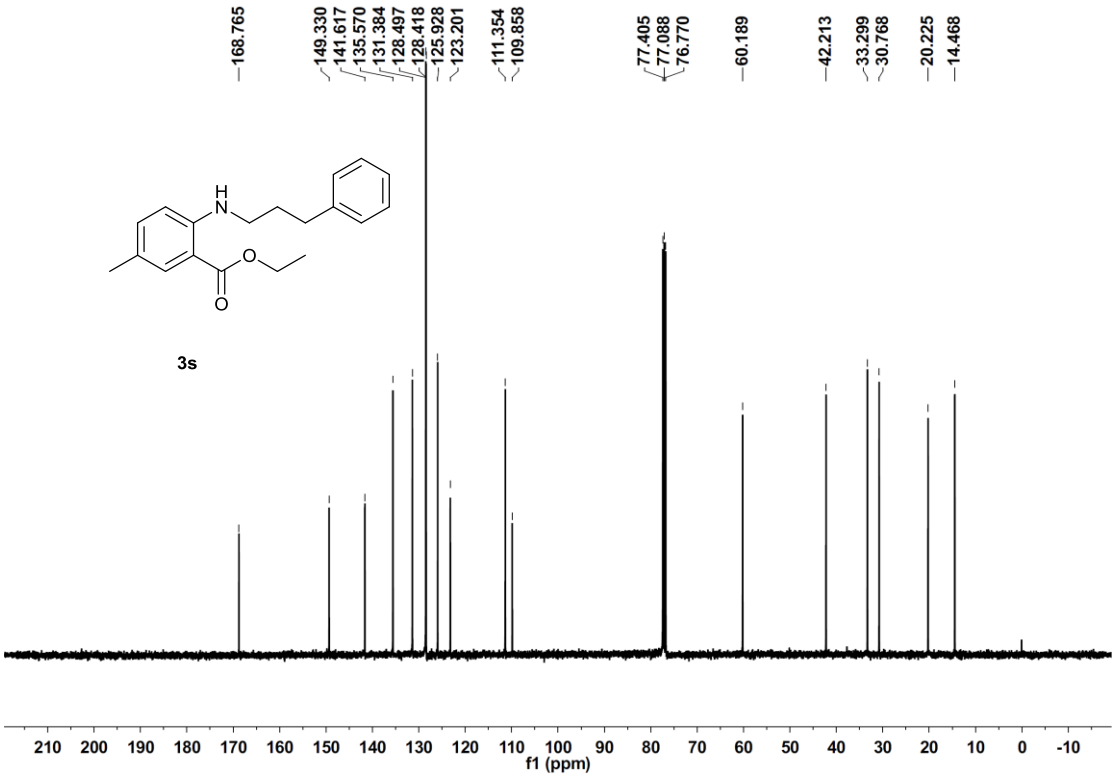
3q


7.750
7.746
7.448
7.291
7.223
7.218
7.201
7.196
6.646
6.625
4.366
4.349
4.331
4.313
3.255
3.244
3.238
3.227
2.267
1.433
1.416
1.398
1.354
1.336
1.319
H
N
O
O
1.02
1.05
1.00
1.05
2.09
2.10
3.18
3.19
3.26
f1 (ppm)

3r


168.730
149.287
135.573
131.319
123.131
111.301
109.699
77.367
77.253
77.050
76.732
60.140
37.552
20.200
14.633
14.436
H
N
O
O
3r
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)


7.781
7.777
7.652
7.358
7.354
7.339
7.326
7.321
7.291
7.268
7.249
7.223
7.217
7.201
7.196
6.626
6.605
4.396
4.378
4.360
4.342
3.248
3.235
3.218
2.828
2.809
2.790
2.287
2.075
2.055
1.458
1.440
1.423
H
N
O
O
3s
1.02
0.97
2.00
3.98
1.00
2.03
2.03
2.05
3.12
2.10
3.04
10.5 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)



Reference

1. T. W. Liwosz, S. R. Chemler, *J. Am. Chem. Soc.* **2012**, *134*, 2020-2023.